Some Simple Fyptamines second edition

Keeper of the Frout Friends

Mydriatic Productions A Better Days ablication

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"It cannot be said too often; the psychedelics issue is one of civil rights & liberties." "The notion of there being illegal plants and animals is both ridiculous & obnoxious." Terence McKenna. The war against nature needs to end. Release the green POWs!

Trout's Notes has been helping build better bridges in plant-human relationships since 1993.

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Trout's ayahuasca book is out of print but a color illustrated second edition of that work is now freely available online at Erowid (www.erowid.org).



"Better living through education & awareness."

Tryptamine:

A type of an indole containing an ethylamine (2-aminoethane) at the 3-position.

Indole

2,3-Benzopyrone. Occurs in coal tar and feces. Word is derived from the Latin Indoles meaning "inborn quality or nature".

If those two definitions mean nothing to you, this is probably not your book. If they excite your imagination then you may have just encountered the single most valuable research reference tool that is available to you outside of your own mind. Some Simple Tryptamines is the

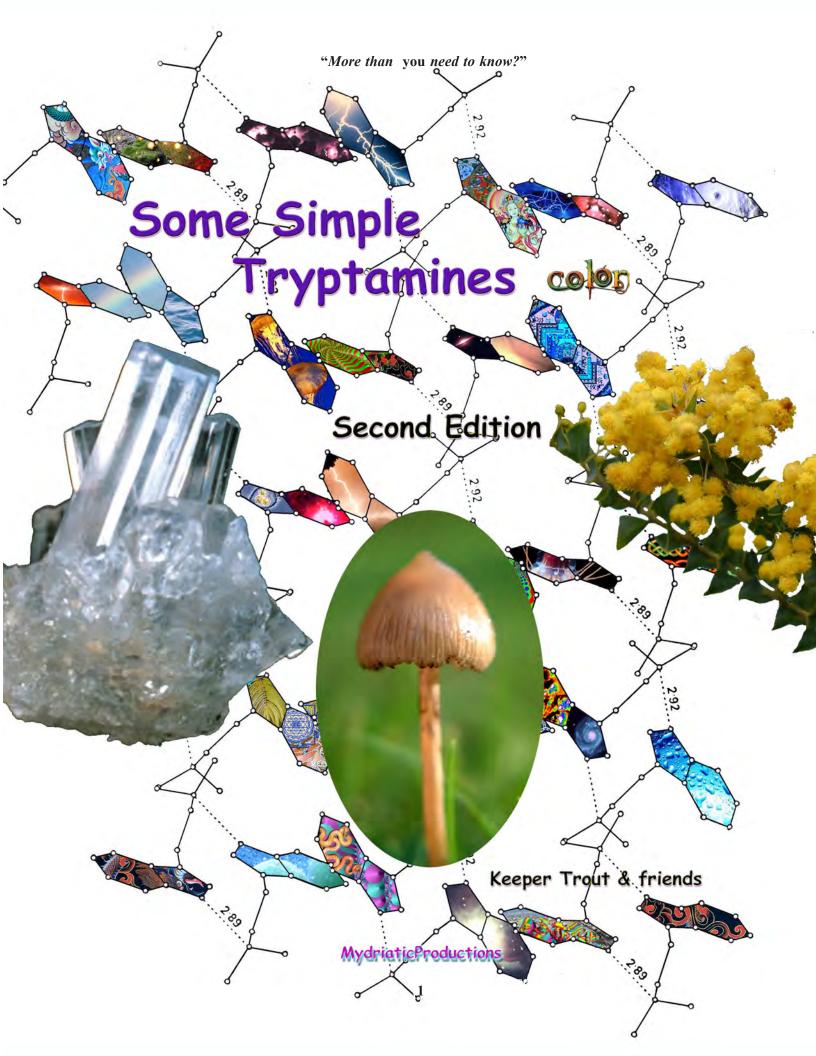
most detailed and accurate data compendium that currently exists concerning the simple tryptamines found in plants, fungi & animal species.

SST2 is not a work intended for a popular audience. It was written for professional researchers and other workers benefitting from a highly specialized and detailed treatment of this multidisciplinary topic. Myriad aspects of the pertinent bio-, phyto-, mycozoo-, physical and analytical chemistries, toxicology & pharmacognosy are interwoven with valuable commentaries, corrections and a wealth of details concerning assorted plant sciences and health related topics. For many people this book is truly more than they will ever need to know.

Some Simple Tryptamines 2nd edition was written for those who are still trying to learn more.



besmanthus leptolobur



Some Simple Tryptamines 🕰 **2nd Edition** A brief overview & resource compendium

Trout's Notes #FS-X7 Revised 12-2006 with minor revisions 5-2007. Created & edited by Keeper Trout with a lot of help from many friends. Copyright ©2007 by Trout's Notes & Mydriatic Productions Revises, updates, merges & replaces ALL FS-X material. (©1993-2004) ISBN 0-9770876-5-4 (softcover); 0-9770876-6-2 (hardcover) Produced by MydriaticProductions; a division of Better Days Publishing Photographs are by Keeper Trout unless indicated otherwise. All photos copyrighted by the photographers & reproduced here with permission.

Cover design by Mulga.

Reformatted for screen viewing and to permit easier online sharing. Please be aware that the resolution is too low for good print quality. High quality print copies of this book in full color are available. I hope you enjoy this work. Many blessings!

Keeper Trout 3 January 2012

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Page 1 images:

Psilocybe semilanceata (The Liberty Cap*): Photo by Dr. P.C. Hickey; DMT crystals: Photo T. Greif & S. Czolowski;

Acacia cultriformis flowers;

DMT crystal packing: after Falkenburg (rendering by Trout):

Photo collage by Trout.

La Liberté image is from Museum of the French Revolution: Vizelle, France [83.314]

* The common name of "Liberty Cap" is believed due to a rough resemblance to a "Phrygian cap".



This soft pointed cap, generally with a peak falling to one side, was given to freed slaves to publicly denote their restored citizenship and, much later, was made famous as a symbol of the French Revolution.

La Liberté

Dedicated with Love to the One Long may we live & prosper; wherever & whoever we are.

IMPORTANT CAUTIONARY STATEMENT TO READERS

All information is contained strictly for informational and educational purposes and should not be construed as advocacy for anyone to violate state or federal laws.

Depending on where a person lives, the following material contains techniques and procedures that might place one in direct violation of state and federal laws if they were put into practice.

Several (four or five) of these substances are currently regarded as dangerous drugs.

Despite a lack of human fatalities and their known safety records exceeding that of many commonly prescribed & over-the-counter pharmaceuticals, they are, in fact, at least potentially, quite dangerous substances, not for their pharmacological or toxic properties but due to the potential actions that may arise from those who quixotically consider them to be dangerous and who are dedicated to MAKING them dangerous.

These zealous peoples' extremely serious and ever-present threat of very real danger should never be underestimated.

Failure to comply with state or federal laws can result in lengthy imprisonment, excessive fines, terroristic home invasions, deliberate terrorism of your family & friends, wanton destruction & vandalism of personal belongings, infliction of immense mental anguish on you & your loved ones, savage beatings & other physical injury, intimidation or harassment of friends or casual acquaintances or even the targeting of them for similar fates, attempted or successful sabotage of careers or business reputation with malicious attacks upon and slanderous accusations against personal character being presented to employers, friends, family or business acquaintances, deliberately brutal murder or injury of pets, eviction from rental properties and/or a complete loss of assets, checking & savings accounts, vehicles, computers, other possessions & real property, child custody; or even worse. Even if you are completely uninvolved and merely the owner of a desirable enough piece of real estate, you may still find yourself being shot in the middle of the night by automatic weapons carrying, night-vision goggled home-invaders as you are trying to put on your pants.

There is no example mentioned above which has not already occurred in the efforts being directed against drug users.

While seemingly unthinkable in any free and democratic society, this is presently the very serious state of reality produced by the current illegality of several of these substances.

Readers should operate under no illusions when reflecting upon the reality of this state-sponsored social purge & cultural cleansing.

The contained information is intended to serve as a reference tool to better enable future research into this important and fascinating area of human consciousness and pharmacognostic science.

We do not advocate the use of illicit —or for that matter, *any*—drugs by uninformed or underinformed individuals.

However, we also recognize that many people will choose to use drugs whether they are informed or not.

We do not intend to encourage or promote drug use. We do want those who are already determined to use these substances, regardless of current legal status, whether planned as sacrament, 'recreation' or experimental material, to be able do so in an informed, knowledgeable and responsible manner.

Our hopes and intentions are that, through education and awareness, more informed choices can be made, thereby minimizing the risks often associated with substance use.

It is with this in mind that we present the following.

ALL Alkaloid Summaries are perennially Incomplete and In Progress.

Non-bold face species names in the occurrence lists denote a conflict, error or lack of proper publication.

This may variously indicate: 1) questions exist; 2) we are unable to locate any verifiable references to support the claim; 3) that the entry represents as-yet unconfirmed preliminary results; or, 4) that it is an erroneous entry published in a secondhand listing in the literature. See comments included in each entry for exact details.

All results of TLC (most graciously performed by my friend Mr. Johnny Appleseed) must be considered to be tentative as results were based entirely on co-tlc with known reference standards and color reactions with Ehrlich's reagents and/or Xanthydrol.

Pure reference material was only available for DMT and 5-Methoxy-DMT.

All potential positive assays for N-Methyltryptamine relied on comparison of chromatographic behavior with isolates of plants known to contain (or sometimes contain) MMT (like *Acacia maidenii*, *Desmanthus illinoensis*, *Desmanthus leptolobus* and *Psychotria viridis*. In all cases the probable identity of MMT was assigned to a particular one of the dark bands that were present at a lower Rf than DMT (intermediate value). Of the bands sometimes seen at a lower Rf than DMT, the band we suspected of being MMT was the only one observed in ALL the species just mentioned. Other low Rf bands sometimes present were never shared by all species. We include our observations here not as proof of MMT's occurrence in any of the plants we mention but rather to stimulate those in a more tenable position to look closer.

It must be emphasized that both alkaloid concentration and composition may vary substantially from one plant to the next, from one part to another within the same plant, and they can also vary by season, or by time of day. Environmental factors, water availability, degree of sun and nutritional factors are also known to affect alkaloid concentration. When a percent is listed, this simply represents what was reported in the literature.

Plant sources for snuffs are highly variable, occasionally ambiguous, and we merged their entries with the probable botanical source. Some of these entries may be erroneous as vouchers were lacking. Botanical sources for Yopo, Paricà and Epéna should be regarded as tentative unless preparation was observed and vouchers made. The presented notions that presence of bufotenine indicates *Anadenanthera* as the source is probably correct but discounts the possibility that other plants may be involved. The paired idea that absence of bufotenine and presence of 5-MeO-DMT indicates *Virola* as the source is also probably correct in most cases but *Anadenanthera peregrina* seeds have been analyzed that showed the presence of 5-MeO-DMT and absence of bufotenine.

Additionally harmine has been found one sample of Paricà co-occurring with DMT, bufotenine and 5-MeO-DMT and in another co-occurring with harmaline and tetrahydroharmine. Similarly a sample of Epéna was reported to contain harmine and tetrahydroharmine.

The frequently presented proposal that **Yopo** be used to refer only to *Anadenanthera* originating snuffs and **Epena** be reserved for the *Virola* based snuffs is a sane one in principle and is indeed applicable to the vast majority of snuffs but many exceptions exist and these designations are not agreed upon by all who use them. Science may define the terms for itself but in light of the ambiguous use by native users, strict definition of the names, as proposed, cannot reflect reality and may instead serve to introduce even more confusion to an already confused area. If those who use the snuffs do not follow the proposals of scientists this presented convention, however rational, seems of dubious usefulness.

Any reference in the bibliography or entry within the occurrence listings that are incomplete was obtained from secondhand sources due to the primary paper being unavailable to us (for one of various reasons).

Abbreviations:

bp= boiling point cm = centimeter gc = gas chromatography gm = gram im = intramuscular ip = intraperitoneal ir = infrared iv = intravenous kg = kilogram kV = kilovolt LD100 = Lethal dose 100% LD50 = Lethal dose 50% ma = milliamp mao = monoamine oxidase maoi = mao inhibitor

ml = milliliter mM = milliMolar mm = millimeter mmp = mixed melting point mp = melting point ms = mass spectroscopy na = not available/applicable nd = not detectednm = nanometer RT = room temperature sc = subcutaneoussp. = species TLC = thin-layer chromatography uv = ultraviolet v = volt



Diplopterys cabrerana

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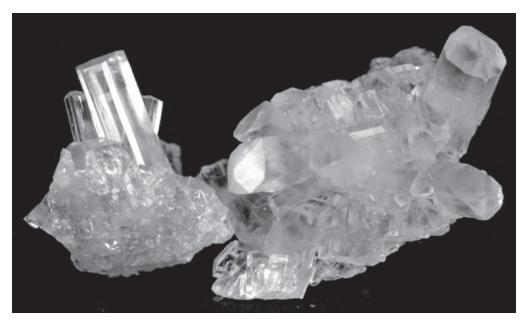
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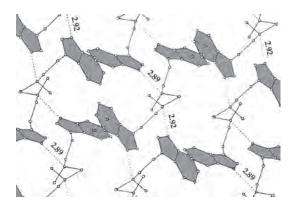


Rock art from the Tassili Plateau in southern Algeria (~5500 years old) Rendering by Kat Harrison; reproduced with permission

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DMT freebase crystals (Photo T. Greif & C. Czolowsky; courtesy of Nick Sand)



DMT crystal packing (After Falkenberg)



Mesquite Left & Below



Trout's Notes on Tryptamines



Tryptamines lacking Ring Substitution







Psychotria alba ripe fruit

Commercially available Psychotria species





Gramine

Gramine is neither a tryptamine nor is it covered in any detail within this work.Due to its frequent presence accompanying some tryptamines, we did want to include some abbreviated information concerning it that might prove of usefulness to those encountering it.
N,N-Dimethyl-1H-indole-3-methanamine, 901; 3-[(Dimethylamino)-methyl]indole; 3-(Dimethylaminomethyl)indole; Donaxine
WLN: T56 BMJ D1N1&1 Hayward: 6R4Y5L(CNM2)=LNHY Usdin & Efron 1979
Chemical Abstracts Registry Number: 000087525 [87-52-5] NIOSH #: NL7525000
Gramine is not a controlled substance:
C ₁₁ H ₁₄ N ₂ MW 174.24 Merck 9 th MW 174.245 Southon & Buckingham 1989
C 75.82%, H 8.10%, N 16.08% Merck 9 th
Free base mp 131-132° (Extracted with ethanol. Concen- tration gave large crystals. Recrystallized from benzene) Pachter <i>et al.</i> 1959 mp 133° (Colorless plates after evaporation of ether) Henry & Leete 1957 mp 133° (Flakes from benzene) Ghosal <i>et al.</i> 1970b mp 134° Boit 1961 mp 138-139° Wassel <i>et al.</i> 1985 mp 138-139° (Shiny flat needles or plates from acetone) Merck 9th
Soluble in chloroform, ethanol, ether Slightly soluble in cold acetone Practically insoluble in petroleum ether, water Merck 9th
Hydrochloride mp 191° (dec.) (from ethanol-E=ether) Soluble in water Merck 9th
Picrate mp 144-145°

mp 144-145° Boit 1961 & Southon & Buckingham 1989

Forms N-oxide similarly to DMT and Bufotenine. See Henry & Leete 1957

Assays for Gramine:

Colorimetric reagents & Reactions: See page 141

TLC & PC: Solvent system Rf Medium Ref. Benzene-Methanol-5% Ammonium hydroxide (10:15:2)0.42 Silica gel 6 n-Butanol-Acetic acid (10:4), saturated with water 0.73 Paper 4 n-Butanol-Acetic acid-Water Paper (120:30:50)0.72 5 [Rf Table Note 3] n-Butanol-glacial Acetic acid-Water (2:1:1) 0.56 Silica gel Kieselguhr (2:1)6 Butanol-Formic acid-Water 9 (16:1:3)0.75 Cellulose *n*-Butanol-Pyridine-Water (60:60:60) 0.70 Paper 5 [Rf Table Note 4] n-Butyl acetate-n-Butanol- Acetic acid-Water (85:15:40:22) 0.53 Paper 3 Chloroform-Methanol (90:10)0.51 Alumina 2 0.53 Alumina 4 Chloroform-Methanol 0.05 (4:1)Kieselgel 1 Chloroform-Cyclohexane-conc. Ammonium hydroxide (20:10:1) (lower phase) 0.0 Silica gel 6 Cyclohexane-Chloroform (1:1)0.0Silica gel 6 Methanol-conc. NH₄OH (29%) (7:1)0.38 Silica gel 9 Methanol-conc. Hydrochloric acid (9:1) 9 0.65 Silica gel Potassium chloride (20% w/v) 0.66 Paper 5 [Rf Table Notes 1, 2, 6] i-Propanol-Ammonia (880)-Water (200:10:20)0.95 Paper 5 [Rf Table Notes 2, 5] i-Propanol alcohol-Ammonia-H₂O (9:1:1) 0.95 Paper 4 i-Propanol -Ethyl acetate-conc. Ammonium hydroxide (120:30:6)0.22 Silica gel 8 *i*-Propanol-Ethyl acetate-conc. NH₄OH-2-Ethoxy-ethanol 0.39 Silica gel (60:15:3:5) 7 n-Propanol-5% Ammonium hydroxide (5:2)0.76 Silica gel 6 Sodium chloride (8% aqueous w/v)-glacial Acetic acid (200:2) 0.67 Paper 5

References:

[Rf Table Note 1, 2, 6]

Audette *et al.* 1970
 Culvenor *et al.* 1964
 Dutta & Ghosal 1967
 Ghosal & Mukherjee 1966
 Jepson in Smith 1969
 Leung *et al.* 1965
 Majak *et al.* 1978
 Williams *et al.* 1971
 Woods & Clark 1971

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Gramine

CC:

Eluted from Brockman alumina with chloroform. Wassel *et al.* 1985

UV:

Kanakoa et al. 1960 Neuss 1964

IR: Neuss 1964

X-ray powder data: Neuss 1964

Synthesis: Kühn & Stein 1937

Activity:

NOT hallucinogenic. Gessner et al. 1961

Pharmacological & physiological properties of Gramine:

- Initially stimulates CNS (clonic convulsions & hyperpnea) and then depresses it.
- Erspamer 1954 Erspamer 1954 discusses pharmacology, physiological properties & toxicology.
- Inhibits norepinephrine reuptake into rat brain synaptic vesicles: Slotkin *et al.* 1978

Toxicity:

Death appears due to respiratory failure.

Gramine and derivatives: Jumping action, clonic and tonic convulsions, but without tremor, were common to all animals before death. Ho *et al.* 1970

The primary observable difference between this and a lethal dose of the tryptamines is that death with the latter is accompanied by tremor.

LD₅₀

44.6 mg/ kg iv in mouse 62.9 mg/ kg iv in rat Erspamer 1954

- AlkaloidLDGramine1225-MeO-gramine1305-Br-gramine75Ho et al. 1970
- It is not clear if the increased toxicity from the 5-bromination of gramine can be extrapolated to the unknown pharmacology of the marine alkaloid 5-bromo-DMT.

For more information see:

Merck Index 9th Edition: Entry 4381 Southon & Buckingham 1989: Entry D-00380

Synthesis of 4 & 5 hydroxylated gramines: Troxler *et al.* 1959



Tryptamine

1H-indole-3-ethanamine; 3-(2-Aminoethyl)indole;
3-(2-Aminoethyl)-indole (Pitman-Moore);
2-(3-Indolyl)ethylamine; Tryptamin (Gr); Triptamina (Sp/Pr/It); Triptaminas (Lith.); T; TPA; Trp; Try.
(Both Trp & Try are also commonly used as the abbreviations for *Tryptophan*)

WLN: T56 BMJ D2Z Hayward: 6R4Y5L(CCZ)=LNHY Entry # 430 Usdin & Efron 1979

CA Reg. # 000061541 [61-54-1] NIOSH # NL4020000

Tryptamine is not a controlled substance

C₁₀H₁₂N₂ MW 160.21 Merck 9th MW 160.218 Southon & Buckingham 1989

C 74.96%, H 7.55%, N 17.49% Merck 9th

Free base:

Cream colored crystals. mp 112-114° Shulgin & Shulgin 1997 Needles from ligroin mp 114.5-115.5° Majima & Hoshino 1925 mp 115° Abramovich & Shapiro 1955 & 1956

mp 115-117° (colorless crystals) Johns et al. 1966

mp 118° Boit 1961; (petroleum ether) Merck 9th

bp 0.15mm 137° Southon & Buckingham 1989

Very sparingly soluble: benzene, chloroform, ether Southon & Buckingham 1989

Soluble in ethanol, acetone.

Practically insoluble: benzene, chloroform, ether, $\rm H_2O.$ Merck 9th

Saavedra & Axelrod 1972 used toluene-*iso*-amyl alcohol (97:3 v/v) during recovery from tissues (said to lessen the presence of other amines.)

Optically inactive. Boit 1961

Chloroform-Water Partition coefficient: 1.37 Gessner & Page 1962

Hydrochloride:

CA Reg. # [343-94-2] NIOSH #: NL4375000 Southon & Buckingham 1989 mp 246° Boit 1961 mp 248° (Colorless needles) Manske 1929 mp 248-249° (Colorless needles) Majima & Hoshino 1925

Picrate:

mp 242-243° Hoshino & Shimodaira 1935 mp 243° Manske 1929 mp 245-246° (from dilute acetone) Abramovich & Shapiro 1955 & 1956 mp 247° dec. (dark red prisms from ethanol) Southon & Buckingham 1989

HFB derivative:

MW 384 mp 103-104° Preparation & isolation Benington *et al.* 1975

Assays for Tryptamine: (Usdin & Efron 1979)

Aures *et al.* 1968
Beckett & Moffat 1968 (Applicable; but did not use for this compound)
Clarke 1969 (See also Clarke 1986)
Iskric *et al.* 1969
Saavedra & Axelrod 1972 presented a sensitive tissue assay but it requires purified NMT from rabbit lungs

Colorimetric reagents: See page 141

TLC & PC:

solvent system	<u>Rf</u>	Medium	Ref
Acetone-i-Propanol-W))
(50:40:7:3)	0.67	Paper	11
Benzene-Methanol-5%	6 Amm	onium hydro:	xide
(10:15:2)	0.27	Silica gel	2
	0.35	Silica gel	4
Butanol-Acetic acid-W	/ater		
(60:15:25)	0.65	SilCel	8
n-Butanol-glacial Acet	ic acid-	Water	
(2:1:1)	0.64	Silica gel-	
		Kieselguhr	(2:1)
		U	4
n-Butanol-Glacial acet	ic acid-'	Water	
(2:1:1)	0.57	Silica gel	2
<i>n</i> -Butanol-Acetic acid-		28	_
(60:15:25)	0.75	Avicel	3a
(00.15.25)	0.72	Cellulose	3a
	0.72	Paper	3a
<i>n</i> -Butanol-Acetic acid-		1 aper	Ju
(4:1:5)	0.63	Paper	1
(4.1.3)	0.67	Paper	12
n Putanal alagial Agat			12
<i>n</i> -Butanol-glacial Acet (120:30:50)	0.72		3b
		Paper	50
<i>n</i> -Butanol-Acetone-Di			2.
(70:70:14:35)	0.98	Avicel	3a
<i>n</i> -Butanol-Acetone-Di			2
(70:70:14:35)	0.94	Cellulose	3a
<i>n</i> -Butanol-Acetone-Di	-		2
(70:70:14:35)	0.95	Paper	3a
n-Butanol-25% Methy		-	
(8:3)	0.89	Paper	1
(3:1)	0.89	Paper	12
n-Butanol-Pyridine-W			
(60:60:60)	0.75	Avicel	3a
n-Butanol-Pyridine-W	ater		
(60:60:60)	0.68	Cellulose	3a
n-Butanol-Pyridine-W	ater		
(60:60:60)	0.83	Paper	3a
n-Butanol-Pyridine-W	ater		
(60:60:60)	0.76	Paper	3b
		_	

Tryptamine

Chloroform-Acetic acid (65:20:10:5) Chloroform-Cyclohexa	0.40 ne-conc	Paper	11 hydroxide
(20:10:1) (lower phase	e) 0.0	Silica gel	4
Chloroform-Methanol	0.0	Silicagei	7
(9:1)	0.07	Alk. silica	6
	0.14	Silica gel	10
Cyclohexane-Chlorofor		0.1. 1	4
(1:1) Ether-Methanol-25% N	0.0 NH OH	Silica gel	4
(17:2:1)	0.48	Kieselgel	9
Ethyl acetate-Methano			<i>.</i>
(80:15:5)	0.32	Silica gel	2
Morpholine			
$(0.1 \text{ M in H}_2\text{O})$	0.64	Silica gel	7
Methanol	0.19	Alk. silica	6
Methanol-Acetic acid-V		AIK. SIIICa	0
(75:10:15)	0.65	Silica gel	8
Methanol-Ammonia (sj	p. gr. 0.		
(100:1.5)	0.22	Alk. silica	6
	0.31	6060	6
D . 1 D 11 W	0.31	6061	6
Pentanol-Pyridine-Wate		D	10
(2:2:1) Potassium chloride	0.53	Paper	12
5% in distilled water	0 56-0	.59 Paper	1
Potassium chloride	0.000	.c> ruper	
20% (w/v) aqueous	0.48	Paper	3b
n-Propanol-1N Acetic a	acid		
(3:1)	0.65	Avicel	
	0.67	Cellulose	
n Drananal () 2NI Amm	0.72	Paper	3a
<i>n</i> -Propanol-0.2N Amme (3:1)	0.90	Avicel	
(5.1)	0.89	Cellulose	
	0.90	Paper	3a
i-Propanol-5% Ammor			
(5:2)	0.44	Silica gel	2
	0.80	Silica gel-	
		Kieselguhr	
Propanol-25% Ammon	ia	(2:1)	4
(5:1)	0.85	Cellulose F	8
<i>i</i> -Propanol-Ammonia (0
(200:10:20)	0.95	Avicel	3a
<i>i</i> -Propanol-Ammonia (0.88) - W	ater	
(200:10:20)	0.89	Cellulose	3a
<i>i</i> -Propanol-Ammonia (8			2.
(200:10:20) (16:1:3)	0.83 0.71	Paper	3a 12
(16:1:3) <i>i</i> -Propanol-Ammonia (0		Paper	12
(200:10:20)	0.87	Paper	3b
<i>i</i> -Propanol-Ethyl acetat			
2-Ethoxy-ethanol		4	
(60:15:3:5)	0.33	Silica gel	5
<i>i</i> -Propanol-Formic acid			2
(40:2:10)	0.77	Avicel	3a
<i>i</i> -Propanol-Formic acid (40:2:10)	-Water 0.81	Cellulose	3a
(10.2.10)	0.01	Contribute	Ju

<i>i</i> -Propanol-Formic acid-Water					
(40:2:10)	0.83	Paper	3a		
n-Propanol-Water					
(3:1)	0.69	Paper	12		
Sodium chloride (8% aqueous w/v)-glacial Acetic acid					
(200:2)	0.51	Paper	3b		
Distilled water	0.29-0	.35 Paper	1		

References:

- Erspamer 1959 (See more in Erspamer 1955)
 Gupta *et al.* 1979 (silica gel 60F-254 with PDAB)
 Jepson 1960: pages 183-212 & (3b) 1969: pp. 243-273 and table 9.3 (See Notes in main Rf table)
 Leung *et al* 1965 (as HCl)
 Majak *et al.* 1978
 Phillips & Gardiner 1969: 6061 = silica gel Eastman chromatogram 6061; 6060 = Silica gel with fluorescence indicator Eastman chromatogram 6060; Alk. silica= silica gel treated with 0.1N sodium hydroxide
 Sanders & Bush 1967
 Stijve et al. 1984
 Wagner & Grevel 1982
 Johns *et al.* 1966
 Smith & Seakins 1976
- 12. Rodnight 1956

Column chromatography:

Johns *et al.* 1966 eluted from weak neutral alumina with chloroform.

HPLC:

Balandrin *et al.*Kysilka *et al.*Kysilka & Wurst 1988 & 1989 Sunshine 1981 Wurst *et al.*UV vs ED: Wurst *et al.*

GLC:

Christian *et al.* 1975 Martin *et al.* 1972 Retention data for 3 isothermal systems: Beckett & Moffat 1968 GLC of HFB derivative: Benington *et al.* 1975 & Christian *et al.* 1975 Vessman *et al.* 1969

GC:

Martin *et al.*Wurst *et al.*Retention times for Amine 220, CHDMS and DEGS columns: Audette *et al.*

UV:

λmax (MeOH): 220, 275, 280, 290 (ε 37900, 6900, 6000, 5500) De Moraes *et al.* 1990 λmax 220, 280 nm (0.1N NaOH) Sunshine 1981 Base: λmax (EtOH): 222, 282, 290 nm (log ε 4.56, 3.78, 3.71) Merck 9th HCl: λ max (95% EtOH): 221, 275, 281, 290 nm (log ϵ 4.52, 3.73, 3.75, 3.69) Merck 9th

Fluorescence:

Fluorescence data: Bridges & Williams 1968 Violet fluorescence under short-wave UV in tlc using PENE. Majak et al. 1978 Fluorescence spectrum at pH 7.4: Activation: 290 nm Emission: 370 nm Gessner & Page 1962 254 nm: Absorbs; 350 nm faint yellow fluorescence Smith & Seakins 1976 Fluorescence maxima: 355 mu. Excitation maxima: 280 mu Fluorescence values decline in acidic systems: Half at pH 2 Burnett & Audus 1994 Udenfriend 1962 has fluorescence spectra (p. 165) & quantitative fluorescence assay (pp. 165-168)

IR:

(cm⁻¹) 751, 811, 882, 941, 1014, 1112, 1128 Shulgin & Shulgin 1997

Mass spectra: See Couch & Williams 1972 MS of HFB derivative: Gupta *et al.* 1979

GC-MS: see De Moraes et al. 1990

NMR:

Cohen *et al.* 1960 De Moraes *et al.* 1990

Synthesis:

Abramovich & Shapiro 1956 Ewins 1911 Jackson & Smith 1965 Majima & Hoshino 1925 Noland & Hartman 1954 Shulgin & Shulgin 1997 Tacconi 1965 Thesing & Schulde 1952 See also Saxton 1965

Another synthetic route for tryptamine would be to enzymatically decarboxylate tryptophan. See Baxter & Slaytor 1972a Christenson *et al.* 1972

Some reported occurrences of Tryptamine:

Tryptamine is widespread in occurrence in both plants and animals. It is often as a trace alkaloid

Only a few representative instances of occurrence are included below:

Smith 1977b lists occurrences in 26 species in 12 genera. Agaricaceae

Amanita citrina (0.0-0.06%) Wurst et al. 1992

Copelandia cyanescens (0.002 & 0.008%) Allen & Merlin 1992a *Psilocybe cubensis* (0.003% & 0.002% & <0.002%) Allen & Merlin 1992a Panaeolus antillarum (<0.002%) Allen & Merlin 1992a Panaeolus sphinctrinus Merlin & Allen 1993 Panaeolus semiovatus Merlin & Allen 1993 Panaeolus subbalteatus Merlin & Allen 1993 Graminae Hordeum vulgare (roots of seedlings) Schneider & Wightman 1974 Leguminosae Acacia acuminata Benth. Main alkaloid in stem and leaf. Amount not given. Broadleafed form gave 0.72% total alkaloid and narrow-leafed form gave 1.5% total alkaloid. Both collected Oct. White 1957: 719 Acacia baileyana F. v M. In some samples of leaves. Minor alkaloid in March collection (9 mg from 300 gm of dry leaf) Not observed in July sample of fresh leaf and Sole alkaloid in October collection of fresh leaf (30 mg from 180 gm) Repke et al. 1973 Acacia caesia (L.) Willd. (= Acacia intsia Willd.) In root/stem-bark. % not given. Ghosal 1972 Acacia cardiophylla A. Cunn. ex Benth Small quantities in stem and leaf. (Amounts not given) White 1957: 719. Acacia cultriformis Cunn. Variable amounts. Detected by White 1957: 718. Also in stem and leaf and in pods as major alkaloid by White 1951: 58. Acacia farnesiana (L.) Willd. In stem-bark Ghosal 1972 [not observed by most investigators] Alkaloid negative in stem, bark, leaf and flowers Fong et al. 1972. Alkaloid negative in stem, leaf and flowers by Smolenski et al. (1973). Ehrlich's negative in seeds, rootbark and bark by J. Appleseed 1994 & 1995. Acacia floribunda Sieb. (= Acacia longifolia var. floribunda F. v. M.) Significant portion of total bases in tops, major alkaloid in flowers (less than 1% by dry weight), White 1944b : 157-162. Acacia leucophloea In root bark, amount not given, Ghosal 1972a Acacia longifolia Willd. Some flowers only. Not stem or leaf. White 1951:58 Acacia nilotica (L.) Delile In leaves (unconfirmed), according to Oliver-Bever 1986 who gives no reference. Negative alkaloid assay of roots, stem-bark and leaves by Odebiyi & Sofowora 1978 Acacia podalyriaefolia Cunn. (A. podalyriifolia) Major alkaloid in stem & leaf. White 1951: 58 & 60. Stem & leaf (0.29% total alkaloids); unripe seedpods (0.11% total alk.) White 1957: 719. From leaves. Balandrin et al. 1978 Leaf and also stem-bark negative for alkaloids by Smolenski et al. 1973

Tryptamine

Acacia pruinosa Cunn.

Major alkaloid in stem and leaf. ("*At most*" 0.04% total alkaloids; although a May sampling by White 1944a was reported as containing 0.09% total alkaloids.) White 1944b: 157-162.

Acacia rigidula Benth.

0.8 ppm in early season/ 21.2 ppm in late season. Fresh leaf, petiole & tender twig. Clement *et al.* 1998 *Acacia vestita* Ker.

In stem and leaf. (Highly variable.) [January sample-0.03% and 0.04% total alkaloid, May sample 0.28% total alkaloid, August sample- 0.08% total alkaloid, October sample- 0.12% total alkaloid. Tryptamine was present as up to 83% of the total alkaloid.] White 1957: 719

Mimosa scabrella DeMoraes et al. 1990

Mimosa somnians (0.026%) Gupta et al. 1979

Petalostylis labicheoides var. casseoides (0.44-0.47% in dry leaf & stem) Johns et al. 1966

Prosopis juliflora (Swartz) DC Saxton 1965

Prosopis nigra (Gris.) Hieron. Moro et al. 1975

Animals:

Gorgonaceae

Villagorgia rubra Espada et al. 1993

Paramuricea chamaeleon Cimino & DeStefano 1978

Salamandridae

Salamandra maculosa Erspamer 1954 [now considered a synonym of Salamandra salamandra]

Triturus cristatus Erspamer 1954

Natural occurence in rat CNS:

Christian et al. 1977 and

Saavedra & Axelrod 1972

(Martin *et al.* 1972 could not detect in rat brain but did in dogs, steers [also Martin *et al.* 1971] & human. Nice summary of earlier work.)

Eccelston *et al.* 1966 could not detect it in guinea pig brains (unless given tryptophan & an MAOI) & found errors in published analysis that could generate false positives.

See also:

Erspamer 1954 Graziano *et al.* 1971 Mears & Mabry 1971 Smith 1977b Stowe 1959



Acacia podalyriifolia

Occurrence in humans:

Tryptamine has been found in normal human blood and urine by Franzen & Gross 1965; Urine: Rodnight 1956

Found in cerebrospinal fluid of normals & psychotics: Corbett et al. 1978 & Narasimhachari et al. 1971b

CSfluid: Christian et al. 1975; Brain: Martin et al. 1972

Activity:

May be active if perfused intravenously. This is a poorly defined area based on the report of Martin & Sloan 1970 using a 250 mg dose.

- This led to some suggestions that tryptamine MIGHT be hallucinogenic in large amounts given IV.
- Shulgin & Shulgin 1997 correctly point out that this conclusion is, at the least, highly questionable. (See Shulgin's comments on the subject)
- It apparently has never been investigated further.

Pharmacological & physiological properties of Tryptamine:

Tryptamine is a hypertensive drug.

- It is also a cholinesterase inhibitor.
- Erspamer 1954 discusses pharmacology, physiological properties & toxicology.
- Produces somatic effects similar to other tryptamines when injected into animals. Saavedra et al. 1970.
- Inhibits norepinephrine reuptake into rat brain synaptic vesicles: Slotkin *et al.* 1978
- Metabolized by both MAO-A & MAO-B. Suzuki et al. 1981.
- MAOI enhances & prolongs activity. Tedeschi et al. 1959.

MAO kinetic constants: Suzuki et al. 1981

- Antagonized by Cyproheptadine & Chlorpromazine but not by Phenoxybenzamine. Martin & Eades 1970.
- Tryptamine induced hypokinesia in rats (tryptamine causes depression not stimulation) is antagonized by Phenethylamine and by Methylamphetamine. Saavedra *et al.* 1970.
- Reverses reserpine-induced depression but only for a short duration. If done after iproniazid, this action was "*markedly potentiated and prolonged*." Tedeschi *et al.* 1959 See also the discussion in Tedeschi *et al.* 1959

Metabolism: Erspamer 1954

Erspamer 1955 reported that rats excrete largely as IAUA (Indoleaceturic acid) with lesser amounts of IAA (Indole acetic acid)



Tryptamine

MMT

Tolerance:

Tryptamine produces a short lived desensitization to itself. Erspamer 1954

Toxicity of Tryptamine:

LD₅₀ (as HCl) 2-1 mM/ kg/ ip/ mouse Shinoda *et al.* 1974

Tedeschi *et al.* 1959 reported some rat deaths occurred at 40 mg/kg iv. This dose produced backward locomotion, Straub tail, bradypnea & dyspnea, severe asymetrical clonic convulsions (& asphyxial convulsions in the ones that died). They noted body tremors, "*bilateral placing-type clonic movements of forepaws*" & hunching of the back at 10-40 mg/kg iv]

For further reading, see:

Merck 9th; Entry# 9456 Shulgin & Shulgin 1997: pp. 579-584; Entry #53 Southon & Buckingham 1989: Entry #T-00366

N-Methyltryptamine

N-Methyl-1H-indole-3-ethanamine, 9 CI;

3-(2-Methylaminoethyl)indole;

Mono methyl tryptamine; Amino-N-methyltryptamine; β-Indolylethyl-β-methylamine; Dipterine; MMT; NMT; MTPA; N-Metiltriptamina (Sp/Pr/It); Metiltriptaminas (Lith.); Tryptamine, N-Methyl-.

[Note: While the abbreviation NMT is commonly encountered, it is potentially confusing as the enzymes used to perform N-methylations are also known as NMT (N-Methyl Transferases) [There are two NMT enzymes; one (PIMT) which methylates Tryptamine and the other (SIMT) which methylates N-Methyltryptamine]

A biosynthetic scheme discussing the use of NMT to synthesize NMT might be far from confusion free.

N-methyltyramine is also known as NMT.

It too is biosynthesized via an NMT that is believed to be distinct from either NMT that methylates tryptamine.]

WLN: T56 BMJ D2M1 Hayward: 6R4Y5L(CCNHM)=LNHY Usdin & Efron 1979 #411

CA Reg. No.: 000061494 [61-49-4]

 $C_{11}H_{14}N_{2}$

MW 174.2 Clarke's 1986 MW 174.245 Southon & Buckingham 1989: # M-00286

Free base

mp 86-87° White prisms of MMT crystallized from light petroleum Fitzgerald & Sioumis 1965 mp 87-88° Yuraschevski & Stepanova 1939 mp 88° Base regenerated from HCl and recrystallized from light petroleum. Arthur *et al.* 1967

mp 88-90 (from ether) Platonova et al. 1958

mp 89° Boit 1961

mp 89-90° Hoshino & Shimodaira 1935

mp 89-90° Fish et al. 1956

mp 90° From a little chloroform with petroleum ether added to induce crystallization. Stellate aggregates of needles. Several isolated crystals showed rectangular form. Manske 1931

mp 90° (87-88°) Prisms from petroleum ether. Southon & Buckingham 1989

mp 91° Needles from benzene Lou et al. 1965

They initially obtained crystals by concentration of a benzene solution, recrystallized from chloroform and again from a concentrated benzene solution, precipitating the crystals by adding petroleum ether to cloudiness.

- mp 168-169° needles from *n*-propanol Filho & Gilbert 1975 [Ed.: ??]
- Vacuum distilled 135-145° at 0.1 mm/Hg yielding a white oil formed white crystals which darkened on exposure to air. Shulgin & Shulgin 1997: pp. 573-574, Entry # 50.

Soluble in CH₂Cl₂

Soluble in water (EtOAc:H₂O partition)

Slowly crystallized after evaporation of water. Filho & Gilbert 1975

Hydrochloride

- mp 175-177° (from Ethanol) Shulgin & Shulgin 1997
- mp 177-178º Yuraschevski & Stepanova 1939
- mp 178° Boit 1961
- mp 178-180° Shulgin & Shulgin 1997
- mp 180° Colorless elongated plates with pyramidal terminations from alcohol-acetone or alcohol-ether. Manske 1931
- mp 180° Southon & Buckingham 1989
- mp 180° Lou et al. 1965
- mp 180-182° (Crystals from acetone/ methanol) Arthur *et al.* (1967

Picrate

- Very sparingly soluble in hot water. Manske 1931
 mp 189-190° Yuraschevski & Stepanova 1939
 mp 190° Boit 1961
 mp 191° After thorough washing with ether.
 picrate formed in alcohol, Benzene was added and the solution reduced to small volume.
 Addition of "*much ether*" induced crystallization.
 Formed large plates "*resembling azobenzene in color*"
 Manske 1931
 mp 192° Lou *et al.* 1965
 mp 193-195° (Red needles)
- Fitzgerald & Sioumis 1965
- mp 193-195° (Red needles)

Southon & Buckingham 1989

mp 193-195° (Orange crystals) Fish et al. 1956

Trout's Notes FS-X7: N-Methyltryptamine

N-Methyltryptamine

Picrolonate

mp 240-243° Fitzgerald & Sioumis 1965 mp 242-243° Yuraschevski & Stepanova 1939 mp 243° Boit 1961

Methiodide mp 208-209° Crystals from methanol Southon & Buckingham 1989

HFB derivative

Oil MW 566 Preparation & isolation Benington *et al.* 1975

Assays for N-Methyltryptamine:

Heating the base in chloroform with slight excess of phenylisocyanate, evaporating to a small volume and then adding ether will cause the formation of large elongated hexagonal plates of the phenylcarbamyl derivative of N-methyltryptamine. They melt sharply at 153° when recrystallized from methanol-ether and give an immediate red color with an orange cast with Ehrlich's reagent. Manske 1931

N-Methyltryptamine and other secondary amines do **not** form N-oxides with peroxide. Fish *et al.* 1955

Colorimetric reagents: See color reactions p. 142

TLC & PC: see Rf table pp. 169-176

HPLC:

Balandrin *et al.* 1978 Kysilka & Wurst 1988 Kysilka *et al.* 1985

GLC:

Christian *et al.*GLC of HFB derivative: Benington *et al.*Vessman *et al.*

GC

Retention times for Amine 220, CHDMS and DEGS columns: Audette *et al.* 1969

UV:

UV λ max (CH₃OH): 220, 275, 280, 290 (= 3400, 5500, 5500, 5000). De Moraes *et al.* 1990 UV λ_{max}^{EOH} : 229, 275, 284, 292 nm. Grina *et al.* 1982

IR:

Graphic portrayal. page 774 in Clarke's 1986. Shulgin & Shulgin 1997: Base: (cm⁻¹): 740, 1018, 1103, 1132, 1161 HCl: (cm⁻¹): 748. 850, 1009, 1104, 1119, 1136

GLC-MS

GLC of HFB derivative: Benington *et al.* 1975 & Vessman *et al.* 1969

GC-MS:

De Moraes et al. 1990

MS:

Shulgin & Shulgin 1997: page 574 of HCl: (m/z) $C_2H_6N^+$ 44 (100%); Indolemethylene⁺ 131, 130 (61%, 51%); parent ion 174 (2%) Williams *et al.* 1971: [m/e 44, 103, 115, 130, 131, 143, 174] Holmstedt & Lindgren 1967: [m/e 44 (base peak), 103, 115, 130, 131, 145, 174 (M+)]. Filho & Gilbert 1975 EIMS: Grina *et al.* (1982) MS of HFB derivative: Gupta *et al.* 1979 MS of TMS derivative: Narasimhachari *et al.* 1971

NMR:

Cohen *et al.*De Moraes *et al.*Filho & Gilbert 1975 Grina *et al.*

Synthesis:

Manske 1931 Hoshino & Shimodaira 1935 Shulgin & Shulgin 1997

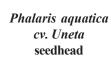
Isolation:

The first isolation of indolethylamines from plants was that of N-Methyltryptamine from *Girgensohnia diptera* Bge. (co-occurring with N-Methylpiperidine) in 1939 by N.K. Yurashevski & S.E. [S.I.?] Stepanov[a?] The alkaloid was named Dipterine in Yurashevskii 1940, and equated with Nmethyltryptamine, previously known as a synthetic compound.

A number of sources state that the first reported isolation was from *Arthrophytum leptocladum* M. Popov (Chenopodiaceae) where it co-occurs with N-methyltyramine and the β -carboline, Leptocladine. (First reported natural occurrence of Leptocladine.) citing Yurashevskii 1939 & 1941 [also given as Yurasheskii] While the 1939 article did report Leptocladine, MMT as was observed in this species, remained an unidentified indole until the 1941 article. 2-methyl-1,2,3,4-tetrahydro- β -carboline was also eventually identified in this species, by Platonova *et al.* 1958

Reported Occurrence of N-Methyltryptamine:

Said to be of frequent occurrence; see Karrer 1958



Plants

Acanthaceae

Justicia pectoralis In leaf. Shulgin & Shulgin 1997

- Justicia pectoralis var. stenophylla
 - In leaf. 31 August 1994 harvest. Faint band corresponding MMT. TLC by Appleseed
- Aizoaceae

Delosperma sp.

- Presence of MMT in Delosperma spp. is unproven, except for the unpublished data involving work done by the Smith, Kline & French Laboratories. (Cited by Raffauf 1970) Smith 1977b cited Rivier & Pilet 1971 and Deulofeu 1973. Both had cited Raffauf 1970.
- Assays by Appleseed 1994-1996:
- Delosperma acuminatum Faint. Sept., Nov. 1994 and Nov. 1995 assays. (Ehrlich's)
- Delosperma brittenae Nov. 1995 assay (Xanthydrol)
- Delosperma cooperi Sept. 1994 and Nov. 1995 assays. (Ehrlich's and Xanthydrol)
- Delosperma esterhuyseniae Nov. 1995 assay (Xanthydrol) Delosperma hallii Nov. 1995 assay. (Xanthydrol)
- Delosperma harazianum Audhali Plateau, Yemen Nov. 1995 assay. traces (Xanthydrol)
- Delosperma hirtum Dec. 94 (Ehrlichs) Nov. 95 assay. traces (Xanthydrol)
- Delosperma klinghardtianum Nov. and Dec. assays. Sole base present. (Xanthydrol and Ehrlich's) Not seen Sept. 96 assay.
- Delosperma litorale Nov. 1995 assay. (Xanthydrol)
- Delosperma pageanum (Same plant tested Christmas 1994) 2 Nov. 1995. Dark band (Xanthydrol)
- Delosperma pageanum (Different plant from same source) Dec. 1994 assay. (Ehrlich's)
- Delosperma tradescantioides Nov. 1994 assay. (Ehrlich's) Suspected MMT was major. All faint.

Calvcanthaceae

- Calycanthus has been listed as containing MMT. This is probably in error. Manske 1931a & 1931c reported MMT as a degradation product of Calycanthine. Unable to locate any reference indicating its actual presence.
- Chenopodiaceae (All traces unless stated)
- Arthrophytum leptocladum M.Popov (Thin-stemmed Haloxylon)
- 0.575% by dry weight in 1 year old Green shoots (Flowering stage) (3.7% total alkaloid content; Major alkaloid. Cooccurring with 2 β-carbolines.) Platonova et al. 1958. In leaf and stem. Smith 1977b cited Yurashevski 1941 Zhur. Obsch. Chem. 11:157 and Rousseau et al. 1966 Bull.Soc.Pharm.Nancy 71:31

Arthrophytum wackchanica [A. wakhanicum?] Smith 1977b cited Orekhov 1955

Girgensohnia diptera Bge.

Yurashevski & Stepanov[a?] 1939 (Smith 1977b)

Haloxylon scoparium

Shulgin & Shulgin 1997

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Hammada leptoclada
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In leaf and stem.
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Orazkulyev et al. 1964 [Grazkuliev et al. 1964 Zh. prikl. Khim. Leningr., 37: 1394; according to Arthur et al. 1967.]

Graminae Hordeum vulgare (roots of seedlings?) Schneider & Wightman 1974 Phalaris arundinacea Amounts not given. Detected by tlc in some cv. Ottawa Synthetic Woods & Clark 1971 Phalaris aquatica cv. AQ-1 Occurrence reported (HPLC). Festi & Samorini 1994b Phalaris aquatica (Commercial) Weak occurrence reported (HPLC). Festi & Samorini 1994b Phalaris tuberosa cv. Australian Commercial Minor alkaloid in seedlings. Mack et al. 1988 A minor alkaloid in 7 day old seedlings. Mulvena & Slaytor 1983 Phalaris paradoxa (Romania) Traces reported (HPLC). Festi & Samorini 1994b Phalaris truncata (France) Traces reported (HPLC). Festi & Samorini 1994b Lauraceae Nectandra megapotamica (Sprg.) Chodat & Hassler 0.57 gm. from 5.7 kg. of finely powdered bark. Filho & Gilbert 1975 Leguminosae Acacia albida Possible but unconfirmed presence. Detected in Fall 1993, Spring, Summer, Fall 1994 and Spring 1995; tlc by Appleseed. Acacia confusa Merrill (=A. richei A.Gray) Unspecified amount in bark. Lou et al. 1965 1.43% in (dried?) root-bark Liu et al. 1977 (55.25% of total alkaloid) Root bark total alkaloid content was 2.58% 0.04% in dried stem Arthur et al. 1967 (8.8 kg. of stems vielded 3.1 gm. MMT & 1.3 gm. DMT.) Acacia maidenii F.vonMueller 0.24% in dry bark. Fitzgerald & Sioumis 1965. [Tentative positive in roots as the major alkaloid. (unconfirmed) tlc by Appleseed 1994] (2 year old seed grown plants) Acacia obtusifolia A.Cunn. Suspected to co-occur with DMT (tlc by Appleseed). Mulga ran hplc-ms and observed what appeared to be a simple tryptamine but was unclear if the identity was this alkaloid or tryptamine as the parent fragments seemed to be lacking; see http://www.lycaeum.org/drugs/plants/tryptamines/acacia/ species.htm & Trout's Notes on Acacias 2004. Acacia rigidula Bentham 4.6 ppm in early spring/ 54.9 ppm in late fall. Fresh leaves, petioles & tender twigs. Clement et al. 1998 Acacia simplicifolia Schinz & Guillaumin (= Acacia *simplicifolia* Druce (?) [= A. simplex?] 1.44% in bark and 0.29% in twigs. Stem bark has 3.6% total alkaloid content. N-Methyltryptamine (40% of total) Twigs have 0.11% total alkaloid content. N-Methyltryptamine (26.3% of total)

Poupat et al. 1976

N-Methyltryptamine

Anadenanthera peregrina (L.) Spegazzini [No.24625; Origin: Boa Vista, Brazil] Traces in dry bark. Agurell et al. 1969 Bark of Brazilian material (as *Piptadenia*). Legler & Tschesche 1963 reported a mixture of 5-MeO-MMT and MMT as comprising 41% of the crude base. as *Piptadenia peregrina* Bark (Colombia in 1956) Holmstedt & Lindgren 1967 Desmanthus illinoensis (Michx.) MacM. 0.11% in Root bark (dried) and 0.0016% in Root wood (dried) Thompson et al. 1987 Desmanthus leptolobus Tentative in dried root bark (present in most but not all specimens) Appleseed tlc 1993-1994. Mimosa ophthalmocentra Mart. ex Benth. 0.0012% dry wt. in roots. Batista et al. 1999 In stem Batista & Almeida 1997 (No quantification) Mimosa scabrella Bentham In bark. De Moraes et al. 1990 (No quantification) Mimosa somnians Humb. & Bonpl. ex Willd 0.029% in dry whole plant. Gupta et al. 1979 Petalostylis labicheoides var. casseoides This is in error [Chemical Abstracts] See Johns et al. 1966a (Included by a number of references including Mears & Mabry in Harbourne et al. 1971: 146) Swainsona galegifolia (Andr.) R.Br. =(S. coronillifolia Salisb.) (Possible assay in stem and leaf. Contradictory results.) tlc by J. Appleseed Tachigalia paniculata Aubl. 0.005% w/w of previously prepared inflorescence extract. No information on plant weight. Svoboda et al. 1979 Malpighiaceae **Diplopterys** cabrerana Traces in leaf. Agurell et al. (1968)a **Mvristicaceae** Virola spp. Virola based snuff: "Epena" [No.24574; Origin: Rio Cauaburi, Brazil] 0.014% N-Methyltryptamine; i.e. 14.3 mg. / 100 gm. of snuff. [2% of 715 mg total alkaloid per 100 gm.] Virola based snuff: "Nyakwána" [No. 24626; Origin: Tototobi, Brazil] Traces Agurell et al. 1969 Snuff: "Epéna" (Virola?) Snuff as prepared by Waica Indians (collected 1965) MMT [with DMT and 5-MeO-DMT] Holmstedt & Lindgren 1967 Virola calophylla Warb. [No.24603; Origin: Manaus, Brazil] Flowering shoots- 0.0077% MMT i.e. 7.7 mg/ 100 gm [4% of 193 mg. of total alkaloids/ 100 gm. of dry flowering shoots] Leaf- 0.0062% MMT i.e. 6.2 mg/ 100 gm [4% of 155 mg. of total alkaloids/ 100 gm. of dry leaves] Agurell et al. 1969

Virola calophylla

Bark collected in Manaus, Brazil during 1964. MMT [with DMT and 5-MeO-DMT] Holmstedt & Lindgren 1967

Virola rufula (A.DC) Warb. [No.24612; Origin: Manaus, Brazil]

Leaf- 0.0059% i.e. 5.88 mg/ 100 gm [6% of 98 mg. of total alkaloids/ 100 gm. dry leaf]

Agurell et al. 1969

Virola sebifera

Present in bark Kawanishi *et al.* 1985 Paste: *Virola sebifera* (DMK-40; Don Marcos no. 1) MMT 1.38 mg/ ml

McKenna et al. 1984a

Virola theiodora Warb. [No.24595; Origin: Manaus, Brazil]

Bark- 0.0025% (i.e. 2.5 mg/ 100 gm) [1% of 250 mg. of total alkaloids/ 100 gm. dry]

Flowering shoots- 0.033% (i.e. 32.9 mg/ 100 gm) [7% of 470 mg. of total alkaloids/ 100 gm. of dry flowering shoots] Agurell *et al.* 1969

Virola theiodora

Bark- 0.0034% [8 mg from 235 gm. of bark]

Cassady *et al.* 1972, also 1971 The latter cited Cassady *et al.* 1970 [Published 1972]

Ochnaceae

Testulea gabonensis Pellegr.

Total alkaloids in Stem bark- 2.5% and in Root bark- 5%.

N-Methyltryptamine was 90% of total. Leboeuf *et al.* 1977 **Rubiaceae**

Psychotria carthaginensis Jacq. ["*Rami appani*"; Culina Indians, Marcos. Collected 4 Sept 1968.] Traces of MMT Rivier & Lindgren 1972

Psychotria viridis Ruiz & Pavon ["*Rami appani*"; Culina Indians, Zapote. Collected 22 July 1968] Traces of MMT and 2-Methyl-1,2,3,4-tetrahydro-β-carbo-

line as minor alkaloids. Rivier & Lindgren 1972

Psychotria viridis Ruiz & Pavon ["*Kawa Kui*"; Sharanahua Indians, Marcos. Collected 7 October, 1968] MMT was major alkaloid in leaf [85% of 0.11% total alkaloids by dry wt.] DMT was absent [12% of 0.11% total] Rivier & Lindgren 1972

Rutaceae

Zanthoxylum arborescens Rose

0.002% in leaf (dry weight) Grina et al. 1982

Animals:

Gorgonaceae

Villagorgia rubra (A sea fan from New Caledonia) Espada *et al.* 1993

Paramuricea chamaeleon

30 mg. of N-Methyltryptamine isolated from 200 grams of coral

Cimino & DeStefano 1978

Occurrence in humans: Found in cerebrospinal fluid of some psychotics and some normal people by Corbett *et al.* 1978.

Oon & Rodnight 1977 thought they observed in psychotics but did not prove.

See Davis 1989 for many more references on the reported natural occurrences of N-methyltryptamine in humans:

MMT

Activity:

Hallucinogenic according to "*Recreational Drugs*" and Clarke's Second; neither cited **any** reference.

Inactive according to nearly everyone else.

Shulgin noted that he received "one report that the smoking of 50-100 mg gave visuals that lasted for maybe 15 seconds." Shulgin & Shulgin 1997

Reported Pharmacognosy:

Inhibits norepinephrine reuptake into rat brain synaptic vesicles: Slotkin *et al.* 1978

Lowers blood sugar. Liu et al. 1977

Inhibits growth of Crithidia fasciculata

(Trypanosomatidae) in brain heart infusion at 6 µg/ml. Filho & Gilbert 1975

Metabolism:

Erspamer 1954

Erspamer 1955 reported that rats excrete largely as IAUA (Indoleaceturic acid) with lesser amounts of IAA (Indole acetic acid).

MAO kinetic constants: Suzuki et al. 1981

Metabolized by both MAO-A & MAO-B. Suzuki et al. 1981



Anadenanthera colubrina var. cebil seeds

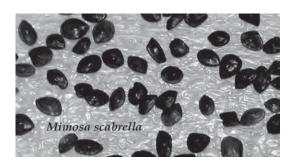


Anadenanthera colubrina seeds The seed imageson this page are not to scale.

Toxicity: No data located

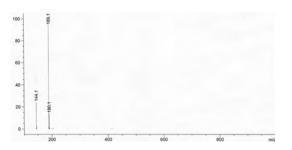
For further reading, see:

Shulgin & Shulgin 1997; entry #50 (pp. 573-574)

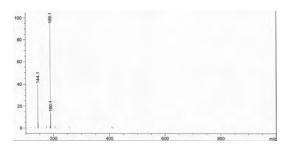




Anadenanthera peregrina seeds



GC-MS of DMT (above) & Mimosa extract (below)



DMT

N,N-Dimethyltryptamine

3-[2-(N,N-Dimethylamino)ethyl]-indole, 9CI; 3-[2-(N,N-Dimethyl)aminoethyl]-indole; N,N-Dimethyl-1H-indole-3-ethanamine; Nigerine; Amino-N,N-dimethyltryptamine; N,N-Dimethyltryptamin / Dimethyltryptamin / Nigerin (Gr); N,N-Diméthyltryptamine/ Nigérine (Fr.); N,N-Dimetiltriptamina/ Nigerina (It/ Pr/ Sp); Dimetiltriptaminas (Lithuania); Desoxybufotenine; DMT (Roche-England); DMTPA; D; Dmitri

WLN: T56 BMJ D2N1&1 Hayward: 6R4Y5L(CCNM2)=LNHY Usdin & Efron 1979

Chemical Abstracts Service Registry Number: 61507 (Sax 6th ed) # 61-50-7 Grina et al. 1982 CA Reg. No.: 004382507 Usdin & Efron 1979 NIOSH #NL7350000 Sax 1984 NX#00740 Sax cited Chemical Systems Laboratory.

Schedule 1 Drug (Federal Controlled Substances Act; registry number 7435) Shulgin 1976

 $C_{12}H_{16}N_{2}$

MW 188.26 Ott 1993/ Merck 9th entry #3253 (#3251 in 11th Edition) / Bergin et al. 1968 MW 188.272 Southon & Buckingham 1989 MW 188.30 Sax 1984 & Clarke's 1986

Elemental analysis: C 76.55%, H 8.57%, N 14.88%

Free base

mp 39-44° Grina et al. 1982

- mp 44° Base recrystallized readily from light petroleum. Rovelli & Vaughn 1967
- mp 44-46° After first vacuum distillation / 44.6-44.8° after second vacuum distillation. Hochstein & Paradies 1957 mp 44-47º Clarke's 1986.

mp 44.6-46.8° Crystals Merck 9th

- mp 44.6-44.8° Crystals from ethanol Ott 1993 & 1996
- mp 45° (crystallized from light petroleum) (reference sample 46.5°) Culvenor et al. 1964
- mp 45.5-46.8° Meckes-Lozoya et al. 1990

mp 45.8-46.8° (as Nigerine/ using xylene; "xilol") Goncalves de Lima 1946

- mp 46-47° When initially eluted from alumina and crystallized from hexane. mp 57.5-58.5° When above was seeded with "authentic" DMT (mp 56-58.5°).
- Fitzgerald & Sioumis 1965

mp 47° Manske 1931

- mp 47° Off-white solid. Shulgin & Shulgin 1997
- mp 47° Ghosal & Mukherjee 1964
- mp 47-49° (from hexane); mp 71-73° (reXtl. from hexane) after seeding with "authentic" DMT (mp 73-74°- obtained from Dr. M.E. Speeter of the Upjohn Company). Fish et al. 1956

mp 48-49° Boit 1961: p.478 mp 48-49° (from hexane containing a little ethyl acetate) Pachter et al. 1959 mp 48.5-49° (petroleum ether: colorless needles) (mp 48-49º (n-hexane: colorless prisms) Ueno et al. 1978 mp 49° Colorless crystals from benzene-hexane [N,Ndimethyl-¹⁴C-tryptamine labeled at 2 position] Baxter & Slavtor 1972 mp 49° Colorless prisms from petroleum ether. Morimoto & Matsumoto 1966 and Morimoto & Oshio 1965 mp 49-50° Shulgin 1976 mp 53.5-57.5° (from hexane) Arthur et al. 1967 mp 58.2° Transparent acicular crystals (Supplied by B. Holmstedt.) Bergin et al. 1968 mp 65.5° Prisms from Schuka Co, Germany. Falkenberg 1972 mp 67° (White crystals from hexane) Shulgin & Shulgin 1997 The product listed from another synthetic route, recrystallized from boiling hexane, showed mp 67-68°.

mp 48-49° (39-44°) Southon & Buckingham 1989

Shulgin & Shulgin 1997 mention the listing of melting points of 58-60° and 64-67° for material offered by the Aldrich Chemical Company.

Vacuum Distillations reported:

170° at 0.01 mm Hochstein & Paradies 1957 80-135° at 0.03 mm Corothie & Nakano 1969 130-140° at 0.1 mm Shulgin & Shulgin 1997

Free base:

Clear thick viscous oil, colorless when pure, often encountered vellow or reddish. Crystallizing colorless or yellowish. (Needles from heptane.) PlPhInc White, pungent smelling, crystalline solid. Shulgin 1976 Transparent acicular crystals (monoclinic) (Supplied by Bo Holmstedt.) Bergin et al. 1968 [Ed: Acicular & monoclinic would seem to be contradictory descriptions.] Density: 1.096 g/cm3 (Observed)/ 1.088 g/cm3 (Calculated) Bergin et al. 1968 Transparent colorless hexagonal prisms (monoclinic) Falkenberg 1972 Crystallized in two separate crystal modifications, both monoclinic. Densities: 1.080 and 1.074 g/cm3 Free base crystals were obtained from Schuka Co, Germany. Falkenberg 1972 3 different monoclinic crystal forms from anhydrous ether, *n*-hexane & anhydrous ether-*n*-hexane (1:2:8). Sand (pers. comm.) Crystals of the base were "quite hygroscopic" Falkenberg 1972 Piezoluminescent when very pure.

Sand & van der Heyden (pers. comm.)

DMT freebase solubility

- Freely soluble in dilute acids (acetic, citric or mineral), [Forms salts with these acids.]
- Soluble in alcohols (methanol or butanol often used experimentally), acetone, chloroform, methylene chloride, xylene, hexane, heptane, ether, carbon disulfide, dioxane, pyridine, tetrahydrofuran, toluene and other organic solvents. Also in acidified alcohols (forms salts)
- Manske 1931 says "It is extremely soluble in all organic solvents with the exception of petroleum ether." [While not as soluble in this solvent, DMT can be dissolved in hot petroleum ether and recrystallized by cooling. DeKorne & others used this solvent to isolate DMT: under the brandname of Coleman Fuel.]

Manske noted extremely soluble in ether even at 0° C.

Insoluble in water. ("*Insoluble*" means less than a gram per liter will dissolve. 45 mg in 50 ml, as encountered in ayahuasca brew is less than a gm/l.)

Chloroform-Water Partition coefficient: 1.7

Gessner & Page 1962 see also Glennon et al. 1979

pKa 8.68 (ethanol-water) Merck 9th

Hydrochloride salt is hygroscopic. Shulgin 1976

- Manske 1931 could not get it to crystallize. Described as a pale yellow resin.
- Shulgin & Shulgin 1997 reported similar problems.
- mp 165-167° (Formed from the free base dissolved in anhydrous ether and then treated with anhydrous hydrogen chloride gas. Recrystallized from benzene-methanol) Shulgin & Shulgin 1997; mentioning a report in the literature.
- mp 165-168° White water soluble crystalline powder. Clarke's 1986.

Picrate

 $[C_{12}H_{16}N_{2}]C_{6}H_{3}N_{3}O_{7}$ MW 417.4

Elemental Analysis of picrate:

Lit.: C, 51.80; H, 4.59; N, 16.78

- Exp.: C, 51.59 [51.48]; H, 4.35 [4.63]; N, 16.74 [16.73] Morimoto & Matsumoto 1966 [Morimoto & Oshio 1965]
- mp 160-168° (from acetone) Corothie & Nakano 1969
- mp 166° dec. Yellow prisms from methanol. Morimoto & Oshio 1965
- mp 166-167° (Yellowish-orange needles from methanolwater.) Ghosal et al. 1971
- mp 166-168° (Yellow picrate from ethanol) Ghosal & Banerjee 1969
- mp 167-167.5° (Orange crystals from ethanol) Culvenor *et al.* 1964
- mp 167-168° (Yellow needles from methanol-Water (1:4) Ueno *et al.* 1978

- mp 167° Pale yellow slender needles. After recrystallization mp 168°. Manske 1931
- mp 168° (Yellow picrate from ethanol.) Banerjee & Ghosal 1969
- mp 168° (From ethanol) Ghosal et al. 1969
- mp 168° Ghosal & Mukherjee 1964
- mp 168-169° Iacobucci & Rúveda 1964 (DMT from bark of *Piptadenia macrocarpa*)
- mp 168-170° (Crude. After 3 recrystallizations from benzene first crop showed mp 171-172°) Pachter *et al.* 1959
- mp 168-170° (Yellow picrate from methanol.) Wassel *et al.* 1985
- mp 168-170° Marini-Bettòlo et al. 1964)
- mp 169° (Yellow needles) also as orange crystals from ethanol mp 167-167.5° (reference sample mp 168.5°) Culvenor *et al.* 1964
- mp 169° Yellow prisms from methanol. Morimoto & Matsumoto 1966
- mp 169° Yellow crystals. Showed "typical" color change during recrystallization.
- Rovelli & Vaughan 1967
- mp 169.5-170.5° Ott 1993 & 1996 and Merck 9th
- mp 169.5-170.5° Hochstein & Paradies 1957
- mp 170° Boit 1961
- mp 170-171° Picrate from ethanol solution separated as red crystals.
- When recrystallized, separated as a mixture of red crystals (mp 171-171.5°) and yellow crystals (mp 170-171°). Further recrystallization yielded only yellow crystals, Fitzgerald & Sioumis 1965
- mp 170-171° Wahba Khalil & Elkheir 1975
- mp 171-172° Yellow (stable) or red (metastable) crystals. Southon & Buckingham 1989
- mp 171-172° Shulgin 1976; also Iacobucci & Rúveda 1964 (DMT from seed pods of *Piptadenia macrocarpa*)

Methiodide

[C₁₂H₁₆N₂] CH₂I MW 330.2

Elemental Analysis of Methiodide: Lit.: C, 47.29; H, 5.80; N, 8.48 Exp.: C, 47.58; H, 5.63; N, 8.30 Morimoto & Matsumoto 1966

- mp 197°"*characteristic micaceous plates*" from methanol. Manske 1931
- mp 197-198° (from acetone) Corothie & Nakano 1969
- Colorless needles from acetone-methanol-ether 207° Morimoto & Matsumoto 1966.
- mp 215-216° Shulgin 1976 & Pachter et al. 1959
- mp 215-216° Southon & Buckingham 1989 & Boit 1961
- mp 216° (Colorless needles from acetone-methanol) Ghosal *et al.* 1971
- mp 216-217° Ott 1993 & 1996 and Merck 9th

DMT

Fumarate

mp 152-152.5° Ott 1993 & 1996

Hydrobromide

mw 269.19 mp 114.5° Transparent light yellow prisms (orthorhombic) (From methanol-ether) Falkenberg 1972 (Unstable in solution)

Oxalate

 $[C_{12}H_{16}N_{2}]C_{2}H_{2}O_{4}$ MW of oxalate 278.3

Elemental Analysis of oxalate: Lit.: C, 60.42; H, 6.52; N, 10.07 Exp.: C, 60.61; H, 6.54; N, 10.06

Colorless needles from methanol 147° Morimoto & Matsumoto 1966

Sulfosalicylate

CA reg. No. 101831-88-3 NIOSH # NL 7450000 Sax & Lewis 7th ed. Entry #DPG000, page 1346. Cited 1970 RPTOAN 33: 180 [CODEN]

Acetate

Weakly basic and chloroform soluble. Ghosal *et al.* 1969 & 1971

HFB derivative

Oil MW 384 Preparation & isolation Benington *et al.* 1975

Assays for DMT:

Usdin & Efron 1979; #384 cited: Der Marderosian *et al.* 1968 Clarke 1969 Udenfriend 1969 Clarke's Second (1986) cited: Walker & Mandell 1979 and Räisänen & Kärrkkïnen 1979

An analytical procedure used in FDA labs:

Using tlc (no heat for drying applied samples) with ammoniaalcohol (1:4), let develop for 10 cm, air dry without heat for around 5 minutes and spray with formaldehyde (40%)-hydrochloric acid (1+3)-alcohol (10:10:20). Heat plate at 100° C for 5-7 minutes.

Yellowish brown spots under long wave UV (~3660 Å) fluoresces in yellow-orange-green region.

Rf is around 0.60.

Sensitivity is 0.4 µg.

- DET behaves similarly. (Rf 0.45)
- Martin & Alexander 1968

Colorimetric reagents: See pages 142-143

TLC & PC: see Rf table p. 169-176

Column chromatography:

DMT (from *Vepris ampody*) eluted from column of alumina (Merck Act. II-III) with benzene-hexane 50:50 Kan-Fan *et al.* 1970 See more in the section entitled "*Abstracted Isolations*"

HPLC:

Balandrin *et al.* 1978
Kysilka & Wurst 1988
Kysilka *et al.* 1985
Verpoorte & Svendsen 1983 (Retention times of DMT and its TMS derivative: p. 175; relative retention times compared to tryptamine: p 155).

GLC:

Christian *et al.*GLC of HFB derivative: Benington *et al.*Christian *et al.*Vessman *et al.*

GC:

Holmstedt 1965
Retention times for Amine 220, CHDMS and DEGS columns: See Audette *et al.* 1969
Verpoorte & Svendsen 1983: Retention times (DMT and TMS derivative: p. 175; relative retention times compared to tryptamine: p 155). Verpoorte & Svendsen 1983

Electrophoresis:

In paper electrophoresis (in a sodium carbonate buffer) it was determined that DMT had a mobility of 4.4 cm. h⁻¹. kV⁻¹. Frahn & Illman 1973

[See Frahn & Mills 1964 & 1959 for a description of the apparatus]

UV:

Absorbs under UV at 254 nm [in morpholine-toluene (1:9)] Alliston *et al.* 1971

Clarke's Second gives:

"Aqueous acid-279 nm (A¹, =327 a), 288 nm." Includes graph of UV Spectrum. page 554. λ_{max} 222 nm, (log ε 4.48), 277(3.77) and 288(3.75) Ghosal *et* al. 1969 λ_{max} 222 -224, 274 & 294 nm Banerjee & Ghosal 1969 λ_{max} 222, 277, 287 & 294 nm Ghosal & Banerjee 1969 λmax 274, 283, 291 mμ (reference material) 275, 283, 291 mμ (isolated material). Fish et al. 1955 λmax (CH₃OH): 220, 280, 290 (= 5500, 5600, 5000) De Moraes et al. 1990 λ_{\max}^{EtOH} : 226, 275 (sh), 279, 284, 293 nm. Grina *et al.* 1982 λ_{\max}^{2} 276, 282 and 290 nm $\lambda_{_{min}}278$ and 287 nm Martin & Alexander 1968 λmax 275, 219 nm (0.1N NaOH) λmax 290, 276, 282 nm (EtOH) Sunshine 1981

Also see Morimoto & Oshio 1965 (graphic)

 λ_{max} of Xanthydrol reactive product (CHCl₃): 510 nm λ_{min} of Xanthydrol reactive product (CHCl₃): 400 nm Gander *et al.* 1976

Fluorescence:

- *"Strongly fluorescent"* in spectrofluorometric assay [Ed.: Note that fluorescence, while native, is not in the visible spectrum]
- $0.1N\,H_2SO_4\,\lambda_{ex}\,279\,nm/\,\lambda_{em}\,352\,nm$ (small amount of photodecomposition)
- 0.001N HCl λ_{ex} 279 nm/ λ_{em} 352 nm (Optimum solvent choice)

0.1M Phosphate buffer (pH 7) $\lambda_{ex} 278 \text{ nm}/ \lambda_{em} 353 \text{ m}\mu$ 1N NH₄OH $\lambda_{ex} 280 \text{ m}\mu/ \lambda_{em} 362 \text{ nm}$ 1N NaOH $\lambda_{ex} 283 \text{ m}\mu/ \lambda_{em} 423 \text{ nm}$ Graphic portrayal of fluorescence spectra. Gillespie 1969 Fluorescence spectrum at pH 7.4: Activation: 295 nm Emission: 370 nm Gessner & Page 1962 Fluorescence maxima: Excitation: 283 mµ; Emission 350 mµ. Fish *et al.* 1955 Excitation: 280 nm; Emission 360 nm. Shah & Hedden 1978

DMT (DET & DPT are all identical) Fluoresces at 340 nm with excitation at 290 nm Slits (Em./Ex.) 10/10

Filter 310 nm Limit 0.5 mcgm Methanol as solvent on Perkin-Elmer MPF-2A fluorescence spectrophotometer DeZan *et al.* 1971

See also:

Bridges & Williams 1968 Holmstedt 1965 William & Bridges 1964

IR:

Clarke's Second: page 554 Grina *et al.* 1982 Morimoto & Oshio 1965 (graphic) Shulgin & Shulgin 1997: (cm⁻¹): 732, 740, 811, 859, 1011, 1037, 1110, 1171

Mass Spectra:

Bellman 1968 [m/e 44, 58, 77, 89, 112, 115, 130, 143 and 188.] Includes graphics.
Clarke's 1986: page 554. [m/z 58, 188, 130, 59, 42, 143, 129, 115]
Holmstedt & Lindgren 1967 [m/e 58 (base peak), 103, 115, 130, 143, 188 (M+)]
Williams *et al.* 1971 [m/e 58, 103, 115, 130. 143, 188]
See also
Couch & Williams 1972 &
Crouch *et al.* 1992
EIMS: Grina *et al.* (1982)
EI-MS and CI-MS:

Shulgin & Shulgin 1997: page 415 (EI-MS: $(m/z) C_3 H_8 N^+$ 58 (100%); Indolemethylene⁺ 130 (10%); parent ion 188 (4%) (CI-MS (with NH₃) M⁺1 at 189 and fragment at 166) Thompson *et al.* 1987 MS of DMT-CNTNF complex: Heacock & Forrest 1973 MS of HFB derivative: Gupta *et al.* 1979 MS of TMS derivative: Narasimhachari *et al.* 1971 Räisänen & Kärrkkïnen 1979 Walker & Mandell 1979

GC-MS: (see p. 222) De Moraes *et al.* 1990

NMR:

Cohen *et al.* 1960 De Moraes *et al.* 1990 Grina *et al.* 1982 Morimoto & Oshio 1965 (graphic) ¹H NMR Torres & Repke 1996

Crystal and molecular structure:

Bergin *et al.* 1968 Falkenberg 1972

Synthesis:

- First synthesized in Manske 1931 See also: Hoshino & Kotake 1935 Fish *et al.* 1956 [Requires lithium aluminum hydride] Shulgin & Shulgin 1997: pp. 412-421, entry #6. [Synthetic
- routes: pages 412-415.]
- An interesting modification can also be found in Tymiak *et al.* 1985 in their synthesis of 5-bromo-DMT which yielded a 1:1 mixture of 5-bromo-DMT and DMT. [This approach requires LAH.]
- 5-Bromo-DMT and 5,6-dibromo-DMT can both readily be hydrogenated to DMT.
- Szara 1956 used the method of Speeter & Anthony to produce DMT for human bioassay. See Speeter & Anthony 1954
- Baxter & Slaytor 1972 includes a synthesis of radiolabeled ¹⁴C-DMT. [as N,N,-

Dimethyl-¹⁴C-tryptamine, labeled at 2 position.] A usual approach.

Another route would be to use the enzyme NMT (N-Methyl Transferase) to convert tryptamine (or Nmethyltryptamine) to DMT.

See Axelrod 1962 & Mack & Slaytor 1979

- For a discussion of what is involved in this conversion; called by Appleseed, "three step synthesis of DMT from tryptophan", See Mack et al. 1988
- Harnessing these enzyme through genetically engineered microbes, as is done routinely for other alkaloids, would be a simple matter for anyone with industrial level funding. Once set up; inexpensive tonnage production is realistic.
- Forms DMT-N-oxide rather easily during extensive experimental manipulations of solutions exposed to air. Fish *et al.* 1955 See after DMT-N-oxide for conversion route.

DMT

Isolations:

- First isolation was as "nigerine" from Mimosa hostilis rootbark. Gonçalves de Lima 1946
- First isolated from plants (and identified as DMT) in 1955 by Fish *et al.* from *Anadenanthera colubrina* var. *cebil* (as *Piptadenia macrocarpa*) and *Anadenanthera peregrina* (as *Piptadenia peregrina*).
- (DMT-N-oxide, bufotenine and bufotenine-N-oxide in the seeds and DMT in the pods.)
- This paper is what stimulated Szara to begin evaluating DMT in himself and friends "courageous enough to volunteer."
- At that time the activity of the snuffs was thought due to Bufotenine and DMT was of unknown pharmacological action.
- Demonstrated hallucinogenic in 1956 by Szara.
- After initially separating their crude alkaloid fraction, Hochstein & Paradies 1957 purified DMT by vacuum distilling at 170° at 0.01 mm. The product was a colorless oil which crystallized spontaneously on standing. They distilled a second time and obtained a product with a tighter mp.
- Corothie & Nakano 1969 used a bath temperature of 80-135° and distilled at 0.03 mm to obtain a slightly yellowish oil from the crude alkaloid they eluted from alumina.
- Mulga reported successful precipitation by dissolving a crude extract residue in a hot petroleum solvent and then freezing.
- Shulgin & Shulgin 1997 vacuum distilled at 130-140° at 0.1 mm to yield a colorless oil that crystallized spontaneously.
- For an early piece on molecular distillation with a plan that any good glass worker and lab technician could master, see Hickman 1937
- See more elsewhere here for various approaches used by multiple workers

Reported Occurrences of DMT: Plants:

Alaricaceae

- *Ecklonia maxima* (Osbeck) Papenfuss Unspecified amount in "*Kelpak*": a commercial seaweed (brown algae) concentrate. Crouch *et al.* 1992. Questioned: Not reproducable for other workers. Tao Jones 2002 (pers. comm.)
 - Agaricaceae

Amanita citrina Gray

- Tyler & Gröger 1964 (German specimens) Traces identified chromatographically.
 - Acanthaceae

Justicia pectoralis

In leaf. Shulgin & Shulgin 1997

Justicia pectoralis

Potential presence of DMT.

Yanonamo snuff prepared from this plant was found to be rich in DMT by McKenna *et al.* 1984b

Justicia pectoralis var. stenophylla Lnd.

DMT in leaf

Schultes & Holmstedt 1968

McKenna et al. 1984b unable to confirm.

Justicia pectoralis var. stenophylla

leaf 31 August 1994 harvest

Bands corresponding to DMT, MMT and another at high Rf.

tlc by J.Appleseed

Aizoaceae

Delosperma spp.

Raffauf 1970 cited unpublished work done by Smith, Kline & French Laboratories.

Raffauf was also cited by both Rivier & Pilet 1971 and Deulofeu 1973.

Smith 1977b cited Rivier & Pilet 1971 and Deulofeu 1973. Assays by J. Appleseed 1994-1996:

Delosperma acuminatum Alicedale

Present in undetermined amount. 5 positive assays over a 15 month period. (Xanthydrol-1 and Ehrlich's-4) (Sept., Nov, Dec.) tlc by Johnny Appleseed (1993-5) Not observed in May assay.

Delosperma cooperi

Sept., Nov. and Dec. assays. 4 positives over a 25 month period. (Xanthydrol-2 and Ehrlich's-2) (Sasha was unable to confirm this using GC-MS on material purchased from Home Depot in Spring.)

Delosperma ecklonis

Nov. 1994, 1995 (2, one year apart) also Sept.

1996 (1-Ehrlich's and 2- Xanthydrol) The first time it was erroneously labeled *D. lydenbergense*

Delosperma esterhuyseniae

Nov. 1995 assay. Faint (Xanthydrol)

Delosperma harazianum Audhali Plateau, Yemen

Nov. 1995 assay. (Xanthydrol) Delosperma harazianum Shibam

Nov. 1995 assay. Faint (Xanthydrol)

Delosperma hirtum

Nov. 1995 and Dec. 1994 assays. Weak DMT band. (Xanthydrol and Ehrlich's)

Delosperma klinghardiana

Sept. 1996 assay. (Xanthydrol) Co-occurrence with 5-MeO. Delosperma pageanum

Dec. 1994 (Ehrlichs) and August 1995 harvest. Good DMT (co-occurring with 5-MeO-DMT) band (Dec. 1994 harvest; same material retested with Xanthydrol in 1996) Cooccurrence also observed in August and December 1995 harvests assayed in Sept 1996 (Xanthydrol)

Delosperma pergamentaceum Numees

Nov. 1995 assay faint (not present in May assay) (Xanthydrol)

Sept 1996 assay decent. Xanthydrol. [No alkaloid observed in Sept 1996 *D. pergamentaceum* Rooilepel.]

Delosperma tradescantioides

Nov. and Dec. 1994 assay Faint (or was it 5-MeO-DMT?) (Ehrlich's)

Graminae

Arundo donax L. In 2 week old flowers. Ghosal *et al.* 1971

In leaf and rhizome. 40 mg from 700 grams of rhizome. Ghosal *et al.* 1969

20 mg from 200 grams of dry plant. (Versus 520 mg of gramine from the same material.) Dutta & Ghosal 1967 DMT in leaf, rhizome and flower.

In numerous assays of this plant we (Appleseed & Trout) observed DMT only once. (New white, skinny (> 2 mm dia.) roots. We repeatedly observed numerous indolic alkaloids (in rhi-Culvenor et al. 1964 zome & growing shoots) but, with this one exception, never Phalaris aquatica DMT. Bromus spp. (Brome grass) DMT appeared potentially present in at least one Bromus sp. (B. breviaristatus) and up to 3 local species of unidentified "Wild Rye" or "Winter Rye" (yes this DOES includes the material used to overseed southern US lawns during winter) for which positive ID is pending. 1996 tlc by J. Appleseed. [The Bromus species was grown by Giorgio Samorini from seeds provided by K. Trout and identified at seed maturity by Dr. Francesco Festi in 1999] Digitaria spp. (Crab grass) We have detected DMT as potentially present in at least one In seedlings. local species; probably D. sanguinalis. Positive identifica-Mack et al. 1988 tion pending. 1996 co-tlc by J. Appleseed. *Hierochloe odorata (Sweet-grass)* Faint band co-chromatographing with DMT. Nov. 1995 tlc with Xanthydrol spray. J. Appleseed. Most references and analysis on Phalaris have been omitted as they are not only voluminous but variable and often ambiguous or contradictory. Please see "Notes on the Genus Phalaris", forthcoming by Trout & friends. An excellent review and summation can be found in the amazing works of Festi & Samorini listed in our references; their reference list almost summarizes all work ever done on the subject. Major base An incomplete overview of Phalaris: Phalaris aquatica L. = Phalaris tuberosa L. Phalaris aquatica L. Major base Present in some clones and varieties.) Frahn & O'Keefe 1971 DMT in leaf. Baxter & Slaytor 1972; Culvenor et al. 1964; Phalaris aquatica JLF Frahn & Illman (1973); Moore et al. 1967; Mulvena & Slaytor Major base 1982; Oram & Williams 1967 and many others. Phalaris aquatica Clone #R16 "large" amount of DMT co-occurring with "trace" amount of 2-Methyl-1,2,3,4-tetrahydro-\beta-carboline. [Clone, designated 369-3, originating with U.S. Regional Pasture Research Laboratory, University Park, Pennsylvania] nate.] Clone #R38 "trace" amount of DMT co-occurring with "large" amount of 2-Methyl-1,2,3,4-tetrahydro-β-carboline. [From Major base "highly diverse source population used in plant breeding and genetic studies at the University of Minnesota, Department of Agronomy and Plant Genetics"] Major base Clone #R504 "large" amount of DMT co-occurring with "intermediate" amounts of hordenine. [Same source as R38] Gander et al. 1976 DMT was not present in all clones they examined (3 out of 12) Phalaris aquatica DMT Mack et al. 1988

Phalaris aquatica

A major alkaloid in all samples he examined [It is not present in all *P. tuberosa* strains]

The usual major alkaloid. Frahn & Illman 1973

Phalaris aquatica var. AQ-1

Higher levels of DMT than are known from any other species Samorini 1992 Personal communication with J. Ott Festi & Samorini 1994 reported in excess of 1% from grass grown in Italy.

Phalaris aquatica cv. AQ-1

Extremely strong occurrence reported (HPLC). Festi & Samorini 1994b

Phalaris aquatica cv. Australian Commercial

- A major alkaloid in 7 day old seedlings.

Mulvena & Slaytor 1983

280 nmol / 100 seedlings

Mulvena & Slaytor 1983

Mature 0.1% dry weight

Baxter & Slaytor 1972

Fresh young seedlings.

- Baxter & Slaytor 1972
- Phalaris aquatica (Commercial)

Weak occurrence reported (HPLC). Festi & Samorini 1994b

Phalaris aquatica cv GB81

Frahn & O'Keefe 1971

- Phalaris aquatica cv "High alkaloid"

5-MeO-DMT & DMT in leaf 17 Sept. 1995 Assay. tlc by J. Appleseed 1995

Phalaris aquatica cv Killer (Killer Phalaris)

DMT was predominate alkaloid in Fall 1994 tlc. [Assays 25 June, 17 Sept., 2 Nov. 1995 showed 5-MeO to predomi-

Phalaris aquatica cv "Low alkaloid"

Frahn & O'Keefe 1971

Phalaris aquatica cv Seedmaster

Frahn & O'Keefe 1971

Phalaris aquatica cv. Sirocco

24 nmol of DMT/ 100 seedlings

Mulvena & Slaytor 1983.

Phalaris arundinacea L.

DMT in leaf and whole plant. Barnes et al. 1971; Culvenor et al. 1964; Gander et al. 1976; Marten et al. 1973; Williams et al. 1971. Present in SOME strains of P. arundinacea but not in most.

DMT

Phalaris arundinacea

Amounts not given. Detected by tlc in some Ottawa Synthetic cv

Woods & Clark 1971

Phalaris arundinacea (France)

Occurrence reported (HPLC).

Festi & Samorini 1994b

Phalaris brachystachys (Portugal)

Extremely strong occurrence reported (HPLC). [Sole alkaloid]

Festi & Samorini 1994b

Positive human bioassays of clones originating from Algeria and Greece has been reported. See DeKorne 1997

Appleseed did not detect in his tlc involving field trials of USDA seeds for PI 202676 or PI 231044 (He found 5-MeO-DMT instead)

Phalaris canariensis (Portugal) Occurrence reported (HPLC). Festi & Samorini 1994b

Phalaris canariensis

Reported in PI 415833 in Appleseed's tlc evaluation of field trials using USDA seeds. Also at lower levels in PI 284185.

Phalaris minor (Portugal) Traces reported (HPLC). Festi & Samorini 1994b

Phalaris paradoxa (Romania) Occurrence reported (HPLC). Festi & Samorini 1994b

Phalaris stenoptera (= *P. aquatica* var. *stenoptera*) Variable amounts. Festi & Samorini 1994)a cited Rendig et al. 1970 as finding 0-60 μ g/ml of expressed juice.

Phalaris truncata (France)

Weak occurrence reported (HPLC). Festi & Samorini 1994b

Phalaris tuberosa L. See as Phalaris aquatica

Phragmites australis (Cav.) Trin ex Steud [= Phragmites communis]

DMT in rhizome. No details of amount included. Wassel et al. 1985

tlc (as P. communis) by J. Appleseed showed it to be weak to absent.

Sorghum halepense [Johnson Grass, Aleppo Grass, Egyptian Millet, Grass Sorghum, Means Grass]

In rhizome but not leaves (see 5-MeO-DMT entry) Most samples run in tlc have shown low concentrations but one was very strong (Harvested August 1995: just above Pedernales River flood plain, west of Johnson Ranch, Stonewall, Texas). Needs 2nd party confirmation. J. Appleseed (1995 and 1996 tlc with Xanthydrol spray.)

Leguminosae Acacia albida

In leaf. Shulgin & Shulgin 1997

Twigs showed faint positive in tlc with Xanthydrol spray. Co-occurrence with suspected 5-MeO-DMT.

Leaf assay 25 August 1994 was negative. J. Appleseed Acacia angustissima

Trace amounts tentatively observed in roots (Unconfirmed) Mar. 1995 tlc by Appleseed. Not observed in second assay.

Acacia baileyana F.Muell.

Trace amounts in seeds. Unconfirmed. tlc by J. Appleseed 1995.

Acacia confusa Merrill [=A. richei A.Grav]

1.15% in (dried?) root bark Liu et al. 1977

0.01% in dry stem-bark Arthur et al. 1967

Acacia cornigera

Presence of DMT in bark indicated but details lacking. Needs confirmation Rätsch 1998.

Acacia difformis

Traces in leaf. Xanthydrol. Both pinnate leaves and phyllodes tested separately. 2 year old plant. Sept. 1996.

Acacia laeta In leaf. In error?

I suspect this to be a misreading of Wahba Khalil & Elkheir 1975 who reported negative results.

Acacia longifolia

Some of the claims appearing in the literature are in error. These *probably* arose from the unfortunate combining of every chemical analysis for all varieties of this species as if they were one and the same (from a tabular summary in a now dated overview and review of herbage biochemistry).

DMT or another substituted tryptamine may indeed eventually be found in the flowers and/or tips but we can locate no conclusive published analysis, only White's intriguing lab observations concerning an unidentified alkaloid(s) that was not tryptamine but reacted intensely with Ehrlich's reagent.

Daniel Siebert (pers comm.) examined this species (roadside plantings in CA) and observed trace amounts of DMT in the aerial parts but did not publish this information.

Acacia maidenii F.vonMueller

0.36% in dry bark. Fitzgerald & Sioumis 1965 Roots- co-tlc with DMT (with Ehrlichs spray reagent) (with MMT) 1994 J.Appleseed (2 year old seed grown plants) Leaf- positive for suspected DMT 17 August 1996. Positive but co-occurring with suspected 5-MeO-DMT 27 October 1995. Co-tlc with Xanthydrol spray. J. Appleseed

Mulga mentions reports of weak results in some isolation attempts.

Acacia mellifera

In leaf. In error?

Same comments as below under A. sieberiana.

Acacia nubica Benth.

0.0016% in dry leaf Wahba Khalil & Elkheir 1975

Acacia obtusifolia A.Cunn. [=Acacia intertexta]

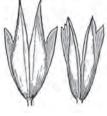
Originally discovered to be present based on serendipitous isolations and multiple human bioassays using misidentified stem-bark. Alkaloid content and composition seasonal; total alkaloid 0.1-0.7% dry wt.

Correct identification was by Mulga.

Later determined by hplc-ms to contain over 90% DMT cooccuring with small amounts of a simple tryptamine (no positive ID- suspected to be either tryptamine or Nmethyltryptamine) and small amounts of leptocladine (Nmethyltetrahydroharman).

From Mulga at http://www.lycaeum.org/drugs/plants/ tryptamines/acacia/species htm & Trout's Notes on Acacias 2004.





Phalaris paradoxa

modified from

Robbins et al. 1951

Acacia phlebophylla F.Muell. (= Acacia longifolia var. phlebophylla (F.Muell.) F.Muell. = Acacia sophorae var. montana F.Muell.)

0.3% in dry leaf. Rovelli & Vaughan 1967

It might be commented that this species sheds its phyllodes annually and said dead dryphyllodes retain their potency.

It might also be commented that this species might be of hybridogenous origin between *A. alpina* and *A. dallachiana* suggesting those two species might be worth looking at.

Acacia polyacantha Willd subsp. campylacantha (Hochst. ex A.Rich) (=Acacia caffra var. campylacantha =Acacia campylacantha =Acacia catechu ssp. suma var. campylacantha)

0.004% in dry leaf. Wahba Khalil & Elkheir 1975

Acacia rigidula Bentham

323.8 ppm in early spring/ 568.4 ppm in late fall. Fresh leaves, petioles & tender twigs. Clement *et al.* 1998 (Some questions exist about disturbing discrepancies contained within this paper as concerns their reference standards.)

Acacia senegal (L.) Willd

0.003% in dry leaf Wahba Khalil & Elkheir 1975

Acacia sieberiana In leaf. In error?

I suspect this to be a misreading of Wahba Khalil & Elkheir 1975 I also suspect that DMT may eventually be found in the roots of this species but thispresently lacks support. *Acacia seval*

In leaf. In error?

Same comments as above under A. sieberiana.

Acacia simplicifolia Schinz & Guillaumin (=Acacia simplex?) (Hortus and Ott refer to as Acacia simplicifolia Druce)

0.81% in bark and 0.007% in twigs. Poupat *et al.* 1976

Acacia sophorae (Labil.) R.Br.

Claimed to have been reported by an unidentified source (see Mulga at Lycaeum address above under *A. obtusifolia*. No references available or published assay located. Needs confirmation.

A strongly suspected species in need of work.

Acacia tortilis

Is **erroneously** included by several sources.

Evidently they misread Wahba Khalil & Elkheir's report of detecting **no alkaloids** in the dried leaves of this species. Wahba Khalil & Elkheir's presentation can be easily misread. J. Appleseed's 1994 assay of aerial parts also detected no alkaloids.

Acacia victoriae

Aerial parts of 1 year old seed grown material. (Unconfirmed) Good banding. 1995 tlc by J. Appleseed

Anadenanthera species. [Biocca Cocco, 1963; Upper Orinoco, El Platanal, Machekototeri]

Seeds

0.001% DMT [1 mg/ 100 gm; Sole alkaloid.] Schultes *et al.* 1977

Anadenanthera species. [Biocca Cocco, 1965; Upper Orinoco, Rio Ocamo]

Seeds

0.006% DMT [6 mg/ 100 gm; Sole alkaloid.] Schultes *et al.* 1977 Anadenanthera species. [G. Seitz, 1965]

Seeds

0.038% DMT [38 mg of total alkaloid/ 100 gm dry. Sole alkaloid]

Seedlings

0.028% DMT [96% of 29 mg of total alkaloid/ 100 gm dry.]

Schultes et al. 1977

Anadenanthera species. [As Piptadenia sp.; Caspar, 1964; Guaporé, Brazil; Tupari.]

Seeds: **0.002%** DMT- [15% of 13 mg of total alkaloid/ 100 gm] Schultes *et al.* 1977

Anadenanthera colubrina

Conflicting reports. Most accounts have found only bufotenine in the seeds but several reports exist claiming the presence of DMT and/or 5-MeO-DMT.

Torres *et al.* 1991 reported the detection of all three in **snuff** powder recovered from archaeological sites in Argentina believed to have been derived from *A. colubrina* seeds. Both *A. colubrina* and *A. colubrina* var. *cebil* occur in Argentina. While it is not clear which Torres and coworkers referred to; the latter is implied. No analysis of seeds or verifiable plant material reported in Torres *et al.* 1991.

Anadenanthera colubrina (Lellozo) Brennan var. cebil (Grisebach) Altschul (= Piptadenia macrocarpa)

Sole alkaloid in pods. Not detected in seeds: Fish *et al.* 1955. Material from both Florida and Brazil were used. Florida material gave weak results. [Reported a total alkaloid concentration of ~ 1.5 -2.0% in seeds. Actual amount of pure alkaloids not gi ven. Pods sometimes were weaker but contained only DMT.]

DMT in both seeds and pods. Iacobucci & Rúveda 1964 (Major alkaloid or only alkaloid in seed-pod (conflicting statements); no amounts given. Argentinian plants used.)

Material collected in Argentina is said to have yielded only bufotenine from the seeds. (Personal communication with researcher requesting anonymity)

Anadenanthera colubrina var. Cebil

DMT 0.06% in seeds from Misión Wichi and 0.05% in pods from Salta but not detected in seed samples from Salta. Traces detected in bark from Cerro San Bernardo (All Argentina) Torres & Repke 1996

Anadenanthera excelsa Grisebach [Argentina]

DMT in seedpods (sole alkaloid present) Iacobucci & Rúveda 1964

Anadenanthera peregrina (L.) Spegazzini [No.24625; Origin: Boa Vista, Brazil]

Bark-0.0004% i.e. 0.42 mg/ 100 gm dry bark [1% of 42 mg. of total alkaloids/ 100 gm. of dry bark] [see 5-MeO-DMT entry]

Leaves- 0.0059% i.e. 5.88 mg/ 100 gm dry leaves [49% of 13 mg. of total alkaloids/ 100 gm. of dry leaves.

Agurell et al. 1969

Sole alkaloid in pods. Not detected in seeds: Fish *et al.* 1955. Material from both Puerto Rico and Brazil were used. Puerto Rican material gave variable results. [Reported a total alkaloid concentration of 1.6% in the seeds. No indication was given of the actual amount of pure alkaloids. The pods were weaker but contained only DMT.]

DMT

Seeds collected in Puerto Rico during 1948 DMT [with Bufotenine] Holmstedt & Lindgren 1967 Seeds collected in western Brazil in the Rio Branco region during 1953 DMT [with 5-MeO-DMT] Holmstedt & Lindgren 1967 Bark collected in Colombia during 1956 DMT [with MMT, 5-MeOMMT & 5-MeODMT] Holmstedt & Lindgren 1967 Anadenanthera peregrina [R.E. Schultes, S. von R. Altschul et B. Holmstedt, sin. num.; La Carolina, Barrio St. Just, near San Juan, Puerto Rico, December 1974. Same colony as Schultes 26363.1 Mature seeds collected in March 1975; hill behind El Comandante horse-racing track. 1975 analysis (5 months after collection): No quantification DMT- 19% of total alkaloid. [1977 analysis of same material could detect only bufotenine] Schultes et al. 1977 Anadenanthera peregrina [R.E. Schultes 26363; La Carolina, Barrio St. Just, near San Juan, Puerto Rico, Dec. 1972] Immature seeds collected December 1972 0.16% DMT [75% of 209 mg of total alkaloid/ 100 gm dry] Seedlings 0.001% DMT [4% of 25 mg of total alkaloid/ 100 gm dry] Pods without seeds 0.001% DMT [8% of 13 mg of total alkaloid/ 100 gm dry] Leaves 0.013% DMT [12% of 107 mg of total alkaloid/ 100 gm dry] Twigs 0.0019% DMT [5% of 38 mg of total alkaloid/ 100 gm drv] Bark [0.41% total alkaloid] 0.02% DMT [5% of 410 mg of total alkaloid/ 100 gm dry.] Roots [0.69% total alkaloid] 0.014% DMT [2% of 699 mg of total alkaloid/ 100 gm dry] Schultes et al. 1977 Anadenanthera peregrina. [R.E.Schultes 24625; Boa Vista, Brazil] Leaves **0.00637%** DMT [49% of 13 mg of total alk./ 100 gm dry] Bark **0.00042%** DMT- [1% of 42 mg of total alk./ 100 gm dry] Schultes et al. 1977 Anadenanthera peregrina. [Abbott Lab., 1948; San Juan, Puerto Rico] Seeds- 0.009% [9 mg of DMT/ 100 gm; Sole alkaloid.] Schultes et al. 1977 Anadenanthera peregrina. [J. Yde, 1964, H4685] Seedlings- 0.001% [1 mg of DMT/ 100 gm; Sole alkaloid.] Schultes et al. 1977 Snuff: "epena" DMT was a minor alkaloid. [5-MeO-DMT was the major] Obtained from the Waica by George Seitz. Bufotenine was

present as a minor component. Because of this, the claimed plant source (Virola) has been questioned. Holmstedt 1965 See comments elsewhere.

Snuff: "epena"

Yanoáma snuff prepared from Piptadenia peregrina. Marini-Bettòlo et al. 1964

Snuff: "epena"

Snuff prepared, by the Ma-hekodo-teri of the Rio Mavaca, from the seeds of an Anadenanthera species.

DMT [with DMT-N-oxide, Bufotenine and Bufotenine-Noxidel

Marini-Bettòlo et al. 1964

Snuff: "paricà" [Snuff as prepared by Piaroa Indians (collected 1955)]

DMT [with Bufotenine and 5-MeO-DMT]

Holmstedt & Lindgren 1967

Snuff: "vopo" [Snuff collected in Colombia (1956)] DMT [with Bufotenine and 5-MeO-DMT] Holmstedt & Lindgren 1967

Snuff: "yopo" [L.Persson, 1966; R. Miriti-Parana Caqueta, Colombia.]

0.016% DMT [16 mg / 100 gm; Sole alkaloid.] Schultes et al. 1977

Snuff [G. Baer, 1964; Brazil; Tupari.]

0.016% DMT [16 mg / 100 gm; Sole alkaloid.] Schultes et al. 1977

Caesalpinia pulcherrima

Strong band in mixed flowers and buds 26 Aug. 1995. tlc by J. Appleseed 1995 [co-occurring with suspected 5-MeO-DMT. Both strong.] An earlier flower sample (dead & dry) showed none.

Calliandra pentandra

Presence of DMT is claimed in Fericgla 1994 based on his bioassay of a Shuar ayahuasca using this as its admixture. Shulgin & Shulgin 1997, on pages 586 and 712, claimed only tetrahydroharmine was detected in this species but included no reference or details.

Desmanthus coolevi

~ 1/2 [] of *D. leptolobus*

tlc and isolation by J. Appleseed 1992.

Desmanthus illinoensis (Michx.) MacM.

0.34% in Root bark (dried) and 0.01% in Root wood (dried) Thompson et al. 1987 [Substantially less is usually encountered. Sometimes none.]

Desmanthus leptolobus

0.14% yield of alkaloid. Identified by Johnny Appleseed 1992. tlc also tested positive 1993- 1995.

Isolated and Bioassayed as pharmahoasca by J. Appleseed on 28 Nov., 1992.

Isolated from Central Texas material and bioassayed as partially crystalline free base. [Identity confirmed in bioassays by others] (1994)

Desmanthus velutinus

some tested +/ more tested -

tlc by J. Appleseed (1992)

Desmodium sp. (Wild local sp.; Austin, Tx.) Faint co-occurrence with suspected 5-MeO-DMT in aerial parts. tlc by J. Appleseed.

Major alkaloid in roots [0.087% by dry weight. Ed.: Procedure likely resulted in some loss. If all of their crude alkaloid and all of their picrate had been used they would have obtained 1.46 gm from 1.6 kg dry roots. i.e. ~ 50 gm of roots for a 45 mg equivalency.] Ueno *et al.* 1978

Minor alkaloid in stem [0.0035%; 380 mg from 10.75 gm of stems] Ueno *et al.* 1978

Desmodium gangeticum DC

Aerial parts [? gm. of thick oil + 0.41 gm (latter as chloroform soluble acetate) obtained from 1 kg of fresh wet material.] Banerjee & Ghosal 1969.

Green Plant (Stem and Leaf) Ghosal 1972a and Ghosal & Bhattacharya 1972; Green material has 3X more alkaloid than if dried.

Roots (Amount not given) Ghosal 1972a and Ghosal & Bhattacharya 1972 [0.38 gm. from 1.6 kg. of dried roots. i.e. 0.02% DMT Ghosal & Banerjee 1969]

Seeds (amount not given) Ghosal & Bhattacharya 1972 Fruit (amount not given) Ghosal 1972a

Desmodium gyrans DC

Leaves (0.004% in dry leaf: 82 mg from 2 kg.) Ghosal *et al.* 1972a

Roots (Minor alkaloid) Ghosal et al. 1972a

Desmodium pulchellum Bentham ex Baker

Whole plant (Minor alkaloid) Ghosal & Mukherjee 1964 (Mention. Ghosal & Mukherjee 1965) (Amount not given. Ghosal & Mukherjee 1966)

Stem and leaf of young seedling [$\sim 0.074\%$ by dry weight; 62% of 0.12% Total alkaloid] Ghosal *et al.* 1972c

Stem and leaf of mature plant [0.294% by dry weight; 21% of 1.4% Total alkaloid] Ghosal *et al.* 1972c

Root of young seedling [~O.27% dry weight; 73% of 0.37% Total alkaloid] Ghosal *et al.* 1972c [i.e. 27 mg. in 10 gm. of dried material]

Root of mature plant [0.451% by dry weight; 41% of 1.1% Total alkaloid] [Also, in same paper: 1.8 kg dried roots yielded 0.7 gm + 0.09 gm; i.e. 0.043%.] Ghosal *et al.* 1972c

Fruit (green) of mature plant [12% of 0.01% Total alkaloid] {~ 0.001% by dry weight} Ghosal *et al.* 1972c

Seeds (ripe) of mature plant [4% of 0.02% Total alkaloid] {~ 0.001% by dry weight} Ghosal *et al.* 1972c

Root, stem-leaf and fruit (Amounts not given) [Ghosal 1972a

Gleditsia triacanthos "Honey locust"

Positive assay (in roots) co-tlc by Appleseed. Plant reacted badly to root sampling (stopped producing any leaves for 18 months) so a second sample was never taken.

Lespedeza bicolor "Bush clover"

Positive tlc assays in seeds, seed pods, stem-bark and roots. co-tlc by Appleseed

Seeds/seed-pods showed same alkaloids as stem-bark but darker and with 3-7 additional bands. (Seeds & pods harvested summer 1994) August stem-bark showed light band. [Successful bioassay of 30 gm of red fall leaves reported

by Wyrm; pers. comm.]

Roots harvested in December showed a positive for DMT and lighter for two other bands. tlc by Appleseed 1994-1995

Some of these results used Ehrlichs spray and there may be confusion with 5-MeO-DMT in seeds and seeds/seed-pods.

Lespedeza bicolor Turcaninow var japonica Nakai

DMT in plant. Goto *et al.* 1958 Major alkaloid in leaf and one of the main alkaloids in the root bark. Root bark showed higher concentration than leaves. Morimoto & Matsumoto 1966

In leaf. Morimoto & Oshio 1965

Lespedeza capitata Rätsch 1998 lists as containing DMT; citing Kindscher 1992.

Mimosa hostilis Benth (Sometimes called *M. jurema*. Considered synonym of *Mimosa tenuiflora* but one of them needs to be described with a subspecific status.)

0.31% alkaloid in root bark (fresh roots obtained 29 October 1942 from Brejo dos Padres) Gonçalves de Lima 1946: 76

0.57% DMT isolated from roots. (Roots were obtained from Prof. Gonçalves de Lima.) [If one calculates the alkaloid content from the crude picrate, their recovery was 0.42% (11.14 grams of DMT from 2.7 kg.).] Page 1286 of Pachter *et al.* 1959 (This account says that 0.51% alkaloid yield was obtained from root bark by Gonçalves de Lima who was unable to identify the alkaloid.)

Mimosa jurema is synonym for Mimosa tenuiflora

Mimosa nigra is synonym for Mimosa tenuiflora

Mimosa ophthalmocentra Mart. ex Benth.

" jurema preta" Used for preparation of jurema

1.6% by dry wt. in roots. Batista et al. 1999

In stem Batista & Almeida 1997

Mimosa pudica "Sensitive plant"

Trace amounts possibly observed in seeds (by tlc) J. Appleseed 1995

None in roots of first year plants.

Roots and also leaves during the second year began to show an alkaloid that co-chromatographed with DMT and showed the same color reaction with Xanthydrol (purple). [See also notes under 5-MeO-DMT entry.] (An additional high Rf band was present in all parts tested.) tlc by J. Appleseed. (1995-1996)

Mimosa scabrella Bentham (= Mimosa bracaatinga Hoehne = Mimosa bracaatinga var. aspericarpa Hoehne = Mimosa secunda Hoehne ex Angely)

In bark. Amount of product not given. Less than 0.0357% by dry wt.

De Moraes et al. 1990

Mimosa somnians

In error? [Not observed by Gupta & coworkers.]

Mimosa tenuiflora (Willd.) Poir. = Acacia hostilis Martius = Mimosa hostilis Benth. [See also entry above] = Mimosa cabrera Karsten = Mimosa nigra J.Huber = Mimosa limana Rizzini = Mimosa maracasensis Harms

~0.03% yield from dry stem bark. Meckes-Lozoya *et al.* 1990

Commercially obtained root-bark (Chiapas, Mexico) showed **four** bands present.

One was DMT, one possibly MMT and the other 2 were high Rf bands; one of which appeared to be present in greater concentrations than DMT. Co-TLC (Xanthydrol), by J. Appleseed 1996.

Ott (1997-1998) reported analysis showing 11% DMT in roots of Mexican material citing unpublished work by Bo Holmstedt in 1983 and by W. E. Sanchez Lemus in 1984. Ott cited personal communication in October of 1996.

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Mimosa verrucosa (purportedly in bark)

- This is included by a number of authorities. I can locate **no published analysis** on any material under this name. The references encountered (when a reference is even included) do not support the claim with analytical work. Usually the reference is Gonçalves de Lima who simply mentions that this plant is used for *vinho da jurema*. (on page 58)
- *Mimosa verrucosa* is said by Da Mota 1991 to be used in making *jurema*, but to have sedative and not hallucinogenic effects.
- Silveira Barbosa 1998 found it in use as a probable DMT containing brew in Brazil but [unlike *M. hostilis*] it appeared to be orally active as a hallucinogen only when an MAOI was coadministered. Her report of full activity with MAOI supports DMT's presence.

Mucuna pruriens

In leaf, stem, seed and root. Bhattacharya *et al.* 1971 In root, stem-leaf, and pod. Ghosal 1972 0.01% in fresh leaves. Ghosal *et al.* 1971d

Mucuna pruriens var. bennetti

Positive assay in seeds. Appleseed 1995

Petalostylis labicheoides var. casseoides Benth.

Traces. Johns et al. 1966a

Piptadenia contorta

In seeds by TLC. Yamasato et al. 1972

- Swainsona galegifolia (Andr.) R.Br. (= Swainsona coronillifolia Salisb.)
 - Positive assay in leaf TLC by J. Appleseed 1994 Conflicting results. First year's growth tested positive in two separate assays, second year's growth tested negative. Plant died during winter after second year; no further assays made.

Wisteria species

Leaf-stem (Plants originally mislabeled; exact species identity not yet determined. Strong positive in 3 tlc assays over a 2 year period. (During years 3 and 4 of growth from seed.) At least 4 other distinct Xanthydrol reactive bands.

Malpighiaceae

- *Banisteriopsis muricata* (Cav.) Cuatr. (=*Banisteriopsis argentea* Spring ex Juss)
- DMT in leaf [48 mg/ 1.8 kg] Ghosal & Mazumder 1971 and Ghosal *et al.* 1971a
- *Diplopterys cabrerana* (Cuatrecasas) Gates [*Misidentified* by Morton as *Banisteriopsis rusbyana* (despite this persistent error, they are **not** synonyms) *see* Gates 1982 for details] [AKA *Chagropanga*, *Chalipanga*, *Oco-yajé*, *Yajé-uco*: Occurs in the Colombian Putumayo; used by Mocoa in Colombia and by the Siona and Secoya in Ecuador. Used far less commonly in Peru than in Colombia and Ecuador. Ott 1994] Leaf 1.46% (1.33%-1.75% spectrophotometer estimate)

Der Marderosian *et al.* 1968a [Sole base present according to Der Marderosian]

1.3% in leaves. Alkaloid content "*largely DMT*" (eastern Ecuador) Der Marderosian *et al.* 1968b

Poisson 1965. DMT was the major base in the leaves. Poisson found 0.64% total bases comprised of DMT; 6.4 gm total bases per kg. He recovered **18 mg of DMT from 2.8 grams of leaves. (3 leaves)**

[He reported β -carbolines in the stems, the major of which he believed to be harmine and smaller amounts of harmaline or 6-Methoxy-N,N-dimethyltryptamine. His extraction route would have been inefficient for harmine.] His material collected in Peru by Claudine Friedberg.

Agurell *et al.* 1968a found 0.4655% in dried leaves (465.5 mg from 100 grams; 4.65 mg per gram) and 0.166% in dried stems. (177 mg from 100 grams.)

[Agurell *et al.* 1968 also detected traces of MMT, Bufotenine, 5-MeO-DMT and N-Methyl-H⁴- β -carboline in the leaves and traces of 5-MeO-DMT and N-Methyl-H⁴- β -carboline in the stem. See also Agurell 1968b.]

Diplopterys cabrerana [Plowman #6040; Tarapoto]

1.58 mg per gm dry weight (SD \pm 0.41) in leaf. [Traces of Bufotenine also present.]

McKenna et al. 1984a

Myristicaceae

Osteophloem platyspermum (DC) Warb.

DMT in bark of Schultes and Rodriguez No. 26126; Origin: Manáos, Brazil. One of 3 alkaloids in 0.62 mg of total alkaloid from 100 grams of dry bark

Holmstedt et al. 1980

[Plowman, Schultes and Tovar # 7095; Origin: Pebas, Peru (Alpha-Helix 1977) assayed negative with Dragendorff and Ehrlich reagents.]

Virola

Snuff: "epéna"

Snuff prepared by Tucano Indians: collected 1965 DMT [with 5-MeO-MMT and 5-MeO-DMT]

Holmstedt & Lindgren 1967

Snuff: "epéna"

Snuff as prepared by Waica Indians (collected 1965) DMT [with MMT and 5-MeO-DMT]

Holmstedt & Lindgren 1967

Snuff: "epéna"

Snuff prepared by Araraibo Indians: collected 1965

DMT [with 5-MeO-DMT]

Holmstedt & Lindgren 1967

Virola based snuff: "epena" [Virola?]

Obtained from Waica by George Seitz. DMT was a minor component. 5-MeO-DMT was the major. Holmstedt 1965. Bufotenine also observed as a minor alkaloid, casting doubts on the presumed botanical origin. This may belong under another entry.

Virola based snuff: "*epena*" [No.24574; Origin: Rio Cauaburi, Brazil]

0.14% DMT i.e 1.43 mg per gm of snuff [20% of 715 mg. of total alkaloids/ 100 gm. of snuff] Agurell *et al.* 1969

- *Virola* based snuff: "*nyakwána*" [No. 24626; Origin: Tototobi, Brazil]
- 1.2% DMT i.e. 12.1 mg per gm of snuff [11% of 11,000 mg. of total alkaloids/ 100 gm. of snuff]

Agurell et al. 1969

Paste: believed from a *Virola* sp. (No voucher; "*oo'-koey*"; La Chorrera)

DMT 0.3 mg/ ml [5-MeO-DMT was major alkaloid at 1.19 mg/ ml]

McKenna et al. 1984a

Virola calophylla Warburg DMT: bark, root, leaf, seed & flower Agurell et al. 1969; Holmstedt et al. 1980; McKenna et al. 1984b DMT in bark and leaf. Holmstedt et al. 1980; McKenna et al. 1984b Virola calophylla Bark collected in Manaus, Brazil during 1964. DMT [with MMT and 5-MeO-DMT] Holmstedt & Lindgren 1967 Virola calophylla [No.24603; Origin: Manaus, Brazil] Bark- 0.008% [8 mg. of alkaloid/ 100 gm. of dry bark: Sole alkaloid DMT] Roots- 0.0009% [0.87 mg. of alkaloid/ 100 gm. of dry roots: Sole alkaloid DMT1 Flowering shoots- 0.185% [96% of 193 mg. of total alkaloids/ 100 gm. of dry flowering shoots] Leaves- 0.15% [149 mg./ 100 gm. of dry leaves: Sole alkaloid DMT] Agurell et al. 1969 Virola calophylloidea Markgraf DMT in bark and leaf. Holmstedt et al. 1980 Virola carinata (Spruce ex Bentham) Warburg DMT in leaf. Holmstedt et al. 1980 Virola divergens Ducke DMT in leaf. Holmstedt et al. 1980 Virola elongata (Spruce ex Bentham) Warb. DMT in bark and leaf. Holmstedt et al. 1980 and McKenna et al. 1984b Virola melinonii (Benoist) A.C.Smith DMT in bark. Holmstedt et al. 1980 Virola multinerva Ducke [No.24614; Origin: Manaus, Brazil] Bark- 0.001% [1 mg./ 100 gm. of dry bark: Sole alkaloid DMT] Root- 0.0004% [0.41 mg / 100 gm. of dry roots: Sole alkaloid DMT] Agurell et al. 1969 Virola multinerva Ducke [No. 24616; Origin: Manaus, **Brazill** Bark- 0.001% [1 mg. / 100 gm. of dry bark: Sole alkaloid DMT] Agurell et al. 1969 Virola multinerva Ducke DMT in bark and root Agurell et al. 1969; Holmstedt et al. 1980 Virola pavonis (DC) Smith DMT in leaf McKenna et al. 1984b Virola peruviana (DC) Warburg DMT in bark Holmstedt et al. (1980 DMT in plant. Part and amount not given. Lai et al. 1973 Virola rufula (DC) Warburg DMT in bark, root and leaf. Agurell et al. 1969; Holmstedt et al. 1980 Virola rufula (A.DC) Warb. [No.24612; Manaus, Brazil] Bark- 0.19% (190 mg. / 100 gm. of dry bark: Sole alkaloid DMT] Root- 0.001% (1.44 mg. / 100 gm. of dry roots: Sole alkaloid DMT] Leaf- 0.09% (92 mg. / 100 gm. of dry leaves: Sole alkaloid DMT] Agurell et al. 1969

Virola sebifera Present in bark Kawanishi et al. 1985 Virola sebifera Aublet DMT in bark Corothie & Nakano 1969 Virola sebifera (DMK-40; Don Marcos no. 1) Paste: DMT 0.1 mg/ ml [with MMT as the major alkaloid; present at 1.38 mg/ ml] McKenna et al. 1984a Virola theiodora (Spruce ex Bentham) Warburg Bark- 0.0017% [4 mg. of DMT in 235 gm. of bark] (MMT and 2 unidentified components present) Leaves assayed negative. Cassady et al. 1971 & 1972. Cassady et al. 1971 cited Cassady et al. 1970 [Published 1972] Virola theiodora [No.24595; Origin: Manaus, Brazil] Bark- 0.13% [52% of 250 mg. total alkaloids/ 100 gm. of dry bark] Root- 0.004% [22% of 17 mg. of total alkaloids/ 100 gm. of dry roots] Flowering shoots- 0.44% [93% of 470 mg of total alkaloids/ 100 gm. of dry flowering shoots.] Leaf- 0.04% [99% of 44 mg. of total alkaloids/ 100 gm. of dry leaves.] Agurell et al. 1969 Up to 0.25% in dry bark and can be twice this in flowers. Shulgin & Shulgin 1997 Virola theiodora [No.24626; Origin: Tototobi, Brazil] Bark- 0.003% in dry bark (5% of 65 mg. of total alkaloids/ 100 gm. of dry bark] Leaf- 0.02% in dry leaves [98% of 21 mg. of total alkaloids/ 100 gm. of dry leaves: i.e. 4.76 grams of dry leaf per milligram of DMT.] Agurell et al. 1969 Virola venosa (Bentham) Warburg [No. 24613; Origin: Manaus, Brazil] Traces of DMT in dry leaves. [1 mg per 100 gm] Bark negative. [5-MeO-DMT in roots.] Agurell et al. 1969 and Holmstedt et al. 1980 Ochnaceae Testulea gabonensis Pellegr. Trace in bark and root bark. DMT & 2 other alkaloids formed 10% of total. (Total alkaloid: 2.5% in Stem bark & 5% in Root bark) Leboeuf et al. 1977 Pandanaceae Pandanus sp. Hyndman 1984 cited personal communication from a D. Culvenor reporting DMT as a minor component among other alkaloids. Pandanus utilis In nuts/ seeds. [Co-occurrence with harmine.] tlc by J. Appleseed 1994. Not confirmed in 1995 assay. Harmine was still present but DMT was not detected in 1995. Pandanus odoratissima In nuts. Observed in hard core but not in fibrous outer nut. Harmine and another β -carboline (blue under UV) were present in both. tlc by J. Appleseed 1995

Polygonaceae

Erigonum sp.

DMT appears **erroneously** in the literature. The reference that was cited, Schroeder 1986, reported N,N-Dimethyl-**tyramine**

[*Eriogonum* spp. include *Buckwheat* and *Umbrella plants*. There are about 150 spp. occurring as wild flowers and cultivars in the west and southwestern US and Mexico. Some are annuals and some are perennials. The only assay I have seen was positive for DMT but in traces. tlc by Appleseed]

Rubiaceae

Antirhea lucida

In roots with gramine, 6-Methoxy-2-methyl-tetrahydro-βcarboline, and N,N-Methyl-3'-indolyl-methyl-5methoxytryptamine. Weniger *et al.* 1995

Psychotria species

Suspected identity. Thought by some to have been analyzed under the misnomer *Prestonia amazonica*. (3 grams isolated from 2 liters of a previously prepared solution.) Hochstein & Paradies 1957

It has been pointed out that the plant in question is described as a twining plant and additionally was 1) given the common name of yage and 2) *Prestonia amazonica* does not grow in the area where the material originated; suggesting that the source material for their analysis might actually have been *Diplopterys* rather than a *Psychotria*.

Other potential candidates for its identity also exist stressing the need for verifiably vouchered material.

Ott 1993 mentions that despite the reasonable assumptions of it being a *Psychotria* or *Diplopterys*, the distinct possibility exists that it may have been an altogether different admixture plant.

Psychotria species [P. viridis?]

"nai kawa" (Cashinahua)

DMT isolated from authenticated material.

Der Marderosian et al. 1969

0.16-0.22% in leaf

Der Marderosian et al. 1970

Psychotria alba

- Thought to contain DMT based on the fact that it is used interchangeably with *P. viridis* by the UdV in Brazil. Published analysis is apparently lacking. Claimed to contain 60% as much as *P. viridis*. Independent analysis failed to detect DMT in at least one commercial strain. (Eel: pers. comm 2001)
- *Psychotria carthaginensis* Jacq. ["*rami appani*"; Culina Indians, Marcos. Collected 4 September 1968.]
- 0.65% DMT in dry leaf. [99% of 0.66% total alkaloid content by dry weight.] Their specimens contained more alkaloid than the *P. viridis* they also analyzed.

"practically all DMT"

Rivier & Lindgren 1972

Many other assays have detected no DMT in this species. (such as McKenna *et al.* 1984a, who analyzed DMCK #109 "yage-chacruna" from Tarapoto, and also Leal & Elizabetsky 1996 Psychotria horizontalis Sw.

May contain DMT due to the unsupported claim (by Duke & Vasquez Martinez 1993) of its application in Ayahuasca preparation but analysis is apparently lacking.

Psychotria marginata Sw.

Suggested to contain DMT due to the unsupported claim (by Duke & Vasquez Martinez 1993) of its application in Ayahuasca preparation but analysis is apparently lacking.

Psychotria poeppigiana

Bioassays indicate a strong presence of DMT. Personal communications with an unnamed source.

Psychotria psychotriaefolia (Seem.) Standley

- [Material **erroneously** identified. Actual identity was later determined to be *P. viridis*.]
 - DMT in leaf along with two non-indolic alkaloids.

Der Marderosian et al. 1969

Psychotria species [probable ID by R.E. Schultes; *"falsa chacruna"* (Shibipo) upper and middle Ucayali also by town dwellers in Iquitos.]

0.8% total crude bases, with DMT was the major alkaloid. Percentage of DMT unspecified. Urzúa *et al.* 1972

Psychotria stenostachya Standl.

May contain DMT based on unsupported claim (Duke & Vasquez Martinez 1993) of its application in preparing Ayahuasca. Apparently lacking analysis.

- Psychotria viridis Ruiz & Pavon
- [AKA "chacruna" (Peru), "sami ruca", "amurucapanga" (Ecuador)]

DMT in leaf Der Marderosian et al. 1970

Psychotria viridis Ruiz & Pavon ["rami appani"; Culina Indians, Zapote. Collected 22 July 1968.]

0.34% DMT in dry leaf [99% of 0.34% total alkaloid content by dry weight.]

Traces of MMT and 2-Methyl-1,2,3,4-tetrahydro- β -carboline as minor alkaloids. [DMT was absent from another specimen of this species.] Rivier & Lindgren 1972

DMT in leaf in "substantial amounts" Co-occurring with traces of MMT and 2-Methyl-1,2,3,4-tetrahydro- β -carboline. Rivier & Lindgren 1972

Psychotria viridis [DMCK #21; Iquitos "chacruna"]

0.16% DMT; 1.58 mg per gm dry weight (SD \pm 0.3) in leaf. [Sole base]

McKenna et al. 1984a

Psychotria viridis [DMCK #108; Tarapoto, "suija"]

0.10% DMT; 1.02 mg per gm dry weight (SD \pm 0.04) in leaf. [Sole base]

McKenna et al. 1984a

Psychotria viridis [DMCK #139; Pucallpa, "*chacruna*"] 0.12% DMT; 1.2 mg per gm dry weight (SD \pm 0.17) in leaf. [Traces of 2-Methyl-1,2,3,4-tetrahydro- β -carboline also present]

McKenna et al. 1984a

Psychotria viridis [probable ID by R.E. Schultes; "*chacruna*" (Shibipo) upper and middle Ucayali also by town dwellers in Iquitos]

0.24% total crude bases, DMT was the major alkaloid. Percentage of DMT unspecified. Said to be distinguishable from the '*false chacruna* based on its profile of unidentified minor bases but the details were not included. Urzúa *et al.* 1972

Rutaceae

Evodia rutaecarpa Hooker f. ex Thomas

- 0.00026% by dry weight in unripe fruit. [7.8 mg from 3 kg.] Yu *et al.* 1997
- *Limonia acidissima* L. (= *Limonia crenulata* = *Hesperethusa crenulata*) [wood-apple, elephant-apple]

0.0045% DMT in dry stems.

Many other compounds present; including N-Acetyl-Nmethyltryptamine, 3-Formylindole & 2-Methyltetrahydro- β carboline.

Other plant parts apparently not tested.

Abu Zarga 1986

Vepris ampody H.Perr.

0.224% DMT in leaf.

Co-occurring with Kokusagine, Dimethoxy-2,4-methyl-10acridone, Evoxanthine and Phenacetamide.

Kan-Fan et al. 1970

Zanthoxylum arborescens Rose

0.09% leaf (dry weight) Many other compounds present. Grina *et al.* (1982)

Zanthoxylum procerum Donn. Sm.

DMT in leaf Ott cited Schroeder 1986

[An odd side note: in tlc run (several times) on *Zanthoxylum americanum* bark, Appleseed saw a band that cochromatographed with DMT but turned a weird orange with Ehrlich's reagent.]

Violaceae

Rinorea viridiflora Rusby

Presence of DMT is claimed in Fericgla 1994 based on bioassay of a Shuar ayahuasca using this as its admixture plant. Analysis is apparently lacking.

Animals:

Gorgonaceae

Paramuricea chamaeleon

Less than 5 mg. of DMT was isolated from 200 grams of this coral by Cimino & DeStefano 1978.

Saavedra & Axelrod 1972 showed that MMT and DMT can be formed in rat brain and that an enzyme is present that is capable of performing this reaction. They also found that *something else is present which inhibits this reaction*.



Occurrence in humans:

- See the review of biogenic amines reported in human body fluids by Bruce Davis 1989.
- Clarke's Second Edition notes that natural endogenous concentrations in plasma are normally less than 0.001 μ g/ml and that im administration of 0.7 mg/kg resulted in an average concentration of 0.1 μ g/ml at 0.17 hour [Ed.: 52.5 mg for a 165 lb. human]. Said to be the time of maximum effect via this route. This is fascinating as it implies that strongly entheogenic activity is a result of elevation of the concentration by less than 100 times that of the naturally occurring baseline.
- DMT was found in normal human blood and urine by Franzen & Gross 1965.
- Sample analysis of human cerebrospinal fluid included DMT. Christian *et al.* 1975.
- Found in cerebrospinal fluid of psychotics and normal people by Corbett *et al.* 1978.
- Narasimhachari et al. 1971a found DMT in schizophrenics but not in normals.
- Narasimhachari *et al.* 1971b reported it in most psychotics but only 2 out of 20 normals.
- Smythies *et al.* 1979 found it at wildly varying levels in both populations.
- Lipinski et al. 1974 found it in some psychotics.
- Oon & Rodnight 1977 thought they observed DMT in psychotics but did not positively prove.
- See also Oon et al. 1977 and his references.
- For additional references on the natural and potential occurrence of DMT in mammals and humans see:
- Beaton & Morris 1984

Christian et al. 1976 & 1977

- Räisänen & Kärkkäinen 1979 (in urine)
- Rosengarten & Friedhoff 1976
- Saavedra & Axelrod 1972
- Tanimukai et al. 1970
- Wyatt *et al.* 1973 (found at variable levels in plasma of both psychotics & normals.)
- For references on occurrence in normal populations versus psychotics, see articles just mentioned, also those by Barker or Christian above, and:
- Davis 1989 [Davis noted that those studies failing to find DMT were the ones that relied on less sensitive assay methods.] Oon *et al.* 1977

See discussion & references in Gillin et al. 1976.

Review: Rosengarten & Friedhoff 1976

Acacia obtusifolia Photo by Zariat

DMT

TD_{L0}

1 mg/ kg Sax 1984 cited Rosenberg et al. 1963

"Toxic Dose Low" i.e. the least amount observed to produce toxic effects. In Sax's opinion; activity itself is apparently what they consider a toxic effect.

Activity:

- Hallucinogenic. Szara 1956 [Usdin & Efron 1979: #384, cited the same]
- Entheogenic at 1 mg./ kg i.m. Ott 1993: #8; cited Szara 1956 and Sai-Halász et al. 1959. Ott 1996 replaces entheogenic with *psychoptic*
- [Relationship between hallucinogenic activity and electronic configuration. Snvder & Merril 1965]

Dose:

Human dose: (Usdin & Efron 1979 #384)

1 mg/ i m. (/ kg?) cited Szara 1961

1-1.2 mg/ i m. (/ kg?) cited Arnold & Hoff 1962

14-70 mg/ i.m. cited Jacob 1966

400 µg/kg smoked/insufflated or IV. Duration 10-15 minutes. Callaway & McKenna 1998

60-100 mg smoked or im or sc

4-30 mg iv

Shulgin & Shulgin 1997: page 415.

Our recommended dosages (for a 150 pound human) is 15-30 mg: smoked; or 35-50 mg: used orally combined with an MAOI.

Duration:

- Variable. Smoked or injected lasts a shorter duration than if taken orally with an MAOI.
- When smoked, effects begin rapidly, peaks for 4-8 (-12) minutes, then drops off rapidly for another 10-20 minutes and usually gone within the hour.
- Baseline is normally completely reestablished within 1-2 hours although some people report a pleasant afterglow with sharpened perception for 1-2 days.
- With an oral dose of 0.48 mg/kg combined with an MAOI (as Hoasca)

 C_{max} (average) 15.8 ± 4.4 ng/ml [See Note]

 T_{max} (average) 107.5 ± 32.5 min.

 $T_{1,2}$ (average) 259.4 ± 207.2 min.

The most intense visual effects were reported to occur between 60 and 120 minutes after drinking the ayahuasca. Callaway et al. 1999

When 0.4 mg/kg was injected intravenously:

- C_{max} (average of 10 subjects) 90 ng/ml (Ranged from 32-204 ng/ ml in their 11 human subjects)

 $T_{max} \sim 2 \text{ min.}$ Their test subjects were moderately intoxicated for 10-20 minutes.

Recovery was complete within 30 minutes although several subjects reported a relaxed "at ease" feeling for another 30 minutes.

Strassman & Qualls 1999 [Note: This is especially interesting in light of Wyatt et al. 1973 reporting 10.6 ng/ml in the blood of one bipolar severely agitated depressed patient who showed levels below the threshold for detection when tested again later (during a less agitated but clearly psychotic period)] Intramuscular injections showed a 2-3 minute onset, peaking

in 10 minutes and was gone at ~ 1 hour. Kaplan et al. 1974

Receptor site specificity:

Agonist at 5-HT_{1A} & 5-HT_{1C} and antagonist at 5-HT₂ (possible partial 5-HT, agonist) Callaway & McKenna 1998 Studies on 5-HT receptor interactions & specificities: Deliganis et al. 1991 Glennon et al. 1979 McKenna et al. 1990

Biochemical & Animal miscellany:

"Preliminary data from rodents," generated by Christian et al. 1976a, suggested "that animals under stress exhibit an increase in the amount of DMT in isolated brain vesicles."

Preadministration increases survival time in mice exposed to lethal radiation. Shinoda et al. 1974

Pharmacology & Pharmacognosy of DMT:

Shulgin 1976: Brief abstract.

Erspamer 1961: Mainly on 5-HT but has some info on pharmacology of DMT.

Heinzelman & Szmuszkovicz 1963 Some pharmacology of DMT. Many synthetic analogs. Fascinating paper.

Pharmacological Study in Humans:

Strassman & Qualls 1994

[Found dose dependent rise in elevation of blood pressure, heart rate, pupil diameter, rectal temperature, β-endorphin levels, corticotropin levels, cortisol levels and prolactin levels. Growth hormone levels were increased equally by all dosages.]

Strassman et al. 1994

See also references contained in both articles.

See also: Strassman 1994 & 1999

Also see discussion in Efron (ed.) 1967: pp. 374-382

[Also the interesting proposal by Callaway 1988]

Metabolism & Excretion:

Räisänen & Kärrkkinen 1979 (humans)

Metabolized by both MAO-A & MAO-B at 20 µM but MAO activity was almost entirely due to MAO-B at concentrations of 100 µM. Suzuki et al. 1981

Effects of MAOI pretreatment: Lu & Domino 1976

See also Ott 1994 & 1999

(Despite this, Barlow 1961, Ho et al. 1970 & Govier et al. 1953 determined it was a poor substrate for MAO)

Freedman et al. 1970 reported DMT showed some apparent MAOI activity in vivo in rat brains.

Clarke's Second (Szara & Axelrod 1959) noted that 33% of a given dose is excreted in human urine at 6 hours as free and conjugated (glucuronide)indol-3-yl acetic acid; less than 0.1% excreted unchanged in urine in 24 hours.

Others have suggested that a significant portion may be removed by active uptake and recycled similar to serotonin rather than metabolized or excreted. However, see Hryhorczuk.

25% was reported to be recovered from human urine as indole acetic acid by Szara & Axelrod 1959

Only 0.07% was recovered from human urine unchanged: Kaplan *et al.* 1974.

Hryhorczuk *et al.* 1986 suggested yet another alternative based on their observation that DMT was largely metabolized (*in vitro*) into Dimethylkynuramine (DMK) via an undefined indoleamine 2,3-dioxygenase when it was incubated with human red blood cells. (Kynuramine itself is active as an alphaadrenergic antagonist but DMK seems to lack evaluation.)

Barker *et al.* 1980 reported that rat brain homogenates metabolized DMT into IAA, DMT-N-oxide, MMT, Tryptamine, 2-MTHBC & THBC. Pretreatment with Iproniazid decreased IAA formation 83%, N-oxide and MMT formation by 90% and prevented the formation of 2-MTHBC suggesting that Iproniazid potentiation and prolongation of the action of DMT is the result of more enzymatic inhibition than simply MAO.

Fish *et al.* 1955 reported that mouse liver homogenates metabolize DMT into the N-oxide (major) with IAA and 2 unknowns. Barker et al. 1978 found that rat liver and brain microsomes metabolize DMT into DMT-N-oxide (major), MMT & formaldehyde. A similar outcome resulted if using DMT-Noxide except for producing DMT as the major. (Suggesting their interconversion.)

Very low excretion rates were noted by Kaplan *et al.* 1974, these being highly variable from one human subject to the next. (154 ng/ml was the highest blood concentration they found after giving 0.7 mg/kg im.)

They also found that the peak blood levels reached varied widely between subjects.

Kaplan noted that since only less than 1.8% of their given dose was present in the blood at any one time, it was unlikely that any DMT that was produced in the brain would ever be detectable in the blood "even if serendipitously the peak time was chosen"

Despite this, peak effects subjectively reported appeared to follow the same time course as peak DMT levels in the blood.

They also found that of DMT recoverable in urine over a 24hr period, 97% was recovered in the first 5.5 hrs.

Their highest DMT recovery from urine was 0.16% of the dose with 0.069% of the injected dose as a mean of 7 subjects.

Kaplan's conclusion was that, even IF DMT turned out to be an endogenous psychotogen, urinary levels of DMT would not be meaningful for comparing psychotics and normals.

Erspamer 1955 reported rats to excrete as IAUA (Indoleaceturic acid) with much smaller amounts of IAA and 2 unidentified metabolites.

Tolerance:

True tolerance is either nonexistant/limited or else extremely short-lived. (See comments on page 231)

Drug interactions: (see more comments on p. 233)

Action inhibited by pretreatment with Ketaserin: Batista & Almeida 1997. Chlorpromazine shows similar actions (See references on page 235)

Sai-Halász 1962 reported that dosages of DMT that were too low to normally produce effects are enabled to become active after pre-dosing with the serotonin antagonist 1-methyl-D-lysergic acid butanolamide (UML-491). (This latter compound is known to have no psychic effects) Interestingly, an exaggerated response (both in per-dose effects and *often* in duration of the peak) also occurs when predosing with LSD. An increase in activity is observable following predosing with *Psilocybe* but without an increase in duration. Observations by Case.

See also Shah & Hedden 1978 for pharmacological observations & some drug interactions.

Pharmacological overview:

DMT biosynthesis, distribution, metabolism, pharmacology and/or evidence for it being a natural neurotransmitter in mammalian nervous systems:

Barker *et al.* 1981 (Also references contained therein.) Christian *et al.* 1976 & 1977 Cohen & Vogel 1972 Rosenberg *et al.* 1963 Smythies *et al.* 1979 Szara 1956

Behavioral effects:

Disruption of learned behavior in Animal studies. Uyeno 1969 Disruption of conditioned avoidance: See Gessner & Page 1962 and Stoff *et al.* 1977

Toxicity:

Mice- Animals assumed abnormal postures and movements (like walking backwards)

Jumping action, clonic and tonic convulsions and tremor were common to all animals before death.

Ho et al. 1970

"In monkeys doses up to 36 mg/ kg intravenously caused clonic spasms followed by loss of equilibrium, erection of hair, mild ptyalism, loss of perception with no loss of consciousness. A dose of 53 mg/ kg was fatal."

Heinzelman & Szmuszkovicz 1963 cited unpublished results of W.A. Freyburger & B.E. Graham at the Upjohn Company.

LD₅₀

0.5-0.25 mM/ kg/ ip / mouse. Shinoda et al. 1974

- 32 mg/kg/ intravenous/ mouse. Sax 1984 cited Chemical Systems Laboratory.
- 110 mg/ kg/ intraperitoneal/ mouse. Ho et al. 1970
- 128 (104-152) mg/ kg/ intraperitoneal/ mouse Batista & Almeida 1997
- 89 (77-101) mg/ kg/ ip/ rat Batista & Almeida 1997

DMT Sulfosalicylate [and DMT itself] said to be poison by iv or ip.

- LD₅₀ (in mouse):
- 153 mg/kg ip
- 69 mg/kg iv
- Sax & Lewis 7th edition. Entry #DPG000, page 1346. Cited 1970 RPTOAN 33: 180 [CODEN]

Suggested reading concerning DMT:

Alexander T. Shulgin & Ann Shulgin 1997 *TIHKAL The Continuation*: Entry #6, pages 412-421

Peter Stafford 1992 *Psychedelics Encyclopedia* 3rd ed.: pages 308-331.

Rick J. Strassmann 1999 DMT: The Spirit Molecule

Infinite Ayes 2001a & 2001b

Jonathan Ott 1993 *Pharmacotheon*: pages 163-197 (also 1996)

See also:

Jonathan Ott 1994 Ayahuasca Analogs

D.M. Turner 1994 *The Essential Psychedelic Guide*: pages 49-58

DMT-metho cation

N,N,N-Trimethyltryptamine; N-methyl cation of DMT

UV: λ_{max} of 218 & 278 nm with shoulder at 288 nm. Frahn & Illman 1973

See also color reaction (p. 143), electrophoresis (p. 208) & isolation using chromatography (p. 189) (or see Frahn & Illman 1973)

The N-methyl cation has been reported in: Graminae

Arundo donax

Rhizome Ghosal 1972a

Phalaris aquatica

(Up to 5% of total alkaloid at certain stages of active growth, such as following the first substantial rains of autumn and early winter)

Frahn & Illman 1973

Leguminosae Desmodium pulchellum

- Roots & Stem-leaf Ghosal 1972a
- Animals

Hylidae

Litoria moorei (30-50 µg/ mg: Roseghini et al. 1976)

N,N-DMT-N-oxide

DMT-N_b-oxide; N,N-Dimethyltryptamine-N-oxide

CA Reg. No: [948-19-6] Southon & Buckingham 1989

Probably considered a controlled substance

MW 204.271 Southon & Buckingham 1989

 $\begin{array}{c} \mathrm{C_{12}H_{16}N_{2}O} \cdot \mathrm{H_{2}O}\\ \mathrm{Fish}\ et\ al.\ 1955 \end{array}$

Elemental analysis

C, 64.84; H, 8.16; N, 12.60 (Calculated) Found: C, 64.27; H, 7.55; N, 12.51. Fish *et al.* 1955

Free base

Basic oil (Weak base. Weaker than DMT, MMT or tryptamine.) Fish *et al.* 1956 Hygroscopic solid Banerjee & Ghosal 1969

Soluble in water. Fish *et al.* 1955 and Ghosal *et al.* 1970b and Banerjee & Ghosal 1969

Soluble in chloroform Banerjee and Ghosal 1969

Insoluble in petroleum but appreciably soluble in petroleum which contains fats. Ghosal & Banerjee 1969

Hydrate

mp 123-128° Fish *et al.* 1955 (Showed one water of hydration)

mp 123-128° Boit 1961/ Southon & Buckingham 1989

Picrate

mp 176-178° Yellow picrate from ethanol. Ghosal & Banerjee 1969

mp 176-178° Yellow needles from methanol. Ghosal *et al.* 1970

mp 178-180° Yellow picrate from ethanol. Banerjee & Ghosal 1969

mp 178-180° Marini-Bettòlo et al. 1964

mp 178-183° Recrystallized from alcohol. Fish *et al.* 1955 mp 178-183° Boit 1961

Acetate is weakly basic. Soluble: water, chloroform Ghosal *et al.* 1971

Assays:

Colorimetric reagents: See color reactions p. 143

TLC & PC: see Rf table p. 169-176

UV:

 λ_{max} 224, 277, 288 and 292 nm. Ghosal *et al.* 1970b λ_{max} 224, 277, and 292 nm Ghosal & Banerjee 1969 λ_{max} 274, 282, 290 mµ (reference material) 274, 282, 291 mµ (isolated material). Fish *et al.* 1955

Fluorescence:

Fluorescence maxima: Excitation: 283 mµ; Emission 349 mµ. Fish *et al.* 1955

Formation & Isolation:

Formed rather easily in DMT solutions exposed to air during extensive experimental manipulations. Fish *et al.* 1955

N-oxides can be recovered from alkaline aqueous solutions by extracting with an equal volume of butanol. (*After* other bases have been extracted with chloroform) This will also recover serotonin if present.

The N-oxide of DMT [or 5-MeO-DMT] can be readily converted to DMT [or 5-MeO-DMT] by dissolving (or extracting) it in aqueous acetic acid, then adding an excess of powdered zinc, stirring for 30 minutes, neutralizing the acetate with ammonia (raise to pH 9) and recovering the DMT base by extracting it from the basic solution with a good organic solvent (i.e. extract three times with chloroform).

IF performing an acetic acid based extract be certain not to use chloroform to defat. [Ed.: Or perhaps also similar chlorinated small hydrocarbons, like methylene chloride?] DMT acetate is both chloroform and water soluble.

Ghosal *et al.* (1971) *Journal of Pharmaceutical Sciences* 60 (8): 1209-1212:

- The viscous brown residue which remained after solvent (ethanol) was removed under reduced pressure was mixed into 2% acetic acid with stirring. (Using 200 ml for 78 grams of residue)
- This was allowed to stand at room temperature overnight and then filtered to remove impurities.
- This acidic solution was extracted with three 50-ml portions of chloroform to remove the weakly basic chloroform soluble acetates.
- After removal of the chloroform, the residue was then dissolved in benzene and chromatographed over a column of alumina.
- Elutants were petroleum ether, benzene, chloroform, methyl alcohol and stepwise increments between them.
- They collected 40 ml fractions.
- DMT was eluted (as a brown oil) with chloroform-methyl alcohol (99:1)
- DMT-N-oxide was eluted with methyl alcohol. [Zinc dust and acetic acid reduced it to DMT.]

Ghosal & Banerjee (1969) *Australian Journal of Chemistry* 22: 2029-2031:

- When defatting with Petroleum ether (60-80°), the Petroleum extract was concentrated to 200 ml and extracted with 2N aqueous citric acid (200 ml). The acidic solution yielded a brown gum.
- This gum was triturated (ground with the solvent in a mortar and pestle) with light petroleum (40-60°)
- The portion which was insoluble in the light petroleum turned out to be DMT-N-oxide.
- [DMT-N-oxide is not soluble in petroleum ether when pure but is soluble in petroleum ether containing fats.]

Banerjee & Ghosal (1969) Australian Journal of Chemistry 22: 275-277:

- The Chloroform layer was then extracted with 2N aqueous acetic acid (100 ml) and both layers saved.
- [DMT and DMT-N-oxide acetates are soluble in both acid and chloroform.]
- **The aqueous layer** was cooled in ice and then made basic to pH 9 with ammonia and extracted with chloroform.
- The chloroform was then evaporated.
- The basic gum (2.5 gm) was chromatographed on a column of Brockman neutral alumina.
- 5-MeO-DMT was eluted with light petroleum-benzene (1:1) [as 570 mg of colorless plates.]
- DMT was eluted with chloroform (as thick colorless oil, amount not given.)
- DMT-N-oxide was eluted with chloroform-methanol (9:1) [as 210 mg as hygroscopic solid.]
- Chromatography using Brockman alumina was then done with **the original chloroform extract** containing the chloroform soluble acetates.
- Chloroform-methanol eluted additional amounts of DMT (410 mg) and DMT-N-oxide [120 mg].

See full details farther below.

DMT free base is yielded by the reduction of DMT-N-oxide with zinc dust and acetic acid

DMT-N-oxide readily forms by the action of peroxide on DMT.

- i.e. 50 mg. of DMT dissolved in 2 ml. of ethanol was treated with 2 ml. of H_2O_2 (1 ml of 30% H_2O_2 in 9 ml. of ethanol.). After two hours (at room temperature) crystallization was induced by adding ether and chilling.
- The granular DMT-N-oxide was removed and recrystallized from ethanol-ether.
- N-Methyl tryptamine is not affected by the addition of peroxide.
- DMT-N-oxide is water soluble.

Fish et al. 1955

Another reduction of DMT-N-oxide was performed by Kawanishi et al. 1985 who were examining Virola sebifera bark.

Reported Occurrences of DMT-N-oxide:

Graminae

Arundo donax

Culm and flowers. Ghosal 1972a

Leguminosae

Acacia caesia (Willdenow) (= Acacia intsia)

- In bark. Further details not given. Ghosal et al. 1970b
- **Anadenanthera colubrina var. cebil** [as Piptadenia macrocarpa]
- In seeds but not in pods. Amount of DMT-N-oxide not given. Total alkaloid content of ~ 1.5 -2.0% determined in some samples: Fish *et al.* 1955. Material from both Florida and Brazil were used.

Anadenanthera peregrina

(Haiti) (Traces in seeds) Paris et al. 1967

In seeds but not in pods. Amount of DMT-N-oxide not given. Total alkaloid content of 1.6% determined in one sample: Fish *et al.* 1955. Used material from both Puerto Rico and Brazil.

Epena

Yanoama snuff prepared from *Piptadenia peregrina*. Marini-Bettòlo *et al.* 1964

Snuff: "Epena"

Snuff prepared, by the Ma-hekodo-teri of the Rio Mavaca, from seeds of an *Anadenanthera* species.

DMT-N-oxide [with Bufotenine, Bufotenine-N-oxide, and DMT]

Marini-Bettòlo et al. 1964

Desmodium gangeticum

Aerial parts [0.033%; 0.21 gm. + 0.12 gm. (latter as chloroform soluble acetate) from 1 kg. of fresh wet material.] Banerjee & Ghosal 1969.

Green Plant (Stem and Leaf) Ghosal 1972a and Ghosal & Bhattacharya 1972 and Ghosal *et al.* 1971

Roots (Amount not given.) Ghosal & Bhattacharya 1972 and Ghosal *et al.* 1972e (0.12 gm. + 0.02 gm. from 1.6 kg. of dried roots.) Ghosal & Banerjee 1969

Fruit Ghosal 1972a

Seeds Ghosal & Bhattacharya 1972 & Ghosal et al. 1970b

Desmodium gyrans

Leaves (0.18+ gm. from 2 kg) Ghosal et al. 1972a

Stem / leaf Ghosal et al. 1970b

Roots. Minor. Amount not given. Ghosal et al. 1972a

Desmodium pulchellum

Whole plant (Minor alkaloid) Ghosal & Mukherjee 1964 (Mention. Ghosal & Mukherjee 1965) (Amount not given) Ghosal & Mukherjee 1966

Trout's Notes FS-X7: N,N-Dimethyltryptamine-N-oxide

DMT-N-oxide

Stem and leaf of young seedling [0.023% by dry weight; 19% of 0.12% Total alkaloid] Ghosal *et al.* 1972c Stem and leaf of mature plant [0.070% by dry weight; 5% of 1.4% Total alkaloid] Ghosal *et al.* 1972c Root of young seedling $[\sim 0.011\%$ by dry weight; 3% of 0.37% Total alkaloid] Ghosal *et al.* 1972c Root of mature plant [0.121% by dry weight; 11% of 1.1% Total alkaloid] [Also, in same paper: 1.8 kg of dried roots yielded 0.18 gm + 0.042 gm; i.e. 0.012%.] Ghosal *et al.* 1972c

Fruit (green) of mature plant [$\sim 0.007\%$ by dry weight; 72% of 0.01% Total alkaloid] Ghosal *et al.* 1972c

Seeds (ripe) mature plant (trace) Ghosal et al. 1972c

Root, Stem-leaf and Seeds Ghosal et al. 1970b

Root, stem-leaf and fruit (Amounts not given) Ghosal 1972a

Desmodium triflorum

Leaf [trace] Ghosal et al. 1972d

Stems [3% of 0.008% total alkaloids] (dry weight) Ghosal *et al.* 1972d

Roots [4% of 0.01% Total alkaloids] {41 mg. from 8.3 kg} (dry weight) Ghosal *et al.* 1972d

Minor in roots. (amount not given) Ghosal et al. 1971

Lespedeza bicolor var. japonica

Rootbark but not leaves. Minor component. Amount not clearly indicated. Morimoto & Matsumoto 1966

Mucuna pruriens

In root, stem-leaf and pod. Ghosal 1972a

In leaf/stem/seed. Ghosal et al. 1970b

0.003% in fresh leaves Ghosal *et al.* 1971

Malpighiaceae

Banisteriopsis argentea Spring ex Juss In stem and leaf: Ghosal 1972a

DMT-N-oxide in leaf. [26 mg from 1.8 kg] Ghosal &

Mazumder 1971 and Ghosal *et al.* 1971

Diplopterys cabrerana as = *Banisteriopsis rusbyana* is listed in literature but lacks a reference.

Diplopterys cabrerana is often equated with Banisteriopsis rusbyana but, according to Bronwen Gates, this is incorrect. A sterile voucher specimen (from Klug) was misidentified by Morton (who was the foremost expert on the genus at that time) as *B. rusbyana* and the material was then analyzed. The mistaken assertion that the analyzed material was in fact *B. rusbyana* served as a basis for subsequent waves of analysis on similarly misidentified material. It is extremely doubtful if *ANY* verified *B. rusbyana* has ever been analyzed

Myristicaceae

Virola sebifera

Present in bark. Kawanishi et al. 1985

Activity:

DMT-N-oxide is entheogenic when smoked. Shulgin 1993: BPC Lecture, Maui, Hawaii, August.

Metabolism & metabolic formation:

Barker et al. 1980 noted it was "essentially resistant to metabolism" by MAO "under aerobic conditions".

They found Iproniazid pretreatment prevented 90% of the metabolism of DMT into DMT-N-oxide from occurring (in rat brain homogenates).

Smith et al. 1962 found MAO effective when anaerobic.

In mouse liver homogenates, the N-oxide was reported to be the major metabolite of DMT (+ IAA & 2 unknowns) by Fish *et al.* 1955.

Barker *et al.* 1978 found this true in the rat as well (accompanied by MMT & formaldehyde) and also found DMT-N-oxide was metabolized into DMT, MMT & formaldehyde (indicating interconversion).

See also: Barker *et al.* 1978 Smith *et al.* 1962 Szara & Axelrod 1959

DET

Diethyltryptamine; 3-(2-Diethylamino-ethyl)indole; 3-(2-Diethylaminoethyl)indole;

N,N-Diethyltryptamine; N,N-Diethyl-tryptamine;

N:N-Diethyltryptamine; N-N Diethyltryptamine;

Tryptamine, N,N-Diethyl;

3-[2-(Diethylamino)ethyl]indole;

Indole, 3-[2-(Diethylamino)ethyl]; Diäthyltryptamin; DET; D.E.T.; DT; T-9

WLN: T56 BMJ D2N2&2 Hayward: 6R4Y5L(CCN(CM)2)=LNHY Usdin & Efron 1979

Chemical Abstracts Registry Number: 61518 [000061518] [61-51-8] Usdin & Efron 1979

MW 216.19 Ott 1996 MW 216.3 Clarke's 1986

 $C_{14}H_{20}N_{2}$

C 77.72%, H 9.32%, N 12.96% Ott 1996

Schedule 1 controlled substance: Ott 1996

Free base:

Orange, oily liquid. Soluble in ethanol, chloroform Clarke's 1986 White waxy crystalline material. Sharp smell mp 84-97° (recrystallized from hexane by seeding) Shulgin & Shulgin 1997 mp 85-89° (from petroleum ether) Ott 1996 Distills at 175-185° at 0.05 mm to yield a white oil that crystallized spontaneously Shulgin & Shulgin 1997 Distilled as clear oil at 0.003-0.004 mm by Barlow & Khan 1959 (temperature not given) Soluble in ether, chloroform, toluene, hot hexane, methylene chloride, dioxane, tetrahydrofuran, alcohols, dilute acids. Prob-

Chloroform-Water partition coefficient: 1.85

ably in acetone as well.

Gessner *et al.* 1968 (This was manually changed in the UT library copy to 6.12)

DMT-N-oxide

Hydrochloride:

(appears unstable; darkens over time): mp 87-89° (white crystalline powder) Clarke's 1986 mp 169-171° (from isopropanol after addition of a few drops conc. HCl) mp 170-171° (From ether saturated with anhydrous HCl and recrystallized from benzene-methanol) Shulgin & Shulgin 1997 mp 172-173° Ott 1996 mp 172-173° (from ethanol-methyl ethyl ketone-ether) Barlow & Khan 1959 Soluble in water

Picrate:

mp 170.5° (from ethanol) Barlow & Khan 1959

Assays:

Usdin & Efron 1979 cited Udenfriend 1969 but we have not been able to obtain a copy.

An analytical procedure for DET as used in FDA labs:

Using tlc (no heat for drying applied samples) with ammoniaalcohol (1:4), let develop for 10 cm, air dry without heat for around 5 minutes and then spray with formaldehyde (40%)-HCl (1+3)-ethanol (10:10:20). Heat plate at 100° C for 5-7 minutes.

Yellowish brown spots that under long wave UV (~3660 Å) fluoresces in yellow-orange-green region.

Rf is around 0.45.

Sensitivity is 0.4 µg.

Martin & Alexander 1968

Colorimetric reagents: See color reaction p. 144

TLC: See Rf Table p. 169-176

UV:

In morpholine-toluene (1:9): Absorbs under UV at 254 nm Alliston *et al.* 1971

UV λ_{max} : 218, 278 (0.1N HCl) Sunshine 1981 (see also p. 89 & 119)

UV λ_{max} : 275, 219 (0.1N NaOH) Sunshine 1981

UV λ_{max} : 281, 289, 297 (0.1N H₂SO₄) Sunshine 1981 See also (graphic) Lee 1985

Fluorescence:

"Strongly fluorescent" in spectrofluorometric assay DET Fluorescence

		(nm)	
	Excite	Emit	
0.1N H ₂ SO ₄	278	355	
0.001N HCI	279	350	
0.1M Phosphate buffer			
(pH 7)	277	355	
1N NH ₄ OH	282	363	
1N NaÕH	282	419	
0.001N HCl (Optimum solvent choice)			
		< · · -	

Gillespie 1969: Graphic of spectra on page 617 Exhibits native fluorescence ($10^{-3}\mu$ HCl) λ_{ex} 279; λ_{em} 352 nm (sensitivity 0.5 ppm) Sunshine 1981 DET (DMT & DPT are identical) Fluoresces at 340 nm with excitation at 290 nm Slits (Em./Ex.) 10/10 Filter 310 nm Limit 0.5 mcgm Methanol as solvent on Perkin-Elmer MPF-2A fluorescence spectrophotometer DeZan *et al.* 1971

IR:

Base (in cm⁻¹) 741, 804, 970, 1018, 1067, 1090, 1120 HCl (in cm⁻¹): 717 (br), 847, 968, 1017, 1110 Shulgin & Shulgin 1997

MS:

Shulgin & Shulgin 1997: (m/z) $C_5H_{12}^{++}86 (100\%)$; indolemethylene⁺ 130 (6%); parent ion 206 (1%)

IR, MS & GC: Clarke's 1986

GLC of HFB derivative of DET: VESSMAN et al. 1969

Synthesis:

Barlow & Khan 1959 (using method of Speeter & Anthony 1954) Shulgin & Shulgin 1997 (2 routes) Speeter & Anthony 1954

Activity: Hallucinogen

Böszörményi *et al.* 1959 (0.65-1.0 mg/ kg/ im; the "*most suit-able*" dose was 0.8 mg/ kg/ im)

Shulgin & Shulgin 1997 (see dosage ranges farther below) Szara & Hearst 1962 (using 1 mg/ kg/ im)

Psychoptic at 1 mg/kg/im Ott 1996 cited Szara & Rockland 1961 & Szara et al. 1966

Szara *et al.* 1966 used 1 mg/ kg/ im and reported quite different (and far more negative) results than Böszörményi. See more comments below.

Szara 1964a showed that DET and other simple alkylated tryptamines "produce a characteristic regional shift in the serotonin distribution in rabbit brains: an increase in the hypothalamus without a significant change in the amygdala-hippocampal region"

Human Dose:

60 mg/ im Usdin & Efron 1979 cited Szara 1964b 14-70 mg/ im Usdin & Efron 1979 cited Jacob 1966 50-100 mg orally (30-40 mg was reported effective when smoked; several accounts used 60 mg iv and /or im) Shulgin & Shulgin 1997

Duration: 2-4 hours

Shulgin & Shulgin 1997

Orally active; earlier comments by Shulgin claiming only parenteral activity were noted to have been erroneous in Shulgin & Shulgin 1997 **EEG:** Böszörményi *et al.* 1959 reported an acceleration in rhythm and a decrease in amplitude similar to that reported for mescaline and LSD.

Reported effects in humans:

After being given DET, one of Böszörményi's subjects remarked "If we could inoculate this into all men, human interrelations would undoubtably improve greatly"

Böszörményi *et al.* **1959:** (Using 30 normal test subjects and 41 mental cases)

Administration time was noted in only one case; that being 10:55 AM.

Intramuscular administration of 0.7-0.8 mg/kg was likened to the effects of a mild to moderate dose of mescaline with some similarities to LSD.

The initial vegetative symptoms, appearing 8-15 minutes after IM injection, were largely perceived of as unpleasant (including vertigo, pupillary dilation, nausea, sweating, tremor, an increased pulse rate and blood pressure) but, in the majority of cases, gave way to a "*meditative, pleasant, euphoric mood, with the contact maintained*." Euphoria was noted in all but 4 subjects.

They reported a "*loosening*" of consciousness, a weakening of concentration ability and disturbances of spatial, temporal & body perceptions. Distortions of spatial perception included planes and outlines, a curving slightly of flat walls, objects appearing farther away or closer than normal. An increased hearing sensitivity was noted in some subjects.

Other symptoms noted included depersonalization, illusions, a loosening of associations that was accelerated and usually whimsical, a tendency towards philosophical thinking, perceptions of approaching "*truth*" or a feeling of getting a truer picture of the world, a drive to speak accompanied by difficulty in wording, colors & visual phenomena including arabesque and abstract patterns, scenic pictures, a brightening of colors and seeing relief in flat surfaces, an "*illusionistic view*" of their surroundings, occasionally the hallucinations of sounds, vertigo, a sense of timelessness, and "*not infrequently*" synaesthesia. The latter (and accompanying visual imagery) was said to frequently be stimulated by music, which was reported as having a "*remarkably pleasant effect*."

Most of their subjects reported a sensation of clouded consciousness or a feeling of being half-asleep that varied during the course of the experience. All of their normal subjects remained contactable during the entire experience.

Some of their subjects showed anxiety or increased activity (often accompanying each other).

A sense of numbness or tingling was commented on but less frequently with DMT.

They also noted an impairment of drawing ability in a schizophrenic painter.

Aftereffects included "slight fatigue, mild headache, depression and insomnia" none of which exceeded one day's duration.

An interesting after effect was that some of their normal test subjects expressed artistic tendencies afterwards that they had not previously shown before.

2 began painting pictures, one wrote a poem and another wrote a short story.

Another interesting feature was that the majority of the pre-

viously uncommunicative, chronic schizophrenics in their study became more communicative, often volunteering information that had not come out in prior therapeutic contacts. A favorable change was also noted in several psychopathic and several severely hysterical patients. It was suggested that DET was superior to barbiturates for "*exploration and for establishing psychotherapeutic contact*"

One woman became talkative after having suffered from catatonic schizophrenia for 5 years; another with an acute paranoid reaction acted out her conflict under the influence of DET and became symptom free afterwards. When later suffering a relapse due to other difficulties she asked for DET because she believed it had been beneficial.

Many of the patients with psychopathology spoke truthfully about their problems but it was noted that many also protested against being given another dose because they felt they had talked too much while under the effects of the DET.

One neurotic subject, described as deeply religious, "*lived through*" an ecstatic union with God during the experience that he felt "*proved his religiousness to be profoundly sincere and not simply apparent*."

Interestingly several subjects likened the experience to the delirium produced by typhus, pneumonia or other trauma capable of inducing such profoundly altered states of consciousness.

Their conclusions were that this substance was superior to LSD or mescaline for psychotherapeutic work due to its combination of effectiveness and short duration (3 hours) and that the effects produced suggested a possible correlation between endogenous "tryptamine derivatives and the exogenous reaction types of mental condition"

Szara 1966: (Using 10 normal test subjects and 10 chronic schizophrenics)

All subjects were given 1 mg/ kg/ IM with a 9AM administration time

Szara *et al.* 1966 made the comment that their subjects showed far more unpleasant side effects and generally disliked the drug in contrast to Böszörményi who primarily used smaller dosages.

Educational levels, prior familiarity with the observers and the environment of administration were noted as potential factors (Szara's *normal* subjects had no music, were described as unemployed, poor & not highly educated. All were from a depressed mining region and were further said to be "*culturally deprived*" with "*little interest in introspection, philosophy, music, art, etc.*" None were experienced with any hallucinogen and were simply told they were going to receive an injection "*which might make them feel different*"

In contrast, Böszörményi's normal subjects had access to music if desired and were mostly friends and colleagues of the same people who dosed them. Those who were not actual colleagues were other professionals or artists. It was not noted if any had familiarity with similar substances or what they were told ahead of time.

Further Szara's subjects were reported to have had negative expectations once the first few subjects had been given the drug and approached it with trepidation expecting an unpleasant experience.

A statistically meaningful correlation between the amount of 6-HydroxyDET formed and the chronological order of admin-

istration was observed in this study suggesting this may have been an actual factor in its formation

A positive correlation was also found between the formation of 6-HydroxyDET excreted in the urine and the autonomic, neurological and psychological changes observed in the subjects. The three showing the highest excretion rates (subjects 5, 6 & 10) were also the ones who reported having the most unpleasant experience.

All of their subjects experienced sweating and dizziness. Most experienced at least some degree of nausea; 5 experienced slight nausea and 4 others vomited.

Distortion of their sense of time was common.

9 out of the 10 experienced a rise in blood pressure with a fall noted in the other.

Pupillary dilation was also common.

A number of bizarre somatic complaints were noted.

Visual distortions, loosening of associative thought, "wavelike exacerbation of all symptomology and rapidly shifting levels of awareness were noted in all subjects." Visual phenomena varied from simple focusing difficulties and light hypersensitivity to hallucinations that were perceived of as real.

Auditory hallucinations were noted in 6 of the 10 and olfactory hallucinations in 1.

Synaesthesia was occasionally noted.

Half of their normal subjects expressed paranoid ideation directed at their observers, two called them "*queers*" and accused them of seduction attempts.

6 of the 10 found the experience moderately to extremely unpleasant. All but two reported at least some anxiety. 4 of the 10 showed psychological reactions to placebo including 2 who found it unpleasant and one who did not but did express paranoid ideation)

They commented that the three subjects showing the most unpleasant experience were also the same ones who showed the highest rise in blood pressure.

[Szara reported 3 of their chronic paranoid schizophrenic subjects became less defensive, more approachable and communicative with their observers. The remainder showed exaggerated versions of their normal behavior. Most of the schizophrenics became pale and shaky. Most complained of feeling sick or else vomited. Tremors were seen in several. 6 of the ten showed an increase in blood pressure, one showed a decrease and the other two remained unchanged. 8 showed definite increases in anxiety levels whereas two seemed to be tranquilized. The least psychotic of the 10 described the experience similarly to the normal subjects.]

Use in psychotherapeutics:

Faillace *et al.* 1970 Vourlekis *et al.* 1967

Reported effects in animals:

Böszörményi et al. 1959:

One dog given the HCl at 1.5 mg/ kg/ IM showed excitation after around 15 minutes, running "*up and down, did not recognize his master, did not listen to his name and refused food*" This lasted for around 15-20 minutes. Similar results were observed in another dog after being given 2 mg/ kg.

The HCl given intravenously to rats produced an excitation that was dose dependent but did not produce convulsions (in doses of 1, 2, 5 & 7.5 mg/ kg). It caused running, the "smaller doses causing them to climb, stand on two legs and retreat backwards." Clonic convulsions resulted from a 10 mg/ kg dose IV but there were no fatalities. After a response time of 15-20 minutes a depressive phase was noted.

LD50:

32 mg/ kg/ IV in mice 24.5 mg/ kg/ IV in dogs Böszörményi *et al.* 1959:

Some Biological trivia:

Szara & Hearst 1962 reported that rats partially metabolize DET into 6-HO-DET (aka 6-HDET), and which was determined was more active than the parent.

He found that it produced behavioral effects similar to DET qualitatively and quantitatively but at lower dosages. (Rats also metabolize DET into 3-IAA which is not active)

After further studying it in humans, they found that, as had been the case for rats, the 6-HO-DET was more potent (some 5-6 times). (See also comments made on Szara *et al.* 1966 above)

Even more interestingly, Szara found that the wide disparity in both threshold and effects noted in rats and humans using DET was directly correlatable to the levels of 6-HO-DET that were excreted in their urine! (In rats, both free and as its glucuronic acid conjugate)

This work suggested that this active metabolite is involved in its activity and the subjective effects experienced from DET may be influenced by the ability of the user to 6-Hydroxylate it. Perhaps also helping to explain the wide individual variability not just in *per dose* response but in duration?

[They also presented a method to take collected urine excreted after the administration of DET to rats (or humans) and incubate it with bacterial glucoronidase allowing the recovery of 6-HO-DET from alkaline solutions with an organic solvent. The rats appeared to excrete it both free and conjugated but it was not clearly stated how humans excrete it. Free was implied but this depends on whether their wording can be taken literally or not.]

One feature that should not be forgotten though, is that while in rats 70% of the administered DET *could* be accounted for, in humans, Szara *et al.* 1966 was completely *unable* to account for an average of **82.6%** (ranging from 71.6% to 88.4%). The highest rates of excretion of DET as the 6-Hydroxylated metabolite was 8.70% in the schizophrenics and 7.6% in a normal subject. The lowest was 2.91 and 2.70% respectively.

[Interestingly they reported a delayed excretion curve for most of the schizophrenics.]

It is also important to remember that over twice as much DET was metabolized to 3-Indole acetic acid than was excreted as the 6-hydroxylated product (whether free or conjugated)

This all clearly needs much more study. (See comments on DMK as a DMT metabolite)

A similar disparity in results has been noted for the 4-substituted DET homologues enjoyed by humans but I have located no metabolic studies. 4-Acetoxy-DET & 4-Hydroxy-DET both have a wide disparity in threshold, activity *and* duration from one user to the next.

Intriguing topic but presently one with many questions and few, if any, answers.

In Holmstedt *et al.* 1967, a fascinating conversation was held on this topic.

DET

In it, it was pointed out, by Szara (a panel member in this discussion), that the activity of this compound had been called into question by Harris Isbell on the basis of finding that 6-Hydroxy-dimethyltryptamine was found inactive in "behavioral tests".

In this same conversation, Dr. Isbell (also a panel member in this discussion) later commented that 6-HO-DMT had been shown to be inactive in doses up to 1 mg/kg in humans.

The obvious question as to why he did not test the 6-HO-DET, itself, was neither raised nor answered.

Or, if he did evaluate it, no one mentioned it.

This is a particularly important issue in my mind as Szara had reported successfully conducting *multiple* double blind tests with it in humans.

Or is something else involved with the 6-HO-DET that is thusfar escaping everyone?

I am at a loss to understand this but am quite fascinated.

Szara also noted that 6-Fluoro-DET prevented this hydroxylation from occurring so the only effects from it were limited to autonomic effects, pupillary changes and changes in blood pressure, but it does not produce the effects hallucinogens are noted for, despite it reaching the brain.

In Szara *et al.* 1967 it was noted that this suggested that 6-Hydroxylation was somehow involved in the activity of DET despite it being a **minor** metabolic pathway in humans.

Vane *et al.* 1959 studied DET along with many other tryptamine derivatives in its effects on the isolated rat stomach strip, before and after MAO inhibition, as compared to serotonin. They found it to be much less active than serotonin, but more so than DMT, and that this activity was **not** affected with an MAOI.

Barlow & Khan 1959 found it to be far less active than serotonin on both the rat uterus and rat fundus strip. It was also reported to be a *"feeble"* serotonin antagonist.

Cerletti *et al.* 1968 found 6-Hydroxy-DMT and 6-Phosphoryloxy-DMT to be practically inactive in the biochemical tests they evaluated except for showing around half as much pressor activity as bufotenine.

They did not test 6-Hydroxy-DET or 6-MeO-DET

Kline *et al.* 1982 found 6-Methoxy-DMT to be much less active than 5-MeO-DMT.

They did not test 6-Hydroxy-DET or 6-MeO-DET

Gessner *et al.* 1968 found 6-Methoxy-DMT less effective than DET or 4-Methoxy-DMT or 5-MeO-DMT at disrupting conditioned avoidance. (They found 5-Methoxy-DET to be more effective than psilocin or DET)

They did not test 6-Hydroxy-DET or 6-Methoxy-DET.

See:

Ott 1996 Entry #5, page 432 Shulgin & Shulgin 1997 Entry #3 pages 396-403 Usdin & Efron 1979 Entry #381, page 125

DPT

3-(2-(N,N-Dipropyl)aminoethyl]-indole;

N,N-Dipropyltryptamine; Tryptamine, N,N-Dipropyl-; 3-[2-(Dipropylamino)ethyl]indole; Indole, 3-[2-(Dipropylamino)ethyl]; Dipropyltryptamine;

Dipropyltryptamin; DPT

MW 244.38 Ott 1996

$$C_{16}H_{24}N_2$$

C 78.64%, H 9.90%, O 11.46% Ott 1996

Not scheduled: Ott 1996

WLN: T56 BMJ D2N3&3 Hayward: 6R4Y5L(CCN(CCM)2)=LNHY Usdin & Efron 1979

Free base:

Soluble in alcohols, chloroform, ether, dioxane Distills as white oil 145-155° at 0.08 mm Shulgin & Shulgin 1997

Hydrochloride:

Soluble in water

mp 174.5-178° (crystals) Ott 1996

mp 174-175° (as fine white powder from 1 gm base dissolved into 5 ml IPA, acidified with conc. HCl and precipitated by the addition of 20 ml anhydrous ether) Shulgin & Shulgin 1997

mp 176-178° (from ethanol-methyl ethyl ketone-ether) Barlow & Khan 1959

mp 178-179° (crystals from benzene-methanol; after precipitation from ether by the addition of anhydrous HCl) Shulgin & Shulgin 1997

pKa of HCl is 8.6

Vane et al. 1959

Assays:

Will react with Ehrlichs & other Indole reagents. Should be purple with Xanthydrol

TLC: See Rf Table p. 169-176

Phillips & Gardiner 1969 used 254 nm to visualize (worked best on 6060) but found Iodine in methanol worked well in all their systems. Thought the best results for speed and effectiveness was 254 nm UV on Eastman Chromatogram 6060 sheets using methanol-ammonia (sp. gr. 0.88) (100:1.5) as the moving phase.

Fluorescence:

See DeZan et al. 1971 under DET above

IR:

HCl: (in cm⁻¹): 759, 774, 831, 987 (br.), 1084, 1101 Shulgin & Shulgin 1997

DPT

Synthesis:

(2 routes): Shulgin & Shulgin 1997

Activity:

Hallucinogen Shulgin & Shulgin 1997 Szara & Hearst 1962 Toad 1999

Dose:

14-70 mg/im Usdin & Efron 1979 cited Jacob 1966

Psychoptic above 1 mg/kg Ott 1996 cited Szara *et al.* 1961 Used in Psychotherapy at 90-100 mg Ott 1996 cited Grof 1977 (See also Grof *et al.* 1973)

Dosage recommendations: (Toad 1999 unless noted otherwise) The free base can be smoked; the hydrochloride should not be.

- [For an example of the conversion to the base, see: http:// www.erowid.org/entheogens/dpt/dpt primer.shtml]
- 20-30 mg of the freebase smoked works well for most but some have needed 100 mg or more.
- 25 mg of HCl is recommended for a starting point for insufflation. There is at least one bad report of too much intensity following insufflation at 60 mg but there is wide variability between people. Some people have reported needing 200 mg insufflated to get effects!
- 12 mg of the HCl given IV was reported to be strongly active. Shulgin & Shulgin 1997
- 15-20 mg im is recommended for novices and 30 mg for experienced travelers. Grof used up to 160 mg im.
- 100-250 mg orally Shulgin & Shulgin 1997. Toad notes that this route requires the most material and shows very unpredictable results between individuals.

Duration:

2-4 hours (500 mg oral lasted 12 hours)

Shulgin & Shulgin 1997

- Onset when smoking may be immediate and effects will last around 20 minutes.
- Onset following insufflation will take up to 15-30 minutes, peak in around an hour then coast down for another 3.
- Onset following IM injection is within 5 minutes, followed by a plateau of more than an hour with a coast down for two more.

Toad 1999

Trip reports:

1999 *The Entheogen Review* 8(2): page 55 (Gwyllm: 35 mg HCl insufflated) & page 56 (Case: 45 mg HCl insufflated) see also www.erowid.org

Use in psychotherapeutics:

Faillace *et al.* 1967 Vourlekis *et al.* 1967 McCabe & Hanlon 1977 Soskin *et al.* 1973 & 1975 Vourlekis *et al.* 1967 Faillace *et al.* 1970 Grof *et al.* 1973a & 1973b Grof & Halifax 1978 Rhead *et al.* 1977 Richards 1975 & 1979/1980 Richards *et al.* 1977 & 1979

Biological trivia:

Vane *et al.* 1959 studied it along with many other tryptamine derivatives concerning its effects on the isolated rat stomach strip, before and after MAO inhibition, as compared to serotonin. They found it to be much less active than serotonin, but more so than either DET or DMT respectively, and that this activity was not particularly affected with an MAOI.

Barlow & Khan 1959 found it to be less active than serotonin on both the rat uterus and rat fundus strip. At higher concentrations it did stimulate the fundus strip preparation but the muscular contractions were much slower than with serotonin as was recovery. Despite showing a high stimulant activity on the rat fundus strip (2.5% that of 5HT), this was not so marked on the rat uterus (0.5% that of 5HT). [On a per molar comparative basis]

See: DPT primer Toad 1999 The Entheogen Review 8(1): 4-6.

Ott 1996 Entry #10, page 434 Shulgin & Shulgin 1997 Entry #9 pages 427-431 Usdin & Efron 1979 Entry #385, page 126

Shulgin reported creating 4-Hydroxy-DPT but found it hard to synthesize; the pharmacology was never fully explored. [#20; pages 479-480 in TIHKAL]

Di-isopropyltryptamine (DIPT)

We do not cover this alkaloid but wanted to include the following for our reader's convenience:

TLC:

- Rf 0.42 in Methanol-Ammonia (sp. gr. 0.88) (100:1.5) on silica gel
- Rf 0.56 in Methanol-Ammonia (sp. gr. 0.88) (100:1.5) on silica gel Eastman chromatogram 6061
- Rf 0.56 in Methanol-Ammonia (sp. gr. 0.88) (100:1.5) on silica gel with fluorescence indicator Eastman chromatogram 6060
- Rf 0.15 Chloroform-Methanol (9:1) on alkaline silica gel treated with 0.1N sodium hydroxide

Phillips & Gardiner 1969



a rooftop Phalaris garden

Lespedamine

1-Methoxy-N,N-dimethyltryptamine; 1-Methoxy-3-(2-(N,N-dimethyl)aminoethyl]-indole; 1-Methoxy-DMT; 1-MeO-DMT; Lespedamin.

Not a controlled substance

C13H18N2O

MW 218.3

C 71.52, H 8.31, N 12.83

Free base:

Distills as colorless oil 113-114° at 0.28 mm. (Also given as 110°/0.2 mm) Morimoto & Oshio 1965

Soluble in methanol, chloroform. Morimoto & Oshio 1965

Hydrochloride:

mp 163-164° (decomp.) colorless feathery needles from benzene-methanol. Morimoto & Oshio 1965

Picrate:

mp 160-162° (decomp.) yellow prisms from methanol. Morimoto & Oshio 1965 mp 163° Morimoto & Matsumoto 1966

Styphnate:

mp 169-170° (decomp.) yellow needles from methanol. Morimoto & Oshio 1965

Methiodide

mp 196° Colorless blades from acetone-ether. Morimoto & Oshio 1965

Assays:

Colorimetric reagents:

Ehrlichs: Wine-red Morimoto & Oshio 1965 Morimoto & Matsumoto 1966 (Its N-oxide also forms a wine-red color with Ehrlich's: Morimoto & Oshio 1966)

Paper chromatography:

Rf: 0.90 in Acetic acid-Butanol-water (1:4:5) Rf: 0.87 in t-Butanol-Water-Formic acid (21:9:0.6) Rf: 0.85 in t-Butanol-Water-Formic acid (21:3:0.6) Rf: 0.93 in Propanol- 28% Ammonia (5:1) Morimoto & Matsumoto 1966

UV:

(MeOH): λ_{max} (log ϵ): 223.4 (4.49), 2788.0 (3.66), 291.0 (3.68) Morimoto & Oshio 1965 NMR & IR: Morimoto & Oshio 1965

Structure:

Morimoto & Oshio 1965 (Proven only by its degradation into DMT via 4 routes)

Synthesis:

Apparently not done.

Isolation:

Morimoto & Matsumoto 1966 Morimoto & Oshio 1965

Occurrence:

Lespedeza bicolor var. japonica

Morimoto & Matsumoto 1966: Almost 2 grams from 9.7 kg dry leaf. Minor alkaloid in rootbark.

Morimoto & Oshio 1965: 0.035% dry wt. (4.31 g from 12.4 kg of dried leaves) [35 mg from 100 gm of dry leaf]

Activity:

Unknown.

Possibly a hallucinogen but actual activity **and** safety **and** dosage **and** pharmacokinetics appear to be unknown and curiously unstudied.

It seems just as likely to be nonhallucinogenic.

The only known human bioassay appears to be that of 30 dry grams of red fall-colored leaves of *Lespedeza bicolor* grown in US which was reported to be successfully ingested as a fully active ayahuasca analog (Personal communication with "Wyrm")

However bear in mind this plant also contains both DMT and Bufotenine and only the *japonica* variety has actually seen any analysis.

Other *Lespedeza* species are also rumored to be active but specific details seem to be thus far lacking.

More work is clearly needed.

Cerletti *et al.* 1968 did not study this compound but did evaluate 1-Methyl-DMT

They reported that, in comparison of 1-**Methyl-DMT** to psilocin, there was an abolishment of any effects on spinal reflexes (based on knee jerk response), 71% as much serotonin antagonism and 74% of the pressor activity of psilocin.

Compare this to comments made under 1-Methylpsilocin & 1-Methylpsilocybin.

1-substitution in general leads to an increase in antiserotonin activity and a decrease of reflex activation when compared to the corresponding unsubstituted homolog. 1-methyl-LSD and 1-acetyl-LSD show a similar increase in antiserotonin activity.

Toxicity:

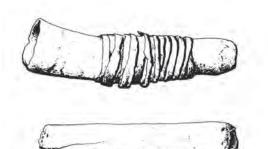
Unknown

LD₅₀: Unknown



Despite Ghosal isolating DMT from the rhizome of *Arundo donax* (in India), Appleseed found it to be lacking from almost all samples examined (US). Appleseed only observed it once; in a sample of young, skinny, white, feathery roots occurring in a rootbound containerized specimen.

"More than you need to know?"



Tubular pipes, puma bone,13 cm & 11.2 cm, Inca Cueva (IC c7), Puna de Jujuy, Argentina (after Fernández Distel 1980: Fig. 5), ca 2130 BC



Tubular pipes, Huachichocana (CH III), Puna de Jujuy, Argentina. Left to right: coll. # 2040, red sandstone, 27.8 cm; coll. # 2039, red sandstone, 34.7 cm; coll. # 2037, andesite, 23.8 cm; coll. # 2038, red sandstone, 22.7 cm; Museo Arqueológico, Tilcara, Jujuy, Argentina. ca 1450 BC

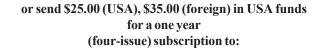
Tryptamine pipes made from puma bone & from stone. Images provided by Manuel Torres in 2005.

THE ENTHEOGEN REVIEW The Journal of Unauthorized Research on Visionary Plants and Drugs

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Will Beifuss, Richard Glen Boire, Jim DeKorne, Earth Erowid, Francesco Festi, Robert Forte, Elizabeth Gips, Alex Grey, Jon Hanna, Albert Hofmann, Ernst Jünger, Thomas Lyttle, Terence McKenna, Dan Merkur, Jean Millay, J.P.
Morgan, Mulga, Jonathan Ott, Dale Pendell, Will Penna, René Rikkelman, Giorgio Samorini, Nick Sand, Alexander Shulgin, Daniel Siebert, Myron Stolaroff, Rick Strassman, Sylvia Thyssen, D.M. Turner, Leander J. Valdés III, R. Gordon Wasson, & numerous anonymous and pseudonymous contributors.

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Journal of Psychoactive Plants ぐ Compounds

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Trout's Notes on Tryptamines



Caesalpinia pulcherrima Above: cultivated in US A plant considered to be "especially sacred to Shiva"









Acacia obtusifolia phyllodes Photos above by Zariat

Note the presence of tiny reddish resinous granules on the irregular margin of the phyllode

Acacia confusa (center left: AUS; Photo by Mulga) (bottom left: seeds)

> Dictyoloma incanescens seeds lower right





A few of the many plants assayed by Johnny Appleseed using TLC



Justicia pectoralis var. stenophylla



from Tin-Tazarift site, Tassili Plateau, southern Algeria *"Round-heads"* pictorial phase 9000-7000 years old Rendering by Giorgio Samorini reproduced with permission

Ancient rock art

4-Substituted Indoles



Psilocybe cyanescens (Australia) Photo by Snu Vooglebreinder

Notice bluing on stem where handled.



Trout's Notes FS-X7





Psilocybe stuntzii Photos (left & above) by JW Allen



Psilocybe samuiensis Photo (above) by JW Allen



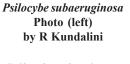


Psilocybe baeocystis Photo (above) by JW Allen

Psilocybe semilanceata Photo (right) by JW Allen

Psilocybe cubensis (Thailand) Photos (left & below) by JW Allen





Psilocybe cubensis or subcubensis? (AUS) Photo (right) by Snu Voogelbreinder

> Psilocybe cyanescens Photo (below) by JW Allen







Some Indoles

4-Hydroxyindole

4-Hydroxyindole; 4-HO-indole; Indole, 4-Hydroxy; 4-Oxy-indol; 4-idrossi-indol

C₈H₇ON

MW 133.11

C 72.16, H 5.30, O 12.02, N 10.52 Stoll et al. 1955

Free base:

mp 97-99° (Hexagonal plates from water) Stoll *et al.* 1955
mp 98° (Elongated needles from boiling petroleum) Beer *et al.* 1948
Soluble in water
Soluble in ether
Readily soluble in "*the usual organic solvents except light petroleum*"
Soluble in boiling petroleum (bp 80-100°) Beer *et al.* 1948

Picrate

red needles from benzene partly yellow at 150°, then charred at 180° Beer *et al.* 1948

Assays & colorimetric observations:

Dark blue with alcoholic ferric chloride Beer *et al.* 1948

Forms a red solution with a strong green fluorescence in Ehrlichs in the cold.

Upon being warmed, it acquires a bluish color, then grows turbid and produces a green precipitate

Beer et al. 1948

A solution of the base in water turns blue when stored.

A dilute aqueous solution will deposit a bluish precipitate over the course of 12 hours.

A 1-2% aqueous solution starts greenish-blue but over the course of 20 minutes changes to deep-bluegreen

Kept for longer it will darken and finally become a dark brown

Beer et al. 1948

UV & IR: Stoll et al. 1955

Synthesis:

Beer et al. 1948 Stoll et al. 1955

> Psilocybe azurescens compared to Psilocybe cyanescens Photos by Dr. P. C. Hickey

C10H9O2N

C 68.6%, H 5.1%, N 8.0% Beer *et al.* 1948

mp 100° (glistening needles: light petroleum (bp 60-80°) Soluble in warm light petroleum Beer *et al.* 1948

4-Acetoxyindole

Synthesis: as the precursor to 4-Hydroxyindole (above) Beer *et al.* 1948

4-Chloro-indole acetic acid methyl ester

4-Chloro-IAA methyl ester

Activity:

Has auxin activity Unlikely to have any type of psychological activity.

Occurrence:

Isolated from immature peas (*Pisum sativum*) by Marumo *et al.* 1968 [*Pisum sativum* also contains serotonin in the leaf. Smith 19977]

4-Hydroxytryptophan

4-Hydroxy-L-tryptophan; 4-HTP

 $C_{11}H_{12}N_2O_3$

MW 220.22

mp 274° Gartz 1985i



4-Hydroxytryptamine

- 3-(2-Aminoethyl)-indol-4-ol;
- 3-(2-Aminoethyl)-4-hydroxyindole;
- 4-Hydroxytryptamine; 4-Oxy-tryptamin; 4-HT
- Very unstable. Apparently *no one* has ever successfully crystallized it.

Unable to crystallize as the free base. Stoll et al. 1955

Unable to crystallize

Distilled as clear oil 150-175° at 0.05 mm but found to be extremely

unstable.

Rapidly darkening on exposure to air. Repke *et al.* 1977b

Oxalate $(C_{10}H_{12}ON_{2})_{2}, C_{2}H_{2}O_{4}$

MW 442.5

mp 269-270° (aggregates of small joined plates from methanol) Stoll *et al.* 1955

As creatinine-sulfate:

mp 250-255° (needles from water diluted with acetone) Stoll *et al.* 1955

Assays:

Colorimetric Reagents or tests:

Gartz 1985i noted that it forms chromophores with Fast Blue B (Echtblau-B) and with Ehrlichs

Keller: (on oxalate) Olive-green changing to Grey-blue. Stoll *et al.* 1955

PDAB: Immediate purple color as spray in TLC

Rapidly changing from light-brown to black on TLC plates with exposure to air. (True whether synthetic or isolated) Repke *et al.* 1977b

As creatinine sulfate on paper:

- Alkaline silver: Black
- Ehrlichs: Rapid. Blue-purple turning Grey then turning Blue

Ninhydrin-Acetic acid: Brown-purple (visible);

Bright Green-blue (UV)

Ninhydrin-Pyridine: Grey-purple

- Pauly's: Reddish-brown Jepson in Smith 1969
- Blaschko & Levine 1960 reported that 4HT forms a Blue color with *Mytilus edulis* gill plate oxidase

TLC & PC:

- Rf 0.77 in *n*-Butanol-Acetic acid-Water (2:1:1) on silica gel GF
- RF 0.55 in *n*-Propanol-6% aqueous Ammonia (5:2) on silica gel GF

Repke et al. 1977b

Very unstable but, if done immediately after its preparation from serotonin, Gartz 1985i found

Rf 0.55 & 0.48 in *n*-Butanol-Acetic acid-Water (2:1:1) on silica gel.

- As creatinine sulfate:
- Rf0.54 in *i*-Propanol-Ammonia (880)-Water (200:10:20) on paper [See Rf table Notes 2, 5]
- Rf 0.48 in *n*-Butanol-Acetic acid-Water (120:30:50) on paper (See Rf table Note 3)
- Rf 0.72 in *n*-Butanol-Pyridine-Water (60:60:60) on paper (See Rf table Note 4)
- Rf 0.33 in Potassium chloride (20% w/v) on paper [See Rf table Notes 1, 2, 6]
- Rf0.34 in Sodium chloride (8% aqueous w/v)-glacial Acetic acid (200:2) on paper [See Notes 1, 2, 6] Jepson in Smith 1969

IR & UV as creatinine sulfate: Stoll et al. 1955

Synthesis:

Gartz 1985i Repke *et al.* 1977b Stoll *et al.* 1955

Occurrence:

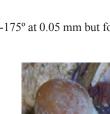
Psilocybe baeocystis Singer & Smith *Psilocybe cyanescens* Wakefield

Repke *et al.* 1977b strongly suspected they observed it in all collections of these two species based on its color change in TLC when exposed to air, its Rf in co-TLC using two solvent systems and its reaction to Ehrlichs.

Activity:

Psychotropic in animals

- Ott 1993 page 319 cited Cerletti *et al.* 1968 (who, using animals and *in vitro* tests, showed *some* similarities in action to psilocin and 4-HO-MMT)
- Haigler & Aghajanian 1977 demonstrated it was far less active than psilocin at inhibiting serotonin receptors.
- Vane *et al.* 1959 studied it along with many other tryptamine derivatives in its effects on the isolated rat stomach strip, before and after MAO inhibition, as compared to serotonin. They found it to be around half as effective as serotonin, and that this activity was not meaningfully affected by an MAOI.
- In comparison to psilocin, Cerletti *et al.* 1968 determined that 4-Hydroxytryptamine decreased spinal reflexes such as knee jerk response (instead of increasing it as psilocin does), had serotonin-like activity (rather than the antagonism of serotonin that psilocin shows), and showed greater pressor activity than psilocin.



Psilocybe cyanescens

(Oakland, California)

4-Hydroxy-MMT

Norbaeocystin

4-Phosphoryloxytryptamine; 3-Aminoethyl-1Hindol-4-ol dihydrogen phosphate ester;
3-(2-Amino)ethyl-4-indolol phosphate ester;
3-(2-Amino)-ethylindol-4-ol dihydrogen phosphate;
4-Hydroxytryptamine phosphate ester;
bis-Desmethylpsilocybine; Desdimethylpsilocybin;
Norbaeocystine; Norbaeocystin; 4-OP-T

WLN: T56 BMJ D2Z FOPQQO Hayward: 6R3R(OPVQ2)Y5L(CCZ)=LNHY Usdin & Efron 1979

Not scheduled: Ott 1996

 $C_{10}H_{13}N_2O_4P$

MW 256.20 Ott 1996

C 46.88%, H 5.11%, N 10.93%, O 24.98%, P 12.09% Ott 1996

Free base:

Soluble in water, methanol mp 188-192° (crystals from methanol) Ott 1996 mp 188-192° (dec) (from *n*-propanol containing 5% ammonium hydroxide [5:2]) Leung & Paul 1968

Assays:

Colorimetric Reagents:

Reacts with both Ehrlich's & a modified Phosphate reagent identically to Psilocybin & Baeocystin. Leung & Paul 1968 Ehrlichs: Violet Prochazka: None Iodine: Brown Gartz 1985h

TLC:

RF 0.17 in *n*-Propanol-5% Ammonium hydroxide (5:2) on Silica gel G-Kieselgur G (2:1) Leung & Paul 1968

A compound suspected to be norbaeocystin was observed at RF 0.08 in *n*-Propanol-6% aqueous Ammonia (5:2) on Silica gel GF Repke *et al.* 1977b

UV & IR (graphic) See Leung & Paul 1968

Synthesis:

Troxler et al. 1959

Isolation: Leung & Paul 1968 (via LC)

Occurrence:

Conocybe smithii Watling

"*very likely*", traces; Repke *thought* it might have been observed in chromatography Repke *et al.* 1977b

Psilocybe baeocystis Singer & Smith

Leung & Paul 1968: 0.007 & 0.009% from same material with altered workup; Repke *et al.* 1977b appears cited in the literature but the paper does not mention observing it. *Psilocybe cyanescens* Wakefield Repke *et al.* 1977b appears cited in the literature but the paper does not mention observing it

Psilocybe semilanceata (Fr.) Kummer

Gartz 1992a; Høiland 1978; ("*very likely*", traces, Repke *et al.* 1977b *thought* it might have been observed in chromatography)

Activity:

Unknown:

Ott 1996 cited Cerletti *et al.* 1968 after claiming it to be a "*probable entheogen*" [Cerletti evaluated 4-Hydroxytryptamine rather than norbaeocystine. See more under it and under 4-OH-MMT below]

See:

Ott 1996 Entry 37, page 448 Usdin & Efron 1979 Entry #417, page 135

4-Hydroxy-MMT

4-Hydroxy-N-methyltryptamine;
3-[2-(Methylamino)ethyl]-1H-indol-4-ol;
3-(2-Methylamino)-ethylindol-4-ol;
Desmethylpsilocin; Desmethyl psilocine;
4-Hydroxy-MMT;
4-HO-MMT

WLN: T56 BMJ D2M1 FQ Hayward: 6R3RQY5L(CCNHM)=LNHY (Extrapolated from other data here)

$C_{11}H_{14}N_2O$

MW 190.3 Troxler *et al.* 1959

Free base:

mp 158-165° Brenneisen *et al.*Amorphous from methanol-acetone. Troxler *et al.*Soluble in methanol. Brenneisen *et al.*Sublimes 120°C at 0.01mm Brenneisen *et al.*

Oxalate:

(C₁₁H₁₄ON₂)₂, C₂H₂O₄ MW 470.5 C 61.3%, H 6.4%, N 11.9% mp 150-152° (Prisms & Plates from methanol) Troxler *et al.* 1959

4-Hydroxy-MMT

Trout's Notes FS-X7

4-Phosphoryloxy-MMT

Assays:

Colorimetric reagents: Keller: Olive-green then grey Van Urk: Blue

Troxler et al. 1959

UV: λ_{max} [log ε]: 223 [4.3], 267 [3.5], 284 [3.4], 293 [3.4] nm Brenneisen *et al.* 1988

IR: (3400 (N-H, O-H); 3290 (N-H, O-H); 2930 (C-H); 1635; 1595; 1500; 1460; 1350; 1260 cm⁻¹) Brenneisen *et al.* 1988

MS: m/z 190 (40% M⁺⁺), 160 (2), 159 (2), 147 (100), 146 (62), 130 (2), 118 (5), 117 (3), 44 (69) Brenneisen *et al.* 1988

NMR: Brenneisen et al. 1988

Synthesis: Brenneisen *et al.* 1988 Troxler *et al.* 1959

Occurrence:

Oddly none reported yet. It is likely to be one of the minor unknown indolic spots observed by a number of workers in TLC

Activity:

Ott 1996 notes Cerletti 1968 reported psychotropic effects in animals.

In comparison to psilocin, Cerletti *et al.* 1968 determined that 4-Hydroxy-N-methyltryptamine decreased spinal reflexes such as knee jerk response (instead of increasing it as psilocin does), had 14% as much antagonism of serotonin as psilocin, and showed 70% as much pressor activity.

(Troxler *et al.* 1959 also synthesized 4-HO-Nethyltryptamine,

In comparison to psilocin, Cerletti *et al.* 1968 determined it had increased spinal reflexes, such as knee jerk response, had around 11% as much antagonism of serotonin activity, and showed as much pressor activity as psilocin.)



Baeocystin

4-Phosphoryloxy-N-methyltryptamine;
3-[2-(Methylamino)ethyl]-1H-indol-4-ol dihydrogen phosphate ester;
3-(2-Methylamino)-ethylindol-4-ol dihydrogen phosphate; Desmethylpsilocybin;
Desmethyl psilocybine; Baeocystine; Baeocystin;
4-OP-MMT

WLN: T56 BMJ D2M1 FOPQQO Hayward: 6R3R(OPVQ2)Y5L(CCNHM)=LNHY Usdin & Efron 1979

 $C_{11}H_{15}N_{2}O_{4}P$

MW: 270.28 Ott 1996

C 48.88%, H 5.59%, N 10.36%, O 23.68%, P 11.46% Ott 1996

Not scheduled: Ott 1996

Free base:

mp 194-205° (both synthetic & isolated) Brenneisen *et al.* 1988

mp 245-248° Repke & Leslie 1977

mp 254-258° (dec.) (crystals from methanol) Leung & Paul 1967

mp 254-258° (from *n*-propanol containing 5% ammonium hydroxide [5:2]) Leung & Paul 1968

Soluble in methanol. Repke & Leslie 1977 Soluble in benzene Brenneisen *et al.* 1988

Assays:

Visible without a reagent and also under UV.

Colorimetric reagents:

Sky blue spot under visible light on Silica gel (no reagent) Beug & Bigwood 1981 Dark blue with Ethanolic Ehrlich's in tlc Repke & Leslie 1977 Ehrlich's: Reddish-violet turning blue-violet Gartz 1989d Showed "a pink to purple to blue color reaction" with Ethanolic Ehrlichs in TLC on silica gel Repke et al. 1977b PDAB dark Blue-purple on silica gel Margot & Watling 1981 Reacts with both Ehrlich's & a modified Phosphate reagent identically to Psilocybin. Leung & Paul 1968 Ehrlichs: Violet Prochazka: Grey Iodine: Brown Gartz 1985h

> Psilocybe cyanescens (Oakland, CA, USA)

Baeocystin

TLC:

Rf 0.34 in *n*-Butanol-Acetic acid-Water (2:1:1) on silica gel G

Margot & Watling 1981

Rf 0.38 in *n*-Butanol-Acetic acid-Water (2:1:1) White 1979

Rf 0.22 in *n*-Butanol-*i*-Propanol-Water (8.5:1:2) on silica gel

Gartz 1989d

Rf 0.22 in *n*-Propanol-5% Ammonium hydroxide (5:2) on silica gel GF

Repke & Leslie 1977 Rf 0.12 in *n*-Propanol-6% aqueous Ammonia (5:2) on silica gel GF (found to separate from other components well in this system) Repke *et al.* 1977b

Rf 0.16 in *n*-Propanol-5% Ammonium hydroxide (5:2) on silica gel G-kieselgur G (2:1) Leung & Paul 1968

Brenneisen *et al.* 1988 used Van Urks as a colorimetric reagent & 254 nm UV to visualize.

Repke & Leslie 1977 used *n*-Propanol-5% Ammonium hydroxide (5:2) on silica gel for preparative tlc. Elution was with methanol containing 5% aqueous ammonia Preparative TLC: Gartz 1986a used *n*-Propanol-Water-Acetic acid (10:3:3) on kieselgel after an initial solvent cleanup

HPLC, HPTLC: Brenneisen et al. 1988

UV:

 $\lambda_{\max} [\log \varepsilon]: 222 [4.2], 268 [3.4], 282 (sh), 290 [3.0] nm Brenneisen$ *et al.*1988

UV identical with PSOP Leung & Paul 1967 (Leung & Paul 1968 has graphic)

Also reported by Christiansen & Rasmussen 1982b

Fluorescence:

Christiansen & Rasmussen 1982b reported that baeocystin fluoresced strongly.

IR: (KBr) 3420 (N-H); 3000; 2740; 2480 (N⁺-H); 1620; 1585; 1505; 1450; 1360; 1250; 1065 cm⁻¹ Brenneisen *et al.* 1988

IR differs from that of PSOP by having broad bands at 3275 and 1640 cm⁻¹ Leung & Paul 1967 (Leung & Paul 1968 has graphic)

MS:

MS (185°C): m/z 190 (15%), 160 (6), 159 (13), 147 (70), 146 (49), 130 (3), 118 (10), 117 (6), 91 (8). 65 (6), 44 (100) Brenneisen *et al.* 1988

m/z 190, 160, 159, 147, 146, 130, 117 & 44 (base peak) Christiansen & Rasmussen 1982b

MS: m/e 190 (8%), 160 (4), 159 (4), 147 (16), 146 (14), 130 (4), 117 (5), 44 (100) [MS shows m/e 44 & 190 (in contrast to 58 & 204 in PSOP)] Repke & Leslie 1977

See also Leung & Paul 1967

When performing MS on extracted PSOP Weeks *et al.* 1979 also found 190 (8%), 44 (100%) which they thought indicated contamination with baeocystin

NMR: Brenneisen et al. 1988

Synthesis:

Brenneisen *et al.* 1988 Troxler *et al.* 1959

Isolation:

Brenneisen *et al.* 1988 Leung *et al.* 1965 Leung & Paul 1967 & 1968 Repke & Leslie 1977

Crude isolation from initial extract using cellulose column with butanol saturated with water as elutant. Separated from PSOP using silica gel column with *n*-propanol-5% ammonium hydroxide (5:1) as elutant.

Leung & Paul 1967

Occurrence:

Conocybe cyanopus sensu Kühner Repke et al. 1977b: 0.04-0.08% in one 4 wk old sample. None in one 183 wk old sample Conocybe smithii Watling Repke et al. 1977b: 0.03-0.1% Copelandia cambodginiensis (Ola'h & Heim) Sing. & Weeks Merlin & Allen 1983: 0.02 & <0.005% Merlin & Allen 1993 Copelandia chlorocystis Singer & Weeks Weeks et al. 1979: presence was indicated Copelandia cyanescens (Berk. & Br.) Sing. Allen & Merlin 1992a: <0.025% Galerina steglichii Besl Besl 1993 Gymnopilus purpuratus (Cooke & Mass.) Singer Gartz 1989a ; Gartz 1991; Gartz 1992b Inocybe aeruginascens Babos Gartz 1987b; 0.08-0.49%; Gartz 1989d: 0.19-0.49%; Gartz 1995: 0.15-0.25%; Stijve & Kuyper 1985: 0.02 & 0.08%; Stijve et al. 1985: 0.02 & 0.08% Inocybe calamistrata (Fr.) Gill. Gartz 1986b: observed; Stijve et al. 1985: Did not detect Inocybe coelestium Kuyp. Stijve & Kuyper 1985: 0.025%; Stijve et al. 1985: 0.025% Inocybe cordyalina var. cordyalina Quélet Gurevich 1995: 0.06 mg/ gm PSOP in pilei; none in stem. Traces in older specimen from another location (Russia); Stijve & Kuyper 1985: 0.007 & 0.0034%; Stijve et al. 1985: 0.007 & 0.092%;

4-Phosphorvloxy-MMT

Stijve & de Meijer 1993: 0.025% MeOH & 0.060% in 75% aq. MeOH sat. w/ potassium nitrate; Gartz 1986b: observed it. Inocybe cordvalina var. erinaceomorpha (Stangl & Vesel.) Kuvp. Stijve & Kuyper 1985: 0.04%; Stijve et al. 1985: 0.04%. Inocybe haemacta (B. & Cooke) Sacc. Gartz 1986b: observed Stijve & Kuyper 1985: 0.034%; Stijve et al. 1985: 0.034% Stijve & de Meijer 1993: 0.003% MeOH & 0.008% in 75% aq. MeOH sat. w/ potassium nitrate. Panaeolus antillarum (Fr.) Dennis Allen & Merlin 1992a: <0.01% Panaeolus subbalteatus (Berk. & Br.) Sacc. Gartz 1989b: 0.08-0.46% in fruiting bodies; 0.12-0.21 in caps & 0.05-0.10% in stems; Gurevich 1995: 11.1 mg/ gm in pilei & traces in stem from Siberia; not detected in the samples from Central Russia except for traces in a small cap; Repke et al. 1977b: 0.0-0.005%, (1 collection was found identical to freshly dried material after 52 wks at RT); Stijve & Kuyper 1985: 0.008-0.033%; Stijve & de Meijer 1993: could not detect. Panaeolus venenosus Murr. Leung & Paul 1968 speculated this may have been an unknown spot reported by Stein et al. 1959 (based on the reported UV [220, 265 & 288 nm] and having a mp >250 dec.) Stein reported it to be water soluble, crystallizable from ethanol, insoluble in nonpolar solvents, possessing acidic & basic functions (unabile to extract it from either acidic or basic solutions) and giving a blue color with Ehrlichs. He concluded that it "seems most likely to be the active compound" in this species (Activity based on bioassays) See comments in Panaeolus subbalteatus entry under Psilocybin. Pluteus salicinus (Pers. ex Fr.) Kummer Gartz 1987a : detected; Stijve & Bonnard 1986; Stijve & Kuyper 1985: 0.0 & 0.008% Psathyrella candolleana (Fr.) Maire Gartz 1986b: observed; Psilocybe argentipes Yokoyama Koike et al. 1981 suspected they observed it but never positively identified it Psilocybe azurescens Stamets & Gartz Gartz 1994; Liggenstorfer & Rätsch 1996 Stamets & Gartz 1995: 0.19-0.41% (Tillamok, OR); 0.21-0.37% (Astoria, OR); Cultivated outdoors: 0.26-0.42% (Germany) & 0.19-0.39% (US). Psilocybe baeocystis Singer & Smith Leung & Paul 1967: 0.03%; Leung & Paul 1968: 0.03 & 0.014% (from same material with altered workup); Repke et al. 1977b: 0.01-0.10% but 0.0 in three samples that were 20 or more wks old. (At least one had showed its presence earlier.) *Psilocybe bohemica* Šebek Gartz 1996(1998): 0.01-0.05%; Gartz & Muller 1989: 0.15-0.21% in mycelium; 0.008-

0.02% in fruit; usually higher in cap than stem; found in all parts of every shroom analyzed; Stijve and Kuyper 1985: 0.01-0.03% Psilocvbe cubensis (Earle) Singer Allen & Merlin 1992a: <0.01%; Repke et al. 1977b: nd-0.1%, (stable after 52 wks stored at -5 C (anhydrous) or after 14 days at RT; undetectable if dried & stored at 22 for 52 wks) Stijve & de Meijer 1993: nd-0.025% (Brazil); Not detected in 2 other strains grown in their lab. Psilocybe cyanescens Wakefield Gartz 1996(1998): 0.01-0.05% (USA); 0.01-0.03% (Europe); Margot & Watling 1981: traces; Repke et al. 1977b: 0.004-0.03% but 0.0% in 3 of 4 samples 66 or more wks old. (At least one had showed presence earlier); Stijve & Kuyper 1985: 0.01-0.03% Psilocybe hoogshagenii var. hoogshagenii (Earle) Singer Stijve & de Meijer 1993: nd-0.014%. Psilocybe liniformans Guzmán & Bas Stijve & Kuyper 1985: 0.005% Psilocybe mexicana Heim Gartz 1994 Psilocybe natalensis Gartz 1994: Gartz et al. 1995: % "similar to Psilocybe cubensis" Psilocybe pelliculosa (Smith) Sing & Smith Repke et al. 1977b: 0.0-0.05%; Beug & Bigwood 1982 and Leung & Paul 1967 and Repke & Leslie 1977 did NOT detect it. Psilocybe samuiensis Guzmán, Allen & Merlin Guzman et al. 1993 Gartz et al. 1994: 0.01-0.05% (collected in Thailand); 0.02-0.05% (cultivated) Psilocybe semilanceata (Fr.) Kummer Brenneisen et al. 1988: recovered 0.33% (Swiss); Christiansen & Rasmussen 1982b: 0.0-0.14% in stems; <0.05-0.28% in caps; <0.05-0.26% total. Up to 0.34% observed; Christiansen & Rasmussen 1983: detected; Gartz 1986a: 0.03-0.38%; Gartz et al. 1994; Høiland 1978; Margot & Watling 1981: over 0.1% but less than 0.3%; Pederson-Bjergaard et al. 1997: detected; Repke & Leslie 1977: found in every specimen tested from US Pacific NW- analytical account: 0.12% yield; Repke et al. 1977b: 0.0-0.17%; & commented on same loss of as was noted above under *cubensis*; Stijve & Kuyper 1985: 0.0-0.36%; Stijve & de Meijer 1993: 0.088% MeOH & 0.14% in 75% aq. MeOH sat. w/ potassium nitrate; White 1979: "provisionally identified" Psilocybe silvatica (Peck) Sing. & Smith Repke et al. 1977b: 0.0-0.02% (noted same potential of loss in storage as mentioned under *cubensis*) Psilocybe stuntzii Guzmán & Ott Repke et al. 1977b: 0.0-0.02% (commented on same potential of loss during storage noted under cubensis) Psilocybe subcubensis Guzmán

Allen & Merlin 1992a: 0.006%.

Baeocystin

Psilocybe cf. *subyungensis* Guzmán Stijve & de Meijer 1993: 0.033%.

Psilocybe uruguayensis Sing. ex Guzmán Stijve & de Meijer 1993: 0.015-0.020%.

Psilocybe weilii Guzmán, Stamets & Tapia Stamets 1996

Psilocybe zapotecorum Heim Stijve & de Meijer 1993: *nd*-0.02%.

Activity:

Psychoactive in animals. Ott 1996 cited Cerletti 1968 [Cerletti *et al.* 1968 evaluated 4-HO-MMT rather than Baeocystine. See comments under its entry]

Dose: Psychoptic in human with a 10 mg oral dose (4 mg threshold) (Ott 1996 cited Gartz, pers. comm.)

See:

Ott 1996: Entry #3, page 431 Usdin & Efron 1979: Entry 367, page 121

Psilocin

4-Hydroxy-N,N-dimethyltryptamine; 3-[2-(Dimethylamino)ethyl]-1H-indol-4-ol 9CI; 3-(2-Dimethylaminoethyl)indol-4-ol; 3-[2-(Dimethylamino)ethyl]indol-4-ol; 3-[2-(Dimethylamino)ethyl] indol-4-ol; 3-(2-Dimethylaminoethyl)-4-hydroxyindole; Tryptamine, 4-Hydroxy-N,N-dimethyl; 3-[2-(Dimethylamino)ethyl]-4-indolol; 4-Indolol, 3-[2-(Dimethylamino)ethyl]; N,N-Dimethyl-4hydroxytryptamine; 4-Hydroxy-N,N-dimethyltryptamine; 4-Hydroxy-ω-N,N-dimethyltryptamine; 4-Hydroxy-N:N-dimethyltryptamine; 4-Hydroxy-dimethyltryptamine; 4-Hydroxydimethyltriptamine (a slightly freudian misspelling encountered in a work for forensic analysts); Psilocyn (a misspelling encountered in law); Psilocine; Psilocin; Psilocina; Psilocinas; Psilotsin (Sandoz); 4-HO-DMT; 4-OH-DMT; 4-OH-DMTPA; CX 59; CX-59 (Sandoz); PSOH; Psc; PI.

WLN: T56 BMJ D2N1&1 FQ Hayward: 6R3RQY5L(CCNM2)=LNHY Usdin & Efron 1979

Chemical Abstracts Registry Number: 520536 [00520536] [520-53-6] NIOSH #: NM 2625000 Sax 1984

Schedule 1 Controlled substance: Ott 1996

C1,H16N,O

Panaeolus subbalteatus Photo by JW Allen

MW 204.27 Merck 9th & Ott 1996 MW 204.30 Clarke's 1986 & Sax 1984 C 70.6%, H 7.9%, N 13.7%, O 7.8% Hofmann *et al.* 1958 C 70.56%, H 7.89%, N 13.71%, O 7.83% Ott 1996 C 70.56%, H 7.90%, N 13.71%, O 7.83% Merck 9th

Free base:

White oil; then crystallizing mp 103-104° (white crystals from ethyl acetate/hexane) Shulgin & Shulgin 1997 mp 173-176° (prisms from ethyl acetate) Troxler *et al.* 1959 mp 173-176° (plates from methanol) Ott 1996 mp 173-176° (white crystals) Clarke's 1986 mp 173-176° (dec.) Perkal 1981

Free base

Very sensitive to oxidation. Hofmann 1971 Forms plates from methanol; very slightly soluble ("*difficultly*") sin water; unstable in solution, especially alkaline solutions. Merck 9th Slightly soluble in water. Soluble in methanol [See note by Kysilka & Wurst 1990 below], ethanol, chloroform. Ott 1996 Soluble in ethanol and in dilute acetic acid (will be as acetate) Clarke's 1986 Soluble in ether. Koike *et al.* 1981 Soluble in dilute acids Soluble in dilute bases Soluble in butyl chloride or chloroform [or others?] Lee 1985

Crystal structureis monoclinic Weber & Petcher 1974

Chloroform-Water Partition coefficient: 5.52

Gessner *et al.* 1968 (this had been manually changed in the UT library copy to 3.30; Migliaccio *et al.* 1981 also gave this latter figure as that reported by Gessner)

Octanol-Water partition coefficient: 0.68 (uncorrected); 1.45 (corrected for ionization) Migliaccio *et al.* 1981

pKa 8.47 (N); 11.33 (OH) Migliaccio *et al.* 1981 (Degrades above pH 7; Casale 1985)

Once provided as a human research material by Sandoz in bulk powder & 1 ml ampuls containing 3 mg/ ml (6 ampuls per box) Scigliano 1968. Sandoz still provides reference standards to researchers in Europe; as does Merck.



4-0H-DMT

Trout's Notes FS-X7: Psilocin

Assays for Psilocin: Colorimetric reagents: See color reactions p. 140

TLC: See Rf table p. 169-176 Horita & Weber 1961 Cinnamaldehyde-HCl on paper

PSOP & PSOH can be separated using Chloroform-Methanol-Ammonia (80:20:10). (PSOP Rf 0.0) Mantle & Waight 1969

Steinigen 1972 detected with 254 nm UV. Alliston *et al.* 1971 used 360 and 254 nm prior to drying and 254 after drying (both prior to application of PDAB) PSOP was said not to fluoresce in their system at 360 and to absorb at 254 nm.

HPLC:

Borner & Brenneisen 1987 Christiansen & Rasmussen 1983 Clarke's (not quantitative) Kysilka & Wurst 1985 & 1989 Perkal 1981& Perkal *et al.* 1980 Thomson 1980 Vanhaelen-Fastré & Vanhaelen 1984 White 1979 Wurst *et al.* 1984 & 1992

GC: Clarke's

UV:

Absorbs (quenches) 254 nm UV Alliston *et al.* 1971 221, 266, 282 & 292 nm (MeOH) Christiansen & Rasmussen 1982a 222, 260, 267, 282, 293 nm (MeOH) Marcano *et al.* 1994 266 nm (aq. acid) Clarke's Second 270, 293 nm (aq. alkali) Clarke's Second

$$\begin{split} \lambda_{\max}: & 222, 260, 267, 283, 293 \text{ nm} (\log \varepsilon \, 4.6, 3.7, 3.8, 3.7, 3.6) \\ & \text{Merck 9th & Perkal 1981} \\ \lambda_{\max}: & [\log \varepsilon]: & 222 [4.63], 268 [3.77], 285 [3.67], 294 [3.64], \\ & (260) [3,72] \text{ Troxler et al. 1959} \\ \lambda_{\max}: & 220, 269, 281, 291 (0.1N \text{ HCl}) \text{ Sunshine 1981 (see also p. 89 & 119 therein)} \\ & \text{UVmax } 222, 260, 267, 283, 293 \text{ nm} \\ & \text{Wurst et al. 1984 (detection limit in HTLC: 40 ng)} \\ \lambda_{\max}: & (\text{MeOH}) & 222, 260 (\text{sh}), 268, 285, 294 \\ & \text{Weeks et al. 1979} \\ & \text{See also (graphic) Lee 1985} \end{split}$$

Fluorescence:

Christiansen & Rasmussen 1982b reported that psilocin fluoresced only weakly.

Marcano *et al.* 1994 reported an "*Obscure bluish-purple*" under UV in TLC.

Perkal *et al.* 1980: Weakly at 312 nm: excitation at 260 nm (in MeOH-water (20:80) containing 0.2% Ammonium phosphate & 0.1% KCl (pH 4.5)

Wurst *et al.* 1984 noted that detection limit in HTLC was a few ng.

MS:

Bellman 1968 (m/e 58, 78, 130, 146, 159, 204) Clarke's 1986 (m/z 58, 204, 59, 42, 30, 146, 77, 44) Shulgin & Shulgin 1997 [m/z $C_3H_8N^+$ 58 (100%); parent ion 204 (15%); indolemethylene⁺ 146 (3%); 159 (2%)] Weeks *et al.* 1979: m/e 204 (21%), 160 (2%), 159 (5%), 146 (8%), 130 (4%), 117 (4%) and 58 (100%) EI-MS (graphic) Casale 1985 MIKES (graphic): Unger & Cooks 1979

GC-MS:

Mehlert *et al.* 1999 Timmins 1984 Wurst *et al.* 1992

GLC-MS: Repke et al. 1977

IR:

Clarke's 1986 (836, 1261, 1236, 1042, 1061, 77) Shulgin & Shulgin 1997 [(in cm⁻¹): 686, 725, 832, 991, 1040 and 1055; the OH stretch is at 3240) Casale 1985 (graphic) Lee 1985 (graphic) Sunshine 1981: #42, p. 257 (graphic)

NMR: Migliaccio et al. 1981

Crystal structure: Weber & Petcher 1974b

Review: Hofmann 1971

Synthesis:

Heim *et al.* 1960 (Sandoz) German Patent 1,087,321 Hofmann *et al.* 1958 & 1959 Shulgin & Shulgin 1997

Formation via the hydrolysis of Psilocybin:

Produced by hydrolysis of psilocybin at 150°. Hofmann *et al.* 1958 Treatment of Psilocybin with 1% HCl formed Psilocin with 1 hr of heating on a boiling water bath. Koike *et al.* 1981 Can also be produced by the action of alkaline phosphatase on PSOP. Horita & Weber 1961b

Isolation:

Hofmann et al. 1958 & 1959

Cold & room temperature methanol have been employed by multiple workers successfully.

Butanol or ethanol combined with acetic acid and water also appears to be an excellent solvent.

For direct consumption, simple extraction with lime juice and water works extremely well (color of solution is tawny not blue.) Mushrooms were soaked for half an hour in methanol (15 ml for 2 gm of material)

The methanol was removed and the residue dissolved in a 0.1N sodium hydroxide solution (25 ml)

This was then extracted with butyl chloride (25 ml)

Evaporation of the butyl chloride apparently left relatively pure Psilocin.

(Alternately the butyl chloride was extracted with a dilute acid and the Psilocin migrated into the acid phase for spectroscopic purposes.)

There was no mention of the % of efficiency for either the initial extraction or any steps of the subsequent isolation process. Lee 1985

Dried powdered mushrooms (2-10 gm) were combined

with dilute acetic acid (100 ml) in a beaker. glacial acetic acid then used to bring the pH to 4.

The beaker was allowed to stand for one hour and then heated for 8-10 minutes in a boiling water bath. (Or until solution temperature reached 70°)

The beaker was then cooled to RT using running water and vacuum filtered through glass wool.

After the filtrate was brought to pH 8 using concentrated ammonium hydroxide, it was quickly extracted twice with 50 ml of ether. (Psilocin degrades at pH >7 hence the need for speed.)

To prevent emulsion formation they recommended a gentle mixing and not shaking.

After separation and combining, the ether is then dried over sodium sulfate, filtered and evaporated without heat under nitrogen.

The greenish residue was crude Psilocin.

White crystals were obtained by recrystallizing from Chloroform-Heptane (1:3).

The product was said to be "*reasonably pure*." Casale 1985 (No indication of efficiency)

Kysilka & Wurst 1990 commented that due to the use of methanol as an extraction solvent by many workers, a lot of the published figures for PSOH content and probably notes of its absence were likely to be low or erroneous. They found that replacement of methanol with 75% aqueous ethanol and use of a longer extraction time (160 minutes) provided them with an increase in yield. See comments page 229

Gartz on the other hand apparently claimed completely opposite results (see p. 229). See also comments concerning Stijve & de Meijer 1993

Hasler *et al.* 1997 found addition of ascorbic acid to PSOH effectively prevented oxidation at RT but was inadequate for use during autoclaving.

Perkal 1981 found that purging with nitrogen dramatically helped prevent degradation of Psilocin. He also found it to be unstable in alkaline solutions but more stable than psilocybin under neutral or acidic conditions. Storage in the dark helped preserve his mushroom's activity even at room tempearture.

Reported Occurrences of Psilocin:

There are only a few instances located in which psilocin was reported without the presence of psilocybin.

See note on Kysilka & Wurst 1990 above and a very different opinion by Gartz in the table on p. 229.

See the rest of the reported occurrences of psilocin

denoted within the psilocybin occurrence list. Conocybe kuehneriana Singer

Ohenoja *et al.* 1987: 0.0% PSOP & 0.004% PSOH *Copelandia cyanescens* (Berk. & Br.) Sing.

Allen & Merlin 1992a made 2 collections in Thailand with trivial amounts of PSOP (<0.025%) in comparison to PSOH (0.40% & 1.05%).

Interestingly, BOTH of their collections contained **Urea** as their major alkaloid (3.3% & 2.0% respectively)

Panaeolus bisporus Bertault & Maleçon Gerhardt Senn-Irlet et al. 1999: 0.41% PSOH & traces only of PSOP. (central Europe)

Psilocybe baeocystis Singer & Smith

Benedict *et al.* 1962a Psilocin was reported without the presence of psilocybin (tryptamine was also present). Other reports where PSOH was the major and PSOP the minor also exist; these are listed under PSOP.

Leung *et al.* 1965 reported only PSOP with with NO PSOH; results that were the exact opposite of Benedict *et al.* 1962a but also analyzed the same material of Benedict (as provided by Benedict) where they found PSOH and could only detect trace amounts of PSOP in that sample.

Psilocybe cyanescens

Wurst *et al.* 1992: 0.0% PSOP & 0.45% PSOH (WA, US: 1984); 0.10% PSOP & 0.47% PSOH (Horni Bradol, Czech. Rep.: 1986)

Activity:

Hallucinogen.

Human dose: 4-8 mg Hofmann et al. 1959

Psychoptic above 6 mg, 2-4 mg threshold (=25 mcg LSD). Ott 1996 cited Abramson & Rolo 1967

Szara 1964 showed that Psilocin and other simple alkylated tryptamines "produce a characteristic regional shift in the serotonin distribution in rabbit brains: an increase in the hypothalamus without a significant change in the amygdala-hippocampal region"

See more comments under Psilocybin pharmacology. While qualitatively the two are nearly identical to each other, there are distinct quantitative differences. Psilocin produces a greater rise in blood pressure, shows over 5X the serotonin antagonism and has over 30 times the pyrogenic activity of Psilocybin.

Also, Stijve 1992 reported more powerful effects when using equivalent amounts of predominately PSOH containing *Copelandia cyanescens* from Thailand compared to the almost entirely PSOP-containing *Psilocybe semilanceata*.

Psilocin

Freedman *et al.* 1970 reported some apparent MAOI activity *in vivo* in rat brains (greater than PSOP)

Dose: 10-20 mg oral Shulgin & Shulgin 1997 300 ig/ kg/ oral Callaway & McKenna 1998

Duration: 3-6 hours Shulgin & Shulgin 1997 & Callaway & McKenna 1998

Reported 1.4X more potent that PSOP (difference in MW) but less stable; the phosphoric acid radical protects psilocybin from oxidation: Hofmann 1971 Wolbach *et al.* 1962 found PSOH was 1.48X PSOP

Pharmacokinetic data (human): Hasler et al. 1997

Tolerance & Drug Interactions: See pp. 231-235

Metabolism & Excretion:

Oddly lacking from the papers we could obtain.

Sticht & Kaferstein 2000 found that most psilocin excreted as psilocin was as the glucoronide conjugate; glucoronidase increasing the recovery dramatically. They reported 0.018 mg/ 1 of free PSOH in serum (vs 0.052 mg/l total) & 0.23 mg/ 1 free vs 1.76 mg/l total in urine. This work was on simple forensic identification. It did not mention other metabolites, include the details of the amount of PSOH ingested or even note the parameters and time of blood or urine collection.

Receptor site specificity:

High affinity for $5HT_{1A}$ (IC₅₀ 190 ± 40 nM), 5-HT₂, 5-HT_{2A} (IC₅₀ 6 ± 0.5 nM) & 5-HT_{2C} (IC₅₀ 410 ± 50 nM) McKenna *et al.* 1990 & Callaway & McKenna 1998

Pyrogenic activity of Psilocin: ED_{+0.5°C} 555 Cerletti *et al.* 1968

LD₅₀: (Usdin & Efron 1979 citing Sandoz) 74 mg/ kg/ iv/ mouse 75 mg/ kg/ iv/ rat 7 mg / kg/ iv/ Rabbit

Toxicology review: Sax 1984 cited 1975 J Med. Assoc. Thailand 58(12) 623



See:

Merck 9th: Entry #7711, page 1027

Ott 1996: Entry #38, page 448 [Merck 11th: 7941; Merck 12th: 8110] Shulgin & Shulgin 1997: Entry #18 page 468-473

Usdin & Efron 1979: Entry #426, page 138

4-Methoxy-DMT

3-[2-(Dimethylamino)ethyl]-1H-indol-4-ol methyl ester;
3-[2-(Dimethylamino)ethyl]-4-indolol methyl ester;
N.N-Dimethyl-4-methoxytryptamine;
4-Methoxy-N,N-dimethyltryptamine;
4-Methoxy-N,N-dimethyl-tryptamine;
4-Methoxy-N,N-dimethyl-4-methoxy;
4-Methoxy-∞-N,N-dimethyl-tryptamin;
4-HO-DMT methyl ester; Psilocin methyl ester;
4-MeO-DMT

WLN: T56 BMJ D2N1&1 FO1

Does not appear to be scheduled.

C₁₃H₁₈ON₂

MW 218.3

C 71.5%, H 8.3%, O 7.3%, N 12.8%

Free base

mp 89-92° (twinned plates from benzene) Troxler *et al.* 1959

Bioxalate

mp 163.5-164.5° Gessner et al. 1968

Chloroform-Water partition coefficient: 2.28

Gessner et al. 1968

Assays:

Colorimetric reagents:

Keller: Olive-brown Van Urk: Blue Troxler *et al.* 1959

Synthesis: Troxler et al. 1959



Psilocybe subcubensis (above) Photo above by JW Allen



Copelandia cyanescens (Maui) Photo by FunGal

Activity:

As is the case for 5-MeO-DMT (when compared to 5-OH-DMT), 4-methoxy-DMT is also apparently an active compound but with decreased visual components (as compared to Psilocin).

McKenna *et al.* 1990 claimed it to have a "*marked* attenuation of the visual component"

Bo Holmstedt [*et al.* 1967] asked the question of why no one had evaluated this compound. Harris Isbell agreed and commented he was "*still waiting animal pharmacology on it*".

Repke *et al.* 1985 cited Gessner *et al.* 1968 as ranking 4-MeO-DMT after DET in potency.

McKenna's conclusion is certainly logical and no doubt correct but thusfar I have been unable to locate any type of indication of its evaluation in clinical investigations or in private human bioassay.

However, Repke *et al.* 1985 did determine that 4-MeO-MIPT lacked the extreme visual phenomena of 4-HO-MIPT in human volunteers.

They commented that Uyeno 1969 & 1971 had reported 4-MeO-DMT to be less active than PSOH with regards to its effects in size discrimination performance in monkeys and on the swimming ability of rodents.



Psilocybe cubensis (Thailand) Photo by JW Allen

Psilocybe azurescens Photo (bottom) by JWAllen Uyeno 1969 found more potent at disruption of learned behavior than DMT and 6-OH-DMT; Uyeno 1971 found it delayed the startled response time much less than PSOP did, but around the same as or slightly less than DMT and more than 6-OH-DMT.

Kline *et al.* 1982 also found 4-MeO-DMT disrupted conditioned behavior. They reported it was less active than 5-MeO-DMT and far more active than 6-MeO-DMT

Gessner *et al.* 1968 SEEMS to found 4-Methoxy-DMT to be *more* effective than either DET or Psilocin at disrupting conditioned avoidance in animals (based on the presented % of CAR failures). (at 10 μ M/kg; i.e. ~2.2 mg/kg) It *seemed* to be on the same order of magnitude as 5-MeO-DMT, in this regard, but there was inadequate data presented for me to be certain of my conclusions. (They also found PSOH to be more effective than DET)

However, the history of failure for non-human animal models when used for predicting the activity of hallucinogens is appalling. Even if activity turned out to be more similar to 5-MeO-DMT than to PSOH, this might be found quite a valuable tool (like 5-MeO-DMT has proven to be)

This substance should be tested in HUMANS where the data is meaningful. A 10 mg dose of it (smoked or insufflated) does not appear to pose any significant risk to a normal human, and would quickly settle the matter, which is only one reason I find its apparent lack of evaluation so puzzling.

Most interestingly, Gessner *et al.* 1968 noted that Chloroform-Water partition coefficients **can't** be directly correlated to activity.



Psilocybe azurescens Photo (top) by Dr. P. C. Hickey



4-AcO-DMT

1-Me-DMT

4-Acetoxy-DMT

4-Acetoxy-N,N-dimethyltryptamine;
3-[2-(Dimethylamino)ethyl]-1H-indol-4-ol acetate ester;
3-[2-(Dimethylamino)ethyl]-4-indolol acetate ester;
N,N-Dimethyl-4-acetoxytryptamine;
Tryptamine, N,N-dimethyl-4-acetoxy;
4-AcO-DMT;
4-HO-DMT acetate ester;
4-Acetoxy-psilocin (error);
Psilocin acetate ester;
PSOA

WLN: T56 BMJ D2N1&1 FOV1 Hayward: 6R3R(OCVM) Y5L(CCNM2)=LNHY Extrapolated from others here

 $C_{14}H_{18}O_2N_2$

MW 246.3 Troxler et al. 1959

C 68.3%, H 7.4%, [N 11.3%], O 13.0%

Free base: mp 92-95° (prisms: ethyl acetate) Troxler *et al.* 1959

Assays:

Colorimetric reagents: Keller: green, then violet Van Urk: negative Troxler *et al.* 1959

Synthesis: Troxler et al. 1959

Activity:

Hallucinogenic. Same effects & dosage as Psilocybin & Psilocin (more stable to oxidation than the latter)

1-Methyl-psilocin

1-Methyl-4-hydroxy-N,N-dimethyltryptamine; 3-[2-(Dimethylaminoethyl)-1-methyl-1H-indol-4-ol; 1-Methyl-psilocin

Galerina autumnalis (deadly)



Galerina autumnalis compared to Psilocybe cyanescens



Psilocybe cyanofibrillosa

WLN: T56 BNJ B D2KH FQ Hayward: 6R3RQY5L(CCNM2)=LNMY (Extrapolated from other examples here)

C₁₃H₁₈ON₂

MW 218.3 Troxler et al. 1959

C 71.5%, H 8.3%, N 12.8% Troxler et al. 1959

Free base:

mp 123-127° (Irregular prisms from Methanol) Troxler *et al.* 1959

Assays:

Colorimetric reagents:

Keller: Grey Van Urk: Green Troxler *et al.* 1959 *Mytilus edulis* gill-plate oxidase: Blue Blaschko & Levine 1960

Synthesis: Troxler et al. 1959

Activity:

Unknown so far as I can tell but should be analogous to 1-Methylpsilocybin.

This compound & 1-Methyl-psilocybin both need more study. See additional comments under 1-Methyl-psilocybin.

Cerletti *et al.* 1968 reported that, in comparison to psilocin, there was an increase in spinal reflexes (based on knee jerk response), a more than tripling of serotonin antagonism and only half the pressor activity.



Unidentified *Psilocybe* sp. in Bay area Photo above by James Edmond

Psilocybin

Psilocybin

3-[2-(Dimethylaminoethyl)-1H-indol-4-ol dihydrogen phosphate ester 9CI; 3-[2-(Dimethylaminoethyl)indol]-4-yl dihydrogen phosphate; 3-(2-Dimethylaminoethyl)indol-4-yl dihydrogen phosphate; 3-2'-Dimethylaminoethylindol-4-phosphate; O-Phosphoryl-4-hydroxy-N,N-dimethyltryptamine; O-Phosphoryl-4-hydroxy-ω-N,N-dimethyl-tryptamine; 4-Phosphoryloxy-N,N-dimethyl-tryptamine; 4-Phosphoryloxy-N,N-dimethyltryptamine; 4-Phosphoryloxydimethyltryptamine; Tryptamine, 4-Hydroxy-N,N-dimethyl phosphate ester; 4-Indolol, 3-[2-(dimethylamino)ethyl], phosphate ester; 3-[2-(Dimethylamino)ethyl]-4-indolol, phosphate ester; N,N-Dimethyl-4-phosphoryloxytryptamine; 4-Phosphoryl-N:N-dimethyltryptamine; 4-Phosphoryloxy-ω-N,N-dimethyl-tryptamin; Psilocin phosphate ester; 4-HO-DMT phosphate ester; dihydrogenphosphate salt of psilocin; Psilocybine; Psilocybin; Psilotsibin; Psilocibina; Psilocibinas; Pcilocibin [sic]; Indocybin; Indocybine; 4-OP-DMT; CY 39 (Sandoz); PSOP; Ps; Psb; PY; P.

WLN: T56 BMJ D2N1&1 FOPQQO Hayward: 6R3R(OP(O-)VQ)Y5L(CCN+HM2)=LNHY Usdin & Efron 1979 Chemical Abstracts Registry Number: 520525 [000520525] [520-52-5] NIOSH #: NM 3150000 Sax 1984

Schedule 1 controlled substance: Ott 1996

 $C_{12}H_{17}N_2O_4P$

MW 284.25 Ott 1996 MW 284.27 Merck 9th MW 284.28 Sax 1984 MW 284.3 Clarke's 1986

C 50.7%, H 6.0%, N 9.9%, P 10.9% Hofmann *et al.* 1958 C 50.70%, H 6.03%, N 9.86%, O 22.51%, P 10.90% Merck 9th

C 50.71%, H 6.03%, N 9.86%, O 22.51%, P 10.90% Ott 1996

Free base:

Stable compound. Colorless crystals. Hofmann 1971 pH 5.2 (in 50% EtOH) Merck 9^{th}

More stable than psilocin. Shulgin & Shulgin 1997

mp 175-180° Repke & Leslie 1977

mp 185-195° (from boiling methanol) Ott 1996

mp 185-195° (from methanol) Picker & Rickards 1970 (Also in Perkal 1981)

mp 185-195° (dec.) (white crystals) Clarke's 1986 mp 190° Mantle & Waight 1969 mp 218-228 (colorless needles from *n*-butanol saturated with water) Koike et al. 1981 mp 219-222° (6-angled plates from methanol) Troxler et al. 1959 mp 200-210° (dec.) Bellman 1968 mp 204-210° (rod-shaped crystals from methanol; after 10 days in a freezer) [co-mp with reference material was 205-210°] Leung et al. 1965 mp 220-228° (crystals from boiling water) Ott 1996 Free base: White crystals "fairly soluble in water". Hofmann 1959 Readily water soluble. Hofmann 1971 Forms crystals from boiling methanol or boiling water; Soluble in boiling water (1:20), Slightly soluble in boiling methanol (1:120); Slightly soluble ("*difficultly*") in ethanol; Practically insoluble in chloroform or benzene. Merck 9th Practically insoluble in ethanol, chloroform or benzene. Hofmann et al. 1958 "practically insoluble in the usual organic solvents" Hofmann 1959 Insoluble in ether. Picker & Rickards 1970 Soluble 1:20 in boiling water; 1:120 boiling ethanol. Perkal 1981 Many workers have successfully used cold or room temperature methanol for isolations. Soluble in dilute acetic acid (as acetate) Clarke's 1986 Soluble in dilute acids Soluble in dilute bases Insoluble in butyl chloride Lee 1985

Base is amphoteric. Hofmann 1959 (Soluble in acids & bases; can't extract from either with organic solvent)

Crystal structure is monoclinic Weber & Petcher 1974

While no precise value was furnished, it appears the pK is around 12 Pederson-Bjergaard *et al.* 1997 pH 5.2 in aq. 50% ethanol. Perkal 1981

Hydrochloride:

Insoluble in acetone; insoluble petroleum ether; soluble in hot ethanol; poorly soluble in cold ethanol. Isolated via crystallization with cold from ethanol. Anon 2003

Once provided as a research material by Sandoz as bulk powder, 10 mg tablets & 1 ml ampuls containing 3 mg/ ml (6 ampuls per box) Scigliano 1968. Sandoz apparently still provides it for reference standard purposes to researchers in Europe; as does Merck.

Unlike psilocin, which is readily oxidized, psilocybin is stable enough to be detectable for many years in herbariums (Dried psilocybin shrooms appear to remain usable for around 9 years or sometimes longer if kept in proper storage; shrooms which contain psilocin often appear to rapidly degrade in storage)

Trout's Notes FS-X7: Psilocybin

4-Phosphoryloxy-DMT

Assays for Psilocybin:

While we include a listing of colorimetric reagents and chromophores, it also should be noted at this point that psilocybin is both visible as a blue spot in most TLC systems and also fluoresces under UV.

Colorimetric reagents: See color reactions p. 145

TLC & PC: See Rf table p. 169-176 for more details

Butanol-Acetic acid-Water (12:3:5) on silica gel was the preferred system of Beug & Bigwood 1981)

- Repke *et al.* 1977b reported *n*-Propanol-6% aqueous Ammonia (5:2) on silica gel GF separated PSOP well from other components.
- Phillips & Gardiner 1969 used 254 nm to visualize (worked best on Eastman chromatogram 6060 and worst on alkalinized silica gel) but found Iodine in methanol worked well

They thought the best results for speed and effectiveness was 254 nm UV on Eastman chromatogram 6060 sheets using Methanol-Ammonia (sp. gr. 0.88) (100:1.5) as the moving phase.

Marcano et al. 1994 viewed their TLC plates under UV and reported observing an "obscure bluish-purple" fluorescence

Chloroform-Methanol (9:1) has also been used for tlc (on alumina & on silica gel) and for LC (on alumina)

While not encountering it as evaluated, we have to wonder about using BAW or IPA-ammonia or methanol-ammonia or just straight methanol for a crude preparative PC (followed by elution of the appropriate band) in those instances when recovery of crude alkaloid(s) is acceptable. Use of 2 dimensional PC with chloroform or methylene chloride for the second solvent would readily separate PSOH from PSOP if desired.

Preparative TLC:

- Gartz 1986a used *n*-Propanol-Water-Acetic acid (10:3:3) on Kieselgel; after an initial solvent cleanup
- Christiansen & Rasmussen 1982a used Methanol-Water-1M Ammonium salts (240:50:10) for preparative chromatography. (Buffered to pH 9.6 with 2N ammonia)

Column chromatography:

Mantle & Waight 1969 used an ion exchange resin to isolate. Cellulose columns were common in the early literature

- Leung *et al.* 1965 used a column of cellulose powder with butanol saturated with water to elute. (They used almost a liter and a half of butanol to recover 4 mg of rod shaped crystals; after using another half liter to first wash the column)
- Leung & Paul 1967 isolated PSOP using a silica gel column with *n*-Propanol-5% Ammonium hydroxide (5:1) as elutant. See also for a nice account for a crude purification of mixed alkaloid using cellulose.

Christiansen *et al.* 1981b: CC on silica gel Weeks *et al.* 1979: CC on silica gel

> *Psilocybe semilanceata* (Netherlands) Photo copyrighted by Perfect Fungi Europe 2005

HPLC:

Beug & Bigwood 1981 Borner & Brenneisen 1987 Christiansen & Rasmussen 1983 Christiansen *et al.* 1981 & 1984 Koike *et al.* 1981 Kysilka & Wurst 1988 & 1989 Kysilka *et al.* 1985 Perkal 1981& Perkal *et al.* 1980 Sottolano & Lurie 1983 Thomson 1980 Vanhaelen-Fastré & Vanhaelen 1984 White 1979 Wurst *et al.* 1984 & 1992



Capillary zone electrophoresis: Pederson-Bjergaard *et al.* 1997

UV: $(\lambda_{max} \text{ in MeOH})$: 219, 266 & 288 nm , 280sh Christiansen & Rasmussen 1982a 221, 227, 268, 279, 290 nm Marcano *et al.* 1994 268 nm (aqueous acid) 269, 282, 292 nm (aqueous alkali) Clarke's Second

- λ_{max} (methanol): 220, 267, 290 nm (log ϵ 4.6, 3.8, 3.6) Merck 9th & Perkal 1981
- λ_{max} (methanol): 221, 268, 280, 290 nm (log ε 4.27, 3.84, 3,74, 3.64) Koike *et al.* 1981
- λ_{max} : 220, 269, 278, 290 (0.1N HCl) Sunshine 1981 (see also pp. 89 & 119)

λmax 269, 278 (sh), 290 nm Picker & Rickards 1970

λmax 220, 267, 290 nm Wurst *et al.* 1984 (detection limit in HTLC was 20 ng)

λmax (MeOH) 222, 260 (sh), 268, 285, 294: Weeks *et al.* 1979 Christiansen & Rasmussen 1982b noted that UV of Baeocystin & PSOP are identical

See also: Lee 1985 & Perkal 1981





Psilocybe azurescens

(Portland, OR)

Photo by Fun Gal

Fluorescence:		
Psilocybin	Fluorescence	
	(nm)	
Solvent		
Ref	Excite	Emit
Sulfuric acid 0.1N	269	339
Hydrochloric acid 0.001N	268	338
Phosphate buffer 0.1M	268	323
(pH 7)		
Ammonium hydroxide 1N	268	325
Sodium hydroxide 1N	272	336
Absolute methanol	272	309
Ammonium nitrate (1N)		
10% in methanol	267	335
1 (C1 :	10001 9 01	

- **1-6** Christiansen & Rasmussen 1982b & Christiansen *et al.* 1981 (psilocybin fluoresced strongly)
- 7 Gillespie 1969 (*"Strongly fluorescent"* in a spectrofluorometric assay. Absolute methanol was felt to be the optimum solvent for this purpose) A small amount of photodecomposition was observed in both phosphate buffer (pH 7) and also in the 1N NaOH. Also noted less fluorescent in the latter.
- Violet fluorescence under UV. Picker & Rickards 1970
- Marcano *et al.* 1994 reported an "*obscure bluish purple*" fluorescence under UV.
- Shows native fluorescence (MeOH) λ_{ex} 277; λ_{em} 309 (sensitivity 1 ppm) Sunshine 1981
- Fluoresced strongly at 335 nm with excitation at 267 (in methanol-water (20:80) containing 0.2% ammonium phosphate & 0.1% KCl (pH 4.5) Perkal *et al.* 1980
- Activation: 295; Emission: 360 nm (pH 7)
- Activation: 295; Emission: 370 nm (pH 2) Gessner *et al.* 1960

Wurst et al. 1984 noted detection limit in HTLC was ng.

PENE should also give fluorescence in TLC but I have not found this evaluated for this compound.

Majak *et al.* 1978 ran on silica gel and viewed fluorescence under short wave UV.

PENE was the solvent system:

iso-Propanol-Ethyl acetate-concentrated Ammonium hydroxide-2-Ethoxy-ethanol (60:15:3:5) [Drying the plates in a cold room is said to prolong the colors.]

IR:

- Clarke's 1986 (Principal peaks at wavenumbers 1105, 1045, 1062, 1183, 1160, 932 (KBr disk)
- Hofmann et al. 1958 has graphic depiction
- Shulgin & Shulgin 1997 ["(in cm⁻¹): 752, 789, 806, 858, 925 and the P=O stretch is at 110; the acidic OH stretches are broad peaks at 2400, 2700 and 3200")
 See also (graphic):
 Sunshine 1981: #18, p. 251
- Lee 1985

MS:

- Bellman 1968 (m/e 58, 78, 130, 146, 159, 204) (same as PSOH)
- Picker & Rickards 1970 [m/e 204, 160, 159, 146, 130, 117, 115, 58]

- Repke & Leslie 1977 [m/e 204 (19%), 160 (4), 159 (3), 146 (6), 130 (3), 117 (2) 58 (100)]
- Weeks *et al.* 1979: m/e 204 (21%), 160 (3%), 159 (4%), 146 (5%), 130 (3%), 117 (4%) and 58 (100%)
- Christiansen & Rasmussen 1982b: m/z 204, 160, 159, 146, 130, 117 & 58 (base peak)
- Clarke's 1986 (m/z 58, 42, 30, 51, 204, 146, 77, 44)

 $\begin{array}{l} \mbox{Shulgin & Shulgin 1997 [m/z C_{3}H_{8}N^{+} 58 \ (100\%); \ parent ion 204 \ (15\%); \ indolemethylene^{+} 146 \ (3\%); \ 159 \ (2\%)] \\ \ (same \ as \ PSOH) \\ \ See \ also \ Koike \ et \ al. \ 1981 \\ \end{array}$

and Unger & Cooks 1979: MIKES

GLC-MS: Repke et al. 1977

GC-MS:

Mehlert *et al.* 1999 Timmins 1984

Structure:

Hofmann *et al.* 1958 Weber & Petcher 1974a

Synthesis:

- Heim *et al.* 1960 (to Sandoz) German Patent 1,087,321
- Hofmann 1959 depicts the route schematically in Bradley *et al.* (eds.)
- Hofmann 1971 also depicts route schematically
- Hofmann & Troxler 1963 (to Sandoz) US patent 3,075,992

Hofmann *et al.* 1958 & 1959 Shulgin & Shulgin 1997

Can be produced by phosphorylation of psilocin with dibenzylphosphorylchloride followed by reductive debenzylation (Hofmann *et al.* 1958 & Shulgin & Shulgin 1997) but Shulgin notes the yield is appalling. Acetylation would seem more sane (See Troxler *et al.* 1959)



1932 postcard provided by James Arthur

Psilocybin

Isolation:

Hatfield *et al.* 1978 Hofmann *et al.* 1958 & 1959

Koike *et al.* 1981 (Using preparative TLC followed by a column of cellulose & also using an anion exchange resin.)
Leung *et al.* 1965 (via LC on a column of cellulose.)
Picker & Rickards 1970 (LC on column of cellulose.)

Crude isolation from initial extract using cellulose column with Butanol saturated with water as elutant. Separated from baeocystin using silica gel column with *n*-propanol-5% ammonium hydroxide (5:1) as elutant. Leung & Paul 1967

"Optimum extraction"

- Homogenizing 2 minutes in 30 ml of methanol per gram of powdered pshrooms recovered all psilocybin. Perkal 1981
- Repke & Leslie 1977 used *n*-propanol-5% ammonium hydroxide (5:2) on silica gel for preparative tlc. Elution was with methanol containing 5% aqueous ammonia.

Many workers have used methanol successfully. It appears that it works fine either cold or at room temperature.

One trend noted in the literature though, after the extraction is complete, the faster it can be processed to pure or concentrated material (with the minimal heat exposure possible) the higher the potential yield appears to be. This is likely due to degradation despite psilocybin being a fairly stable compound.

Butanol or ethanol combined with acetic acid and water also appears to be an excellent extraction solvent.

Wurst *et al.* 1984 made the statement that "Boiling of fruit bodies in water results in a quantitative extraction of psilocybin. The subsequent extraction ... with methanol did not yield even traces." but provided no further detail.

For direct consumption, the simple extraction of chopped or shredded mushrooms with water and lime juice (or citric acid) works extremely well (color of the resulting solution is tawny not blue) Heat & cooking time should be minimized (2X of 15 minutes or less of simmering each) as prolonged cooking is neither needed nor desirable. Works well in a microwave if allowed to stand following the each heating.

Hofmann *et al.* 1963 used a methanol extract, concentrated it to a low volume and cleaned it with petroleum ether. Heim & Hofmann 1958a had taken a residue from a methanol extract and cleaned it up with a successive series of solvents: petroleum ether, chloroform & chloroform-ethanol. The remaining residue was taken into the least possible amount of water and more contaminants precipitated with the addition of absolute alcohol. The residue remaining after the evaporation of the filtrate, was then passed through a column of cellulose using butanol saturated with water to elute and selection made for the appropriate fractions. For smaller quantities they used butanol saturated with water on ascending paper chromatography to separate.

Gartz 1986a used dried powdered shrooms (2 gm) and extracted for 4 hours with petroleum ether (30 ml) and etherchloroform (1:1) (30 ml) prior to soaking the recovered mushroom mass in methanol (100 ml) for 24 hours and isolated using preparative TLC with *n*-propanol-water-acetic acid (10:3:3) on 30 cm of kieselgel.

- Neal *et al.* 1968 found that using 10 ml of methanol for each dried and powdered gram of shroom shaken (mechanical) for one hour and this performed twice produced a clear solution but exhausted the marc of any detectable tryptamines.
- Pederson-Bjergaard *et al.* 1997 used dried pulverized mushrooms (100 mg) in 3 ml of methanol in an ultrasonic bath for 15 minutes; repeated with 2 ml of methanol, to obtain a 98% extraction efficiency.
- Christiansen *et al.* 1981 found methanol (hot) worked acceptably but only 91% of the psilocybin was extracted. They raised this to 98% by using 10% 1N ammonium nitrate in methanol. (They used 2X 30 minutes extraction times with a rotary mixer using a high potency mushroom.)
- (They also determined heat-drying shrooms removed 92% water by weight.)
- Christiansen & Rasmussen 1982a used hot methanol to extract & Methanol-Water-1M Ammonium salts (240:50:10) for preparative chromatography. (Buffered to pH 9.6 with 2 N Ammonia.)
- "Psilocybin extraction": Taken from Anonymous 1992 as posted on the Internet. Printed in *The Entheogen Review* 3(2): 4.
- Claimed to be derived from page 97 in Ola'h, Gyorgy 1970 *Le Genre Paneolus: Essai taxonomique et physiologique.* (This work is currently unavailable to me so unable to verify.)
- 1. Dry the mushrooms (caps or mycelium).
- 2. Crush or grind to a powder.
- (Note: For each of the following solvents use twice the dry weight of the powder or enough to cover the powder well.)
- 3. Shake then allow to stand 30 minutes in Chloroform.
- 4. Filter and discard the Chloroform.
- 5. Shake then allow the residue to stand in Acetone
- 6. Filter and discard the Acetone.
- 7. Shake then allow the residue to stand in Methanol.
- 8. Filter.
- 9. Repeat 7 and 8 twice more.
- 10. Discard residue.
- 11. Combine Methanol extracts; evaporate to dryness.
- The final product is a crude extract said to be a whitish-grey crystalline substance.
- [Psilocin, if present, will be lost in step 4.]

See Note G: page 227.

Hofmann 1971 comments; "*The synthetic production of psilocybin is much more rational than obtaining it from the mush-rooms*." [He was working with shrooms that contained 0.2-0.4% psilocybin.]

Beug & Bigwood 1981 commented that extraction at higher temperatures or in a Soxhlet caused partial or complete loss of psilocin but generally less than a 20% loss of psilocybin. Methanol extracts stored at -5° showed little changes after more than a year but within a few month storage at room temperature lead to a total loss of psilocin with some loss of psilocybin.

- These two observations should be kept in mind for the following section.
- Interestingly, it appears that dried mushrooms with PSOH should be kept stored under freezing conditions, to prevent degradation, while those with ONLY PSOP or trivial amounts of PSOH can keep their potency (when dried) for many years even at room temperature.
- A useful piece of trivia to keep in mind for dosage estimations: 0.01% is 10 mg per 100 gm; 0.1% is 100 mg per 100 gm (10 mg per 10 gm); 1.0% is 1000 mg per 100 gm (10 mg per gram).







Comments on the Occurrence of Psilocybin listings: This list was drawn largely from Ott 1996 & Voogelbreinder 2002; with added details & updates.

Occurrences for psilocybin and psilocin are merged as they are not simply variable but so far the vast majority of all reported occurrences of psilocin have been co-occurring with at least traces of psilocybin.

Not all occurrences of psilocybin have been accompanied by psilocin so it seemed most useful to preserve this presentation in the same format used by Ott; with some modifications.

ALL percentages given are as % of the dry weight unless specifically stated to the contrary.

If only one figure is given this was Psilocybin with no mention of Psilocin.

The following reflects only what has been reported; actual values can vary wildly, even within a single collection, even in mushrooms growing next to each other.

The reasons for this are not clearly understood, despite differences in P or N availability being suspected, but such variations are quite common in the plant world, just usually not to as extreme of a degree as is common for these wondrous creatures.

Non-experienced authors commonly present this as an indication of the extreme danger of these mushrooms but most of their users have learned to cope with it just fine.

The phrase "*psilocybian latent*" indicates the author making the claim found PSOP/PSOH only in some specimens and a total absence in others. In some of these instances a high degree of variability is obvious but some have been proposed as potentially reflecting misidentification of specimens within larger collections. According to communication with JW Allen, misidentification of fungus species even by highly noted experts is far from uncommon.

All references lacking detail were unavailable or not adequately translated by press-time.

Synonyms are listed only in those cases where published analysis was presented under the earlier name. Only minimal effort was made to decipher the taxonomic conflicts since there appear to be so many varying opinions among the experts; even concerning the ability of other experts to make correct identifications of what they analyzed (or sometimes their ability to competently perform analysis!)

The following uses the names listed or synonyms proposed when we found them. Consult the most recent works you can find for current synonyms.

Perhaps the most important note was made by Jonathan Ott, in a letter received in 1995; "*To be sure, the number increases all the time!*"

Psilocybe cyanescens (Oakland, CA, USA)

Trout's Notes FS-X7: Psilocybin

4-Phosphoryloxy-DMT

Reported occurrences of Psilocybin Agrocybe farinacea Hongo Koike et al. 1981: 0.2-0.4%. Agrocybe sp. (unident.) Ohenoja et al. 1987: 0.003% PSOP & no PSOH. Conocybe cyanopus (Atkinson) Kühne Benedict et al. 1962b; Benedict et al. 1967: PSOP identified but no PSOH; by TLC; Beug & Bigwood 1982: PSOP at 0.93% but not PSOH; Christiansen et al. 1984: 0.33-0.55% PSOP & 0.004-0.007% PSOH: Gartz 1985f: observed; Ohenoja et al. 1987: 0.45% PSOP & 0.07 PSOH in a fresh specimen. Conocybe kuehneriana Singer Ohenoja et al. 1987: NO PSOP & 0.004% PSOH . Conocybe smithii Watling Benedict et al. 1967: PSOP identified; no PSOH (TLC) Copelandia anomala Murrill? Merlin & Allen 1993 cited Pollock 1976 but did not include it in their references. Copelandia bispora (Malencon & Bertault) Singer & Weeks Merlin & Allen 1993 cited Malecon & Bertault 1975. Copelandia cambodginiensis (Malencon & Bertault) Sing. & Weeks Merlin & Allen 1983: 0.55 & 0.13% PSOP & 0.3-0.5 & 0.13% PSOH (O'ahu); Ola'h 1968; Ott & Guzmán 1976; Ola'h 1969 Said to have determined this to be "psilocybian" but included no details. Copelandia chlorocystis Sing. & Weeks Weeks et al. 1979: 0.46% PSOP & 0.29% PSOH. Copelandia cyanescens (Berk. & Br.) Sing. Allen & Merlin 1992a: <0.025% & <0.025% PSOP & 0.40 & 1.05% PSOH: Fiussello & Ceruti Scurti 1972; Heim et al. 1966: Heim et al. 1967; KvW 2002: 0.5-2.95% (compost grown); Ola'h 1968; Ola'h 1970; Merlin & Allen 1983: 0.6% PSOP & 0.2% PSOH (pooled sample). PSOH & PSOH observed in chromatography in all 7 specimens examined (O'ahu); Ola'h 1969 Said to have determined this to be "psilocybian" but included no details; Stijve & de Meijer 1993: 0.05 PSOP & 0.10% PSOH (MeOH) vs. 0.09% PSOP & 0.33% PSOH (Optimized). Copelandia tropicalis (Ola'h) Sing. & Weeks Ola'h 1968; Ola'h 1970; Ola'h 1969 Said to have determined this to be "psilocybian" but included no details. Galerina steglichii Besl Besl 1993; Gartz 1994. Gerronema fibula (Bull. ex Fr.) Singer Gartz 1986b: PSOP & no PSOH observed; Stijve & Kuyper 1988 could not detect it.

Gerronema solipes See as Gerronema swartzii Gerronema swartzii (Fr. ex Fr.) Kriesel Gartz 1986b: PSOP & no PSOH observed; Stijve & Kuyper 1988 could not detect it. Gymnopilus aeruginosus (Fr. ex Fr.) Kriesel Hatfield et al. 1977: PSOP detected (chromatography); Hatfield et al. 1978 detected PSOP chromatographically in 3 systems; It was not observed by Koike et al. 1981 Gymnopilus lateritius (Pat.) Murrill ? Allen et al. 1992 is cited for this? I could only find Gymnopilus leteoviridis [sic] Thiers. in this paper. Gymnopilus liquiritiae (Fr.) Karst. Koike et al. 1981: 0.012-0.029%: Japan; Hatfield et al. 1978: could not detect it: US. Gymnopilus luteus (Peck) Hesler Hatfield et al. 1977: PSOP detected (chromatography); Hatfield et al. 1978: PSOP detected chromatographically in 3 systems: not present in all specimens. Gymnopilus purpuratus (Cooke & Mass.) Singer Gartz 1989a; Gartz 1991; Gartz & Müller 1990; Kreisel & Lindequist 1988: Presence was reported based on chromatography; Gurevich 1995 did not observe (Russian). Gymnopilus sapineus (Fr.) Maire Gurevich 1995 did not observe (Russian); Guzman et al. 1998 (2000) lists it with no detail. Gymnopilus spectabilis (Fr.) A.H. Sm. (=Pholiota) Successful bioassays: Michigan, USA. Conflicting reports. Negative analysis may reflect seasonal or regional differences: more work is needed. Hatfield et al. 1977: PSOP detected chromatographically: Michigan); Hatfield et al. 1978: PSOP detected chromatographically in 3 systems: not present in all specimens. Present in 50% of eastern US collections and in none of the western US collection they looked at; Hatfield & Brady 1969; Stijve & Kuyper 1988; It was not observed by Christiansen et al. 1984 or Koike et al. 1981 or Kusano et al. 1986:. Has a more complex chemistry than other psilocybian mushrooms and subjective effects suggest other active components are also present WHEN they are active. See Voogelbreinder for a full discussion. In the interesting account of Walters 1965, a woman taking "a few nibbles" of what was believed to be this species reported: "...most glorious visions of color and sounds of music, but with no feeling of discomfort whatsoever." She reportedly told the person attending to her that "... if this was the way one died of mushroom poisoning, she was all for it." Gurevich 1995 did not detect it (Russian). Gymnopilus validipes (Peck) Hesler Hatfield et al. 1977: PSOP isolated at 0.12%; did not appear to detect PSOH; accidental positive bioassay reported;

(Hatfield et al. 1978: same data as 1977)

Gymnopilus viridans Murrill

Hatfield et al. 1978: PSOP detected (chromatography).

Hygrocybe psittacina

Gartz 1986b: PSOP & PSOH observed;

Stijve & Kuyper 1988 could not detect it.

Inocybe aeruginascens Babos

Drewitz 1983;

Gartz 1986c noted PSOP content was affected by the composition of the substrate and could be affected (maximized) by its available nutrients;

Gartz 1987b: 0.16-0.84% PSOP & NO PSOH;

Gartz 1989d: 0.26-0.52%;

Gartz 1995: listed 0.16-0.50%;

Gartz & Drewitz 1985 & 1986;

Gurevich 1995 reported in Russian material;

Semerdzieva *et al.* 1986: 0.11-0.38% PSOP & 0.0 PSOH in specimens (0 to 18 years old); 0.22% PSOP in the 18 yr old one;

Stijve & Kuyper 1985 & Stijve *et al.* 1985: 0.085 & 0.28% PSOP & 0.0 & 0.008% PSOH;

Wurst *et al.* 1992: 0.03-0.36% PSOP & 0.0-0.02% PSOH. *Inocybe calamistrata* (Fr.) Gill.

Gartz 1986b: PSOP & PSOH observed;

Stijve et al. 1985 could not detect it.

Inocybe coelestium Kuyp.

Stijve & Kuyper 1985 & Stijve *et al.* 1985: 0.035% PSOP & no PSOH.

Inocybe cordyalina var. cordyalina Quélet

Ballero & Contu 1998: 0.021% PSOP & 0.01% PSOH (Sardinia);

Gartz 1986b: PSOP & no PSOH observed;

Stijve & Kuyper 1985 & Stijve *et al.* 1985: 0.011% & 0.032% PSOP & no PSOH;

Stijve & de Meijer 1993: 0.023% (MeOH) & 0.03% (Optimized MeOH) PSOP but no PSOH;

Gurevich 1995: 0.02 & 0.04 mg / gm (Russian).

Inocybe cordyalina var. *erinaceomorpha* (Stangl & Vesel.) Kuyp.

Stijve & Kuyper 1985 & Stijve *et al.* 1985: 0.1% PSOP & no PSOH.

Inocybe haemacta (Berk. & Cooke) Sacc.

Gartz 1986b: PSOP & PSOH observed;

Stijve & Kuyper 1985 & Stijve *et al.* 1985: 0.17% PSOP & 0.0% and 0.02% PSOH (respectively);

Stijve & de Meijer 1993: 0.023% (MeOH) & 0.03% (Optimized MeOH) PSOP but no PSOH;

Stribrny et al. 2003: PSOP & PSOH observed.

Inocybe tricolor Kühner

Gartz 1993.

Panaeolina castaneifolius (Murrill) Bon

Ballero & Contu 1998: 0.07% PSOP & 0.04% PSOH (Sardinia) [as *P. papilionaceous*);

Ola'h 1968 & Ola'h 1970;

Ola'h 1969 Said to have determined this to be "*psilocybian latent*" but included no details;

Gurevich 1995 did not observe (Russian).

Panaeolina foenisecii (Fr.) Maire

Ballero & Contu 1998: 0.06% PSOP & 0.04% PSOH (Sardinia);

Fiussello & Ceruti Scurti 1972 reported PSOP;

Ohenoja *et al.* 1987: found 0.03% PSOP & no PSOH in 2 out of 19 specimens using HPLC (Finland);

Ola'h 1968; Ola'h 1970;

Robbers *et al.* 1969: 0.17% PSOP in specimen from Lafayette, Indiana. PSOP also identified in another from Canada but not in material from Washington.

They based their ID on 1-D & 2D co-TLC using 2 solvent systems, color with Ehrlich's (pink turning violet then blue), & MS m/e 284)

Another indole was present but not identified;

Ola'h 1969: determined this to be "*psilocybian latent*" but included no details. See Endnote B page 219;

Christiansen *et al.* 1984: NO PSOP, hplc (Norway);

Gurevich 1995 could not detect (Russian):

Ott & Guzmán 1976: NO PSOP in TLC (Mexico);

Allen & Merlin 1991 claim to have "*proven*" it does not contain psilocybin (?!) [how 'proven' was not mentioned]; Allen & also Stijve suspect that the earlier reports were based on false positives involving mixed populations with misidentified specimens of *P. subbalteatus*;

See Allen's review at http://www.erowid.org/plants/mushrooms/mushrooms article4.shtml

Gartz 1985g: Analyzed 100 specimens from 20 locations and could find NO PSOP or PSOH (German); Stijve & de Meijer 1993 could not detect (Swiss); Stijve *et al.* 1984 had 5 volunteers bioassay up to 40 grams of dried material for no effect. Their analysis found serotonin & tryptophan but detected neither PSOP nor PSOH in 15 separate collections made from Switzerland, Austria, USA (Olympia, Washington), Australia, France, Spain, England & the Netherlands.

They also noted that many lots of their collections, made by people "more or less" familiar with the species, often contained individuals of similar species and the need to double check the identification of each and every one, BEFORE drying, was requisite.

However, no later worker has ever looked at ANY material from Indiana or Finland or Sardinia. nor at material vouchered by any of those workers reporting positive results.

The suspicions above are quite reasonable, but more work is still needed before any solid conclusions are drawn.

Panaeolus africanus Ola'h

Ola'h 1968;

Ola'h 1970;

Ola'h 1969 Said to have determined this to be "*psilocybian latent*" but included no details.

Panaeolus antillarum (Fr.) Dennis

Allen & Merlin 1992a: <0.01% PSOP & <0.01% PSOH; Kusano *et al.* 1986: 0.045-0.083% PSOP;

Stijve & de Meijer 1993 could not detect (Brazil).

Panaeolus ater (Lange) Kühner & Romagnesi

Ola'h 1968; Ola'h 1970;

Ola'h 1969 Said to have determined this to be "*psilocybian*" but included no details;

Gurevich 1995 did not observe (Russian).

Panaeolus bisporus Bertault & Maleçon Gerhardt

Senn-Irlet *et al.* 1999: 0.41% PSOH & traces only of PSOP. (central Europe);

Panaeolus cambodginiensis See as Copelandia cambodginiensis

Panaeolus campanulatus Panaeolus subbalteatus (Berk. & Broome) Sacc. Fiussello & Ceruti Scurti 1972; Beug & Bigwood 1982: 0.16-0.65% PSOP but no PSOH; Kusano et al. 1986: 0.04-0.05% PSOP in Japanese collection (anion exchange chromatography & HPLC); (Sardinia): Stijve & de Meijer 1994 found PSOP (Brazilian); Fiussello & Ceruti Scurti 1972: Beug & Bigwood 1982 did not detect PSOP or PSOH in 2 specimens from US; Gurevich 1995 did not observe (Russian); Neal et al. 1968 did not detect either PSOP or PSOH in a gm in a small cap; saprophytic culture. Panaeolus castaneifolius See as Panaeolina castaneifolius recreation in Oregon."; Panaeolus cyanescens See as Copelandia cyanescens Panaeolus fimicola (Fr.) Gillet Ballero & Contu 1998: 0.14% PSOP & 0.03% PSOH & HPLC): (Sardinia); Ola'h 1968; [See Note A; page 225] Ola'h 1970; men; Ola'h 1969 Said to have determined this to be "psilocybian Ola'h 1968; [See Note A; page 225] latent" but included no details; Stijve & de Meijer 1994 found PSOP (Brazilian). (Mexico); Panaeolus goosensiae Ola'h Merlin & Allen 1983: PSOP & PSOH totaling less than 0.01%. but he included no actual details; Panaeolus microsporus Ola'h & Cailleux Ola'h 1968; [See Note A; page 225] Ola'h 1969 Said to have determined this to be "psilocybian the same collection when freshly dried; Stijve & Kuyper 1985: 0.08-0.14% PSOP & no PSOH; latent" but included no details. Panaeolus olivaceus Möller Ohenoja et al. 1987: 0.005% PSOP & no PSOH in a fresh (Brazilian); specimen. Panaeolus papilionaceus (Fr.) Quélet Purportedly used by Portuguese witches: Rudgley 1978; See page 225]; as Panaeolus campanulatus. Panaeolus retirugis (Fr.) Quélet species observed near Chicago, Illinois exhibited bluing. Ballero & Contu 1998: 0.12% PSOP & 0.05% PSOH (Sardinia); Fiussello & Ceruti Scurti 1972. Pluteus atricapillus (Secr.) Singer Panaeolus semiovatus Kusano et al. 1986: 0.0007-0.001% PSOP (Japan) via anion either specimen, exchange chromatography & HPLC. Gurevich 1995 did not observe (Russian). Panaeolus sphinctrinus (Fr.) Quélet Pluteus cyanopus Quelet Ballero & Contu 1998: 0.11% PSOP & 0.08% PSOH Amirati et al. 1989; (Sardinia): Gitte et al. 1983. Heim & Hofmann 1958(1959); Pluteus glaucus Singer Ola'h 1968; Ola'h 1970; [See Note A; page 225] Stijve & de Meijer 1993: 0.28% PSOP & 0.12% PSOH (Bra-Ola'h 1969: Designated as "psilocybian latent"; zilian material). Ola'h reported both PSOP & PSOH in Heim & Hofmann's material via TLC, CC & spectral analysis; Guzmán et al. 1976 reported that it was used as a recreational drug in Oregon with users eating doses of up to 250 carphophores, but in Washington what appeared to be this species had been tried by users and was found to be inactive; Kusano et al. 1986: 0.0.014-0.017% PSOP in Japanese collection (using anion exchange chromatography & HPLC); Gurevich 1995 did not observe (Russian); Ott & Guzmán 1976: NO PSOP in TLC (Mexico); Stein et al. 1959: Found NO PSOP in paper chromatography; observed 3 spots at higher Rf; one was bluish-grey with Ehrlichs;

Stein 1960 also mentioned no PSOP was observed (3 spots observed at higher Rf).

Ballero & Contu 1998: 0.31% PSOP & 0.11% PSOH

Gurevich 1995 reported "considerable amounts" in Russian specimens; Siberia: 3.6 mg/ gm in pileus & 1.7 mg/ gm in stipe; Central Russia: 0.5 mg/gm in pileus & stipe & 1.2 mg/

Guzman et al. 1976 claimed this species "is widely used for

Kusano et al. 1986: 0.061-0.70% PSOP in 3 highly variable Japanese collections (using anion exchange chromatography

Ohenoja et al. 1987; 0.06-0.14% PSOP & 0.0-0.004 PSOH in 3 dried specimens; 0.01% PSOP & no PSOH in a fresh speci-

Ott & Guzmán 1976: PSOP identified (TLC); No PSOH

Ola'h 1969 said he had determined this to be "psilocybian"

Repke et al. 1977b noted that one specimen showed identical PSOP levels after 52 wks of storage as it did specimens from

Stijve & de Meijer 1993: 0.033-0.080% PSOP & no PSOH

Stein et al. 1959: NO PSOP in paper chromatography; (4 spots at higher Rf; 2 were blue with Ehrlichs) [See Note B;

A perhaps important note was made by Singer & Smith 1958 commenting that only 1 out of "hundreds" of this

Panaeolus tropicalis Ola'h See as Copelandia tropicalis Panaeolus venenosus Murr. See as Panaeolus subbalteatus

Ohenoja et al. 1987: 0.004 & 0.005% PSOP & no PSOH in



Psilocybe cubensis cultivation

Pluteus sp. (aff. glaucus Singer)

Stijve & de Meijer 1993: 0.15% PSOP & 0.10% PSOH (Brazilian material).

Pluteus nigroviridis Babos

Stijve & Bonnard 1986.

Pluteus salicinus (Pers. ex Fr.) Kummer Ballero & Contu 1998: 0.009% PSOP & 0.007% PSOH (Sardinia);

Christiansen et al. 1984: 0.35% PSOP & 0.011% PSOH;

Gartz 1987a: 1.20-1.57% PSOP & No PSOH in caps; 0.48-1.14% PSOP & NO PSOH in stems;

Ohenoja *et al.* 1987: 0.21% & 0.30% PSOP & 0.0% & 0.05% PSOH:

Saupe 1981: PSOP & PSOH noted (chromatography); Stijve & Bonnard 1986;

Stijve & Kuyper 1985: 0.05-0.25% PSOP & NO PSOH; Stribrny *et al.* 2003: PSOP & PSOH observed.

Psathyrella candolleana (Fr.) Maire

Ballero & Contu 1998: 0.007% PSOP & 0.002% PSOH (Sardinia);

Gartz 1986b: PSOP & no PSOH observed;

Koike et al. 1981: 0.08-0.15%;

Ohenoja et al. 1987: 0.004% PSOP & 0.005% PSOH;

Gurevich 1995 did not observe (Russian); Ott & Guzmán 1976: NO PSOP in TLC (Mexico);

Stijve & Kuyper 1988: unable to detect any PSOP.

Psilocybe acutissima See as Psilocybe yungensis

Psilocybe arcana Yokoyama

Stribrny *et al.* 2003: 0.01-1.15% PSOP & 0.01-0.85% PSOH.

Psilocybe argentipes Yokoyama

Koike *et al.* 1981: 0.003-0.53%: isolations yielded 0.03% & 0.34% PSOP (both from same site harvested 3 months apart but used different workups) they found no PSOH;



Dried Psilocybe semilanceata (Oregon)

Kusano *et al.* 1986: 1.21-1.35% PSOP in material from 1985; 0.002-0.55% in material from 1979 (Their high testing material from 1985 relied on TLC densitometry whereas the others used anion exchange resin chromatography & HPLC but no details provided);

Yokoyama 1976 appears cited in the literature but simply described this species and noted it hallucinogenic. They did not perform any analysis.

Psilocybe atrobrunnea (Lasch.) Gillet Høiland 1978 reported PSOP; Christiansen *et al.* 1980 & 1984: unable to detect it; Leung *et al.* 1965: unable to detect it;

Guzman 1987 suspected Høiland analyzed another species rather than this one and suggested theirs might be an unknown species that was closer to *P. serbica*.

Psilocybe aucklandii Guzmán, King & Bandala

Guzmán *et al.* 1991 & Johnson & Buchanan 1995: staining greenish-blue: The latter said it was found to contain PSOP/PSOH but included no details.

Psilocybe aztecorum var. *aztecorum* Heim emend. Guzmán Heim & Hofmann 1958: 0.2% PSOP & traces of PSOH (collected in 1956):

Heim & Hofmann 1958(1959); Hofmann et al. 1959.

Psilocybe aztecorum var. bonetii Guzmán

Ott & Guzmán 1976: PSOP identified by TLC; No PSOH (Mexico).

Psilocybe azurescens Stamets & Gartz

Gartz 1994;

Liggenstorfer & Rätsch 1996;

Stamets & Gartz 1995: Tillamook, Oregon: 1.20-1.71% PSOP & 0.25-0.34% PSOH; Astoria, Oregon: 1.18-1.78% PSOP & 0.19-0.38% PSOH; Outdoor cultivated & Naturalized: Germany: 1.17-1.62% PSOP & 0.26-0.42% PSOH; US: 1.21-1.72% PSOP & 0.24-0.38% PSOH.

Psilocybe baeocystis Singer & Smith

Benedict *et al.* 1962a: **NO** PSOP; ONLY PSOH accompanied by tryptamine;

Benedict *et al.* 1962b; Article stolen from UT library. Unable to learn more at present;

Beug & Bigwood 1981: 0.15-0.85% PSOP (0.2% was typical) & 0.0-0.13% PSOH (averaging 0.15%);

Beug & Bigwood 1982: 0.15-0.85 % PSOP & 0.0-0.59% PSOH;

Leung *et al.* 1965: PSOP; NO PSOH; accompanied by Tryptamine. They also examined the same material that had been analyzed by Benedict *et al.* 1962a and found PSOH was the major alkaloid and PSOP was present only as traces; Leung & Paul 1967: 0.04%;

Leung & Paul 1968: isolated but not quantified.

Psilocybe bohemica Šebek

Gartz 1996(1998): 0.34-0.91% PSOP & 0.0-0.04% PSOH; Gartz & Miller 1989: 0.11-0.1.34% PSOP & 0.0-0.02% PSOH; PSOH present in 10 of 23; PSOP higher in caps than stems, in 2 of the 4 evaluated separately they were twice as high but close in the other two;

Semerdzieva *et al.* 1986: 0.25-1.14% PSOP & 0.0-0.07% PSOH; specimens 0-11 years old; highest concentration in the one analyzed soonest after harvest;

Stijve & Kuyper 1985: 0.28-0.80% PSOP & 0.0-0.02% PSOH;

Stribmy *et al.* 2003: 0.1-0.63% PSOP & 0.17-1.27% PSOH; Wurst *et al.* 1984: 1.14% PSOP & 0.02% PSOH (2 yr old), 0.46% PSOP & 0.07% PSOH (3 yr old), 0.63% PSOP & 0.06% PSOH (7 yr old), 0.50% PSOP & 0.07% PSOH (10 yr old) [While an age related decline was claimed, and very likely, no direct correlation can be extracted from the figures due to incomplete documentation, different harvest dates

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and different strains], 0.74% PSOP & 0.03% PSOH in the caps with 0.54% PSOP & 0.03% PSOH in the stem (4 yr old), 0.82% PSOP & 0.04% PSOH in the caps with 0.32% PSOP & 0.04% PSOH in the stem (7 yr old), 0.44% PSOP & 0.05% PSOH in the caps with 0.32% PSOP & 0.07% PSOH in the stem (10 yr old); Wurst *et al.* 1992: 0.046-1.14% PSOP & 0.02-0.48% PSOH.

Kysilka & Wurst 1990: Conventional approach (with MeOH) PSOP (0.932%) & PSOH (0.041%);

Improved (aqueous EtOH & longer extraction) PSOP (1.223%) & (Optimized MeOH) PSOH (0.448%);

Gartz 1994b disputed this and claimed pure methanol was the ideal solvent; we are presently unable to resolve the disparity in opinions. (*See* **p. 229 herein.**)

Psilocybe bonetii See as Psilocybe aztecorum var. bonetii Psilocybe caerulescens var. caerulescens Murrill

Heim & Hofmann 1958: Reported isolation of PSOP; Heim & Hofmann 1958(1959); Hofmann *et al.* 1959: have same data;

Singer & Smith 1958a reported it bluing;

Stein *et al.* 1959: Identified (paper chromatography); Stein 1960: Reported PSOP was observed;

Stijve & de Meijer 1993: 0.1-0.22% PSOP & *nd* -0.25% PSOH (Brazilian material).

Psilocybe caerulipes (Peck) Saccardo Leung *et al.* 1965: PSOP; traces of PSOH.

Psilocybe caeruloannulata Singer ex Guzmán Stijve & de Meijer 1993: 0.055-0.30% PSOP & 0.20-0.23% PSOH (Brazilian material).

Psilocybe callosa (Fr. ex Fr.) Quelet Leung *et al.* 1965: PSOP; NO PSOH; Benedict *et al.* 1967: PSOP but no PSOH (TLC); Singer & Smith 1958a reported it to be bluing.

Psilocybe candidipes See as Psilocybe zapotecorum

Psilocybe collybioides Singer & Smith

Southcott 1974 reported Rickard as finding psilocybin but Guzmán 1983 felt identification was in error. Singer & Smith 1958a described it bluing or greening; Perkal 1981 noted to be collected, dried, crushed, en-

capsulated and sold in the 1972 Tasmanian blackmarket. *Psilocybe coprinifacies* (Roll.) Pouzar

Auert et al. 1980;

Semerd×ieva & Nerud 1973;

Semerd×ieva et al. 1986;

[Wurst *et al.* 1984 appears listed in the literature but simply referred to previous work.]

Psilocybe coprophila (Bull. ex Fr.) Kummer

This entry may be erroneous on my part. Kusano *et al.* 1986 reported 0.08-0.15% PSOP in Japanese *Psilocybe caprophila* which I have not found listed as a species name yet. Its inclusion here is on the basis of my assumption *caprophila* was a typo. Margot & Watling 1981 looked at Australian *Psilocybe coprophila* and found **no** PSOP but did note the presence of an unidentified indole at >0.1% & <0.3%.

Psilocybe cubensis (Earle) Singer

Allen & Merlin 1992a: 0.042% & 0.08% & 0.08% PSOP & 0.58% & 0.20% & 0.19% PSOH;

Bigwood & Beug 1982: found great variability in repeated flushes within single strains [0.0-0.32-1.3% PSOP & 0-0.26% PSOH] but PSOP levels usually had decreased by the 4th flush while PSOH, though lacking in some in the first flush generally appeared and rose with successive flushes. Only able to get 5 flushes in most cases. 5th & 6th flushes, if occurring were higher than the 4th and some other earlier flushes but not as high as the peak value of the earlier flushes. There was no clear pattern from one culture to the next beyond that. In analysis of the first flush only of three strains they found: 0.97% PSOP & 0.0% PSOH in the caps with 0.42% PSOP & 0.35% PSOH in the stem of their "M.R." strain; 0.57% PSOP & 0.01% PSOH in the caps with 0.57% PSOP & 0.0% PSOH in the stem of an Amazonian strain from near Pulcalpa, Peru; 0.76% PSOP & 0.0% PSOH in the caps with 0.47% PSOP & 0.04% PSOH in the stem of a strain from Ecuador; An evaluation of 5 "street samples found variable levels of 0.07-0.62% PSOP and 0.0-0.03% PSOH;

Gartz 1987a: PSOP higher in caps (1.20-1.57%) than stems (0.62-1.14%). DID NOT find PSOH;

Gartz 1989e: 0.01-0.20% PSOP & 0.0-0.15% PSOH in cultured pshrooms but 0.46-0.61% PSOP & 2.1-3.3% PSOH if cultures supplemented with tryptamine;

Gartz & Müller 1989; only mentions work from 1987a: did not look at this species;

Heim & Hofmann 1958: 0.25% PSOP & traces of PSOH (collected in 1956); 0.24% in the same but cultured in France (Mexico); 0.5% PSOP & 0.1% PSOH (collected in 1957: Thailand); 0.3% PSOP & traces PSOH (collected in 1957: Cambodia); 0.2% PSOP (same Cambodian collection cultivated in France);

Hofmann et al. 1959;

KvW 2002: 0.5-0.9% total alkaloid (smartshop fungi) no significant differences based on flush;

Margot & Watling 1981: traces of PSOP & no PSOH (Australian herbarium specimen);

Repke *et al.* 1977b: 0.168% PSOP & 0.42% PSOH in GLC-MS quantification. Higher proportion of PSOH to PSOP: confirmed by TLC. They wondered if due to partial hydrolysis during isolation.);

Repke *et al.* 1977b noted PSOP & PSOH were stable for 52 wks stored at -5° C under anhydrous conditions and the same as in freshly dried (14 days) but undetectable if dried and stored at 22° for 52 wks;

Stein 1960 reported that PSOP was observed (2 spots observed at higher Rf);

Stein *et al.* 1959: Identified PSOP in paper chromatography An interesting account of a bad trip was discussed in Stein 1958, after eating 5 gm of *cubensis* that he fried in butter, but it should be noted that this was most likely NOT helped by his dropping reserpine 38 minutes after eating pshrooms;

Stijve & de Meijer 1993: Wild: 0.1-0.36% PSOP & 0.2-0.6% PSOH (Brazil); Labgrown: 0.12% vs. 0.15% PSOP & 0.05% vs 0.50% PSOH (Mexican str.); 0.12% vs. 0.15% PSOP & 0.10% vs. 0.33% PSOH (Amazon: str.) ["vs" refers to the use of MeOH vs. 75% aq. MeOH sat. w/ potassium nitrate for PSOP & 75% EtOH for PSOH (overnight soakings)];

Trout's Notes on Tryptamines: 4-Substitution







Psilocybe cyanofibrillosa Bay area Photos by James Edmond







Jackson & Alexpoulos 1976 found that the Texas *cubensis* has a shorter life cycle (faster to fruit) than the Mexican *cubensis* and noted this showed a perfect adaptation for the cycle of harsh winters and intermittent droughts of Texas when contrasted with the more tropical and lush environment in Mexico.

Psilocybe cyanescens Wakefield

Benedict et al. 1962b [stolen; could not confirm];

Beug & Bigwood 1982: 0.15-1.68% PSOP & 0.06-0.96% PSOH;

Ballero & Contu 1998: 1.24% PSOP & 0.72% PSOH (Sardinia);

Gartz 1996(1998): 0.41-0.98% PSOP & 0.23-0.93% PSOH (USA); 0.33-0.71% PSOP & 0.02-0.05% PSOH (Europe); also reported a rise in PSOP levels in flush 3 & 4 and in PSOH in flush 4 (USA);

Margot & Watling 1981: >0.3% PSOP & traces PSOH;

Singer & Smith 1958b reported it bluing;

Stijve & Kuyper 1985: 0.20-0.85% PSOP & 0.04-0.36% PSOH;

Stribrny *et al.* 2003: 0.13-1.84% PSOP & 0.28-1.81% PSOH; Unger & Cooks 1979: Using MS/MS & MIKES;

Wurst *et al.* 1992: 0.0% PSOP & 0.45% PSOH (WA, US: 1984) 0.10% PSOP & 0.47% PSOH (Horni Bradol, Czech. Rep.: 1986).

Psilocybe cyanofibrillosa Stamets & Guzmán

Stamets *et al.* 1980: 0.005-0.21% PSOP & 0.04-0.14% PSOH. Found that two 2-yr old herbarium specimens had lost most of their alkaloid content; Internet accounts and a friend's collection witnessed in 2003 evidenced this species usefulness in successful human bioassays inOakland and elsewhere in the SF Bay Area. It has been pointed out that the specimens collected do not precisely match the description and may be an undescribed species

Psilocybe eucalypta Guzmán & Watling

Margot & Watling 1981: over 0.10 and under 0.3% PSOP & no PSOH;

(Johnson & Buchanan 1995: stained greenish-blue.)

Psilocybe fimetaria (Orton) Watling

Benedict *et al.* 1967: PSOP identified but no PSOH; by TLC. *Psilocybe hoogshagenii* var. *convexa* Guzmán

Heim & Hofmann 1958: 0.6% PSOP & 0.1% PSOH (collected in 1956);

Heim & Hofmann 1958(1959): same data; Hofmann *et al.* 1959 (same data).

Psilocybe hoogshagenii var. hoogshagenii Heim

Stijve & de Meijer 1993: 0.15-0.30% PSOP & 0.20-0.30% PSOH (Brazilian material).

Psilocybe isauri Sing. See as Psilocybe yungensis

Psilocybe liniformans var. americana Guzmán & Stamets Stamets et al. 1980: 0.59-1.28% PSOP & no PSOH. Herbarium specimens reported to retain potency.

Psilocybe liniformans var. *liniformans* Guzmán & Bas Stijve & Kuyper 1985: 0.16% PSOP & NO PSOH.

Psilocybe mairei Singer

Auert et al. 1980;

Ballero & Contu 1998: 0.0% PSOP & 0.0% PSOH (Sardinia); Semerdzieva & Wurst 1986.

Psilocybe makarorae Johnson & Buchanon

Johnson & Buchanan 1995: reported it stained greenish-blue and noted it was found to contain PSOP/PSOH but included no details. Margot & Watling found no indoles in aged herbarium material.

Psilocybe mexicana Heim

Heim & Hofmann 1958& 1958(1959): See Hofmann *et al* 1958 below;

Hofmann & Troxler 1959: identified PSOH following an earlier observation that an unidentified alkaloid co-occurred with PSOP. The identity of the species was not specifically stated in this paper;

Hofmann et al. 1958: 0.4% PSOP & traces of PSOH;

Hofmann et al. 1959 (same as 1958);

Stein *et al.* 1959: Identified PSOP in paper chromatography; Stein 1960 Reported that PSOP was observed (2 spots observed at higher Rf);

Collected and sold to tourists by children in Guatemala: Lowy 1977.

Psilocybe muliercula See as Psilocybe wassonii

Psilocybe natalensis Gartz, Reid, Smith & Ecker Gartz 1994;

Gartz et al. 1995: PSOP, PSOH & Baeocystin "in concentrations similar to Psilocybe cubensis..."

Psilocybe pelliculosa (Smith) Sing & Smith

Beug & Bigwood 1982: PSOP at 0.12-0.71% but no PSOH; Repke & Leslie 1977: Recovered 0.08% PSOP: observed traces of PSOH;

Singer & Smith 1958b: slight bluing or greening;

Tyler 1961: PSOP identified chromatographically but did not observe PSOH.

Psilocybe pseudobullacea (Petch) Pegler

Marcano *et al.* 1994: PSOP & PSOH present but not quantified.

Psilocybe pugetensis Harris nom. nud. See as Psilocybe stuntzii Psilocybe quebecensis Ola'h & Heim

Heim et al. 1967;

Ola'h & Heim 1967.

Psilocybe samuiensis Guzmán, Allen & Merlin

Gartz *et al.* 1994: 0.23-0.90% PSOP & 0.05-0.81% PSOH (Thailand); 0.36-0.73 PSOP & 0.21-0.52% PSOH (cultivated); caps determined to contain more PSOP than the stems; Guzmán *et al.* 1993.



Psilocybe subcubensis (Thailand) Photo by JW Allen

Psilocybe semilanceata (Fr.) Kummer [Note C: p. 225]

Benedict *et al.* 1967: PSOP identified but no PSOH; by TLC; Beug & Bigwood 1982: PSOP at 0.69-1.28 % but not PSOH) *"typically in excess of 10 mg per gm"*;

Christiansen *et al.* 1981a: 0.72-1.01% PSOP; PSOH in traces; Christiansen *et al.* 1981b: 0.71-1.96% PSOP;

Christiansen & Rasmussen 1982b: 0.15-1.05% PSOP in stems; 0.19-1.10% PSOP in caps; 0.17-1.08% PSOP total & no PSOH; Looking at stored herbarium material, they found 1.42% & 0.75 in 4 year old samples, 0.63% in a 5 yr old; 0.61% & 0.55% in 6 yr old; 0.87% in an 8 yr old; 0.41 & 0.65% & 0.64% in 13 yr old; 0.54% in a 14 yr old; 0.29% in a 22 yr old; 0.12% in a 26 yr old; 0.15% in a 27 yr old; 0.38% & 0.10% in 29 yr old and 0.05% in a 30 yr old sample;

Christiansen & Rasmussen 1983: PSOP detected as major alkaloid but not quantified; PSOH present at 0.002%; Gartz 1985a;

Gartz 1985g;

Gartz 1986a: 0.19-1.45% PSOP & NO PSOH; smaller caps often had higher concentrations than larger caps but the highest PSOP TOTAL content was found in some, not all, of the larger caps; interestingly some apparently blued and others did not but Gartz found no correlation between bluing and PSOP content;

Gurevich 1995 reported in Russian specimens;

Hofmann *et al.* 1963: 0.25% PSOP isolated; no mention of PSOH;

Høiland 1978;

Jokiranta *et al.* 1984: 0.62-2.37% PSOP & 0.0-0.02% PSOH; concentration of PSOP was inversely proportional to weight but absolute values of PSOP content were directly proportional to weight (i.e. smaller caps had a higher concentration of PSOP but larger caps had more total PSOP content); pooled samples of unselected caps showed 1.68 & 1.53% PSOP (average weight loss during drying was 90%);

Tao Jones (2002 pers. comm) commented that he found 10 yr old specimens that were quite potent; noting this species retains potency much longer than others;

Mantle & Waight 1969: 0.15% PSOP & no PSOH;

Margot & Watling 1981: over 0.3% PSOP & traces of PSOH; Ohenoja *et al.* 1987: 0.20 & 0.87% PSOP & 0.0 & PSOH in 2 dried specimens and 0.19-0.82% PSOP & 0.003-0.025% PSOH in 3 fresh specimens; They also found 0.84% PSOP & no PSOH in 11 yr old specimen; 0.57% PSOP in a 33 yr old specimen; 0.014% PSOP in a 118 yr old specimen and none in a 144 yr old specimen;

Pederson-Bjergaard et al. 1997: detected;

Repke & Leslie 1977: recovered 0.36%; no mention of PSOH; Semerdzieva *et al.* 1986: 0.91-1.05% PSOP & 0.0-0.12% PSOH in specimens, 0 to 12 years old; highest concentration was in a two-year old specimen;

Stijve & Kuyper 1985: 0.05-1.70% PSOP & 0.0-0.02% PSOH;

Stijve & de Meijer 1993: 0.39% vs. 0.47% PSOP & no PSOH in either (Swiss) [MeOH vs. 75% aq. MeOH saturated with potassium nitrate for PSOP & 75% EtOH for PSOH (O/N soaks)];

Støbrný *et al.* 2003: 0.12-0.51% PSOP & 0.06-0.27% PSOH; White 1979: Identified PSOP & PSOH: no quantification;



cultivated Amazonian Psilocybe cubensis (dried)

Wurst *et al.* 1984 (1.05% PSOP & 0.12% PSOH (4 yr old), 0.91% PSOP & 0.09% PSOH (14 yr old); 0.83% PSOP & 0.08% PSOH in the caps with 0.33% PSOP & 0.10% PSOH in the stem (3 yr old), 0.74% PSOP & 0.68% PSOH in the caps with 0.45% PSOP & 0.04% PSOH in the stem (15 yr old).)

Wurst *et al.* 1992: 0.76-1.05% PSOP & 0.09-0.12% PSOH (Czech. Rep.)

Psilocybe semilanceata var. caerulescens See as Psilocybe callosa

Psilocybe semperviva See as Psilocybe hoogshagenii var. convexa

Psilocybe serbica Moser & Horak

Moser & Horak 19968: PSOP & PSOH identified chromatographically;

Semerd×ieva & Nerud 1973.

Psilocybe silvatica (Peck) Sing. & Smith ? Is listed in the literature but the references cited did not report PSOP or PSOH; see in bluing list p. 81.

Psilocybe stuntzii Guzmán & Ott

Beug & Bigwood 1982: PSOP at 0.0-0.36%; PSOH at 0.0-0.012%;

Guzmán & Ott 1976: Identified PSOP by chromatography. Did not observe PSOH but noted possibility it may have been present when collected and degraded.

Psilocybe strictipes Smith See as Psilocybe callosa

Psilocybe subaeruginascens var. *subaeruginascens* Höhne

Koike et al. 1981: 0.017-0.018%;

Singer & Smith 1958b: bluing.

Psilocybe subaeruginosa Cleland

Perkal *et al.* 1980: 0.01-0.2% PSOP with traces of PSOH;

Perkal 1981: 0.06-1.93% PSOP & 0.0-0.17% PSOH (Victoria, Australia);

Picker & Rickards 1970: 0.45% PSOP isolated; PSOH was not detected;

Voogelbreinder 2002 reported it to sometimes be much stronger than believed in human bioassays (AUS);

Margot & Watling 1981: no indoles (old material).

Psilocybe subcaerulipes Hongo

Kusano *et al.* 1986: 0.34-0.81% PSOP (material from Hiroshima in 1981);

Koike *et al.* 1981 did not detect in a mycelial culture of this species;

Yokoyama 1973 had reported positive bioassays but later decided (Yokoyama 1976) that it was a similar but new species which was designated *Psilocybe argentipes*. See more under.

Psilocybe subcubensis Guzmán

Allen & Merlin 1992a: 0. 37% PSOP & 0.26% PSOH; Marcano *et al.* 1994: PSOP & PSOH present but not quantified.

Psilocybe cf. subyungensis Guzmán

Stijve & de Meijer 1993: 0.50% PSOP & 0.40% PSOH (Brazilian material).

Psilocybe tampanensis Guzmán & Pollock

Gartz et al. 1994: 0.34-0.68% PSOP & 0.21-0.52% (?) (sclerotia grown on malt agar & Lolium seed); 0.41-0.61% PSOP & 0.11-0.32% PSOH (sclerotia grown on Lolium seed); (? reflects statement of PSOP saying "malt agar and Lolium seed" & PSOH saying simply "malt agar");

Guzmán & Pollock 1978: Reported as bluing & Pollock bioassayed successfully but no analysis; KvW 2002: 0.3 % total alkaloid (sclerotia); Peele 1985;

Ott 1996: Apparently rare in nature. Thought to have been collected only the one time. Now widely cultivated for deliberate use. (as sclerotia & fruit).

Psilocybe thailandensis Guzmán & Allen

Stijve & de Meijer 1993: 0.055% vs. 0.075% PSOP & 0.1 vs. 0.6% PSOH [MeOH vs. 75% aq. MeOH saturated with potassium nitrate for PSOP & 75% EtOH for PSOH (O/N soaks)].

Psilocybe uruguayensis Sing. ex Guzmán Stijve & de Meijer 1993: 0.085-0.14% PSOP & *nd*-0.01% PSOH (Brazilian material)

Psilocybe wassonii Heim

Escalante & López 1971; Escalante *et al.* 1973;

Heim & Wasson 1958[1959]; Singer & Smith 1958b: Bluing.

Psilocybe zapotecorum Heim

Heim & Hofmann 1958: 0.2% PSOP & no PSOH (collected in 1956); Heim & Hofmann 1958(1959); Hofmann *et al.* 1959: same data;

Ott & Guzmán 1976: PSOP identified by TLC; No PSOH (Mexico);

Singer & Smith 1958b: Bluing;

Stijve & de Meijer 1993: 0.06-0.30% PSOP & 0.05-1.0% PSOH (Brazilian material).



Psilocybe cyanofibrillosa



Unidentified *Psilocybe* sp. Bay area Photo by James Edmond



Psilocybe cyanescens (AUS) Photo by Snu Voogelbreinder

Species that are suspected to be active, based largely on bluing:

The following are, so far as we can discern, presently unanalyzed or in some instances their analysis failed to find anything active.

Some of Guzmán's suspects were based on relationships to known actives with bruising either not noted or absent.

As above, only minimal effort was made to assign synonyms or sort out taxonomic conflicts since there are so many varying opinions. The following are names listed or synonyms proposed when encountered. The final disposition is in the hands of the experts. Let's hope some day some semblance of a consensus is reached.

Species without references were from Ott 1996 who cited: Allen et al. 1991 & 1992; Gartz 1985d; Guzmán 1983; Guzmán et al. 1988 & 1991; Levine 1967; Merlin & Allen 1993; Johnson & Buchanan 1995; Stamets 1996.

Copelandia affinis Horak (Allen 2001)

Copelandia lentisporus (Ew.Gerhardt) Guzmán (Allen 2001) = Copelandia mexicana Guzmán

Copelandia tirunelveliensis Natarajan & Raman (Allen 2001)

Copelandia westii (Murrill) Singer

Gymnopilus braendlei (Peck) Hesler (Singer 1978)

Gymnopilus intermedius (Sing.) Sing. (Singer 1978)

Gymnopilus luteoviridis Thiers (Singer 1978: bluing; Hatfield *et al.* 1978 could not detect PSOP)

Gymnopilus purpuratus var. pampeanus

Gymnopilus subpurpuratus Guzmán-Davalos & Guzmán (greenish-bruising: Allen *et al.* 1992)

Mycena amicta (Active compounds remain unisolated in the *Mycena* spp.)

Mycena cyanescens

Mycena pura

Naematoloma popperianum Singer (Singer 1973; Singer 1978)

Panaeolina microsperma Natarajan & Raman (Allen 2001)

Panaeolina rhombisperma Hongo (Allen 2001)

Panaeolus rubricaulis Petch (Allen 2001)

Panaeolina sagarae Hongo (Allen 2001)

Panaeolina venezolanus Guzmán (Allen 2001)

Psilocybe acutipilea (Speg.) Guzmán (Guzmán 1983 suspected)

Psilocybe angustipleurocystidiata Guzmán (Guzmán 1983)



dried Hygrophorus conica (JLF)

Psilocybe antioquensis Guzmán, Saldarriaga, Pineda, Garcia & Velázquez

Psilocybe aquamarina (Pegler) Guzmán (Guzmán 1995) Psilocybe armandii Guzmán & Pollock

Psilocybe atlantis Guzman et al. (Guzman et al. 2002) [A successful human bioassay was reported but unpublished]

- Psilocybe banderillensis Guzmán (Guzmán 1983)
- Psilocybe brasiliensis Guzmán (Guzmán 1983)

Psilocybe brunneocystidiata Guzmán & Horak (Guzmán 1983)

Psilocybe caerulea (Kriesl) Noordeloos (Stamets 1996)

Psilocybe caerulescens var. *murrill* (Material collected Atlanta GA, USA; ID by Guzman: pers. comm. Craig 2004)

Psilocybe caeruleoannulata Singer ex Guzmán (Guzmán 1983)

Psilocybe carbonaria Singer (Guzmán 1983 suspected due to relationships)

Psilocybe chiapanensis Guzmán (Guzmán 1995)

Psilocybe columbiana Guzmán (Guzmán 1983)

Psilocybe dumontii Sing. ex Guzmán (Guzmán 1983)

Psilocybe fagicola Heim & Cailleux (Guzmán 1983)

Psilocybe fagicola var. mesocystidiata Guzmán (Guzmán 1983)

Psilocybe farinacea Rick ex Guzmán (Guzmán 1983)

Psilocybe fuliginosa (Murr.) Smith (Guzmán 1983)

Psilocybe furtadoana Guzmán (Guzmán 1983)

Psilocybe galindii Guzmán (Guzmán 1983)

Psilocybe goniospora (Berk. & Broome) Singer

Psilocybe graveolens Peck (Guzmán 1983)

Psilocybe guatapensis Guzmán, Saldarriaga, Pineda, Garcia & Velázquez

Psilocybe guilartensis Guzmán, Tapia & Nieves-Rivera

Psilocybe heimii Guzmán (Guzmán 1983)

Psilocybe heliconiae Guzmán, Saldarriaga, Pineda, Garcia & Velázquez

Psilocybe herrerae Guzmán (Guzmán 1983)

Psilocybe hispanica Guzmán

Psilocybe inconspicua Guzmán & Horak (Guzmán 1983 suspected based on relationships)

Psilocybe indica Sathe & Daniel

Psilocybe isabelae Guzmán

Psilocybe jacobsii Guzmán (Guzmán 1983)

Psilocybe jaliscana Guzmán

Psilocybe laurae Guzmán (Guzmán 1998: suspected based on relationships; Allen 2001)

Psilocybe lonchophorus (B. & Br.) Horak ex Guzmán

Psilocybe maire Singer (Guzmán 1983; Stamets 1996)

Psilocybe mammillata (Murr.) Smith (Guzmán 1983)

Psilocybe meridensis Guzmán (Guzmán 1995)

Psilocybe mescaleroensis Guzmán *et al.* (Guzmán *et al.* 2007b) [Traditonal use asserted.]

Psilocybe moseri Guzmán (Guzmán 1995)

Psilocybe novae-zelandiae Guzmán & Horak (*zealandiae*) (Stained greenish-blue for Johnson & Buchanan 1995; Reportedly used in New Zealand: Allen *et al.* 1991; Guzmán *et al.* 1991 referred to as nonblueing and placed in a section of the genus with no active species. Johnson & Buchanan and Guzmán may have looked at different species but there is not enough information available for us to sort this out.) Psilocybe ochreata (Berk. & Broome) Horak ex Guzmán (Guzmán 1983 suspected based on relationships.) Psilocybe ovoideocystidiata Guzmán & Gaines (Guzmán et al. 2007a) Psilocybe papuana Guzmán & Horak (Guzmán 1983) Psilocybe paulensis (Guzmán & Bononi) Guzmán Psilocybe pericystis Singer Psilocybe pintonii Guzmán (Guzmán 1983) Psilocybe pleurocystidiosa Guzmán (Guzmán 1983) Psilocybe plutonia (Berk. & M.A. Curtis) Sacc. (Guzmán 1983 suspected based on relationships.) Psilocvbe portoricensis Guzmán, Tapia & Nieves-Rivera Psilocybe pseudoaztecorum Natarajan & Raman (Natarajan & Raman 1985) Psilocybe puberula Bas & Noordel. Psilocybe ramulosa (Guzmán & Bononi) Guzmán Psilocybe rostrata (Petch) Pegler Psilocybe rzedowskii Guzmán (Guzmán 1983) Psilocybe sanctorum Guzmán Psilocybe schultesii Guzmán & Pollock (Guzmán 1983 suspected based on relationships.) Psilocybe septentrionalis (Guzmán) Guzmán Psilocybe sierrae Singer (Singer 1978) Psilocybe silvatica Peck (Guzmán 1983 notes as bluing & to be hallucinogenic but lacking analysis; 2 papers by Repke have also been cited: one did not examine it & the other study looked only for baeocystin; Singer & Smith 1958b did not note bluing.) Psilocybe singeri Guzmán (Guzmán 1983) Psilocybe subacutipilea Guzmán, Saldarriaga, Pineda, Garcia & Velázquez Psilocybe subaeruginascens Höhne var. septentrionalis Guzman Psilocybe subfimetaria Guzman & Smith (Guzmán 1983) Psilocybe subtropicalis Guzmán (Guzmán 1995) Psilocybe subyungensis Guzmán (Guzmán 1983) Psilocvbe subzapotecorum Guzmán Psilocybe uxpanapensis Guzmán (Guzmán 1983) Psilocybe veraecrucis Guzmán & Perez-Ortiz (Guzmán 1983) Psilocybe villarrealiae Guzmán (Guzmán 1998 suspected based on relationships) Psilocybe wassoniorum Guzmán & Poll. (Guzmán 1983)Psilocybe weldenii Guzmán (Guzmán 1983) Psilocybe wrightii Guzmán (Guzmán 1983) Psilocybe xalapensis Guzmán & Lopez (Guzmán 1983)

For thoughts and references concerning Blueing see Note D on page 226

Use reported but analysis either lacking or in conflict:

Conocybe siligineoides (Heim 1956b: Reportedly used in Mexico.)

Coprinus atramentarius (Kucharz *et al.* 1999: 30-50 caps eaten by teenagers in Poland for drug effects. Time course & description of short-lived nonfatal "*poisoning*" sounds suspiciously psilocybian or, some have suggested, placeboid.)

Gymnopilus luteofolius (J.W. Allen; Internet post 2001: Successfully bioassayed. Hatfield *et al.* 1978 were unable to find any active alkaloids.)

Mycena cyanorrhiza **Quélet**)Allen *et al.* 1992: Bluing but lacks analysis. Positive human bioassay by Allen.)

- Pluteus atricapillus var. ealensis (Horak 1978)
- *Psathyrella sepulchralis* (Singer *et al.* 1958a: Reportedly used in Mexico; Guzmán suspected the identity was erroneous: Ott & Guzmán 1976.)
- *Psilocybe australiana* Guzmán & Watling (Guzmán 1983: bluing; Allen *et al.* 1991 indicated use; Margot & Watling 1981 failed to detect any active alkaloids.)
- *Psilocybe barrerae* Cifuentes & Guzmán emend Guzmán [Guzmán 2000]
- *Psilocybe caerulescens* var. *ombrophila* (R.Heim) Guzmán (= *P. mixaeensis*) (Heim & Wasson 1958: Blueing species reported used in Mexico; Lipp 1991.)
- *Psilocybe cordispora* **R.Heim** (Heim 1956b: Reported used in Mexico; Lipp 1990; Lipp 1991; Miller 1966.)
- *Psilocybe* aff. *cyclonic* Stamets. (Material believed to be this species was found active in a human bioassay in 2005.)
- Psilocybe fagicola var. fagicola R.Heim & Cailleux (Heim & Cailleux 1959: No analysis but apparently used in Mexico)
- Psilocybe gallaeciae Guzmán & M.L.Castro (Guzmán & Castro 2003: No analysis but used as a recreational hallucinogen in Gallacia.)
- *Psilocybe kumaenorum* **R.Heim** (Guzmán 1983: bluing; Heim 1978; Heim *et al.* 1967; Reportedly used in New Zealand: Allen *et al.* 1991.)
- *Psilocybe* sp. (An unidentified tropical-appearing species fruiting in the Bay was found to be active in human bioassays.)
- *Psilocybe tasmaniana* Guzmán & Watling (Guzmán 1983 suspected based on relationship rather than bluing; (Allen *et al.* 1991 indicated use; Margot & Watling 1981 failed to detect any active alkaloids.)
- *Psilocybe venenata* (S.Imai) Imaz. & Hongo (Singer & Smith 1958a/b & Guzmán 1983: bluing; Imai 1932 reported as causing a "*special intoxication*"; Matsuda 1960: Not actually studied but believed to be psilocybian.)
- *Psilocybe yungensis* Singer & A.H.Smith (Used in Mexico and successfully bioassayed by Wasson in 1958: Heim & Wasson 1958; Wasson 1959a; Hofmann failed to detect active alkaloid in dried material: Heim & Calleux 1959; Heim & Wasson 1958.)

See Note E on page 226

Reviews:

Cerletti 1959 Hofmann 1958 & 1971



Unidentified *Psilocybe* sp. Bay area Photos by James Edmond





Trout's Notes on Tryptamines: 4-Substitution









Psilocybe cyanescens Bay area Photos above & upper left by James Edmond

Psilocybe semilanceata (Netherlands) Photo copyrighted by Perfect Fungi Europe 2005 Photo second from top on left

Hildesheim Cathedral, Germany from Samorini 2002





TD_{Lo} (from Sax 1984)

- Toxic Dose Low i.e. the least amount required to produce "toxic" effects in humans.
- (Activity itself is apparently considered to be a toxic effect in Sax' view. Sax cited the references listed.)

Hollister *et al.* 1962 reported primarily effects such as anxiety, euphoria, introspection & visual effects lasting around 4 hours. The values given by Sax below were the lowest doses administered by Hollister and were nowhere indicated by Hollister to be the lowest doses capable of producing effects. Hollister's study evaluated activity rather than toxicity.

Wolbach *et al.* 1962 similarly evaluated activity rather than toxicity and used dosage levels of 37.5, 75 & 150 μ g/ kg; all were administered IM

- 75 µg/kg Wolbach et al. 1962
- $60 \,\mu\text{g} / \text{kg}$ Hollister *et al.* 1960 (oral)
- 37 µg / kg Hollister *et al.* 1960 (parenteral)
- 130 μg/kg Sax 1984 cited 1967 Proc. Eur. Soc. for the Study of Drug Toxicity 8: 59.

Activity:

- Hallucinogen. 4-8 mg Hofmann et al. 1959
- Hofmann 1971 refers to this amount as a "*medium oral dose*" roughly equivalent to 2 grams of dried *Psilocybe mexicana*.
- Found to cause "definite mental changes" at 2 mg/ 70 kg or above by Isbell 1959 (testing 0.5-8.0 mg/ 70 kg and giving 3 dosage levels of PSOP (4, 6 & 8 mg/ 70 kg), 2 dosage levels of LSD (1.0 & 1.5 μ g/kg) and placebo-in randomized order with 8 AM administration to each of 7 "volunteers;" chosen from the black male "former drug addict" portion of the prison population who were serving time for "violation of United States narcotics laws" Use of incarcerated prisoners hardly qualifies as use of volunteers free from duress!)

A human CNS and psychotropic. Sax 1984

Psilocybin was also reported to show anticholinesterase activity in Bhattacharya & Sanyal 1971. (Citing Richter & Crossland 1950 which seems too early.)

Believed to be converted to psilocin in vivo.

- Shulgin & Shulgin 1997 notes this was apparently assumed and taken for granted, based on the chemistry, before any actual study was performed in humans.
- The subject was more recently studied by Hasler *et al.* 1997: who proved a rapid and extensive conversion of PSOP to PSOH (after both oral and IV administration) but was unable to demonstrate either the absence of PSOP or its complete conversion due to lacking an effective assay for determining plasma levels.
- They suggested that the extremely high plasma clearance of PSOH following IV administration of PSOP might be explained by an incomplete conversion of PSOP to PSOH but were unable to prove or disprove it.
- [For whatever reason this detail seems to have been missed by most people commenting on this report.]
- They estimated an absolute bioavailability of 52.7% for PSOH; following oral administration of PSOP.
- The same is assumed for the acetoxy ester. However, in another related material (4-HO-DIPT) studied via multiple human bioassays, evaluating both acetate ester and parent compound, users reported subjective differences and most of those expressing a preference preferred the acetoxy.

- Whether the acetate esters reach the brain intact clearly needs to be studied and not simply assumed that they do not. Toad 2001
- There are also some clear quantitative biological differences that suggest the conclusion of complete synonymity may be premature. (See comments farther below; and also those made above: under psilocin.)

Dose: 10-20 mg oral Shulgin & Shulgin 1997

- Hasler *et al.* 1997 found 3 mg IV was considered to be too much after one evaluation in one subject (causing vertigo, vomiting, cardiovascular side-effects, derealisation & depersonalization. This was reported as unpleasant and fear-inducing "*due to a complete loss of contact with reality*" (They used naive subjects.) They did not repeat this dosage again and used 1 mg IV for their work.
- "Psychoptic" above 10 mg [Ott 1996: Delay *et al.* 1958 used 10 mg; Delay et al. 1959 suggested 15 mg as an uppermost limit]; threshold 3.4 mg [Ott 1996 cited Abramson & Rolo 1967]; 6 mg/b.i.d. Usdin & Efron 1979 cited Paterson 1963; 0.2 mg (? /kg?) Usdin & Efron 1979 cited Arnold & Hoff 1962.
- 3.5-14.6 mg/po Usdin & Efron 1979 cited Jacob 1966 5-14 mg/ im Usdin & Efron 1979 cited Jacob 1966.
- Fisher 1965 suggested that 15-30 mg be used for the initial experience in people experienced with LSD but that 50-70 mg was recommended if intended to serve as an LSD substitute.
- He described 2-8 mg as a low dose and 20-40 mg as a *"standard initial psychedelic experience*".
- Fisher commented that with the right environment and mental setting *"rather amazing states of expanded consciousn*ess" could result with as little as 1 or 2 mg in an experienced individual.
- Fisher also noted that the highest known human dose was 120 mg. It is quite likely that this has been exceeded in unreported human bioassays.
- Wolbach *et al.* 1962 determined 1 μ g/kg of PSOH was equivalent to 1.48 μ g/ kg of PSOP.

Duration: 3-6 hours Shulgin & Shulgin 1997

Receptor site specificity:

High affinity for 5HT₁₄ & 5-HT,

Callaway & McKenna 1998

See also Almaula et al. 1996b (or McBride 2000)

Biochemical & Animal miscellany:

- Monnier 1959 noted that, in rabbits, the stimulating action on the somatic behavior and electrical brain activity is not due to an activation of the reticular arousal system but rather due to a depression of the mediothalamic recruiting and moderating system.
- Freedman 1963 showed that after 90 minutes, 50 mg of PSOP had caused an 11% rise in brain serotonin and a 20% fall in norepinephrine; in rabbits. [The 2% fall in epinephrine mentioned in the literature was apparently a typo.]

Freedman et al. 1970 reported some apparent MAOI activity in vivo in rat brains

- An unsurprising observation is that spiders given 6 grams per kg of psilocybin did not build webs.
- Of the spiders given 600 mg/kg only 5% built webs. It's quite amazing that 5% COULD build webs.
- 54% of the spiders built webs after 150 mg/ kg.
- It was noted that the length of the strands were shorter but not thicker and, unlike mescaline, it did not change the regularity of the webbing angles; **IF** the spiders built webs while they were tripping. (Oral in a sugar solution)
- See Christianson et al. 1962 (They concluded that motivation was impaired more than performance, a feature commented upon by Hollister in human trials)

Jackson & Alexpoulos 1976 note that cows are quite fond of *P. cubensis* and regularly graze them in the early morning before they can be picked by humans but show no visible effects. I have to question this. On a number of occasions cows have been observed, with that certain look in their eyes. These were hesitant to move but when forced to showed panicky, clumsy movements & stumbling far in excess of what was usual; causing me to wonder if they weren't tripping.

"Clinical Syndrome" resulting from Psilocybin administration:

Wolbach et al. 1962 reported that effects began in a few minutes after IM administration, peaked in around 30 minutes and had largely subsided in less than 4 hours. from Hollister et al. 1960:

- During the first half hour: Anxiety, restlessness, dizziness, giddiness, nausea, abdominal discomfort, shivering, muscle aches, twitches, weakness and numbness of lips.
- During the second half hour: Yawning, tearing, sweating, flushed face, incoordination, tremulous speech, decrease in concentration & attention, slowness of thought, depersonalization, dreaminess, feelings of unreality, increase in hearing, and visual effects including blurring, colors brightening, outlines becoming more sharply defined, more persistent after-images and closed-eye visual patterns.
- During the second hour: Perception of a slowing of the passage of time, impairment of distance perception, wavelike motion of surfaces, euphoria, increased perception, a meditative state, and increased visual effects (mostly closed-eye) including colored patterns & shapes.
- Hours 3 and 4: A diminishment and nearly complete disappearance of the aforementioned effects.
- Hours 4-12: Usually normal.
- After effects were said to include headache, fatigue and a contemplative state.
- Less commonly encountered side-effects included, transient sexual feelings, a decrease in appetite, difficulty in breathing, uncontrollable laughter, numbness, tingling and synesthesia (perception of a sensory crossover involving two or more different senses).

- They observed that the drug "impaired motivation more than performance as the ratio of correct responses to line drawings attempted remained constant even though the number attempted was reduced."
- In comparison to LSD, they noted that psilocybin produced a "dreamy, introspective state at dose levels which did not produce predominant somatic effects nor marked impairment of mental functions" and that the "whole effect" produced was "more agreeable" than that of LSD.

Summary of pharmacological properties of PSOP & PSOH:

from Cerletti et al. 1968:

No significant effects on isolated organs except for antagonizing 5HT in isolated rat uterus (Also reported that PSOP showed no antagonism of epinephrine, acetylcholine or histamine)

Increases blood pressure

Increases heart rate

Increases respiration

- Increases pupillary diameter
- Increased body temperature
- Increases blood sugar levels
- Increases spinal reflexes
- Increases hexobarbital induced sleeping time (Gessner et al. 1960 found it also increased the toxicity of the barbiturate as 11 out of their 21 animals died within an hour of the barbiturate administration)

Activation of EEG

- Decreases reaction time
- Decreases motor activity
- Decreases isolation-induced fighting behavior

Contraction of nictitating membrane

Despite largely showing responses and effects qualitatively similar to PSOH, PSOP also showed some clear differences.

In comparison to psilocin, psilocybin had only half the pressor activity, 18% as much serotonin antagonism, and only 3% as much pyrogenic activity.

Interestingly, despite the 4-acetate, the 4-phosphate and the 4-benzoic acid esters of psilocin being physiologically active, the sulfate ester was found to be inactive.

[When comparing bufotenine to ITS phosphoryloxy ester, Cerletti found it increased the suppressive effects on knee jerk response (unlike the 4-substituted alkaloids mentioned above, bufotenine shows a short-lived inhibition of this response rather than a lengthy increase of it, as is characteristic of psilocin), it showed trivial serotonin antagonism (less than 1% as much as psilocin) in contrast to bufotenine's serotonin-like action, it had no pyrogenic activity (bufotenine has trivial pyrogenic effects; 1% as much as psilocin) and 33% more pressor activity than bufotenine (6% more than psilocin].

See also the comments on pages 231-235 concerning tolerance, cross-tolerance and synergy with other drugs.

Hollister et al. 1960 reported:

Significant elevation of blood pressure in 3 out of 16 subjects (27 trials total)

Dilation of pupils

- Increase in deep tendon reflexes in 19 trials (often clonic in character)
- Incoordination judged to be more subjective than objective

Reduction in excreted urinary inorganic phosphorus Reduction in total circulating eosinophils No remarkable change in SGO-T titer No remarkable change in serum cholesterol No remarkable change in serum alkaline phosphatase No remarkable change in electroencephalogram In one subject, after receiving divided increasing doses

for 21 straight days, a 0.203 mg/kg dose had no effect.

PSOH levels & Pharmacokinetics: (after administration of PSOP to humans)

Parameter	Route		
	Oral	IV	
Dose (Av)	0.224 mg/ kg	1 mg	
	(~15 mg)		
T _{max} (Av.)	105 min	1.9 min	
max	(85-180)	(0.7-2.5)	
C _{max} (Av.)	8.2 ng/ ml	12.9 ng/ ml	
max	(4.8-12.3)	(7.1-23.1)	
T _{1/2} (Av.)	163.3 min	74.1 min.	
/*	(106.0-272.2)	(49.8-97.5)	
Bioavailability	52.7% (Av.)	NA	
(Absolute)	(29.7-68.6)		
Hasler et			

Pharmacological overview:

Cerletti 1959 Delay *et al.* 1958 & 1959 Gessner et al. 1960 (rats excrete ~11% unchanged) Hofmann 1971 Hollister 1961 Ott 1996 Weidmann *et al.* 1958

Many pharmacological, physiological, psychopharmacological, & therapeutic reports exist : (A very

few of the many)

See Passie 1997 for *many* more. Aguilar 1963 Aldhadeff 1962 & 1962a & 1962b Alnaes 1965 & 1967 Clark 1967/1968 Clark 1970 Cwynar & Rydzynski 1966 David & David 1961 Delay *et al.* 1958a & 1958b & 1959a & 1959b & 1959d & 1959e & 1963 Duche 1961 Eberle & Leuner 1970 Eberle 1973



4-Phosphoryloxy-DMT

Fernandez-Cerdeno & Leuner 1965 Fisher 1963 & 1965 Grossbard 1989 Hasler et al. 1997 Hebbard & Fischer 1966 Heimann 1962 Horita & Weber 1962 Holfeld 1961 Isbell 1959 Kristensen 1963 Laatsch 1996 Leary 1962, 1964 & 1969; Leary et al. 1963, 1964 & 1965; Learv & Metzner 1968 Leuner 1963 & 1967; Leuner & Holfeld 1962 Malitz et al 1960 Martindale & Fischer 1977 Massoni & Lebensohn 1964 Pahnke 1967 Passie 1995a & 1995b Quetin 1960 Rinkel et al. 1960 Roquet & Favreau 1981 Roquet et al. 1975 Rydzynski & Gruszczynski 1978 Rydzynski et al. 1968 Schultz-Wittner 1989 Sercl et al. 1961 Sherwood et al. 1982 Sjoberg 1965 Stevenin & Benoit 1960 & 1962 Unger 1963 & 1964 Vernet 1960 Volterra 1967 Wolbach 1962



Psilocybe semilanceata (above) Photo by Dr. P. C. Hickey (below) Photo by JW Allen



Rick Strassman is either planning or conducting more recent pharmacological evaluations with psilocybin but this work is neither completed nor published.

Metabolism: PSOP is primarily converted to PSOH and 4-HIAA in humans. Hasler *et al.* 1997 Metabolism & excretion in rats: Gessner et al. 1960 We have locate nothing meaningful concerning human excretion (Sticht & Kaferstein found any excreted PSOH was largely as the glucoronide. See p. 62)

Toxicity:

Cerletti 1959 reported that giving psilocybin IV to mice up to 200 mg/ kg produced no deaths and only a few animals died after 250 mg/ kg/ iv

> Psilocybe atlantis lower left: Atlanta, Georgia Photo by Craig

4-Phosphoryloxy-DMT

- Delay *et al.* 1959c reported 200 mg/ kg/ intravenous as the LD50 in mice (i.e. only ~30% as toxic as intravenous LSD in their studies.)
- No real idea where the discrepancy arises but a logical GUESS would be that different times of day were chosen for the administrations.
- Gessner et al. 1960 reported that 1 of their rats given 100 mg/ kg/ ip died 10 days later with similar symptoms as are noted under bufotenine below. It seems likely the death did not involve the drug

LD₅₀

275 mg/ kg/ iv/ mouse 420 mg/ kg/ ip/ mouse Merck 9th 285 mg/ kg/ iv/ mouse 280 mg/ kg/ iv/ rat 12.5 mg/ kg/ iv/ Rabbit Usdin & Efron 1979 cited Sandoz

It should be noted that an oral LD50 should be expected to be higher still and a fatal dose from the Psilocybin present in a mushroom would require more than a person could physically eat, even if using the most potent species known.

Interestingly, psilocybin appears to be one of the *least* toxic of all the major hallucinogens. As Schultes & Hofmann put it: psilocybin is 2.5 times less toxic than mescaline and 50 times more potent.

Gessner *et al.* 1960 found 100 mg/kg/ip caused no deaths, with recovery after several hours, but one rat died 10 days later from complications thought linked.

Despite the fact that the active hallucinogenic components of mushrooms are moderately nontoxic this should not be extrapolated to mean that the mushrooms themselves are necessarily safe in large amounts.

Allergic reactions have been reported in some people (Horner *et al.* 1995) and the presence of other more toxic components cannot be ruled out (Ott 1996) so prudence and moderation are always recommended with fungal ingestion.

This is especially true with those that lack both a detailed analysis AND a history of safe human use.

It should also be remembered that fatal misidentifications are quite possible and NO ONE should ever eat ANY mushroom UNLESS they are absolutely certain of its identity.

This is true as well for dried mushrooms offered for sale. This author has encountered unidentified but clearly **non***cubensis* specimens within street samples of dried field collected mushrooms that were primarily *cubensis* and sold as such.

If the reader ever has **any** doubt **DO NOT EAT THAT MUSHROOM!** "When in doubt, toss it out"

The most reliable method for obtaining mushrooms is to cultivate them yourself. The only real (and very serious) danger that MOST people taking this approach will face is being arrested if noticed to be doing so.

It is also important to remember that, despite the fact psilocybin is unlikely to result in death at high dosages, it can cause people intense psychological distress and, like ALL of the major hallucinogens, has the potential for producing severe mental problems for mentally ill or otherwise unstable individuals; usually of brief duration but sometimes more persistent.

Of course, I did like Jeff Rense's admonition to young people not to eat these mushrooms because they may realize just how much of their potential that modern society has cheated them out of. While perhaps this may in fact **be** a quite distressing discovery, NOT knowing scarcely seems a better choice.

Toxicology review:

Sax 1984 cited 1975(?) J Med. Ass. Thailand 58(12) 623

See:

Merck 9th: Entry #7712, pages 1027-1028 Ott 1996: Entry #39, page 449 [Merck 11th: 7942; Merck 12th: 8111]

Shulgin 4 Shulgin 1997: Entry #18 page 468-473

Usdin & Efron 1979: Entry #427, page 138

Aeruginascin

Not positively identified structurally.

Possible identity is thought to be the methylphosphate ester of psilocin. (Ott 1996 cited Gartz 1992a)

Soluble in methanol.

Assays:

Colorimetric reagents:

Ehrlich's : Reddish-violet (Similar to the initial color of baeocystine or psilocybine but not turning blue-violet as those two do) Gartz 1989d

TLC:

Rf 0.12 in *n*-Butanol-Water-*i*-Propanol (8.5:2:1) on silica gel. Gartz 1989d reported to effectively separate from Psilocybin & Baeocystin

UV:

Shows strong absorption at 267 nm similar to Psilocybin Gartz 1989d

Isolation (via HPLC & preparative TLC): Gartz 1989d

Activity:

Its presence is said to add an "*always euphoric*" mood to the mushrooms. Gartz 1989d

Occurrence of Aeruginascin:

Inocybe aeruginascens Babos

Gartz 1987b: occurrence noted; Gartz 1989d: 0.14-0.35%

- Comprises a substantial proportion of the alkaloids.
- These mushrooms stain greenish (as do a fair number of other psilocybin containers). Ott 1996

Aeruginascin

Trout's Notes FS-X7

Note:

The methylester of psilocybin can be produced by direct methylation with diazomethane. It was synthesized by Hofmann as part of his total synthesis and structural elucidation of Psilocybin

C 70.8%, H 6.8%, N 9.0%, P 9.9%

C₁₂H₁₆ON

Hofmann et al. 1958

1-Methyl-psilocybin

1-Methyl-4-phosphoryloxy-N,N-dimethyltryptamine; 1-Methyl-psilocybin; CMY (Sandoz); CMY-16 (Sandoz)



Assorted dried *Psilocybe* species Photos thanks to RKundalini & friends requesting anonymity

WLN: T56 BNJ B D2KH FOPQO&O Hayward: 6R3(OP(OVQ)Y5L(CCN+HM2)=LNMY Usdin & Efron 1979

 ${\rm C}_{13}{\rm H}_{19}{\rm N}_{2}{\rm O}_{4}{\rm P}$

MW 298.3 Troxler et al. 1959

C 52.3% H 6.4% N 9.4% P 10.4%

Free base:

mp 255-257° (from methanol) Troxler et al. 1959

Sandoz once provided as 1 ml ampuls containing 3 mg/ ml. Scigliano 1968

Assays:

Colorimetric reagents:

Keller: gradually becoming bright Blue-green. Van Urk: Negative Troxler *et al.* 1959

Synthesis: Troxler et al. 1959

Activity:

- Hallucinogenic activity claimed by Usdin & Efron 1979; citing Scigliano 1967 who only noted it was an experimental research compound offered by Sandoz.
- Compare the following with comments under 1-methylpsilocin.
- Cerletti *et al.* 1968 reported that, in comparison to psilocin, there was an abolishment of any effects on spinal reflexes (based on knee jerk response), 44% as much serotonin antagonism and 30% of the pressor activity of psilocin.
- Supposedly has seen clinical trials but I have been thusfar unable to locate even a single account.
- Methylation of the one position in 5-MeO-DMT produces a compound with "only mild "stimulant" (amphetamine-like)" effects at 10 mg/kg. in rats. (Smythies et al. (1970) It too appears to lack any human evaluation.

See:

Usdin & Efron 1979 Entry #373, page 123

CZ 74

4-Hydroxy-N,N-diethyltryptamine (Sandoz);
3-[2-(Diethylamino)ethyl]-1H-indol-4-ol;
3-[2-(Diethylamino)ethyl]-4-indolol;
4-Indolol, 3-[2-(Diethylamino)ethyl];
N,N-Diethyl-4-hydroxytryptamine;
Tryptamine, N,N-diethyl-4-hydroxy;
4-Hydroxy-N-diethyltryptamine; diethyl homologue of psilocin;
4-HO-DET; 4-OH-DET; CZ 74; CZ-74

(Sandoz); HT (This is far more commonly applied to serotonin in the literature)

WLN: T56 BMJ D2N2&2 FQ Hayward: 6R3RQY5L(CCN(CM)2)=LNHY Usdin & Efron 1979

Not scheduled: Ott 1996

 $C_{14}H_{20}N_{2}0$

MW 232.3 Troxler *et al.* 1959 MW 232.45 Ott 1996

C 72.4%, H 8.7%, N 12.1%, O 6.9% Troxler *et al.* 1959 C 72.34%, H 8.67%, N 12.1%, O 6.88% Ott 1996

Free base:

mp 103-104° (White crystals room ethyl acetate/ hexane) Discolored rapidly in presence of air. Best stored in inert atmosphere at –30°) Shulgin & Shulgin 1997 mp 104-105° (crystals from acetone) Ott 1996

mp 104-106° (crystals from ethyl acetate) Troxler *et al.* 1959 mp 105° Gartz 1989c

Distilled 170-180° at 0.001 mm. Troxler et al. 1959

Free base:

Soluble in chloroform, tetrahydrofuran Shulgin & Shulgin 1997

Once provided by Sandoz as 1 ml ampuls containing 3 mg/ ml. Scigliano 1968

Assays:

Colorimetric reagents:

Ehrlichs: Gartz 1989c Keller: Green-blue Van Urk: Violet Troxler *et al.* 1959

TLC:

Rf 0.27 in Butanol-Acetic acid-Water-*i*-Propanol (8:2:5:1) on silica gel

Gartz 1989c

Rf 0.45 in *n*-Propanol-Acetic acid-Water (10:3:3) on silica gel. Gartz 1989c



Psilocybe cyanescens Photo by Dr. P. C. Hickey

UV:

λmax (MeOH) 223, 260, 267, 282, 293 nm Gartz 1989c

Synthesis:

Troxler *et al.* 1959 Shulgin & Shulgin 1997 Gartz 1989c used alkaline phosphatase to cleave CEY 19 into CZ 74.

Occurrence:

Psilocybe cubensis (Earle) Singer

Gartz 1989c isolated up to **3.3%** CZ 74 after feeding the mycelia with DET.

It is **NOT** normally present in this mushroom. Interestingly, NO DET was found remaining in the mycelia or present in the mushrooms.

Caps were smaller than usual and instead of bruising dark blue they bruised blue-green.

Activity:

Clearly a hallucinogenic material. Leuner & Baer 1965, Baer 1967

Psychoptic in same range as psilocin Ott 1996 citing Leuner & Baer 1965.

Dose: 10-25 mg oral Shulgin & Shulgin 1997

Duration: 4-6 hours Shulgin & Shulgin 1997

- Average duration 3.5 hours; minimum 1 hr 20 minutes to a maximum of 6 hours.
- Duration differed based on both the dosage AND the individual.

No discernible difference between CEY 19 and CZ 74 was observed (in 80 sessions involving 36 naive subjects) All administrations were via intramuscular injection.

Leuner & Baer evaluated 0.05-0.26 mg/kg with 0.20 mg/kg considered the dose producing average effects. (High dose was 40 mg) Leuner & Baer 1965

Baer 1967 gives more or less the same account but states most doses lasted 3-4 hours and gave their high value as 0.28 mg/ kg/ im.

He also noted that 14% of the 80 cases of administration had "*cosmic mystic experiences*" but that it produced NO effects in another 14%.

While the *majority* of those having cosmic-mystic experiences were theologically inclined it is also interesting that few of them were not.



Psilocybe subcubensis (Thailand) Photo by JW Allen

Receptor site specificity:

High affinity for $5HT_{1A}$ (IC₅₀ 1370 ± 472 nM), 5- HT_{2A} (IC₅₀ 14 ± 4 nM) & 5- HT_{2C} (IC₅₀ 680 ± 50 nM) McKenna *et al.* 1990

Physiological, psychological or therapeutic studies and/or applications:

Alnaes 1965 & 1967 Baer 1967a & 1967b Leuner & Baer 1965 Leuner 1967 & 1968 Passie 1995a & 1995b Schultz-Wittner 1989 See Passie 1997 for more.

See:

Ott 1996 Entry #6, page 432 Shulgin & Shulgin 1997 Entry #16 pages 461-465 Usdin & Efron 1979 Entry 378 (page 124)

4-Acetoxy-DET

4-Acetoxy-N,N-diethyltryptamine; 3-[2-(Diethylamino)ethyl]-1H-indol-4-ol acetate ester; 3-[2-(Diethylamino)ethyl]-4-indolol acetate ester; N,N-Diethyl-4-acetoxytryptamine; Tryptamine, N,N-Diethyl-4-acetoxy; 4-Acetoxy-DET; 4-AcO-DET; CZ 74 acetate ester; "Aces"

WLN: T56 BMJ D2N2&2 FOV1 Hayward: 6R3R(OCVM)Y5L(CCN(CM)2)=LNHY Extrapolated from others here

Not scheduled

 $C_{16}H_{22}O_2N_2$

MW 274.3 (Approx. extrapolated from other values)

Free base:

As encountered commercially has been a beige powder with a fairly bitter taste. Insoluble (or poorly soluble?) in water, Soluble in dilute acids, alcohol and in ginger ale. Oxidizes darker to brown unless kept dry & frozen. When exposed to heat and moisture forms a dark brown goo.

Hydrochloride rapidly decomposes in solution. Mostly from Toad 2001

Activity:

Hallucinogenic. Roughly equipotent to CZ 74 with same duration & effects. Toad 2001 **Dose:** 10-25 mg oral Shulgin & Shulgin 1997 For injection, smoking or insufflation we would suggest starting low (10 mg; perhaps 5 mg for someone lacking any experience with it) then increasing the dosage (by 2-5 mg) only as the effects and familiarity develops.

Duration: 4-6 hours Shulgin & Shulgin 1997

See comments under CZ 74

Oral doses of 22 mg or less usually have produced a roughly 2 hour peak with a rapid coast down. 25 mg or above also showed a 2 hour peak but with discernible effects that can persist another 4 hrs.

Shows wide variability between individuals even with a given dosage. Duration can be as little as 90 minutes or several times that.

Very friendly in the 18-22 mg range.

Can cause depersonalization, possible ego-death and major boundary dissolving in some people above 25 mg.

See:

Shulgin & Shulgin 1997 Entry #16 pages 461-465 Toad 2001 Also comments on oxidation in Note F on page 226

Also comments on oxidation in Note F on page 226



Psilocybe cyanofibrillosa (East Bay area)



CEY 19

4-Phosphoryloxy-N,N-diethyltryptamine (Sandoz); 3-[2-(Diethylamino)ethyl]-1H-indol-4-ol dihydrogen phosphate ester;

3-[2-(Diethylamino)ethyl]-4-indolol, phosphate ester; N,N-Diethyl-4-phosphoryloxytryptamine; Tryptamine, N,N-Diethyl-4-phosphoryloxy; 4-Indolol, 3-[2-(diethylamino)ethyl], phosphate ester; 4-Phosphoryloxy-ω-N,N-diäthyltryptamin (Sandoz); 4-Hydroxy-N-diethyltryptamine-O-phosphate; diethyl homologue of psilocybin; 4-HO-DET phosphate ester; 4-Phosphoryloxy DET; CZ 74 phosphate ester; 4-OP-DET; CY 19, CEY 19; CEY-19 (Sandoz); PT

WLN: T56 BMJ D2N2&2 FOPWQ Hayward: 6R3R(OPV2Q)Y5L(CCN(CM)2)=LNH Usdin & Efron 1979

Not scheduled: Ott 1996

 $C_{14}H_{21}N_2O_4P$

MW 312.3 Troxler *et al.* 1959 MW 312.31 Ott 1996

C 53.8%, H 6.8%, N 9.0%. P 9.9% Troxler *et al.* 1959 C 53.84%, H 6.78%, N 8.97%, O 20.49%, P 9.92% Ott 1996

More stable than CZ-74 when exposed to air. Shulgin & Shulgin 1997

Free base:

mp 260-263° (from methanol) Troxler *et al.* 1959 mp 261-263° Gartz 1989c

Once provided by Sandoz as 1 ml ampuls containing 3 mg/ ml. Scigliano 1968

Assays:

Colorimetric Reagents for CEY 19: Ehrlichs: Red-violet Gartz 1989c Keller: Blue-violet Van Urk: Pinkish-brown Troxler *et al.* 1959 TLC: Rf 0.10 in Butanol-Acetic acid-Water-i-Propanol (8:2:5:1) on silica gel Rf 0.17 in *n*-Propanol-Acetic acid-Water (10:3:3) on silica gel Gartz 1989c

UV:

λmax (MeOH) 221, 267, 280. 290 nm Gartz 1989c

Synthesis:

Troxler *et al.* 1959 Shulgin & Shulgin 1997

Occurrence:

Psilocybe cubensis (Earle) Singer

Gartz 1989c isolated **0.01-0.8%** CEY 19 after feeding the mycelia with synthetic DET.

CEY 19 is **NOT** normally present in this or any other mushroom but can be created by the mushroom.

NO DET was found in either the mycelia or the mushrooms. Caps were smaller than usual and bruised blue-green instead of dark blue.

Activity:

Clearly a hallucinogenic material. Leuner & Baer 1965, Baer 1967, Psychoptic in same range as psilocybin. Ott 1996 cited Leuner & Baer 1965

Dose: 10-25 mg oral Shulgin & Shulgin 1997

Duration: 4-6 hours Shulgin & Shulgin 1997 See more detailed comments above under CZ 74 [See also comments made under 4-Acetoxy-DET & under DET]

Pharmacological effects:

Cerletti *et al.* 1968 determined it had increased spinal reflexes, such as knee jerk response, around 11% as much antagonism of serotonin activity, and showed 84% as much pressor activity, as psilocin.

See:

Ott 1996 Entry #7, page 433. Shulgin & Shulgin 1997 Entry #16 pages 461-465. Usdin & Efron Entry #372, page 122.



Unidentified *Psilocybe* sp. Bay area Photo courtesy of James Edmond



Psilocybe subaeruginosa Photo by Snu Voogelbreinder



Psilocybe cubensis Photo (above) by Ringworm (SE, USA) Photo (below right) by JW Allen (Thailand)





Psilocybe azurescens (left & above) Photos by Dr. P. C. Hickey







Psilocybe cyanescens Photo (above) by JW Allen

Psilocybe cyanescens Photo (left) by Dr. P. C. Hickey



Psilocybe liniformans var. americana Photo by JW Allen



Assorted *Psilocybian* species (Thailand) Photo by JW Allen

Trout's Notes on Tryptamines



Unidentified frog napping underwater at the Audubon Aquarium of the Americas (Photo by a friend)

The 5-Substituted Tryptamines





A factory sleeps.



Phalaris aquatica (Pt. Reyes, CA) above





Desmodium gangeticum right-hand column





Brominated Tryptamines

5-Bromotryptamine

5-Bromo-1H-indole-3-ethanamine, 9CI; 5-Bromo-3-(2-aminoethyl)indole

C₁₀H₁₁BrN₂ MW 239.114

Southon & Buckingham 1989 Entry B-00186: page 159 cited Djura 1980

5,6-Dibromotryptamine

5,6-Dibromo-1H-indole-3-ethanamine, 9CI; 5,6-Dibromo-3(2-aminoethyl)indole

CA Reg. No: [41115-69-9]

$$C_{10}H_{10}Br_2N_2$$

MW 318.010

mp 110-120°

MS & NMR Van Lear et al 1973

Alkaloid from Caribbean sponge *Polyfibrospongia maynardi* Hyatt

Shows antibiotic activity against gram negative and gram positive bacteria in vitro but not in vivo. Van Lear *et al.* 1973

Southon & Buckingham 1989: Entry D-00199

5,6-Dibromo-N-methyltryptamine

C₁₁H₁₂Br₂N₂ MW 332.037

CA Reg. No: [41115-68-8]

mp. 132-134°

MS & NMR Van Lear et al 1973

Alkaloid from Caribbean sponge *Polyfibrospongia maynardi* Hyatt

Shows antibiotic activity against gram negative and gram positive bacteria *in vitro* but not *in vivo*. Van Lear *et al.* 1973

Southon & Buckingham 1989: Entry D-00199

5-Bromo-DMT

5-Bromo-N_b,N_b-dimethyltryptamine; 5-Bromo-N,N-Dimethyl-1H-indole-3-ethanamine, 9CI; 5-Bromo-3-(2-dimethylaminoethyl)-indole; Tryptamine, 5-Bromo-N,N-dimethyl-.; 5-Br-DMT

C₁₂H₁₅BrN₂ MW 267.168

CA Reg. No: [17274-65-6] Entry #B-00186 Southon & Buckingham 1989

mp 90-92° From methanol. Tymiak *et al.*mp 98-99° Crystals (MeOH aq.) Djura *et al.*Soluble in ethanol, ethyl acetate Djura *et al.*Soluble in methylene chloride Debitus *et al.*

UV:

λmax (MeOH) 225 nm (ε 39000), 285 (4400), 305 (2700) Djura *et al.* 1980 λmax (MeOH) 227 nm (ε 24000), 282 (5500) Tymiak *et al.* 1985

IR:

Djura *et al.* 1980 (CHCl₃): 3500-3200, 1475 cm⁻¹ Tymiak *et al.* 1985 (CHCl₃): 3400, 3300-3100, 1480-1430 cm⁻¹

MS:

Djura *et al.* 1980: m/e 268, 266 (5%), 188 (10%), 58 (100%)

GC-MS: Tymiak et al. 1985

NMR:

Djura *et al.* 1980 Tymiak *et al.* 1985

Synthesis:

Tymiak et al. 1985

Isolation:

Djura *et al.* 1980 Tymiak *et al.* 1985

Occurrence:

Smenospongia aurea [voucher 77-107] collected by SCUBA at Glover and Light House Reefs, Belize. (Only found in one of two specimens collected)

Djura *et al.* 1980 noted that the brominated tryptamines did not occur in all specimens examined.

It was postulated that they might be produced by symbionts that cannot live in all of the environments tolerated by the sponges.

Eudisoma fragum

A sea-squirt (Polycitoridae) from New Caledonia Yielded 0.02% dry wt Debitus *et al.* 1988

5-Br-DMT

Djura *et al.* 1980; examined two specimens of *Smenospongia aurea* (AKA *Aplysina aurea*), one of which was found to contain 5-bromo-DMT to the extent of 0.68% by dry weight while the other specimen did not contain 5-bromo-DMT and were distinguished by instead containing 8-epichromazonarol. The two compounds did not co-occur in any given specimen.

All sponges were collected in the Caribbean [at Glover and Lighthouse Reefs, Belize], using SCUBA, at minus 20 meters.

Sponges were homogenized and Soxhlet extracted with ethanol. After the solvent was removed the residue was partitioned between ethyl acetate and water. They used two 250 ml portions of ethyl acetate and 100 ml of water. After drying the ethyl acetate over sodium sulfate it was evaporated to a brown oil.

A column of silica gel separated the alkaloids. The series of elutants started with Hexane with 30% ethyl acetate proceeding through ethyl acetate and ended up with methanol in ethyl acetate. Ethyl acetate eluted the brominated tryptamines. Both were recrystallized from aqueous methanol.

In several cases they started elution with hexane and followed with ether before ethyl acetate. In all cases ethyl acetate eluted the two tryptamines.

Tymiak *et al.* 1985 found BOTH 5-bromo-DMT and 5,6-dibromo-DMT to be present in a sponge originally identified as *Aplysina lacunosa* but which was later reevaluated to be *Smenospongia aurea*. Concentrations were not reported.

The sponge was collected by SCUBA at -3 to -7 meters at Turneffe Island, Belize; 17°11'18"N, 87°55'36"W.

[All other samples of *Aplysina* spp. including *A*. *lacunosa* showed a wide variety of other alkaloids to be present while this specimen showed only two bands prompting their reevaluation and analysis.]

Fresh material was (Soxhlet) extracted with ethanol. Residue was partitioned between ethyl acetate and water. Ethyl acetate dried over sodium sulfate and evaporated. Crude extract chromatographed on silica gel and eluted with ethyl acetate. Crystallized from aqueous methanol.

Recovered 0.63%

Hydrogenation of 5-Br-DMT and also 5,6-dibromo-DMT gave DMT. Djura *et al.* 1980

Simple tlc can rapidly differentiate useful sponges from other sponges.

5-Bromo-DMT is possibly active but we have been unable to locate any direct pharmacological data in humans.

Ho's evaluation of gramine analogs *suggests* that the 5-bromo compound *may* be more toxic than either the parent compound (DMT) or the 5-methoxy analog (5-MeO-DMT).

Note:

Marine organisms frequently contain materials toxic to skin or eyes. Exposure may be by touch or by the organism squirting liquid. When dealing with unknown organisms the assumption of toxicity should be automatic and protective clothing, gloves and eye protection be worn. See Wright 1999 for tips and advice.

5,6-Dibromo-DMT

N_b,N_b-Dimethyl-5,6-dibromotryptamine; 5,6-Dibromo-N_b,N_b-dimethyltryptamine; 5,6-Dibromo-N,N-dimethyltryptamine, 9CI; Tryptamine, 5,6-Dibromo-N,N-dimethyl-.

 $C_{12}H_{14}Br_{2}N_{2}$

MW 346.064

CA Reg. No: [72853-80-6] Entry # D-00199 in Southon & Buckingham 1989

mp 113-115° Crystals (aq. MeOH) Djura *et al.* 1980 and also Tymiak *et al.* 1985

Soluble in Ethanol, Ethyl acetate Djura et al. 1980

UV:

λmax (MeOH) 230 nm (ε 40100), 285 (4900), 300 (3300) Djura *et al.* 1980 λmax (MeOH) 207 nm (ε 9600), 232 (30800), 291 (6300) Tymiak *et al.* 1985

IR:

Djura *et al.* 1980 (CHCl₃): 3500-3100, 1450 cm⁻¹ Tymiak *et al.* 1985 (CHCl₃): 3400, 3300-3100, 1480-1430 cm⁻¹(? Same figures given for 5-Br-DMT)

MS:

Djura *et al.* 1980: m/e 348,346, 344 (5%), 290, 288, 286 (5%), 266, 264 (5%), 58 (100%)

NMR:

Djura *et al.* 1980 Tymiak *et al.* 1985

Isolation:

Djura *et al.* 1980 Tymiak *et al.* 1985

Occurrence:

Smenospongia echina (AKA Polyfibrospongia echina) contained 5,6-dibromo-DMT in a yield of 0.95% by dry weight. (Collected at Puerto Morelos, Mexico yielded 0.88%) Djura et al. 1980 Also in Smenospongia aurea

Also in Smenospongia au

5.6-DiBr-DMT

5-HO-Tryptamine

Activity: Antimicrobial activity: Tymiak et al. 1985

See comments under 5-bromo-DMT and under "*Hydrogenation*".

Serotonin

3-(2-Aminoethyl)-1H-indol-5-ol, 9CI; 3-(2-Aminoethyl)-5-hydroxyindole; 3-(α-Aminoethyl)-5-hydroxyindole; 5-Hydroxy-3-(α-aminoethyl) indole; 3-(2-Aminoethyl)-5-indolol; Antomoqua; DS substance; Enteramin; Enteramine; Hippophain; Substanz DS; Thrombocytin; Thrombetonin; Thrombotonin (Warner-Lamb, Geigy); Tryptamine, 4-Hydroxy-; 5-Hydroxytryptamine; 5-HT; 5HT; 5-OH-TPA [Creatinine sulfate: SCS, Antemovis (Vister)]

WLN: T56 BMJ D2Z FQ Hayward: 6RRRQRY5L(CCZ)=LNHY Usdin & Efron 1979

CA Reg. No: [50-67-9] NIOSH #NM 2450000 Southon & Buckingham 1989

Serotonin is not a controlled substance

C₁₀H₁,N,O

MW 176 Buckingham *et al.* 1982 MW 176.21 Merck 9th MW 176.218 Southon & Buckingham 1989

C 68.16%, H 6.86%, N 15.90%, O 9.08% Merck 9th

Free base:

Purification by sublimation 150°/ 10-4 mm yielding a pale brown glass. Harley-Mason & Jackson 1954
Pure base is colorless. Thewalt & Bugg 1972
Soluble in dilute acids and butanol.

Chloroform-Water Partition coefficient: 0.62 Gessner & Page 1962

Hydrochloride:

CA Reg. No: 000153980 Usdin & Efron 1979 mp 167-168° Soluble in water. Light-sensitive. Aqueous solutions stable at acidic pH. Merck 9th & Southon & Buckingham 1989

Creatinine sulfate complex

[971-74-4] NIOSH #NM 2550000 Plates+ 1 H₂O mp 214-216° dec. **O-Sulfate** [16310-20-6] C₁₀H₁₂N₂O₄S MW 256.276 Crystals (H₂O) mp 192-194° Southon & Buckingham 1989

Hydrogen oxalate.

Pale buff microcrystals recovered. Recrystallized from ethanol-ether (charcoal) as colorless crystals 195-197° dec. Harley-Mason & Jackson 1954

Oxalate:

Large plates mp 194-196° dec. Harley-Mason & Jackson 1954

Picrate:

Orange-red needles (from aq. MeOH) 196-197° dec. (sintering at 130°). Harley-Mason & Jackson 1954

Crystal structure of Serotonin picrate monohydrate: Large red prisms (monoclinic) Thewalt & Bugg 1972

Assays for Serotonin:

Aures *et al.* 1968 Bogdanski *et al.* 1956 Curzon & Green 1970 Gessner *et al.* 1960 Iskric *et al.* 1969 Miller & Maickel 1970 Modell 1964 Snyder *et al.* 1965 Thompson *et al.* 1970 von Redlich & Glick 1969 Welch & Welch 1969

Colorimetric reagents: See color reactions p. 146

TLC & PC:

Serotonin:			
<u>Solvent</u>	<u>Rf</u>	<u>Medium</u>	<u>Ref</u>
Acetone-i-Propanol-Water-Ammonia (0.880)			
(50:40:7:3)	0.63	Silica gel	(9)
Benzene-Methanol-5% Ammonium hydroxide			
(10:15:2)	0.18	Silica gel	(1)
	0.21		(4)
n-Butanol-glacial Acetic acid-Water			
(120:30:50)	0.48	Paper	(3b)
n-Butanol-glacial Acetic acid-Water			
(2:1:1)	0.55	Silica gel	(1)
n-Butanol-glacial Acetic acid-Water			
(2:1:1)	0.61	Silica gel-	
		Kieselguhr	
		(2:1)	(4)

<u>Solvent</u>	<u>Rf</u>	<u>Medium</u>	<u>Ref</u>
n-Butanol-Acetic a	icid-Wate	r	
(4:1:5)	0.46	Paper	(15)
	0.54	Paper	(2)
			(11)
<i>n</i> -Butanol-Acetic a			
(60:15:25)	0.52	Avicel	(3a)
	0.50	Cellulose	(3a)
	0.57	Paper	(3a)
<i>n</i> -Butanol-Acetic a	0.50	SilCel	(11)
(4:1:1)	0.58	Paper	(13)
(4.1.1)	0.58	Silica gel	(15)
n-Butanol-Acetone	-Diethyl:	-	
(70:70:14:35)	0.92	Avicel	(3a)
(, ,)	0.85	Cellulose	()
	0.85	Paper	
Butanol		- « _F • ·	
saturated with 3%	Ammonia		
	0.30	Kieselgel	(10)
Butanol-Ethyl acet	ate-water		
(70:60:25) [v/v]	0.15	Alumina	(10)
n-Butanol-Pyridine	e-Water		
(60:60:60)	0.66	Avicel	(3a)
	0.65	Cellulose	(3a)
	0.79	Paper	(3a)
	0.70	Paper	(3b)
Chloroform-Acetic			(0)
(65: 20: 10: 5)			(9)
Chloroform-Cyclob	iexane-co	onc. Ammoniu	m hydroxide
(20:10:1)	0.0	0:1:1	(A)
(lower phase) Chloroform-Metha	0.0	Silica gel	(4)
(9:1)	0.03	alk. Silica gel	(8)
Chloroform-Metha			(8)
(10: 4: 1)	0.36	Silica gel	(5)
Cyclohexane-Chlo		Silled ger	(5)
(1:1)	0.0	Silica gel	(4)
Ether-Methanol-25			
(17:2:1)	0.65	Kieselgel	(15)
Ethyl acetate-Meth	nanol-58%	6 NH ₄ OH	
(80:15:5)	0.18	Silica gel	(1)
Methanol		-	
	0.15	alk. Silica gel	(8)
Methanol-Acetic a			
		Silica gel	(11)
Methanol-Ammoni			
(100:1.5)	0. 20	Silica gel (8)
	0.24	6061	
	0.24	6060	
Methanol-Methyl			(10)
(1:1) Marmhalina	0.05	Alumina	(10)
Morpholine (0.1 M in Water)	0.67	Silica gel	(6)
Potassium chloride		Silica gei	(6)
(20% w/v)	0.34	Paper	(3b)
<i>n</i> -Propanol-1N Ace		1 aper	(50)
(3:1)	0.43	Avicel	(3a)
(5.1)	0.47	Cellulose	(3a)
	0.53	Paper	(3a)
n-Propanol-0.2N A			()))))))))))))))))))
(3:1)	0.62	Avicel	(3a)
. /	0.63	Cellulose	(3a)
	0.68	Paper	(3a)
n-Propanol-Ammo	onia	-	
(8:3)	0.48	Paper	(12)

<u>Solvent</u>	<u>Rf</u>	<u>Medium</u>	<u>Ref</u>	
n-Propanol-Ammo	<i>n</i> -Propanol-Ammonium hydroxide (1N)			
(5:1)	0.53	Paper	(15)	
	0.66	Paper/		
		Silica gel	(13)	
n-Propanol-5% An	nmonium	hydroxide		
(5:2)	0.63	Silica gel-	(7)	
	0.75	Kieselguhr	(4)	
		(2:1)		
Propanol-Ammon	ia			
(5:1) [v/v]	0.60	Cellulose	(10)	
	0.65		(11)	
i-Propanol-5% Am	imonium	hydroxide		
(5:2)	0.38	Silica gel	(1)	
i-Propanol-Ammo	nia	-		
(8:2)	0.48	Paper	(2)	
<i>i</i> -Propanol-Water-Ammonia (0.88)				
(200:20:10)	0.66	Avicel	(3a)	
	0.65	Cellulose	(3a)	
	0.61	Paper	(3a)	
	0.56	Paper	(3b)	
i-Propanol-Formic	acid-Wa	iter		
(40:2:10)	0.50	Avicel	(3a)	
	0.56	Cellulose	(3a)	
	0.63	Paper	(3a)	
Sodium chloride (8% aqueous w/v)-Acetic acid				
(200:2)	0.34	Paper	(3b)	
Sulfurous acid				
(0.1 M in Water)	0.81	Silica gel	(6)	

References:

- 1. Gupta et al. 1979 (Silica Gel 60F-254)
- 2. Kveder & McIsaac 1961
- 3a. Jepson 1960 (as creatinine sulphate)
- 3b. Jepson 1969 (as creatinine sulphate)
- 4. Leung et al 1965 (as creatinine sulfate)
- 5. McKenzie et al. 1975
- 6. Sanders & Bush 1967
- 7. Neal et al 1968
- 8. Phillips & Gardiner 1969: Silica gel was silica gel; 6061 was silica gel Eastman chromatogram 6061; 6060 was silica gel with fluorescence indicator Eastman chromatogram 6060; Alk. silica gel was pretreated with 0.1N sodium hydroxide
- 9. Smith & Seakins 1976: p. 146
- 10. Stijve 1979
- 11. Stijve et al. 1984
- 12. Taborsky & McIsaac 1964
- 13. Tyler & Gröger 1964
- 14. Tyler & Malone 1960
- 15. Wagner & Grevel 1982
- Welsch & Batty 1963 used 95% Ethanol-Ammonium hydroxide (95:5) (More: Erspamer 1955; Rodnight 1956)

DNS derivative:

- <u>Rf</u> <u>Solvent system</u>
- 0.11 Benzene-Triethylamine (4:1)
- 0.49 Chloroform-*n*-Butyl acetate (5:2)
- Huebert & Boulton 1979

Weir & Tyler 1963: quantitative chromatographic assay

Column chromatography:

Use of Sephadex ion exchanger for separation: Aures *et al.* 1968b Separation using a combination of DEAE-cellulose, Sephadex and Amberlite columns. Iskric *et al.* 1969

HPLC:

Kysilka & Wurst 1988 & 1989 Kysilka *et al.*Stijve *et al.*Wurst *et al.*

GLC:

GLC of HFB derivative: Benington *et al.* 1975 & Vessman *et al.* 1969

GC:

Verpoorte & Svendsen 1983 (relative retention times compared to tryptamine: p 156) Wurst *et al.* 1992

UV:

Absorbs under 254 nm UV (shows up as dark spot) λ max (MeOH): 275 (300 sh) nm. McKenzie *et al.* 1975

Fluorescence:

Like other 5-hydroxyindoles it fluoresce pink under 365 nm mercury lamp. Not sensitive but fairly specific.

Serotonin and ninhydrin in acetic acid under 365 nm UV is much more sensitive but not specific.

(Primary indoles give an intense greenish-blue fluorescence on paper.)

Udenfriend 1962 (also has fluorescence spectra (p. 81) & quantitative fluorescence assay (pp. 171-172).

von Redlich & Glick 1969 also has fluorometric assay. Activation: 305 nm; Emission: 355 nm (pH 7.4)

Gessner & Page 1962 Activation: 310; Emission: 360 nm (pH 7)

Activation: 295; Emission: 540 nm (pH 2)

Gessner et al. 1960

Faint blue fluorescence under 350 nm UV; absorbs under 254 nm UV.

Smith & Seakins 1976: p. 146.

Visible fluorescence in 3M HCl- λ max 554 nm.

UV fluorescence in 0.01M Tris buffer at pH 7.4- λmax 338 nm.

Chen 1968

Blue fluorescence: Kveder & McIsaac 1961

Fluorescence maxima: 340 nm.

Excitation maxima: 295 nm

Burnett & Audus 1994

Serotonin fluoresces at 550 m μ in 3N HCl: Activation maxima 295 nm

Dilute acid or neutral pH: fluoresces at 330 nm when activated at 295 nm.

Udenfriend et al. 1955b

Fluorescence spectrum of OPD-serotonin Thompson *et al.* 1970.

Assays using spectrophotometer:

Udenfriend et al. 1955a & 1955b

Bogdanski et al. 1956

The last of these presents a fluorimetric assay which is capable of quantitative estimations.

They found that serotonin is activated at 295 nm and emits fluorescent light which shows a maximum at 550 nm (in 3N HCl).

Their procedure can detect as little as $0.1 \ \mu g$ but requires $0.3 \ \mu g$ for an accurate assay.

Welsch & Batty 1963 used this for quantitative assays, reading at 540 nm when wavelength for excitation was near 300 nm (in 3N HCl).

They also reported that the presence of bufotenine could interfere with this assay.

Spectrofluorometric assay (in 3N HCl using 550 nm) Gillespie 1969.

IR:

Stoll et al. 1955 (graphic)

MS:

Couch & Williams 1972 MS of HFB derivative: Vessman *et al.* 1969

Synthesis:

Hamlin & Fischer 1951 Speeter et al 1951 & 1953

While synthetic routes exist, another route for obtaining serotonin would be to decarboxylate 5-Hydroxy-L-tryptophan (5-HTP). See Baxter & Slaytor 1972a

and Christenson et al. 1972

Occurrence:

Serotonin is widespread in the plant kingdom, Smith 1977 lists 38 species from 20 families of plants.

A very few instances of its reported occurrence:

Agaricaceae

Amanita citrina (*A. mappa*) Tyler & Gröger 1964 (German specimens) ID via chromatography.

Amanita porphyria Tyler & Gröger 1964 (German specimens) Identified chromatographically.

Copelandia cambodginiensis Merlin & Allen 1993

Copelandia cyanescens Allen & Merlin 1992a: 0.033 & 0.026% [Merlin & Allen 1993: observed]; Stijve & de Meijer 1993: 0.02% (MeOH) & 0.06% (Optimized).

Panaeolina foenisecii Stijve et al. 1984: HPLC & TLC; Stijve & de Meijer 1993: 0.22% (MeOH) & 0.50% (Optimized) Swiss; 0.25% Brazil; [5HTP was the major in both collections].

Panaeolus antillarum Allen & Merlin 1992a: 0.015%; Stijve & de Meijer 1993: 0.035%.

Panaeolus campanulatus Tyler & Malone 1960 & Weir & Tyler 1963 (1.2 mg per gm)

Panaeolus semiovatus Merlin & Allen 1993

Panaeolus sphinctrinus Merlin & Allen 1993

Panaeolus subbalteatus Merlin & Allen 1993; Stijve & de Meijer 1993: 0.058-0.097% [5HTP was the major].

Leguminosae

Albizzia julibrissin Durazz. (4.7 µg/ gm secondary pulvini; 2.7 µg/ gm laminae; 3.4 µg/ gm rachillae) Applewhite 1973

Serotonin

Mimosa tenuiflora (0.001% in stem-bark) Meckes-Lozoya et al. 1990

Phaseolus multiflora (0.6 μ g/ gm pulvini; 1 μ g/ gm laminae; 1 μ g/ gm petioles) Applewhite 1973

Prosopis juliflora Saxton 1965

Samanea saman (4 µg/ gm secondary pulvini; 2 µg/ gm rachillae; *nd* in laminae) Applewhite 1973

Pisum sativum (14 days light: $0.9 \ \mu\text{g}/\text{gm}$ of pulvini; 1 $\mu\text{g}/\text{gm}$ of stems; *nd* in laminae; 6 days dark: $0.6 \ \mu\text{g}/\text{gm}$ of hooks & internodes, *nd* in terminal buds) Applewhite 1973

Passifloraceae

Passiflora quadrangularis (1 µg/ gm of tendril; *nd* in laminae) Applewhite 1973

See also: Mears & Mabry 1971 Smith 1977b Udenfriend 1962: 173

Animals:

Serotonin is extremely widely distributed in the animal kingdom. We include only a few examples.

It is found in ganglia and nerve cords of some invertebrates (such as insects, molluscs, crustaceans, tunicates [Erspamer 1961], cockroaches [Martin & Downer 1989] and also horseshoe crabs [Roberts et al 1983]); outside of nerve tissues in many places, such as: the venoms of some amphibians, some reptiles, many arachnids including spiders, centipedes & scorpions, some bees & some vespids (earlier claim of presence in only social, not solitary wasps shown incorrect in Welsch & Batty 1963), & stinging caterpillar hairs, [Deulofeu & Rúveda 1971, Edery et al. 1978, Erspamer 1961, Erspamer & Vialli 1951, Green & Odell 1984, Janghi 1984, Kawamoto & Kumada 1984, Nakajima 1984, Piek 1984, Raskova 1971, Schenberg & Pereira Lima 1978, Welsch & Batty 1963, Welsch & Zipf 1966]; in the venom apparatus of some insects, mollusks & coelenterates [Erspamer 1961]; in the salivary glands of cephalopods such as Octopus vulgaris [Erspamer 1961, Raskova 1971:58]; Gastropods such as Murex & Conus [Raskova 1971 p. 52]; Coelenterates/Cnidarians (corals, sea fans, sea anemones & jellyfish) [Cimino & De Stefano 1978, Erspamer 1961, Raskova 1971: 23, Shulman et al. 1957]; Tunicates [Erspamer 1961]; Crustaceans [Erspamer 1961] See also Erspamer 1954 & Udenfriend 1962: 173

Arachnids:

- Found in some spider venoms (at low concentrations: often co-occurring with other bioactive amines such as histamine, GABA, glutamic acid and other amino acids):
- *Atrax robustus* (Sydney Funnel-web spider) Present in small amounts in the venom of the male (Venom of female has 5-methoxytryptamine) Male has the more toxic venom despite the female secreting greater amounts. Geren & Odell 1984

Lycosa erythrogantha

1.5-2% of dry venom wt. Tu 1977 **Pamphobetes nigriventor** Green & Odell 1984 **Pamphobetes tetracanthus** (Bird Spider Tarantula) Geren & Odell 1984 **Phoneutria fera** 0.5-2.7% of dry venom wt. Tu 1977 See also Schenberg & Pereira Lima 1978 Welsch & Batty

Cimino & DeStefano commented 5HT is found in higher amounts in terrestrial than aquatic amphibians. (citing Welsch & Zipf)

Animals:

Names followed by a plus sign(s) (+) are from Daly & Witkop 1971

+ 1-100 μg / gm of skin ++ 100-1000 μg / gm of skin

+++ 1-10 mg/gm of skin

Bufonidae

Bufo alvarius + 4.0-6.0 mg/ gm of nonglandular skin. Erspamer *et al.* 1965

Bufo americanus + Bufo arenarum ++ Bufo coccifer ++ Bufo berghei ++ Bufo bufo bufo + Bufo bufo formosus + Bufo bufo gargarizans + Bufo calamita + Bufo coccifer ++ Bufo cognatus ++ Bufo fernandezae ++ Bufo fowleri + **Bufo funereus** ++ Bufo granulosus + **Bufo haematiticus** +++ Bufo hemiophrys ++ Bufo kisolensis ++ Bufo luetkeni ++ Bufo major ++ Bufo marinus ++ Bufo marmoreus ++ **Bufo mauretanicus** ++ Bufo microscaphus ++ Bufo paracnemis ++ Bufo perplexus ++ Bufo pygmaeus + Bufo regularis +++ Bufo speciosus +++ Bufo spinulosus + Bufo terrestris +++ Bufo typhonius + Bufo valliceps +++ Bufo viridis + Bufo woodhousei +++ Atelopodidae Melanophryniscus moreirae +

Discoglossidae Bombina bombina + Bombina variegata ++ Discoglossus pictus ++ Leptodactylidae Cyclorana alboguttatus + Eleutherodactylus martinicensis + Lechriodus fletcheri+ *Leptodactylus americanus* ++ [*See note below] *Leptodactylus caligonus* ++ [*See note below] Leptodactylus curtus + [now considered a synonym of Leptodactylus labrosus] *Leptodactylus laticeps* ++ (Erspamer *et al.* 1964) Leptodactylus melanonotus + *Leptodactylus occidentalis* +++ [now considered a synonym of Leptodactylus melanotus] *Leptodactylus pentadactylus* ++ (Erspamer *et al.* 1964) *Leptodactylus rubido* + [now considered a synonym of Leptodactylus rhodonotus] Pleuroderma bufonina + Thoropa militaris + Rhinoderma darwinii ++ Hylidae Hyla arborea + Litoria aurea + Litoria caerulea ++ Litoria gilleni + Litoria gracilenta ++ *Litoria infrafrenata* ++ Litoria latopalmata + Litoria nasuta + Litoria lesueuri ++ Litoria pearsoniana + Litoria peronii ++ Litoria rothii + Pipidae Xenopus laevis + Ranidae Rana esculenta + Rana dalmatina + Rana japonica + Rana labrosa + [now Laliostoma labrosum] Rana latastei + **Rana madagascariensis** + [now Aglyptodactylus madagascariensis] Rana nigromaculata + Rana pipiens ++ Rana palustris + Rana rugosa ++ Rana sphenocephala ++ Rana sylvatica ++ Rana temporaria ++ For still more (toads, frogs & a salamander), see also: Cei et al. 1968, Deulofeu & Rúveda 1971, Erspamer 1954 and Roseghini et al. 1976 & 1986.

Serotonin is also found in blood GI tract, nervous system, spleen and elsewhere in vertebrates.

It is one of the major neurotransmitters in humans.

* Note: We were unable to locate these two names.

Reviews:

Borne 1994 Erspamer 1954 & 1961 Bradley *et al.* 1992

Activity:

Orally inactive: dosages to 1 gm.. Chilton *et al.* 1979 Sjoerdsma *et al.* 1958 found it inactive orally up to 50 mg even in presence of an MAOI. (But noted that infusions of serotonin are poorly tolerated.)

Pharmacological & physiological properties:

Potent vasoconstrictor. Freyburger et al. 1952

Serotonin has been reported to show anticholinesterase activity [Bhattacharya & Sanyal 1971 cited Langemann 1954; Erspamer 1954 has more references.]

5HT also shows radioprotective effects from X-radiation. Erspamer 1954

Erspamer 1954 discusses pharmacology, physiological properties, toxicology, biosynthesis and metabolism.

Metabolism was studied in Erspamer 1955.

Interestingly, both serotonin and serotonin receptors are highest in the gastrointestinal tract.

Pharmacology, metabolism & excretion in rats: Gessner *et al.* 1960

See also p. 224 in Tedeschi et al 1959.

Reviews of serotonin, its receptors & ligands:

Fozard & Saxena 1991

Glennon et al. 1991; Glennon & Dukat 1995

Hardman & Limbird 1996

Huang & Julius 1991

Olivier et al. 1997

Peroutka 1991 & 1997

Sanders-Bush & Canton 1995

Tolerance:

Produces short-lived desensitization to itself. Erspamer 1954

Pharmacological overview: See Erspamer's reviews.

There is a wealth of published papers as concerns the pharmacological, physiological & toxicological properties, drug interactions (it potentiates compounds as disparate as barbiturates and adrenaline), metabolism, and many other aspects of serotonin including its role in depression & both physical and mental health.

These alone would form a nice sized library.

Consult Erspamer's reviews and those included in our references for a small but sizable portion of them.

A comprehensive bibliography on serotonin would form a rather large book.

Toxicity:

Southon & Buckingham 1989 describe as "*Highly toxic*" and an experimental teratogen. Toxicity is enhanced by anaesthetics [& vice versa].

Death is due to respiratory failure. Erspamer 1954

Trout's Notes FS-X7

5-HO-MMT

Serotonin

LD₅₀

Base: 160 mg/ kg intravenous in mouse 750 mg/ kg intramuscular in mouse 868 mg/ kg intraperitoneal in mouse 30 mg/ kg intravenous in rat 117 mg/ kg subcutaneous in rat Usdin & Efron 1979 citing Barnes & Eltherington 1965 160 mg/ kg intravenous in mouse >868 mg/ kg subcutaneous in mouse 30 mg/ kg intravenous in rat ~117 mg/ kg subcutaneous in rat Freyburger *et al.* 1952 Hydrochloride: 81 mg / kg iv in mouse. 601 mg /kg sc in mouse

Mashkovsky & Arutyunyan 1964

Tedeschi *et al.* 1959 reported "*marked cyanosis followed by severe asphyxial clonic convulsions and death*" at 5 mg/ kg iv in rats.

5HT given at 0.05-0.1 mg/ kg in an anaesthetized animals (rabbit) causes immediate stimulation & respiratory arrest in 50% of them. Erspamer 1954

For more information see:

Merck 9th: Entry #8209 Southon & Buckingham 1989: Entry #H-00352

5-OH-N-Methyltryptamine

N-Methyl-serotonin;

3-[2-(Methylamino)ethyl]-1H-indole-5-ol; 5-Hydroxy-N-methyltryptamine; N-Methyl-5-HT; N-Methyl-5-hydroxytryptamine; MeSer; 5-HMT.

Freebase:

Amorphous Stoll *et al.* 1955 Soluble in acetone, methanol *etcetera*.

Hydrogen oxalate:

mp 153-156° Stoll et al. 1955

Assays:

Chromophoretic reagents: See pages 169-176

TLC & PC for N-methyl-5-hydroxytryptamine:

Solvent		Medium	Ref	
Acetone-i-Propa	nol-Water-	 Ammonium hydrox 	xide	
(50:40:7:3)	0.26	Paper	(6)	
n-Butanol-Acetic	acid-wate	er		
(4:1:1)	0.66	Paper	(5)	
(4:1:5)	0.47	Paper	(7)	
<i>n</i> -Butanol-glacial Acetic acid-Water				
(120:30:50)	0.56	Paper	(1)	
Butanol saturated with 3% Ammonia				
	0.50	Silica/ Kieselgur	(3)	
n-Butanol-Pyrid	ine-Water	-		
(60:60:60)	0.72	Paper	(1)	
Chloroform-Acetic acid-Methanol-Water				
(65:20:10:5)	0.32	Paper	(6)	

Methanol-Chloroform (latter with 1% NH4OH)				
(9:1)	0.08	Silica gel	(4)	
Morpholine				
(0.1 M in Water)	0.60	Silica gel	(2)	
aqueous Potassium	chloride			
20% (w/v)	0.38	Paper	(1)	
i-Propanol-Ammo	nia (880)	-Water		
(200:10:20)	0.89	Paper	(1)	
(16:1:3)	0.66	Paper	(7)	
n-Propanol-1N Ammonium hydroxide				
(5:1)	0.73	Paper	(5)	
Propanol-Ammonia				
(5:1)	0.80	Cellulose	(3)	
n-Propanol-Water				
(3:1)	0.53	Paper	(7)	
Sodium chloride (8% aqueous w/v)-glacial Acetic acid				
(200:2)	0.42	Paper	(1)	
		-		

References:

1. Jepson in Smith 1969 & 1960 (as oxalate)

- 2. Sanders & Bush 1967
- 3. Stijve 1979
- 4. Torres & Repke 1996
- 5. Tyler & Gröger 1964
- 6. Smith & Seakins 1976
- 7. Rodnight 1956

Fluorescence:

Absorbs under 254 nm UV (shows up as dark spot) Faint yellow fluorescence under 350 nm UV Smith & Seakins 1976: p. 146.

Udenfriend 1962 states that 5-Hydroxyindoles fluoresce pink under a 365 nm mercury lamp.

GC: Wurst et al. 1992

MS of TMS derivative: Narasimhachari et al. 1971

¹H NMR: Torres & Repke 1996

First isolated by Erspamer & Vialli 1951 & 1952

Synthesis: Stoll et al. 1955

Reported occurrences of 5-OH-MMT: Agaricaceae

Amanita citrina

Tyler & Gröger 1964 (German specimens) Identified chromatographically. Wurst et al. 1992: 0.0-0.039%

Amanita porphyria

Tyler & Gröger 1964 (same notes as under *A. citrina*) Wurst et al. 1992: 0.0 & 0.072%

Graminae

Hordeum vulgare

(roots of seedlings) Schneider & Wightman 1974 Leguminosae

Desmodium pulchellum

Ghosal & Mukherjee 1966 (In root Ghosal 1972a) *Anadenanthera colubrina* var. *cebil* (= *P. macrocarpa*) Iacobucci & Rúveda 1964 *Anadenanthera peregrina* Legler & Tschesche 1963

5-HO-MMT

5-HO-DMT

Animals: Names followed by a plus sign(s) (+) are from Daly & Witkop 1971 +1 - 100µg / gm of skin ++ 100-1000 μ g / gm of skin +++ 1-10 mg / gm of skin Bufonidae Bufo alvarius + (30-40 µg/gm dried skin. Erspamer et al. 1965) Bufo americanus + Identified by Erspamer 1954. Deulofeu & Rúveda 1971 (Also in Erspamer 1961) Bufo arenarum ++ (? listed in Erspamer (1961) Bufo bufo bufo (Identified by Erspamer (1954). Deulofeu & Rúveda (1971) (Also in Erspamer 1961) Bufo bufo formosus + Bufo bufo gargarizans + Bufo calamita + Identified by Erspamer 1954. Deulofeu & Rúveda 1971 Bufo canaliferus ++ Bufo coccifer + Bufo debilis + Bufo fernandezae ++ Bufo fowleri + Identified by Erspamer 1954. Deulofeu & Rúveda 1971 (Also in Erspamer 1961) Bufo gargarizans Identified by Erspamer 1954. Deulofeu & Rúveda 1971 Bufo granulosus + Bufo hemiophrys ++ Bufo ictericus + Bufo luetkeni + Bufo major ++ Bufo marinus ++ Identified by Erspamer 1954. Deulofeu & Rúveda 1971 Bufo mauretanicus (? listed in Erspamer 1961) Bufo microscaphus ++ Bufo paracnemis ++ Bufo pygmaeus ++ Bufo speciosus ++ Bufo terrestris + Bufo viridis + Identified by Erspamer 1959. Deulofeu & Rúveda 1971 (Also in Erspamer 1961) Bufo woodhousei ++ Atelopodidae Melanophrynscus moreirae + Leptodactylidae Cyclorana alboguttatus + (50-100 µg/ mg: Roseghini et al. 1976) Cyclorana cultripes (20 & 180 µg/ mg: Roseghini et al. 1976) Cyclorana platycephalus (0 & 80 µg/ mg: Roseghini et al. 1976) Leptodactylus melanonotus + Hylidae Litoria adelaidensis (75 & 140 µg/ mg: Roseghini et al. 1976) Litoria booroolongensis (50 µg/ mg: Roseghini et al. 1976) Litoria freycineti (65 µg/ mg: Roseghini et al. 1976) Litoria moorei (40-200 µg/ mg: Roseghini et al. 1976) Litoria peronii + (12 µg/ mg: Roseghini et al. 1976) Litoria pearsoniana (10 µg/ gm in one collection of 117 skins but 0 in 4 other collections totalling 811 skins: Roseghini et al. 1976) Nyctimystes disruptus (400 µg/ mg: Roseghini et al. 1976) Ranidae Rana temporaria +

Bufotenine

3-[2-(Dimethylamino)ethyl]-1H-indol-5-ol, 9CI; 3-(2-Dimethylaminoethyl)-5-hydroxyindole; 3-(2-Dimethylaminoethyl)-5-indolol; 3-(β-Dimethylaminoethyl)-5-hydroxyindole; N,N-Dimethyl-5-hydroxytryptamine; 5-Hydroxy-N,N-dimethyltryptamine; N,N-Dimethylserotonin; Mapine; Mappine; Cohoba; Bufotenin; 5-OH-DMTPA; BT. WLN: T56 BMJ D2N1&1 GQ Hayward: 6RRRQRY5L(CCNM2)=LNHY Usdin & Efron 1979: Entry #371 CA Reg. #000487934 [487-93-4] NIOSH #NM 2800000 Schedule 1 Drug (Federal Controlled Substances Act; registry number 7433) Shulgin & Shulgin 1997 C₁,H₁₆N,O MW 204.26 Merck Index 9th Edition MW 204.27 Bergin et al. 1968 MW 204.271 Southon & Buckingham 1989: page 162; Entry # B-00202 MW 204.30 Sax & Lewis 7th edition, Vol. 2. Page 1346; Entry #DPG109 C 70.56%, H 7.90%, N 13.72%, O 7.83% Free base: Transparent prisms (monoclinic) (Supplied by B.Holmstedt) Bergin et al. 1968 Density: 1.205 g/cm³ (Observed)/ 1.191 g/cm³ (Calculated) Bergin et al. 1968 Purification by sublimation 160°/ 10-4 mm. Harley-Mason & Jackson 1954 mp 123-124° From concentrated ethyl acetate. This was recrystallized several times. Their reference sample showed mp 146-147°. When low melting form was seeded with higher melting form and recrystallized it showed the higher melting point. Iacobucci & Rúveda 1964 mp 123-125° Dutta & Ghosal 1967 mp 125-126° (from acetone/ether) Barlow & Khan 1959 mp 130-131° (from Bufo viridis viridis). Jensen & Chen 1932 mp 138-140° Crystallized from ethyl acetate. Neuss 1964 mp 138-140° Prisms from ethyl acetate. Stoll et al. 1955 mp 138-140° Massive prisms from acetone. Troxler et al 1959 mp 146° Ghosal & Mukherjee 1964

mp 146.5° Bergin *et al.* 1968

- mp 146-147° Boit 1961 & Wieland et al. 1931
- mp 146-147° Speeter & Anthony 1954
- mp 146-147° Pachter et al. 1959
- mp 146-147° Stout prisms from ethyl acetate. Merck 9th

Bufotenine

mp 149° (recrystallized twice from ethyl acetate) Alvarez Periera 1957 bp_{0.1} 320° Southon & Buckingham 1989 & Merck 9th

Practically insoluble in water. Freely soluble in alcohol, less so in ether. Soluble in dilute acids and alkalis. Merck Ninth Soluble in ethanol, chloroform and ethyl acetate. Pachter *et al.* 1959 Soluble in butanol. Tyler & Gröger 1964

Chloroform-Water partition coefficient: 0.67

Gessner & Page 1962 [Serotonin = 0.62] 0.06 (apparent)

11.2 (corrected for ionization) Migliaccio et al 1981

Octanol-Water partition coefficient: 0.16 Migliaccio et al 1981

pKa 9.67 (N); 10.88 (OH) Migliaccio et al 1981

Flavianate:

mp 130-131°/ mp 131-133° Red needles from water. Deulofeu & Duprat 1944

Fumarate :

mp 228-230° dec. From ethanol. Barlow & Khan 1959

Hydrogen oxalate:

Needles (from MeOH) 89-90°. Stoll et al. 1955 Also Boit 1961

Mono-oxalate:

mp 93-94° Pink needles Shulgin & Shulgin 1997

Oxalate (unspec.):

mp 81-82°. Torres & Repke 1996 mp 93-94° (from ether-acetone) Wieland *et al.* 1931 [Same

mp as Handovsky 1920]

mp 178° Wieland *et al.* 1931 (Shulgin & Shulgin 1997 note that this may have been the bioxalate.)

Monopicrate:

Yellow crystals which change to a red modification at 120-140°, then mp 179-180° Merck 9^{th}

mp 177-178° Yellow crystals. Hoshino & Shimodaira 1935 and Hoshino & Shimodaira 1936

mp 177-178º Marini-Bettòlo et al. 1964

mp 177.5 ° Wieland & Wieland 1937

Yellow picrate from EtOH. mp 178°. (Eluted from Brockman alumina with CHCl₃-MeOH (99:1) Wassel *et al.* 1985

mp 178°. Both red (long needles) and yellow (short prisms) melting at this temperature.

Red picrate changing to yellow at 140°.

Wieland *et al.* 1931 (cited Handovsky as reporting 168°) Yellow picrate (from ethanol) mp 178° Ghosal *et al.* 1969

Yellow monopicrate (from water) mp 180°. Deulofeu & Duprat 1944

Picrate: (unspec.)

mp 176-178º Marini-Bettòlo et al. 1964

- Red needles (from MeOH) mp 177° dec. Harley-Mason & Jackson 1954
- mp 177° and mp 178° were reported from red needles; isolated from toads (recrystallized from methanolether) Jensen & Chen 1936

mp 178-179° Red crystals from methanol. Giesbrecht 1960 cited Stromberg 1954 as finding 176-170°

mp 179-180° Wieland & Motzel 1953

mp 174-175° (from seeds) mp 174-175.5° (from seed pods) Iacobucci & Rúveda 1964

mp 197° Jensen & Chen 1932

Dipicrate:

Red dipicrate mp 172-173°. Boiling in benzene produced yellow monopicrate mp 179°. Deulofeu & Duprat 1944

mp 174° Wieland & Wieland 1937

Red dipicrate mp 174°. Deulofeu & Duprat 1944

Red crystals from MeOH. mp 176-177° Merck 9th mp 177-178° Boit 1961

mp 175-177° Dutta & Ghosal 1967

mp 176-177° Dark red. Wieland & Motzel 1953

mp 177-178° Ghosal & Mukherjee 1964

mp 177-178° Red prisms from methanol. Hoshino & Shimodaira 1935 & 1936

Picrolonate:

mp 120-121° Small yellow prisms mp 110° Recrystallized from ethanol mp 120-121°. Deulofeu & Berinzaghi 1946. Also Boit 1961

mp 130-131° Jensen & Chen 1932

mp 183-184° (Both from experimental product and from reference material.) Iacobucci & Rúveda 1964

Methiodide:

C₁₃H₁₉IN₂O

Colorless crystals ("*drusen*") mp 209°. Wieland *et al.* 1931 mp 210° Iacobucci & Rúveda 1964 Colorless prisms; mp 210-211° Wieland & Motzel 1953 mp 210-211° (from ethanol) Barlow & Khan 1959 mp 213-215°. Deulofeu & Duprat 1944 Stout prisms from MeOH. mp dec. 214-215° Merck 9th Stout prisms mp 214-215° Stoll *et al.* 1955

Assays for Bufotenine:

Usdin & Efron 1979 cited: Clarke 1969 Udenfriend 1969 (See also Clarke's 1986)

Colorimetric reagents: See color reactions p. 147

TLC & PC: see Rf table pp. 169-176

Column chromatography:

Eluted from column of Alumina with Ethyl acetate by Pachter *et al.* 1959

Does not adsorb on Alumina so eluted with first Ethanol eluates. Erspamer *et al* 1964

HPLC:

Balandrin et al. 1978 Kysilka & Wurst 1988 & 1989 Kysilka et al. 1985 Stijve et al. 1984 Wurst et al. 1992

GLC:

Christian et al. 1975 GLC of HFB derivative: Benington et al. 1975

GC:

Audette et al. 1969: retention times for CHDMS and DEGS columns Verpoorte & Svendsen 1983 (relative retention times compared to tryptamine: p 156) Wurst et al. 1992 (UV vs ED)

UV:

 λ max 220, 265 nm (log ε 4.0, 3.7) Merck 9th λmax 224 nm (log ε 4.46), 278 nm (3.93), 294 nm (3.90) and 305 nm (3.52) Ghosal et al. 1969 λmax 225, 280, shoulder 303 nm (log ε 1.35, 3.83, 3.71) (EtOH) Stoll et al. 1955 λmax 277, 296 nm (log ε 3.74, 3.67) (0.1N HCl) Stoll et al. 1955 λmax 218, 276, 323 nm (log ε 4.37, 3.74, 3.65) (0.1N NaOH) Stoll et al. 1955 λ max 228, 278, 300, shoulder at 315 nm (log ε 4.35, 3.83, 3.70) (95% EtOH) DeBudowski et al. 1974 λmax 279, 301, 314 nm (reference material) 279, 301, 315 nm (isolated material) Fish et al. 1955 λmax [log ε]: 225 [4,35], 280 [3.83], (303) [3.71] Troxler et al 1959 λmax 273, 303 nm Marini-Bettòlo et al. 1964 Neuss (1964) λ 23, 277, 300 nm See also Sunshine 1981 Wieland & Motzel 1953 Fluorescence:

Spectrofluorometric assay (in 3N HCl using 550 nm): Gillespie 1969 Activation: 310 nm; Emission: 360 nm (pH 7.4) Gessner & Page 1962 Activation: 310; Emission: 360 nm (pH 7) Activation: 310; Emission: 360 nm (pH 2) Gessner et al. 1960 Fluorescence maxima:

Excitation: 300 nm; Emission 340 nm. Fish et al. 1955 Reported to have a weak violet fluorescence Culvenor et al. 1964 Absorbs under 254 nm UV (i.e. shows up as a dark spot). Faint yellow fluorescence under 350 nm UV. Smith & Seakins 1976: p. 146.

Media and pH of solvent will affect its fluorescence. Fluoresces at 540 nm with excitation at 295 nm using 0.1N HCl as solvent. Fluoresces at 340 nm with excitation at 300 nm using ethanol as solvent. [Perkin-Elmer MPF-2A fluorescence spectrophotometer] DeZan et al. 1971 See also William & Bridges 1964

IR:

DeBudowski et al. 1974 (in CHCl.) 3500, 3025, 2950, 2875, 2860, 2800, 1738, 1660, 1638, 1597, 1480, 1380 cm⁻¹ See also Neuss 1964 Stoll et al. 1955 Sunshine 1981

MS:

MS of TMS derivative: Narasimhachari et al. 1971 Räisänen & Kärrkkinen 1979

NMR:

DeBudowski et al. 1974 Migliaccio et al 1981 ¹H NMR: Torres & Repke 1996

X-ray powder data: See Neuss 1964

Structure:

Structure elucidated by Wieland in 1931

Synthesis:

Hoshino & Shimodaira 1935 & 1936 Harley-Mason & Jackson 1952 & 1954 Shulgin & Shulgin 1997: pp. 473-478, entry #19. Synthetic route on pages 473-474. Speeter & Anthony 1954 Speeter 1955 [U.S. pat. 2,708,197 (1955 to Upjohn)] Stoll et al. 1955 Troxler et al. 1959 Bufotenine (and sulfuric acid) are produced from bufoviridine when warmed with dilute HCl. Habermehl 1974

Another route would be to use the enzyme NMT (N-methyl -transferase) to convert serotonin to bufotenine. See Axelrod 1962 and Mack & Slaytor 1979

Bufotenine

Isolations:

First isolation was from *Bufo vulgaris*: Handovsky 1920 (BUT he proposed an incorrect structure)

From **Ch'an Su** (Chinese medicinal dried toad venom.) Jensen & Chen 1932

Also isolated (from *Amanita*) Wieland *et al.* 1934 & Wieland & Wieland 1937

First isolation with a correct structural identification of Bufotenine was from *Amanita citrina* by Wieland & Motzel 1953 *Anadenanthera peregrina* by Stromberg 1954

Bufo alvarius by Erspamer *et al.* 1967

Reported Occurrences of Bufotenine:

Agaricaceae

Amanita spp. (All low concentration)

Amanita citrina (Schff.) S.F.Gray (A. mappa (Batsch ex. Fr.) Ouelet)

Wieland & Motzel 1953 (European specimens) 0.045% (Stijve 1979 noted their inefficient procedure may have only recovered 10% of what was there.)

Catafolmo & Tyler 1961 (North American specimens) Tyler 1961 (North American specimens)

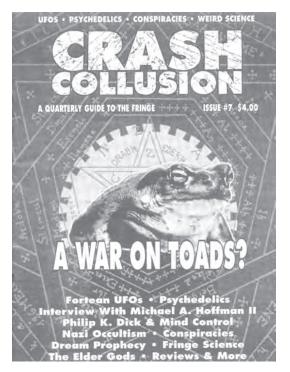
Tyler & Gröger 1964 (German specimens) Identified chromatographically. (Mycelium shown to contain 0.03%)

Stijve 1979 (German, Dutch & Swiss specimens). Bufotenine was the major. Bufotenine concentrations were estimated from 0.70-1.5% using GLC and from 0.4-1.3% using tlc. Isolation: 0.8% in cap, 1.5% in stalk & 0.065% in bulb.

Wurst et al. 1992 (0.0-1.899%)

Amanita muscaria

Wieland & Motzel 1953 (observed) Catalfomo & Tyler 1961 could not verify Stijve 1979 could not detect it



Bufo alvarius depicted above Cover design and "Sparky"'s photo by W. Nation

Amanita pantherina Wieland & Motzel 1953 (observed) Brady & Tyler 1959 could not detect. Stijve 1979 could not detect it Amanita porphyria Catafolmo & Tyler 1961 (North American specimens) Tyler 1961 (observed) Tyler & Gröger 1964 (German specimens) Identified chromatographically. Wurst et al. 1992 (0.374 & 0.617%) Amanita rubescens Wurst et al. 1992 (0.018-0.020%) Stijve 1979 could not detect it. Amanita tomentella Tyler 1961 (observed) Overview & summary: see Catalfomo & Eugster 1970 Graminae

Arundo donax

In leaf and rhizome. 180 mg from 700 grams of rhizome. Ghosal *et al.* 1969 128 mg from 200 grams of dry plant. 110 plus 18 mg. Dutta & Ghosal 1967 In flowers. Ghosal *et al.* 1971 *Phalaris arundinacea* (France) Traces reported (HPLC) Festi & Samorini 1994b *Phalaris canariensis* (Portugal) Traces reported (HPLC) Festi & Samorini 1994b *Phalaris minor* (Portugal) Traces reported (HPLC) Festi & Samorini 1994b *Phalaris paradoxa* (Romania)

Traces reported (HPLC)

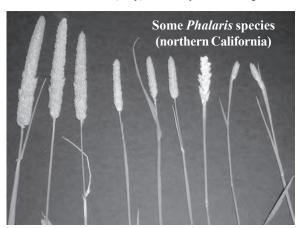
Festi & Samorini 1994b

Phalaris truncata (France) Traces reported (HPLC) Festi & Samorini 1994b

Phalaris tuberosa (= *P. aquatica*)

Present in all fresh samples they examined but not in all dried samples and if so was present in considerably reduced amounts. Culvenor *et al.* 1964

Festi & Samorini 1994b reported traces both in commercial material and in **AQ-1** (Italy). HPLC by Fabio Calligris.



P. aquatica cv stenoptera (left); P. minor (center)

Phalaris tuberosa var. Australian Commercial

Trace. Baxter & Slaytor 1972b.

Phragmites australis (Cav.) Trin. ex Steud Rhizome. Not quantified. Wassel *et al.* 1985 Lauraceae

Umbellularia californica Shulgin & Shulgin 1997

Leguminosae

Anadenanthera spp.

- Analyzing seeds, known to be used in preparing paricá, collected in Brazil during the first half of the 1800s, DeSmet & Rivier 1987, reported finding only bufotenine. They thought that any other tryptamines may have degraded over time. They noted that Schultes *et al.* 1977, reported that 2 year old *Anadenanthera* seeds were observed to go through a similar change in composition while in storage and that Spruce's 1854 collection of *A. peregrina* seeds also yielded only bufotenine. [Identification of *Anadenanthera* species cannot reliably be made from seeds alone.]
- [It should be mentioned that Holmstedt & Lindgren 1967 detected both DMT and bufotenine in seeds that had been collected in 1948 and DMT and 5-MeO-DMT in seeds collected in 1956. Similarly Torres *et al.* 1991 detected DMT, 5-MeO-DMT and bufotenine in snuff dated to 780 AD]

Snuff: "epena"

Obtained from the Waica by George Seitz. Bufotenine was a minor component, Holmstedt 1965. Because of this, the claimed plant source (*Virola*) has been questioned. [Seitz claimed to have witnessed the preparation of the material from *Virola*. In light of the ambiguities uncovered in snuff analysis, we do not think this can automatically be considered to be an *Anadenanthera* snuff without further study. Our suspicion is that the botanical sources of snuffs remain incompletely characterized.] 5-MeO-DMT was the major alkaloid. (DMT also present as a minor)

Snuff: "epena"

Snuff prepared, by Ma-hekodo-teri of the Rio Mavaca, from seeds of an *Anadenanthera* species. Bufotenine [with Bufotenine-N-oxide, DMT and DMT-N-oxide]

Marini-Bettòlo et al. 1964

Snuff: "yopo"

Snuff prepared, by Pixasi-teri (or Bisashi-teri) of Upper Orinoco, from an *Anadenanthera* species seeds Bufotenine [5-MeO-DMT also present]

Marini-Bettòlo et al. 1964

Snuff: "yopo"

Snuff collected in Colombia (collected 1956) Bufotenine [DMT and 5-MeO-DMT also present]

Holmstedt & Lindgren 1967

Snuff: "paricà"

Snuff prepared by Piaroa Indians (collected 1955) Bufotenine [DMT and 5-MeO-DMT also present] Holmstedt & Lindgren 1967

Anadenanthera colubrina

2.1% yield from seeds collected in autumn near Rio de Janeiro. Voucher prepared. Pachter *et al.* 1959

Anadenanthera colubrina

Conflicting reports. Most accounts found only bufotenine in the seeds but several reports exist of the additional presence of DMT and/or 5-MeO-DMT. Torres *et al.* 1991 reported the detection of all three in snuff powder recovered from archaeological sites in Argentina believed to have been derived from *A. colubrina* seeds. Both *A. colubrina* and *A. colubrina* var. *cebil* occur in Argentina.

While it is not clear which Torres and coworkers referred to; the latter is implied.

No analysis of seeds or verifiable plant material were reported in Torres *et al.* 1991

Modern unpublished analysis of seeds from Argentinean *A. colubrina* var. *cebil* found only bufotenine in material that proved active in humans used as a snuff or when smoked.

Anadenanthera colubrina var. cebil (= Piptadenia macrocarpa)

Present in seeds. Not detected in pods: Fish *et al.* 1955. Material from both Florida and Brazil were used.

In seeds and lower in seed pods. Iacobucci & Rúveda 1964

- *Anadenanthera colubrina* var. *Cebil* Bufotenine 12.4% in seeds from a shaman's garden at Misión Wichi [Highest reported concentration of bufotenine in a plant], 4.41% in a sample of seeds from Salta, 3.51% (seeds) and 0.05% (pods) in a second Salta collection.
- Only traces were found in bark from Cerro San Bernardo (All in Argentina). Torres & Repke 1996
- Anadenanthera excelsa (as Piptadenia excelsa) In seeds. Lower concentration than *P. macrocarpa*. Iacobucci & Rúveda 1964
- Anadenanthera falcata (as Piptadenia falcata) Major alkaloid in seeds. Giesbrecht 1960
- Anadenanthera peregrina

1.1% in seeds. Periera 1957

Major alkaloid in seeds. (Haiti) Paris et al. 1967

Present in seeds. Not detected in pod: Fish *et al.* 1955. Material from both Puerto Rico and Brazil were used Alvares Pereira & deOliveira 1961 Stromberg 1954

as *Piptadenia peregrina* Seeds collected in Puerto Rico during 1948 Bufotenine [DMT also present] Holmstedt & Lindgren 1967

Anadenanthera peregrina [R. Spruce #119; Rio Negro, Brazil, collected in 1854] (1977 analysis) Seeds: 0.6% Bufotenine [614 mg/ 100 gm dry; Sole alkaloid.]

Schultes et al. 1977

Anadenanthera peregrina [R.E.Schultes, S.von R.Altschul & B.Holmstedt, sin. num.; La Carolina, Barrio St. Just, near San Juan, Puerto Rico, December 1974. Same colony as Schultes 26363.]

Bufotenine

1972c

Mature seeds collected in March 1975; hill behind El Comandante horse-racing track. 1975 analysis (5 months after collection): No quantification Bufotenine- 80% of total alkaloid. 1977 analysis of same material: 3.5% Bufotenine [3523 mg/ 100 gm dry; Bufotenine was sole alkaloid present.] Schultes et al. 1977 Anadenanthera peregrina [R.E. Schultes 26363; La Carolina, Barrio St. Just, near San Juan, Puerto Rico, Dec. 1972] Immature seeds collected December 1972 0.01% Bufotenine [6% of 209 mg of total alkaloid/ 100 gm dry] Seedlings 0.00025% Bufotenine [1% of 25 mg of total alkaloid/100 gm dry] Pods without seeds 0.00013% Bufotenine [1% of 13 mg of total alkaloid/100 gm dry] Twigs 0.0004% Bufotenine [1% of 38 mg of total alkaloid/100 gm dry] Roots [0.69% total alkaloid] 0.007% Bufotenine [1% of 699 mg of total alkaloid/100 gm drySchultes et al. 1977 Anadenanthera peregrina [Collected in southern Venezuela.] Seeds- 7.5% bufotenine Schultes et al. 1977 cited Chagnon et al. 1970 & 1971 Snuff: "vopo" Believed to have originated from A. peregrina seeds; showed only bufotenine- 160 mg from 6 gm of snuff (2.67%). De Budowski et al. 1974. Another Yopo sample showed only 5-MeO-DMT and was thought derived from a Virola species instead. Snuff: "epena" Yanomamo snuff prepared from Piptadenia peregrina. Marini-Bettòlo et al. 1964 Anadenanthera peregrina (L.) Spegazzini [No.24625; Origin: Boa Vista, Brazil] Traces in dry bark. Agurell et al. 1969 Desmodium caudatum DC Major alkaloid in stem (0.04% by dry weight; If they used all of their picrate they would have recovered 4.3 gm of bufotenine base from 10.75 kg of stems.) Ueno et al. 1978 **Desmodium** gyrans Leaf (68 mg. from 2 kg. of dry leaves.) Ghosal et al. 1972a Desmodium pulchellum Whole plant (Minor alkaloid) Ghosal & Mukherjee 1964; (Mention) Ghosal & Mukherjee 1965; (Amount not given) Ghosal & Mukherjee 1966 Stem and leaf of young seedling [~ 0.011% by dry weight; 9% of 0.12% Total alkaloid] Ghosal et al.

Stem and leaf of mature plant [0.112% by dry weight; 8% of 1.4% Total alkaloid] Ghosal *et al.* 1972c Root of mature plant. (Trace) Ghosal et al. 1972c Root and stem-leaf (Amount not given) Ghosal 1972a Mimosa somnians May be in error? Not observed by Gupta and coworkers Lespedeza bicolor Turcaninow var japonica Nakai Present in both leaf and root bark. Morimoto & Matsumoto 1966 Mucuna pruriens In root, stem-leaf and pod. Ghosal (1972)a Piptadenia communis In seeds [with "related substances". Much lower concentrations than *peregrina* or *Cebil*] von Reis Altschul 1964 page 7 cited a letter from M.S. Fish dated 7 January 1958. Piptadenia contorta In seeds [with "related substances". Much lower concentrations than *peregrina* or *Cebil* von Reis Altschul 1964 page 7 cited a letter from M.S. Fish dated 7 January 1958. Also by Yamasato et al. 1972 (TLC) Piptadenia leptostachya In seeds [with "related substances". Much lower concentrations than *peregrina* or *Cebil*] von Reis Altschul 1964 page 7 cited a letter from M.S. Fish dated 7 January 1958. Piptadenia moniliformis In seeds by TLC. Yamasato et al. 1972 Malpighiaceae Diplopterys cabrerana [Plowman #6040; Tarapoto, "Chagro-panga"] Traces of Bufotenine were present in leaf. McKenna et al. 1984a Agurell et al. 1968a also reported traces in leaf. Myristicaceae Osteophloem platyspermum (DC) Warb. [Schultes & Rodriguez No. 26126; Origin: Manáos, Brazil]

Bufotenine in bark. One of 3 alkaloids in 0.62 mg of total alkaloid from 100 grams of dry bark. This is the only report of this compound being observed in a member of the Myristicaceae. Holmstedt *et al.* 1980

[Plowman, Schultes & Tovar # 7095; Origin: Pebas, Peru (Alpha-Helix 1977) assayed negative with Dragendorff and Ehrlich reagents.]

Virola sebifera

The listing of this compound for this species **IS in error.** We suspect that it stems from Holmstedt's analysis of Epená claimed by Seitz to have originated from *Virola*.

This is the lone claim of bufotenine in the genus and it is considered by most not to represent a *Virola* based snuff for this reason.

We encountered one string of claims of bufotenine showing up as a minor base in analysis of this plant. Kawanishi *et al.* 1985 made the claim for this species citing Schultes & Holmstedt 1971. Schultes & Holmstedt mention this in passing as part of an included quote taken from Corothie & Nakano 1969. Schultes & Holmstedt deliberately deleted the portion of the quote that implied that Holmstedt found this alkaloid in the species. Holmstedt analyzed *epena* snuff.

Corothie & Nakano 1969's wording could be taken to mean that Holmstedt reported this but do not include a reference (#14) for the statement.

The only paper of Holmstedt (#6 on their list) that they list is Holmstedt's analysis of Seitz's *Epena* snuff. [i.e. Holmstedt 1965]

At no point during that particular study did Holmstedt analyze plant material of *Virola sebifera* and detect this compound.

Urticaceae

Urtica pilulifera Shulgin & Shulgin 1997

Animals:

Gorgonaceae

Paramuricea chamaeleon

10 mg. of bufotenine isolated from 200 grams of coral Cimino & DeStefano 1978

Names followed by plus sign(s) from Daly & Witkop 1971:

+1-100 μ g / gm of skin

- ++ $\,$ 100-1000 μg / gm. of skin
- +++ 1-10 mg / gm. of skin

Bufonidae

"Toads"

Wieland et al. 1934 and Wieland & Wieland 1937

Ch'an Su (Chinese pharmaceutical preparation of toad venom) Isolated by Jensen & Chen 1932

Bufo sp. Wieland & Wieland 1937

Bufo alvarius Wieland & Wieland 1937

Cutaneous glands were found to contain 0.8 to 5 mg/gm. Non-glandular skin was found to contain 0.33-2.15 mg/gm Erspamer *et al.* 1965

Up to 3 mg per gram of dried skin.

Concentrations ranged from less than 0.1 mg to 5.0 mg per gram of large cutaneous glands (averaging 1.2 milligrams/ gm. glandular tissue) and from 0.17-2.2 mg per gram (averaging 0.9 mg/ gram) in the rest of the dry skin. Erspamer *et al.* 1967

Bufo americanus + Identified by Erspamer 1954 (Also listed in Erspamer 1961)

Bufo arenarum +++ Isolated by Jensen & Chen 1932 Wieland *et al.* 1934 recovered 5.1 mg/dried skin. (Also listed in Erspamer 1961)

Bufo boreas +++

Bufo bufo bufo ++ Isolated by Handovsky 1920 Males found to contain 510 μg/animals (0.3% in dry secretions. Females: 90 μg/animal (0.33% in dry secretions) by Wieland & Behringer 1941. (Also listed in Erspamer 1961) Bufo bufo formosus ++

Bufo calamita ++ Identified by Erspamer 1954

Bufo chilensis Deulofeu & Rúveda 1971

Bufo crucifer + Identified: Alvares Pereira & deOliveira 1961. Isolated: Deulofeu & Duprat 1944

Bufo debilis + Bufo fernandezae ++ **Bufo formosus** Isolated by Ohno et al. 1961 Bufo fowleri + Identified by Erspamer 1954 Also listed in Erspamer 1961 Bufo granulosus + Bufo hemiophrys ++ Bufo ictericus + Bufo luetkeni + Bufo major ++ **Bufo marinus** + (Despite the unsupported claims that this species is a good source of bufotenine, it possesses only low concentrations WHEN it is even present. For example: Cei et al. 1968 found it was present in only some collections and even when present it was sometimes only in some individuals.) Bufo marmoreus ++ Bufo melanostictus + Bufo microscaphus + Bufo paracnemis +++ Isolated by Deulofeu & Mendive 1938 & by Deulofeu & Duprat 1944 Bufo perplexus +++ Bufo punctatus + Bufo pygmaeus ++ Bufo spinulosus ++ Bufo terrestris + Bufo trifolium ++ Bufo viridis +++ Identified by Erspamer 1959, who found 630 μ g/gm of dried skin (0.06% in fresh skin.). (Also in Erspamer 1961: 640 μ g/ gm of fresh skin.) Bufo viridis viridis Isolated: Jensen & Chen 1932 & 1936 Bufo vulgaris (Bufo bufo bufo) Handovsky 1920 (Also even earlier by Phisalix & Bertrand 1893 but they did not fully characterize.) Isolated by Jensen & Chen 1936. Atelopodidae Melanophryniscus moreirae +++ Melanophryniscus stelzneri + Hylidae Litoria adelaidensis (0 & 100 µg/gm: Roseghini et al. 1976) Litoria chloris (0 & 20 µg/gm: Roseghini et al. 1976) Litoria dentata ++ (0.75 mg/gm: Roseghini et al. 1976) Litoria ewingii (0.87 & 2.3 mg/ gm: Roseghini et al. 1976) Litoria gracilenta + (25 µg/gm: Roseghini et al. 1976) *Litoria lesueurii* + (5 µg/ gm: Roseghini *et al.* 1976) Litoria moorei (0 & 0 & 8 µg/ gm: Roseghini et al. 1976) Litoria pearsoniana +++ (0.75-7 mg/gm: Roseghini et al. 1976) Litoria peronii + (10 & 15 µg/ gm: Roseghini et al. 1976) Litoria rubella + (10 & 60 µg/ gm: Roseghini et al. 1976) Litoria thesaurensis (35 µg/gm: Roseghini et al. 1976)

Bufotenine

Nyctimystes kubori (85 µg/gm: Roseghini et al. 1976)

Phyllomedusa rohdei (10-25 µg/gm: Roseghini et al. 1986) Ranidae

Rana temporaria +

Occurrence in humans:

Bufotenine is found in normal human blood and urine; Franzen & Gross 1965

Angrist et al. 1976 found that while the overall values were higher in their patient group (especially patients with acute psychosis, in women patients and in those patients with a high suspiciousness rating), there was no statistically significant difference between bufotenine blood levels of psychotic patients and normal subjects.

Kärkkäinen & Räisänen 1992 also detected endogenous bufotenine in normal humans.

See also Räisänen & Kärkkäinen 1979.

Narasimhachari et al. 1971b reported it was more common in psychotics than normals; also see Sitaram's papers.

For more references on its reported occurrences in humans: See Davis 1989.

Reviews:

McLeod & Sitaram 1985 See also Erspamer 1954

TD_{L0}

 $57 \,\mu\text{g}/\text{kg}$ intravenous in humans [= Toxic dose low: i.e. the lowest amount to produce 'toxic' effects. For Sax and many others, simple activity of the hallucinogens is apparently considered to be a toxic effect.] Sax cited Fabing & Hawkins 1956.

Activity:

Hallucinogenic: Fabing & Hawkins 1956; Southon & Buckingham 1989; Merck 9th; Usdin & Efron 1979

It should be noted that Fabing & Hawkins used iv administration, Turner & Merlis gave it im. The latter reported no effects when pure bufotenine was given nasally at dosages up to 40 mg. Chilton et al. 1979

Said to produce psychotropic effects when small amounts given IV. Sax & Lewis 7th page 1346.

This remains in debate. It is now widely accepted that Bufotenine is inactive as a hallucinogen.

It is noteworthy that NONE of those published papers which claimed it to be either hallucinogenic or nonhallucinogenic had ever evaluated it in themselves so these reports lacked any truly meaningful evaluation.

However recent bioassays indicate that Bufotenine is in fact active, as smoking or snuffing of LARGE amounts of crushed Anadenanthera colubrina seeds has been reported to be active in multiple bioassays involving a decent number of individuals. Analysis of the seeds had shown only Bufotenine to be present. (RM: personal communication at Mind States in 1997.)

Direct experiences with smoked Bufo marinus venom (US) [a very poor source of bufotenine when it contains it all all!] and smoked Anadenanthera colubrina var. cebil seeds (Argentina), produced an initial vague stimulation

followed by an unpleasant oppressive feeling, a restricted feeling of breathing, extreme salivation to the point of drooling, nausea, lolling of the head, ataxia, upper body tension, headache, mydriasis, mental confusion with a center of clarity, inability to think purposefully leading to a light stupor, lethargy and a tiredness leading to a light fitful sleep. Duration was around an hour. It was claimed by an informed individual that an excessive dosage level was to blame for this reaction. In 2001, smaller amounts of Anadenanthera colubrina

seeds were found highly effective as a visionary compound; with a distinctive tryptamine signature; snuffed (3) or smoked (<1 seed). (Justin Case 1995-2001)

For a summary of the evidence (if not **proof**) for its activity see: McLeod & Sitaram 1985 & Ott 2001a & b

Dose:

Human dose:

- 2-16 mg. Fabing & Hawkins 1956; 2-16 mg/kg/iv Usdin & Efron 1979 cited the same.
- 70 mg. Arnold & Hoff 1962 (Usdin & Efron 1979 cited Udenfriend 1969)
- Said to not be hallucinogenic in Ott 1993 & 1996
- However, Ott 2001a & b painstakingly determined that bufotenine free base was active & effective when smoked (2-8 mg), insufflated (40-100 mg), or taken orally (100 mg) or sublingually (50 mg) or rectally (50 mg + 10 mg)harmaline) Ott noted a marked potentiation when small amounts of harmine or harmaline HCl (10 mg or less) were combined with the bufotenine.
- Rather than hypothetical or secondhand assessments, Ott's conclusions were based on firsthand direct evaluation, we therefore consider Ott to be the best authority on the subject.
- (See also the otherwise excellent accounts in Lyttle et al. 1996 & McBride 2000 & Weil & Davis 1994 wherein the perils of drawing pharmacological conclusions bereft of direct experience are rather clearly illuminated.) See also Chilton et al. 1979 for a discussion.

- Psychoactive 10-12 mg. i.m.; 10 mg. i.v. Turner & Merlis 1959 and Fabing 1956. Ott 1993 & 1996
- 50 µg/kg smoked/insufflated. Duration 10-20 minutes. Callaway & McKenna 1998

Duration:

Complete recovery within 1-2 hours.

Receptor site specificity:

Agonist at 5-HT_{1A}, 5-HT_{1C}, 5-HT_{1D} & 5-HT₂ Callaway & McKenna 1998 See also: Almaula et al 1996 a & 1996b Choudary et al. 1993 Glennon et al. 1979 McBride 2000 McKenna et al. 1990 Peroutka 1991 Roth et al. 1997

5-HO-DMT

Biochemical & Animal miscellany:

Nialamide, an MAO inhibitor, increases the urinary excretion of endogenously produced Bufotenine. Kärkkäinen & Räisänen 1992

No effects on conditioned avoidance: see Gessner & Page 1962

Pharmacology & Pharmacognosy:

Found to have *in vitro* and *in vivo* **anti-cholinesterase** activity similar to, but 20-30 times weaker than, physostigmine. [Psilocybin & LSD were also reported to show anticholinesterase activity: Bhattacharya & Sanyal 1971 who cited Richter & Crossland 1950.]

Erspamer 1954 commented that bufotenine is a stronger cholinesterase inhibitor than serotonin

Bufotenine at 4 mg/kg iv in dogs:

Pseudoparaplegic splaying of hindlegs, salivation, pilomotor response, an "*unearthly howling*" (for better part of 3 hours), indifference to noxious stimuli and apparent inability or unwillingness to defend itself when attacked by other dogs.

Pharmacological evaluation in animals:

Oxytocic (half that of serotonin), raised blood pressure, showed BOL inhibition and increased behavioral disturbances. Gessner *et al.* 1961

Half the oxytocic activity of serotonin Taborsky & McIsaac 1964

Erspamer 1954 for animal pharmacology.

Metabolism: see Erspamer 1954

Distribution, metabolism & excretion in rat:

Sanders & Bush 1967

Pharmacology, metabolism & excretion in rats: Gessner *et al.* 1960 (Found it potentiates barbiturate induced sleeping time more than serotonin and less than PSOP.)

Excretion rates in humans: Räisänen & Kärrkkinen 1979

Physiological properties & toxicology: Fabing & Hawkins 1956

MAO kinetic constants: Suzuki et al. 1981

Preferentially metabolized by MAO-A at 20 μ M but some MAO activity was due to MAO-B at concentrations of 1000 μ M. Suzuki *et al.* 1981

Tolerance: No information located. If any tolerance develops it will be extremely short-lived.

Pharmacological overview:

Erspamer 1961 was cited by Culvenor *et al.* 1964f Bonhour *et al.* 1967 Gessner *et al.* 1960 (also metabolism; excretion) McBride 2000 Ott 2001a & 2001b Sanders-Bush *et al.* 1976

Toxicity:

Jumping action, clonic & tonic convulsions and tremor common to all animals before death. Ho *et al.* 1970 Death due to respiratory failure. Erspamer 1954 Said to be poison when given IP. (says same about DMT) Emits toxic nitrogen oxide fumes when heated to decomposition. Sax & Lewis 7th page 1346.

LD₅₀

290 mg/kg/ip/mouse; Ghosal et al. 1969.

290 mg/ kg ip in mouse; Ho et al. 1970.

Cerletti 1959 claims ip LD50 for mouse reported in the region of 25-30 mg / kg by Erspamer 1954 but we were unable to locate these values within this work. Guinea pigs lived after being given 20 mg.

Mice died after being given 0.5 mg/ kg IP (??!)

Erspamer 1954 (citing Handovsky 1920).

Gessner *et al.* 1960: 125 mg/ kg/ ip of bioxalate proved fatal to rats in >30 min. (marked by extreme rigidity).

If given in 6 divided doses, this was not fatal to 5 rats which appeared to recover. However, **a week later** 4 out of the 5 rats went on to develop prostration, flaccidity, dehydration, thoraco-lumbar hyperflexion (kyphosis), cervical extension and loss of 1/3 of their body weight. They then died.

The fifth rat apparently was fine. See comments elsewhere here on this topic.

Bufotenine-N-oxide

CA Reg. # 1019-44-9

C₁₂H₁₆N₂O₂ MW 220.271 Southon & Buckingham 1989: # B-00202

mp 211-214° (no water of hydration) [After being washed with ether and dried at RT]

Recrystallizing or drying at 80° in vacuo; gave varying mp.

Fish *et al.* 1955 Rods mp 217° (From MeCO₂-EtOH) Ghosal *et al.* 1969 mp 214-215° Dutta & Ghosal 1967

Assays for Bufotenine-N-oxide:

Colorimetric reagents: See color reactions p. 148

TLC & PC: See Rf table p. 169-176

UV:

λmax 278, 301, 314 nm (reference material) 278, 302, 314 nm (isolated material) Fish *et al.* 1955

Fluorescence:

Fluorescence maxima: Excitation: 308 nm; Emission 330 nm. Fish *et al.* 1955

Prepared as for DMT-N-oxide. Reconverted to bufotenine with zinc dust and acetic acid. Fish *et al.* 1955

Reported Occurrences of Bufotenine-N-oxide: Agaricaceae Amanita citrina (Schff.) S.F. Gray (low concentration) Tyler & Gröger 1964 (German specimens) Identified chromatographically. Stijve 1979 (Minor alkaloid) Amanita porphyria (low concentration) Tyler & Gröger 1964 (German specimens) Identified chromatographically. C₁₂H₁₈N₂O Leguminosae Anadenanthera species "epena" snuff prepared, by the Ma-hekodo-teri of the Rio Mavaca, from seeds. Bufotenine-N-oxide [Accompanied by Bufotenine, DMT & DMT-N-oxide] Marini-Bettòlo et al. 1964 nine) Anadenanthera colubrina var. cebil In seeds. Iacobucci & Rúveda 1964 Anadenanthera colubrina var. cebil Present in seeds. Not detected in pods: Fish et al. 1955. Material from Florida and Brazil were used. In seeds. Iacobucci & Rúveda 1964 Indide Piptadenia excelsa In seeds. Lower concentration than P. macrocarpa. Iacobucci & Rúveda 1964 Flavianate Anadenanthera peregrina (As Piptadenia peregrina) Present in seeds. Not detected in pods: Fish et al. 1955. (Material from both Puerto Rico and Brazil) Minor alkaloid in seeds. (Haiti) Paris et al. 1967 Oxalate epena Yanomamo snuff prepared from Piptadenia peregrina. Marini-Bettòlo et al. 1964 Picrate Desmodium caudatum Minor root alkaloid [0.03%; 496 mg from 1.6 kg] Minor stem alkaloid [0.004%; 447 mg from 10.75 kg of stems.] Ueno et al. 1978 Desmodium pulchellum Stem and leaf of mature plant. [Trace] Root of mature plant. [Trace] Ghosal et al. 1972c **Mvristicaceae** Virola sebifera The listing of this compound in this species appears **Picrolonate** to be in error. We suspect that it stems from Holmstedt's analysis of Epena snuff claimed to have originated from Virola. Holmstedt ran Bufotenine-Noxide as a reference material only. Activity: Activity may be similar to Bufotenine if smoked.

Bufotenidine

metho cation. Bufotenine Cinobufagin, 3-[2-(Trimethylamino)ethyl]-1H-indol-5-ol (cation); N, N, N - Trimethyl - 5 - hydroxytryptamine;

N₄-Methyl-Bufotenine, inner salt; BTD

(5-Hydroxy-hypaphorine appears in the literature but this name is *incorrect*).

Chemical Abstracts Registry Number: [487-91-2]

MW 218.298

Base is soluble in acetone, methanol

Hydriodide (produced by action of methyl iodide on bufotemp. 209° Southon & Buckingham 1989 mp 215° Deulofeu & Duprat 1944 mp. 217º Boit 1961 mp 209-210° Ghosal et al. 1969

mp 216-217° Wieland & Wieland 1937

mp. 195-200°C Wieland & Wieland 1937 mp 200° dec. Wieland et al. 1931

mp. 96.5°C Wieland et al. 1934

mp. 198°C Wieland et al. 1934

mp 198° Wieland et al. 1931

mp 198-200° (Red picrate from aqueous ethanol) Ghosal et al. 1969 (198° when recrystallized)

Reddish-yellow (crude from EtOH)

mp. 198-200° Orange needles when recrystallized from aqueous alcohol) Ghosal et al. 1970b. Same figure is given by Boit 1961 [mp. 198° given as literature figure (citing Jensen & Chen 1936]

mp 198-200° Ghosal et al. 1970

mp. 255° Boit 1961

Yellow prisms 253-255° (From 50% Ethanol; Fine yellow needles mp 255°) Deulofeu & Berinzaghi 1946

Assays for Bufotenidine:

Colorimetric reagents: See page 148

TLC & PC: (Ghosal *et al.* 1969) Rf 0.18 in n-Butyl acetate-n-Butanol-Acetic acid-Water (85:15:40:22) [Also in 1970] Rf 0.16 in Methanol on silica gel G (also used for preparative TLC)

Bufotenidine

UV:

λmax 218-220 (4.55), 284-288 nm (3.98) Ghosal *et al.* 1969

Isolation: [from Deulofeu & Rúveda 1971]

Ch'an Su Chen et al. 1931 & Wieland et al. 1931

Bufo gargarizans & Bufo fowleri Jensen & Chen 1932

Bufo bufo bufo $(170 \ \mu\text{g} / \text{animal by Wieland$ *et al.* $1934) and (males / 84 \ \mu\text{g per animal and 0.53% dried secretion and from females / 170 \ \mu\text{g per animal and 0.62% of dried secretion}) Wieland & Behringer 1941$

Bufo formosus at 2% of dried secretions by Ohno & Komatsu 1957

Xenopus laevis by Jensen 1935

Reported Occurrences of Bufotenidine: Plants:

Graminae

Arundo donax (rhizome) Ghosal 1972a & Ghosal *et al.* 1970 (A major component of 2.3 grams of basic gum obtained from 700 grams of rhizome. Ghosal *et al.* 1969)

Animals:

Names followed by a plus sign(s) (+) are from Daly & Witkop 1971

```
1-100
                    \mu g / gm of skin
++
          100-1000 \mu g / gm of skin
+++
          1-10
                    mg / gm of skin
 Bufonidae
Bufo americanus + (Erspamer 1954; & in 1961)
Bufo arenarum ++ (Also listed in Erspamer 1961)
Bufo boreas +
Bufo bufo bufo + (Also listed in Erspamer 1961)
Bufo bufo formosus ++
Bufo bufo gargarizans + (Erspamer 1954)
Bufo calamita + (Erspamer 1954)
Bufo canaliferus +
Bufo coccifer +++
Bufo fowleri + (Also listed in Erspamer 1961)
Bufo hemiophrys +++
Bufo marinus + (Cei et al. 1968 found it in only one out of
 11 collections)
Bufo melanostictus +++
Bufo microscaphus +++
Bufo paracnemis ++ (Erspamer 1954)
Bufo spinulosus ++
Bufo terrestris +++
Bufo trifolium ++
Bufo variegatus ++
Bufo viridis ++ (Erspamer 1954; listed in 1961)
Bufo woodhousei +++
  Hylidae
Cyclorana alboguttatus (30-100 µg/ gm: Roseghini et al. 1976)
Cyclorana platycephalus (0-40 µg/ gm: Roseghini et al. 1976)
Litoria adelaidensis (0.3-1.2 mg/gm: Roseghini et al. 1976)
Litoria angiana (65 µg/ gm: Roseghini et al. 1976)
Litoria aurea (25-60 µg/ gm: Roseghini et al. 1976)
Litoria booroolongensis (1.85 mg/ gm: Roseghini et al. 1976)
Litoria citropa (1.3 mg/ gm: Roseghini et al. 1976)
Litoria cyclorhynchus (0.8 mg/ gm: Roseghini et al. 1976)
Litoria ewingii (270 & 280 µg/ gm: Roseghini et al. 1976)
Litoria glandulosa (6 mg/ gm: Roseghini et al. 1976) [now
considered a synonym of Litoria subglandulosa]
Litoria latopalmata (30 & 190 µg/ gm: Roseghini et al. 1976)
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Litoria micromembrana (60 µg/ gm: Roseghini et al. 1976) Litoria moorei (3.7-5.5 mg/ gm: Roseghini et al. 1976) Litoria pearsoniana (0-350 µg/gm: Roseghini et al. 1976) Litoria raniformis (1.6 mg/ gm: Roseghini et al. 1976) Litoria thesaurensis (350 µg/ gm: Roseghini et al. 1976) Nyctimystes disruptus (4.3 mg/ gm: Roseghini et al. 1976) Nyctimystes kubori (250 µg/ gm: Roseghini et al. 1976) Nyctimystes tympanocryptis (5.1-9.5 mg/ gm: Roseghini et al. 1976) [now considered a synonym of Nyctimystes dayi] Nyctimystes vestigea (65-550 µg/ gm: Roseghini et al. 1976) [now considered a synonym of Nyctimystes dayi] Leptodactylidae Lantodactulus caligonus + [Linable to locate this name]

Leptodactylus caligonus + [Unable to locate this name] Leptodactylus curtus + (15 µg/ gm: Roseghini et al. 1986) [now considered a synonym of Leptodactylus labrosus] Leptodactylus melanonotus + (25 µg/ gm: Roseghini et al. 1986) Leptodactylus pentadactylus ++ (nd in Erspamer et al 1964) Leptodactylus pentadactylus dengleri (550 & 600 µg/ gm:

Roseghini *et al.* 1986; Erspamer et al. 1964)

Leptodactylus podicipinus podicipinus (20 µg/ gm: Roseghini et al. 1986)

Leptodactylus rubido + (40-45 µg/ gm: Roseghini et al. 1986) [now considered a synonym of Leptodactylus rhodonotus] Leptodactylus vilarsi (1.45 mg/ gm: Roseghini et al. 1986) [now considered a synonym of Leptodactylus stenodema]

Pelobatidae

Scaphiopus hammondii hammondii (70 µg/ gm: Roseghini *et al.* 1986) [now known as **Spea hammondii**]

Pipidae Xenopus ++

Xenopus laevis (Deulofeu & Rúveda 1971 cited Jensen 1935 (Erspamer 1961: strong concentration.)

Pharmacological & physiological properties of Bufotenidines:

Erspamer 1954 discusses pharmacology, physiological properties & toxicology.

Ghosal et al. 1969 evaluated in animals; reported to have curariform activity on isolated muscles and to block acetylcholine induced spasms. Neither death nor ataxia was noted in mice at 2 mg/ kg/ ip.

Toxicity:

Lethal dose: (due to respiratory failure) 10 mg/ kg/ ip/ mouse Ghosal et al. 1969

See also:

Southon & Buckingham 1989: Entry # B-00202



Bufothionine

Bufoviridine

Bufoviridine

Bufotenine-O-sulfate: 3-[2-(Dimethylamino)ethyl]-1H-indol-5-ol sulfate ester; BT-S

mp. 210-212° Erspamer 1959

Base is soluble in acetone, methanol

Isolation:

Erspamer 1959: from: Bufo viridis.

Reported Occurrences of Bufoviridine:

Names followed by a plus sign(s) (+) are from Daly & Witkop 1971 +1-100 μg / gm of skin ++ 100-1000 μg / gm of skin +++ 1-10 mg / gm of skin

Bufonidae

Bufo alvarius + 15-20 µg./gm. dried skin; Erspamer et al. 1967 Bufo calamita ++ (Erspamer 1959) Bufo debilis ++ Bufo perplexus + Bufo punctatus +++ Bufo spinulosus ++ Bufo viridis ++ (Also listed in Erspamer 1961; with 430 µg/ gram of fresh skin) Hylidae

Litoria pearsoniana (30-600 µg/ mg: Roseghini et al. 1976)

Activity:

Apparently unevaluated in humans, it is suggested to be inactive in animal studies.

Oddly, despite the 4-acetate, the 4-phosphate and the 4-benzoic acid esters of psilocin all being physiologically active, its 4-sulfate ester is inactive which further suggests that the sulfate ester of bufotenine may prove to be inactive as well. Cerletti et al. 1968

Bufothionine

Dehydrobufotenine-O-sulfate; Dehydrobufotenine sulfate; Dehydrobufotenine-O-hydrogen sulfate inner salt; Sulfate ester of dehydrobufotenine

C₁,H₁₄O₄N,S

MW 282.314

Free base:

mp 249° Deulofeu & Duprat 1944 colorless prisms mp 250° (darkening from around 240°) Deulofeu & Duprat 1944 mp 250° Boit 1961 (also Deulofeu & Rúveda 1971 citing

- Wieland & Vocke 1930)
- mp ca. 250° (with decomp.) Southon & Buckingham 1989 [Entry #D-00063]

mp 252° Deulofeu & Duprat 1944

Base is soluble in acetone, methanol

HCl

mp 244° Deulofeu & Duprat 1944

Heating with dilute hydrochloric acid will yield dehydrobufotenine and sulfuric acid.

Assays for Bufothionine:

Colorimetric reagents: See color reactions p. 148

TLC & PC:

- Rf 0.35 (streaking) in i-Propanol-Ammonia (880)-Water (200:10:20) on paper
- Rf 0.47 in n-Butanol-Acetic acid-Water (120:30:50) on paper
- Rf 0.72 in *n*-Butanol-Pyridine-Water (60:60:60) on paper
- Rf 0.43 in Potassium chloride (20% w/v) on paper
- Rf 0.49 in Sodium chloride (8% aqueous w/v)-glacial Acetic acid (200:2) on paper Jepson 1969

Reported Occurrences of Bufothionine:

Names followed by a plus sign(s) (+) are from Daly & Witkop 1971

- + $\mu g / gm of skin$ 1-100
- ++ 100-1000 µg / gm of skin
- +++ 1 - 10mg/gm of skin

Bufonidae

Bufo arenarum (7.1 mg./dried skin: Deulofeu & Rúveda 1971 cited Wieland et al. 1934)

- Bufo arenarum ++
- (Also listed in Erspamer 1961)
- Bufo boreas ++
- Bufo bufo bufo ++
- Bufo bufo formosus ++
- Bufo canaliferus ++

Bufo chilensis (Deulofeu & Rúveda 1971; Deulofeu & Duprat 1944)

- Bufo crucifer (Deulofeu & Rúveda 1971; Deulofeu &
- Duprat 1944)
- Bufo debilis ++
- Bufo fernandezae +++
- Bufo formosus (2 mg./dried skin) (Deulofeu & Rúveda 1971 cited Wieland & Vocke 1930)

Bufo granulosus +++

Bufo ictericus ++

Bufo major ++

Bufo marinus ++

Bufo paracnemis ++ (Deulofeu & Rúveda 1971;

Deulofeu & Duprat 1944)

Bufo punctatus +++

- Bufo pygmaeus ++
- Bufo spinulosus +++
- Bufo spinulosus (Deulofeu & Rúveda 1971; Deulofeu &

Acris crepitans + (Deulofeu & Rúveda 1971 cited

Duprat 1944)

Bufo typhonius ++

Bufo viridis + Hvlidae

Erspamer & Vialli 1952)

Dehydrobufotenine

"Constit. of the Japanese toad" Southon & Buckingham 1989

Activity is unknown and perhaps doubtful. See comment under Bufoviridine above.

Dehydrobufotenine

Chemical Abstracts registry number: [17232-69-8] Southon & Buckingham 1989: Entry #D-00063; 303

NIOSH # UY9454800

C1,H14N,O

MW 202.255

C 65.45%, H 7.27%, N 12.72% (calc.)

Free base:

mp. 199° & 218° Boit 1961 mp. 218°C Wieland & Wieland 1937 double melting point of 202 and 217° [mp 198° & 218°] Ghosal *et al.* 1969

Water soluble Ghosal *et al.* 1970b Soluble in ethanol Ghosal *et al.* 1969 Base is soluble in acetone, methanol

Hydrochloride:

mp 237-238° dec. Southon & Buckingham 1989 mp 237-238° (Needles from ethanol) Ghosal *et al.* 1969 mp 240-241° Deulofeu & Duprat 1944 mp 242° Wieland & Vocke 1930 mp 243° Deulofeu & Duprat 1944 mp 244° Boit 1961

Flavianate:

mp 260-265° Deulofeu & Rúveda 1971 cited Wieland et al. 1934

Hydriodide:

mp 243-245° (Light grey rods from aqueous ethanol.) Ghosal *et al.* 1969

Picrate:

- Yellow picrate (from absolute alcohol) mp 187°. (from 50% ethanol) mp 147-150°. Boiling with mineral acids will convert to higher melting point. Deulofeu & Duprat 1944
- mp 182-184° Ghosal *et al.* 1970b cited a literature value of mp. 183-184°, by Jensen & Chen 1936

mp 186° Wieland & Wieland 1937

mp 189° Boit 1961

mp 182-184° (Yellow needles from aqueous ethanol) Ghosal *et al.* 1969

Picrolonate:

mp 300°C Deulofeu & Rúveda 1971 Yellow prisms from 50% ethanol. Darkening 275°; mp above 300° Deulofeu & Berinzaghi 1946

Sulfate: mp. 209° Boit 1961

Assays:

Colorimetric reagents:

Dragendorff's- Orange Ehrlich's - Negative α-Nitroso-β-naphthol-nitrous acid - Violet Ghosal *et al.* 1970b [After extracting from *Arundo donax* & regenerating the base from the reineckate over a De-Acidite FF column.] Jepson & Steven's test: Negative. Ghosal *et al.* 1969 Diazotized *p*-nitroaniline - Orange Diazotized sulphanilic acid - Brown-red NNCD reagent - Orange-yellow Erspamer 1959

TLC:

Rf of 0.06-0.08 on Whatman #1 paper. [*n*-Butyl acetate-*n*-Butanol-Acetic acid-Water (85:15:40:22)] Rf 0.04 with Methanol on silica gel G. Ghosal *et al.* 1969

UV:

λmax 218-220 (4.55), 285 nm (3.98) Ghosal *et al.* 1969

PMR:

Marki *et al.* 1961 Robinson *et al.* 1961

Reported Occurrences of Dehydrobufotenine: Graminae

Arundo donax (440 mg [impure] from 700 grams of rhizome) Ghosal et al. 1969 [Possible presence in Phragmites communis rhizome is suggested in Wassel's account but unconfirmed.] Animals: Names followed by a plus sign(s) (+) are from Daly & Witkop 1971 1-100 $\mu g / gm of skin$ +++ 100-1000 μ g / gm of skin 1-10 mg/gm of skin +++ Bufonidae Bufo fernandezae +++ Bufo americanus + (Identified by Erspamer 1954). (Also listed in Erspamer (1961) Bufo arenarum ++ (Isolation by Wieland et al. 1934). (Also listed in Erspamer (1961) Bufo boreas + Bufo bufo ++ (Identified by Erspamer 1954). (Also listed in Erspamer (1961) Bufo bufo formosus + Bufo calamita (Identified by Erspamer 1954). Bufo chilensis (Isolation: Deulofeu & Duprat 1944)

Dehydrobufotenine

Bufo cognatus +

Bufo crucifer (Isolation: Deulofeu & Duprat 1944) Bufo debilis +++ Bufo fowleri + (Identified by Erspamer 1954). (Also listed in Erspamer 1961) Bufo granulosus +++ **Bufo haematiticus +++** Bufo hemiophrys +++ Bufo ictericus +++ Bufo major ++ Bufo marinus +++ (Isolation of 6 mg./animal by Marki et al. 1932). ["Large amounts in parotid glands of **Bufo** marinus." Daly et al. 1993] Bufo melanostictus + **Bufo paracnemis** ++ (Identified by Erspamer 1954) (Isolation by Deulofeu & Duprat 1944) **Bufo punctatus +++** Bufo pygmaeus +++ Bufo regularis (Isolation by Jensen 1935) Bufo speciosus + *Bufo spinulosus* +++ (Isolation by Deulofeu & Duprat 1944) Bufo terrestris + Bufo typhonius ++ Bufo valliceps (Isolation by Jensen & Chen 1932) **Bufo viridis** + (Identified by Erspamer 1954) (Also listed in Erspamer 1961) Bufo woodhousei ++

Activity: Unknown.

Toxicity:

Lethal dose in mice is around 120 μg subcutaneously. Death occurs with clonic convulsions.

Daly et al. 1993: 262

See also Ghosal *et al.* 1969 but keep in mind that Ghosal tested this only as part of an alkaloidal fraction also containing bufotenidine and did not test it on its own (only doing so with bufotenidine). Assertions to the contrary which appear in the literature are in error or at least lack a primary reference.

Dehydrobufotenine is a cyclized form of bufotenine. Like bufothionine is not only substituted at the 5 position but is also substituted at the 4 position, this being where the aliphatic nitrogen forms a quaternary bond.

5-Methoxytryptamine

5-Methoxy-1H-indole-3-ethanamine;

3-(2-aminoethyl)-5-methoxyindole;

5-Hydroxy-tryptamine methyl ether;

Serotonin methyl ether; 5-MeO-TPA; 5-MeO-T; 5-MT; MT; (as HCl): Meksamin, Mekasamin, Mexamine (U.S.S.R.).

WLN: T56 BMJ D2Z GO1 Hayward: 6RR(OM)RRY5NHL=L(CCZ)Y Usdin & Efron 1979: #404

CA Reg. # [608-07-1] NIOSH # NL 4055000 Southon & Buckingham 1989

5-Methoxytryptamine is not a controlled substance

C₁₁H₁₄N₂O

MW 190.24 Merck 9th # 5878 MW 190.244 Southon & Buckingham 1989

C 69.44%, H 7.42%, N 14.73%, O 8.415 Merck 9th

Free base:

mp 120-121° Abramovich & Shapiro 1955 & 1956 and Kveder & McIsaac 1961

mp 120-121° (from chloroform-ligroin) Späth & Lederer 1930: page 2108.

mp 120-121° Southon & Buckingham 1989

mp 121-122° Crystals from ethanol Merck 9th

mp 121.5-122.5° Pale yellow prisms from benzene. [Also mp 115-117° from ethyl acetate and Skellysolve B (chilled overnight)]

Szmuszkovicz et al. 1960

mp 122-123° Hoshino & Kobayashi 1935 (Says Spath & Lederer 1930 found mp 120-121 and Wieland *et al.* 1934 reported mp 121-122°.)

Hydrochloride

C₁₁H₁₄N₂O. HCl mp 245° Tabular crystals from alcohol. Hoshino & Kobayashi 1935 Crystals dec. 248° Merck 9th

Picrate:

Magnificent ruby red crystals 214-215° "unter Aufschäumen schmolzen" Späth & Lederer 1930

mp 219° Abramovich & Shapiro 1955

- mp 219° (dec.) Deep red flat prisms. Abramovich & Shapiro 1956
- mp 220-221° (dec.) Hoshino & Kobayashi 1935

Assays for 5-Methoxytryptamine:

Usdin & Efron 1979 cited: Aures *et al.* 1968 Miller & Maickel 1970 Iskric *et al.* 1969 See also: Prozialeck *et al.* 1978

Colorimetric reagents: See color reactions p. 148

TLC & PC:

5-Methoxytryptamine: Solvent system Medium Ref <u>Rf</u> Acetone-i-Propanol-Water-Ammonia (0.880) (50: 40: 7: 3) 0.66 Silica gel (5)Benzene-Methanol-5% Ammonium hydroxide (10:15:2)0.25 Silica gel (1)*n*-Butanol-Glacial acetic acid-Water (2:1:1)0.57 Silica gel (1)n-Butanol-Acetic acid-Water (4:1:5)0.64 Paper (3)

5-Methoxytryptamine

n-Butanol-Acetic acid-Water (4:1:5)0.64 Paper (6)*n*-Butanol-glacial Acetic acid-Water (120:30:50)0.64 Paper (HCl) (2) n-Butanol-sat. w/ N HCl 0.32-0.36 Paper (HCl) (7) *n*-Butanol-Methylamine (25-30%) (8:3) 0 78-0 82 Paper (7)n-Butanol-Pyridine-Water (60:60:60)0.72 Paper (HCl) (2) Chloroform-Acetic acid-Methanol-Water (65: 20: 10: 5) 0.40 Silica gel (5)Ethyl acetate-Methanol-58% Ammonium hydroxide (80:15:5)0.30 Silica gel (1)aqueous KCl 0.30 20% (w/v) Paper (HCl) (2) *i*-Propanol-5% Ammonium hydroxide Silica gel (5:2) 0.44 (1)i-Propanol-Ammonia (8:2) 0.76 Paper (3)*n*-Propanol-Ammonia (8:3) 0.76 Paper (6)i-Propanol-Ammonia (880)-Water (200:10:20)Paper (HCl) (2) 0.76 Morpholine (0.1 M in Water) 0.65 Silica gel (4)8% (w/v) aqueous NaCl-glacial Acetic acid (200:2)0.35 Paper (HCl) (2) **References:** 1. Gupta et al. 1979 (silica Gel 60F-254 with PDAB) 2. Jepson 1960 & 1969 3. Kveder & McIsaac 1961 4. Sanders & Bush 1967 5. Smith and Seakins 1976: p. 146. 6. Taborsky & McIsaac 1964 7. Erspamer 1955

Column chromatography:

Separation using a combination of DEAE-cellulose, Sephadex and Amberlite columns. Iskric et al. 1969

HPLC:

Kysilka & Wurst 1988 Kysilka et al. 1985

GLC:

GLC of HFB derivative: Benington et al. 1975 & Vessman et al. 1969

GC:

Audette et al. 1969: Retention times for Amine 220, CHDMS and DEGS columns.

Verpoorte & Svendsen 1983: Relative retention times compared to tryptamine: p 155

UV:

λmax 223 (25,250), 227 (6,300), 296 (5,050), f308 (3,450) Szmuszkovicz et al. 1960

Fluorescence:

Absorbs under 254 nm UV (shows up as a dark spot) Faint Purple fluorescence under 350 nm UV Smith & Seakins 1976: p. 146. Visible fluorescence in 3M HCl- λ max 547. UV fluorescence in 0.01M Tris buffer at pH 7.4- λmax 350 nm. Chen 1968

IR: see Szmuszkovicz et al. 1960

MS:

Agurell et al. 1969 [m/e 160 (base peak), 30, 117, 145, 146, 161, 190 (M+)] See also Couch & Williams 1972

Synthesis:

Abramovich & Shapiro 1955 Asero et al. 1952 Hoshino & Kobayashi 1935 Kveder & McIsaac 1961 Majima & Hoshino 1925 Spath & Lederer 1930: p. 2108 Supniewski & Misztal 1961 (Included in synthesis of melatonin) Szmuszkovicz et al. 1960 (Two routes included in synthesis of melatonin) Wieland et al. 1934

Reported occurrence of 5-Methoxytryptamine:

So far, reports of its occurrence have been rare

- 5-MeO-tryptamine is found in the Sydney Funnel-web spider: Atrax robustus. Traces of 5-Methoxytryptamine are secreted in the venom of the female. The male has serotonin.
- Female secretes more poison. Male is more toxic. Geren & Odell 1984
- Atrax robustus venom also contains GABA. Tu 1977

In plants:

Graminae

Phalaris aquatica var. Australian Commercial

Traces. Baxter & Slaytor 1972b. (as *P. tuberosa*) Phalaris aquatica cv. Australian Commercial

A minor alkaloid in 7 day old seedlings. Mulvena & Slaytor 1983.

- While this compound has also been listed as being reported in the Apocynaceous Catharanthus roseus, this claim is in error. Naaranlahti et al. 1987, used 5-MeO-tryptamine only as an internal reference standard during HPLC analysis and they did not actually detect it in the plant.

Myristicaceae Virola peruviana (DC) Warburg

5-Methoxytryptamine in wood. No amounts given. Lai et al. 1973

Occurrence in humans:

Reported in human blood and urine by Erspamer *et al.* 1965 [Gessner *et al.* 1968 cited Siegal 1965]

5-Methoxytryptamine was found in normal human blood and urine by Franzen & Gross 1965

Found in cerebrospinal fluid of some psychotics and some normal people by Corbett *et al.* 1978

See also:

Beck & Jonsson 1981 Bosin & Beck 1979

DeMontigny & Aghajanian 1977 Heinze et al. 1980

Koslow 1976 Pevet 1983

Prozialeck et al. 1978

Activity:

Proposed as a potentiator for hypnotics and sedatives. Claimed to be more effective than serotonin.

Merck 9th: #5878 cited Mashkovshy and Arutyunyan 1963 Hallucinogenic according to Usdin & Efron 1979 #404 who

cite <u>ANIMAL</u> studies by Gessner *et al.* 1961. THIS CANNOT BE CONSIDERED TO BE ANY SORT OF PROOF!

See also: DeMontigny & Aghajanian 1977 Heinze *et al.* 1980 Koslow 1976

Biochemical miscellany:

MAO kinetic constants: See Suzuki et al. 1981

Preferentially metabolized by MAO-A at 20 μM but some MAO activity was due to MAO-B at concentrations of 1000 μM. Suzuki *et al.* 1981

5-Methoxytryptamine showed an IC₅₀ of 31.0 (μ M) in its inhibition of [³H]Imipramine binding in human platelets. [IC₅₀ is the concentration required to inhibit 50% of the specific binding.] Langer *et al.* 1984

Summary of pharmacological properties of 5-Methoxytryptamine:

Pharmacological evaluation in animals:

Oxytocic (70% that of serotonin), biphasic action on blood pressure, showed BOL inhibition and increased behavioral disturbances. Gessner *et al.* 1961

70% the oxytocic activity of serotonin

Taborsky & McIsaac 1964

Inhibits norepinephrine reuptake into rat brain synaptic vesicles: Slotkin *et al.* 1978

- Preadministration increases survival time in mice exposed to lethal radiation (if given within 30 minutes before exposure) Shinoda *et al.* 1974
- 20 mg/ kg caused an increase in 5-HIAA in rat brain but precise values unclear. Freedman *et al.* 1970
- Rats given the HCl at 1 gm by mouth or 10 mg/ kg sc (immediately after a water load) showed blocked diuresis for 4-5 hours, "*intense cutaneous vasodilation, accompanied by itching and the evacuation of abundant formed faeces.*" Erspamer 1955

In mice (as hydrochloride; subcutaneous or oral):

100 mg/ kg- Depression, ataxia, edema of the soft tissues of head and diarrhoea.

 $200\mathchar`-400$ mg/ kg- Depression, ataxia, tremor and diarrhoea were pronounced.

Effects lasted 2 hours.

5-10 mg/ kg produced a marked general depression lasting 2-3 hours; some animals showed increased salivation, vomiting and defecation. Mashkovsky & Arutyunyan 1964

90% of a given dose disappeared from plasma & tissues within 60 minutes. Showed apparent analgesic effects in mice as well as vasodilation.

Iproniazid increased the effects. Vogel 1969

Excretion, distribution and metabolism in rats: Kveder & McIsaac 1961

Erspamer 1955 noted what was thought to be 5-MeO-IAA in rat urine, accompanied by two faint spots.

Incubation with mescaline in rat brain homogenates showed no effects. Shah & Gulati 1974

Pharmacological overview: Mashkovsky & Arutyunyan 1964

Toxicity

LD₅₀:

- In mice (as hydrochloride):
 - 5mM/ kg/ intraperitoneal/ mouse. Shinoda *et al.* (1974) 102.5 mg / kg intravenous in mice. (Death occurred in
 - rats at 50 mg/ kg. intravenous.)

620 mg/ kg subcutaneous

580 mg/ kg orally

- Mashkovsky & Arutyunyan 1964
- As Hydrochloride:
- $LD_{50} 60 \text{ mg/ kg}$ intravenous in mouse

 LD_{50}^{50} 750 mg/ kg subcutaneous in mouse

 LD_{50} 600 mg/ kg subcutaneous in rat

Erspamer 1954

5-Methoxy-N-methyltryptamine

5-Methoxy-3-[2-(methylamino)-ethyl]indole;

3-(2-Aminopropyl)-5-methoxyindole;

- 5-Hydroxy-N_b-Methyl-tryptamine methyl ether;
- N-Methyl-5-methoxytryptamine;
- 5-Methoxy-amino-N-methyl-tryptamine;
- N,O-Dimethylserotonin; 5-MeO-MMT;

5-MeO-NMT; N,O-DMS; Nor-5-MeO-DMT

WLN: T56 BMJ D2M1 GO1 Hayward: 6RR(OM)RRY5NHL=L(CCNHM)Y Usdin & Efron 1979: #403

CA Reg. No.: 002009032 [2009-03-2]

5-methoxy-N-methyltryptamine is not a controlled substance

C₁₂H₁₆N₂O MW 204.271 Southon & Buckingham 1989: #H-00352

Free base:

mp 88-91° Shulgin & Shulgin 1997: pp. 546-547, entry #42.
mp 90-93° Shulgin & Shulgin 1997
mp 99-102° Colorless prisms from benzene. Legler & Tschesche 1963
mp 101.7° Transparent orthorhombic prisms. (Supplied by B. Holmstedt) Bergin *et al.* 1968
Vacuum distilled as pale yellow oil at 0.05 mm with a 150° bath temperature. Wilkinson 1958

Oil Boit 1961

Pale yellow oil. Wilkinson 1958. Pale yellow feathery crystals. Majak & Bose 1977 Soluble in ether. Shulgin & Shulgin 1997 No doubt soluble in methanol and in acetone.

Transparent prisms (orthorhombic) (Supplied by B.Holmstedt) Bergin *et al.* 1968) Density: 1.196 g/cm³ (0bserved)/ 1.182 g/cm³ (Calculated) Bergin *et al.* 1968)

Hydrochloride:

- mp 164-166° Legler & Tschesche 1963
- mp 165-166° Boit 1961
- mp 166-167° Prisms from methanol/ ether Southon & Buckingham 1989
- mp 166-167° (Prisms from ethanol-acetone) Wilkinson 1958

mp 167° (Needles from methanol) [Crude HCl was precipitated from ether with hydrogen chloride.] Ghosal & Mukherjee 1966

Insoluble in ether. Wilkinson 1958.

Picrate:

mp 216-220° Legler & Tschesche 1963 mp 220° Red picrate from MeOH (mp 222° with

"authentic") Ghosal et al. 1969

mp 220-221° (dec.) (Orange red prisms) Wilkinson 1958

mp 220-222° Red picrate Wassel *et al.* 1985

mp 222° (Orange-red picrate from methanol) [Crude picrate from acetone] Ghosal & Mukherjee 1966

Oxalate:

mp 209-210° Iacobucci & Rúveda 1964 mp 223-226° Legler & Tschesche 1963

Assays:

Colorimetric reagents:

See also the color reactions on p. 152 for more. Brown with Millon's reagent

Green in solution with *p*-dimethylaminobenz-aldehyde (0.125 gram in 65% sulfuric acid containing 0.1% v/v of 5% ferric chloride soln.). The green became blue when diluted with water.

Gave a deep blue color with *p*-dimethylaminobenzaldehyde in ethanol containing hydrochloric acid.

Blue spot with *p*-dimethylaminobenzaldehyde in cyclohexane when followed with hydrogen chloride treatment.

5-MeO-MMT gave a blue color with sodium nitroprusside and acetone but not with acetaldehyde. (Said to be characteristic of secondary amines.) [citing Kharichkov 1906] [All other references to Sodium nitroprusside reagent involving aldehyde imply that it would react blue with 5-MeO-MMT or most other secondary amines. Have not yet obtained a copy of Kharichkov 1906.]

No reaction with ferric chloride solution. Wilkinson 1958

TLC & PC: See Rf table p. 148

Preparative TLC:

Preparative tlc using Butanol-Acetic acid-Water (12:3:5) Shulgin & Shulgin 1997

Column chromatography:

Eluted from Brockman alumina with CHCl₃-MeOH (1: 9) Wassel *et al.* (1985) Eluted from alumina with chloroform. Wilkinson 1958

GC:

UV:

 λ_{max} 224 nm (log e 453) and 288 (3.82) (*Arundo donax*) Ghosal *et al.* 1969

 λ_{max} (EtOH) 223, 276 and 292 nm (*D. pulchellum*) Ghosal & Mukherjee 1966

 λ max 275.6 and 309 nm (ϵ 1 6470 and 3790) experimental / 275 and 309 nm (ϵ 1 6470 and 3790) with synthetic material. Wilkinson 1958

MS:

Williams *et al.* 1971 [m/e 44, 103, 117, 160, 161, 173, 204] see also Holmstedt & Lindgren 1967

Synthesis:

Shulgin & Shulgin 1997 [from 5-MeO-DMT and also from 5-Methoxytryptamine.]

Wilkinson 1958 [from 5-Methoxytryptamine]

Audette *et al.* 1969: Retention times for Amine 220, CHDMS & DEGS columns:

5-MeO-MMT

Isolation:

First isolated from *Phalaris arundinacea* found growing on the laboratory grounds of Wellcome Research Laboratories, Beckenham, Kent, England by Wilkinson 1958.

Reported Occurrences of 5-Methoxy-Nmethyltryptamine:

It would be a surprise if this alkaloid does not show up in the **Aizoaceae**; once someone starts looking.

Graminae

Arundo donax In flowers: Ghosal et al. 1971 In leaf: Ghosal et al. 1969 In rhizome: 16 mg from 700 grams. Ghosal et al. 1969 In dried plant in traces: Dutta & Ghosal 1967 Phalaris arundinacea L. Wilkinson 1958 first reported the natural occurrence of this alkaloid Also reported in some Norwegian P. arundinacea by Hovin et al. 1980 [also reported in a number of northern European P. arundinacea genotypes not included in this list.] Phalaris arundinacea Amounts not given. Detected by tlc in some Ottawa Synthetic cv. Woods & Clark 1971 Phalaris arundinacea (British Columbia) 0.001% by fresh weight (co-occurring with hordenine and gramine) Majak & Bose 1977 Phalaris arundinacea Beaverlodge, Alberta September harvest (Gramine present in all; DMT, bufotenine and 5-MeODMT absent in all) NRG 741 Sample 1 (0.115% dry wt.) NRG 741 Sample 2 (0.067% dry wt.) Castor (Mean of 18 plots- 0.025% dry wt.) [Range-0.009-0.051% dry wt.] Majak et al. 1978 Phalaris tuberosa cv. Australian Commercial In seedlings. Mack et al. 1988 Phalaris aquatica cv. Australian Commercial A minor alkaloid in 7 day old seedlings. Mulvena & Slaytor 1983. Phalaris arundinacea var. Frontier Minor. Audette et al. 1969 Phragmites australis (Cav.) Trin. ex Stend In rhizome. No details included. Wassel et al. 1985 Leguminosae Anadenanthera colubrina var. cebil [as Piptadenia *macrocarpa*] 0.1% isolated as oxalate from dry bark. Iacobucci & Rúveda 1964 Anadenanthera peregrina (L.) Spegazzini [No.24625; Origin: Boa Vista, Brazil] Bark- 0.0015% [15 mg. of 5-Methoxy-Nmethyltryptamine/ 100 gm. of dry bark] (36% of the total bark alkaloid.)

Leaf- Trace [13 mg. of total alkaloid/ 100 gm. of dry leaves: of which a trace was 5-methoxy-Nmethyltryptamine] Agurell *et al.* 1969 Bark of Brazilian *Piptadenia peregrina*.

Legler & Tschesche 1963 reported a mixture of 5-MeO-MMT and MMT as comprising 41% of the crude base. as *Piptadenia peregrina*

Bark collected in Colombia during 1956

5-MeO-MMT Holmstedt & Lindgren 1967

Anadenanthera peregrina. [R.E.Schultes 24625; Boa Vista, Brazil]

Bark **0.015%** 5-MeO-MMT [36% of 42 mg of total alkaloid/ 100 gm dry] Schultes *et al.* 1977

Desmodium gyrans

Leaf (97 mg. from 2 kg. dry) Ghosal et al. 1972a

Desmodium pulchellum

Whole plant (Minor alkaloid) Ghosal & Mukherjee 1964 (Amount not given. Ghosal & Mukherjee 1965) Stem-leaf (Amounts not given) Ghosal 1972a

Stem and leaf of young seedling [~0.010% by dry weight; 8% of 0.12% Total alkaloid] Ghosal *et al.* 1972c Stem and leaf of mature plant [0.154% by dry weight; 11% of 1.4% Total alkaloid] Ghosal *et al.* 1972c Root of young seedling. [Trace] Ghosal *et al.* 1972c Seeds (ripe) of mature plant [~0.002% by dry weight; 8% of 0.02% Total alkaloid] Ghosal *et al.* 1972c

Mimosa somnians

May be **in error?** Not observed by Gupta and coworkers

Myristicaceae

Virola based snuff: "*nyakwána*" [No. 24626; Origin: Tototobi, Brazil]

Traces [11,000 mg of total alkaloid / 100 gm. of snuff, of which a **trace** was 5-methoxy-N-methyl-tryptamine] Agurell *et al.* 1969

Virola rufula (A.DC) Warb. [No.24612; Origin: Manaus, Brazil]

Roots- 0.00576% [5.76 mg/ 100 gm. of dry roots] (4% of total alkaloid)

Agurell et al. 1969

Virola theiodora Warb. [No.24595; Origin: Manaus, Brazil]

Roots- 0.00255% [2.55 mg/ 100 gm. of dry roots] (15% of 17 mg total alkaloid/ 100 gm of dry root) Agurell *et al.* 1969

Identified in: Bufo alvarius;

20-23 μ g/gm. dried non-glandular skin. Erspamer *et al.* 1965

10-15 µg/gm dried skin Erspamer et al. 1967

Activity:

Not known. Shulgin & Shulgin 1997

Hallucinogenic according to Usdin & Efron 1979: #403, who cited Wilkinson 1958

This cannot be considered as any indication of proof, or even an evaluation, as Wilkinson simply mentioned an earlier report of the occurrence of "*staggers*" in sheep

5-MeO-MMT

5-MeO-DMT

confined to pastures of *Phalaris tuberosa*. *Besides* the fact that a direct causative link between this compound and the development of '*Phalaris* staggers' has **never** been investigated, much less proven (as Wilkinson performed absolutely NO type of pharmacological assessment); Wilkinson isolated this compound from *Phalaris* arundinacea.

Animal studies mentioned in Ghosal 1972a strongly *suggest* hallucinogenic activity.

Biochemical & Animal miscellany:

1% the oxytocic activity of serotonin

Moderate potency at disrupting conditioned behavior in rats. Less so than either bufotenine or 5-MeO-DMT. Taborsky & McIsaac 1964

Pharmacological studies:

Marczynski 1959 & 1960 Taborsky & McIsaac 1964

Metabolism:

Taborsky & McIsaac 1964

For further reading, see:

Shulgin & Shulgin 1997 Entry # 42; pp. 546-547.

5-Methoxy-N,N-dimethyltryptamine

3-[2-(N,N-Dimethyl)aminoethyl]-5-methoxy indole;
N,N-Dimethyl-5-methoxytryptamine;
5-Methoxy-3-[2(dimethylamino)ethyl]indole;
3-[2-(N,N-Dimethylamino)ethyl]-5-methoxy-indole;
Bufotenine methyl ether; O-Methyl-bufotenine;
O-Me-bufotenine; Bufotenine O-methyl ether;
Bufotenin-methyl-äther; Bufotenin methyl ester;
N,N-Dimethylserotonin;
5-Methoxy-N,N-dimethyl-tryptamine;
5-Methoxy-N,O-Timethyl-tryptamin;
5-Methoxy-N,O-TMS; OMB;
5-MeO-DMTPA;
M (used by Ott; also used by Shulgin as an abbreviation for mescaline and by several workers as an abbreviation for MDMA); CT 4334 (Inst. Pasteur).

WLN: T56 BMJ D2N1&1 GO1 Hayward: 6RR(OM)RRY5NHL=L(CCNM2)Y Usdin & Efron 1979: #401

CA Reg, No.: 001019450 [1019-45-0]

NIOSH # NL 7380000 Southon & Buckingham 1989: #B-00202

5-MeO-DMT is not a controlled substance

(At least not presently in the US; apparently it IS controlled in Japan, Germany and Australia.)

MW 218.17 Ott 1993 and Merck 9th

MW 218.298 Southon & Buckingham 1989

MW 218.30 Bergin et al. 1968; also Legler & Tschesche 1963

 $C_{13}H_{18}N_2O$

Elemental Analysis:

- Lit.: C, 71.52; H, 8.31; N, 12.83
- Exp.: C, 71.53; H, 8.49; N, 12.66
- Morimoto & Matsumoto 1966

C 71.51%, H 8.32%, N 12.84%, O 7.33% Ott 1993

Free base:

Prisms mp 65-66 [Recrystallized from ether-hexane (1:1) mp 66-67°]

Balsam & Voigtlander 1978

- mp 66-67° (Colorless prisms from ether-petroleum ether) Hoshino & Shimodaira 1936
- mp 67-68° Colorless solid (from ether-petroleum ether; 1:1) Miles *et al.* 1987
- mp 67-68° Short colorless prisms from benzene-cyclohexane. Legler & Tschesche 1963
- mp 67.5-68° Colorless prisms from hexane. Erspamer *et al.* 1967
- mp 67.5-68.5° Prismatic crystals from hexane Ott 1993
- mp 67.5-68.5° (Prisms from hexane containing some ether) Pachter *et al.* 1959
- mp 68° Transparent monoclinic prisms (dimeric) (Supplied by B. Holmstedt)

Bergin et al. 1968

- mp 68° Boit 1961
- mp 68° Colorless prisms from petroleum ether. Morimoto & Matsumoto 1966
- mp 69° Flakes from ether/ light petroleum Ghosal & Mukherjee 1964

and also Ghosal & Mukherjee 1966

- mp 69° Southon & Buckingham 1989
- mp 69° (Colorless plates from light petroleum-benzene (1:1) Banerjee & Ghosal 1969
- mp 69-70° (from hexane) Shulgin & Shulgin 1997

bp, 208-210° Southon & Buckingham 1989

- Vacuum distilled at 208-210° at 4 mm. Hoshino & Shimodaira 1936
- [Hoshino & Shimodaira also prepared from 5-MeO-DMT metho chloride by distilling in a strong vacuum at 240-250°.] Vacuum distilled at 160-170° at 0.6 mm. Crystallized on cooling. Shulgin & Shulgin 1997

Free base:

Off-white solid. Pungent but not ethereal like DMT, slightly dusty sweet smell.

Soluble in chloroform

Banerjee & Ghosal 1969

Soluble in ether and methylene chloride.

Pachter et al. 1959

Soluble in acetone, methanol and ethanol. Erspamer *et al.* 1967

Density: 1.123 gm/cm³ (Observed)/ 1.122 gm/cm³ (Calculated) Bergin *et al.* 1968

Chloroform-Water Partition coefficient: 3.30

Gessner *et al.* 1968 6.4 Gessner & Page 1962 see also Glennon *et al.* 1979

pKa 9.3 Ghosal & Mukherjee 1964

Hydrochloride:

 $C_{13}H_{18}N_2O.$ HCl MW 254.76 Highly birefringent colorless flat prisms (from ether/ ethanol 0° C) (Orthorhombic) $D_x = 1.218 \text{ gm/cm}^3$ $D_m = 1.221 \text{ gm/cm}^3$ mp 153° Falkenberg & Carlström 1971 mp 145-146° (from ethanol-ether) Shulgin & Shulgin 1997 [Formed by dissolving base in ether and passing a stream of

[Formed by dissolving base in ether and passing a stream of hydrogen chloride gas through the solution.]

Oxalate:

mp 172-175° Legler & Tschesche 1963 mp 172-176° (dec.) Sanders & Bush 1967 mp 173° (Ott 1993) (bioxalate) Gessner *et al.* 1968 mp 174° Gessner & Page 1962

mp 173° (bioxalate) Gessner et al. 1968

Monopicrate:

mp 176-177° Yellow Picrate mp 174-175°. Hoshino & Shimodaira 1936

Picrate:

Picrate Stout needles mp 166-168°. Recrystallized from methanol-acetone (1:1) mp 173-175. (mp 171-173° Synthesized from bufotenine) Balsam & Voigtlander 1978 mp 168° Orange picrate from EtOH (Reference material had mp 172°, mmp 168°) Banerjee & Ghosal 1969 mp 172° (orange-yellow needles from MeOH) Ghosal & Mukherjee 1964 & 1966 mp 173-175° Legler & Tschesche 1963 mp 175-176° Yellow needles from methanol. Morimoto & Matsumoto 1966 mp 175-176° Ott 1993 mp 175-176° (dec) (Brownish-red crystals slowly recrystallized from boiling water.) (Rapid recrystallization gave yellow-orange crystals and slightly lower mp) Erspamer et al. 1967 [brownish-red monopicrate assumed.] mp 175.5-176° (from ethanol) Culvenor et al. 1964 mp 176-177° Orange-yellow crystals (MeOH/ Me₂OH) Southon & Buckingham 1989 mp 176-177° Deulofeu & Rúveda 1971 cited Hoshino & Shimodaira 1936 mp 176-177º (Orange yellow crystals. mp after three recrystallizations from methanol-acetone) Pachter et al. 1959

mp 178° Takagi et al. 1979

Methiodide:

mp 181-182° Plates from methanol-acetone Ghosal & Mukherjee 1964 mp 181-182° (acetone-methanol 9:1) Ghosal & Mukherjee 1966 mp 183° (Colorless prisms) Hoshino & Shimodaira 1936 mp 183° Ott 1993 and Boit 1961 mp 186-188° Legler & Tschesche 1963

Metho cation:

Monopicrate: mp 170-171 Dipicrate: mp 103-104° Red needles

Flavianate:

mp 242° Red needles from methanol-water. Trimethylammonium chloride: mp 144° Hoshino & Shimodaira 1936 Says Wieland found 233°. Only Wieland citation included is Wieland *et al.* 1934.

Assays:

Analysis-: Pachter *et al.* 1959 cited Abramovitch & Shapiro 1956

Colorimetric reagents: See color reactions p. 149 Blue with 0.1% Xanthydrol. Gander *et al.* 1976

TLC & PC: See the Rf table p. 169-176 See also Holmstedt 1965

Preparative TLC:

Purified by preparative TLC using AcEt-*i*PrOH-10% NH₄OH (45: 35: 10) on Merck GF₂₅₄ Takagi *et al.* 1979 Preparative tlc on silica gel using Chloroform-Methanol-1N Ammonium hydroxide (80:15:1) (Rf 0.15) Miles *et al.* 1987

Column chromatography:

Eluted from Brockman alumina with benzene (from *D. pulchellum*) Ghosal & Mukherjee 1964

Eluted from Brockman neutral alumina with light petroleumbenzene (50:50).

Crystallized from same as colorless plates. (From *D. gangeticum*) Banerjee & Ghosal 1969

5-MeO-DMT was "*easily recovered*" from a 98% ethanol eluate using a column of alumina Erspamer *et al.* 1965

HPLC:

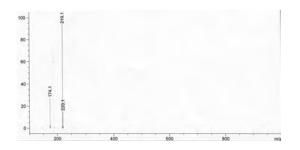
Balandrin *et al.* 1978 Kysilka & Wurst 1988 Kysilka *et al.* 1985

GLC:

Christian *et al.* 1975 GLC of HFB derivative: Benington *et al.* 1975 & Christian *et al.* 1975 & Vessman *et al.* 1969

MS: (see page 232)

5-MeO-DMT



GC-MS of 5-MeO-DMT

GC:

Holmstedt 1965

Audette *et al.* 1969: Retention times for Amine 220, CHDMS and DEGS columns

Verpoorte & Svendsen 1983 (relative retention times compared to tryptamine: p 156)

UV:

- $λ_{max}$ [log ε] (MeOH) = 278 [3.76], 296 [3.66] and 309 nm [3.54] Morimoto & Matsumoto 1966
- $\lambda_{\min} (\text{Ethanol}) \ 228 \ (\log \epsilon \ 4.35), \ 278 \ (\log \epsilon \ 3.83), \ 298 \ (\log \epsilon \ 3.70 \ \text{and shoulder at } 310 \ \text{nm. De Budowski} \ et \ al. \ 1974$
- λ_{max} (EtOH) 224, 277, 295 nm (log ε 4.46, 3.84, 3.76) Ghosal & Mukherjee 1964
- $λ_{max}$ (EtOH) 224, 277, 294 nm (log ε 4.45, 3.82, 3.77) Banerjee & Ghosal 1969
- λ_{max} (EtOH) 224, 277, 296 nm (log ϵ 4.46, 3.84, 3.76) Ghosal & Mukherjee 1966
- $\lambda_{max}(EtOH)$ 222, 278, 296, 309sh μm (log ϵ 2.51, 6.30, 5.10, 3.50)
- λ_{\min} (EtOH) 212, 248, 294 nm (log ε 2.18, 1.75, 5.05) Pachter *et al.* 1959

See also Erspamer et al. 1967

- λ_{max} of Xanthydrol reactive product (in CHCl₃): 590 nm
- λ_{\min} Xanthydrol reactive product (in CHCl₃): 425-500 nm. Gander *et al.* 1976

Fluorescence:

Pale yellow fluorescence under UV on paper. Banerjee & Ghosal 1969 [Fluorescence was not observed on tlc plates. Personal communication; J. Appleseed]

Fluorescence spectrum at pH 7.4:

Activation: 305 nm

Emission: 360 nm

Gessner & Page 1962

[Use of UV to localize spots was mentioned in Sanders & Bush 1967 but it was not clear if they did so using fluorescence or quenching.] See also Holmstedt 1965

IR:

De Budowski *et al.* 1974: (KBr) (chloroform) maxima at 3500, 3025, 2910, 2870, 2830, 2790, 1660, 1625, 1595, 1485, 1460, 1450 cm⁻¹ Erspamer *et al.* 1967 Ghosal & Mukherjee 1966 Williams *et al.* 1971 [m/e 58, 103, 117, 131. 143, 160, 173, 204, 218]
Holmstedt & Lindgren 1967
Couch & Williams 1972
Ikhiri *et al.* 1987
EI-MS: Jossang *et al.* 1991
MS of HFB derivative:
Vessman *et al.* 1969
MS of TMS derivative:
Narasimhachari *et al.* 1971
Walker & Mandell 1979
Mass fragmentation: Holmstedt & Lindgren 1967

GLC-MS:

Miles *et al.* 1987 m/e 218 (11%), 173 (2%), 160 (5%), 117 (4%), 103 (2%), 58 (100%)

NMR:

CDCl₃ De Budowski *et al.* 1974: page 577 ¹H-NMR. Balsam & Voigtlander 1978 Ikhiri *et al.* 1987 Jossang *et al.* 1991 Ghosal & Mukherjee 1964 (NMR, PMR)

Structure:

Crystal and molecular structure see: Falkenberg & Carlström 1971

Synthesis:

Benington 1958 Gessner & Page 1962 Gessner *et al.* 1968 used the method of Speeter & Anthony; reporting a 81% yield. Hoshino & Shimodaira 1936 Shulgin & Shulgin 1997: pages 531-532. Skaltsounis *et al.* 1983 Stoll *et al.* 1955

Obtained via two routes:

- 1) 5-Methoxyindolyl-3-acetonitrile \rightarrow 5-methoxyindolyl-3acetic acid \rightarrow 5-methoxytryptophol \rightarrow β -(5-methoxy-3indolyl)ethylbromide \rightarrow 5-methoxy-N,N-dimethyltryptamine. [route requires dimethylamine]
- 2) 5-Methoxytryptamine was reacted with methyl iodide and anhydrous sodium carbonate to yield β -(5-methoxy-3indolyl)ethyltrimethyl ammonium iodide which was then refluxed with an alcoholic solution of silver chloride yielding the chloride which was then heated in a strong vacuum at 240-250° to give 5-MeO-DMT.

Synthesis via methylation of bufotenine. Balsam & Voigtlander 1978

Can be obtained by direct O-methylation of bufotenine. While this can be performed with ease, another interesting approach would be to use the enzyme hydroxyindole-*O*-methyl transferase. See Axelrod & Weissbach 1961 for purification route.)

5-MeO-DMT

Another route would be to use the enzyme NMT (Nmethyl transferase) to convert serotonin (or 5-Hydroxy-N-methyltryptamine) to bufotenine then use the Omethyltransferase to obtain 5-MeO-DMT.

Similarly 5-MeO-tryptamine or 5-MeO-MMT could be converted to 5-MeO-DMT by the enzyme NMT.

See Axelrod 1962

and Mack & Slaytor 1979

Isolations:

First isolated from plants (*Dictyoloma incanescens* DC) by Pachter *et al.* 1959.

Isolation from *Bufo alvarius* by Erspamer *et al.* 1965 & 1967

Isolation from *Anadenanthera peregrina* by Legler & Tschesche 1963

Also see:

Baudouin et al. 1981

Erspamer et al. 1967

Ghosal & Mukherjee 1966

Ikhiri et al. 1987

Highest concentration thusfar reported from plants is the 0.678% reported from the dried roots of *Anadenanthera peregrina* in Schultes *et al.* 1977.

Highest reported concentration in nature; *Bufo alvarius* 5-15% in glandular tissues. Erspamer *et al.* 1967. In 1965 they had reported 6-16%.

Reported occurrences of 5-MeO-DMT:

Agaricaceae

Amanita citrina Gray

Tyler & Gröger 1964 (German specimens) Traces identified chromatographically.

Amanita porphyria (Fries) Secretan

Tyler & Gröger 1964 (German specimens) Traces identified chromatographically.

ANGIOSPERMS

Acanthaceae

Justicia pectoralis

In leaf. Shulgin & Shulgin 1997

Justicia pectoralis var. stenophylla

Leaf (dry) sampled 2 Nov. 1995 showed a faint blue band that co-chromatographed with 5-MeO-DMT. Plant from LER. tlc by Appleseed 1995. (Xanthydrol) We wonder if the above might not actually be the same alkaloid as McKenna observed rather than 5-MeO-DMT. While tlc has indicated DMT traces in this SAME plant, this was our only sampling to show this compound and no DMT.

Peristrophe hyssopifolia

Leaves (dry) showed a faint blue band that cochromatographed with 5-MeO-DMT in 1996 assays. Commercial plant. tlc by J. Appleseed 1996 (Xanthydrol) This plant resembles *Justicia pectoralis* in both general appearance (not color) and coumarin-like smell.

Aizoaceae

Aerial portions or whole plants used. Assays by J. Appleseed 1994-1996. (All identifications of 5-MeO-DMT used Xanthydrol.) These appear to be the only reports of 5-MeO-DMT occurring in the Aizoceae.

Delosperma acuminatum

May 1995 assay, Faint (Xanthydrol) Nov. 1995 assay: Dark blue and purple band corresponding to DMT and 5-MeO-DMT. (Xanthydrol) Other earlier positives used Ehrlich's.

Delosperma brittenae?

2 Nov. 1995 sample. Very nice dark band with Xanthydrol.

Delosperma cooperi

May 1995 assay (two sources) also in 2 Nov. 1995 sample. 3 positives total with Xanthydrol. Other positives using Ehrlichs; co-occurrence with DMT seen in 2 Nov. 1995 sample.) [Sasha did not confirm on material from another source.]

Delosperma ecklonis

Nice single blue band. Xanthydrol. Sept. 1996.

Delosperma hallii

2 Nov. 1995 sample. Dark band with Xanthydrol. *Delosperma harazianum* Audhali Plateau, Yemen

2 Nov. 1995 sampling. Dark band with Xanthydrol. (Co-occurring with DMT) Not observed In *D. harazianum* Shibam.)

Delosperma klinghardtiana

Nice band. Xanthydrol. Sept. 1996. (Co-occurrence with DMT)

Delosperma litorale

2 Nov. 1995 sample. Dark band with Xanthydrol. *Delosperma nubigenum*

9 May 1995. Weak 5-MeO band. (Xanthydrol)

Delosperma pageanum

(Same individual plant tested Dec. 1994) Positives: May and 2 Nov. 1995. [Faint in Nov. Good in May] (Xanthydrol.). Material harvested Aug. and Dec. 1995 tested positive (with DMT co-occurrence); Sept. 96 (also using Xanthydrol). Assay with Ehrlich's had shown decent band at this Rf in 5 Dec. 1994 sampling.



Graminae

5-MeO-DMT was detected in decent amounts in two abundant local weedy grasses which we have not yet been identified. 1996 tlc by J. Appleseed.

Arundo donax

Leaf and flower. Ghosal 1972a

Bromus spp. (Brome grass)

5-MeO-DMT appeared potentially present in at least some specimens of one *Bromus* sp. (*B. breviaristatus*) 1996 tle by J. Appleseed. [Species was grown by Giorgio Samorini from seeds provided by Trout and identified at seed maturity by Dr. F. Festi in 1999.]

Digitaria spp. (Crab grass)

5-MeO-DMT appeared potentially present in at least one local species; probably *D. sanguinalis*. Positive identification pending. 1996 tlc by J. Appleseed.

??? spp. (Wild Rye, Winter Rye, Rye Grass)
5-MeO-DMT appeared to be potentially present in several local species (2-4 spp.; including both annual & perennial ryes. Including the haying material called "coastal") 1996 tlc by J. Appleseed.

Phalaris data below is incomplete. We have dealt with this in some depth in a separate piece.

Alkaloid concentrations and proportions are highly variable from year to year and show dramatic seasonal fluctuations.

Concentrations between plant parts and first growth versus regrowth are also very different.

In many populations there may be marked differences in both the amounts present and in actual alkaloid profile from one plant to the next. (i.e. plants in the same population and arising from the same seeds may show completely different chemistry, not simply differing concentrations.)

See the amazing Festi & Samorini pieces listed and also our work on *Phalaris* for a review and overview of what is known so far.

Phalaris aquatica var. **AQ-1** (Italy) Weak occurrence reported (HPLC).

Festi & Samorini 1994b

Phalaris aquatica JLF (As *P. tuberosa*) Strong in leaf. 2 Nov. and 17 Sept. 1995. Dec 1995 Assay. tlc by J. Appleseed 1995

Killer Phalaris (At one point this was synonymous with **cv. Uneta** but has been in uncontrolled propagation for along enough, this is no longer certain.)

5-MeO-DMT predominated in 25 June, 17 Sept., 2 Nov. 1995 samples. [DMT was predominate alkaloid in Fall 1994 tlc.]

Phalaris aquatica

A major alkaloid in all samples they examined. Culvenor *et al.* 1964

Phalaris aquatica

Clone #R5 "large" amount of DMT co-occurring with "trace" amount of 2-methyl-1,2,3,4-tetrahydro- β -carboline.

[Clone, designated 405-9, originating with U.S. Regional Pasture Research Laboratory, University Park, Pennsylvania] Clone #R37 "trace" amount of 5-MeO-DMT co-occurring with "intermediate" amounts of hordenine and "large" amounts of 6-methoxy-2,9-dimethyl-1,2,3,4tetrahydro-β-carboline. [From "*highly diverse source* population used in plant breeding and genetic studies at the University of Minnesota, Department of Agronomy and Plant Genetics".] Clone #R51 "large" amount of 5-MeO-DMT as sole observed alkaloid. [Same source as R37] Clone #R96 "large" amount of 5-MeO-DMT co-occurring with "trace" amounts of hordenine and "trace" amounts of 6-methoxy-2,9-dimethyl-1,2,3,4tetrahydro-β-carboline. [Same source as R37] Gander et al. 1976. 5-MeO-DMT was not present in all clones examined (4 out of 12) Frahn & Illman 1973 Phalaris aquatica 5-MeO-DMT in leaf. 0.01-0.28% in material from California. Festi & Samorini 1994a cited Welch 1971 Phalaris aquatica cv. Australian Commercial [CPI 119305] A major alkaloid in 7 day old seedlings. Mulvena & Slaytor 1983 150 nmol / 100 seedlings. Mulvena & Slaytor 1983 In seedlings. Mack et al. 1988 Mature 0.05% dry wt. Baxter & Slaytor 1972 Phalaris aquatica cv. Sirocco 51 nmol / 100 seedlings. Mulvena & Slaytor 1983 Major base. Frahn & O'Keefe 1971 5-MeO-DMT in leaf. Ott 1994 cited Culvenor et al. 1964; Baxter & Slavtor 1972; Frahn & Illman 1973; Moore et al. 1967; Mulvena & Slaytor 1982; Oram & Williams 1967 Phalaris arundinacea 5-MeO-DMT in leaf and whole plant Barnes et al. 1971; Culvenor et al. 1964; Gander et al. 1976; Majak & Bose 1977; Majak et al. 1978; Marten et al. 1973; Williams et al. 1971. Many others. 0.0002-0.0067% in material from British Columbia. [Majak & Bose 1977] 0-0.02% in material from Minnesota. NRG741 was strongest of those tested & NRG721 the weakest. [Majak et al. 1978] Festi & Samorini 1994a Phalaris arundinacea JLF tlc by Johnny Appleseed Phalaris arundinacea L. P.I. 172442 Turkey (cv. "Turkey Red") 0.0025% to 0.045% total alkaloid by wet weight. 5-MeO is predominate alkaloid J.Appleseed (undated manuscript); "Ayahuasca analog plants of the temperate zone." see also Integration.) tlc by Johnny Appleseed: fall 1994, 25 June, 17 Sept., 2 Nov. 1995.

Sept 1996.

Phalaris arundinacea Amounts not given. Detected by tlc in some Ottawa Synthetic cv. Woods & Clark 1971 Phalaris brachystachys Reported in PI 202676 & PI 231044 Appleseed tlc evaluation of field trials using USDA seeds. Phalaris canariensis Reported in **PI 167261** in Appleseed's tlc evaluation of field trials using USDA seeds. Also at lower levels in PI 284185. Phalaris canariensis (Portugal) Traces reported (HPLC). Festi & Samorini 1994b *Phalaris stenoptera* (= *P. tuberosa* var. *stenoptera*) Variable amounts. Festi & Samorini 1994b cited Rendig et al. 1970 as finding 135-264 μ g/ml of expressed juice. *Phalaris truncata* (France) Traces reported (HPLC). Festi & Samorini 1994b Phalaris tuberosa L. (see as Phalaris aquatica) Sorghum halepense (Johnson Grass, Aleppo Grass, Egyptian Millet, Grass Sorghum, Means Grass) Low levels of 5-MeO-DMT in leaf only. 1996 tlc by J. Appleseed. (Xanthydrol) Samples collected spring and summer: Central Texas. Lauraceae Umbellularia californica (Hook & Arn.) Nutt. Unspecified concentration of 5-MeO-DMT in stembark. Needs confirmation. Rätsch 1998 Leguminosae Our reports concerning Acacia are the only mention that we can locate indicating that this alkaloid may occur in this genus. All assays by us must be regarded as tentative pending isolation and characterization All identification based on co-tlc with known reference standards and blue color with Xanthydrol spray. Acacia albida Traces tentatively observed in twigs. 5 Oct. 1995. tlc by J. Appleseed 1995 (Xanthydrol) [with suspected DMT] Acacia angustissima Trace amounts tentatively observed in roots (unconfirmed) in March 1995. tlc by J. Appleseed. Not observed in second assay. Trace amounts in seeds. tlc by Appleseed 1995 (all with Xanthydrol) Acacia auriculiformis Trace amounts tentatively observed in stem-bark (25 April 1995). tlc by J. Appleseed 1995 (A band at this Rf was also seen in roots [mislabeled as Guaiacum] in 2 Sept. 1994 assay but used Ehrlichs reagent which does not differentiate between DMT and 5-MeO-DMT.) Acacia cultriformis Blue band co-chromatographing with 5-MeO-DMT (Xanthydrol) Seen in branch stems, and in phyllodes, also in flowering spikes. Commercial florist's material.

Acacia difformis

Blue band co-chromatographing with 5-MeO-DMT (Xanthydrol) Roots of two year old seedlings. tlc by J. Appleseed 1996. Also in stems Sept 1996. Not observed in roots Sept. 1996.

Acacia farnesiana

Traces tentatively observed in green fruit. Not present in ripe fruit. (Xanthydrol) Also contained a suspected β -carboline. (blue under UV) 24 July 1995. tlc by J. Appleseed 1995.

Acacia maidenii

Traces observed in wood (October 1995);

Observed in twigs (26 July in phyllode and in mixed phyllode and twigs 27 Oct 1995) tlc by J. Appleseed 1995 (Xanthydrol) Co-occurring with suspected DMT in all samples of phyllodes/twig but sole alkaloid seen when just using twigs. Not observed in bark or root bark.

[Note: *Acacias* such as this one do not produce leaves except when young or stressed. What are thought of as being leaves are actually phyllodes; a specially modified petiole (the short stalk at the leaf base.)] *Acacia nilotica*

Acacia nilotica

Faint traces observed in stem, roots and leaves. Assayed separately (Sept 1996; Xanthydrol)

Acacia obtusifolia

In some samples as a minor component. Apparently unpublished work. Pers. comm. with Snu Voogelbreinder. Needs confirmation.

Appeared present in one sample of leaves/twigs looked at by Mulga; Not observed in bark or root bark.

Acacia victoriae Strong blue band co-chromatographing with 5-MeO-

DMT (Xanthydrol) Roots of two year old seedlings. tlc by J. Appleseed 1996.

Albizia procera

Suspected 5-MeO-DMT was seen with 2 other bands in a large sample of leaf. Three year old seedling.) 1996 tlc by J. Appleseed (Xanthydrol)

No alkaloids observed in stem, root or root bark in 1994.

Anadenanthera species. [G. Seitz, 1965]

Seedlings

0.001% 5-MeO-DMT [4% of 29 mg of total alkaloid/ 100 gm dry.]

Schultes et al. 1977

Anadenanthera species. [As Piptadenia sp.; Caspar, 1964; Guaporé, Brazil; Tupari.]

Seeds:

0.11% 5-MeO-DMT [85% of 13 mg of total alkaloid/ 100 gm]

Schultes et al. 1977

Anadenanthera colubrina

5-MeO-DMT reported in snuff thought to originate from this species. Conflicting reports. Most accounts have found only bufotenine in the seeds but several reports exist claiming the presence of DMT and/or 5-MeO-DMT.

Torres et al. 1991 reported the detection of all three in snuff powder recovered from archaeological sites in Argentina believed to have been derived from A. colubring seeds. Both A. colubring and A. colubring var. cebil occur in Argentina. While it is not clear which Torres and coworkers referred to; the latter is implied. No analysis of seeds or verifiable plant materials were reported in Torres et al. 1991. Anadenanthera peregrina (L.) Spegazzini [No.24625; Origin: Boa Vista, Brazil]: Bark- 0.025% [25 mg. of 5-Methoxy-N,N-dimethyltryptamine/ 100 gm. of dry bark] Leaf- 0.006% [6 mg. of 5-Methoxy-N,N-dimethyltryptamine/ 100 gm. of dry leaves] Agurell et al. 1969 (Bark- as *Piptadenia peregrina*) Legler & Tschesche 1963 examined bark and reported 5-MeO-DMT formed 32% of the crude base. A mixture of 5-MeO-MMT and MMT comprised 41%. Used Brazilian material. Seeds collected in western Brazil in the Rio Branco region during 1953 5-MeO-DMT Holmstedt & Lindgren 1967 Bark collected in Colombia during 1956 5-MeO-DMT Holmstedt & Lindgren 1967 Anadenanthera peregrina [R.E. Schultes, S. von R. Altschul et B. Holmstedt, sin. num.; La Carolina, Barrio St. Just, near San Juan, Puerto Rico, December 1974. Same colony as Schultes 26363.] Mature seeds collected in March 1975; hill behind El Comandante horse-racing track. 1975 analysis (5 months after collection): 5-MeO-DMT- 1% of total alkaloid. 1977 analysis of same material could detect only bufotenine (80% of total alkaloid in 1975). Schultes et al. 1977 Anadenanthera peregrina [R.E. Schultes 26363; La Carolina, Barrio St. Just, near San Juan, Puerto Rico, Dec. 1972] Immature seeds collected December 1972 0.04% 5-MeO-DMT [19% of 209 mg of total alkaloid/100 gm drySeedlings 0.024% 5-MeO-DMT [95% of 25 mg of total alkaloid/100 gm dry] Pods without seeds

0.012% 5-MeO-DMT [91% of 13 mg of total alkaloid/100 gm dry]

Leaves 0.094% 5-MeO-DMT [88% of 107 mg of total alkaloid/ 100 gm dry]

Twigs 0.0357% 5-MeO-DMT [94% of 38 mg of total alkaloid/ 100 gm dry]

Bark [0.41% total alkaloid]

0.39% 5-MeO-DMT [95% of 410 mg of total alkaloid/ 100 gm dry.] Roots [0.69% total alkaloid]

0.678% 5-MeO-DMT [97% of 699 mg of total alkaloid/ 100 gm dry]

Schultes et al. 1977

Anadenanthera peregrina. [R.E.Schultes 24625; Boa Vista, Brazil]

Leaves

0.00624% 5-MeO-DMT [48% of 13 mg of total alkaloid/ 100 gm dry]

Bark

0.025% 5-MeO-DMT [59% of 42 mg of total alkaloid/ 100 gm dry]

Schultes et al. 1977

Snuff: "epena"

Obtained from the Waica by George Seitz. 5-MeO-DMT was the major alkaloid.

Bufotenine was present as a minor component. Because of this, the claimed plant source (*Virola*) has been questioned. Holmstedt 1965

Many snuff using people use both snuffs, although many have preferences one way or the other. While idle speculation; we wonder if preparation might not have involved both plants, or if the snuff witnessed as made from one source (*Virola*) may not have been contaminated with residuals of another snuff (*Anadenanthera*) during processing or storage, involved the admixture of other Myristicaeaous plants or as-yet unidentified plants, or perhaps have been derived from an altogether different but as yet unknown source.

Snuff: "paricà"

Snuff as prepared by Piaroa Indians (collected 1955) 5-MeO-DMT [with DMT and Bufotenine]

Holmstedt & Lindgren 1967

Snuff: "yopo"

Snuff prepared, by the Pixasi-teri (or Bisashi-teri) of Upper Orinoco, from the seeds of an *Anadenanthera* species.

5-MeO-DMT [with Bufotenine]

Marini-Bettòlo et al. 1964

Snuff: "yopo"

Snuff collected in Colombia (collected 1956) 5-MeO-DMT [with DMT and Bufotenine]

Holmstedt & Lindgren 1967

Caesalpinia gilliesii

In stem-bark (with 1 other band) 1996 assays. In roots. (Nice band with two others present) June 1995, also 1996. tlc by J. Appleseed (Xanthydrol)

Caesalpinia pulcherrima

In flowers and buds. (26 August 1995) tlc by J. Appleseed 1995. [August 1994 of a small sample of dead flower petals did not detect this alkaloid but did show a faint indolic band at a lower Rf.]

In roots (nice band) June 1995, also 1996. tlc by J. Appleseed 1995 (Xanthydrol)

Desmodium sp. (Wild species, not yet positively identified; Austin, Tx.)

Trace amounts in aerial portions (co-occurring with suspected DMT).

tlc with Xanthydrol spray, 24 June 1995.

Desmodium gangeticum DC

Aerial parts (0.057% by wet weight; 0.57 gm. from 1 kg. of fresh wet material.) Banerjee & Ghosal 1969 Stem-leaf Ghosal 1972a

Green Plant (Stem and Leaf) Ghosal & Bhattacharya 1972

Our assays of seed grown plants detected 5-MeO-DMT as present in trace amounts in seeds

(May 1995 assay).

No alkaloids were observed in this species until after it was 2 years old.

5-MeO-DMT was observed in small amounts in roots and also in stems (May 1995);

Also in leaf Feb. 1995 (using large red leaves left from winter) and May 1995 using normally colored new but full sized leaves.

Leaves also tested positive (faint) in Nov. 1995. tle by J. Appleseed using Xanthydrol.

Desmodium gyrans DC

Leaves (35+ mg. from 2 kg) Ghosal *et al.* 1972a In stem/ leaves Ghosal 1972a Roots Ghosal 1972a

Desmodium pulchellum Bentham ex Baker

Whole plant (0.2-0.25% by dry weight.) Ghosal & Mukherjee 1964 (Major alkaloid. Ghosal & Mukherjee 1965) (Amount not given. Plates crystallized from 8.36 grams of impure chromatographic fraction residue; from 4 kg of dried whole plant.) Ghosal & Mukherjee 1966

Stem and leaf of young seedling [Trace.] Ghosal *et al.* 1972c

Stem and leaf of mature plant [0.476% by dry weight; 34% of 1.4% Total alkaloid] Ghosal *et al.* 1972c

Root of mature plant [0.132% by dry weight; 12% of 1.1% Total alkaloid] [Also, in same paper; 1.8 kg. of dried roots yielded 0.23 gm; i.e. \sim 0.013% by dry weight.] Ghosal *et al.* 1972c

Seeds (ripe) of mature plant [0.002% by dry weight; 10% of 0.02% Total alkaloid] Ghosal *et al.* 1972c Root, stem-leaf and flower (Amounts not given) Ghosal 1972a

Desmodium racemosum Thunberg

Whole plant. (Amount not given) Hsü *et al.* 1982 cited Hsü 1970 [Source article has not been located. Title is suspect.]

Lespedeza bicolor Turcaninow var japonica Nakai

In leaf and root bark. Smith cited Goto *et al.* 1958 Present in root bark (Less than 0.1%). Morimoto & Matsumoto 1966

Lespedeza bicolor

Our assays did not detect any alkaloids in any parts during the first year. (1994) We did detect 5-MeO-DMT in both seeds and mixed seeds and pods. (May 1995) tlc by J. Appleseed. See comments under DMT as we may have mistaken the two as tlc used Ehrlichs.

Our reports of the occurrence of 5-MeO-DMT in the genus *Mimosa* are the only reports of this alkaloid in this genus that we can locate.

All assays must be regarded as tentative identifications pending isolation and characterization.

Mimosa pudica

In low amounts in stem and leaf after the first year (November harvest of 15 month old plants).

Concentrations were higher in the roots (August harvest). Assays 2 Nov. 1995

Very young seedlings (whole plant) tested in 1996 showed a very dark suspected 5-MeO-DMT band. In branches; 1996 assays.

tlc by J. Appleseed 1995-6.

[DMT did not start to show up in assays until after second year, at which time it was present in leaf and root.]

Mimosa somnians

Maybe appearing **in error**? Not observed by Gupta and coworkers

Mimosa tenuiflora (Willd.) Poir. The listing of 5-MeO-DMT **is in error**. Meckes-Lozoya ran it as a pure reference sample only.

Mucuna pruriens

0.01% in fresh leaves Ghosal *et al.* 1971 In leaf, seed, stem and roots. Ott cited Bhattacharya *et al.* 1971; Ghosal 1972a; Ghosal *et al.* 1971 In root, stem-leaf and pod Ghosal 1972a

Piptadenia see Anadenanthera

Linaceae

Hugonia oreogena Schlecter Traces of 5-MeO-DMT in rootbark. (0.5 mg from 140 grams) Ikhiri et al. 1987

Malpighiaceae

Diplopterys cabrerana (Cuatrecasas) Gates (misidentified as **Banisteriopsis rusbyana see** Gates 1982) Traces of 5-MeO-DMT in leaf and 0.0035% in dried stem. Agurell *et al.* 1968a

Myristicaceae

Horsfieldiana superba (Hk. f. et Th.) Warb. (Collected in September at Sandakan, Sabah, Malaysia.) 5-MeO-DMT as minor leaf alkaloid [0.0007%; 20 mg from 2.8 kg of leaves.] Roots and bark apparently unexamined.

Jossang *et al.* 1991

Iryanthera ulei Warburg

Trace amounts in bark Holmstedt et al. 1980

Osteophloem platyspermum (DC) Warb. [Schultes & Rodrigues #26126]

One of 3 alkaloids in 0.62 mg of total alkaloid from 100 grams of dry bark. (Brazil);

Other material: [Schultes & Tovar #7095 (Peru)] tested negative with Ehrlich's and Dragendorff's. Holmstedt *et al.* 1980

McKenna *et al.* 1984b tested a samples of leaves [DMK-78] but failed to find. Did report N-Methyltryptophan methyl ester

Virola based arrow poison (Yanomamö).

8% 5-MeO-DMT by dry wt. Macrae & Towers (1984) cited Galeffi *et al.* 1983

Virola [?]

Snuff: "epéna"

Snuff as prepared by Araraibo Indians (collected 1965) 5-MeO-DMT [with DMT]

Snuff as prepared by Tucano Indians (collected 1965) 5-MeO-DMT [with DMT and 5-MeO-MMT] Snuff as prepared by Waica Indians (collected 1965) 5-MeO-DMT [with MMT and DMT] Holmstedt & Lindgren 1967 Virola based snuff: "epena" [No.24574; Origin: Rio Cauaburi, Brazil] 0.5% 5-MeO-DMT [515 mg. of 5-Methoxy-N,Ndimethyltryptamine / 100 gm. of snuff] Agurell et al. 1969 Virola based snuff: "nyakwána" [No. 24626; Origin: Tototobi, Brazil] 9.68% 5-MeO-DMT [9,680 mg. of 5-Methoxy-N,N-dimethyltryptamine/ 100 gm. of snuff] Agurell et al. 1969 Yopo snuff Suspected to have originated from Virola (based on chemistry- not vouchered) showed only 5-MeO-DMT- 26 mg from 1.8 gm of snuff (1.4%). De Budowski et al. 1974 Paste: Virola sp. (No voucher; "oo'-koey"; La Chorrera) 5-MeO-DMT 1.19 mg/ ml DMT also present at 0.3 mg/ ml McKenna et al. 1984a Virola calophylla Bark collected in Manaus, Brazil during 1964. 5-MeO-DMT Holmstedt & Lindgren 1967 Virola calophylla Warb. [Schultes No.24603; Origin: Manaus, Brazil]: Bark- 0.0008% [0.81 mg. of 5-MeO-DMT/ 100 gm. of dry bark] Traces in dry roots. 130 µg from 100 grams of dry bark. [Not observed in flowering shoots or in leaves.] Agurell et al. 1969 [Prance #14947. Origin: Rio Cuieras, Brazil]: In bark (low levels). In roots (even lower levels) Holmstedt et al. 1980 [DMK-46]: In bark. McKenna et al. 1984b Virola calophylla [Collected on March 12; Santa Rose, Iquitos, on Rio Momon in Peru.] Wood was found to contain: 5-MeO-DMT (major alkaloid) co-occurring with 2-Methyl-6-methoxy-1,2,3,4-tetrahydro-β-carboline and Nicotine. No amounts were given. Miles et al. 1987 Virola calophylloidea Markgraf [Plowman, Schultes and Tovar #7093; Origin: Pebas, Peru.] DMT in bark Holmstedt et al. 1980 Virola elongata (Spruce ex Bentham) Warb. [5-MeO-DMT has not been reported from all specigm.] mens analyzed.] Bark-0.245 mg of 5-MeO-DMT/ gm dry bark. Macrae & Towers 1984 cited McKenna et al. 1984a



Acacia victoriae seeds

[Plowman, Schultes and Tovar #6920; Origin: Pebas, Peru.]:

Present in resin and phloem (Not in leaves) Holmstedt et al. (1980)

[Plowman, Schultes and Tovar #7263; Origin: Pebas, Peru.]:

Present in paste and phloem.

[Plowman, Schultes and Tovar #7092; Origin: Pebas, Peru.]: Trace in bark. Holmstedt et al. 1980

[DMK-59]:

In bark (not in leaves-DMT and MMT present in leaves). McKenna et al. 1984b

[DMK-67] and [DMK-68] and [DMK-69]:

5-MeO-DMT in leaf. (Negative assay in bark) McKenna et al. 1984b

Paste: Virola elongata

(DMK-59; Alfredo Moreno no. 1) 5-MeO-DMT 2.03 mg/ ml (Sole base present) McKenna et al. 1984a Virola multinerva Ducke [No.24614; Origin: Manaus, Brazil] Roots- 0.00059% [0.59 mg. of 5-MeO-DMT/ 100 gm. of dry roots] Agurell et al. 1969 Virola multinerva Ducke [Many collections of this species were reported devoid of alkaloid. Others contained only traces of DMT.] [Schultes No. 24616; Origin Manáos, Brazil]: Trace amounts in dry roots. (590 µg per 100 grams) Holmstedt et al. 1980 Virola peruviana (DC) Warburg In bark and paste Holmstedt et al. 1980 5-MeO-DMT in plant. Part and amount not given. Lai et al. 1973 Virola rufula (A.DC) Warb. [Schultes No.24612; Origin: Manaus, Brazil] Bark- 0.008%? [8 mg. of 5-methoxy-N,N-dimethyltryptamine/ 100 gm. of dry bark] (given in Holmstedt et al. (1980) as 190 mg per 100 grams of dry bark which, if true, would be 0.19%.) Roots- 0.135% [135 mg. of 5-methoxy-N,N-dimethyltryptamine / 100 gm. of dry roots] Agurell et al. 1969 Virola sebifera Aublet [DMK-40]: In bark. McKenna et al. 1984b

Virola theiodora Warb. [Schultes No. 24595; Origin: Manaus, Brazil] All by dry weight.

Bark- 0.11% [108 mg. of 5-MeO-DMT/ 100 gm.] Roots- 0.011% [11 mg. of 5-methoxy-DMT/ 100

Traces in leaves

Agurell et al. 1969

5-MeO-DMT

[Schultes No. 24626; Origin: Tototobi, Brazil]

Bark- 0.062% [62 mg. of 5-MeO-DMT / 100 gm.]

Agurell et al. 1969

Ott 1993 cited Holmstedt 1965 [Schultes No. 24613; Origin: Manaus, Brazil]

Roots- 0.001% [1 mg per 100 gm.]

Leaves- <0.001% [Less than 1 mg per 100 grams. (Holmstedt *et al.* 1980 listed only DMT in leaves for this same collection)]

Virola venosa (Bentham) Warburg [No. 24613; Origin: Manaus, Brazil]

Roots- 0.001% [1 mg per 100 gm.]

Leaves- <0.001% [Less than 1 mg per 100 grams. (Holmstedt *et al.* 1980 listed only DMT in leaves for this same collection)] Agurell *et al.* 1969

Agurell *et al.* 1969

Virola sp. indet. aff. *venosa* [Plowman, Schultes and Tovar (7094]; Origin: Pebas, Peru]:

Bark- 0.0042% [4.18 mg per 100 grams of dry roots] Leaf- 0.0003% [0.29 mg per 100 grams of dry leaves] Holmstedt *et al.* 1980

Rutaceae

Dictyoloma incanescens DC

0.04% isolated from dry bark collected in winter near Rio de Janeiro. Voucher prepared. Pachter *et al.* 1959

Dutaillyea drupacea (Baillon) Hartley

New Caledonia

5-MeO-DMT was the sole alkaloid (98%) in the leaves- 0.04%. [The published value of 0.4%, repeatedly appearing in the literature, **IS A TYPO**] 5-MeO-DMT was absent from trunk-bark Baudouin *et al.* 1981

Dutaillyea oreophila (Baillon) Sevenet-Pusset New Caledonia

Leaves- 0.02% 5-MeO-DMT by dry weight. Major alkaloid (40% of 0.05% total alkaloids) Co-occurring with 2-Methyl-6-methoxy-tetrahydro- β -carboline (0.005% dry wt.), Hordenine (0.0125% dry wt.) and Kokusagine (0.0125% dry wt.)

5-MeO-DMT was absent from trunk-bark Baudouin *et al.* 1981

Evodia rutaecarpa Bentham [also Hooker f. ex Thomas]

In unripe fruit. Takagi *et al.* 1979 recovered 30 mg from 10 kg unripe fruit. Yu *et al.* 1997 reported 0.00015% by dry weight in the unripe fruit. [15 mg from 3 kg.]

Also present in the roots. Shulgin & Shulgin 1997 *Meliocope leptococca* (Baill.) Guillaumin [= *Evodia leptococca* Baill.]

5-MeO-DMT (0.21% dry wt.) is the major alkaloid (35% of 0.61% total alkaloids) in aerial parts Accompanied by 0.03% 5-MeO-DMT-N-oxide and 0.024% 2-Methyl-6-methoxy-tetrahydro- β -carboline, Acronydine (0.61%), Kokugasiginine (0.183%), Acronycidine (0.012%), Melicopicine (0.043%), Melicopidine (0.018%) and Acronycine (0.006%). Skaltsounis *et al.* 1983

Acacia cultriformis flowering

Pilocarpus organensis Occhioni & Rizzini

In leaf (Main alkaloid- 0.41%. Total combined alkaloid content of 1.06%) Balsam & Voigtlander 1978 Shulgin & Shulgin 1997 and Ott 1994 both noted that other *Pilocarpus* species are known to contain the poisonous cholinergic Pilocarpine.

Zygophyllaceae

Tribulus terrestris

Whole plant (trace) Dec. 1995 assays. In assays of isolated parts it was observed only in the seeds (faint). 1996 assays by Appleseed. All collections in summer.

Animals:

Bufonidae

Bufo alvarius

- Cutaneous glands were found to contain as much as 60 to 160 mg per gram of dry tissue
- 1.0-3.5 mg/ gm of non-glandular skin. Erspamer *et al.* 1965
- As much as 5-15% of parotid and coxal glands by dry weight.
- Ranged from 50-150 mg per gram of large cutaneous glands (dry) and from 0.42-3.5 mg per gram in the rest of the dry skin.

Erspamer et al. 1967 (occurs with its N-sulfate)

Occurrence of 5-MeO-DMT in humans:

- Sample analysis of human cerebrospinal fluid included 5-MeO-DMT. Christian *et al.* 1975
- Found in cerebrospinal fluid of some psychotics and a few normal people by Corbett *et al.* 1978. Narasimhachari *et al.* 1971b reported it was more common in psychotics than normals
- Also detected in some patients by Smythies *et al.* 1979 but it is not clear in their account exactly which subjects showed its presence and which did not.







Bufo alvarius (female)



For some related studies concerning the natural occurrence of 5-MeO-DMT in humans: Guchhait 1976 (produced *in vitro* using human pineal glands recovered during autopsies)

Rosengarten & Friedhoff 1976 Saavedra & Axelrod 1972 Smythies *et al.* 1979

Bufo alvarius (male)

5-MeO-DMT

Activity:

Hallucinogenic. Usdin & Efron 1979 #401 cited Gessner *et al.* 1961

"Hallucinogenic" No colors or visuals (except at higher doses than many people will tolerate at which point visuals are both pronounced and intense) but most decidedly a hallucinogen.

Occasional brief but <u>TRUE</u> hallucinations. True hallucinations being distinguished from phosphene originating patterns and colors and similar visual phenomena by being open-eyed waking visual or auditory hallucinations with no immediate awareness of unreality. On the rare occasions that we experienced them, it was always been just after the peak has passed and seemingly normal consciousness had returned or was returning.

Despite the above observations, when mixed with *Cannabis* or harmala alkaloids there is a radical enhancement of both the intensity of colors and the movement within the phosphene field, especially if fairly large dosages (~20 or so mg) are administered over the course of a few minutes by smoking it suspended on the herb.

Occasional auditory hallucinations (especially when mixed with *Cannabis*).

Distressing if optimal dosage exceeded. Under 10 mg. for most (if taken all-at-once).

Dramatic amyl nitrate intensity rush at onset.

Incredible mental stimulation and sense of well-being if within the individual's optimum dosage window. Well loved by some, detested by many. [I suspect excessive dosages to be the culprit in this last case.]

Amounts too low to normally produce effects have been found to be fully active after pre-dosing with small amounts of LSD-25.

Observations by Trout

See Ott 2001a & 2001c for many more observations and bioassay details. Ott reported it active orally, insufflated, via buccal absorption and greatly increased in effects by the addition of even small amounts of harmala alkaloids.

Dose:

200 µg/kg smoked/insufflated or IV. Duration 10-20 minutes.

Callaway & McKenna 1998

5-10 mg. smoked "Entheogenic" Ott 1993 & "Psychoptic" 1996: Entry #9 citing Shulgin in DeSmet 1983

6-20 mg smoked.

2-3 mg iv

Shulgin & Shulgin 1997: Entry #38, pages 531-538.

Ott 2001c conducted an interesting series of bioassays:

- 30 mg oral: onset in 12-18 min, peak in 40 min., fading at 48 min., over in an hour. Found to be equipotent with 10 mg 5-MeO-DMT combined with 40 mg harmine (other subjects required 60 mg of harmine).
- Shulgin had reported 35 mg oral was inactive suggesting some people require larger doses of the tryptamine.
- 10 mg insufflated (0.14 mg/ kg) was found to be the "*visionary threshold*" & dramatic potentiation reported with 5, 10 or 20 mg of harmaline HCl. onset in 3-4 min., peak in 35-40 min., fading by 50 min, over in 60-70 minutes.

10 mg sublingually was found indistinguishable from 10 mg insufflated. Found was potentiated with similarly small doses of harmine or harmaline. 5 mg with the MAOI was found equipotent with 10 mg by itself. [Interestingly Ott found 7.5 mg of harmaline was active via this route when taken alone but harmine was not. (As HCl but dose given is as "*base-equivalent*".) Oddly Ott found both inactive when insufflated.]

Ott has stated that as little as 5 mg of 5-MeO-DMT is perceptible when used as pharmahuasca. [i.e. Co-administered with an effective dose of harmala alkaloid(s).] See more details in Ott 2001 a & c.

Duration:

Shulgin & Shulgin 1997 1-2 hr (When smoking onset is rapid, with a peak that will pass within a very few minutes; baseline often in 30-60 minutes.) See observations by Ott above for other routes.

Receptor site specificity:

Full agonist at 5-HT_{1A}, 5-HT_{1C}, 5-HT_{1D} & 5-HT₂ Callaway & McKenna 1998 5-HT receptor interactions & specificities:

McKenna *et al.* 1990

See also:

Glennon *et al.* 1979 & 1994 Sanders & Bush 1967

Biochemical & Animal miscellany:

5-MeO-DMT is an effective anti-feedant for insects. Miles *et al.* 1987

Pharmacology & pharmacognosy of 5-Methoxy-N,Ndimethyltryptamine:

Effects on conditioned avoidance (disruption):

Gessner & Page 1962

Gessner et al. 1968

Pharmacological evaluation in animals:

- Oxytocic, biphasic action on blood pressure, BOL inhibition & radically increased behavioral disturbances. Gessner *et al.* 1961
- 5-MeO-DMT shows 8% of the oxytocic activity of serotonin. Taborsky & McIsaac 1964

Distribution, metabolism & excretion (in rat): Sanders & Bush 1967

Preferentially metabolized by MAO-A:

Squires 1975

Suzuki et al. 1981

Biosynthesis and/or excretion:

Guchhait 1976 Mandel & Walker 1974 Rosengarten & Friedhoff 1976

Tolerance:

It is unclear if any tolerance develops to 5-MeO-DMT. If it does, it is *extremely* short-lived as repeated administrations appear to possess full activity; as does administration of a large dose via a steady series of small doses. See comments p. 231-232.

Pharmacological overview:

Benington et al. 1965

Erspamer 1961 was cited by Culvenor *et al.* 1964. [Main focus is on 5-HT but does include a few points of interest]

Falkenberg & Carlström 1971 cited Gessner & Page 1962 and Gessner 1970

In animals

Ahlenius & Larsson 1991 DeMontigny & Aghajanian 1977

Holmstedt & Lindgren 1967

Toxicity:

Animals assumed abnormal postures and movements (like walking backwards)

Jumping action, clonic and tonic convulsions and tremor were common to all animals before death.

Ho et al. 1970

"Toxic agent causing staggers-like poisoning of sheep in Australia." Southon & Buckingham 1989

Despite its frequent presentation as an established fact, this is currently **supposition** rather than proven.

Superficial evidence of symptomology has been repeatedly used to claim this despite the complete and utter failure to produce chronic stagger effects, or degenerative damage such as lesions in the livers or nervous systems of animals, by administration of pure DMT and/or 5-MeO-DMT (even if given in fatal doses).

Intensive breeding efforts to produce low tryptamine strains **OF** *Phalaris ARUNDINACEA* were undertaken, in the US, ostensibly to decrease animal fatalities, in spite of the following demonstrable facts:

1. Highest numbers of animal deaths occur during the times when the tryptamine content was proven to be lowest (by separate workers in Australia and US),

2. "Low alkaloid" strains of *Phalaris aquatica* are found to produce higher numbers of dead livestock than "*high alkaloid*" strains when compared directly,

3. *Phalaris arundinacea* has NEVER caused any occurrence of staggers in the US; and only two incidences worldwide [Simpson *et al.* 1969 & Ulvund 1985] despite it clearly being on record as deliberately cultivated & utilized for forage for over 200 years.

Many workers appeared to operate as if the causative link between 5-MeO-DMT and *Phalaris* staggers was already proven (almost as soon as the presence of dimethylated tryptamines was first reported)

The known ability of DMT and 5-MeO-DMT to produce a green pigmentation *in vitro* using enzyme preparations of cells has caused a similar green pigmentation found in animals which died from chronic *Phalaris* staggers to become both regarded as diagnostic evidence and proof that such methylated tryptamines are the causative agents despite Gallagher's utter and complete failure to observe this pigmentation in any animals afflicted with acute or peracute staggers (This was true whether the animals recovered or died).

It must be remembered that Gallagher was **only** able to mimic some of the acute stagger symptoms with the N-dimethylated tryptamines and was entirely unable to reproduce either the chronic effects or degenerative neurological changes like lesions. I might also mention that bufotenine was reported by Gallagher to be more active than DMT and produces a brown pigmentation (as does serotonin) under similar *in vitro* conditions.

[Using bufotenine bioxalate, Gessner et al. 1960 reported that deaths of rodents given intraperitoneal administrations of 6 divided doses, of what WOULD have been a lethal dose of given all at once, were delayed by around a week with an apparent return to normalcy after the acute effects wore off. They could not find anything unusual except for "acute thoracic kyphosis accompanied by a cervical hyperextension of the spine" as well as dehydration and loss of around 1/3 of the total body weight. (4 out of 5 rats died this way) The length of time suggests it might be worth looking into some sort of secondary problem resulting from the mechanical aspects of the interperitoneal injection itself in terms of actual site of delivery or infection potential? I have witnessed this performed on rats a good number of times. A quick jab somewhere into the belly is about all the care in target selection that is usually given. It might be added that said rats are not totally compliant in this matter as evidenced by their struggling and screams.]

Work by Gallagher is claimed to have established lethality using oral administration of pure compounds, but it must be stressed that at no point in Gallagher's work is it actually stated that any death or deaths were produced by oral administration although he does claim effects began in 6 minutes via this route. [As opposed to the 6-12-(72) hours known to be required for onset of symptoms, and up to months before death, after grazing. Recovery from the effects of administered 5-MeO-DMT is complete within an hour.]

Interestingly, Gallagher also states that bufotenine readily crosses the blood brain barrier.

Clearly something needs closer scrutiny.

One idea commonly tossed around is that the presence of β carbolines orally activate DMT/5-MeO-DMT. While logical, this property is entirely unevaluated for any of the β -carbolines reported from *Phalaris*. Directly toxic effects *are* suspected from 1 or 2 of the quaternary β -carbolines.

The action of the quaternary N-methylated tryptamines that are known to sometimes represent up to 5% of the alkaloids present in *P. aquatica* (under poorly defined circumstances) must also be considered due to their potential for cyclization.

IF the β -carbolines turn out to possess MAOI properties, their interaction with many other compounds present must also be considered.

Other alkaloids known to also be (at least sometimes) present in *Phalaris*: 3-methylindole (known to produce Bovine Pulmonary Emphysema in cattle), indoleacetic acid, 5-methoxyindoleacetic acid, tryptophan, 5-methoxy-tryptophan, (the preceding 4 can be readily converted to 3-methylindole by the gut flora of cattle but not by sheep or goats), gramine, substituted gramine derivatives and hordenine.

Hordenine and gramine are both known to produce toxic effects in livestock and the combination of hordenine with an MAOI would be more likely to precipitate a hypertensive crisis, than combining tryptamines with MAO inhibitors. [Also more likely than if combining mescaline with an MAOI]

Another factor to consider is the known anticholinesterase activity of bufotenine and several of the *Phalaris* β -carbolines; see Ghosal *et al.* 1977.

The potential presence of lysergic acid type fungal products, from not only grass ergotisms but also fungal endophytes of common pasturage components, would also contribute to toxicity, especially if combined with an MAOI.

Combine this gut-full of chemicals with yet other compounds found in the mixed pasturage *Phalaris* invariably occurs in - *Agrostis, Bromus, Carex, Cyperus, Fescue, Scirpus, Sorghum, Tribulus,* Ryegrasses etc.. - all of which have been incredibly dismissed as trivial; yet, all are known to produce alkaloids toxic to livestock; most inducing stagger effects on their own], add the potential metabolites produced by gut flora, and there rapidly appears to be **many** potential sources of toxicity and contributing factors possibly involved with *Phalaris*; all of which, for the largest part, have been entirely disregarded in the peculiar quest to blame dimethylated tryptamines.

My best guess is a complex of drug interactions may be involved; hordenine, lysergic acid derivatives, β carbolines & mixed indoles are hardly a recommended combination.

Incredibly, perhaps due to the politics involved, we still do not fully understand this <u>economically impor-</u> tant disorder despite over **3 decades of work and a hell of a lot of money** thrown at it attempting to address the tryptamine "*problem*".

Amazingly, Gallagher went on to further conclude that exposure to DMT/5-MeO-DMT produced a situation where later stimulation by dogs would induce <u>delayed</u> death due to a sensitization to adrenaline somehow caused by <u>prior</u> exposure to the tryptamines.

Another not so insignificant point is that *Phalaris* staggers are known to be produced by strains that do not contain appreciable amounts of the N-dimethylated tryptamines, containing instead β -carbolines and gramine and/or hordenine.

Other workers have put considerable effort into linking decreased palatability with the tryptamines claiming the decrease in palatability was evidence of toxicity; on the assumption that sheep would supposedly prefer to eat nontoxic plants.

If sheep were truly this discriminating, it would seem that *Phalaris* staggers and a host of other livestock poisonings would be less of a problem. [I must note that, like hordenine and gramine, 5-MeO-DMT is a proven feeding deterrent. Due to their TASTE.]

The majority of the evidence is strongly against the chronic form of *Phalaris* staggers syndrome being a product of tryptamine ingestion but few seem to care for discovering the truth; politics rule.

Consult our forthcoming work on the Genus *Phalaris* for more details of some perplexingly bad science.

See Festi & Samorini and/or Shulgin & Shulgin 1997 for alternate scenarios that are AT LEAST as plausible, if not far better supported.

More recent work strongly suggests that one or more of up to several novel furanobisindoles may be involved in the toxicity. Several have been characterized. See the recent published accounts by N. Anderton, C.A. Bourke, S.M. Colegate and R. Oram for more details concerning their research.



Generalized structure of furanobisindoles

LD₅₀ of 5-MeO-DMT

115 mg/ kg ip in mouse Ho *et al.* 1970 LD_{50} iv is said to be ¹/₄ oral but the claim lacked a reference.

There is more recently one human death claimed to be associated with 5-MeO-hoasca but we suggest that this intriguing account needs a closer look as nothing concrete about the actual cause of death was established.

See Sklerov et al. 2005.

5-MeO-DMT-N-oxide

 $C_{13}H_{18}N_2O_2$

MW 234.297 Southon & Buckingham 1989: #B-00202

Free base:

Pale violet oil

Pale brown gum (eluted from Brockman alumina with MeOH).

Dull red color under UV.

Hygroscopic Ghosal & Mukherjee 1966

- Water soluble; Soluble in dilute acetic acid Ghosal & Mukherjee 1965 & 1966
- Soluble in methanol and chloroform Banerjee & Ghosal 1969
- Soluble in chloroform Ghosal & Mukherjee 1965

Picrate

mp 158° Southon & Buckingham 1989

mp 158° Crimson-red picrate (Fine needles from

ethanol) Ghosal & Mukherjee 1965 mp 158-159° Red picrate from ethanol

Banerjee & Ghosal 1969

Assays:

Colorimetric reagents: See p. 149

TLC & PC: See RF table p. 169-176

NMR: Skaltsounis et al. 1983

Formation &/or Isolations (5-MeO-DMT ⇔ 5-MeO-DMT-N-oxide interconversions):

- 5-MeO-DMT (50 mg.) in ethanol (2 ml.) was treated with 3 ml. of a solution of H_2O_2 in ethanol (2 ml. of 30% H_2O_2 in 8 ml. ethanol). The mixture was kept at room temperature for two hours then diluted with ether (5-MeO-DMT-N-oxide separated as a flocculent solid) Ghosal & Mukherjee 1966
- An aqueous solution of 5-MeO-DMT-oxide was acidified with acetic acid, reduced with zinc dust, the solution brought up to pH 9 with ammonia then extracted with CHCl₂.
- "Almost quantitative recovery of the base in the form of 5-Methoxy-N,N-Dimethyltryptamine was effected." [See procedure under DMT-N-oxide] Ghosal & Mukherjee 1965
- An aqueous solution of 5-MeO-DMT-N-oxide was treated with ferrous sulfate at 60° and yielded formaldehyde, and a mixture of 5-MeO-DMT and 5-MeO-MMT Ghosal & Mukherjee 1965
- 5-MeO-DMT-N-oxide (64 mg) in aqueous acetic acid (5 ml.) and ferrous sulfate heptahydrate (198 mg.) in water was kept 40 minutes over a steam bath (60-65°) after which the mixture was cooled in ice.

Solid NaOH was added to bring pH up to 12.

- Liberated bases extracted with CHCl₃. The chloroform was washed with a little cold water, dried and solvent removed.
- The residue chromatographed on Brockman alumina (18x1 cm.)
- Benzene-CHCl₃ (80:20) eluted 6-Methoxy-2-methyl-H⁴- β -carboline.
- Ether-MeOH (90:10) eluted 5-MeOMMT and 5-MeODMT.
- (Separated as picrates via fractional crystallization from methanol. 5-MeO-MMT picrate was the more soluble.) Ghosal & Mukherjee 1966

Reported Occurrences of 5-MeO-DMT-N-oxide: Leguminosae

Desmodium gangeticum

Aerial parts [0.18 gm. from 1 kg of fresh wet material] Banerjee & Ghosal 1969 Stem-leaf Ghosal 1972a Green Plant (Stem and Leaf) Ghosal & Bhattacharya 1972

Desmodium gyrans

Leaves (trace) Ghosal et al. 1972a

Desmodium pulchellum

Whole plant (Minor alkaloid) [First reported occurrence of this alkaloid.] Ghosal & Mukherjee 1965 [Minor alkaloid: 17 mg. as an impure violet oil, contaminated with gramine, was obtained from 4 kg. of dried whole plant. Ghosal & Mukherjee 1966] Stem and leaf of mature plant [0.070% by dry weight; 5% of 1.4% Total alkaloid] Ghosal *et al.* 1972c Stem-leaf (Amounts not given) Ghosal 1972a and Ghosal *et al.* 1972e

Lespedeza bicolor var. japonica

Identified in root bark. Morimoto & Matsumoto 1966

Mucuna pruriens

Leaf and fruit. Shulgin & Shulgin 1997 Rutaceae

Meliocope leptococca (Baill.) Guillaumin [= Evodia leptococca Baill.]
0.03% 5-MeO-DMT-N-oxide by dry weight in the aerial parts. Skaltsounis *et al.* 1983

Activity:

Should be active if smoked but we have found no reported evaluations.

O-Methyl-nordehydrobufotenine

6-Methoxy-5-methyl-1,2,3,4,5tetrahydropyrrolo[4,3,2-d,e]-quinoline

Hayward: 6RRR(OM)Y 5NMLLY5=NHY WLN: T566 1A L CM HN&T&J H JO1

 $C_{12}H_{14}N_{2}O$

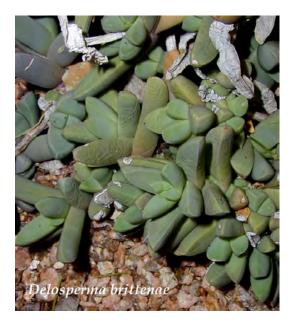
Activity:

Hallucinogenic in animals.

409 in Usdin & Efron 1979; citing Lee et al. 1969

Despite not actually ever being proven to be a hallucinogen in either animals or humans, this is interesting as, while it remains unreported as a naturally occurring alkaloid, this molecule is a simple rearrangement product of dehydrobufotenine.

It suggests that it too may be physiologically active albeit possibly moderately toxic.



Trout's Notes on Tryptamines: 5-Substitution







Several surprising positive TLC assays; uncovered by Appleseed



Caesalpinia pulcherrima (above)

Caesalpinia gilliesii (below)



"More than you need to know?"



Justicia pectoralis flowering

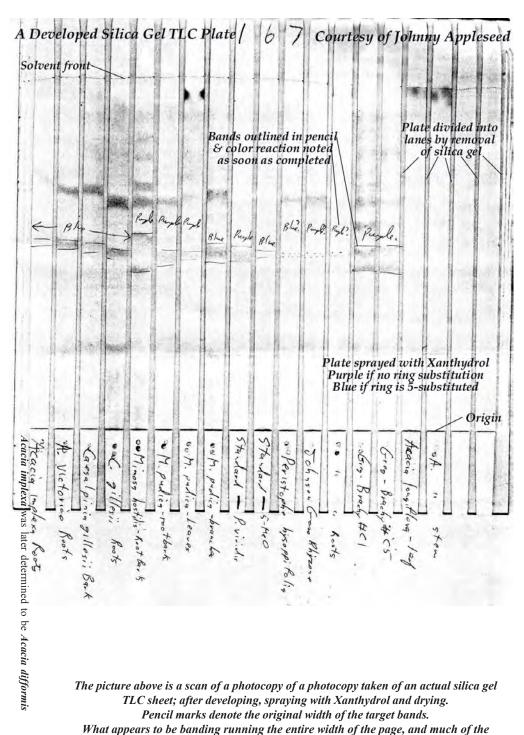
Psychotria viridis Photo upper right by Des Tramacchi

Desmodium pulchellum Photo below by Des Tramacchi









Assays for some Indoles

shadowing, are actually artifacts of the photocopier reproduction.

Trout's Notes FS-X7



Acacia farnesiana



Digitaria sanguinalis (Crabgrass)







Calliandra pentandra



Color reactions & chromophoretic reagents

As reported for some Indolic Compounds.

Color names are very subjective; dependent upon many variables including, but not limited to, cultural and personal definitions. I left them entirely as described. [A densitometer will make it much more accurate; See Ehman.]

p-Dimethylaminobenzaldehyde is used in a variety of concentrations and forms (PDAB reagent, Ehrlich's [also many different versions], Van Urk's etc...) When there is a variance, formulation was as given by the reference cited. See details and test procedure under the listed reference in the enclosed list of Chromophoretic Reagents. Test results were taken from the literature. Any errors, present there, will be intact here.

Reagent	Reaction	[Medium]	Ref.	
Gramine				
PDAB	No immediate color	[on paper]	[19]	
	Pink	[on paper]	[55]	
Dragendorff's	Orange	[on paper]	[19]	
Ehrlich's	Slowly turning Pink	[silica gel & cellulose]	[56]	
	Weak. Slow brown	[on paper]	[40]	
	Weak. Slow brown	[on silica gel]	[56]	
	Pink	[on silica gel]	[1]	
	No immediate color, slowly turns ye	llow	[9]	
Fluorescence with PENE	Violet (under 254nm UV)	[silica gel (using PENE)]	[30]	
diazotized p-Nitroaniline	Mauve	[Avicel]	[29]	
Nitrite-Nitric acid	Brown (under UV)	[on paper]	[40]	
	Yellow-brown (Visible)	[on paper]	[40]	
Xanthydrol	Pink (also by Appleseed)	[silica gel & cellulose]	[56]	
-	No reaction	[on paper]	[33]	
Van Urk-Salkowski	Reddish blue (Pansy Violet)	[when dry, on silica gel]	[10]	
	Bluish Violet (Methyl violet)	[when wet, on silica gel]	[10]	
<u>Tryptamine</u>				
DMCA	Purple red	[on silica gel]	[46]	
Ehrlich	Purple (1 hr)/ Blue (overnight)	[on silica gel]	[41]	
	Reddish-purple	[on paper]	[40]	
Ethanolic PDAB	Purple	[pure compound]	[42]	
Fluorescamine	Aquamarine under 360 nm UV	[on Kieselgel]	[58]	
	Violet (under 254nm UV)	[silica gel (using PENE)]	[30]	
NNCD reagent	Orange-yellow; Orange	[on paper]	[11; 11b]	
Ninhydrin	Brown-red	[on paper]	[51]	
Ninhydrin-Acetic acid	Bright Green-blue (sea-green)	[on paper under UV]	[40]	
i (inity and i lootio uola	Brownish purple (Visible)	[on paper]	[40]	
Ninhydrin-Cadmium acetate				
	Pink-Gray	[on silica gel]	[41]	
Ninhydrin-Pyridine	Reddish-purple	[on paper]	[40]	
diazotized p-Nitroaniline	Orange-yellow	[on paper]	[11]	
Pauly's	Yellow	[on paper]	[11]	
	Faint Brown-red (5 min)	[on silica gel]	[41]	
PDAB-TS	Yellow-green	[pure compound]	[42]	
o-Phthalaldehyde	Faint orange under 350 nm UV	[on silica gel]	[41]	
-	No reaction	[on silica gel]	[2]	
Van Urk-Salkowski	Blue (Princes Blue)	[when dry, on silica gel]	[10]	
	Blue (Oriental Blue)	[when wet, on silica gel]	[10]	

Trout's Notes FS-X7

Reagent	Reaction	[Medium]	Ref.	
<u>MMT</u>				
CAS	Brilliant yellow under UV	[on silica gel]	[31 a]	
Chloranil	Gray (light)→Gray	[on silica gel]	[26]	
CNTNF	Gray	[on silica gel]	[26]	
PDAB	Blue	[on silica gel]	[7]	
Ehrlich's Reagent	Blue-Gray	[on silica gel & cellulose.]		
	Reddish purple	[(as HCl) on paper]	[40]	
Fluoranil	Blue	[on silica gel]	[26]	
Fluorescence with PENE	Violet (under 254nm UV)	[silica gel (using PENE)]	[30]	
HNS	None	[on silica gel]	[26]	
Iodoplatinate	Purple-blue	[on silica gel]	[7]	
Acidified iodoplatinate	Positive	[on silica gel]	[8]	
Marquis Test	Orange	[on pure compound.]	[8]	
Ninhydrin-Acetic acid	weak Yellow-green (UV)	[(as HCl) on paper]	[40]	
	Brownish-purple (Visible)	[(as HCl) on paper]	[40]	
Ninhydrin-Pyridine	weak Brown	[on paper]	[40]	
diazotized <i>p</i> -Nitroaniline	5	[on paper]	[11b]	
NNCD reagent	weak Orange	[on paper]	[11b]	
Sodium nitroprusside Sulfuric acid test	Blue Yellow	[tlc]	[4]	
		[on pure compound]	[8]	
TACOT	Orange-pink→Pink	[on silica gel]	[26]	
TCNE	Gray	[on silica gel]	[26]	
TetNF	Brown	[on silica gel]	[26]	
TNB	$Yellow \rightarrow Yellow (light)$	[on silica gel]	[26]	
TNF No without have 1	Brown	[on silica gel]	[26]	
Xanthydrol VanUrk's	Purple	[on silica gel & cellulose]	[56]	
Valiotk S	Purple	[pure, paper & silica gel]	[28]	
DMT				
Chloranil	None	[on silica gel]	[26]	
CNTNF	Gray (light)	[on silica gel]	[26]	
PDAB (as 0.5 gm. in 50 m	l of EtOH-Sulfuric acid (6:4) – freshl	y prepared)		
Dissolved alkaloid forms red solution,				
	turns to violet when diluted with wa		[8]	
PDAB	Blue	[on paper]	[19]	
	Blue	[on silica gel]	[7]	
	Violet	[on silica gel]	[3]	
DMBA	Green [Dark blue in few hr.]	[on silica gel; under UV]	[52]	
Dragendorff's	Positive with spray	[on silica gel]	[8]	
	Red-Brown	[on paper]	[9]	
Ehrlich's	Orange Reddish purple	[on silica gel] [(as acetate) on paper]	[21] [40]	
Limiter S	Blue	[on silica gel]	[21]	
	Purple	[on silica gel]	[21] [8]	
	Blue-Gray	[silica gel & cellulose]	[56]	
	Mauve turning Dark Blue	[on paper]	[9]	
Ethanolic PDAB	Purple	[pure compound]	[42]	
Fluoranil	Purple	[on silica gel]	[26]	
Fluorescence with PENE	Violet (under 254nm UV)	[silica gel (using PENE)]	[30]	
HNS	None	[on silica gel]	[26]	
			-	

Reagent	Reaction	[Medium]	Ref.
DMT	(continued)		
Iodine vapor	Red-brown	[on paper]	[9]
Iodoplatinate	Purple	[on silica gel]	[39]
I II II III III	Blue	[on silica gel]	[7]
Acidified Iodoplatinate	Positive	[on silica gel]	[8]
Marquis Reagent	GreenYellow	[silica gel]	[52]
Marquis Reagent	Gray brown	[on silica gel]	[8]
Marquis Test	Orange	[on pure compound]	[8]
Ninhydrin	Slight purple-Gray	[on silica gel]	[39]
	nt present) [Most investiga		
	rith tertiary amines, purple b		
Ninhydrin-Acetic acid	No fluorescence (UV)	[(as acetate) on paper]	[40]
5	No color (Visible) [(as acet		[40]
HNO ₃ atmosphere	Yellow	[on silica gel]	[39]
α -Nitroso- β -naphthol-nit			
1 1	Negative	[on silica gel]	[21]
	Weak Brown	[on paper]	[9]
		orted no reaction with this	
diazotized p-Nitroaniline		[on paper]	[11b]
NNCD reagent	weak Orange	[on paper]	[11b]
PDAB-TS	Yellow	[pure compound]	[42]
Sodium acetate solution,	2,6-Dibromo-p-benzoquino		
, ,	Orange brown	[on silica gel]	[18]
TACOT	Purple (light)	[on silica gel]	[26]
TCBI	Brown-green	[on silica gel]	[29, 53]
TCNE	Brown (light and fading)	[on silica gel]	[26]
TetNF	Brown (light)	[on silica gel]	[26]
TNB	Yellow→Brown	[on silica gel]	[26]
TNF	Brown (light)	[on silica gel]	[26]
	used successfully by many		
Van Urk Reagent	Blue	[tlc]	[8]
Xanthydrol	Purple	[silica gel & cellulose]	[56]
	Purple	[tlc/ on paper]	[8, 13]
	Pink	[on paper]	[14]
	Lavender	[on paper]	[15]
		[on haber]	[10]
DMT-N-oxide			
PDAB	Mauve to Blue	[on paper]	[19]
PDAB & HCl gas	Violet	r. L.L. 1	[36]
Dragendorff's	Dirty orange	[on silica gel]	[21]
Ehrlich's	Cherry Red	[on silica gel/ on paper]	[21, 20]
α -Nitroso- β -naphthol- ni			1 7 9
1 1	Negative	[on silica gel]	[21]
	Dull brown	[on paper]	[20]
DMT methocation			
Xanthydrol	Purple	[on paper]	[13]
5	1		

Reagent	Reaction	[Medium]	Ref.
DET			
CNTNF	Gray (light)	[on silica gel]	[26]
DMBA	Green [Dark blue in few hrs]	[silica gel; 366 nm UV]	[52]
Dragendorff	Positive	[silica gel]	[8]
Ehrlich's	Reddish purple	[(as HCl) on paper]	[40]
	Violet; deep blue after storage	[on kieselguhr]	[17]
Ethanolic PDAB	Purple	[pure compound]	[42]
Fluoranil	Purple	[on silica gel]	[26]
acidified IPA	Positive	[silica gel]	[8]
Mandelin's test	$Greygreen \rightarrow Yellow$	[pure compound]	[8]
Marquis reagent	GreenYellow	[silica gel]	[52]
marquis reagent	Brown	[silica gel]	[8 <u>]</u>
Marquis test	Yellow→Brown	[pure compound]	[8]
-		[(as HCl) on paper]	
Ninhydrin-Acetic acid	No fluorescence (UV)	[(as HCl) on paper]	[40] [40]
PDAB	No color (Visible) Violet	[(as FICT) on paper] [on silica gel]	[40] [3]
PDAB-TS	Yellow	[pure compound]	[3] [42]
TACOT	Purple (light)	[on silica gel]	[42]
TCNE	Brown (light and fading)	[on silica gel]	[26] [26]
TetNF	Brown (light)	[on silica gel]	[26]
	Yellow—Brown		
TNB		[on silica gel]	[26]
TNF Vanthudnal	Brown (light)	[on silica gel]	[26]
Xanthydrol	Purple (assumed)	[on silica gel]	NA
Psilocin			
DMBA	Purple-blue (faster with heat)	[silica gel; under UV]	[52]
DMCA	Greenish-grey	[silica gel]	[46]
Ehrlichs	Bluish purple	[on silica gel]	[31]
	Darker purple than PSOP	[on silica gel]	[6]
	Red-violet	[on Kieselgel]	[58]
Ehrlichs, modified	Blue-violet	[on paper]	[22]
,	Blue-purple	[on silica gel]	[43]
Ethanolic PDAB	Deep blue	[pure compound]	[42]
Keller	Olive-green turning Grey	[pure compound]	[50]
Marquis test	Green-brown	[pure compound]	[44, 8]
Mytilus edulis gill plate	oxidase		
	Blue	[on pure compound]	[61]
None	Sky blue	[on silica gel]	[6]
α -Nitroso- β -naphthol-n	itrous acid Brownish-orange	[on paper]	[62]
Pauly's	Orange-red	[on paper?]	[5]
Pauly's	deep Red-orange turning Red-brow	vn [on silica gel]	[20]
PDAB	Blue-violet	[on paper?]	[3]
	Blue	[on paper?]	[2b]
	Blue-grey	[on silica gel]	[3]
	Purple tinged with red	[on silica gel]	[32]
	immediate strong Blue	[on silica gel]	[54]
	Grey-Violet	[on Kieselgel GF]	[44]
PDAB-TS	Brown	[pure compound]	[42]
pTSA	Similar to Ehrlichs but brownish c		[6]
Van Urk	Grey-blue	[pure compound]	[50]
	Faint blue	[in tlc]	[8]

Trout's Notes on Tryptamines: Color Reactions

Reagent	Reaction	[Medium]	Ref.
Psilocybin			
DMBA	Blue (faster with heat)	[on silica gel; under UV]	[52]
DMCA	Reddish	[on silica gel]	[46]
DMCA	Violet	[on cellulose]	[46]
Ehrlichs	Reddish-purple eventually turning	purple	
		[on silica gel]	[31]
	Reddish-violet turning blue-violet	[on silica gel]	[25]
	Pinkish brown then brownish purpl	le	
		[silica gel & cellulose]	[6]
	Pink turning violet	[on Kieselgel & on paper]	[37]
	Red-violet	[on Kiesel gel]	[58]
	Violet	[in tlc]	[16]
Ehrlichs, modified	Violet	[on paper]	[22]
	Brown	[on silica gel]	[43]
Ethanolic PDAB	Purple	[pure compound]	[42]
Keller	Red-violet	[on paper]	[50]
Iodine	Brown	[in tlc]	[16]
IPA	PurpleBrown	[on silica gel]	[7]
Marquis reagent	Orange	[on silica gel]	[52]
Marquis test	Orange	[pure compound]	[8]
None	Sky-blue	[on silica gel]	[6]
Pauley's	No reaction	[on silica gel]	[54]
PDAB	Purple-blue	[on silica gel]	[7, 25]
	Blue-grey	[on silica gel]	[3]
	dark Blue to Purple	[on silica gel]	[32]
	Reddish-purple fading to Violet	[on silica gel]	[24]
	Dark Blue	[on silica gel GF]	[38]
	Reddish-grey	[on Kieselgel GF]	[44]
PDAB-TS	Yellow-green	[pure compound]	[42]
Prochazka	Grey-blue	[in tlc]	[16]
pTSA	Similar to Ehrlichs but with a brown	nish cast	
		[as dip on silica gel]	[6]
Van Urk	Pinkish-brown; very weak	[on paper]	[50]
Van Urk	Grey-Violet	[in tlc]	[8]

Absorbs (quenches) at 254 nm UV **[2]** (Christiansen & Rasmussen 1982b used 254 nm for UV detection) (2% Iodine in Methanol as a spray will also work)

Also reacts with Fentons Reagent (i.e FeSO₄ followed by 5M H₂O₂ sprayed on paper. But some details aren't clear enough due to my lack of fluency in German. See Venker & Schmidt 1962 for details)



Psilocybe cubensis Photo copyright Perfect Fungi Europe Reproduced with permission



Photo by Anonymous

Reagent	Reaction	[Medium]	Ref.
<u>Serotonin</u>			
	nce Bright yellow under 254nm UV	[on Avicel]	[29]
Alkaline Silver	Black	[creatinine sulfate: paper]	[40]
Brentamine Fast Red B	Yellow	[on paper]	[27]
trans Cinnamaldehyde	Cherry-Red [Reddish]	[on silica gel and others]	[45]
DMCA	Blue	[on paper]	[23]
DMCA	Bright Blue	[on silica gel]	[46]
PDAB	Blue	[on paper or with tlc]	[12]
PDAB	Purple	[on paper]	[60]
Echtrotsalz B	Pinkish violet	[on paper]	[12]
Ehrlich	Blue (1 hr)/ Purple (overnight)	[on silica gel]	[41]
	Rapid. Bluish-purple \rightarrow Gray \rightarrow Blue		[40]
	Blue	[on paper]	[27, 49]
	Pink-violet turning intense deep blu		
, , , _		[as oxalate: on paper]	[24]
Fast Blue salt B	Reddish-purple [Purple-violet]	[on silica gel and others]	[45]
Fast Blue BB salt	Purple	[on silica gel]	[34]
Fluorescamine	Aquamarine under 360 nm UV	[on Kieselgel]	[58]
	Pale yellow (under 254nm UV)	[silica gel (using PENE)]	[30]
Gibbs	Violet blue	[on paper]	[12]
	Blue	[on paper]	[49]
Naphthanil diazoblue B	Blue	[on paper]	[27]
Ninhydrin-Acetic acid	Brownish-purple [Visible]	[creatinine sulfate: paper]	[40]
Ninhydrin-Cadmium ace	Bright Green-blue [UV] tate Reddish-Gray (5 min.)	[creatinine sulfate: paper] [on silica gel]	[40] [41]
Ninhydrin-Pyridine	Gray-purple	[creatinine sulfate: paper]	[41] [40]
diazotized <i>p</i> -Nitroaniline		[on paper]	[40] [11b]
NNCD	Peach red	[on paper or with tlc]	[11b] [11b, 12]
Pauly	Wine red	[on paper]	[110, 12]
1 uury	Dark reddish violet	[on silica gel and others]	[45]
	Reddish-brown	[creatinine sulfate: paper]	[40]
	Orange (5 min)	[on silica gel]	[41]
PDAB	Purple	[on silica gel]	[34]
o-Phthalaldehyde	Strong yellow under UV (350 nm)	[on silica gel]	[41]
Sulphanilic acid/ Na nitri		[on paper]	[27]
Van Urk-Salkowski	Violet blue (Bluebird Blue)	[when dry, on silica gel]	[10]
	Bluish Green (Capri Blue)	[when wet, on silica gel]	[10]
<u>5-OH-MMT</u>			
	nce Bright yellow (under 254nm UV)		[29]
Alkaline Silver	Black	[as oxalate on paper]	[40]
trans Cinnamaldehyde	Cherry-Red [Reddish]	[on silica gel and others]	[45]
PDAB	Blue	[on paper or with tlc]	[12]
Echtrotsalz B	Pinkish violet	[on paper]	[12]
Ehrlich	Gray-pink (1 hr); Blue (overnight)	[on silica gel]	[41]
	Gray-purple	[oxalate on paper]	[40]
Fast Blue salt B	Reddish-purple [Purple-violet]	[on silica gel and others]	[45]
	Pale yellow (under 254nm UV)	[silica gel (using PENE)]	[30]
Gibbs Ninbudrin Apotio poid	Violet blue	[on paper]	[12] [40]
Ninhydrin-Acetic acid	Weak brown [Visible] No fluorescence [UV]	[as oxalate on paper]	[40] [40]
Ninhydrin-Cadmium ace		[as oxalate on paper] [on silica gel]	[40] [41]
Ninhydrin-Pyridine	Weak brown	[as oxalate on paper]	[41] [40]
rannyarm-r yranne	weak brown	[us oxatate on paper]	נידן

Trout's Notes on Tryptamines: Color Reactions

diazotized p-Nitroanilin	[on paper]	[11]	
α -Nitroso- β -naphtholnitro	ous acid Bright violet	[on paper]	[62]
NNCD	Peach red	[on paper or with tlc]	[12]
Pauly	Wine red	[on paper]	[12]
	Dark reddish violet	[on silica gel and others]	[45]
	Orange (5 min)	[on silica gel]	[41]
	Reddish brown	[oxalate on paper]	[40]
o-Phthalaldehyde	Strong yellow under UV (350 nm)	[on silica gel]	[41]

Bufotenine

Duiotenine			
	nce Bright yellow (under 254nm UV		[29]
Alkaline Silver	Rapid. Black	[as acetate on paper]	[40]
trans Cinnamaldehyde	Cherry-Red [Reddish]	[on silica gel and others]	[45]
Cinnamaldehyde + HCl	Orange	[paper]	[59]
DMCA	Violet	[on silica gel]	[46]
PDAB	Blue	[on paper or with tlc]	[12]
	Blue	[on paper]	[19]
	Purple	[paper]	[59]
Dragendorff	Red-Brown	[on paper]	[9]
2	Orange		[63]
Echtrotsalz B	Pinkish violet	[on paper]	[12]
Ehrlich's	Blue-Purple	[on silica gel & cellulose]	[56]
	Dark Blue	[on paper]	[9]
	Rapid. Bluish-purple→Blue	[as acetate on paper]	[40]
	Red-Pink (1 hr)/ Blue (overnight)	[on silica gel]	[41]
Ethanolic PDAB	Blue	[pure compound]	[42]
FeCl3 Test	Blue	[pure compound]	[63]
Fast Blue salt B	Reddish-purple [Purple-violet]	[on silica gel and others]	[45]
	Pale yellow (under 254nm UV)	[silica gel (using PENE)]	[30]
Gibbs	Violet blue	[on paper]	[12]
	cetate White on turquoise backgrou		[45]
Iodine vapor	Red-brown	[on paper]	[9]
Keller	Reddish turning Blue	[on pure compound]	[50]
Mytilus edulis gill plate of		[on pure compound]	[61]
Ninhydrin-Acetic acid	No reaction [Visible]	[as acetate on paper]	[40]
Tunnyurni-Accile acid	No reaction [UV]	[as acetate on paper]	[40]
Ninhydrin-Cadmium acet		[on silica gel]	[41]
α -Nitroso- β -naphtholnit		[on paper]	[9]
a ranoso p naphaionna	Bright violet	[on paper]	[62]
NNCD	Peach red	[on paper or with tlc]	[12]
Pauly	Wine red	[on paper]	[12]
1 duly	Orange (5 min)	[on silica gel]	[41]
	Dark reddish violet	[on silica gel & others]	[45]
	Reddish-brown	[as acetate on paper]	[40]
	Orange-pink	[paper]	[59]
PDAB-TS	Green	[pure compound]	[42]
<i>o</i> -Phthalaldehyde	Strong yellow under UV (350 nm)	[on silica gel]	[41]
Sulphanilic acid	Red-brown	[on paper]	[40]
TCBI	Brown-green	[?]	[47]
Thies & Reuther's React	-	[paper]	[59]
Van Urk	Blue	[on pure compound]	[50]
Van Urk-Salkowski	Violet blue (Cornflower Blue)	[when dry, on silica gel]	[10]
, an one ownowohi	Bluish Green (Capri Blue)	[when wet, on silica gel]	[10]
Xanthydrol	Blue [Ed.: This report has been questione		[1 6]

Reagent <u>Bufotenine-N-oxide</u>	Reaction	[Medium]	Ref.
trans Cinnamaldehyde Fast Blue salt B	Cherry-Red [Reddish] Reddish-purple [Purple-violet]	[on silica gel & others] [on silica gel & others]	[45] [45]
Pauly	Dark reddish violet	[on silica gel & others]	[45]
Bufotenidine Dragendorff's	Orange	[on paper]	[20]
Ehrlich's	Blue	[on paper]	[20]
α -Nitroso- β -naphtholnitro		[on paper]	[20]
		[on paper]	[=•]
<u>Bufothionine</u> Ehrlich's	Weak. Blue-purple	[on paper]	[40]
	Weak. Dide pulpie	[on pupor]	[10]
5-Methoxytryptamine			
	nce Bright yellow (under 254nm UV)) [on Avicel]	[29]
Ehrlich	Reddish-pink (1 hr)/ Blue (overnight	ht) [on silica gel]	[41]
	Rapid. Bluish-purple→Blue-Gray	[as HCl on paper]	[40]
	Blue	[on paper]	[27, 49]
Fluorescence with PENE	Pale yellow (under 254nm UV)	[silica gel (using PENE)]	[30]
Gibbs'	No reaction	[on paper]	[49]
Naphthanil diazoblue B	No reaction	[on paper]	[27]
Ninhydrin-Acetic acid	Brownish-purple	[as HCl on paper]	[40]
Ninhydrin-Cadmium ace	e s ([on silica gel]	[41]
Ninhydrin-Pyridine	Brownish-purple [Visible]	[as HCl on paper]	[40]
4	Bright Green-blue [UV]	[as HCl on paper]	[40]
diazotized <i>p</i> -Nitroaniline		[on paper]	[11b]
NNCD reagent	Orange-red	[on paper]	[11b]
Pauly	Faint pink (5 min)	[on silica gel]	[41]
<i>o</i> -Phthalaldehyde	Strong blue under UV (350 nm) ite Yellow	[on silica gel]	[41]
Sulphanilic acid/ Na nitri Van Urk-Salkowski		[on paper]	[27]
vali UIK-Saikowski	Blue (Princes Blue) Bluish-green (Langite Green)	[when dry, on silica gel] [when wet, on silica gel]	[10] [10]
	Biuisii-green (Langite Oreen)	[when wet, on since ger]	
5-MeO-MMT			
Acid-Induced Fluorescer	nce Bright yellow (under 254nm UV)[on Avicel]	[29]
PDAB (0.125 grams in 65%	% sulfuric acid containing 0.1% v/v of 5%	ferric chloride)	
	Green solution which turns blue whether the solution which turns blue whether the solution wh	hen diluted with water	[55]
PDAB	Blue	[on paper or with tlc]	[12, 19]
	Deep blue	[on paper]	[55]
Ehrlich's	Royal blue	[on silica gel & cellulose]	[56]
	Blue	[on paper]	[49]
Echtrotsalz B	Light brown-yellow	[on paper]	[12]
Ferric chloride soln.	No reaction		[55]
	Pale yellow (under 254nm UV)	[silica gel (using PENE)]	[30]
Gibbs	No reaction No reaction	[on paper]	[12] [40]
Millon's	Brown	[on paper]	[49] [55]
NNCD	Orange brown	[on paper or with tlc]	[35] [12]
Pauly	Light olive yellow	[on paper]	[12]
Sodium nitroprusside	[Should test blue with this reagent		[]
(and acetone)	Blue	L	[55]
(and acetaldehyde)	None		[55]
Xanthydrol	Blue	[on silica gel & cellulose]	[56]

Trout's Notes on Tryptamines: Color Reactions

Reagent 5-MeO-DMT	[Medium]	Ref.	
	ce Bright yellow (under 254nm UV)	[on Avice]]	[29]
Chloranil	None	[on silica gel]	[26]
CNTNF	Gray	[on silica gel]	[26]
PDAB	Blue	[on paper]	[19]
	Blue	[on paper or with tlc]	[12]
Dragendorff	Red-Brown	[on paper]	[9]
e	Orange precipitate	[in ethanol solution]	[35]
Echtrotsalz B	Light brown-yellow	[on paper]	[12]
Ehrlich's	Royal blue	[silica gel & cellulose]	[56]
	Dark Blue	[on paper]	[9]
Fluoranil	Purple	[on silica gel]	[26]
Fluorescence with PENE	Pale yellow (under 254nm UV)	[silica gel (with PENE)]	[30]
Gibbs	No reaction	[on paper]	[12]
HNS	None	[on silica gel]	[26]
Iodine vapor	Red-brown	[on paper]	[9]
NNCD	Orange brown	[on paper or with tlc]	[12]
α-Nitroso-β-naphthol- ni		[on paper]	[9]
Pauly	Light olive yellow	[on paper]	[12]
TACOT	Purple (light)	[on silica gel]	[26]
TCNE	Green (light and fading)	[on silica gel]	[26]
TetNF	Purple (light)	[on silica gel]	[26]
TNB	Yellow→Brown	[on silica gel]	[26]
TNF	Brown (light)	[on silica gel]	[26]
Van Urk-Salkowski	Blue (Cornflower blue)	[in ethanol solution]	[35]
Xanthydrol	Blue	[silica gel & cellulose]	[56]
	Blue	[on paper]	[15]
5-MeO-DMT-N-oxide			
PDAB	Cherry-Red to Blue	[on paper]	[19]
PDAB	Blue	[on paper]	[36]





cultivated ornamental growing in Austin, Texas Psychotria carthaginensis unripe fruit (Oz) (on the left)

Reagent	Reaction	[Medium]	Ref.
Ibogaine			
Chloranil	None	[on silica gel]	[26]
CNTNF	Grey turning Purple	[on silica gel]	[26]
Ethanolic PDAB	Green-blue	[pure compound]	[42]
Fluoranil	None	[on silica gel]	[26]
HNS	None	[on silica gel]	[26]
PDAB	None	[on silica gel]	[3]
PDAB-TS	Yellow-green	[pure compound]	[42]
TACOT	Light purple fading to nothing	[on silica gel]	[26]
TCBI	Brown-green	[on silica gel]	[53]
TCNE	Brown turning yellow	[on silica gel]	[26]
TetNF	Grey-brown turning Brown	[on silica gel]	[26]
TNB	Brown turning light brown	[on silica gel]	[26]
TNF	Brown	[on silica gel]	[26]
Eluorogoog of 222 mm	with availation at 200 mm		

Fluoresces at 332 nm with excitation at 298 nm

Slits (Em./Ex.) 8/8; Filter 310 nm; on Perkin-Elmer MPF-2A fluorescence spectrophotometer

Limit 1 mcgm (Methanol as solvent)

DeZan et al. 1971

<u>LSD</u>

Ethanolic PDAB	Blue	[pure compound]	[42]
Chloranil	Light brown turning brown	[on silica gel]	[26]
CNTNF	Grey turning purple	[on silica gel]	[26]
DMBA	Blue (faster with heat)	[silica gel; under UV]	[52]
Fluoranil	Purple turning light brown	[on silica gel]	[26]
HNS	None	[on silica gel]	[26]
Marquis reagent	Grey	[silica gel]	[52]
PDAB	Purple	[on silica gel]	[3]
PDAB-TS	Purple	[pure compound]	[42]
TACOT	Pink	[on silica gel]	[26]
TCBI	Grey-brown	[on silica gel]	[53]
TCNE	Brown fading to nothing	[on silica gel]	[26]
TetNF	Light Grey turning Grey	[on silica gel]	[26]
TNB	Brown	[on silica gel]	[26]
TNF	Brown	[on silica gel]	[26]
F1 (1 1 1 1 1		10111 1 0// INVIENT	

Fluoresces (bright blue) under both 360 nm and 254 nm UV **[3]** Under 366 nm UV **[52]** This is true whether using pure material or running in tlc.

Fluoresces at 400 nm with excitation at 320 nm

Slits (Em./Ex,) 10/6; Filter 350 nm; on Perkin-Elmer MPF-2A fluorescence spectrophotometer

Limit 0.05 microgram (Methanol as solvent)

DeZan et al. 1971

See also Phillips & Gardiner 1969 for Assays on LSD & related compounds

Note though, that fluorescence of this or the fluorescent tryptamines is accompanied by at least some degree of degradation and, while often beautiful to look at, such viewing under a fluorescent light should be kept to a minimum or when absolutely required (such as when windowpane has been spilled on a shag carpet)

Neither of these two compounds are covered in this work.

The listed color reactions above are included only for the reader's convenience.

An antique 25 mg ampoule of Delysid (Sandoz's LSD-25) from those days when it was still a legal item of commerce. Above right are gels from Tennessee, USA - now illegal in many places. Both photos thanks to anonymous readers



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Phalaris aquatica cv. Harding grass floret & seed

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Panaeolus subbalteatus Photo above by JW Allen



Mimosa tenuiflora rootbark (Chiapas, Mexico) above & below







Anadenanthera peregrina seedpods & established plant (Oz) Photos above by Snu Voogelbreinder

Copelandia cambodginiensis (Thailand) Photo by JW Allen



Chromophoretic Reagents; Preparations & Assays

It is important to remember that most of these have multiple formulations.

Those included below are what were presented by the authors reporting the listed chromophores (if they included a specific version of the formula)

If these are available their use should be favored over other formulations when attempting to reproduce the published observations.

Also caution should be exercised concerning the color names as this can potentially vary from one researcher to the next depending on a variety of elements; from the reagent formulation to the solid phase chosen (the substrate it was performed on), to the assay procedure itself to their cultural definition of color names, and other factors.

Prior familiarity with their use using known reference standards is highly recommended

Both IPA & Dragendorff's should show a positive reaction for ALL of the indoles despite us not finding individual applications mentioned for every substance. Both form reversible chromophores that can subsequently be degraded with an alkali and the base recovered with an organic solvent allowing their use in preparative chromatography.

Phosphomolybdic acid is also said to form a precipitate decomposable with strong base for alkaloid recovery.

Vanillin reagent should also work fine but forms an irreversible color reaction. All three of these are largely nonspecific meaning they will not distinguish between most of the individual compounds but can prove valuable when used in tlc or pc. Iodine is even more nonspecific and will react with everything covered here.

Gibbs reagent, alkaline silver reagent, sulphanilic acid reagent and Pauly reagent are useful for detecting the phenolic indoles such as bufotenine or psilocin or serotonin; as they will show no reaction with the nonphenolics like 5-MeO-DMT or psilocybin or DMT.

Silicotungstic acid reagent should lend itself nicely to field screenings for all simple indoles (and can provide crude quantitative estimations with a little practice) but so far as we can tell it has not specifically been applied to the pshroom indoles.

HOWEVER, since the active pshroom species usually blue (or green) upon bruising or cutting they have a built-in but not infallible field screening assay. (Some Boletes bruise blue that do not contain any psilocybian alkaloids and those active mushroom species lacking psilocin and containing only psilocybin do not bruise blue.)

Many of these were used as a TLC or PC spray. This is indicated either along with the chromophores listed above or in the recipe for their preparation under the individual author cited as reporting the listed chromophore.

Dryness is incredibly important for silica gel plates used as solid phases in TLC. Storage in a desiccator or warm oven is recommended after the required oven drying prior to use. Presence of even traces of moisture can cause poor performance and uneven results between tests or within a single plate!

DANGER!

Be very cautious when performing assays.

Many require toxic compounds, caustic agents and/or strong acids that can cause serious injury to eyes, lungs and other body parts.

Vapors, mists or direct contact can be extremely hazardous.

Do not attempt to perform ANY assays without FIRST understanding safe chemical handling practices.

Acid-induced Fluorescence:

Majak & Bose 1977:

Viewed under 254 nm UV after exposure to HCl vapor or when running in acidified solvents [MeOH conc. HCl (9:1) or butanol-2M HCl (1:1; upper phase) Run on avicel or avicel-silica gel 7.]

Bright yellow fluorescence with all **5-methoxy-** and **5-hydroxy-**tryptamines.

Alkaline Silver reagent:

Jepson in Smith 1969:

1) 1 drop of silver nitrate (as a saturated solution in water) is mixed with 10 ml of acetone

2) 0.5% solution of sodium hydroxide in 80% ethanol.

Part 1 should be freshly mixed and applied as a dip. The acetone is then blown off and finally it is dipped in part 2.

If Indoles with a free oxidizable phenol are present immediate black of dark brown spots will form.

Tryptamines with a free phenolic group, such as serotonin and bufotenine, show an immediate black spot. Other tryptamines may take several minutes to react. Reacts with a variety of oxidizable phenols and enols.

Cannot be used on chromatograms run in solvent systems involving chloride ions.

Can be used after Ninhydrin-Pyridine but a very rapid darkening of the background occurs.

Brentamine Fast Red B:

Kveder & McIsaac 1961: 0.1% aqueous solution of Brentamine Fast Red B salt, followed by saturated NaHCO,

CAS:

Svoboda *et al.* 1979: N-Methyltryptamine exhibits a brilliant yellow color in long wave UV when visualized with 1% ceric ammonium sulfate in 85% phosphoric acid (heated at 110°, 10 min.).

trans-Cinnamaldehyde:

Stijve 1979: A 1% ethanolic solution was prepared immediately before use and applied as a spray. The plate was developed by placing in a tank with HCl vapors. (They bubbled air through concentrated hydrochloric acid). [See also Ehrlichs etc.] Sensitivity of up to 0.1 mg.

Chloranil reagent:

- Heacock & Forrest 1973; 1 gram of chloranil in 100 ml of Acetonitrile. Freshly prepared before use.
- Chloranil: colors with tryptamines were relatively weak and slow to develop.

CNTNF reagent:

- Heacock & Forrest 1973: 1 gram of 9dicyanomethylene-2,4,7-trinitrofluorene in 100 ml of acetonitrile. Freshly prepared before use.
- **DMBA reagent:** See also more under Ehrlich's, Van Urk's & PDAB.
- Van Welsum 1973: 0.5 gm of PDAB is dissolved in 103 ml of conc. Sulfuric acid-water (53:50). 0.5 ml of Fe₃Cl₂ solution is then added. (10.5% aqueous solution of Fe₃Cl₂.)] (Color develops faster with heating.)

DMCA:

p-Dimethylamino-cinnamaldehyde

(aka 4-Dimethylaminocinnamaldehyde)

- Jepson's version of the DMCA reagent (Modified Ehrlich's Reagent): (as given by Harley-Mason & Archer 1958)
- If used for dipping the formula should be modified to 1% of solid in concentrated hydrochloric acid. It should be diluted 1:4 with acetone immediately before use.
- 2 grams of *p*-dimethylaminocinnamaldehyde are dissolved in 100 ml of 6N hydrochloric acid and 100 ml of ethanol. Used as spray.

[Harley-Mason & Archer dissolved 2 gm in 100 ml of 6N HCl and ethanol.]

- Said to be more sensitive than Ehrlich's with PDAB but less selective.
- Jork et al. 1994: p-dimethylaminocinnamaldehyde hydrochloric acid reagent:

500 mg of *p*-dimethylamino-cinnamaldehyde dissolved in 50 ml of hydrochloric acid and brought to 200 ml with ethanol for use as a spray reagent. Will keep several days in refrigerator.

Kveder & McIsaac 1961 and Taborsky & McIsaac 1964 used as a 0.5% solution in 1.5N hydrochloric acid.

Stijve *et al.* 1984: 0.5 gm of DMCA, mixed with 10 ml fuming sulfuric acid, then mixed with 50 ml of methanol. Colors may vary depending on the solid phase. Optimal visibility took 10-15 minutes. It could be accelerated by slightly heating with a hair drier.

Dragendorff's reagent:

Clarke's: Dissolve 1 gram bismuth subnitrate in 3 ml. of 10M hydrochloric acid with the aid of heat, dilute to 20 ml with water, and dissolve into the mixture 1 gram of potassium iodide. If black bismuth tri-iodide separates, add 2M HCl and more potassium iodide to dissolve.

Dissolve the sample in 3 drops of 2M hydrochloric acid, add 2 to 3 ml of the reagent and dilute to 10 ml with water.

May be applied after Ninhydrin.

[Forms reversible precipitate; see pages 158-159] Can also be used as a spray in TLC.

Gibbons & Gray 1998: 10 ml of 40% aqueous KI added to 10 ml of basic bismuth subnitrate (0.85 gm dissolved in 10 ml of acetic acid and 50 ml of distilled water) Resulting solution (1 part) diluted with acetic acid (2 parts) & water (10 parts) Heating or decolorizing plate with ammonia vapor may enhance.

Echtrotsalz B reagent:

Erspamer *et al.* 1967: 0.3% solution in 50% ethanol of Echtrotsalz B Fluka (diazotized 1-amino-2-methoxy-4-nitrobenzene) followed by sodium carbonate.

Ehrlich's reagent:

- (*p*-dimethylamino-benzaldehyde [PDAB] or *p*dimethylamino-cinnamaldehyde [PDAC]). (See also DMBA, PDAB & Van Urk's)
- [Jork points out that while the hydrochloric acid version of this is properly known as Ehrlich's and the sulfuric acid version is properly known as Van Urk's there unfortunately is no standard usage of the names in the literature and different preparations are variously referred to as one or the other.]
- Can be obtained commercially as a ready-made product or it can be made as needed.
- Once chromatograms are developed, spots should be gently outlined with a soft lead pencil, as colors often change and/or fade.
- Several points should be noted with Ehrlich's according to Clarke's:

1. Rate of color development (Immediate, rapid, slow or delayed).

2. The Initial color.

3. Any changes in colors. (Color change from A to B will be shown as $A \rightarrow B$.)

4. The permanence of the color.

Some colors only form overnight. While unusual, this is diagnostic for certain compounds (like tetrahydroharmine).

Note: Pyrroles and some phenols also react with Ehrlich's. Ehrlichs can be used after Ninhydrin-pyridine but will be

- less sensitive for any compounds that reacted with the Ninhydrin-Pyridine.
- If used after Ninhydrin-acetic acid, any compounds that reacted with the Ninhydrin-acetic acid will not react with Ehrlichs.
- Can be used before sulphanilic acid but all acid must first be blown off or the plate allowed to stand for several hours before the sulphanilic acid reagent is applied.
- *p*-Dimethylaminobenzaldehyde (0.5 gm. in 50 ml of ethanol-sulfuric acid (6:4) - freshly prepared) Gives red, purple or blue color with indoles.
- Baxter & Slaytor 1972 used Ehrlich's oversprayed with 4.5% sodium nitrate. (Said to be blue with all tryptamine and tryptophan derivatives.)
- Beug & Bigwood 1981: 10% PDAB in concentrated HCl was made into a spray for TLC by combining the Ehrlich's reagent with acetone (1:4) (viewed directly and then under UV). Used on silica gel & microcrystalline cellulose.
- Fish *et al.* 1956 used as 0.5% PDAB in 1N hydrochloric acid (on paper).
- Gibbons & Gray 1998: First, spray with solution of 1 gram PDAB in 100 ml conc. HCl-methanol (3:1), then spray with (?g) PDAB in 100 ml ethanol. Place in tank saturated with HCl vapor for 5 minutes or spray with 25% HCl. Warm plates gently.
- Guzmán & Ott 1976: (Modified Ehrlichs) 10% PDAB in concentrated HCl; diluted with acetone (1:4). Applied as a dip after plates were first air dried.
- Jork *et al.* 1994: 1-5 grams of PDAB in 50 ml of 25% hydrochloric acid and 50 ml of ethanol (96 or 100%). Methanol or *iso*-propanol can be used instead of ethanol. Solution is stable for several weeks.
- Also 10 grams of PDAB in concentrated hydrochloric acid diluted 1 volume with 4 to 10 volumes of acetone just before use (not stable).
- Margot & Watling 1981: 5% PDAB in 50% sulfuric acid and exposed to HCl vapor overnight.
- Picker & Rickards 1970: 2% PDAB in 1N HCl.
- Repke & Leslie 1977: 2% PDAB in ethanol and exposed to HCl vapors.
- Smith & Seakins 1976: a 10% weight to volume solution of p-dimethylamino-benzaldehyde is first made with concentrated hydrochloric acid. One volume of this is mixed with 4 volumes of acetone immediately before use. The plate should be sprayed and observed for color reactions, then placed in a desiccator and colors noted after one hour and again after 18 hours. They recommend that two observers note the colors.
- Stamets *et al.* 1980 used same Ehrlichs as Guzmán & Ott 1976 but applied as a spray after examining their plates under UV.
- Touchstone 1992: 10% of PDAB in concentrated hydrochloric acid. Mix 1:4 with acetone before use. Color development within 20 minutes after spraying.

- Waldi 1962: Ehrlich's: 1% solution of *p*-dimethylaminobenzaldehyde in 50 ml of hydrochloric acid with 50 ml of ethanol then added. After applying as a spray, placed for 3 to 5 minutes in an atmosphere of HCl vapors.
- Modified Ehrlich's: 500 mg of PDAB dissolved in 100 ml of hot cyclohexane. Placed in tank of HCl vapor after spraying.
- Woods & Clark 1971: 0.7 grams of *p*dimethylaminobenzaldehyde was dissolved in 150 ml conc. hydrochloric acid then made up to 250 ml with distilled water. They recommended that it be at least a week old before using.

Ethanolic PDAB See under PDAB

- **Fast Blue B:** (di-*o*-anisidine tetrazolium chloride) (i.e. Echtblau B)
- Stijve: 0.5% in 50% aqueous ethanol. Should be freshly prepared before use. Visibility is sometimes improved by exposure to ammonia fumes. Detection limit is 0.2 µg.

Fast Blue BB salt (diazotized 4-benzoylamino-2,5diethoxy aniline):

- McKenzie *et al.* 1975 used as 1 gram dissolved in 10 ml of methanol.
- **Ferric Chloride:** Useful for differentiating phenolic compounds like psilocin (does not react with the others).
- Clarke's: Dissolve sample in ethanol and add ferric chloride solution (red, orange, green, blue, violet or brown indicates phenolics).

Fluoranil reagent:

- Heacock & Forrest 1973: 1 gram of fluoranil in 100 ml of acetonitrile. Freshly prepared before use.
- Fluoronil was the best (of the reagents they used) for the tryptamines (0.1 μ g sensitivity).

Fluorescamine:

Wagner & Grevel 1982 used a 0.002% solution of Fluorescamine in water-free acetone as a tle spray on Kieselgel. Viewed under 360 nm UV.

Fluorescence with PENE:

- Majak *et al.* 1978: PENE: *i*-Propanol-Ethyl acetate-conc. NH_4OH -2-Ethoxy-ethanol (60:15:3:5) was used as a tlc solvent system to enhance (prolong) fluorescence.
- Viewed under shortwave UV. TLC run on silica gel. Drying plates in a cold room also prolonged colors. Gramine, MMT and DMT gave a violet fluorescence. 5-hydroxy and 5-methoxyindoles give pale yellow fluorescence on silica gel with PENE.
- Unable to find any evaluation of the 4-substituted indoles using this system but it is certainly suggested.

Froehde's reagent:

- Also known as sulfomolybdic acid reagent. (Useful for some β-carbolines) From CRC handbook: Dissolve 10 grams of molybdic acid or sodium molybdate in 100 ml of concentrated sulfuric acid.
- Svendsen & Verpoorte 1983 give as 1% ammonium molybdate in concentrated sulfuric acid. Colors are observed immediately after spraying and again after heating for 5 minutes at 105°.
- **Gibbs reagent:** (Useful for differentiating phenolic compounds; does not react with nonphenolics.)
- Erspamer *et al.* 1967: 0.05-0.1% alcoholic solution of dichloroquinone chlorimide (followed by sodium carbonate.
- Gasparic & Churacek 1978: 0.4% 2,6-dibromoquinone-4-chlorimide in methanol (prepared immediately before use) After spraying expose to ammonia vapor or spray with 10% sodium carbonate solution.
- Jepson 1969: 0.05% 2,6-dichlorobenzonquinone-4-Nchloro-imine in absolute alcohol [In dark is stable for 2 weeks.] This is applied and allowed to dry. It is then followed with sodium borate buffer 4.75 g. per cent at pH 9.3.
- Taborsky & McIsaac 1964 used a 2% ethanolic solution of 2,6-dichloroquinoneimide followed by NaHCO₂ (saturated aqueous solution).
- Gibbs' reagent cannot be used in systems containing diethylamine; Svendsen & Verpoorte 1983.

HNS reagent:

Heacock & Forrest 1973: Saturated solution of 2,2',4,4',6,6'-hexanitrobenzene in acetonitrile. Freshly prepared before use. Weak reaction with tryptamines.

Human serum/ Indoxyl acetate:

- Stijve 1979: (Tests for cholinesterase inhibitors such as bufotenine.)
- 1% aqueous solution of lyophilized human serum was freshly prepared and sprayed on plate until well wetted but not dripping. It was then incubated 20-30 minutes at 37°C.
- 10 mg indoxyl acetate was dissolved in 1 ml of ethanol, then mixed with 9 ml of distilled water at 40°C. This was sprayed and again incubated. Bufotenine shows as a white spot on a light blue (turquoise) background. Detection limit- $0.1 \mu g$.

Iodine:

Phillips & Gardiner 1969 used a 2% solution of iodine in methanol as a TLC spray and found it worked in all systems they evaluated. They noted that it could be used to differentiate tertiary amines from primary & secondary amines. The first are said to give an orange color that fades to a permanent, very faint yellow. The latter initially started as a pale yellow that darkened before fading to a permanent fawn coloration.

- This is a general spray reagent commonly used for alkaloids. Iodine is getting harder to find due to legal restrictions aimed at explosive & drug manufacture.
- Waldi noted that plates could also be placed into a closed vessel containing a few iodine crystals in the bottom. When heated over a water bath, the vapor given off form brown chromophores with most organic compounds.

Iodoplatinate reagent (IPA):

- Brown *et al.* 1972 lightly sprayed with IPA (iodoplatinate); which was made of equal parts of 0.3% hexachloroplatinic acid and 6% aqueous potassium iodide. Colors were allowed to develop for 5 minutes.
- Schnoll *et al.* 1972 used iodoplatinate reagent as their final spray (after Ninhydrin and HNO_3 tests): Prepared from 250 mg. platinic chloride and 5 gm potassium iodide in 100 ml. distilled water.

Touchstone 1992: Solution A: 5% platinic chloride in water,

Solution B: 10% aqueous KI,

Mix 5 ml of A, with 45 ml of B, and dilute to 100 ml

- with water.
- Addition of hydrochloric acid as 1 to 10 parts of solution will increase sensitivity.

It has some advantages over many other reagents:

- a) It is about as sensitive as Dragendorff's (0.01 to 0.1 μ g can be detected.)
- b) It is nondestructive & forms reversible chromophores; allowing the material on the chromatogram to be recovered if desired. This makes it a valuable reagent for locating and identifying zones during preparative chromatography. The chromophore can be scraped from the plate, extracted, decomposed with a base and the alkaloid recovered with simple organic solvent extraction.
- Comments:
- Formamide impregnated plates will interfere with alkaloid detection but this can be overcome by spraying with 0.25% sodium nitrite in 0.5% hydrochloric acid to convert the formamide into formic acid which will not interfere with potassium iodoplatinate reagent.
- If the blue-violet background resulting from the presence of starches interferes with viewing of developed colors, it can be decolorized by spraying with a sodium hydrogen sulfite solution.
- Use of diethylamine in a solvent system will decrease the sensitivity of both Dragendorffs and potassium iodoplatinate reagents due to a darkening of the background.

Svendsen & Verpoorte 1983

For some of these alkaloids, (such as the major pshroom alkaloids), this is not a problem since they are visible in visible light (and also fluoresce under UV); thus needing no reagent to MAKE them visible. For many other tryptamines, it's a different story.

To avoid confusion stay aware that the abbreviation IPA is also commonly used for *i*-propanol (Isopropyl alcohol).

Acidified Iodoplatinate:

- Smith & Seakins 1976: 9 grams of potassium iodide are first dissolved in 200 ml of distilled water and then 10 ml of chloroplatinic acid is added. Three volumes of this solution are added to One volume of conc. hydrochloric acid. It should be mixed with the acid just before spraying.
- Reacts with a wide variety of alkaloids and compounds; from indoles, caffeine and methaqualone to benzodiazepines.

Can be applied after Ninhydrin and Dragendorff's.

Reacts with a wide variety of alkaloids and compounds; from indoles, caffeine and methaqualone to benzodiazepines.

Keller assay:

Troxler *et al.* 1959: 0.2 mg of the substance is dissolved in 1 ml ferric acetate containing 0.5% ferric ions, which is then underlaid with 1 ml concentrated sulfuric acid and thoroughly shaken.

Mandelin's test:

Clarke's: 0.5 gm of ammonium vanadate is dissolved in 1.5 ml of water and then diluted with sulfuric acid to 100 ml. The solution is then filtered through glass wool.

Add a drop of reagent to sample on a white tile.

- Care must be taken in interpretation as hydrochlorides give a red color when using this reagent.
- Marquis reagent and test: (AKA Sulfuric acid-Formaldehyde test)
- Clarke's: Mix 1 volume of formaldehyde solution with 9 volumes of sulfuric acid.

Add a drop of reagent to sample on a white tile.

- Clarke's 1986 also mentions this should be poured rather than sprayed on plates to avoid generating a dangerous aerosol of concentrated acid mist.
- Van Welsum 1973: 10 drops of a 40% solution of formaldehyde added to 10 ml of concentrated sulfuric acid and applied as a spray reagent.

Mayer's reagent:

- CRC Handbook: Dissolve 1.358 grams of HgCl₂ in 60 ml of water and pour into a solution of 5 grams of KI in 10 ml of water. Bring up to 100 ml with water. Forms a white precipitate with most alkaloids.
- Rovelli & Vaughan 1967 said that DMT gave very strong test with Mayer's reagent in standard field test for alkaloids based on Culvenor & Fitzgerald 1963. Soluble mercury salts are highly toxic. This and Millon's reagent should only be prepared and used by the experienced.

Millon's reagent:

CRC Handbook: Dissolve 1 part mercury in 1 part cold fuming nitric acid.

Dilute with twice the volume of water.

Let stand several hours and decant clear solution.

Clarke's: 3 ml of mercury is dissolved in 27 ml of fuming nitric acid. Add to an equal volume of water with stirring. To test- Add 0.5 ml of reagent to sample with warming. Red or orange indicates phenolic. Not all phenolics react.

Naphthanil diazoblue B:

Kveder & McIsaac 1961: Naphthanil diazoblue B (tetrazotized di-*O*-anisidine), 3% solution, plus borate buffer, pH 9 (3:2)[see also Fast Blue B]

Ninhydrin: [AKA Triketohydrindane hydrate]

- Ninhydrin is very useful as TLC spray to differentiate primary amines from secondary amines (which show different colors) and tertiary amines (which do not show any color).
- The reported colors though are highly variable depending on the source of the information, the actual target compounds & the reagent formulation (many exist and it is readily available commercially)
- It is a very valuable reagent for this purpose but we would suggest prior familiarity with its use using known reference materials.
- Clarke's: Dissolve 0.5 grams of ninhydrin in 40 ml of Acetone.

Dissolve the sample in methanol, place 1 drop of the solution on a filter paper, add one drop of the reagent and dry in a current of hot air. The samples should then be examined under UV. [Will not react with all indoles.]

- Schnoll *et al.* 1972 used Ninhydrin as a spray reagent [after checking for a color change in HNO₃ and before iodoplatinate]. He used a concentration of 500 mg of ninhydrin in 100 ml of acetone. After spraying it was heated in an oven for 5 minutes at 100° C.
- Gibbons & Gray 19998 recommended 0.3 g ninhydrin in 100 ml butanol & 3 ml acetic acid.
- Culvenor *et al.* 1964 claims that ninhydrin shows no color with tertiary amines (like DMT) and a purpleblack color with primary (like tryptamine) and secondary amines (like MMT).
- Clarke's claims that ninhydrin gives violet or pink with primary and yellow with secondary amines.

Ninhydrin-Acetic acid:

- Jepson 1960: 9 volumes of ninhydrin (0.2% in acetone) are mixed with 1 volume of glacial acetic acid just before use.
- After dipping, the acetone is blown off and the paper heated 110° for 2 min. High concentration are visible (brownish-pink) in normal light but much more sensitive (100X) when viewed under UV (Wood's light). Tryptamines show greenish-blue (sea-green) under UV, whereas N-substituted tryptamines do not. N-alkyltryptamines show visible color while N,N-dialkylated tryptamines do not.

No reaction with tryptophans.

Tryptamines show strong visible color and fluorescence.

- N-alkyl show strong visible color and little fluorescence. N,N-dialkyl shows no visible color and no fluorescence.
- Jepson 1969

Ninhydrin-Cadmium acetate reagent:

- Smith & Seakins 1976: 20 ml of cadmium acetate (0.5 g.) in glacial acetic acid, 30 ml of distilled water and 450 ml of acetone are mixed for a stock solution. Just prior to use, aliquots should be measured and solid ninhydrin added to make a 0.2% w/v solution of Ninhydrin.
- After spraying, the plate is heated at 60° for 15 minutes.
- Colors are recorded after plates has cooled to room temperature.

Ninhydrin-Pyridine:

Jepson 1969: 0.2% ninhydrin in acetone that has had a few drops of pyridine added immediately before use. [Will not react with all indoles. Reaction is slow in cold; fast when heated. Reacts with MMT but not DMT.]

α -Nitroso- β -naphthol reagent:

- Gasparic & Churacek 1978: First spray with 0.01% solution of α -nitroso- β -naphthol and then with 10% HNO₃ and heat at 90° C.
- Ivor Smith 1969: 0.1% α-nitroso-β-naphthol in ethanol (9 volumes is mixed with concentrated nitric acid (1 volume) immediately before use. The paper is dipped and allowed to dry 2 to 3 minutes at room temperature then is heated for 2-3 minutes at 105° C. Colors are destroyed by overheating. Low sensitivity- 20 µg sample required.
- Specific for 5-hydroxyindoles. Forms violet chromophore.

NNCD [Heinrich & Schuler NNCD reagent]:

Erspamer *et al.* 1967: 0.1-0.3% solution of 2-chloro-4-nitrobenzenediazonium-naphthalene-2-sulphonate.

HNO₃ (Nitric acid) atmosphere:

Schnoll *et al.* 1972 placed their chromatogram in a tank with a HNO₃ atmosphere for 3 minutes (noting any color changes).

Pauly's reagent:

- Erspamer *et al.* 1967: an aqueous solution of diazotized sulfanilic acid (see below for proc.) followed by 3-5% aqueous sodium carbonate.
- Smith & Seakins 1976:

The three solutions should be made and kept separate until use.

A: Sodium nitrite in a 5 per cent solution in water. B: Sulphanilic acid in a 10% weight to volume solution with concentrated hydrochloric acid. 45 ml of this solution is mixed with 350 ml of distilled water. [Ivor Smith 1969 gives as 9 gm of sulphanilic acid in 90 ml of hydrochloric acid and 90 ml of water.]

C: Anhydrous sodium carbonate in a 10 per cent weight to volume solution with water.

- One volume of A should be added to one volume of B being certain that the temperature does not rise above 20° C. Allow it to then stand at a temperature below 20° C for 5 minutes. After this, carefully add two volumes of C and spray the tlc plate with the resulting solution. Colors should be recorded 5 minutes after spraying.
- Pauly's reagent (sulphanilic acid reagent) can be applied after Ehrlichs but all acid must be blown off the plate first or it should be allowed to stand for several hours before applying Pauly's.
- Weeks *et al.* 1979: Used Krebs *et al.*'s formula from Egon Stahl 1969,

PDAB reagent: See also Ehrlich's and Van Urks

- Alliston *et al.* 1971: 5% PDAB in methanol-HCl (1:1). Ran tlc, on silica gel with a fluorescence indicator, in morpholine-toluene (1:9). Using 360 & 254 nm UV to observe (before dry), then again at 254 nm after drying; prior to spraying with this reagent.
- Benedict et al. 1962: 2% PDAB in 1N HCl as spray
- Brown *et al.* 1972: 0.8 grams of PDAB in 10 ml of sulfuric acid and 90 ml of ethanol. Sprayed on silica gel plates which are then heated for 10 minutes at 105° C.
- Clarke's *p*-dimethylaminobenzaldehyde reagent: Solution A: 1 gram of PDAB in 25 ml of concentrated hydrochloric acid and 75 ml of methanol. Solution B. 1 gram of PDAB in 100 ml of 96% ethanol.
- Spray plate with A. Warm plate and then spray with B. Place in a tank saturated with HCl vapor for 3 to 5 minutes or spray with 25% HCl.
- Erspamer *et al.* 1967: 1-2% alcoholic solution of *p*dimethylaminobenzaldehyde followed by exposure to HCl vapors in a glass cabinet.
- Gartz 1986a: 125 mg of PDAB, 0.1 ml of a saturated solution of FeCl,, in 100 ml of 65% sulfuric acid.
- Gupta *et al.* 1979: 2% of PDAB in concentrated hydrochloric acid-ethanol (1:1).
- Hatfield *et al.* 1978: 2% PDAB in con. HCl-ethanol (1:1).
- McKenzie *et al.* 1975: One gram PDAB in 25 ml of 10M HCl and 75 ml of methanol.
- Repke & Leslie 1977 used 2% PDAB in Ethanol as spray followed by exposure to HCl vapor.
- Repke *et al.* 1977b: 1% PDAB in ethanol containing 5% HCl as spray, followed by exposure to HCl vapors & Recommending 1) preparation of solvent immediately prior to use, 2) Solvent equilibrated in tank for only 10 minutes prior to the run, and 3) Minimum of 6 hr exposure to HCl vapors to maximize color development.
- Smith 1981 (page 173): Ethanolic PDAB: Dissolve 2 gm PDAB in 50 ml of ethanol and dilute to 100 ml with concentrated HCl.

- Touchstone 1992: One gram of PDAB in 30 ml of ethanol, 3 ml of conc. hydrochloric acid and 180 ml of *n*butanol, as spray.
- Weeks *et al.* 1979: PDAB 2% PDAB in HCl-ethanol (1:1).
- Wilkinson 1958 found that *p*-dimethylamino-benzaldehyde in ethanol containing hydrochloric acid gave a deep blue color with 5-MeO-MMT, a purple reaction with tryptamine and also showed a pink reaction with gramine.
- On paper, *p*-dimethylaminobenzaldehyde in cyclohexane and followed with hydrogen chloride treatment showed a blue spot with 5-MeO-MMT; Wilkinson 1958.
- Wilkinson 1958 describes 5-MeO-MMT as forming a green solution with PDAB (as 0.125 gram in 65% sulfuric acid containing 0.1% v/v of 5% ferric chloride solution). The green became blue when diluted with Water.
- Wilkinson also tested fractions to locate 5-MeO-MMT by mixing with PDAB in 65% sulfuric acid; looking for a green solution which became blue when diluted with water.

PDAB-TS:

Smith 1981: Dissolve 125 mg PDAB in a cool solution of 65 ml of sulfuric acid in 35 ml of water. Then add 1-2 drops of FeCl₃-T.S. (from 9 gm of FeCl₃ in 100 ml of water).

o-Phthalaldehyde reagent:

- Smith & Seakins 1976: 1 volume of a 0.2% W/V solution of *o*-phthalaldehyde in concentrated hydrochloric acid is added to 1 volume of methanol immediately before use.
- After spraying, the plate is heated at 100° for 15 minutes.
- Colors when viewed under 350 nm UV are recorded after plates has cooled to room temperature.
- 5 ng sensitivity for serotonin and bufotenine.

Phosphomolybdic acid:

Svendsen & Verpoorte 1983: 0.5% in 50% nitric acid. Gibbons & Gray 1998: 0.5% (w/v) in ethanol. Heat plate at 100°C until colors appear.

p-Dimethylaminocinnamaldehyde-Hydrochloric acid reagent: See under Ehrlich's reagent above

Prochazka reagent: (aka Formaldehyde-HCl reagent) Waldi: 10 ml of a 35% Formaldehyde solution is combined with 10 ml of concentrated HCl and 20 ml of Ethanol.

It should be freshly prepared before use. After spraying, the plates should be heated to 100° for 5 minutes and then viewed under longwave UV. Indoles fluoresce brightly yellow to orange to green. Exposure to *aqua regia* may intensify colors.

pTSA:

Beug & Bigwood 1981: toluene-*p*-sulfonic acid (anhydrous) [i.e., *p*-toluenesulfonic acid] as a 20% solution in methanol. This was applied as a dipping solution on silica gel & microcrystalline cellulose plates.

Silicotungstic acid reagent:

- Said by Rovelli & Vaughan 1967 to give a strong reaction with DMT in a standard field test for alkaloids, based on Culvenor & Fitzgerald 1963. (*Thanks to Mulga for details!*)
- 50 gm of sodium tungstate is dissolved in 400 ml cold water.

[To prepare sodium tungstate: tungsten (the metal) or tungsten carbide powder (an abrasive) is placed on a fire brick surface and heated with a gas torch until it glows red. Once started the oxidation will continue through the material until most of it is reduced to a yellow-green powder. It should then be stirred and set to glowing again to completely oxidize the tungsten.

Add 23 (or 230) grams to a solution containing 8 (or 80) grams of sodium hydroxide. Boil until the tungsten oxide is dissolved. (This may get violent so use a large enough container.)

Reduce liquid until platelike crystals appear on the surface. Remove from heat and allow to crystallize. Remove the sodium tungstate crystals and allow to dry. (Discard the caustic soda solution.)]

- Add 6N HCl dropwise until neutral to litmus. IF any white precipitate forms it should be redissolved by swirling.
- An excess of silicic acid hydrate (freshly precipitated) is then added.

[To prepare silicic acid hydrate: Dissolve sodium silicate into a minimum amount of cold water and make neutral to litmus by adding HCl dropwise. After 15 minutes add a small excess of HCl.

Decant solution and wash precipitate one or two times with cold water. Again decant. (This may ruin the glassware.)

- Remove the freshly precipitated Silicic acid hydrate from the container.]
- Boil the mixture for around 2 hours. Keep it acidic by periodically adding small amounts of HCl. This is done until the solution no longer precipitates tungstic acid on addition of dilute HCl. Filter solution to remove undissolved material.
- Concentrate solution as far as possible and shake it in a separatory funnel with 1/3rd of its volume of ether.
- Add small quantities of ice-cold conc. HCl and shake vigorously after each addition. If necessary, use an icebath to maintain a cool solution temperature.
- The liberated acid forms an oily adduct with the ether and sinks to the bottom of the solution. Continue adding acid until no more droplets of the oily adduct are produced.
- Allow solution to clarify and separate the oily adduct from the aqueous and ether phases.

There are 2 routes that can be used at this point to obtain the free silicotungstic acid:

1) Shake adduct with equal volumes of clean water.

- A stream of air [or nitrogen] directed through the mixture will drive off the ether. The resulting clear aqueous solution can then be placed into a desiccator over sulfuric acid to remove the water and crystallization of the free acid will occur. Heat must not be used to remove the water.
- Residual HCl can be driven off by passing a stream of air through a test tube containing the crystals. The largest crystals can be used as they are. Redissolving into water, filtering and recrystallizing will provide a more pure product.
- 2) The adduct can be decomposed at 40°C until all ether is removed. A stream of air passed through the slowly solidifying residue in a test tube will remove excess HCl.
- The resulting residue is dissolved in water, filtered and recrystallized.
- Reagent is prepared for use by adding 12 grams of free silicotungstic acid crystals to 100 ml of water. See also <u>http://lycaeum.org/~mulga/silico.html</u>[if this goes off-line try at <u>www.erowid.org</u>].

Sodium acetate, 2,6-Dibromo-*p*-benzoquinone-4chlorimine and Iodine:

Genest & Hughes 1968:

- Plates were first sprayed lightly with 10% aqueous sodium acetate, the immediately with 2,6-dibromo-*p*-benzo-quinone-4-chlorimine (B.D.H. Ltd.) (one per cent in ethanol), carefully avoiding overspray.
- They then were placed in a tlc tank containing 2 grams of iodine crystals which were distributed in two small Petri dishes placed at the bottom (iodine vapor atmosphere). Colored spots appeared promptly.
- Plates which run in solvent system C [containing acetic acid] were aerated until the acidic background became dispelled and were then sprayed twice with the aqueous sodium acetate before application of the 2,6-dibromo-*p*-benzoquinone-4-chlorimine and exposure to iodine vapors.
- The sodium acetate solution and the 2,6-dibromo-*p*-benzoquinone-4-chlorimine remained usable for 5 days if refrigerated.

Sodium nitroprusside reagent:

- A blue color which was said to develop with sodium nitroprusside and acetone but not with acetaldehyde was said to be characteristic of secondary amines Wilkinson 1958 cited Kharichkov 1906.
- Baxter & Slaytor 1972 cited Feigl 1954, for the use of sodium nitroprusside as visualization reagent in tlc for Nmethyltryptamines (blue).
- Waldi 1962: 5 grams of sodium nitroprusside is dissolved in 100 ml of 10% aqueous acetic aldehyde.

One volume of this is mixed with 1 volume of 1% sodium carbonate just before use. Used as a spray.

Said to react with secondary aliphatic amines (such as MMT or 5-MeO-MMT).

Sulfuric acid test:

Clarke's Second: Concentrated sulfuric acid is applied directly to the sample on a white tile or in a test tube. (Check also under UV.)

Dilute Sulfuric acid:

Clarke's Second: 10% w/w sulfuric acid is prepared by carefully mixing 104 grams of concentrated sulfuric acid to 896 grams of water and allowing it to cool.

Sulfuric acid-Ethanol spray reagent:

- Clarke's : 10 ml of concentrated sulfuric acid gradually added to 90 ml of ethanol.
- Van Welsum 1973: Concentrated sulfuric acid and 96% ethanol are combined in equal parts.
- Sulphanilic acid reagent: See also as Pauly's reagent. (Pauly's is what is generally used.) Useful for differentiating or detecting phenolic alkaloids like psilocin.
- Baxter & Slaytor 1972 used diazotized sulphanilic acid for phenolic indoles (pink color).
- CRC: 0.5 grams of sulfanilic acid is dissolved in a mixture of 15 ml of glacial acetic acid and 135 ml of recently boiled water.
- Kveder & McIsaac 1961: 0.5% aqueous solution of sodium nitrite and 0.1% solution of sulfanilic acid in 0.15N hydrochloric acid (1:30).

TACOT reagent:

- Heacock & Forrest 1973: 0.01 gram of tetranitro-2,3:5,6-dibenzo-1,3a,4,6a-tetra-azapentalene in 100 ml of acetonitrile. Freshly prepared before use.
- **TCBI** should be an *excellent* TLC reagent and one that will probably show differentiation between MOST if not all of the compounds listed here.
- Unfortunately Vinson & Hooyman 1975 did not evaluate it for any of them.
- Svendsen & Verpoorte 1983: 100 mg of N,2,6-trichloro*p*-benzoquinone imine is dissolved into 100 ml of a solution of chloroform-dimethyl sulphoxide (9:1) saturated with sodium bicarbonate. If stored in brown glass at 4°C; the solution is stable for 4 months.
- Applied as spray; chromophores developed by heating chromatogram 1-2 minutes at 110°C.
- Vinson & Hooyman 1975 recommended the plates be dried in an oven at 110°C for 5 minutes prior to spraying. After spraying lightly with the reagent, the plates were placed in the 110°C oven for 1 or 2 minutes. They noted that occasionally it was necessary to respray and reheat to optimize colors.

TCNE reagent:

Heacock & Forrest 1973: 1 gram of tetracyanoethylene in 100 ml of acetonitrile. Freshly prepared before use.

TetNF reagent:

Heacock & Forrest 1973: 1 gram of 2,4,5,7-tetranitro-9fluorenone in 100 ml of acetonitrile. Freshly prepared before use.

TNB reagent:

Heacock & Forrest 1973: 1 gram of 1,3,5-trinitrobenzene in 100 ml of acetonitrile. Freshly prepared before use.

TNF reagent:

Heacock & Forrest 1973: 1 gram of 2,4,7-trinitro-9fluorenone in 100 ml of acetonitrile. Freshly prepared before use.

Vanillin reagent: Made variously.

- Clarke's: 1 gram of vanillin is dissolved into 20 ml of Sulfuric acid (warming if necessary)
 - Add 2 drops to sample. Heat in water bath at 100° C for 30 seconds and note any color. Cool and dilute with a few drops of water and note any change in color.
- Jork et al. 1994:
- One gram of vanillin in 70 ml of 96% ethanol is treated cautiously with:
- a) 10 ml of concentrated sulfuric acid OR
- b) 10 ml of 85% ortho-phosphoric acid OR
- c) 10 ml of concentrated hydrochloric acid.
- The plate should be viewed and then heated to 70°. In the case of the phosphoric acid version, colors may only appear once heated. It is said to be the most sensitive of the three versions. Other modifications exist for specific purposes.
- Also made for use as a spray reagent by dissolving 1 to 5 grams of vanillin in 100 ml of hydrochloric acid or 50% methanolic hydrochloric acid or 4 grams in concentrated sulfuric acid.
- Touchstone 1992: Sulfuric acid version: 3 grams of vanillin in 100 ml of absolute ethanol. Add 0.5 ml of sulfuric acid and stir well. Spray and heat at 120°.
- Smith 1969: Vanillin 10% w/v in ethanol (2 volumes combined with 12N hydrochloric acid (1 volume).
- Vanillin-phosphoric acid reagent was used by Paris and coworkers.

Vanillin & sulfuric acid is used by some.

Vanillin in the presence of concentrated hydrochloric acid gave purple and blue with 3-alkyl indoles according to Ghosal & Mukherjee 1966.

Van Urk reagent:

- Clarke's: One gram of *p*-dimethylamino-benzaldehyde is dissolved in 100 ml of ethanol and 10 ml of concentrated hydrochloric acid is then added.
- Jork *et al.* 1994: 50 mg of *p*-dimethylaminobenz-aldehyde dissolved in 1 ml of concentrated sulfuric acid and brought to 100 ml with 95% ethanol.
- Smith & Seakins 1976: One gram of *p*-dimethylaminobenzaldehyde is dissolved in 34 ml of concentrated hydrochloric acid plus 50 ml. of ethanol and 16 ml. of water.
- This is normally only used on tlc plates.

- Colors should be viewed immediately under UV and then plates heated for 5 minutes in an oven and viewed for colored spots.
- Troxler et al 1959: 0.5 g *p*-dimethylaminobenzaldehyde, 100 ml water & 100 ml concentrated sulfuric acid.
- **To assay:** 1 mg of the substance is dissolved in 1 ml of ethanol and mixed with 2 ml of Van Urk's reagent then exposed to a quartz (mercury vapor) lamp for 10 minutes.
- Williams *et al.* 1971 used the formulation of Waldi 1962: One gram of *p*-dimethylamino-benzaldehyde is dissolved in 50 ml of hydrochloric acid and then 50 ml of ethanol is added.

After spraying it is heated at 50° C for 5 minutes and *aqua regia* vapors are blown across surface. (i.e. 3 parts hydrochloric acid and 1 part nitric. [Ed. This is a dangerous reagent. Other alternatives are recommended.])

Van Urk-Salkowski reagent:

Ehman 1977:

- Solution A: 1 gram of *p*-dimethylamino-benzaldehyde dissolved in 50 ml of hydrochloric acid to which is then added 50 ml of absolute ethanol. (Stable several months in brown glass.)
- Solution B: 2.03 grams of FeCl₃. $6H_2O$ in 500 ml of H_2O and 300 ml of concentrated sulfuric acid.
- A and B are mixed 1:3 then sprayed on developed silica gel chromatograms. The plate is then heated for 5 minutes at 100°C and washed several times with distilled water before being blotted dry.
- Once mixed the reagent is stable several weeks at room temperature.
- Has been successfully used with a densitometer for quantification of alkaloids.
- (Clarke's points out that the modern use of densitometers, [especially coupled with HPTLC] has brought the range of accuracy for quantitative estimates using tlc into that of GC and HPLC.)

Xanthydrol:

- Baxter & Slaytor 1972 describe as pink with unsubstituted indoles and blue with 5-substituted indoles.
- Clarke's: 0.1% xanthydrol reagent (0.1 g xanthydrol in 95 ml EtOH and 5 ml concentrated HCl). Will give a purple color with DMT and a blue color with methoxylated compounds such as 5-MeO-DMT.
- [Ivor Smith 1969 recommends 0.2 grams of xanthydrol, 90 ml. of ethanol and 10 ml. of concentrated HCl.]
- [Pyrroles and some phenolic acids also react with xanthydrol.]
- Frahn & Illman 1973 and Frahn & O'Keefe 1971 give as 0.1% xanthydrol in ethanol containing 5% syrupy phosphoric acid [0.1 gm in 95 ml of absolute ethanol and 5 ml of syrupy phosphoric acid] used as a spray reagent on dried paper pherograms. After spraying the dried paper they were heated 7 minutes at 110°. The first describes DMT as purple with this reagent and the second as pink.

- Gander *et al.* 1976 gives as 0.1 gram of xanthydrol in ethanol-11.7N hydrochloric acid (19:1) Same as Clark above.
- McComb *et al.* 1969: Used as 1% xanthydrol and 10% trichloroacetic acid in absolute methanol as a dip.
- Xanthydrol reaction reported to be temperature dependent. See also Dickman & Crockett 1956.
- Woods & Clark 1971 recommends that it be freshly prepared.
- We have not yet encountered it applied for any 4-substituted compounds and have no clue what color would result (blue?). It is quite valuable for substances like DMT, DET, DTP and similar.
- In tlc assays, of Appleseed, DMT and MMT were decidedly purple and 5-MeO-DMT was distinctly blue. Both purified plant materials and known reference standards for DMT and 5-MeO-DMT were used. Instances with both alkaloids co-chromatographing showed bands zoned with both colors.

CAUTION:

Always avoid skin contact with reagents; many contain caustic, corrosive and/or toxic components.

Be careful of not only liquids but vapors, sprays and mists.

The dangers of inhaling strong acids; whether as fumes (very bad) or aerosols (even worse) cannot be stressed enough.

An acquaintance who suffered acid vapor inhalation, due to exposure during precious metal refining, discovered his treatment to be surgical removal of the affected lung tissue.

Many common compounds can cause blindness if exposure is not treated immediately.

In most cases, as long as you can adequately wash the chemicals out of the eyes within several seconds of exposure, no permanent harm will result. There are MANY exceptions.

Solvents are frequently immediately toxic upon direct exposure but often have even more serious delayed effects. This can include organ damage or cancer stimulation or both.

Use adequate ventilation (preferably a fume hood), respiratory protection if needed, splash approved eye protection and long black lab rubber, PVC or nitrile gloves. (The glove material can vary depending on the solvent.)

An emergency shower is an excellent safety precaution. It should be easily locatable & operable with closed eyes, and located immediately adjacent to the lab.



Phalaris brachystachys modified from Robbins *et al.* 1951

Potassium iodoplatinate

Some advantages over many other reagents:

- a) It is about as sensitive as Dragendorff's (0.01 to $0.1 \,\mu g$ can be detected.)
- b) It is nondestructive; allowing the material on the chromatogram to be recovered if desired. This makes it a valuable reagent for locating and identifying zones during preparative chromatography.

Comments:

- Formamide impregnated plates will interfere with alkaloid detection but this can be overcome by spraying with 0.25% sodium nitrite in 0.5% hydrochloric acid to convert the formamide into formic acid which will not interfere with potassium iodoplatinate reagent.
- If the blue-violet background resulting from the presence of starches interferes with viewing of developed colors, it can be decolorized by spraying with a sodium hydrogen sulfite solution.
- Use of diethylamine in a solvent system will decrease the sensitivity of both Dragendorff's and potassium iodoplatinate reagents due to a darkening of the background.

Svendsen & Verpoorte 1983

To recover alkaloids from tlc plates after using potassium iodoplatinate

If using another reagent it is crucial that one determine it does not react irreversibly with the alkaloid in order to produce a chromophore!

If it does; it cannot be recovered:

1) Dry plate.

- 2) Scrape off spot containing alkaloid and place into a test tube.
- 3) Add a few drops of a sodium sulfite solution (saturated) and then 1 ml. of 0.5N sulfuric acid. (If necessary, the mixture can be heated to decolorize the solution.)
- 4) Saturate the resulting solution with sodium chloride.
- 5) Basify solution with a strong ammonia solution.
- 6) Extract with butanol-chloroform (1:9) or chloroform or diethyl ether to recover the alkaloid.
- A recovery rate of 70-80% can be expected for primary and secondary amines.

Svendsen & Verpoorte 1983 cited Holdstock & Stevens 1975.

To recover alkaloids from Dragendorff precipitates

(Dragendorff's test is performed in an acidic solution)

Treat with sodium carbonate and recover the liberated alkaloid by extracting with ether.

Silva et al. 1998

(Phosphomolybdic acid is said to also form precipitates that can permit alkaloid recovery. Bearing in mind that the molybdenum can prove toxic if not removed.)

Rapid Field Test for Alkaloids

From Culvenor & Fitzgerald 1963

(Thanks to Mulga & friends to providing us with a nice work-up on the details!):

Grind 2-4 grams of plant material with clean sand and a little chloroform (in a mortar & pestle). To the resulting thick slurry add 10 ml ammoniacal chloroform [6.71 ml ammonium hydroxide in 993.29 ml of chloroform (0.1N)] and macerate.

Draw off chloroform and filter into a test tube. Add 0.5-1 ml of dilute sulfuric acid (2N) to the test tube, shake and allow to separate.

Remove aqueous layer with a pipette (use cotton wool as a crude filter). Place 2 or 3 drops in a clean test tube.

Add either Mayer's reagent or silicotungstic acid reagent to form a precipitate.

The relative amount of the precipitate can provide a rough quantitative approximation of the alkaloid content if a baseline is first established using various concentrations of pure reference standards or known materials.

More on using this for a rough quantitative estimate: http://www.lycaeum.org/~mulga/quantitative html



Psilocybe mexicana (cultivated) Photos by Dr. P. C. Hickey





Several useful techniques for crude estimations of alkaloid content

Dried & milled grass was Soxhlet extracted with methanol. After removal of solvent under reduced pressure, the residue was extracted with dilute sulfuric acid.

Aqueous solution then brought to pH 9 with ammonia and extracted with chloroform.

After removal of the chloroform they were brought to a standard volume with chloroform.

Titration was done using 0.01N p-toluene sulfonic acid with dimethyl yellow as an indicator to give a quantitative estimation of the total alkaloid present.

Culvenor et al. 1964

McComb *et al.* 1969 used known amounts and a spectrophotometer to first determine a curve in order to estimate concentrations for their unknowns.

[They cut bands out of their developed chromatograms and extracted; performing colorimetric estimations while in solution.]

A similar approach was used by Der Marderosian *et al.* 1968 to estimate the DMT content in "*Banisteriopsis rusbyana*" [actually *Diplopterys cabrerana*] leaves using PDAB & a spectrophotometer.

They first established a Beer's law line using known amounts of pure DMT under the same conditions (absorbance read at 600 nm).

Mulvena & Slaytor 1983 identified alkaloids by co-tlc and used the color reactions obtained with the Van Urk-Salkowski reagent coupled with a scanning densitometer to estimate concentration. [citing Ehmann 1977]



female *Bufo alvarius* (above)



Anadenanthera colubrina (Oakland, California)

Trout's Notes on Tryptamines



Acacia obtusifolia seedling



The future begins with the seeds that are planted today.



Above shows same seedling on left but 4 days later Juvenile leaves will disappear as the plant grows. Phyllodes are modified petioles rather than true leaves.



Psilocybe cubensis (USA) Photo by Ringworm



Diplopterys cabrerana (Ecuador) Photo by Bobby Brown



Acacia obtusifolia (Australia) Photo by Snu Voogelbreinder

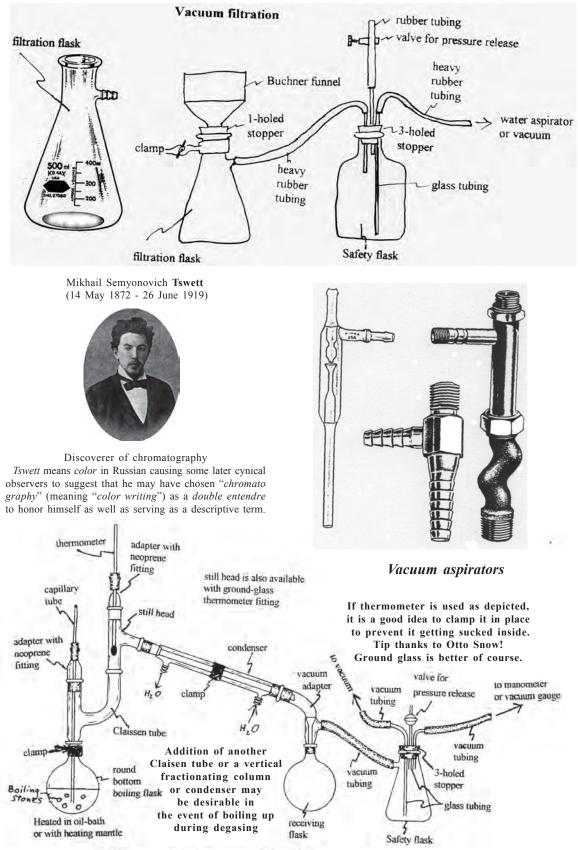


(Austin, Texas)

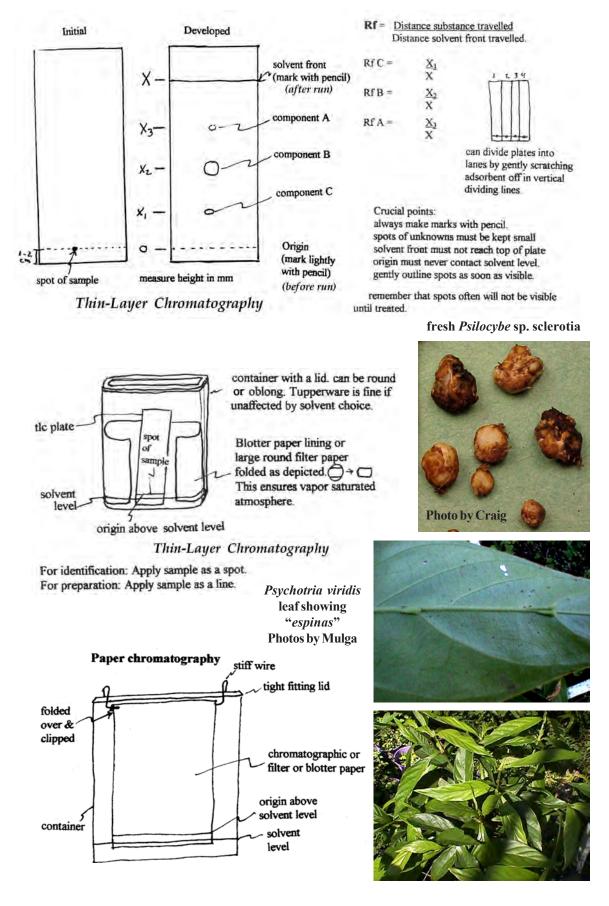


from a major hardware chain

Several plants containing simple tryptamine derivatives



Vacuum distillation apparatus (typical)



Reported Rf Values for Some Selected Tryptamines

Assembled by Keeper Trout

(no attempt has been made to separate isomeric forms of some of the alcohols.)

B: DMT [D [B]: DMT-N-Oxide E:		[D]: PSO E: 5-OH	D: PSOH [D]: PSOP E: 5-OH-DMT [E]: 5-OH-DMT-N-oxide		F: 5-MeO-MMT G: 5-MeO-DMT [G]: 5-MeO-DMT-N-oxide			
<u>Solvent System</u>				<u>Rf of Al</u>	kaloid			
[Medium]	А	В	С	D	E	F	G	Reference
[meanin]	1	[B]			E [E]	1	[G]	Reference
		[D]	[C]	[D]	[E]		[G]	
Acetic acid (2N) [Silica Gel] Acetone- <i>i</i> -Propanol-Water-	na	0.50	na	na	0.65	na	0.60	10
conc. Ammonia (50:40:7:3)	na	na	na	na	0.54	na	na	52
[Silica Gel] Ammonium chloride (3% aqueous solution) [Paper]	na	na	na	na	0.51	na	na	9
Conc. Ammonium hydroxide- <i>p</i> -Dioxane-Acetone (2.5:45:5.5) (v/v) [Silica Gel]	na	na	0.21	0.20	na	na	na	50
t-Amyl alcohol-Formic Acid- Water (10:1:10) [Paper]	na	0.78	na	na	na	na	na	43
Benzene-Ethanol- 30% Methylamine (22:7:1)	na	na	na	na	0.66	na	0.79-0.8	0 14
[Silica Gel] Benzene-Ether (1:4) saturated with 18M Ami	0.5 nonium	0.9 hydroxide	na	na	na	na	na	35
[Alumina] Benzene-Ethyl acetate (3:1)	003	na	na	na	na	na	na	34
[Silica Gel]								
Benzene-Methanol (90:10) [Alumina] Benzene-Methanol-	na	0.45	na	na	na	na	na	47
5% Ammonium hydroxide	0.20	0.46	na	na	0.36	na	0.44	28
(10:15:2)	na	0.61	na	0.57	0.43	na	na	33
[Silica Gel]				[0.01]				33
n-Butanol-Acetic acid-Water	na	0.48	na	na	na	na	0.42	18
(80:3:17)	na	0.48	na	na	0.27	na	0.38	9
[Paper] <i>n</i> -Butanol-Acetic acid-Water (10:1:10)	na	na	na	na	na	na	0.66	43
[Paper] <i>n</i> -Butanol-glacial Acetic acid- Water (5:1:2)	na	na	na	na	0.54	na	na	27
[Paper] <i>n</i> -Butanol-Acetic acid-Water (4:1:1)								
[Paper]	0.61	na	na	na	na	na	na	34
[Paper]	na	0.60	na	na	na	na	na	11
[Silica gel G & Paper]	na	0.85 [na]	na	na	0.69 [0.45]	na	0.78	58 58

Notes for this section are on page 176

A: MMT B: DMT [B]: DMT-N-Oxide C: DET [C]: DPT	B: DMT [B]: DMT-N-Oxide C: DET		D: PSOH [D]: PSOP E: 5-OH-DMT [E]: 5-OH-DMT-N-oxide		F: 5-MeO-MMT G: 5-MeO-DMT [G]: 5-MeO-DMT-N-oxide			
Solvent System				Rf of A	lkaloid			
[Medium]	А	В	С	D	E	F	G	Reference
[wieulum]	А					r		Kelefence
Butanol-Acetic acid-Water (12:3:5)		[B]	[C]	[D]	[E]		[G]	
[Paper]	na	na	na	na	na	0.58	na	63
[Paper]	na	na	na	0.72	na	na	na	49
	na	na	na	0.75	na	na	na	29
	na	na	na	[0.44]	na	na	na	29
	na	na	na	[0.42] [0.43]	na	na	na	49 49
[Cellulose]	na	na	na	0.78	na	na	na	6
[Silica gel]	na	na	na	[0.48] 0.36 [0.16]	na	na	na	6 6
	na	na	na	0.75	na	na	na	38
	na	na	na	0.72	na	na	na	38
				[0.44] [0.42]				38 38
[SilCel]	na	na	na	na	0.40	na	na	55b
<i>n</i> -Butanol-Acetic acid-Water (4:1:2)	na	0.74 [0.82]	na	na	na [na]	na	0.72 [0.89]	5
[Paper] n-Butanol-Acetic acid-Water (120:30:50)								
[Paper]	na	0.78	na	na	na	na	0.72	31
[Paper] Note 3	0.78	0.92	0.85	na	na	na	na	51
[Silica Gel]	na	0.86	na	na	na	na	na	31
n-Butanol-Acetic acid-Water	na	na	na	na	0.51-0.5	6 0.62-0.	65 0.67-0.	69 14
(40:10:50)	na	0.87	na	na	0.60	na	0.78	41
	na	na	na	na	0.47	na	na	67
	na	na	na	na	na	0.68	na	57
[Paper]		[0.86]			[0.67]		[0.81]	41
n-Butanol-Acetic acid-Water (40:10:50) [Silica Gel]	na	na	na	na	0.40	na	0.40	14
n- Butanol-Acetic acid	na	0.67	na	na	0.62	na	na	15
(10:4); saturated with water	0.74	0.72	na	na	na	na	na	16
[Paper]	na	0.74	na	na	0.63	0.70	0.72	23
		[0.76]			[0.72]		[na]	15
		[0.80]			[na]		[na]	16
		[0.81]			[na]		[0.89]	23
n-Butanol-Acetic acid-Water	na	na	na	[0.19]	na	na	na	45
(24:10:10)	na	na	na	0.50-0.5	54 na	na	na	65
[Kieselgel G]				[0.22-0.	33]			65
<i>n</i> -Butanol-glacial Acetic acid-W (2:1:1)	ater							
[Silica gel]	0.52	0.47	na	na	0.46	na	0.47	28
[Silica gel]	na	na	na	0.46	na	na	na	6
				[0.21]				6
[Silica gel]	na	na	na	0.52	na	na	na	30 30
[Silica gel]	0.52	0.43	na	0.40	0.37	na	na	39 39
[Silica gel]	na	na	na	0.58	na	na	na	64 64
[Silica gel-Kieselgur (2:1)]	na	0.51	na	0.55	0.46	na	na	33 33
Butanol-Acetic acid-Water				[0.15]				
(4:2:1) [Silica gel]	na	0.23	na	na	na	na	na	11

Trout's Notes on Tryptamines: Rf Table

A: MMT B: DMT [B]: DMT-N-Oxide C: DET [C]: DPT		D: PSOI [D]: PSO E: 5-OH [E]: 5-OI	N-oxide	F: 5-MeO-MMT G: 5-MeO-DMT [G]: 5-MeO-DMT-N-oxide xide					
Solvent System				Rf of A	lkaloid				
[Medium]	Α	B [B]	C [C]	D [D]	E [E]	F	G [G]	Reference	
<i>t</i> -Butanol-Acetic acid-Water (70:2:29)									
[Paper]	na	080	na	na	0.70	na	0.75	10	
[Silica Gel] Butanol saturated with	na	050	na	na	0.30	na	0.40	10	
3% Ammonia	na	na [na]	na	na	0.65 [0.46]	na	na	55a	
[Silica/ Kieselgel] Butanol-Acetic acid-Water- i-Propanol (8:2:5:1)	na	na	0.18	0.21 [0.16]	na	na	na	19	
[Silica gel] Butanol-Ethyl acetate-Water (70:60:25) [v/v]	na	na [na]	na	na	0.76 [0.25]	na	na	55a	
[Alumina] t-Butanol-Formic acid-Water (21:0.6:3) [Paper]	na	0.79 [0.84]	na	na	0.61 [0.71]	na	0.74 [0.81]	41	
<i>t</i> -Butanol-Formic acid-Water (21:0.6:9) [Paper]	na	0.81 [0.82]	na	na	0.63 [0.69]	na	0.71 [0.80]	41	
<i>t</i> -Butanol-Formic acid-Water (207:6:87) [Silica Gel]	na	0.69 (hydrogen oxalate)	na n	na	na	na	0.73	31	
<i>t</i> - Butanol -Formic acid-H ₂ O (207:6:87)	na 0.70	0.71 0.66 [0.72]	na na	na na	0.51 na [0.62]	na na	na na	15 16 15	
[Paper] Butanol-Formic acid-Water (16:1:3) [Cellulose]	0.75	[0.74] 0.75	na	na	0.45	0.65	0.65	16 60	
<i>n</i> -Butanol-2N HCl (1:1, upper phase) [Cellulose]	0.6	0.6	na	na	0.31	0.43	na	36a	
Butanol-sat. w/ N HCl [Paper]	0.46-0	.50 0.46-0.50	na)	na	na	na	na	13b	
<i>n</i> -Butanol-30% Methylamine (80:30)	na	na	na	na	0.81-0.8	8 0.92-0.9	94 0.92-0.9	3 14	
[Paper] <i>n</i> -Butanol-30% Methylamine [Silica Gel]	na	na	na	na	0.91	na	0.97	14	
<i>n</i> -Butanol- <i>i</i> -Propanol-Water (8.5:1:2)	na	na	na	[0.16]	na	na	0.97	19	
[Silica Gel] <i>n</i> -Butanol-Pyridine-Water (15:10:10:3)	na	na	na	0.55 [0.10]	na	na	na	30	
[Silica gel] <i>n</i> -Butanol-Pyridine-Water (60:60:60)	0.82	0.82	0.83	na	0.79	na	na	51	
[Paper] Note 4 <i>n</i> -Butanol saturated with Water	na	na	na	0.50 [0.04]	na	na	na	29 29	
[Paper]	na	na	na	0.44 [0.07] [0.09]	na	na	na	49 49 45	

A: MMT B: DMT [B]: DMT-N-Oxide C: DET [C]: DPT		D: PSOI [D]: PSO E: 5-OH- [E]: 5-OI)P ·DMT	N-oxide	F: 5-MeO-MMT G: 5-MeO-DMT [G]: 5-MeO-DMT-N-oxide				
<u>Solvent System</u>				<u>Rf of A</u>	lkaloid				
[Medium]	A	B [B]	C [C]	D [D]	E [E]	F	G [G]	Reference	
<i>n</i> -Butyl acetate- <i>n</i> -Butanol- Acetic acid-Water	na na	0.55 0.53	na na	na na	0.26 na	0.51 na	na 0.51	12 5	
(85:15:40:22)	na na na	0.74 0.58 na	na na na	na na na	0.27 na [0.18]	0.58 na na	na na na	24 21 25	
[Paper] Chloroform	na	[0.89]	na	na	na	na	na	21	
[Alumina] [Paper] [Paper]	0.4 0.00 na	0.5 0.92 (ref) 0.94 (exp)		na na na	na na na	na na na	na na na	17 4 4	
Chloroform-Acetic acid-Methar (65: 20: 10: 5) [Silica gel]	na na	er na	na	na	0.15	na	na	52	
Chloroform-Cyclohexane- conc. Ammonium hydroxide (20:10:1) (lower phase) [Silica gel]	na	0.02	na	0.06 [0.0]	0.0	na	na	33	
Chloroform-Diethyl ether- Methanol-25% Ammonia (75:25:5:1) [Silica gel]	na	0.43	0.58	0.58 [0.03]	na	na	na	61	
Chloroform-Methanol (95:5) [Alumina]	na	na	na	na	0.15	na	0.70	10	
Chloroform-Methanol (90:10)		0.16	0.16					1	
[Silica Gel] [Alk. Silica Gel Note 8]	na na 0.05	0.16 0.09 0.16	0.16 0.10 0.16 [0.39]	na 0.09 na [0.01]	na na 0.07	na na na	na na na	1 8 44 44	
[Alumina] [Alumina]	na	0.66 [0.91]	na	na	na [na]	na	na	9 23	
Chloroform-Methanol (1:1) [Alumina] Chloroform-Methanol-	0.3	0.7	na	na	na	na	na	17	
Acetic acid (75:20:5) [Silica Gel]	na	0.10	na	na	na	na	na	20	
Chloroform-Methanol saturated with Ammonia (20:1) [Silica Gel]	na	na	na	na	0.17	na	0.48-0.5	0 14	
Chloroform-Methanol (9:1) [Alk. with NH3: Note 9] [Silica Gel] Chloroform-Methanol-	na	0.53	na	na	0.25	na	na	62	
Ammonium hydroxide (49:7:1) [Alumina]	0.4	0.9	na	na	na	na	na	35	
Chloroform-Methanol- 1N Ammonium hydroxide (80:15:1) [Silica gel]	na na	na na	na na	na na	na na	na na	0.36 0.15	47 40	

A: MMT B: DMT [B]: DMT-N-Oxide C: DET [C]: DPT		D: PSC [D]: PS E: 5-OF [E]: 5-C	OP	N-oxide	F: 5-M G: 5-M [G]: 5-			
<u>Solvent System</u>				Rf of A	lkaloid			
[Medium]	Α	B [B]	C [C]	D [D]	E [E]	F	G [G]	Reference
Chloroform-Pyridine (6:1)	0.32	0.56	na	na	0.14	0.36	0.59	3
[Paper; Formamide impregnat Cyclohexane-Toluene-Diethylar (75:15:10)		0.09	0.15	0.05	na	na	na	8
[Alk. Silica Gel: Note 8]	nu	0.09	0.15	0.02	nu	nu	nu	0
Ethanol-Ammonia	na	0.93	na	na	na	na	0.90	5
(4:1) [Paper]		[0.63] [0.58]			[na]		[0.59]	5 21
Ethyl acetate-Chloroform-Meth	anol	[0.50]						21
(2:2:1)	na	0.05	na	na	na	na	na	11
[Silica gel]								
Ethyl acetate-Methanol- 58% Ammonium hydroxide								
(80:15:5)	0.29	0.58	na	na	0.40	na	0.55	28
[Silica Gel]								
Ethyl acetate- <i>n</i> -Propanol-								
28% Ammonium hydroxide	0.21	0.49						7
(40:30:3) [Silica Gel]	0.21	0.48 [0.0]	na	na	na	na	na	7
Ethyl methyl ketone-Dimethylf	ormami		nia solutic	on				
(13:1.9:0.1) (Note 10)	na	0.35	na	na	na	na	na	20
[Silica Gel]								
Methanol								
[Silica Gel]	0.12	na	na	na	na	na	na	34
[Alk. Silica Gel: Note 8]	0.12	0.27	0.29	na	0.21	na	na	44
[Alumina]	0.3	0.7	na	na	na	na	na	17
[Paper] (reference material) [Paper] (Experimental values)	0.22 0.21	0.62 0.61	na	na	na	na	na	4 4
Methanol-Acetic acid-Water	na na	na	na na	na na	na na	na na	na 0.58	47
(75:10:15)	na	na	na	[0.25]	na	na	na	37
[Silica gel]								
Methanol-conc. Ammonium hyc (100:1.5)								
[Silica Gel]	0.16	0.35	0.38 [060]	[0.04]	0.32	na	na	44 44
[6061]	0.21	0.41	0.46 [0.65]	[0.09]	0.35	na	na	44 44
[6060]	0.21	0.41	0.46 [0.65]	[0.09]	0.35	na	na	44 44
[Alk. Silica Gel: Note 8]	0.18	0.40	0.46	0.39 [0.05]	na	na	na	8 8
دد	na	na	0.29	na	na	na	na	44
[Kieselgel GF]	na	0.34	na	[0.05]	na	na	na	54
Methanol-conc. Ammonium hyc		10 C		0.45				6
(98.1:1.5) [Silica Gel] Methanol-Ammonium	na	na	na	0.45 [0.14]	na	na	na	6 6
hydroxide (29%) (7:1) [Silica gel]	0.25	0.50	na	na	0.45	0.25	0.50	60
Methanol-conc. Hydrochloric (9:1)	0.75	0.60	na	na	0.60	0.75	0.60	60
[Silica Gel]								

A: MMT B: DMT [B]: DMT-N-Oxide C: DET [C]: DPT	D: PSOH [D]: PSOP E: 5-OH-DMT [E]: 5-OH-DMT-N-oxi			F: 5-MeO-MMT G: 5-MeO-DMT [G]: 5-MeO-DMT-N-oxide I-oxide					
<u>Solvent System</u>				Rf of All	bioles				
[Medium]	А	В	С	D	E	F	G	Reference	
		[B]	[C]	[D]	[E]	F	[G]		
Methanol-Chloroform	na	0.93	na	na	na	na	na	20	
(1:1)									
[Alumina]									
Methanol-Chloroform	na	0.16	na	na	na	na	na	1	
(1:9) [Silica Cal with fluorescence in	diaatarl								
[Silica Gel with fluorescence in Methanol-Chloroform-	lateator								
Acetic acid	na	0.47	na	na	na	na	na	26	
(75:25:15)		[0.88]			[na]				
[Silica Gel]									
Methanol-Methyl ethyl ketone	na	na	na	na	0.50	na	na	55a	
(1:1)		[na]			[0.05]				
[Alumina] Methyl ethyl ketone- <i>t</i> -Butanol-		0.02			0.80			15	
Diethylamine-Water	na 0.94	0.92 0.95	na na	na na	0.89 na	na na	na na	15 16	
(40:40:4:20)		[0.70]			[0.46]			15	
[Paper]	[0.68]							16	
Morpholine	na	0.34	na	na	0.43	na	0.25	48	
(0.1 M in water)									
[Silica Gel G]		0.54	0.61	0.46					
Morpholine-Toluene (1:9)	na	0.54	0.61	0.46	na	na	na	1	
[Silica Gel with fluorescence in	ndicatorl								
<i>n</i> -Pentanol-Pyridine-30% Meth	-	Water							
(40:40:1:10)	na	na	na	na	0.72-0.74	0.63-0.6	6		
							0.82-0.88	14	
[Paper]					0.72.0.72	0.20.0.4	2		
Potassium chloride	na	na	na	na	0./2-0./3	0.39-0.42	2 0.46-0.47	14	
(20% w/v)	0.57	0.60	0.62	na	0.43	na	na	51	
[Paper] [Notes 1, 2, 6]									
n-Propanol-Acetic acid-Water	na	na	na	0.36	na	na	na	19	
(10:3::3)				[0.23]					
[Silica gel] <i>n</i> -Propanol-Ammonium hydrox	ida								
(8:3)	na	na	na	na	na	0.86	na	57	
[Paper]	nu	nu	nu	nu	nu	0.00	nu	01	
Propanol-Ammonium hydroxide	e na	na	na	na	0.90	na	na	55a	
(5:1) [v/v]		[na]			[0.45]			55a	
[Cellulose]					0.90			55b	
<i>n</i> -Propanol- 1N Ammonium		0.07			0.05		0.00	-0	
hydroxide	na	0.97	na	na	0.85 [0.50]	na	0.92	58	
(5:1) [Silica gel G & Paper]		[na]			[0.50]				
<i>n</i> -Propanol-Ammonium									
hydroxide	na	0.91	na	na	na	na	0.90	31	
(5:1)									
[Silica gel]	na	na	na	na	na	na	0.13	47	
[Silica gel]	na	na	na	[0.05]	na	na	na	38	

Trout's Notes on Tryptamines: Rf Table

A: MMT B: DMT [B]: DMT-N-Oxide C: DET [C]: DPT		D: PSO [D]: PSO E: 5-OF [E]: 5-O	ОР	N-oxide	G: 5-Me	eO-MMT eO-DMT deO-DMT	'-N-oxide	
Solvent System				Rf of A	lkaloid			
[Medium]	Α	В	С	D	Е	F	G	Reference
		[B]	[C]	[D]	[E]	F	[G]	
n-Propanol-1N Ammonium	na	0.86	na	na	0.82	na	na	15
hydroxide (5:1)	0.75	0.83	na	na	na 0.77	na	na	16 9
[All on Paper]	na	na [0.56]	na	na	[0.35]	na	na	9 15
		[0.59]			[0.35] [na]			16
	n 9	na	na	0.85	na	na	n 9	49
	na	IIa	na	[0.05]	IIa	na	na	49
	na	na	na	0.86	na	na	na	29
				[0.05]				29
Propanol-28% Ammonium								
hydroxide	na	0.93	na	na	0.89	na	0.90	41
(5:1)		[0.68]			[0.54]		[0.64]	
[Paper]								
n-Propanol-5% Ammonium h (5:2)	iyaroxiae							
[Silica Gel]	0.40	0.53	na	na	0.47	na	0.52	28
[Silica Gel]	0.40	0.55	Па	[0.27]	0.47	IIa	0.52	20 45a
[Silica gel]								43a 32
[Silica Gel]				[0.19] [0.12]				30
[Silica gel/Kieselguhr (2:1)]	20	n 0	n 0	0.79	20	n 0	20	42
[Silica gel/Kieseiguili (2.1)]	na	na	na	[0.19]	na	na	na	42
[Silica gel/Kieselguhr (2:1)]	na	0.79	na	0.76	0.74	na	na	33
				[0.15]				33
n-Propanol-6% Ammonium h	nydroxide							
(5:2)	na	na	na	[0.16]	na	na	na	45b
[Silica Gel]								
<i>n</i> -Propanol-Water-Ammoniu hydroxide	m na	na	na	0.58	na	na	na	6
(500:188:12)	na	Πά	па	[0.11]	IIa	па	Πά	0
[Silica gel]				[0.11]				
<i>n</i> -Propanol-Water-Ammoniu	m							
hydroxide	na	na	na	0.82	na	na	na	6
(150:50:10)				[0.16]				
[Silica gel]								
<i>i</i> -Propanol-Ammonium hydroxide (880)-Water	0.88	0.92	0.98	na	0.90	na	na	51
(200:10:20)	0.00	0.72	0.90	nu	0.90	nu	nu	01
[Paper] [Notes 2, 5]								
<i>n</i> -Propanol-Ammonium								
hydroxide (880)-Water								
(16:1:3)	na	na	na	na	0.86	na	na	67
[Paper]								
<i>i</i> -Propanol-Ammonium hydroxide-Water	110	0.89	na	na	na	na	n 9	11
(10:1:1)	na	0.89	na	na	na	na	na	11
[Paper]								
<i>i</i> -Propanol-Ammonium								
hydroxide-Water	na	0.91	na	na	0.82	0.86	0.97	23
(9:1:1)	na	0.91	na	na	0.83	na	na	22
		[0.63]			[na]		[0.56]	23
[Paper]		[0.63]			[na]		[na]	22

A: MMT B: DMT [B]: DMT-N-Oxide C: DET [C]: DPT		[D]: PS E: 5-01	D: PSOH [D]: PSOP E: 5-OH-DMT [E]: 5-OH-DMT-N-oxide			F: 5-MeO-MMT G: 5-MeO-DMT [G]: 5-MeO-DMT-N-oxide				
<u>Solvent System</u>				<u>Rf of A</u>	lkaloid					
[Medium]	Α	В	С	D	Ε	F	G	Reference		
		[B]	[C]	[D]	[E]	F	[G]			
n-Propanol-Acetic acid-Water										
(10:3:3)	na	na	0.30	na	na	na	na	19		
[Silica gel]										
<i>i</i> -Propanol-Ethyl acetate-conc.		5	oxide							
(120:30:6)	0.16	0.34	na	na	na	0.13	0.28	59		
[Silica Gel]										
<i>i</i> -Propanol-Ethyl acetate-conc.	Ammor	nium hydr	oxide-2-Et	thoxy-etha	nol					
(60:15:3:5)										
[Avicel-Silica gel]	na	na	na	na	na	0.51	0.83	36a		
[Silica gel]	0.23	0.57	na	na	na	na	na	36b		
<i>i</i> -Propanol-Water				FO (2)						
(9:1)	na	na	na	[0.63]	na	na	na	53		
[Paper]										
<i>n</i> -Propanol-Water (3:1)					0.59			67		
	na	na	na	na	0.39	na	na	0 /		
[Paper] Sodium chloride (8% aqueous w	/w)									
glacial Acetic acid	0.57	0.60	0.63	na	0.47	na	na	51		
(200:2)										
[Paper] [Note 1, 2, 6]										
Sulfurous acid	na	0.37	na	na	0.45	na	0.27	48		
(0.1 M in water)										
[Silica Gel G]										
Toluene-Morpholine	na	0.54	na	na	na	na	na	56		
(9:1)										
[Silica gel]										
Distilled Water	na	na	na	na	0.25-0.3	0 na	na	13		
[Paper]										

Rf table Notes

Note 1: These are corrosive to metal parts and cannot be dried with a fan as the paper becomes fragile but provide very compact excellent spots and very constant Rf values

Note 2: Using Whatman No. 1 Chromatography Paper on a Universal Apparatus with ascending frame using a 8 to 9 inch run from the origin and performed at room temperature.

Note 3: 7 hours for 8 inch rise. 15 to 30 minutes for removal with fan.

Note 4: 8 hours for 8 inch rise. 15 to 30 minutes for removal with fan.

Note 5: 8 hours for 8 inch rise. 1/2 hour to remove with fan

Note 7: One hour to dry

Note 8: Alkalinized Silica Gel; treated with 0.1M KOH and dried

Note 9: Chloroform contained 1% Ammonium hydroxide

Note 10: Ammonia sp. gr. 0.90

An odd note with no good place for it:

Debenzylation can be done with Hydrogen and Palladium-Charcoal (10%) at atmospheric pressure.

[For the decomposition of the benzoyl- intermediary used in some synthetic approaches to tryptamines.]

Methanol was used as the solvent and mixture warmed if necessary. Barlow & Khan 1959



Phalaris canariensis modified from Robbins *et al.* 1951

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Phalaris aquatica

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Psilocybe subaeruginosa (Australia) Photos by R. Kundalini (L) & Snu Voogelbreinder (R)

Acacia obtusifolia flowering





Reported Chromatographic Assay Data and Alkaloid Tests. Some procedures and results taken from the literature.

Assembled by Trout 1996

Agurell et al. (1969) Acta Chemica Scandinavica 23 (3): 903-916: Agurell recommends Chloroform-Pyridine (6:1) on Formamide impregnated paper (FCP) or silica gel G with

Methanol-Acetic acid-Water (75:10:15) for the separation of tryptamines and β -carbolines

Alkaloid	Ikaloid <u>Fluorescence Maxima</u> in ethanol in 3M HCl		Rf in FCP	
	nm	nm		
Tryptamine (T)	(???)	0.07		
MMT			0.32	
DMT			0.56	1.1
Serotonin			0.00	1
5-OH-MMT			0.04	1
Bufotenine			0.14	
5-MeO-MMT			0.36	
5-MeO-DMT	335	520	0.59	911
7-Methoxy-DMT	355		OT 1	
4-Benzoyl-DMT	345	_		
2-Methyltetrahydro-		_		
β -carboline (MTHC)	350		No color with Ehrlich's	
2-Methyl-6-methoxy-1,2,3,4	4-tetrahydro-	β-carboline	Sec. St.	91 6 7 A P 2
(6-MeO-THC)	335	515	No color with Ehrlich's	
6-MeO-DMTHC	335	520	No color with Ehrlich's	
Tetrahydroharmine	355	-	[McKenna notes no immediate reaction but tu robin's-egg blue when left overnight]	urning

N-Methyltetrahydroharmine 355

TLC System used by Johnny Appleseed:

Methylene chloride: Methanol: concentrated Ammonia (80:15:1) on silica gel.

Arthur et al. (1967) Australian Journal of Chemistry 20: 811-813:

Chromatography on paper:	$\underline{\mathbf{R}}\mathbf{f}$			
	Methanol	Chloroform		
MMT (experimental)	0.21	0.00		
MMT (authentic)	0.22	0.00		
DMT (experimental)	0.61	0.94		
DMT (authentic)	0.62	0.92		

Banerjee & Ghosal (1969) Australian Journal of Chemistry 22: 275-277:

Using paper chromatography with Whatman no. 1 paper and Dragendorff's &Ehrlich's reagents. Solvent systems:

A: n-Butyl acetate-n-Butanol-Acetic acid-Water (85:15:40:22.

B: *n*-Butanol-Acetic acid-Water (4:1:2)

C: Ethanol-Ammonia (4:1)

C. L'unanoi Ammonia (=	ŧ.ı)		
Alkaloid	Rf A	Rf B	Rf C
DMT	0.53 (0.58) [†]	0.74	0.93
DMT-N-oxide	na (0.89)† (0.88)*	0.82	0.63 (0.58)*
5-MeO-DMT	0.51	0.72	0.90
5-MeO-DMT-N-oxide	na	0.89	0.59
5*D.C (11 C) 1	0 D : 10(0 *DC	. 11	C1 1 . 1 10

[*Rf reported by Ghosal & Banerjee 1969; *Rf reported by Ghosal et al. 1970; using the same system and medium.]

Baxter & Slaytor (1972b) Phytochemistry 11: 2767-2773:

Using Phalaris tuberosa var. Australian Commercial.

They separated the alkaloid from the plant material by homogenizing in 80% ethanol, heating 15 minutes on a steam bath and centrifuging.

Basic compounds were separated by used of a cellulose phosphate cation exchange column (in H+ form) and eluted with Ethanolconc. Ammonium hydroxide (4:1). After removal of the alcohol, the alkaloids were separated from amphoteric compounds by extracting with Ethyl acetate.

They used two dimensional tlc to separate the alkaloids. (Merck A1 precoated silica gel F_{254} plates). Isopropanol-conc. Ammonium hydroxide (8:1:1) was used first and then *n*-Butanol-Acetic acid-Water (12:3:5).

They found that GLC with W-98 on Diatoport S 80-100 mesh did not separate DMT from tryptamine or 5-MeO-DMT from 5-MeO-tryptamine.

Psychotria viridis New plant. sprouting from a leaf cutting. Photo above by Mulga

(Multiple offshoots can occur when the leaf is folded accordianlike or rolled like a short cigar before being planted.)

Trout's Notes FS-X7: Chromatographic Assay Data and Alkaloid Tests

An interesting screening was presented by Brown et al. (1972) Journal of Chromatography 64: 129-133: Their sample was dissolved in 95% ethanol for application.

Using silica gel plates without a fluorescent marker (Merck, 0.25 mm; used 5x20 and also 20x20) they activated by heating the plates (before use) at 105° for 60 minutes and then stored in a desiccator over silica gel to keep dry.

[When activating plates they should be allowed to cool in a desiccator as well as being stored there.]

Two duplicate plates were made for each sample.

Their solvent was Ethyl acetate-n-Propanol-28% Ammonium hydroxide (40:30:3).

Plates were allowed to develop around 70 minutes (solvent traveled 10 cm.) and they then first checked under a UV light (254 nm) for fluorescence and then lightly sprayed with IPA (Iodoplatinate); Colors were allowed to develop 5 minutes. A second plate was then sprayed with p-Dimethylaminobenzaldehyde reagent, heated at 105° for 5 minutes and read.

	Fluorescence	<u>Rf</u>	<u>Color with IPA</u>	<u>Color with</u> PDAB
DMT	None	0.48	Blue	Blue
LSD (pure)	Blue	0.69	Purple	Blue
(LSD as street samples)	Blue	0.69	Purple-brown	Blue

Clarke's Isolation and Identification of Drugs in Pharmaceuticals, Body Fluids and Post-Mortem Materials:

Solvent systems: (All using Silica Gel G; dipped in or sprayed with 0.1M Potassium hydroxide in Methanol & dried.) A: Methanol-concentrated Ammonium hydroxide (100:1.5)

B: Cyclohexane-Toluene-Diethylamine (75:15:10) Rf

C: Chloroform-Methanol (90:10)

Alkaloid

	Α	В	С
DMT	0.40	0.09	0.09

Culvenor et al. (1964) Australian Journal of Chemistry 17: 1301-1304:

Working with	th Phalo	uris they used a	scending paper	r chromatography in Butanc	ol-Acetic acid-Water (80:3:17).
Alkaloid	<u>Rf</u>	Iodine vapor	<u>Dragendorff</u>	Ehrlichs	α <u>-Nitroso-β-naphthol</u>
Bufotenine	0.27	Red-brown	Red-Brown	Dark Blue	Violet
5-MeO-DMT	0.38	Red-brown	Red-Brown	Dark Blue	Weak Brown [†]
DMT	0.48	Red-brown	Red-Brown	Mauve turning Dark Blue	Weak Brown*†
There were se	varal at	har alkalaida pr	acont which the	w did not identify but gave	Pf and color reactions

There were several other alkaloids present which they did not identify but gave Rf and color reactions. Bufotenine was present in all fresh samples but not all dried samples and if so was present in considerably reduced amounts.

*Ivor Smith and [†]Ghosal have reported no reaction with this reagent.

 α -Nitroso- β -naphthol-nitrous acid is said not react with most of the tryptamines. [Culvenor observed a weak reaction but it is unclear if this was due to impurities or another reason.]

It will react with bufotenine and with serotonin. It will not react with their O-methyl ethers. [See note on Culvenor above.] It also reacts with hydroxylated phenethylamines such as tyramine (but not with their methoxylated analogs).

It can be used to differentiate p-hydroxyphenethylamines from hydroxytryptamines due to the fact that it produces a reddish purple color with the *p*-hydroxyphenethylamines that fades to yellow over time while the violet produced by hydroxytryptamines is stable for long periods.

De Budowski et al. (1974) Il Farmaco (Edizione Scientifica) 29 (8): 574-578:

Using reference materials with Van Urk's reagent to visualize.

A: Chloroform-Methanol (95:5)

B: 2N Acetic acid

C: t-Butanol-Water-Acetic acid (70:29:2))

Solvent System		<u>Rf</u>		
-	DMT	Serotonin	Bufotenine	5-MeO-DMT
A: (on alumina)	na	na	0.15	0.70
B: (on silica gel)	0.50	0.80	0.65	0.60
C: (on silica gel)	0.50	0.70	0.30	0.40
C: (on Whatman No.1 paper)	0.80	0.65	0.70	0.75

Dutta & Ghosal (1967) Chemistry and Industry 2046-2047:

Paper: n-Butyl acetate-n-Butanol-Acetic acid-Water (85:15:40:22) on Whatman No. 1

Alkaloid	<u>Rf on paper</u>
Gramine	0.53
DMT	0.55
Bufotenine	0.26 & 0.27
5-MeO-MMT	0.51



seeds are to scale

Ehman (1977) Journal of Chromatography 132: 267-276: On silica gel with Van Urk -Salkowski Reagent: Gramine: Reddish violet (Pansy Violet) when dry. Bluish Violet (Methyl Violet) when wet. **Tryptamine:** Blue (Princes Blue) when dry. Blue (Oriental Blue) when wet. 5-Hydroxytryptamine: Violet blue (Bluebird Blue) when dry. Bluish Green (Capri Blue) when wet. 5-Methoxytryptamine: Blue (Princes Blue) when dry. Bluish Green (Langite Green) when wet. **Bufotenine:** Violet blue (Cornflower Blue) when dry. Bluish Green (Capri Blue) when wet. (Color names from Horticultural Colour Chart by the British Colour Council. Vol. I: 1938 ; Vol. II: 1941.) This is an interesting approach which could prove most useful for

better defining colors of specific chromophores. The digital scanning color densitometers that are in current use in the lithographic industry could provide far more accurate color definition than the color charts used above.

It was noted in Clarke's second edition that use of chromophoretic methods combined with tlc and densitometers had increased the accuracy of quantitative estimates into the range previously attainable via GC and HPLC.



Acacia maidenii with green fruit

Erspamer et al. (1967) Biochemical Pharmacology 16 (7): 1149-1164: For Paper they used Whatman No. 1.

Ran at 18° C for 10-30 hours.

Solvent Systems used in paper chromatography:

A: n-Butanol-Acetic acid-Water (40:10:50)

- B: 1-Pentanol-Pyridine-30% Methylamine-Water (40:40:1:10)
- C: n-Butanol-30% Methylamine (80:30)
- D: 20% Potassium chloride
- For tlc they used Merck Kiesel-gel G, at room temperature.

After drying the plates were sprayed with the NNCD reagent or the PDAB to visualize.

Solvent Systems used in tlc:

- E: n-Butanol-Acetic acid-Water (40:10:50)
- F: n-Butanol-30% Methylamine (80:30)
- G: Chloroform-Methanol saturated with NH₃ (20:1)
- H: Benzene-Ethanol-30% Methylamine (22:7:1)

Solvent System

Rf or color reaction of Alkaloid

<u>Sorvent System</u>			<u>KI UI CUIUI TEACTION UI AIKAIUU</u>				
		Serotonin	5-MeO-				
			tryptamine	5-OH-MMT	Bufotenine	5-MeO-MMT	5-MeO-DMT
	А	0.43-0.49	na	0.46-0.50	0.51-0.56	0.62-0.65	0.67-0.69
	В	0.56-0.59	na	0.55-0.62	0.72-0.74	0.63-0.66	0.82-0.88
	С	0.59-0.66	na	0.77-0.82	0.81-0.88	0.92-0.94	0.92-0.93
	D	0.34-0.38	na	0.37-0.40	0.72-0.73	0.39-0.42	0.46-0.47
	E	0.59-0.63	0.59-0.62	na	0.40	na	0.40
	F	0.75-0.78	0.87-0.90	na	0.91	na	0.97
	G		0.16	na	0.17	na	0.48-0.50
	Н	0.43-0.47	0.67-0.68	na	0.66	na	0.79-0.80
	Reagent:						
	Pauly	Wine red	na	Wine red	Wine red	Light olive yellow	Light olive yellow
	NNCD	Peach red	na	Peach red	Peach red	Orange brown	Orange brown
	Echtrotsalz B	Pk Vt	na	Pk Vt	Pk Vt	Light Brown Yellow	Light Brown Yellow
	Gibbs'	Violet blue	na	Violet blue	Violet blue	No reaction	No reaction
	PDAB	Blue	na	Blue	Blue	Blue	Blue

Pk Vt = Pinkish violet

Trout's Notes FS-X7: Chromatographic Assay Data and Alkaloid Tests

Fish et al. (1955) Journal of the American Chemical Society 77: 5892-5895: Developing systems:

A: Propanol-1N Ammonium hydroxide (5:1).

Used S&S 507 paper, unwashed, for DMT and Whatman 3MM paper, washed, for Bufotenine.

B: Methyl ethyl ketone-t-Butyl alcohol-Diethylamine-Water (40:40:4:20). [Used S&S 507, unwashed.]

C: *n*-Butyl alcohol-Acetic acid (10:4); saturated with water. [Used Whatman 3MM, washed.]

D: t-Butyl alcohol-Water-Formic acid (207:87:6). [Used S&S 507, unwashed.]

Was allowed to develop for 16 to 64 hours depending on paper and solvent system. Used a 0.5% solution of *p*-Dimethylaminobenzaldehyde in 1N Hydrochloric acid to visualize.

Alkaloid	Rf				UV λ_{max}	Fluorescence	maxima (nm)
	<u>A</u>	<u>B</u>	<u>C</u>	D	(Ethanol)	Excitation	Emission
DMT	0.86	0.92	0.67	0.71	274, 283, 291 (R)	283	350
	[0.83]*	[0.95]*	[0.72]*	[0.66]*	275, 283, 291 (I)		
DMT-	0.56	0.70	0.76	0.72	274, 282, 290 (R)	283	349
-N-oxide	[0.59]*	[0.68]*	[0.80]*	[0.74]*	274, 282, 291 (I)		
5-OH-DM1	0.82	0.89	0.62	0.51	279, 301, 314sh (R)	300	340
					279, 301, 315sh (I)		
Bufotenin	e 0.35	0.46	0.72	0.62	278, 301, 314sh (R)	308	330
-N-oxide					278, 302, 314sh (I)		
5 m m m m m m m m m m m m m m m m m m m	F.O	50 0 171	50 - 131	50 -071			

[**MMT**] [0.75]* [0.94]* [0.74]* [0.70]*

*[589R paper in same systems; Fish et al. (1956) Journal of the American Chemical Society 78: 3669-3671.]

R = Reference material

I = Isolated material

Fitzgerald & Sioumis (1965) Australian Journal of Chemistry 18: 433-434:

tle on alumina:		
Developer	Rf of MMT	Rf of DMT
Chloroform	0.4	0.5
Methanol	0.3	0.7
Chloroform-Methanol (1:1)	0.3	0.7

Gander et al. (1976) Phytochemistry 15: 737-738:

Whatman 3mm paper with n-Butanol-Acetic acid-Water (80:3:17) as the developer for preparative chromatography.AlkaloidRfXanthydrolXanthydrol reaction products

N,N-Dimethyltryptamine	0.48	Lavender	λ _{max} 510 nm	λ _{min} 400 nm
5-Methoxy-N,N-dimethyltryptamine	0.42	Blue	590 nm	425-500 nm
2-Methyl-6-methoxy-1,2,3,4-THβC	0.47	Blue-Violet	560 nm	425-500 nm
2-Methyl-1,2,3,4-tetrahydro-β-carbolin	ne 0.51	Lavender	505 nm	427 nm

They point out that at least some suspected occurrences of DMT in *P. arundinacea* were actually 2-Methyl-THβC. Xanthydrol: 0.1 gram in Ethanol-11.7N Hydrochloric acid (19:1)

Genest & Hughes (1968) The Analyst 93 (1109): 485-489:

Using plates of Silica Gel G (Merck) or Alumina G (Merck). Plates activated before use by heating at 110° C for 1 hour. Used Sodium acetate solution, 2,6-Dibromo-*p*-benzoquinone-4-chlorimine and Iodine to visualize. Solvent Systems:

A. Ethyl methyl ketone-Dimethylformamide-Ammonia solution (sp. gr. 0.90) (13:1.9:0.1) on silica gel G.

B. Methanol-Chloroform (1:1) on alumina G.

C. Chloroform-Methanol-Acetic acid (75:20:5) on silica gel G.

Ghosal & Mukherjee (1964) Chemistry and Industry 1800:

Used Isopropanol-Ammonia-Water (9:1:1) on paper.

Alkaloid	Rf
Bufotenine	0.83
DMT	0.91
DMT-N-oxide	0.63

Perkal (1981) Master's thesis:

Using silica gel-kieselguhr (2:1)								
RF	1	2	3					
Psilocybin	0.01	0.15	0.15					
Psilocin	0.57	0.55	0.76					
Baeocystin	-	0.36	0.16					
Norbaeocystin	-	0.58	0.17					
Dimethyltryptamine	0.61	0.51	0.79					
DMT & pailogin are not again, concrehig in their systems								

DMT & psilocin are not easily separable in their systems.



Benzene-Methanol-5% Ammonium hydroxide (10:15:1)
 n-Butanol-glacial Acetic acid-Water (2:1:1)

3 n-Propanol-5% Ammonium hydroxide (5:2)

Ghosal & Mukherjee (1966) Journal of Organic Chemistry 31: 2284-2288:

Used preparative tlc to separate a mixture of Bufotenine, DMT and DMT-N-oxide.

They streaked a sample of the residue onto Whatman No 1 paper and ran in Isopropyl alcohol-Ammonia-Water (9:1:1).

They had determined Rf with a marker strip (finding DMT-N-oxide- 0.63, Bufotenine- 0.82 and DMT- 0.91 respectively) and then cut the appropriate zones from the rest of the paper, eluting each alkaloid with ethanol.

A: Isopropyl alcohol-Ammonia-Water (9:1:1) [Chromatography on Whatman No. 1 paper.]

B: n-Butanol-Acetic acid (10:4), saturated with water. [Chromatography on Whatman No. 1 paper.]

C: Chloroform-Methanol (90:10) [tlc on alumina.]

Visualization was with 0.5% p-Dimethylaminobenzaldehyde as spray reagent.

<u>Alkaloid</u>	<u>Rf</u>			Color with PDAB	Other	
	Α	В	С			
Gramine	0.95	0.73	0.53	No immediate color	Orange with Dragendorff's	
DMT	0.91	0.74	na	Blue		
DMT-N-oxide	0.63	0.81	na	Mauve to Blue		
Bufotenine	0.82	0.63	na	Blue		Ph
5-MeO-MMT	0.86	0.70	na	Blue		
5-MeO-DMT	0.97	0.72	na	Blue		
5-MeO-DMT-N-oxide	0.56	0.89	0.91	Cherry-Red to Blue		R
Synthesized:						
6-Methoxy-2-methyl- I	H ⁴ -β-car	boline	0.97	No color	Intense blue with	
					Hopkin-Cole's glyoxalic reag	gent



Phalaris caroliniana modified from Robbins *et al.* 1951

Ghosal et al. (1969) Journal of Medicinal Chemistry 12: 480-483:

Paper: *n*-Butyl acetate-*n*-Butanol-Acetic acid-Water (85:15:40:22) on Whatman No. 1 tlc: Methanol on silica gel G.

<u>Alkaloid</u>	Rf on paper	<u>Rf on Silica Gel</u>
5-MeO-MMT	0.58	na
DMT	0.74	na
Bufotenine	0.27	na
Bufotenidine	0.18	0.16
Dehydrobufotenine	0.06	0.04



Phalaris minor modified from Robbins *et al.* 1951

Ghosal et al. (1971) Journal of Pharmaceutical Sciences 60 (8): 1209-1212:

Using silica gel G chromatoplates and Methyl alcohol-Chloroform-Acetic acid (75:25:15) as developer.

<u>Alkaloid</u> N,N-DMT DMT-N-oxide	<u>Rf</u> 0.47 0.88	Dragendorff's Orange Dirty orange	<u>Ehrlich's</u> Blue Cherry Red	\underline{Other} α -Nitroso- β -naphtholnitrous acid - Negative α -Nitroso- β -naphtholnitrous acid - Negative
N-Methyl- tetrahydroharman 5-Methoxy-	0.51	Orange	Negative	α -Nitroso- β -naphtholnitrous acid - Negative
tetrahydroharmar	n 0.62	na	na	Fröhde- Navy blue Hopkin-Cole- Purple
Harmine Harmaline	0.73	Orange na	Negative na	α -Nitroso- β -naphtholnitrous acid - Dull violet
Tetrahydroharmine	0.66	Orange	Negative	α-Nitroso-β-naphtholnitrous acid- Dull violet Fröhde- Navy blue Hopkin-Cole- Purple

Ghosal and coworkers reported that indole-3-alkylamines and tetrahydro- β -carbolines showed a light-blue fluorescence under UV on papers, aromatic β -carbolines showed violet and 3,4-dihydro- β -carbolines showed a sea-green fluorescence.

(It must be noted that these colors are not the same on alumina and DMT does not fluoresce. at visible wavelengths.) Harmine shows an indigo-blue fluorescence in acid solutions which becomes yellow-green in alkaline solutions. The transition occurs within the pH interval 7.2-8.9 (from Marion p. 394).

Banerjee & Ghosal found that crudely purified 5-MeO-DMT has a pale yellow fluorescence under UV and 5-MeO-DMT-N-oxide has a dull red color under UV (both were on paper; Appleseed could not observe fluorescence in TLC on silica gel.)

Bufotenine is said to show a weak pink fluorescence under UV; according to Udenfriend.

Gupta et al. (1979) Journal of Natural Products 42 (2): 234-236:

Using pure reference materials Ran on silica gel 60F-254 (E.M. Reagents)

Used PDAB reagent to visualize.

Solvent Systems:

A: 1-Propanol-5% Ammonium hydroxide solution (10:15:2) [5% or 58%?]

B: Benzene-Methanol-5% Ammonium hydroxide solution (10:15:2) [5% or 58%?]

C: Ethyl acetate-Methanol-58% Ammonium hydroxide solution (80:15:5)

D: 1-Butanol-glacial Acetic acid-Water (2:1:1)

Alkaloid	Rf A	Rf B	Rf C	Rf D
Tryptamine	0.44	0.27	0.32	0.57
N-Methyltryptamine	0.40	0.20	0.29	0.52
N,N-Dimethyltryptamine	0.53	0.46	0.58	0.47
Serotonin	0.38	0.18	0.18	0.55
Bufotenine	0.47	0.36	0.40	0.46
5-Methoxytryptamine	0.44	0.25	0.30	0.57
5-Methoxy-N,N-dimethyltryptamine	0.52	0.44	0.55	0.47



Acacia obtusifolia seeds

Holmstedt (1965) Archives Internationales de Pharmacodynamie et de Thérapie 156 (2): 285-305:

Paper; Used *n*-Butanol-Acetic acid-Water (120:30:50). Developed for 16 hours, Using Silica Gel for tlc (run in cold room for 3-1/2 to 4 hours);

tlc A: Used *n*-Butanol-glacial Acetic acid-Water (120:30:50).

tlc B: *n*-Propanol-Ammonia (5:1).

tlc C: *t*-Butanol-Water-Formic acid (207:87:6).

tie C. <i>i</i> Butanoi Water i offine dela (207.07.0).									
Alkaloid	Rf on paper	Rf with A	Rf with B	Rf with C					
5-Meo-DMT*	0.72	0.71	0.90	0.73					
DMT (as hydrogen oxalate)	0.78	0.80	0.91	0.69					
Bufotenine	0.60	Not run							
Bufotenine-N-oxide	0.65	Not run							
*(Identity is inferred based o	n his further an	alytical work)							

Mack & Slaytor (1979) Phytochemistry 18:1921-1925:

They used the to separate the tryptamines according to methyl groups with neutral alumina F_{254} (Type E) (Merck 5550/0025) in Benzene-Ether (1:4) saturated with 18M NH₄OH.

They found that it did not separate the unsubstituted alkaloids from their 5-methoxy derivatives.

Tryptamine	Rf 0.1
MMT	Rf 0.5
DMT	Rf 0.9

Mulvena & Slaytor (1983) Phytochemistry 22 (1): 47-48:

All indoles in *Phalaris tuberosa* were found to be adequately separated by two dimensional tlc.

They used an initial extraction of homogenizing in 0.01M HCl and extracting twice with chloroform.

After adjustment of acid solution to pH 10 with NaOH they extracted three times with ethyl acetate.

The residue from the ethyl acetate was taken up in methanol and applied to Merck silica gel plates (20X20).

These were first developed with Chloroform-Methanol-Ammonium hydroxide (80:15:1) and then with *n*-Butanol-Ethanol-Ammonium hydroxide (20:2:1)

They identified the alkaloids by co-tlc and used the color reactions with the van Urk-Salkowski reagent coupled with a scanning densitometer to estimate the concentrations. [cited Ehmann 1977 Journal of Chromatography 132: 267]

Pachter et al. (1959) Journal of Organic Chemistry 24: 1285-1287:

Using Whatman 3mm paper in t-Amyl alcohol-Formic Acid-Water (10:1:10) found DMT had Rf 0.78

Rovelli and Vaughan (1967) Australian Journal of Chemistry 20: 1299-1300:

Using tlc on alumina with Benzene-Methanol (90:10) they reported that DMT had an Rf of 0.45

Schnoll et al. (1972) Journal of Psychedelic Drugs 5 (1): 75-78:

Using silica gel G with fluorescence indicator

Solvent Systems:

1 Concentrated Ammonium hydroxide-p-Dioxane-Acetone (2.5:45:5.5) (v/v)

2 Concentrated Ammonium hydroxide-Chloroform-p-Dioxane-Acetone (2.5:45:4:47.5) (v/v)

Rf values determined in solvent system 1, allowed to front at 10 cm for 5 minutes.

They first examined the plate under UV, recording fluorescence and quenching at 254 nm (shortwave) and

fluorescence at 366 nm (longwave)

It was then placed in a tank with a HNO₃ atmosphere for 3 minutes (noting any color changes)

It was then sprayed with Ninhydrin and placed in an oven at 100° C for 5 minutes.

Ninhydrin spray reagent (500 mg. Ninhydrin in 100 ml of acetone)

Trout's Notes on Tryptamines: Chromatographic Assay Data and Alkaloid Tests

They used Iodoplatinate reagent as the final spray. Iodoplatinate spray reagent (250 mg. Platinic chloride and 5 gm Potassium iodide in 100 ml. distilled Water Sample was also run using solvent system 2

Substance	$Rf \pm SD$	254nm	366n m	HNO,	Ninhydrin	Iodo-	$Rf \pm SD$	
	Solvent 1			atm.		platinate	Solvent 2	
DMT	$0.21 \pm .04$	quenching	blue-green	yellow	b	purple	0.20 ± 05	
1 11 1 .		1.		EX. () 1				

b slight purple-grey depending on amount present. [Most authors say no reaction.]

Sensitivity between 1 and 10µg.

Dr. *A.T. Shulgin* (1993) recommended Ethyl acetate-Methanol-Ammonia (170:20:2) as a developing solvent for tlc (alumina on glass plates). He suggested adding more methanol if 5-MeO-DMT is present; to separate from DMT.

Ivor Smith (1969) Chromatographic and Electrophoretic Techniques: (Many more indoles are listed by Smith, but he does not include 5-MeO-DMT)

Using Whatman No. 1 chromatography paper on a Universal apparatus with ascending frame using a 8 to 9 inch run from the origin and performed at room temperature.

A: Isopropanol-Ammonia (880)-Water (200:10:20) [8 hours for 8 inch rise. 1/2 hour to remove with fan.]

B: n-Butanol-glacial Acetic acid-Water (120:30:50) [7 hours for 8 inch rise. 15 to 30 minutes for removal with fan.]

C: n-Butanol-Pyridine-Water (60:60:60) [8 hours for and 8 inch rise. 15 to 30 minutes for removal with fan.

D: 20% aqueous Potassium chloride [KCl] (weight/volume) [One hour to dry]

E: 8% aqueous Sodium chloride (weight/volume)-glacial Acetic acid (200:2) [One hour to dry.]

The last two of these are corrosive to metal parts and cannot be dried with a fan as the paper becomes fragile but provide very compact excellent spots and very constant Rf values.

Alkaloid			RF			Ehrlich's	Other				
	Α	В	С	D	Е						
DET (HCl)	0.98	0.85	0.83	0.62	0.63	Reddish purple					
Bufotenine	0.90	0.60	0.79	0.43	0.47	Reddish purple then blue-purple	Sulphanilic acid: Red-brown				
DMT	0.92	0.74	0.82	0.60	0.62	Reddish purple					
MMT	0.88	0.78	0.82	0.57	0.57	Reddish purple	Ninhydrin-Pyridine: Weak reaction				
DMT & MN	DMT & MMT were as acetate										

MMT can also be differentiated from DMT as MMT reacts with Ninhydrin-Acetic acid. DMT does not react.

Williams et al. (1971) Crop Science 11: 213-217:

Using Brinkman pre-coated silica gel G plates in Isopropanol-Ethyl acetate-concentrated Ammonium hydroxide (120:30:6) with Van Urk's reagent to visualize. [citing Waldi 1965: 490]

They were unable to get complete separation but used this solvent system as it separated the alkaloids into "groups".

Reference alkaloid	<u>Rf</u>
Gramine	0.22
Tryptamine	0.20
MMT	0.16
DMT	0.34
5-Methoxytryptamine	0.19
5-MeO-MMT	0.13
5-MeO-DMT	0.28



Woods & Clark (1971) Crop Science 11:121-122:

Solvent Systems: (15 cm run on each)

A: Methanol-strong Ammonia solution (29%) (7:1) on Merck silica gel G plates.

B: Methanol-concentrated Hydrochloric acid (9:1) on Merck silica gel G plates.

C: Butanol-Formic acid-Water (16:1:3) on Mackery Nagel MN300 cellulose plates

Using reference material.

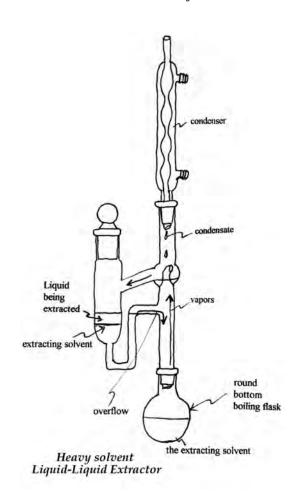
Alkaloid		Rf		Ehrlich's	Xanthydrol
	Α	В	С		•
Gramine	0.38	0.65	0.75	Slowly turning Pink	Pink
MMT	0.25	0.75	0.75	Blue-Gray	Purple
DMT	0.50	0.60	0.75	Blue-Gray	Purple
Bufotenine	0.45	0.60	0.45	Blue-Purple	Blue
5-MeO-MMT	0.25	0.75	0.65	Royal blue	Blue
5-MeO-DMT	0.50	0.60	0.65	Royal blue	Blue
6-Methoxy-2-methyl-1,2,3,4-					
tetrahydro-β-carboline	0.55	0.65	0.65	Blue-Gray turning brown (Poor reaction)	Purple
2,9-Dimethyl-6-methoxy-					
1,2,3,4- tetrahydro-β-carboline	0.55	0.55	0.65	Blue-Gray turning brown (Poor reaction)	Purple

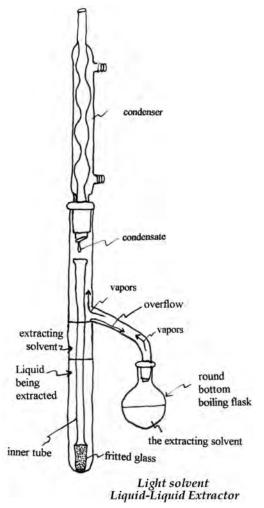


Acacia confusa



Cultivated as an ornamental in Oakland, California





Abstracted Isolations of Some Indoles

Maximum load for preparative tlc on silica gel is around 5 mg per cm of width on a 1.0 mm thick layer. Poole & Poole 1981: 727

[Maximum load on a single sheet of blotting paper is said to be around 100 mg.]

Smith 1977b

"Isolation of tryptamines from plant material may involve extraction of alkaline macerates $[Na_2CO_3 \text{ or } NH_4OH]$ with CHCl₃ or Et₂O. Purification may also be achieved with cation exchange resins. TLC or PC are used for the separation of complex mixtures with the Salkowski.

Ehrlich (p-dimethylamino-benzaldehyde or -cinnamaldehyde), Pauly or Dragendorff chromogenic reagents. The nitronaphthol reagent may be used for 5-hydroxyindoles. The tryptamines do not require prior derivatization for separation by GLC."

Arthur et al. (1967) Australian Journal of Chemistry 20: 811-813:

Using stems of Acacia confusa.

They first extracted the material with petroleum ether and then with ethanol.

The ethanol was concentrated under reduced pressure to a brown syrup. This was then brought to pH 8 [should be pH 8.6 for full recovery of DMT] with a sodium carbonate solution.

The resulting basic solution was then extracted with chloroform.

The chloroform was then extracted with 2N hydrochloric acid.

The aqueous acidic solution was basified with Sodium hydroxide and exhaustively extracted with chloroform.

The residue from evaporation of the chloroform was purified by re-extracting into an acid solution from chloroform.

An acetone solution of the alkaloids containing hydrochloric acid deposited N-methyltryptamine as the hydrochloride.



Compiled, edited & annotated by Keeper Trout

After recovery of the remaining alkaloids they were then reacted with acetic anhydride which does not affect DMT but converts MMT into an acetyl derivative which is non-basic.

The remaining MMT was separated from DMT by partitioning between chloroform and dilute hydrochloric acid. DMT went into the acid and MMT stayed in the chloroform as the acetyl derivative.

The basic fraction which was eventually recovered was chromatographed over alumina to yield crystalline DMT.

[It should be obvious that the return of DMT via their procedure was incomplete, however the separation they used for DMT and MMT may be useful in conjunction with other techniques.]

Banerjee & Ghosal (1969) Australian Journal of Chemistry 22: 275-277:

Using *Desmodium gangeticum* [green plant.]

[Air dried had 0.01 to 0.03% of total alkaloid. Fresh has three times as much alkaloid as dry. Fresh also has more uncharacterized alkaloids but dry has higher proportion of 5-MeO-DMT.]

Fresh material (1 kg. wet wt), macerated in a Waring blender with chloroform (3 liters) and 15N ammonia (50 ml). Allowed to soak at room temperature, with occasional shaking, for a week.

After filtering the two phases were separated.

Chloroform layer then extracted with 2N aqueous acetic acid (100 ml) and both layers saved.

[DMT and DMT-N-oxide acetates are soluble in both acid and chloroform.]

The aqueous layer was cooled in ice and then made basic to pH 9 with ammonia and extracted with chloroform.

The chloroform was then evaporated.

The basic gum (2.5 gm) was chromatographed on a column of Brockman neutral alumina.

5-MeO-DMT was eluted with light petroleum-benzene (1:1) [as 570 mg of colorless plates.]

DMT was eluted with chloroform (as thick colorless oil, amount not given.)

DMT-N-oxide was eluted with chloroform-methanol (9:1) [as 210 mg as hydroscopic solid.]

5-MeO-DMT-N-oxide was eluted with methanol. (as 180 mg as pale brown gum.)

Chromatography using Brockman alumina was then done with the original chloroform extract containing the chloroform soluble acetates.

 N_b -Methyltetrahydroharman was eluted by chloroform. [30 mg almost crystalline material.]

Chloroform-methanol eluted additional amounts of DMT (410 mg) and DMT-N-oxide [120 mg].

6-Methoxy-2-methyl- β -carbolinium cation was recovered from the aqueous mother liquor of the aforementioned separation allowed recovery of 40 mg via the reineckate.

[Note: in seed grown plants, alkaloids did not begin to show up in measurable quantities until after several years of growth. Unpublished tlc by J. Appleseed.] Cei *et al.* 1968 commented that 80% acetone proved to be the best solvent they used for isolating indolealkylamines but it should be noted that the residues recovered from acetone extracts can also potentially contain MANY other undesirable compounds if further cleanup is not performed and acetone itself can be harmful to some alkaloids due to the potential for imine formation. However, unlike the first point, the last point does NOT appear to be an issue for the stable tryptamines. However, limited tests of this solvent for the extraction and recovery of DMT have not appeared promising.

Corothie & Nakano (1969) Planta Medica 17 (2): 184-188:

Using dried and milled bark from Virola sebifera.

They extracted exhaustively with methanol at room temperature until Meyer's reagent no longer gave positive tests on the methanol extracts.

They concentrated their combined extracts in vacuo to a brown tar.

The residue was extracted several times with 2% hydrochloric acid and the solutions filtered.

The residue was then partitioned between chloroform and 2% hydrochloric acid.

The aqueous solutions were combined and basified with 25% ammonia.

Aqueous solution then extracted with chloroform.

After washing the chloroform solution with water, they dried it with a desiccant and evaporated in vacuo to a sticky basic fraction. (tlc showed only one alkaloid to be present.)

The basic fraction (33 grams) was dissolved in benzene and chromatographed on Merck alumina, activity II-III (660 grams).

Chloroform eluted the DMT which was distilled with a bath temperature of 80-135° at 0.03 mm.

Their end product was a slightly yellowish oil.

Culvenor et al. (1964) Australian Journal of Chemistry 17: 1301-1304:

Using P. tuberosa grass from Glenroy SA

260 pounds dry weight equivalency of partly dried grass gave 19 grams of crude alkaloid

Culvenor extracted with methanol, removed the solvent under pressure and extracted the residue with dilute sulfuric. To convert any N-oxide which were present one portion was made 2N with sulfuric acid and stirred for 4 hours with an excess of zinc dust. Both portions were then basified with ammonia and extracted with chloroform. The resulting residues were compared. [They found no N-oxides to be present]

The crude alkaloid was subjected to a 72 tube counter current distribution between chloroform and 0.2N hydrochloric acid.

1-0.2 grams was a nonbasic material

2-170 mg contained Rf 0.90

3-4.1 grams contained Rf 0.56, 0.48, 0.40

4- From tubes 67-72 [yielding a residue of 14.5 grams which contained alkaloids with Rf 0.50 and 0.41, see assay section for solvent system.] were then subjected to partition chromatography on a column of Pyrex glass powder moistened with potassium buffer at pH 8.1 (320 ml for 1880 grams of glass).

Elution with 7.6 liters of 60-80° light petroleum eluted impure DMT (Rf 0.50) which was then dissolved in ether and recovered via its picrate.

[650 mg was recovered from a 4 gram sample of the 14.5 gram residue. [Total of 2.356 grams recoverable via this route from the 14.5 gram residue.] (2.7 grams was also eluted as a mixture of DMT and 5-MeO-DMT.)

Preparative chromatography on paper was used to separate 5-MeO-DMT from DMT in the Rf 0.41 material.

They found alkaloid content was higher in material which was immediately placed in alcohol when harvesting.

They also noted that while air dry samples of *P. tuberosa* contain 0.05% to 0.08% total alkaloids, fresh grass may contain twice as much alkaloid than would be present if it was dried. In addition there is a higher proportion of bufotenine and a lower proportion of the uncharacterized indoles of high Rf present.

[Ghosal noted that in *D. gangeticum* leaf, the alkaloid content was three times as high when fresh and green than if allowed to dry but that dry material had a higher proportion of 5-MeO-DMT]

Dutta & Ghosal (1967) Chemistry and Industry 2046-2047:

Using Arundo donax.

200 grams of dried plant defatted with benzene and extracted for 8 hr in a Soxhlet.

After removal of the alcohol under reduced pressure it was treated with 3% aqueous acetic acid and filtered.

It was cooled and then neutralized with ammonia to pH 9.

The resulting aqueous solution was then extracted with chloroform.

(Much polymeric material was present that was insoluble in the chloroform.)

The residue which resulted from removal of the chloroform was further separated using a column of Brockman alumina.

CHCl,-benzene (50: 50) eluted 520 mg of Gramine.

Chloroform eluted 20 mg of DMT

Chloroform-methanol (99:1) eluted 110 mg of Bufotenine

5-MeO-N-methyltryptamine was separated from bufotenine by fractional crystallization of picrates from EtOH as bufotenine picrate was only sparingly soluble in EtOH.

[While they did not specifically state it in this piece, the procedure they used with the rhizomes in 1969 used a sequence of petroleum ether / petroleum ether-benzene (90:10, 80:20, 50:50) / benzene / benzene-chloroform (95:5, 90:10, 80:20, 50:50, 25:75) / chloroformmethanol (99:1, 98:2, 95:5) and methanol.]

Fish et al. (1955) Journal of the American Chemical Society 77: 5892-5895:

Using Piptadenia macrocarpa obtained in Florida.

Dried material (450 grams) was ground and stirred with a solvent mixture of chloroform (1950 ml), tetrahydrofuran (1050 ml) and ammonium hydroxide (225 ml) for 1 hour at 40° C.

It was then filtered and the organic solvent mixture washed with an 8N ammonium hydroxide solution.

The volume was reduced to around 50 ml under reduced pressure then diluted with chloroform.

The resulting solution was extracted several times with 2N hydrochloric acid.

The combined aqueous extracts were then made basic with sodium carbonate. This solution was exhaustively extracted with chloroform (until the aqueous solution no longer reacted with Ehrlichs)

After drying with magnesium sulfate, the chloroform evaporated to yield 1.4 gm of a black gum.

This was purified by applying small samples dissolved in ethanol-chloroform to washed Whatman 3mm paper and using preparative ascending paper chromatography.

A propanol-ammonia system was used for the preparative chromatography. [When performing such an operation it will be found useful to apply the sample as a line rather than a spot.]

A strip was tested with Ehrlich's to determine the Rf of the zones and the paper was cut to separate the alkaloids which were then eluted with ethanol.

Fitzgerald & Sioumis (1965) Australian Journal of Chemistry 18: 433-434:

Using Acacia maidenii bark.

Bark was milled and percolated with warm methanol (40°) using method described by Fitzgerald 1963.

The methanol was removed in a climbing film evaporator and diluted with water.

The aqueous solution was basified with ammonia and extracted with chloroform.

The chloroform was then extracted with dilute Sulfuric acid. The acidic fraction was basified and again extracted into chloroform.

Evaporation of chloroform gave the crude bases.

Separation of MMT and DMT:

Short path distillation of crude base at 160°/ 4 mm and crystallization of the distillate

from light petroleum gave the higher melting compound (MMT) in a somewhat impure state.

Separation was best effected by chromatography over alumina.

Chloroform eluted DMT and chloroform-methanol (1:1) eluted MMT.

> Image provided by James Arthur



Canterbury Psalter 1100 CE

Frahn & Illman (1973) Journal of Chromatography 87: 187-191:

Using Phalaris aquatica (as P. tuberosa)

They used neutral polystyrene resin to separate the alkaloids. [as Porapak Q (80-100 mesh)] Effluents were followed by use of a UV spectrophotometer. They used an example of mixed alkaloid to demonstrate:

DMT- metho cation came off first and showed a λ_{max} of 218 and 278 nm with a shoulder at 288 nm.

DMT came off separately with another elutant and showed a similar spectrum.

They suspended the Porapak in acetone for half an hour, washed it thoroughly with freshly boiled water and allowed it to settle into a 1 cm ID glass column to a depth of 3 cm. [This was used in their example where they separated a total of 3.3 mg of alkaloid combined with 38 mg of mixed salts, for demonstration purposes. It can be scaled up for larger amounts.]

After resin was equilibrated with 0.1M ammonium hydroxide the alkaloid mixture was dissolved in 6 ml of sodium hydroxide and applied to the top of the column.

Eluting with 50 ml of 0.1M ammonium hydroxide desalted the mixture

[This ready ability to effectively desalt alkaloid solutions may prove most applicable to such known high mineral salt containing plants as those *Delospermas* species known to contain DMT and/or 5-MeO-DMT (identified by co-tlc) or toxic salt accumulators such as the *Astragalus* species, some of which are indicated (also by tlc) to contain 5-MeO-DMT or even to potentially recapture and recycle sacramental materials such as *Psilocybe* mushrooms or mescaline within entheogenic communities. While part is metabolized, a portion of each of these two passes unchanged with the urine.]

DMT metho cation was effectively eluted with methanol-0.2M ammonium hydroxide (1:1) using a total of 90 ml. They collected 5 ml fractions and set the flow rate at 0.4 ml/min..

[They noted that DMT could be eluted with this also if a much greater quantity was used but that the methocation cleared the column before elution of DMT began. They changed solvents to allow a sharper peak of elution.)

DMT eluted with methanol-water (3:1) using 50 ml total.

The column was cleared with methanol before being re-equilibrated with ammonium hydroxide.

[They found it effectively separated 5-MeO-DMT, 2MTC and 6M2MTC from their metho cations under experimental

conditions.

Gramine metho cation was decomposed under alkaline conditions.] [Niederwieser &

Giliberti 1971 were cited for procedure.]

cultivated shrooms

Photo lower right by JWAllen



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Frahn & O'Keefe (1971) Australian Journal of Chemistry 24: 2189-2192:

Using Phalaris tuberosa cv. Seedmaster:

12 kg of fresh grass (2.4 kg dry weight equivalency) was macerated with 40 liter of cold 0.1N HCl. After filtering it was neutralized with 17N NaOH and brought to pH 10 with so-dium carbonate.

Solution was then saturated with sodium chloride and exhaustively extracted with chloroform.

The chloroform was dried over magnesium sulfate and evaporated to 9.8 grams of tar.

The tar was thoroughly suspended in 35 ml of 0.1N HCl and filtered.

The filtrate was made alkaline with ammonia and extracted with chloroform to give 2.4 grams of a mixture of crude bases.

A 0.6 gram sample of the 2.4 grams of crude bases was separated using a 107x1.5 cm column of Bio-Rex 70 cation exchange resin (Bio-Rad Laboratories, California) and equilibrated in the H+ form with formic acid-ammonium formate buffer (pH 3; 0.2M with respect to the formate)

Elution was carried out with the same buffer until they had collected 220 tubes each with 15 ml.

They then eluted with the same buffer but at pH 2 and collected 50 more tubes.

They followed fractionation with a spectrofluorometer using; range of λ excitation 285-310 nm, range of λ emission 338-360 nm.

The tubes were combined as indicated below and made alkaline with NaOH, extracted with chloroform.

The residue from the chloroform was identified using tlc and paper electrophoresis.

Tubes 100-108 contained a total of 20 mg of gramine

Tubes 110-180 contained a total of 212 mg of DMT [Total 848 mg from 12 kg of fresh grass/ equivalent to 2.4 kg dry material]

Tubes 200-220 contained 32 mg that was a mixture of 5-MeO-DMT and 2-methyl-1,2,3,4-tetrahydro- β -carboline (N_b-methyltetrahydro- β -carboline) and 2-methyl-6-methoxy-tetrahydro- β -carboline

Tubes 240-270 contained a total of 15 mg that was a mixture of 2-methyl-6-methoxy-tetrahydro- β -carboline and an unidentified base.

A small amount additional 2-methyl-6-methoxy-tetrahydro- β -carboline was recovered by clearing the column with 0.1N HCl.

Ghosal & Banerjee (1969) Australian Journal of Chemistry 22: 2029-2031:

Using Desmodium gangeticum roots.

Dried and milled material (1.6 kg) was defatted with petroleum (60-80°). The petroleum extract was then concentrated to 200 ml and extracted with 2N aqueous citric acid (200 ml).

The acidic solution yielded a 400 mg of a brown gum.

This gum was triturated (i.e. ground with the solvent in a mortar and pestle) with light petroleum (40-60°) which extracted 280 mg of phenethylamine as a light brown oil.

The portion that was insoluble in the light petroleum turned out to be 120 mg of DMT-N-oxide.

[DMT-N-oxide is not soluble in petroleum when pure but is soluble in petroleum containing fats.]

The remaining plant material was dried and then extracted with hot ethanol. The residue after evaporation of the alcohol was dissolved in 2N acetic acid, filtered, and then extracted at pH 4 with chloroform [acetic acid being used to convert alkaloids to acetates].

[Only phenethylamines and hypaphorine were recovered from their higher pH extracts.]

Removal of the chloroform gave a 900 mg of a brown solid.

This was dissolved in 10 ml of ethanol and 500 mg of N-methyltyramine separated when it was cooled.

The mother liquor was concentrated and the residue chromatographed over a column of alumina

Chloroform eluted 380 mg of DMT as a thick brown oil.

Chloroform-methanol eluted 20 mg of DMT-N-oxide. (Procedural details not given as such in this paper were added from Ghosal *et al.* 1972e)

[When not attempting to recover DMT-N-oxide from the defatting solution, and recovering from the ethanolic extract via the reineckate, Ghosal and coworkers, (1970b), did not report the recovery of DMT but recovered 70 mg of DMT-N-oxide (from ~2 kg of dried material).]

Ghosal & Mukherjee (1964) Chemistry and Industry 1800:

Using whole dried plant of Desmodium pulchellum

They extracted with alcohol containing acetic acid and separated using column chromatography over Brockman alumina. (Found 0.3% total alkaloids.)

Elution with benzene gave 5-methoxy-N,N-dimethyl-tryptamine (major base - 0.2-0.25%),

Elution with ether-methanol (1:1) gave bufotenine, DMT and DMT-N-oxide as minor components. (Total yield of 0.0018% as a brown gum.)

Subsequent elution with methanol yielded two other bases.

Alkaloid profile and content are highly variable based on plant part and age.

See "*Trout's Notes on the Genus Desmodium*" for a breakdown on this potentially valuable plant.

See below for more detail on the above separation.

Ghosal & Mukherjee (1966) Journal of Organic Chemistry 31: 2284-2288:

Same as Ghosal & Mukherjee 1964.

Dried and finely ground whole plant (4 kg.) was defatted with Benzene by refluxing 8 hours in a Soxhlet.

They then extracted by percolation at room temperature for 4 weeks using alcohol (95% ethanol) containing 2% acetic acid.

The alcohol was reduced to a viscous brown slurry under vacuum and the resulting slurry (170 gm) was poured, with stirring, into 2% aqueous acetic acid (200 ml) and allowed to stand overnight.

After being filtered it was shaken with three 500 ml portions of chloroform (removing 1.7 grams of material.) [It should be noted that DMT-N-oxide and DMT would be components of said chloroform soluble acetates.]

The pH was then raised to 9 with ammonia [full extraction of 5-MeO-DMT needs 9.3 or above] and it was again extracted with chloroform.

After being washed with water and dried over calcium chloride the solvent was removed under reduced pressure.

The residue (18 grams) was dissolved in 10 ml of methanol and introduced into a 35 X 4 cm column of Brockman alumina.

Using 100 ml portions of each solvent they eluted with: petroleum ether (40-60°)

petroleum ether-benzene (90:10) (80:20) & (50:50)

benzene

benzene-ether

and methanol

They collected 40 ml fractions.

5-MeO-DMT was obtained as colorless plates via recrystallization, from ether-light petroleum, of the 8.36 grams of residue left by the removal of solvent from fractions 2 through 11.

5-MeO-MMT was obtained by taking a sample of the residue from fractions 27-33, dissolving it in ether and passing hydrogen chloride gas through it. The crude hydrochloride was recrystallized from methanol as needles. [It was

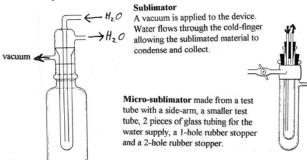
also recoverable via the picrate when said residue was dissolved in acetone.]

Bufotenine, DMT and DMT-N-oxide had also cochromatographed in fractions 27-33. They streaked a sample of the residue onto Whatman No 1 paper and used preparative tlc in isopropyl alcohol-ammonia-water (9:1:1) to separate. They determined Rf (0.82, 0.91 and 0.63 respectively) with a marker strip and cut the zones from the paper, eluting each alkaloid with ethanol.

Gramine and 5-methoxy-DMT-N-oxide was recovered from fractions 37-40.

The residue (322 mg) from fractions 37-40 was dissolved in 2 ml of methanol and chromatographed on cellulose powder, using ~30 grams of Whatman ashless standard grade cellulose powder. [They cite Bartlett *et al.* (1963) *J. Org. Chem.* 23: 1445 for the procedure]. Flow rate was adjusted to 2-3 ml per minute and they again collected 40 ml fractions. Ethyl acetate eluted gramine and acetone-water (90:10) eluted 142 mg of a brown oil.

The water soluble portion of this was exhaustively extracted with chloroform to yield 17 mg of a pale violet oil when the chloroform was removed. It was primarily 5-MeO-DMT-Noxide with some gramine as a contaminant. It was further purified via its picrate.



Ghosal et al. (1969) Journal of Medicinal Chemistry 12: 480-483:

Using Arundo donax rhizome.

Dried and milled material (700 grams) was extracted by percolation with 95% ethanol at room temperature for one week.

After reduction of the ethanol to a viscous consistency (112 grams of material) under reduced pressure, it was poured into 2% acetic acid with stirring and allowed to stand overnight.

After filtering it was extracted with three 500 ml portions of chloroform.

The chloroform was evaporated and the residue taken up into benzene and extracted with 100 ml of 0.1M citric acid.

The acidic aqueous solution was basified with ammonia and extracted with chloroform. After removal of the chloroform 40 mg of thick brown oil remained. Crude DMT was sole component.

They used a more thorough workup to recover the other alkaloids in the original solution but did not identify any additional DMT.

Ghosal et al. (1971) Journal of Pharmaceutical Sciences 60 (8): 1209-1212:

Using Banisteriopsis argentea leaves.

Dried and milled material was defatted with petroleum ether (60-80°)

It was then Soxhlet extracted with ethanol for 16 hours and filtered.

The viscous brown residue which remained after solvent was removed under reduced pressure was mixed into 2% acetic acid with stirring. (Using 200 ml for 78 grams of residue)

This was allowed to stand at room temperature overnight and then filtered to remove impurities.

This acidic solution was extracted with three 50-ml portions of chloroform to remove the weakly basic chloroform soluble acetates.

After removal of the solvent, the residue was dissolved in benzene and chromatographed over a column of alumina.

Elutants were petroleum ether, benzene, chloroform, methyl alcohol and stepwise increments between them.

They collected 40 ml fractions.

DMT was eluted (as a brown oil) with chloroform-methyl alcohol (99:1)

DMT-N-oxide was eluted with methyl alcohol. [Zinc dust and acetic acid reduced it to DMT.]

[N-Methyltetrahydroharman had eluted in the first few fractions of chloroform while all other contained alkaloids used acid base partitioning of the original acidic solution, precipitation of their Reineckates and chromatography over Florisil etc.. for purification.]

Psilocybe semilanceata Center photo by JW Allen

sublimator & microsublimator (left)



Hochstein & Paradies (1957) Journal of the American Chemical Society 79: 5735-5736:

Used what probably was either *Diplopterys cabrerana* or *Psychotria viridis* but which had been furnished to them as an aqueous extract and misidentified as *Prestonia amazonica*. They used sodium hydroxide to bring the pH to 10.5 and extracted three times with chloroform. After washing with water

the chloroform was evaporated *in vacuo* to a viscous residue. This was then vacuum distilled at 170° at 0.01 mm to yield DMT as a colorless oil that crystallized spontaneously on

It was further purified by a second vacuum distillation.

standing.

Holmstedt (1965) Archives Internationales de Pharmacodynamie et de Thérapie 156 (2): 285-305:

Using snuff samples, Holmstedt extracted similar to Fish *et al.* 1955 but used smaller quantities of solvent proportional to the dried material.

He further purified by use of preparative paper chromatography.

Using Whatman 3 mm paper 0.05 to 0.1 ml of the alkaloid fraction was applied along the origin of the chromatogram with a width of 20 to 25 mm. Three spots like this were applied with a distance of 60 to 70 mm between them.

Holmstedt used 20% KCl in water to develop the paper using descending chromatography.

After it was dried, the edges were cut off and developed with Ehrlich's reagent. They were then held on each side of the rest of the sheet to determine the positions of the alkaloids.

The alkaloids were then separated by cutting the paper and eluted with 30 ml of absolute alcohol

Holmstedt et al. (1980) Botanical Museum Leaflets Harvard University. 28 (3): 215-234:

Dried and powdered plant material (primarily from *Virola* spp.) was extracted with methanol which was first filtered and then evaporated.

Equal volumes of chloroform and 0.1N hydrochloric acid were added to the residue and after shaking, the organic layer was discarded. After washing the aqueous later with chloroform the aqueous layer was made alkaline (to pH 9.0; this should be higher if extracting 5-MeO-DMT) with sodium carbonate (Na₂CO₂).

The alkaline aqueous solution then was extracted with chloroform. The free bases were recovered by evaporating the chloroform. The residue was dissolved in methanol-chloroform (1:1) for spotting for tlc.

tlc was used to separate the components. They used silica gel G plates (Merck No. 5748) with methanol-ammonia (99:1) as the developing solvent. Dragendorff's was used to locate the alkaloids and Ehrlich's to distinguish the tryptamines.

> Justicia pectoralis var. stenophylla Photo by Des Tramacchi

Iacobucci & Rúveda (1964) Phytochemistry 3: 465-467:

Using Argentinian *Piptadenia macrocarpa* (i.e. *Anadenanthera colubrina* var. *cebil*)

They first ground the plant material, covered it with 1N hydrochloric acid and stirred for 12 hours at room temperature. This was filtered and repeated three more times.

The combined extracts were adjusted to pH 9 with solid sodium carbonate and then exhaustively extracted with ethyl acetate.

After drying with a desiccant, the ethyl acetate was evaporated to a residue.

They dissolved the residue that they had obtained from an extract of the seeds and passed it through a column of activity grade III neutral alumina. After concentration and being allowed to stand at 0° C overnight, Bufotenine crystallized.

The mother liquor was brought to a residue, dissolved in chloroform and then chromatographed on a column of activity grade III neutral alumina.

Elution was first done with chloroform to yield DMT and then with chloroform-ethanol (95:5). The last of these eluted bufotenine, bufotenine-N-oxide and a third 5-oxytryptamine which was not evaluated further (as determined by use of DCC).

Similarly the residue resulting from a seed pod extract was dissolved in chloroform and chromatographed on a column of activity grade III neutral alumina.

Elution was first done with chloroform to yield DMT and then with chloroform-ethanol (99:1) and with chloroform-ethanol (95:5). The last of these eluted bufotenine.

Macrae & Towers (1984) Journal of Ethnopharmacology 12 (1): 75-92.

In their study of *Virola* bark, (5-MeO-DMT was the sole alkaloid present) they extracted the alkaloidal fraction by exhaustively extracting the milled bark at 20° C with methanol. When the alcohol was removed the residue was suspended in water and acidified to pH 3 with hydrochloric acid and extracted with diethyl ether. The aqueous solution was then basified to pH 12 with lye (sodium hydroxide) and extracted with dichloromethane (methylene chloride). Evaporation of the solvent yielded the alkaloid fraction.



Niederwieser & Giliberti (1971) Journal of Chromatography 61: 95-99 reported excellent results using the neutral polystyrene resin Porapak Q both to desalt and to fractionate a variety of indoles.

Elution was with distilled water and aqueous acids. Alkaline solutions eluted hydroxyindolic acids.

0.1 N HCl most strongly absorbed acidic compounds.

Bases were most strongly absorbed with 0.1N NaOH.

Using Porapak Q, 150-200 mesh obtained from Waters Associates in Framingham, Mass.

Resin was allowed to swell in acetone and washed excessively with water prior to use. It must be kept wet.

Pachter et al. (1959) Journal of Organic Chemistry 24: 1285-1287:

Starting with a 900 gram sample of dried bark of *Dictyoloma incanescens* from Brazil.

They extracted for 8 hours with 3 liters of boiling petroleum ether (bp $30-60^{\circ}$) and filtered.

The bark was then heated under reflux while stirring for 3 hours with 2 liters of ethanol containing 10% aqueous ammonia.

This was then repeated twice with fresh solvent but for extraction periods of 8 hours.

The ethanolic extracts were combined and concentrated under vacuum to 500 ml, acidified with 5% tartaric acid, diluted with one liter of water and filtered with the aid of Supercel.

The resulting solution was made basic with concentrated ammonium hydroxide and then extracted three times with 500 ml portions of ethyl acetate.

When evaporated left 4.4 grams of residue.

The residue was dissolved into 20 ml of methylene chloride and extracted three times with 20 ml portions of 5% hydrochloric acid.

After combining, the acid fractions were brought to pH 9 [this is enough for an excellent recovery but it should have been slightly higher for full recovery; pH 9.3 or above.] with concentrated ammonium hydroxide and extracted four times with 50 ml portions of ether. After combining, the ether fractions were dried with magnesium sulfate and evaporated to dryness.

They recovered 1.1 grams of crude alkaloid (0.12%) which was purified via its picrate.

[i.e. By dissolving the crude alkaloid in methanol and adding an equal weight of picric acid dissolved in methanol. The picrate was recrystallized three times from methanol-acetone, treated with alkali to yield the free base which crystallized. It was recrystallized from hexane containing some ether.]

This is a final recovery of 0.023% of very pure 5-methoxy-N,N-dimethyltryptamine. [210 mg, from 900 grams of bark.]

They had a yield of 0.04%. as calculated from the picrate. [They obtained 0.7 grams of the picrate.]

Ott commented on the existence of 2 unpublished analytical accounts claiming up to 11% DMT from the rootbark of *Mimosa tenuiflora* from Chiapas.

See the entry under DMT occurrences herein.

Pachter et al. (1959) Journal of Organic Chemistry 24: 1285-1287:

Using Mimosa hostilis roots.

2.7 kg of ground roots exhaustively extracted with ethanol.

After removal of alcohol, the residue was stirred with 500 ml of 5% aqueous ammonia and 2 liters of chloroform.

They used a centrifuge to separate the layers. The resulting emulsion was extracted several times with aqueous ammonia and chloroform until it did not contain any alkaloids.

The combined aqueous extracts were then extracted three additional times with one liter portions of chloroform.

All chloroform extracts were combined, concentrated and extracted with 2% hydrochloric acid until exhausted of alkaloids (They used Mayer's reagent to check)

The combined acid fractions were brought to pH 9 with concentrated ammonium hydroxide and then extracted three times with 500 ml portions of chloroform.

They used a centrifuge to separate the emulsions.

After drying over magnesium sulfate the chloroform fractions were evaporated to dryness yielding 18.5 grams of a crude brown alkaloid.

Part of the crude alkaloid was dissolved in boiling ether and filtered to remove black material.

The ether was then evaporated to dryness and the residue dissolved in 30 ml of methanol.

An excess amount of picric acid [based on weight of crude alkaloid. They only used 12 grams of the crude alkaloid and added 15 grams of picric acid.] was dissolved in 75 ml of methanol and added to yield a crystalline picrate which separated from the solution. [12 grams of crude alkaloid yielded 21.9 grams of picrate of which they recovered a total of 20.7 after three recrystallizations from Hexane. 16.9 grams as first crop and 3.8 as second.] This was filtered and dried.

It was converted to the free base which was then crystallized from hexane containing a little ethyl acetate.

10 grams of the picrate yielded 3.3 grams of base.

The DMT content of the roots was found to be 0.57% as calculated from the picrate.

If they had used all of their crude base, their yield would have been 10.53 grams; a final return of 0.39% of highly purified DMT.

Rovelli & Vaughan (1967) Australian Journal of Chemistry 20: 1299-1300:

Using Acacia phlebophylla leaves.

Dried and milled material was macerated repeatedly with methanol.

After concentration the extract had an equal volume of dilute sulfuric acid added and was then filtered.

The resulting aqueous solution was made basic with ammonia and extracted with chloroform.

The chloroform was then extracted with dilute sulfuric acid which was again basified with ammonia and extracted with chloroform.

The chloroform was evaporated to yield a crude alkaloid fraction.

This was then purified by eluting from a column of neutral alumina with benzene-methanol (98:2)

Thompson et al. (1987) Journal of Agricultural and Food Chemistry 35: 361-365:

Using dried and pulverized roots of *Desmanthus illinoensis*.

They had originally used methanol for the extraction. They apparently extracted this with both hot and room temperature aqueous solutions but are not clear on this point. They passed the aqueous extracts, after concentrating under reduced pressure, through Celite to remove precipitated chlorophyll.

This could prove most useful if working with *Phalaris* spp.

The aqueous solution was adjusted to pH 7 and extracted with methylene chloride to obtain a neutral fraction.

The pH was then adjusted to 3 and again extracted to yield a fraction with no activity.

The pH was then raised to 11 and again extracted to yield a basic fraction.

DMT was recovered from both the neutral and the basic fractions.

They used DCC and HPLC to purify.

Used Vanillin-Sulfuric acid reagent to visualize for tlc

Ueno et al. (1978) Chemical and Pharmaceutical Bulletin 26 (8): 2411-2416.

Using the roots of Desmodium caudatum.

They used methanol to extract the dry chips of root. This was evaporated to a residue.

The residue was partitioned into a mixture of ethylacetate and water. [We suspect part of their product was lost with the ethyl acetate at this step.]

The water was then extracted with butanol.

After filtration the butanol extract was dissolved into water [butanol removed first??] and the aqueous solution made basic with sodium bicarbonate.

The precipitate that formed was collected by filtration and extracted with chloroform. The filtrate was then also extracted with chloroform.

After removal of the chloroform the residue was suspended in 5% aqueous hydrochloric acid and added to a solution saturated with picric acid. The precipitate was collected by filtration, washed with benzene and chromatographed on a column of silica gel.

DMT picrate was first eluted with benzene-ethyl acetate (13:7) and then recrystallized from methanolwater (1:4). It was then dissolved in a 1% potassium carbonate solution (w/v) with warming and extracted with chloroform. After removal of the chloroform the base was recrystallized as colorless needles from petroleum ether.

Wahba Khalil & Elkheir (1975) Lloydia 38: 176-177:

Used 1.5% concentrated ammonia (28%) in methanol as a solvent for preparative tlc of DMT from their crude alkaloid residue. They used methanol to elute. DMT was the only alkaloid present.

Wassel et al. (1985) Scientia Pharmaceutica 53: 169-170:

Using *Phragmites australis* they first defatted with *n*-hexane and dried the material.

They extracted with methanol in a Soxhlet

The extract was concentrated under reduced pressure and 4% acetic acid added.

It was then made alkaline with $\rm NH_4OH$ and extracted with chloroform.

They used a column of Brockman Al₂O₃ CHCl₃ eluted gramine middle CHCl₃ eluted DMT CHCl₃-Methanol (99:1) eluted bufotenine CHCl₃-Methanol (10:90) eluted 5-MeO-MMT Methanol eluted an unidentified base.

Woods & Clark (1971) Crop Science 11:121-122:

Working with Phalaris.

They Soxhlet extracted with 95% ethanol, filtered and removed the solvent under vacuum

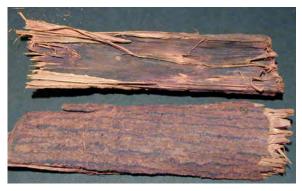
The residue was dissolved by alternately shaking with equal portions of 2N sulfuric acid and chloroform, using two portions of each. The chloroform, containing pigment was discarded.

They then saturated the aqueous layer with sodium chloride and made it basic above pH 11 with 40% sodium hydroxide.

The aqueous solution was then extracted with chloroform which was taken to dryness to yield a residue containing crude alkaloids.



Acacia obtusifolia bark Photo by Zariat



Virola calophylla bark

A few of the many plants assayed by Johnny Appleseed









Trout's Notes #FS-X4 Edited by Keeper. Trout. Portions indicated were written by Justin Case. ©1997 by Trout's Notes Revised 2000 & 2001 ©2002 by MydriaticProductions/ Better Days

Tryptamines from plants. Extraction, isolation & manipulations.

Disclaimer & Cautionary Statement

All information is contained strictly for informational purposes and should not be construed as advocacy for anyone to violate state or federal laws.

The following section contains techniques and procedures that might place one in direct violation of state and federal laws if they were put into practice. The information is contained strictly for informational and educational purposes.

Direct applications in areas where the law does not allow such manipulations can result in serious punishments including loss of property and lengthy imprisonments.

Use of the first person pronoun, whether singular or plural is not intended to represent any particular single person or group of people. Use of a fictitious, composite narrator was considered the only feasible way to discuss this subject without identifying a lot of people who may have knowingly broken the law and then shared what they learned with us.

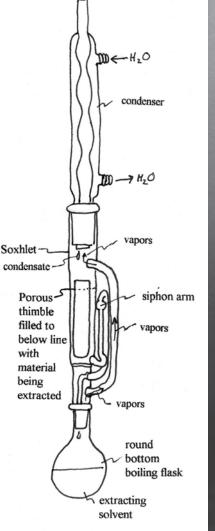
We do not advocate the use of illicit —or for that matter, *any*— drugs by uninformed or underinformed individuals.

However, we also recognize that many people will choose to use drugs whether they are informed or not.

We do not intend to encourage or promote drug use. We do want those who are already determined to use these substances, regardless of current legal status, whether planned as sacrament, 'recreation' or experimental material, to be able do so in an informed, knowledgeable and responsible manner.

Our hopes and intentions are that, through education and awareness, more informed choices can be made, thereby minimizing the risks often associated with substance use.

It is with this in mind that we present the following.



Rendering by JC.

Photos thanks to our friends at UT Austin



"More than you need to know?"



Acacia phlebophylla



Acacia obtusifolia

Acacia phlebophylla

Photos by Des Tramacchi

Extraction of tryptamines & other simple alkaloids; through manipulation of their free base⇔salt conversions

by Justin Case

Before we begin, we'll need to discuss a few basic concepts and some important terminology. I will attempt to keep it elementary and assume total ignorance on the part of the reader so that I don't omit anything important. For those readers who are well versed, either skip ahead or bear with me.

First, alkaloids are called bases because they are basic in pH.

pH is a measurement of the relative concentration of acidity or basicity (concentration of hydronium ions; i.e. another name for positively charged hydrogen ions, H^+) [Note 1] It is an inverse logarithmic scale (sort of like the Richter scale, except backwards) which means that one pH unit of decrease is 10 times the concentration of the number immediately above it, 2 pH units difference is 100 times larger, 3 are 1000 etc...

The scale runs from 1 to 14; with 7 being neutral. Down to 1 is acidic (the most hydronium ions) and up to 14 is basic (the most negatively charged ions, usually, but not always, hydroxyl radicals (OH^-).

Alkaloids in their natural state are usually bases rather than salts. This is what is called a free base state. i.e. it is not part of a salt and has a naturally basic state.

If one takes an acid and a base, one molecule of each [Note 2], such as mixing hydrochloric acid (muriatic acid) and sodium hydroxide (Lye), the acid and base combine to form a salt (in this case sodium chloride, i.e. table salt) and water. [Note 3] (This is also called neutralizing the base.)

Salts usually dissolve freely in water. Most free bases do not. [There are exceptions for both.]

Free bases usually dissolve freely in organic solvents. Most salts do not. [There are exceptions for both.]

Alcohols quite often dissolve in both. (I don't want to get off-track by getting into this yet.)

If one adds an acid to a base, the salt it forms depends upon the acid. Hydrochloric acid forms the hydrochloride salt. Sulfuric acid (battery acid) forms the sulfate salt. Citric acid (like in lemon or lime juice) form the citrate. Ascorbic acid forms the ascorbate. Acetic acid (vinegar) forms the acetate. Tartaric acid: the tartrate; Picric: the picrate...etc..

If one takes a salt and adds a base to it sufficient to raise the pH past a point known as the ${}_{P}K_{A}$ (this can be different for each base) the base that was part of the salt is "liberated" as the free base. (Also called regeneration of the base.) The base used for this must be stronger in basicity than the substance that it is liberating.

This is why with DMT (pKa 8.68) one can use nearly any strong base but with mescaline (pKa somewhere \sim 9.6) it is usually recommended that one use ammonia or lye for ease of liberation.

For DMT or 5-MeODMT (pKa 9.3) one can use Am-

monia (Ammonium hydroxide), Sodium carbonate (washing soda), Sodium bicarbonate (baking soda) (while it is claimed by others to work, I have never actually tried using baking soda), sodium hydroxide (household lye) or potassium hydroxide. The last two or the first one are the best. Do not, however, use ammonium carbonate in alkaloid solutions as they are incompatible.

I have heard it said that you can also use large amounts of household ammonia but I personally would be concerned about all the detergents and additives. Besides, smaller volumes are easier for most people to handle. 10% ammonium hydroxide is available in some areas (sold as *Janitor's Extra Strength Ammonia* or *Industrial Strength Ammonia*).

Ammonium hydroxide (usually available as a 30-35% solution of ammonia in water) or lye will both burn your skin if they get on it. On contact they will feel slick (soapy- as they react with fatty acids in your skin and membranes to literally make soap!) and must be immediately washed off until the slick feeling is gone. Base burns can become quite severe if not immediately dealt with.

Gloves should be worn and washed before removing them. Splash approved goggles should be worn also since splatters can blind you. If you get ANY chemical in your eyes flush with water for 10 to 15 minutes then get prompt medical attention. [i.e. Flush your eyes with water until EMS arrives.]

Lye is easy to find at most grocery stores Do not just get Drano! You do not want any other ingredients to be present. It will say Household LYE on the can and list it as the only ingredient. Red Devil is a common and good brand, but there are many others.

To use, place a jar in a sink or bathtub (PYREX is best as it will get hot) [Quart Mason jars work but put in a sink anyway just in case]. It will definitely get hot and may break. Fill it about 2/3 to 3/4 of the way with water. Pour about half the can of lye in, stir briefly with a steel spoon (long handled) be careful not to drip anywhere when removing. A thick white layer of undissolved lye will be on the bottom. Leave it be. Cover (do not tighten lid) and let stand until cool (or ready for use). It is best to do this in advance, having a quart or two made up at all times.

When adding the lye, choking fumes will be generated. DO NOT BREATHE THEM OR ALLOW CONTACT WITH EYES.

Use a fan, or vent it somehow, and **dump it in, stir briefly** while holding your breath and run, opening up the windows and airing out the place before returning. Once dissolved, the fumes will no longer be present (for the largest part). Before you put it up; wait until the jar has cooled enough to touch, put a tight lid on it and wash off any lye that might have gotten on the outside of the jar. Label it so no one accidentally opens it and gets hurt. Keep all chemicals locked up and out of the reach of children. Always label ALL solutions. Do not ever trust your memory. Wax or grease pencils work better than pens and markers.

By the time it is ready to use there will probably be nothing left on the bottom of the jar. If white lye crystals are still there it simply means that you have added more than will dissolve. This will not interfere with anything. Similarly a layer of carbonate may form on the bottom due to carbon dioxide dis-

See also the approach on page 228

Notes for this section are on pages 209-210

solved in the water (more so in some towns than others). This is not a problem for DMT extraction but, if desired, you can decant (pour) the clear solution into a new jar to eliminate the bottom layer (left behind).

Lye can also be made the old fashioned way, if necessary, by repeatedly leaching out wood ashes with a water solution which will grow stronger with each leaching. [Use new ashes as needed.] Some plants produce a more basic ash than others. This is beyond the scope of this text to discuss in depth. See under soap making in 'self-reliance' handbooks or works on early pioneer technology.

Also, the basic ashes used for snuff making will work; such as are extracted from plant ashes, or obtained via calcining [Note 4] calcium carbonate sources such as snail shells, clam shells or even limestone. Manfred Junius (*Practical Plant Alchemy*) gives a good discussion on calcining and preparing basic salts from the ash residues.

Calcium hydroxide is also available in an industrial grade as 'slaked lime' or 'hydrated lime' and used for making cement. If this is to be used a solution should be made, tightly sealed and allowed to settle overnight. The top solution should be decanted before use to eliminate the layer of solid which will form. If there is any frothy or scummy-looking material present on the surface, skim it off.

10% (or weaker) sodium hydroxide solutions are also readily available to winemaking supply stores. This can work although a stronger solution will be found more useful due to the decreased volume required.

If you can get ammonia you can use it just as it is. (Usually sold as ammonium hydroxide (ammonia dissolved in water)-commonly ~30 or 35% although 55% is sometimes available)

Supposedly the carbonates will work as well. If the solution to be made basic is a water solution the base can be added as a powder otherwise it will need to first be mixed with water. It may be a good idea to add it as a solution regardless. I cannot vouch for their effectiveness, they supposedly work but I have no experience using them for this purpose. [Do not, however, use ammonium carbonate with any solution containing alkaloids!]

Ammonia or lye both work great!

Sodium carbonate is available at most pool supply centers as "*pH Up*". Always check the label to be sure this is actually the active ingredient and that no other chemicals are present!

One drawback to carbonates is that they generate considerable gas when neutralized with acids. Usually, when using carbonates, the solution should first be cooled in ice before being neutralized. (Stand the vessel in a larger container packed with crushed ice until quite cold.) Foaming may also be a problem when using carbonates; introduce them gradually, and use a vessel considerably taller than the level of solution.

Calcium hydroxide will also have this tendency as it will draw carbon dioxide out of the air and at least partially convert to calcium carbonate when it is in solution or if the powder is allowed much air contact. It also tends to generate heat during neutralization and should be cooled prior to addition of acid. Do not allow it to boil up and foam over or product will be lost. Work with cold solutions and add gradually and cautiously. [If neutralizing a carbonate solution with an acid use a similar approach.] Lye and ammonia are much better choices if available.

A pH meter would be extremely helpful; otherwise use litmus paper or other indicator and add extra base to be sure it is basic enough but we'll come back to this later. <u>A problem with</u> <u>litmus</u> [Note 5] and phenolphthalein [Note 6] is that both turn colors **before** the necessary point for full liberation of DMT free base. This means that you should add extra base. A useful approach would be to first do a solvent extraction of the basic solution at around pH 8 or 9 and then raise the pH to 10 or above and extract again.

A few more phrases you will need to be familiar with (perhaps obvious to most):

Basic solution: A solution that is basic in pH.

Acidic solution: A solution that is acidic in pH.

Aqueous solution : Water based solution, it can be neutral, acidic or basic.

Organic layer: The layer of solvent be it Benzene,

Chloroform or other nonaqueous solvent. Organic means that it is based on carbon. The organic layer may form on top of the aqueous solution or on the bottom of it depending on relative molecular weights and densities. The common parlance 'organic' is an arbitrary corruption of the word. In *every* sense both Acetone and Toluene are organic.

Mother liquor: a solution which has given birth to crystals. These can generally be concentrated further for a second crop.

That said, back to the story....

The initial extraction can be done a number of ways.

An organic solvent can be used (we'll discuss solvents below) or an alcohol such as methanol or ethanol can be used. Many people add 3-5% acid to the alcohol. The organic solvent will extract the free base as is [prior basification may be needed in some species; see below]. The alcohols will also unless an acid is added. Then it will be extracted as the salt corresponding to the acid selected.

To extract; the plant material needs merely be soaked tightly closed in the solvent for several days to a week. The solvent should then be strained off (remember that many organic solvents will dissolve things of plastic such as funnels. Even if they do not dissolve it fully, plastic material will end up in your product. Not good if you plan to smoke it.

Many plants extract better if dried first.

[Others should be extracted fresh. Some, it will depend on what you are after. For example if extracting DMT from *Phalaris* it should be preserved in alcohol immediately upon harvest and processed as fresh plant material. *Desmodium gangeticum* leaf should also be processed fresh if wanting to maximize the DMT yield. BUT, If wanting 5-MeO-DMT, *Phalaris* should be allowed to dry first, as should *Desmodium gangeticum*. Total alkaloid yield will be lower in both of these materials if dry plants are used but the relative ratio of 5-MeO-DMT will be proportionally higher. Apparently, enzymatic activity affects the alkaloids both immediately after harvest and during drying. The addition of alcohol **during the act of harvesting** will stop said enzymatic activity and in the case of *Phalaris* will ensure highest possible recovery of alkaloid. But, even material preserved in alcohol will soon show loss of alkaloid content so processing should begin as soon as possible.]

Once dry, grind finely and add solvent, be it alcohol or an organic solvent.

[If using fresh material, it will need to be chopped, crushed or blenderized in a blender that is not affected by the solution used for the initial extraction. A hand held homogenizer [like a Tissu-izer; not a plastic Bemix] is very useful or a stainless steel Waring blender is also an excellent choice but if necessary, the material can be finely chopped, like salsa or pesto, using a knife or scissors or food processor.]

If using dry material: 'rehydration' (with the solvent of choice) prior to beginning is crucial.

To accomplish this, add enough liquid to cover by at least an inch and shake well. Shake frequently while the dry material absorbs the solvent as it will swell substantially in the process of rewetting. Once the plant stops absorbing the solvent add enough to cover it by 1/8 to a maximum of 1/2 an inch. [Obviously, skip to this point if using fresh material.]

After soaking overnight or even for a week the solvent is filtered off of the plant material (Do not squeeze at this point.) If fresh solvent is added once or twice more almost total extraction should occur. The third extraction will recover only a few percent and can be omitted. Only after the final extraction is complete, squeeze out everything you can from the pulp. Squeezing all liquid from the pulp at every step will create an unnecessarily large volume of solvent to deal with.

In the case of long soaking times try to keep the container out of light as much as possible [Note 7]. A dark cupboard works as does wrapping in aluminum foil. This isn't always necessary but some molecules are unstable when exposed to light while in solution. Aluminum foil is cheap, and placing in a cupboard costs nothing. [We used to use aluminum foil when running platinum catalyzed hydrogenation of folic acid (notoriously light sensitive) and never had any problems with the reactivity of the resulting tetrahydrofolate; our assay reagent. We prepared this regularly in quantity and never used anything more than a tight wrapping of foil around the reaction flask.]

While soaking in a solvent at room temperature works just fine; gentle heating, short pressure cooking, percolation, Soxhlet or other reflux extraction (as in an ISO-2), microwaving and/or ultrasonification all will decrease the time needed for extraction. In all cases, filtration (or centrifugation) will readily separate the spent pulp from the alkaloid-rich 'tea'.

A neat trick used by friends, for crude filtration of small volumes, utilizes a press-type coffee maker.

At this point the solvent can be removed (evaporated) This is a must if alcohol is used as alcohol can dissolve in water and many organic solvents making an inseparable mess. Especially if alcohol is used, removal under reduced pressure is recommended.

If an organic solvent was used then you can directly use it [Note 8] and now extract it with an acidic solution as is described below. Defatting is not feasible if this is the route chosen. If defatting is necessary, it can be performed by extracting the resulting acidic aqueous solution with an organic solvent. Usually defatting is not crucial unless there is a high lipid component to the plant material such as would be the case if seeds were being extracted. [If this were the case, I would suggest moistening slightly with an acidic solution such as citrus juice and then directly defatting the ground seeds or taking an alternate route altogether.]

A safe and simple approach is to start by gently cooking the plant material out with an acid such as lemon or lime juice in water (12-15-20 minutes per cooking). The resulting citrate salts are extremely water soluble. A couple of such short cookings and strainings will remove most of what is there.

(Nylon mesh straining bags are available from wine making suppliers and work very well. [Never use such plastics with solvents. Most can dissolve part of the plastic.] Cotton cloth also works. Clean; no dye or color. An old clean T-shirt or cotton sheet works well. Cheese cloth is too coarse and flimsy.) If the material is finely powdered clogging filters can be an issue if Whatman filters and vacuum filtration is not available..

A three step process that can readily and effectively deal with this:

1) Use a fine stainless steel filter to do an initial rough separation of solid materials. Empty and rinse it clean as often as it clogs.

2) Use a cloth filter, emptying it and rinsing clean each time it clogs. Paper coffee filters can work but clog rapidly and must be replaced frequently during the process. They also let a lot of fine particles pass through.

3) Permit the liquid to settle (ideally in a refrigerator) and decant or pipette off in order to remove the finest particulates that pass through the filter

Despite them being rather stable, overcooking can damage the alkaloids; especially in the presence of acid.

In the case of extracting with citrus juice, what one ends up with is a water solution containing the alkaloid(s) as salts (mainly as their citrates). This is the tea that many drink with added beta carbolines, such as *Peganum harmala* or *Banisteriopsis caapi*.

If making such an ayahuasca analog it is recommended that the harmine source be either cooked out separately and the two combined or else harmine be purified via Hasenfratz's method, weighed out and added to the drink. [Hasenfratz's method can be found in Trout's Notes on Ayahuasca & likely on the web.]

If extracting for purification rather than hoasca, do another prolonged simmering (for around an hour) after the first couple short extractions. This will ensure that you have exhausted your material.

If *Desmanthus* roots are used fats and oils are minimal. However if *Phalaris* is used there are fats, waxes, pigments and other materials present which can interfere with ease of the rest of the process so defatting should be done. Omit this if *Desmanthus* is used. [Another variation to accomplish this could be that if you first extracted with a solvent such as methylene chloride, it could be removed and the residue taken up into alcohol or an acidic solution and then filtered to remove any insoluble material. The reverse will also work.]

In a later section, the reader will find abstractions of the procedures used by professional researchers in dealing with a number of different plants. They may find that by picking and choosing particular pieces from these they can adapt the techniques to fit their own unique situation, available materials and equipment.

Justin Case

To defat; add an organic solvent to the alkaloid containing acidic solution above (always check to be sure it is acid; and below pH5) You will need one of the following: (preferably) a separatory funnel, a flask with stopper or glass stopper, or mason jars, or another jar with lids. The organic solvents will dissolve the lining, often even the paint, inside lids! To avoid this cover the top of the mason jar with heavy duty aluminum foil, smooth it down and then carefully add the lid making sure it is tight. Replace it as often as necessary. Turn the jar upside down, then right side up, many times (50-100), over and over, slowly and smoothly. Wear gloves in case of leakage. You want to mix the liquids thoroughly. If you shake it vigorously a thick emulsion may form which may or may not separate unless you have a centrifuge handy. The two liquids will separate into two layers. If there is a third and there normally is; the middle funky looking layer is the emulsion. It is usually present, you just want to minimize it. Do not discard emulsion layers as a fair amount of alkaloid can get tied up there. Label them, set to one side and deal with them later.

Remove and discard [Note 9] the organic solvent containing the dissolved fats.

One point which must be stressed is that while any food safe acid will work, acetic acid should not be used in combination with chloroform during defatting (possibly also true of methylene chloride?).

While it will work, DMT acetate is somewhat soluble in chloroform and will be at least partially extracted into it during defatting.

Another point is that if using a plant source with DMT-Noxide, while it is not soluble in petroleum ether, it is soluble in petroleum ether that contains fats [Note 10].

[See a later section for some potential approaches to recover possible products present in the defatting solution.]

We would recommend converting the N-oxides to their free base before attempting defatting or purification. i.e. extract with acetic acid [extract with 3% to straight 5% vinegar] and reduce the N-oxides by adding an excess of powdered zinc and allowing it to react (see elsewhere here), before proceeding to the next step. [Use ammonia as your base if going this route.]

Now the fun begins.

Since salts aren't soluble in the solvent they are still left in the tea (the acidic "aqueous solution")

Now add enough base to raise the pH up to <u>above</u> 9 (above 9.3 for 5-MeO-DMT). The aqueous solution will go dark or at least darken. This is good. When you are sure it is basic enough (Don't go overboard or you will have a large volume to deal with!) add an organic solvent like before when we defatted. Repeat the mixing process.

This time since the addition of base has turned the salt into the free base the alkaloids will migrate into the organic solvent. Drain off or pour off what you can of the solvent and <u>save it</u>.

Add more solvent and repeat. You can even do it a third time! BUT once you add base you should finish this part of the extraction within ~ 6 hours. Otherwise degradation of the alkaloids may begin to occur. (This is not much of an issue with DMT but is for mescaline and some other alkaloids.) It will not affect what is in the solvent so after the final time just let the

whole thing stand over night or even a couple days to get the best separation you can on the last extraction. When done, throw away the strongly basic water solution. Flush it down the toilet to help keep your pipes clean!

As mentioned briefly earlier, one could also use a pH of around 9 for the first extraction, then raise it to over 10 for the second (some raise it even higher for the final extraction.)

It is worth remembering that bufoteine is amphoteric so will not effectively recovered by a solvent at around pH 11.

(Some people have success with vigorous shaking, then letting it sit for weeks (or months) and draining off the solvent as it settles. This is OK when processing large quantities of fairly stable molecules such as DMT but for most it is risky (legally) to keep such a thing around for any length of time. Another problem is this only works well for solvents that settle to the bottom. It clearly is not acceptable for other less stable molecules such as mescaline which are not stable when kept for long periods in a strongly alkaline aqueous solution. Psilocin is even more unstable in an alkaline solution.)

Evaporating the organic solvent will leave behind a thick oil. It may be clear to yellow depending on how pure. Probably green to black in the case of *Phalaris*!

[If you originally extracted with alcohol you can supposedly filter it through a short column of diatomaceous earth or Celite to remove the chlorophyll. We have never tried this.]

Keep the syrup in a warm (not hot) dry place. If it is allowed to sit you should see DMT crystals starting to form within a day or so. Maybe not. It sometimes doesn't like to crystallize unless you already have a few crystals to drop in as seeds. If you do, it will crystallize within a few hours; often starting in moments. Cover loosely with paper to keep dust out.

Or dissolve the syrupy residue in a little grain alcohol and mix with an herb which can be smoked.

Allow it to dry, with frequent stirring, and Voilà!

It is generally not a good idea to go directly from an organic solvent onto a smoking material like a dried food-safe herb. Sometimes residual traces and foul tastes remain.

If possible, it is also a good idea to "*dry*" the solvent before it is evaporated. This can be done by adding a small amount of desiccant to the solution (just enough to almost cover the bottom), swirling gently and filtering it to remove the desiccant. Alternately the solution can be poured through a column of the desiccant. Calcium or magnesium sulfates are good choices and easy to get [**Avoid CaCl**₂] Be sure they are anhydrous and intended for use as desiccants. It is possible to heat them to dehydrate them but heating must not get too high. Follow the instructions on the label.

Another approach is to wash the organic solvent solution with a **strong** lye solution to remove any traces of water from it.

A molecular sieve could also be added and then filtered off as a final step. These are small bead-like materials with precisely graded pores. They are added to the solution, swirled and removed by decanting or filtration. They can also be used in a column; with the liquid then poured through the column. Be certain the pore size is correct for retaining water [#3A or #4A] This is best done after other preliminary drying with another desiccant Most people omit drying the solvent entirely. It is of great benefit if you are trying to get your material to crystallize but not crucial if you are just suspending it on another material to smoke.

As mentioned, once the organic solvent has been evaporated the residue can be dissolved in a little grain alcohol and mixed with a material for creating a smoking blend. Use only enough alcohol to get the material to dissolve. If impure and solvent extracted there may be alcohol insoluble material present, this is inert and should be filtered out before mixing with the herb. [Wet the filter paper with alcohol before filtering.]

It is best if the product is weighed out and added to a known portion of herb so you can regulate the dosage. 1 gram of pure DMT mixed with 9 grams of herb will make 10 grams of 10% DMT smoking material. Smoking 100 milligrams of the herb will roughly equate to 10 milligrams of DMT less what is lost as side smoke. 20% is actually a bit more useful in this regard but it is strictly a matter of personal approach and taste. [A 20% blend is created by combining 2 grams DMT with 8 grams of herb or 200 mg with 0.8 grams of herb.]

We find 20% to be an excellent proportion for 5-MeO-DMT as it readily permits eyeballing of individual doses.

For DMT afficionados desiring full effectiveness in a joint, we would suggest creating an approximately 50% blend. Before you pass it to anyone, be certain to inform them about its contents.

A strong DMT joint can be a rude surprise for someone thinking they were smoking only pot. Potentially even distressing depending on what other substances might already be in their system

To add to an herb, dissolve the DMT in the **least amount possible** of ethyl alcohol (Everclear). Add with an eyedropper. Once completely dissolved, stir in the weighed and cleaned herb. It should absorb all the liquid that is present.

If not, too much alcohol was used and the herb will need frequent and repeated stirring as it dries.

Stir and mix the herb until homogenous. Repeat this occasionally as it dries. Allow to dry thoroughly before smoking. Do not use heat for drying unless necessary.

The intensity of the experience is much less when smoking it on an herb as opposed to using the free base but some extremely interesting and protracted experiences are possible.

Another useful route is to do the initial extraction with alcohol, evaporate it to a dark sticky residue (preferably under a vacuum) or thick viscous liquid and then dissolve this as best that you can by mixing with a dilute acid solution (2-10% acid or 20-30% lemon or lime juice in water). This should then be well filtered before being made basic [Note 11] and extracted with an organic solvent as above. Be absolutely certain, when doing this, that all the alcohol is removed or (depending on the solvent of choice) it may help the solvent and the aqueous phase (water solution) mix so there might be no separation ever. (IF this becomes an issue try adding salt - to the point of saturation-to the solution.)

If doing the first extraction with an organic solvent, it may be helpful to break apart any organic acid-conjugates, that might tie up part of the alkaloids (this is crucial in some species), by first pre- **moisten**ing the plant material with a little ammonia and a small bit of solvent and allowing it to stand for a few hours before adding the extraction solvent, then, after soaking or percolation to extract, filter off the solvent, take it to a residue, dissolve the residue in a dilute acidic solution, filter and treat as above.

Some add magnesium oxide to the plant material, moisten and mix them thoroughly and then take it to dryness, prior to beginning their extraction.

Solvent extraction can be carried at room temperature by simply soaking several days to a week or the material can be percolated or Soxhlet extracted with heat or even mechanically at room temperature.

Unless well equipped and having access to huge acreage, *Desmanthus* is not practical for producing any commercial quantity but ANYONE can easily turn a pile of plants into a small personal amount of more useful substance. About a half pound of cleaned & dried *D. leptolobus* root bark can yield about 200 mg of crystalline solid. The largest component is DMT.

This 200 mg. may take 8 to 12 hours of labor to dig, clean and reduce to powdered root bark and about two days of effort to process under primitive conditions. The current street price of several hundred per gram (for 1000 mg. of pure) almost sounds like a bargain! About 1 hour of the plant processing time will be actually spent digging, the rest will be cleaning and peeling. If using air assisted washing techniques, the time can be reduced. If a person actually has a lab, processing time will be far less. The above estimate is for work done under primitive conditions with minimal equipment.

Back to the question of solvents

Many will work. What is chosen will depend on a number of factors that will vary according to the individual situation.

Toxicity and flammability may factor into the decision; ease of removal and cost may also be factors. Usually the most important factor for our consideration, due to modern day repression, is availability.

If one uses methylene chloride or chloroform (the first moderately easy to get; the second more difficult) the organic solvent will separate as the layer on the **bottom**. (Both evaporate rapidly and won't burn but are slightly polar and dissolve more water than do nonpolar solvents.)

If one uses petrochemicals (hexane, xylene, benzene, toluene etcetera) or ether; they will separate as the layer on **top**. (All catch fire readily but are more nonpolar)

Helpful Tips

It is always a good idea to label everything even if it is just as a, b or c and save them ALL until you are completely finished with the entire process and absolutely certain about what can be thrown away. It is too easy to discard the wrong solution especially at first if unfamiliar with the procedure.

It is also possible to recover a higher yield by assiduously eliminating sources of careless waste such as inadequately extracted solutions and mother liquors.

Justin Case

Carefully wiping up even small spills for recovery (always working in a clean area) and rinsing any glassware used for handling or manipulating concentrated solutions can also contribute to a substantial decrease in loss.

Acetone can work for many tryptamines but will also extract **many** undesirable contaminants (including tannins) so its suitability will be limited by the material of choice. It mixes readily with both water and many solvents so can't be used in acid-base partitioning. It CAN be used to obtain a residue that can then be purified further. Limited trials for DMT have not been reported to be promising.

Alcohols:

Ethanol is readily available as 190 proof grain alcohol. Do not use less than 140 proof (70%) for extraction purposes.

Be certain that ALL alcohol is gone before processing a residue as the presence of alcohol may interfere with the separation of your aqueous and organic layers. [The repetition of this is not accidental.] The slow evaporation of alcohol is one reason it is not more widely used. A vacuum, rotary, climbing film or flash evaporator is very handy and not hard to construct. They can speed the removal of alcohol without harming the alkaloids.

Methanol is also available and very effective but far more toxic to work with. Fumes are dangerous and so is skin contact. Methanol may not just kill you. It may also leave you alive and blind or brain damaged. Methanol is also known to adversely affect some alkaloids and additionally will usually leave plasticizers behind in the residue. As these are suspected contributors to a variety of problems this is not a good thing.

Pure **Isopropanol** or **Butanol** may be available in some areas. DO NOT use 35 or 75% rubbing alcohol from the drug store. Only use pure isopropyl (99% is readily available). Isopropanol is often used for cleaning and sterilization in commercial edible mushroom cultivation and is specifically sold for this purpose. It is also available from larger printing supply houses for cleaning ink from printing presses and desktop printers.

Chloroform works well but is dangerous to human livers & is not always readily available.

Coleman Fuel is recommended in the counterculture literature but I have some concern about what happens to the added rust inhibitors. Some solvents such as deodorized paint thinners use lead salts. There is every indication that they may end up in your product. [Note 12] Besides, it apparently is in the same range of hydrocarbons as **Petroleum ethers**. Sure, when dealing with small amounts of alkaloids and a decent volume of solvent, this will effectively recover some product but it still is a poor choice of solvent, except as a last resort, since DMT is far better dissolved by many other things.

Dioxane, while one of the best solvents for dissolving DMT, is incredibly dangerous. It is toxic enough to **kill you by breathing it**, may form explosive products during prolonged storage and should be avoided. It is an exceedingly hazardous material and said to leave a residue that is toxic if injected. Like **Tetrahydrofuran**, another excellent but potentially dangerous solvent for DMT, it is difficult to obtain.

Ethyl acetate is a good solvent but fairly expensive and may not be readily available for many people.

Ether may or may not be easy to get depending on where you are. It is readily available in some areas, especially northern states where it finds usefulness to get older cars started in cold weather. [Note 13]

Hexane or Heptane are easier to find. [These are the petroleum distillates that are 2 and 1 carbon units less, respectively, than octane.] Hexane is getting more difficult to purchase in large quantity due to antidrug manufacture efforts aimed at reducing precursor availability. [Note 14] All petroleum distillates have the tendency to leave strong and bad tastes in the final product. This can be helped by dissolving the residue in alcohol, filtering off any insoluble materials, evaporating with frequent stirring in a large glass dish or better still use xylene or toluene which leave no such residual tastes once removed.

Hexane, or heptane are both easy to get in some areas. If not, look at the label on **rubber cement thinners**; many are one or the other. Go to stationary or paper supply especially the larger office supply and read the label on their rubber cement thinners. Keep looking until you find a good brand. Ross, for example, uses hexane. Sanford's is heptane and works well. This seems to vary regionally. It will come in pint or quart cans, Maybe gallon sized if you are lucky. While not great solvents for tryptamines, they can in fact work fine for obtaining small personal sized amounts.

Methylene chloride (AKA Dichloromethane: Dichlor; DCM) is commonly used. It is used in paint strippers (DO NOT use paint strippers as they contain **many** other things) and to clean out glass tubing used in neon signs. It is also used to chemically "weld" plastics. Most liquids sold for this purpose are pure methylene chloride. [Always check for other ingredients.]

It is usually available from chemical suppliers no questions asked but it is often mentioned in the counter culture literature so it is getting more monitored by some companies.

If using methylene chloride it is a good idea not to buy more than you will be able to use in a few months. It is so volatile that even a tightly closed gallon bottle will often evaporate to dryness in about a year. A metal drum will last longer if stored in a cool place.

Toluene (**Toluol**), which can be picked up by the gallon at larger paint supply and hardware stores, works quite well. It is often used as a fast drying solvent in enamels and lacquers (known as *"fast thinner"* among auto spray painters) Purchases can apparently be reported if 55 gallons or more.

Xylene (Xylol) works very well and is sold as a medium evaporating thinner, a remover for dried latex paint and for removing oily residues from aluminum or galvanized metal before painting, as it dries clean and will not leave a residue behind. It is readily available by the gallon at larger painting supply companies and some larger hardware stores. It finds many uses and is available through many sources. It is used as a thinner for background enamel paint on jewelry and for dissolving Canadian Balsam when mounting slides for microscopy.

There are sometimes restrictions on sales of xylene and toluene to limit their inhalant use by teenagers.

Tryptamine solutions in most organic solvents will be found to be stable for many months or even years. Fumes can easily give you away. Heptane or Hexane smell like Coleman fuel or paint thinner and don't have the same what-the-hell-is-that type smell that many solvents do.

All, except Methylene chloride and Chloroform, are inflammable [Note 15] (Ether is absurdly inflammable and flash fires can start from even things like sparks inside the motor of an appliance or a pilot light. A pilot light can ignite petroleum distillate fumes as well if concentrated enough.).

<u>All are toxic</u> [Note 16] and require good ventilation so you don't breathe them. (Work in front of a fan vented to the outdoors or better still use or construct a fume hood. If adequate venting is not possible; use an approved FULL-FACE respirator.)

Leave the area if you are feeling headaches, confused, dizzy or light-headed and do not return until your head clears. Be careful. ether can knock you out cold if you are careless. The distance between feeling 'buzzed' and being unconscious is not a long one. (Ether also smells distinctive and can be smelled for blocks, people associate smells like ether or ammonia with drug labs!)

If solvents stored in plastic containers are used, plasticizers, such as dioctylphthalate ester, can be a serious contaminant of the resulting extract. (A major problem with both methanol or chlorinated hydrocarbons)

While they can be removed with reverse phase column chromatography, prior distillation of the solvent, or filtering through porous alumina (Silva *et al.* 1998), a better solution is to avoid using such solvents.

Iodine precipitation

An interesting use of iodine precipitates to recover indole alkaloids from dilute and/or thick solutions was patented in 1972 by a Hungarian ergot producer.

The isolation scheme consists of adding 100 ml of a solution containing 20% potassium iodine and 10% iodine to 10 liter of alkaloid bearing solution with stirring.

The precipitate is collected by filtration. The alkaloid is regenerated by dissolving in 1 L chloroform and shaking with 500 ml of a solution of 5% sodium hydroxide and 10% sodium thiosulfate. The chloroform is then separated, dried over anhydrous sodium sulfate and the solvent removed under reduced pressure. Efficiency for the use of this to recover lysergic acid amide as a crystallizing residue was reported to be 65% from a fermentation broth containing 1.5 mg per ml

This is reported to reduce the solvents used and the resulting volumes of liquids required when working with dilute aqueous solutions (such as fermentation broths) by several orders of magnitude.

Scale the aboveup or down to suit the situation

It is also implied to work for any indole alkaloid suggesting that available biomass containing trace amounts of interesting indole alkaloids such as ibogaine or DMT might become more functional plant sources.

See Richter Gideon 1972 British patent #1374343

Assorted simple tryptamine containers



Psilocybe cyanescens Photo by Dr. P. C. Hickey







Copelandia cyanescens (NSW, Oz) Photo by Anonymous

A few words on Emulsions

A good centrifuge may be of usefulness for emulsion separations. (Run, using precisely balanced containers and an appropriate rotor, until liquids separate) Be certain you are familiar with its safe operating procedures and limitations.

An interesting approach found mentioned is breaking emulsions in a rotary vacuum evaporator. Obviously, care must be taken not to remove the solvent while doing this. [There has been suggestions that simply carefully degassing the liquid under a vacuum would help but know no one who has actually tried it.]

Emulsions can also sometimes be broken by filtering through a filter paper or glass wool, pouring the mixed solution down a clean glass rod, swirling them gently in a large glass container, pouring them down the side of a rotating clean and dry glass container (prior warming of the glass may help) or by gently mixing the emulsion with an additional amount of solvent and/or aqueous solution.

Many workers in laboratory situations repeatedly extract the emulsions with solvent and aqueous solutions until it is exhausted of alkaloid.

Another approach that can work quite well in many cases of seemingly unbreakable emulsions, especially if the problem is the result of the presence of water due to using a mildly polar solvent like ether or ethyl acetate or chloroform, or if there is a small bit of alcohol remaining in the initial extract, is to add salt (sodium chloride) to the point of saturation.

Separating and combining all similar emulsions will make them much easier to handle and process.

The most helpful approach to dealing with emulsions is to minimize them by avoiding vigorous shaking and minimizing the amount of air that is mixed with them during partitioning.

A prolonged gentle mixing, as little turbulence as possible) is more of a pain initially but in the long run can save a lot of time and product.

The actual degree to which emulsions will be found problematic will depend both on how mixing is approached and the specific choices of solvents and base.

NEVER FORGET!

In this day and age a simple thing like isolating an alkaloid [even if strictly for personal and sacramental use] is considered drug manufacture and is punishable by incredibly harsh mandatory minimum sentences.

In some states even simple possession of some specific pieces of labware is also a crime.

Know your state and local laws and take whatever precautions are necessary to keep yourself safe.

A valuable work for further details is Zubrick 2001

Tannins

Tannins are known to form precipitates with alkaloids, especially with alkaloid salts. (Due to the ready formation of hydrogen bonding)

Yields can be decreased if extracting bark or roots with high tannin levels.

We would suggest first **moistening** (only till damp not soggy) the material with a mixture of ammonia and ether (or ammoniacal chloroform [Note 17]) or mixing the plant material with a solid base (such as calcium or magnesium oxide or similar) and then extracting with an organic solvent like benzene, toluene, chloroform, or ether.

After removal of the organic solvent, the residue should be redissolved in benzene, toluene, xylene, or ether and filtered.

Tannins are more or less insoluble in these solvents. (The less polar the solvent; the less soluble the tannins)

Tannins are very soluble in water, glycerol, alcohols, and acetone.

They are incompatible with basic solutions or alkaloid salts. For this reason, some older workers pretreated their plant material with magnesium oxide or similar alkali prior to alkaloid extraction. (Moisten material, mix with magnesium oxide, calcium hydroxide, sodium carbonate or something similar, dry and then extract with an organic solvent.)

Tannins can be decomposed using mineral acids and it appears that the use of a strongly acid solution (pH1) for extraction is adequate to prevent problems when subsequently basifying.

This approach (extracting at pH 1 with aqueous HCl acidification prior to a normal recovery approach via neutralization and solvent extraction has gotten favorable reviews on the web. [See **page 228** for details]

The plant extract could also be passed through a short column of polyvinyl-pyrrolidone (PVP) to remove tannins. Similarly DE52 (a cellulose-based ion-exchange material) can be used as a column to remove tannins (if using methanol extracts)

Silva et al. 1998 & Van Middlesworth & Cannell 1998

Tannins will extract to a degree into chloroform. They can be effectively removed by washing the chloroform with a 0.1% sodium chloride solution and then drying the chloroform with anhydrous Na_2SO_4 for several hours. Silva *et al.* 1998

Other potential concerns

Alkaloids are also sometimes present in plants as **organic acid** conjugates.

Basification prior to beginning the extraction will convert them to a form that can be more readily recovered with organic solvents. Use of an aqueous acid for the initial extraction works well also.

High salt levels can be removed using Porapak Q packed in a column. See Niederwieser & Giliberti 1971

Chlorophyll and other plant pigments can sometimes be problems.

Simple solvent partitioning is often effective at removing them. Filtering the solution through a short column or Buchner funnel filled with Celite (diatomaceous earth) is said by many to remove them from alcoholic solutions.

Some workers have similarly reported success using a column of neutral alumina to retain chlorophyll.

Lead acetate precipitation is commonly employed in laboratories for this purpose (it can remove both fatty acids and pigments) but is unsuitable for most people's use due to potential toxicity.

Activated charcoal can work for removing chlorophyll but may also remove part of the alkaloids! Silva *et al.* 1998

Oxalates & oxalic acid can be a serious problem in some plants.

Oxalic acid can be removed from a concentrated alcoholic plant extract by precipitating it via the addition of a calcium hydroxide solution. (It precipitates as calcium oxalate which can be removed by filtration or careful decanting)

Shankland et al. 1998

Useful trivia concerning DMT & some related compounds

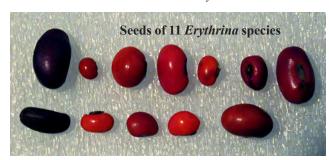
DMT is readily purified by molecular distillation or by vacuum distillation of the crude extract. Redistillation can yield extremely pure product.

After separating their crude alkaloid fraction, Hochstein & Paradies 1957 purified DMT by vacuum distilling at 170° at 0.01 mm. The product was a colorless oil which crystallized spontaneously on standing. Performed a second time to obtain a product with a tighter mp.

Corothie & Nakano 1969 used a bath temperature of 80-135° and distilled at 0.03 mm to obtain a slightly yellowish oil from the crude alkaloid they eluted from alumina.

Shulgin & Shulgin 1997 vacuum distilled using 130-140° at 0.1 mm.

Molecular stills are commercially available. A frequent form involves a spinning heated disc to volatize the material and a condenser plate a short distance away. This takes advantage of a very short distillation path being achievable when volatizing only very small quantities at a time and by use of a preheated carrier oil such as cottonseed oil. As only small fractions are



heated to a volatizing temperature and the shock of the temperature transition is lessened due to preheating of the carrier, degradation is minimized. The action is continuous and is amazingly efficient and effective for processing large volumes of highly purified material.

For an early piece on molecular distillation with a plan that any good glass worker and lab technician could master, see Hickman 1937

Another interesting piece of trivia is that compounds like Abrine (N-methyl-tryptophan) and dimethyltryptophan can be decarboxylated to MMT & DMT, respectively, by heating in a vacuum.

Hoshino 1935 reported that heating Abrine at 320° at 12 mm yielded CO, and MMT.

N,N-Dimethyltryptophan methyl ester occurs in several plants; such as *Pultenaea altissima* [at a concentration of 0.28% crude base by dry weight].

This base is readily hydrolyzed to N,N-dimethyltryptophan by heating with 1% aqueous NaOH for 3 minutes at 90°C.

See Fitzgerald 1963 or Ghosal et al. 1971c

Tryptamine or MMT can be methylated with methyl iodide in an alcoholic solution. It is a messy reaction and has a poor yield.

Trimethyltryptamine and trimethyltryptophan (hypaphorine) can both be demethylated by converting them to their methiodide, converting this to its chloride with silver chloride and heating in a strong vacuum.

This also is a messy procedure with a poor yield but can readily be used to make DMT.

Manske 1931c gives a detailed discussion of what is involved using tryptamine as a starting point. (See also Shulgin & Shulgin 1997)

What is interesting is that, while it is likely to produce low returns, if it was modified to use hypaphorine as a starting material this [rather dangerous] alkaloid is readily available in a number of plants. In some cases it occurs in surprisingly high concentrations.

For example, hypaphorine has been reported:

- at **5.8%** in seeds of *Erythrina acanthocarpa* E.Mey. in Folkers *et al.* 1941
- at **6.7%** in the seeds of *Erythrina pallida* Britton & Rose in Folkers & Shavel 1942
- Ghosal *et al.* 1972a reported a recovery of 1.2 grams from 500 grams of the roots of *Desmodium gyrans*.
- Many *Erythrina species* seeds have been reported to contain around 1-2%.

Found in seeds of EVERY *Erythrina* species analyzed although most contained far lower concentrations.

There are some reports indicating that *SOME Erythrina* species may be used as entheogens. It is not known what preparation is involved, but processing which led to demethylation and decarboxylation would produce smokable DMT.

Hypaphorine is a dangerous convulsant poison so this is not an area for random experimentation or bioassay.

Hydrogenation of Brominated Tryptamines

Hydrogenation of brominated-DMT gave DMT. Ex.: 15 mg. of 5,6-dibromo-DMT (0.04 mMol.) in anhydrous methanol (3 ml.) containing 5 mg. of 10% palladized charcoal was stirred under an atmosphere of hydrogen for 18 hours

Catalyst was removed by filtration and the solvent evaporated to obtain DMT in quantitative yield.

The same was true of 5-bromo DMT

Djura et al. 1980

Hydrogenation is easy to do if one has common sense and is safety minded. For a person who is set up for hydrogenation the source animals (sponges) could provide an excellent source of DMT.

Separation of some indoles using electrophoresis

The following procedure was used by Fish *et al.* 1956: Fish found N-methyltryptamine to be a stronger base than N,N-dimethyltryptamine; N,N-dimethyltryptamine-N-oxide was found to be a weak base.

Using a hanging paper design, paper ionophoretic separation was achieved using a 0.01M sodium borate buffer as the electrolyte. The buffer was allowed to rise via capillary action to wet the entire paper. (Using washed Whatman 1 paper)

Then the separation was carried out at 300 v and 3 milliamps for 5 hours, at pH 9.3. (Run at room temperature: 22-23°.)

This separation should be performed first with a small amount of material and visualized with Ehrlich's reagent or Xanthydrol to confirm and establish the values of the migration distance before using this technique for preparative separations. Alternately a narrow strip can be cut from one end (vertically) and developed with a chromogenic agent to find the desired band.

Preparative separations should be applied as a long thin streak rather than a spot. 3 mm paper will carry a larger load than 1 mm thick paper. It will be necessary to use multiple papers separately to obtain any quantity.

[Usually 100 mg or less is considered to be the limit for 1 mm thick paper.]

Once the separation is complete, great care must be taken to ensure that the power supply is off and disconnected to avoid electrocution hazard.

The area of maximum density can be separated from the sheet of the paper by cutting it free with scissors.

Dissolving the target alkaloid in a small amount of solvent such as methanol or methylene chloride and recrystallizing or dissolving in a nonpolar solvent and washing with water should suffice to remove any traces of the buffer.

Ionophoretic Mobility Ratios For Some Indole Bases [from Fish et al. 1956]

Compound	Rm	Cm traveled
Urea (used as a marker)	0.03	3.5 ± 0.3
N,N-DMT-N-oxide	0.00	3.2 ± 0.1
N,N-Dimethyltryptamine	0.93	14.3 ± 0.1
Tryptamine	0.87	13.7 ± 0.3
N-Methyltryptamine	1.00	15.2 ± 0.1
Conditions:		

- 300v / 3ma / for 5 hours with 0.01 M sodium borate buffer solution at pH 9.3 and washed Whatman 1 paper, at room temperature (22-23°).
- Paper electrophoretic separations were used to identify alkaloids. Frahn & Illman cited Frahn & Mills 1964 for a description of the apparatus.
- [See also Frahn & Mills 1959]
- After removal of the solvents [from above] under reduced pressure they dissolved the residue in 0.05M acetic acid and applied 1 μ l to a paper strip for electrophoresis in a sodium carbonate buffer.
- They ran against standard solutions of 0.2M of each respectively.
- They determined that DMT had a mobility of 4.4 cm. h^{-1} . kV^{-1} .
- DMT metho cation had mobility of 1.6 cm. h⁻¹ kV⁻¹. Frahn & Illman 1973

From Erspamer et al. 1967:

- A high voltage electrophoresis apparatus from L. Hormuth, Inh. W.E. Vetter (Heidelberg) was used. It was said to have been run according to Wieland & Pfeiderer.
- They used thick Machery No. 214 paper (Nagel & co., Düren) and pure reference materials, running the compounds separately.

Buffers:

- pH 1.2: glacial Acetic acid-85% Formic acid-distilled Water (15:25:110)
- pH 5.8: 99% Pyridine-glacial Acetic acid-distilled Water (15.5:1.8:135)
- pH 7.9: Triethylamine-distilled Water (5:95) (CO₂ was bubbled through until desired pH was reached)

Relative migration rates (of pure compounds) towards cathode (using serotonin as 1):

Alkaloid	pH 1.2	<u>pH 5.8</u>	<u>pH 7.9</u>
Bufotenine	0.98-1.00	1.00	1.60-1.70
Bufoviridine	0.06-0.16	0.12-0.16	0.10-0.20
5-MeO-DMT	1.05	1.10	1.50-1.65
Dehydro-			
bufotenine	0.96-1.00	0.86-0.90	0.77-0.84
Bufo-			
thionine		0.13-0.18	0.10

Ivor Smith 1969 cited Ivor Smith *et al.* 1967 for: Electrophoresis & electrophoresis chromatography.

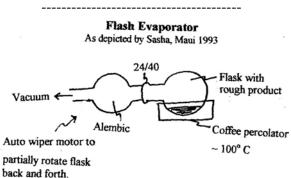
Notes for **Isolation & Separation Section**

1 If one takes a hydronium ion (H[']) and a hydroxide ion (OH⁻) and places them near each other, they will interact to balance each other's imbalance of charge and in doing so will form a molecule of water (HOH, i.e. H₂O)

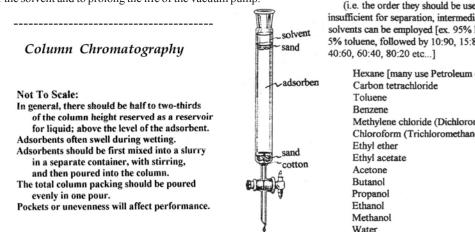
Actually while this is a convenient way of viewing the situation, the truth is a little more complex. A positively charged hydrogen ion is not stable on its own and will not exist that way for long. It therefore joins with water to form the hydronium ion which is more properly expressed H₂O⁺. When this joins with a hydroxyl, two (2) water molecules are actually formed.

2 Usually - I don't want to get too far into the subject of "equivalencies" where some molecules of acid, such as the sulfate, take two molecules of monobasic base to form a neutral salt. All the bases we will deal with are monobasic. It would only be of a crucial nature if you were titrating bases or figuring molecular weight. In the first case it is a subject you would already know, in the second case molecular weight of bases and their salts are readily available and can be simply looked up. We discuss this in somewhat more detail in Sacred Cacti Second Edition.

3 HCl (hydrochloric acid; hydrogen chloride) + NaOH (sodium hydroxide; lye) = H_2O (water) + NaCl (salt; the chloride salt of sodium)



When removing solvents under reduced pressure it is always a very good idea to install a solvent trap in the line; both to recover the solvent and to prolong the life of the vacuum pump.



The above is a generic diagram; your actual apparatus will probably look quite different.

4 Burning to white powder or powderable material. This can be repeatedly dissolved in water, filtered and evaporated to dryness to highly purify.

5 Red paper turns blue with bases/ blue litmus turns red with acids-

"Bases "B" blue/ Acids "R" Red"

It is actually more pink than red.)

6 Phenolphthalein (formerly the active ingredient in Ex-Lax) This is readily available via most wine making supply stores to check pH, and turns deep pink to purplish-red with bases.

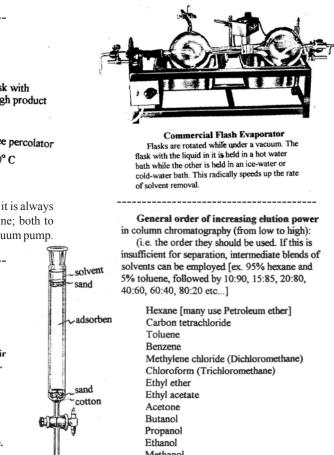
[Purple cabbage cooked in water will also yield a functional but short-lived acid/base indicating solution. This requires a small bit of solution be sacrificed for each pH assessment. Use something white for easy viewing of color.]

7 Despite being rather stable, DMT should be kept from exposure to light and air as much as is possible. This is true if crystalline, an oil or if in solution.

8 It may advantageous to reduce the volume of the solvent to make it easier to handle. Do not allow it to get thick when doing so.

9 This solution should be discarded.

However, for now, LABEL IT, and set it to one side until you are certain that you are entirely finished. It is easy to discard the wrong solutions. It may also be found economical to recover used solvents by distillation and reuse them.



Some Simple Tryptamines

10 Be aware that if petroleum ether is used to defat, and both part of your product is present as DMT-N-oxide **and** fats are simultaneously present in any quantity, much of the DMT-Noxide will be lost with the defatting solution. Similarly if acetic acid is used for acidification and chloroform is the organic solvent, both DMT acetate and DMT-N-oxide acetate will be at least partially present in BOTH the chloroform and in the acid solution. Hydrochloric or sulfuric or citric would all be better choices in conjunction with chloroform. Of course if this is being run as a preparative procedure and analytical quantitative extraction is not an issue, loss just means less product. If a person is operating under crude and primitive conditions and is simply attempting to provide themselves with sacramental material for personal use then, many times, loss of part of the product is an acceptable waste.

11 Filtering the citrus juice before addition will help later filtration.

12 If you decide to use Coleman Fuel at least wash it with water several times before use and discard both the water and the thin emulsion layer. I would recommend not using it unless you have no choice. Petrochemical products sometimes used as rust inhibitors are only partially removed by washing (they will be in the emulsion). Lead salts used for this same purpose can be removed by repeatedly washing with an aqueous acid solution but a better approach in both cases is to choose a different solvent.

13 If this is the source for your ether be certain to wash it with water before using. This is done by gently shaking it together with an equal volume of water, letting them settle and draining and discarding the water and emulsion layer. This should be done two or three times. Ideally the water layer should be clean.

A better approach would be to purify it by distillation but this is a potentially dangerous operation best left to experts. See note 12 concerning rust inhibitors.

If in doubt about the purity of your solvent, choose a different one.

14 A fact that such short sighted regulation ignores is that precursor restriction does not reduce the amount of a drug available, it simply increases the numbers of analogs which hit the streets as other similar reactants are substituted. Many of these find their first pharmacological evaluations in their consumers, some with disastrous results.

Some absurdly potent narcotics in the fentanyl series were actually created as a direct result of such moronic laws. Such precursor laws most seriously affect the otherwise law abiding and do little or nothing against organized criminals.



15 'Inflammable' means it will burn. 'Nonflammable' means it will not. 'Flammable' is a *bastard* word people made up because some people thought 'Inflammable' meant it would not burn. 'Inflammable' means it is capable of becoming 'Inflamed'. 16 Chloroform and methylene chloride are known liver toxins and have been proven to be carcinogens in some animal species and are suspected as such in others. Methylene chloride (DCM) is further known to be metabolized into carbon monoxide within humans; damage to the heart is a real possibility from overexposure and ANY exposure to this solvent should be avoided by people with preexisting heart conditions

As was noted earlier for methanol, industrial grades are also likely to leave plasticizers behind in the residue.

Petrochemical solvents are known to cause damage to the liver, kidneys, spleen, bone marrow and long term or repeated exposure will result in brain and nervous system damage. If you have any doubt about this, just get to know a lifelong house or enamel painter.

17 Prepare by shaking chloroform with concentrated ammonium hydroxide, allowing them to separate and discarding the aqueous ammonia layer.





Acacia maidenii flowers, buds & ripe fruit

Acacia phlebophylla phyllodes Photo by Mulga

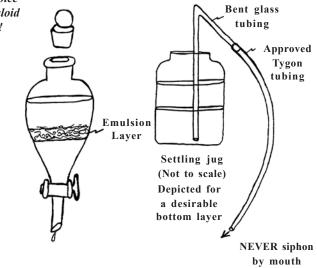
Separation

Hexane Heptane Petroleum ether Coleman fuel Xylene Toluene Benzene Ethyl acetate Ether Whether you want the top or bottom layer will depend on both the solvent choice and whether your alkaloid is a salt or free base!

ALLFLOAT

WATER [Acetone & some small alcohols MIX with water]

Methylene chloride (DCM) Chloroform Carbon tetrachloride Chlorinated hydrocarbons Most dry-cleaning solvents ALL SINK Settling jug could also have a 2-hole stopper sealing the top and a short second tube used to blow into (for starting the siphon action)





A convenient way to hold a separatory funnel during manipulations

Separatory funnel tips

Be sure drain is closed before adding liquid. To vent & prevent pressure buildup during neutralization: periodically invert & release gas using stopcock while drain is pointed upwards. Use of a ringstand and a clamp are highly recommended while waiting for separation. Careful prolonged mixing will produce less emulsion than will shorter vigorous shaking. Liquids can not effectively drain out of the stopcock unless the stopper is removed first. Drain off a bottom layer; decant a top layer. In a pinch; a large glass pipette (50, 100 or 250 ml) equipped with a rubber bulb can also work just fine



Separatory Funnels
Photos thanks to our friends at UT Austin

What's wrong with the lower right picture?

A Simple Alkaloid Volatizer

It was been brought to our attention that our high court has decided it is against the law to sell any "obviously non-tobacco" smoking paraphernalia.

Ignoring for the moment the inherent conflict of interest and drug monopoly involved, we are disturbed at inherent potential limitations on personal religious and spiritual freedoms.

In response, we would like to offer the following:

The "volatizer" can be a **large** (8 inch) Pyrex test tube or **small** flask of Pyrex. (Basically anything with a neck that can have a sample placed in it and is capable of being stoppered and heated from beneath.).

It will need a two holed rubber stopper (cork **can** work if necessary), a #4 stopper in the case of an 8 inch test tube, and two short pieces of 1/4" O.D. heavy wall glass tubing.

One, 6 to 8 inch straight piece, will extend through the stopper and go about three quarters of the way to the bottom of the flask or test tube. This will serve as the air INTAKE.

The other 6 to 8 inch piece should be slightly bent (somewhere between 30 and 60 degrees) near one end. The end of the tube nearest the bend should be inserted into the rubber stopper so that it protrudes slightly out the bottom of the stopper (the long portion staying above the stopper). This is the stem of the volatizer (mouthpiece).

When one inserts glass tubing through rubber stoppers, wet the outside of the tubing with water first. Then, being certain to grasp the tubing very close to the end being inserted, gradually feed the tube through the stopper with a gentle rotating or twisting motion. It will slip through quickly and easily. Holding the tubing any distance from the stopper and attempting to simply push it through can cause very serious puncture wounds if the tubing breaks.

The straight piece should be inserted first and the stem second.

A major problem with DMT pipes is the accumulation of DMT condensing in the stem over time. Eventually it gets in one's mouth during inhalation. DMT has a burning, strongly basic taste so this is not pleasant. This pipe can be readily disassembled to have the stem cleaned without losing the accumulated condensate on the vessel walls. (The material cleaned out of the stem can be recovered if one so desires.) Ethyl alcohol (95% or 190 proof; i.e., Everclear or other brands of pure grain alcohol) or ethyl alcohol with about 30%, by volume, of 5-9% vinegar added or IPA can be used to rinse (or soak) the stem clean. Warming will speed the process. If pure alcohol is used it can simply be evaporated in the test tube itself for recovery. If acidified alcohol is used it should be processed via removal of the alcohol, suspension of the residue in acidified water, basification and solvent partition as dealt with elsewhere

Once the glass tubes are in place and the device assembled to ensure a good fit, the stopper should be removed and everything allowed to dry before use. After careful measuring of the dosage, the sample is placed in the bottom of the test tube (or flask) and the stopper replaced.

In the case of DMT, 15-30 mg is well liked by most novices, (1 to 2 match-head sized portions) and 50 mg. is not. (For 5-MeO-DMT; divide numbers by three.)

The test tube (or flask) is now gently heated with an alcohol lamp or other sustainable flame until the sample melts, boils up, collapses forming a mist and then begins to fill the chamber with smoke. [A microtorch will volatize it much faster.]

Only when the chamber begins to fill with smoke should the inhalation begin. Beginning to inhale while the mist is still present wastes both lung space and material as a lot will condense on the cooler sections of the glass walls.

One needs to inhale slowly and fully (like a long sip through a very small straw); trying to get every bit of the harsh smoke into the lungs. (It actually has a pleasant taste for something so reminiscent of burning plastic or mothballs.) If there is more than can be taken as one hit, remove heat when halfway through the inhalation as the heat retained by the glass will keep making smoke for a short while. Overheating rapidly destroys the material; producing a foul taste and nitrogen oxides.

If one has done it correctly there will be no need to take a second hit or to hold it in for more than a few seconds (DMT absorbs rapidly). Not everyone has good breath control. If one doesn't get "in" with one hit, they can (and should) try again rapidly.

T. McKenna recommended simply taking three rapid lungfulls of DMT in quick succession when smoking DMT. (This always works despite the amount wasted.)

If a couple of rapid hits don't do it or if more than a minute or two have elapsed since first beginning to inhale, we would suggest not smoking any more for around an hour as this can cause a waste of material without optimum results. Best results come from allowing a day to a week between doses but access can be achieved with less than an hour between doses (even less with 5-MeO).

See more comments below under "Tolerance"

Nausea and somatic distress may become progressively more pronounced each time DMT is repeated; often exceedingly so if administered more frequently than recommended above.

Have a surface such as a folded-up towel, hot pad or piece of wood handy for placing the pipe on. If hot glass is placed on a metal, tile or other cool surface it may cause it to break. Another approach is to use a clamp and a ring stand.

This is such a simple idea and parts so easy to find that we can't imagine that it hasn't been thought of it before. If we, somehow, are the first; we freely give this idea over into the public domain and grant permission for anyone to use this idea in any way that serves their purposes so long as no copyright is attempted and we are held blameless for the outcome of their actions. We do not advocate the breaking of laws but rather a redress and repeal of the presently unjust laws.

Explore these inner pharmacodimensions with care. For those who have never experienced DMT or 5-MeO-DMT, read everything that you can before proceeding.

Make sure this is done in a safe and protected spot. Be aware that you may be incapacitated and usually unable to interact with your surroundings during the peak. (You may not even be aware of them.)

These are not recreational drugs. Their use must not be taken casually or lightly. People who attempt to use large amounts will often never try the alkaloid again.

This is not an experience for everyone.

Many individuals would be much happier if not going at all.

In one study evaluating the effects of DMT only 17 out of 40 entheogen experienced volunteers were willing to repeat the experience.

These points cannot be stressed enough. Read everything you can BEFORE beginning.



Above: Heat sources Alcohol lamp (L); Butane torch (R) Below: An "oil" pipe; popular & functional but not ideal Photos by friends



A couple of comments about smoking DMT (or 5-MeO-DMT)

Smoking DMT is not like smoking pot. You do not want to puff, mix the smoke with air, try to get it 'lit' (even if it is on pot) nor do you want to try and hold in the smoke for long periods. The idea is not one of attaining a chosen level by gauging it as you go. It is one of administering an effective dosage by packing as much into the lungs as fast as is possible.

The idea is that one can either cross the line or not cross it. The two are very different states. If you do not cross the line, you stay here, in an extremely altered, intensely colorful and entheogenic state, but conscious of all somatic distress. The ideal is to reach a state where you are no longer aware of your body.

The idea is to get enough DMT into your system to enable you to break through and make the trip before the brief-lived "tolerance" (see p. 231) can build up and prevent full access. Within a very short time, a person will be physically incapable of anything. Get the most possible into your system in as short of a time as possible. The shorter the time of administration is, the more effective the experience.

Even larger amounts taken more slowly can have less effects. A rapid series of several deep inhalations, none but the last being held in for more than a few seconds, is the most efficient way for most people to experience full effects. (As mentioned , a slow, deep, full lungfull can accomplish the same thing if breath control is used.)

If larger amounts are smoked by holding in the hits and breathing in between (like when smoking DMT on pot) the subsequent experience lasts longer but can never quite reach the same peaks of a full blown DMT adventure.

Both have their own unique vantage points.

Smoking tryptamines on herbs also enables them to be used in places where smoking a free-base pipe is not feasible and to regulate lower dosage levels.

We actually would recommend suspension on an herb like mullein as a good approach for dealing with 5-MeO-DMT. If a fairly large amount is administered repeatedly over a period of a some minutes (smoking it suspended on pot) it can positively break some reality barriers without the overwhelming distress of smoking the same amount all at once.

Smoking it with pot does cause a marked enhancement of the visual field with eyes closed but 5-MeO-DMT does not cause the appearance of 'patterns-and-colors' or any of the striking visuals associated with DMT (except at high dosages).

On the other hand, auditory and visual TRUE hallucinations have not been uncommon for us with 5-MeO. A true hallucination being one that appears and seems to be something that is really there with the cognitive senses seemingly clear. [Thought real at the time; at least momentarily] In our experience the most likely appearance of such hallucinations visually is accompanying or just after the **return** to (seemingly) normal consciousness.

We have often wondered if its difference from DMT is due to its similarity to serotonin. In serotonin the side chain is close to being in the same generalized plane as the rest of the molecule. Similarly with 5-MeO-DMT. With DMT, however, the side chain approaches a 90° angle vertical from the generalized plane of the indole.)

When heating DMT, care should be taken not to heat it too rapidly or to overheat it. It will take on a foul taste and may change the character of the experience. Heating to decomposition produces toxic nitrogen oxides (brown smoke) so it is not a good idea to smoke the charred remains.

Another point that is extremely important:

When smoking DMT do not try to see how far it can take you (unless well experienced).

If you do not already know this, DMT can, and often will, take you farther than you can possibly imagine. The use of large dosages will not physically hurt someone but many times, it will be the last time that person EVER does DMT.

Numerous users have been reported to stop using any and all drugs entirely after experiencing too large of a dose of DMT, DET, DPT or similar tryptamines.

The intensity is quite overwhelming at normal dosages, it is suggested that a person first familiarize themselves with effective levels before attempting to 'turn up the volume'. (DMT does not have a linear volume setting.)

5-MeO-DMT (or DPT) can be physically and overwhelmingly distressing if the optimal dosage is exceeded.

Despite the fact that DMT, 5-MeO-DMT & Bufotenine have all been shown to occur in humans naturally, these drugs demand respect.

This cannot be stressed enough.

Perhaps results from an animal study might be helpful for the uninitiated:

Using a "mongrel" dog.

At 5 mg/kg iv- panting and muscular rigidity began before the needle was withdrawn. The dog was howling and baying within 1 minute.

"It assumed a spread-eagle stance, with its abdomen pressed to the floor, and resisted efforts to disturb its equilibrium. The hair did not stand erect. Pulse and respiration were rapid, pupils dilated and eyes were open but the animal did not appear to see." Urination and defecation occurred immediately after the injection. The symptoms became less severe after an hour and "the dog howled only occasionally". The dog was weak but apparently normal two hours later.

"In monkeys doses up to 36 mg/ kg intravenously caused clonic spasms followed by loss of equilibrium, erection of hair, mild ptyalism, loss of perception with no loss of consciousness. A dose of 53 mg/ kg [iv] was fatal."

Heinzelman & Szmuszkovicz 1963 cited unpublished results of W.A. Freyburger & B.E. Graham at the Upjohn Company.

Other ingestion methods (applicable to the tryptamines) are discussed in Lazar 2001: including insufflation (snuffing), use of nasal sprays and smoking the free base alkaloids.

Snuffing should not be done using purified free base tryptamine alkaloids due to the basic pH.

Conversion of a salt to the free base:

Alkaloid salt was dissolved in water (using heat if necessary but, if so, using only the minimal amount required).

The solution was placed in a separatory funnel; ammonia was then added dropwise until pH 8-9 was reached.

At this point chloroform was added, swirled and thoroughly shaken for 5-10 minutes.

Once the layers separated, the chloroform was removed. Fresh chloroform was added and the shaking repeated.

(Adding more ammonia as required to keep the pH at 8-9.) Chloroform extracts were combined and evaporated.

The oil remaining was thoroughly mixed with dried parslev and stored in an airtight container.

A different organic solvent than chloroform could have been used and/or the chloroform evaporated, the free base dissolved in a bit of alcohol & added to the herb.

Above was taken from Toad's comments in MS 2001.

Use of a strong sodium bicarbonate solution and toluene works fine too!

Making a smoking mixture:

For easier & precise dosaging, the oil could have been weighed and then placed on weighed herbal material.

This works great for crystalline material as well.

The freebase should be placed in a small glass bowl and then dissolved in the least amount of 95% ethanol possible. (Warming it with a bowl of hot water if required)

99% Isopropanol also works fine.

For a 20% smoking mixture: 8 parts of dried, cleaned herb would be added to every 2 parts of alkaloid. (800 mg of herb for 200 mg of freebase alkaloid or 4 gm of herb for every 1 gram of freebase alkaloid)

The mixture should be allowed to evaporate without heat and repeatedly stirred during drying.

If using a 20% smoking mixture, a 10 mg dose could be achieved by smoking 50 mg of herb. [If 50%: 25 mg/50 mg.]

Some use *Cannabis* but for a clearer experience one can employ parsley, or mullein, or another nontoxic herb.

A nice approach some use is having pure shredded *caapi* form the substrate for their smoking blend.

Friends in Oz utilize an extremely nice blend using *Banisteriopsis caapi* leaf and *Acacia obtusifolia* isolate and suggest that 70:30 is better than 50:50.

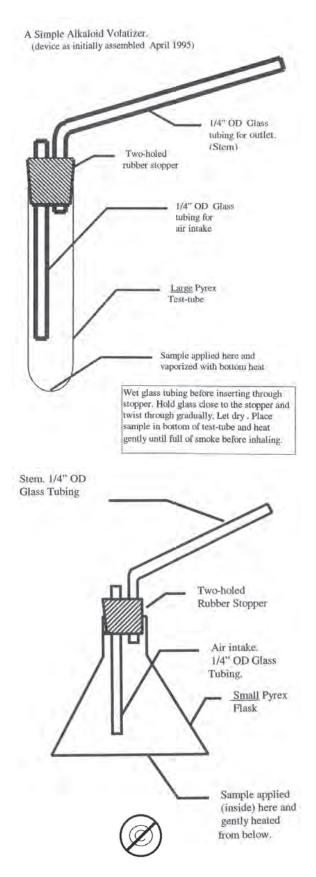
Justin Case's smoking blend:

A dry herbal mix is made from combining 4 parts of *mullein*, 2 parts *raspberry* leaf, 2 parts *spearmint* leaf and 1 part shredded *caapi* vine, 1 part shredded *caapi* leaf. An optional addition is 2 parts *damiana* leaf.

DMT is added to the herb mix as was mentioned above. Use one gram of herbal mix to one gram of DMT.

It is suggested that this be used to create joints approximately 1 gram in size rolled from extra long papers and using a rolled cardboard "backstop" inserted as the mouth piece. This can help prevent liquefying DMT from contacting the lips during smoking.

One good lungfull is often all that is required for, or desired by, some people but the undaunted might prefer taking 2 or 3 in rapid succession before passing the joint.



Volatizers are copyright-free designs

The Machine is a modified crack pipe produced by an Australian friend. It can deliver more volatized alkaloid plus seems gentler on the lungs than the other base or oil pipes evaluated sofar.

It uses a bit of extremely coarse steel scrubber as is used for scrubbing pans (rinse any rust inhibitor clean with acetone or alcohol and dry before use); this is stuffed into a small bottle with a neck. [Avoid steel wool as it can ignite and burn.] Too small of a neck will restrict airflow adversely.

A single serving cognac bottle works great. (Some even have a handy preformed weak spot in the bottom)

Repeated tapping with scissors or a knife tip can soon generate a small hole in the bottom. (See inset) (A diamond drill works great as well) Any broken glass bits should be washed clean with running water.

This hole will serve as the mouthpiece.

To use: place the material on top of the steel turnings. Gently heat it until it soaks into the opening (as shown here). Once this is done, the neck is pointed downwards and heated with a lighter while a slow but steady hit is taken through the hole in the bottom. Do not overheat the steel. (Replace it often.) One slow full lungfull is all it takes for most people. See Note H on page 227



Other devices for smoking free base alkaloids:

See Hempburn's 'hot knives" article on page 230



More free-base smoking devices (Top to Bottom):

A 'crack' pipe similar to the Machine (bulbous end is the mouthpiece)

A 'classic' DMT pipe.

An 'economy vaporizer'



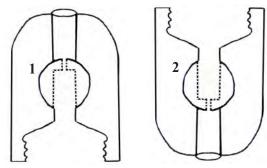
A glass light bulb with inner parts removed and cleaned well is classic old school. A glass tube (not plastic) should be used to inhale the smoke. A small glass bottle with a bit of wire twisted around the neck for a handle works too. Material is placed inside and heated from underneath. Smoke is sucked off through the glass tube as it is formed.



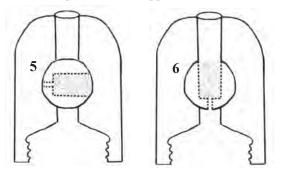


A useful device for tryptamine salts:

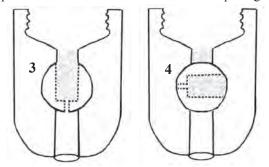
A 'bullet'. A device measuring out small aliquots of powdered material (inside of a cavity in the valve) permitting easy measurement for insufflation. In this model the handle points in the same direction as the valve opening.



turned upside down & tapped to fill chamber



bullet is inverted & valve rotated to expose the charge



valve turned to center position to secure contents

Contents are insufflated up one nostril to clear chamber of the dose.

'Bullet' device screws on to the top of standard small glass vials. It disassembles for easy cleaning. Remember different sized chambers OR densities provide different doses. Know your tools!





Jurema preta rootbark (Brazil)



Desmanthus leptolobus roots (Manor, Texas, USA)

Psychotria viridis [Note the 'espinas'] Photo by Mulga

> Various sources of N,N-dimethylated tryptamines





Diplopterys cabrerana leaves (Ecuador)



Mimosa tenuiflora rootbark (Chiapas, Mexico)

Useful properties of some solvents commonly used in chromatography

		L			· ··· · · · ·]	
Solvent [Order for elut (Note 2)]	Polarity index ion	Dielectric constant (Note 1)	Solubility in water % w/w	Boiling point (C)	Flash point (C)	LD_{50} or Lethal concentrations for mice or rats in air (ppm)
Acetic acid	6.2	6.15	100	118°	None	Causes skin burns
Acetone [10]	5.1	20.7	100	56.5°	-20°	10.7 ml/ kg/ oral / rat
Acetonitrile	5.8	37.5 [38.8]	100	81.6°	12.8°	3.8 gm/ kg/ oral / rat
Benzene [5]	2.7	2.284	0.18 (Note 3)	80.1°	10-12°	3.8 ml/ kg/ oral / rat
<i>n</i> -Butanol [11]	3.9	17.8	7.81 (Note 4)	117-118°	36-38°	4.36 gm/ kg/ oral / rat
Carbon tetrachloride [3]	1.6	2.238	0.08 (Note 5)	76.7	None	~10,000 ppm Known liver toxin (Note 6)
Chloroform [7]	4.1	4.8	0.815 (Note 7)	61-62°	None	Known liver toxin (Note 8)
Cyclohexane	0.2	2.023	0.01	80.7°	-18°	60-70 mg/liter of air in mice
Diethylamine	na	na	100	56.3° [55.5°]	<-6.7°	540 mg/ kg/ oral/ rats
Diethyl ether [8]	2.8	4.335 (9.)	6.89 (Note 10)	34.6°	-45° (Note 11)	
Dimethylamin (anhydrous)	e na	na	100	6.88° (Note 12)	0°	4.0 gm/ kg/ iv/ rabbit
Dimethyl sulfoxide	7.2	4.7 [4.5]	100	189°	95°	>20 gm/ kg/ oral / rat
Dimethyl- formamide	na	6.4	100	155° [153°]	67°	1122 mg/ kg/ oral / mouse (Note 13)
Dioxane	4.8	2.209	100	101.1°	5-18°	6.0 gm/ kg/ oral / rat 5000 ppm Known human toxin (Note 14)
Ethanol [13]	5.2	24.30	100	78.5°	9-11°	13.7 gm/ kg/ oral / rat
Ethyl acetate [9]	4.4	6.02	8.7 (Note 15)	77°	7.2°	11.3 gm/ kg/ oral / rat
Heptane	0.0	-	0.0003	98.4°	-1°	15,900 ppm

[Bracketed values are from Merck Index or as noted]

Solvent properties continued:

Solvent [Order for elut [(Note 2)]	Polarity index ion]	Dielectric constant (Note 1)	Solubility in water % w/w	Boiling point (C)	Flash point (C)	LD ₅₀ or Lethal concentrations for mice or rats in air (ppm)
Hexane [2]	0.0	1.890	0.001	69°	na	40,000 ppm
Methyl ethyl- ketone	4.7	18.5	24 (Note 16)	79.6°	1.67°	6.86 ml/ kg/ oral / rat
Methanol [14]	5.1	32.63 (25°) 33.62 (20°)	100	64.7°	12°	< 30 ml can kill human Usual fatal human dose: 100-250 ml. (Note 17)
Methylene chloride [6]	3.1	9.08	1.6 (18.)	39.75°	None	1.6 ml/ kg/ oral / rat Liver toxin (Note 19)
<i>i</i> -Propanol [12]	3.9	18.2	100	82.5°	11.7°	5.8 gm/ kg/ oral / rat 100 ml can be fatal to humans
n-Propanol	4.0	20.1	100	92° [97.2°]	22°	1.87 gm/ kg/ oral / rat
Pentane [1]	0.0	1.844	0.004 (0.36 gm/l; 16°C	36° C)	-40°	128,200 ppm
Pyridine		12.3	100	115.5°		4000 ppm. Liver toxin
Tetrahydrofura	n 4.0	na	100	65°	1°	Known human toxin
Toluene [4]	2.4	2.379	0.51	[66° Merck; 110.6°	6-10°	7.53 ml/ kg/ oral / rat
Water [15]	9.0	80.37 (20°) 78.54 (25°)	100	100°	None	None
Xylene	2.5	p = 2.27; m = 2.374; o = 2.568 (Note 20)	0.018	137-140° p-: 137-138; m-: 139.3; o-: 144 (N	29°	Unclear. Thought a little bit less toxic than benzene

Please note that ALL solvents are toxic (except perhaps for water) Many, if not most, of the solvents listed above can seriously damage the brain, central nervous system, liver, kidneys, spleen, bone marrow and other vital organs



with either prolonged or repeated exposures. Many are suspected or proven carcinogens in laboratory animals. Eye, skin & respiratory protection are absolutely required to help minimize the risk of serious injury or damage

synthetic DMT

Notes for Solvent properties table

1 All at 20°C, except for acetone, benzene, dioxane, ethanol, ethyl acetate and both propanols, which were measured at 25° C

2 Please note:

This order of increasing elution power is not agreed upon in all texts nor recommended in all applications.

In general, they should be used in the order of increasing polarity.

However, the specific type and form of the column packing material of choice can affect the order that is used.

3 1 ml is soluble in 1430 parts water

- 4 9.1 ml will dissolve in 100 ml of water at 25°
- 5 1 ml is soluble in 2000 parts water
- **6** Known to cause human liver & kidney damage. Confirmed carcinogen in animals.

There is one known case that a human was reported to develop a liver tumor after acute exposure.

To quote SAX "respiratory or dermal exposure should be avoided by all means"

7 1 ml is soluble in 200 parts water; 25°C

8 Known toxic action on heart, liver & kidneys.

Suspected, as with similar chlorinated solvents, of being a liver carcinogen. (Proven as such in some but not all animals. Produces birth defects in chickens.)

Humans metabolize methylene chloride into *carbon monoxide* so anyone with heart problems should take pains to avoid *ANY* exposure to this solvent.

9 4.197 at 26.9°C

10 Ether can dissolve up to 1.2% water at 20°C; Water can dissolve up to 8.43% at 15°; 6.05% at 25°.

HCl increases water solubility.

11 Shaken under totally dry conditions, ether can generate enough static electricity to start a fire



Acacia cultriformis (cultivated as ornamental in Austin, Texas)

12 Gas at room temperature.

Normally marketed as a compressed liquid or else as a 33% aqueous solution

13 Prolonged exposure to as low as 100 ppm has been shown to produce liver injury

14 Human deaths via industrial inhalation have resulted due to liver and kidney necrosis

15 1 ml dissolve in 10 ml of water at 25°.

More soluble at lower temperatures and less soluble at higher temperatures.

16 1 part is soluble in \sim 4 parts water; less soluble at higher temperatures

17 Chronic, or acute, exposure can cause brain damage, visual impairment or blindness

(Injury may be permanent)

18 1 part is soluble in around 50 part water

19 Also known to attack the eyes & nervous system

20 Commercial xylene (Xylol) is a mixture of three isomers (*p*-, *o*- and *m*-xylene) with *m*-xylene predominating.

21 *p*-Xylene 137-138°; *m*-Xylene 139.3°; *o*-Xylene 144°



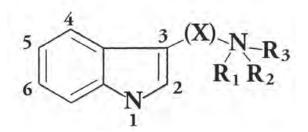
Trout's Notes on Tryptamines



Acacia obtusifolia (NSW, Australia)

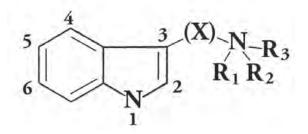


Diplopterys cabrerana



Indolic Structural table

	Aromatic		(Indole) N		-(X)-	(Amine) N				
Position:	4	5	6	1		R1	R2	R3+		
Name										
[Page#] Gramine	Н	Н	Н	Н	-CH ₂ -	-CH,	-CH,	na		
[11]	11	11	11	11	-0112-	-CII ₂	-0112	Πa		
Tryptamine	Н	Н	Н	Н	$-CH_2CH_2$ -	Н	Н	na		
[13]										
N-Methyltryptamine (MMT)	Н	Н	Н	Н	-CH,CH,-	-CH,	Н	na		
[17]					2 2	3				
N,N-Dimethyltryptamine										
(DMT) [22]	Н	Н	Н	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	na		
DMT-N-oxide	Н	Н	Н	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	-0		
[38]										
N,N-Diethyltryptami (DET)	ne H	Н	Н	Н	-CH,CH,-	-CH,H,	-CH,H,	na		
[41]						0112115	0112115	nu		
N,N-Dipropyltryptam					~~~~					
(DPT) [45]	Н	Н	Н	Н	-CH ₂ CH ₂ -	$-CH_{3}H_{7}$	$-CH_{3}H_{7}$	na		
Lespedamine	Н	Н	Н	H ₃ CO-	-CH,CH,-	-CH ₃	-CH ₃	na		
[47]				5	2 2	2	2			
4-Hydroxyindole [53]	HO-	Н	Н	Н	Н	na	na	na		
4-Acetoxyindole	AcO-	Н	Н	Н	Н	na	na	na		
[53]										
4-Hydroxytryptamin	е НО-	Н	Н	Н	CUCU	Н	Н			
(4HT) [54]	п0-	п	п	П	-CH ₂ CH ₂ -	П	п	na		
Norbaeocystin	O ₄ PO-	Н	Н	Н	-CH ₂ CH ₂ -	Н	Н	na		
[55] 4 OH MMT	ЦО	ш	TT.	TT	CUCU	CII	TT			
4-OH-MMT [55]	HO-	Н	Н	Н	-CH ₂ CH ₂ -	-CH ₃	Н	na		
Baeocystin	O ₄ PO-	Н	Н	Н	-CH ₂ CH ₂ -	-CH ₃	Н	na		
[56]										
Psilocin (4-Hydroxy-DMT)	HO-	Н	Н	Н	-CH,CH,-	-CH ₃	-CH ₃	na		
[59]	110					0113	0113	nu		
4-Methoxy-DMT	H ₃ CO-	Н	Н	Н	-CH ₂ CH2-	-CH ₃	-CH ₃	na		
[62] 4-Acetoxy-DMT	AcO-	Н	Н	Н	-CH ₂ CH ₂ -	-CH ₃	-CH,	na		
[64]	100-	11	11	11	C11 ₂ C11 ₂ -	U11 ₃	C113	nu		
1-Methyl-Psilocin	HO-	Н	Н	H_3C -s	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	na		
[64] Psilocybin	O PO-	Н	Н	Н	-CH,CH,-	СН	СН	n 9		
[65]	0 ₄ r0-	11	11	11	-CII ₂ CII ₂ -	-CH ₃	-CH ₃	na		
1-Methyl-Psilocybin	O ₄ PO-	Н	Н	H ₃ C-	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	na		
[88]										





Indolic Structural table

	Aromatic		(Indole) N		-(X)-	(Alkylamine) N		
Position:	4	5	6	1		R1	R2	R3+
Name [Page#]								
CZ-74 (4-Hydroxy-DET)	HO-	Н	Н	Н	-CH ₂ CH ₂ -	$-C_2H_5$	$-C_2H_5$	na
[88] 4-Acetoxy-DET [90]	AcO-	Н	Н	Н	-CH ₂ CH ₂ -	$-C_2H_5$	$-C_2H_5$	na
CEY-19 [91]	O ₄ PO-	Н	Н	Н	-CH ₂ CH ₂ -	$-C_2H_5$	$-C_2H_5$	na
5-Br-Tryptamine [95]	Н	Br	Н	Н	-CH ₂ CH ₂ -	Н	Н	na
5,6-DiBr-Tryptamine [95]		Br	Br	Н	-CH ₂ CH ₂ -	Н	Н	na
5-Br-MMT [<i>na</i>]	Н	Br	Η	Н	-CH ₂ CH ₂ -	-CH ₃	Н	na
5,6-DiBr-MMT [95]	Н	Br	Br	Н	-CH ₂ CH ₂ -	-CH ₃	Н	na
5-Br-DMT [95]	Н	Br	Н	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	na
5,6-DiBr-DMT [96]	Н	Br	Br	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	na
Serotonin [97]	Н	HO-	Η	Н	-CH ₂ CH ₂ -	Н	Н	na
N-Methylserotonin [102]	Н	HO-	Η	Н	-CH ₂ CH ₂ -	-CH ₃	Н	na
Bufotenine [103]	Н	HO-	Н	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	na
Bufotenine-N-oxide [111]	Н	HO-	Η	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	-0
Bufotenidine [112]	Н	-O-	Η	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	-CH ₃ ⁺
Bufoviridine [114]	Н	O ₃ SO-	Н	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	na
Bufothionine [114]	Н	O ₃ SO-	Н	Н	-CH ₂ CH ₂ - (N-bound to C#4)	-CH ₃	-CH ₃	—C#4
Dehydrobufotenine [115]	Н	·O-	Н	Н	-CH ₂ CH ₂ - (N-bound to C#4)	-CH ₃	-CH ₃	—C#4
5-Methoxytryptamin [116]		H ₃ CO-	Η	Н	-CH ₂ CH ₂ -	Н	Н	na
5-Methoxy-MMT [118]	Н	H ₃ CO-	Η	Н	-CH ₂ CH ₂ -	-CH ₃	Н	na
5-Methoxy-DMT [121]	Н	H ₃ CO-	Н	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	na
5-Methoxy-DMT-N-02 [135]	Н	H ₃ CO-	Н	Н	-CH ₂ CH ₂ -	-CH ₃	-CH ₃	—0
5-Methoxy-nordehyd [<i>na</i>]	robufote H	nine H ₃ CO-	Н	Н	-CH ₂ CH ₂ - (N-bound to C#4)	-CH ₃	Н	—C#4

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Notes & Commentary

Note A (from page 72)

The UT subscription for *Comptes Rendus* stopped prior to his other papers so we have no further information at the moment except from the work with Robbers *et al.* 1969 & his identification comments under *subbalteatus*.

Note B (from page 72)

A quite odd, though fascinating, account of some truly 'experimental medicine', can be found in Stein 1959 who was treating a patient for "homosexual thoughts" This was said to be "cured", with such thoughts only slightly reasserting themselves, by prescribing a combination of Amphetamine, Amytal, Iproniazid & Reserpine. At this point, the patient was given 1.5 gm of Panaeolus venenosus which produced "a strong, favorable and euphoriant effect." He was later given 1.4 grams of Psilocybe caerulescens but reported it was "different, relatively hallucinogenic, and much less pleasant." (I bet that it was; considering the multiple drug interactions that would have been occurring!) Stein found P. venenosus clearly different from Psilocybe and mainly stimulant/euphoriant.

Stein *et al.* 1959 gave both fungi to associates; concluding the *Panaeolus* was more pleasant and relaxed in feeling but *Psilocybe* caused disorganization "*associated with a feeling of panic and disagreeable intoxication.*"

In summary, Stein noted (after commenting on his "considerable experience with psychoneuropharmacological (psychoneurotropic) material") that "it is unwise and perhaps unnecessary to try to correlate clinical observations where unknown chemicals or chemical complexes are ingested" and "Some of the effects observed here ... with Psilocybe are probably due to the psilocybin already isolated from Psilocybe mexicana ... which probably also obtains in Psilocybe caerulescens." Apparently based on its similarity to serotonin, he concluded: "However it seems unlikely that psilocybin will be found to have caused the signs of mydriasis or cerebral or cortical intoxication. Probably several chemical compounds will be found to be psychoneurophysiologically effective in each significant mushroom. In addition, undoubtable, the relative differences in the biochemistry which obtains from individual to individual may account further for differences elicited, alleged or reported, including extrasensory perception and hallucinosis, in situations where so-called "sacred" mushroom material has been ingested."

Stein 1960 discusses all of this a bit more and notes that the decision to administer mushrooms to his patient under treatment was voluntary on the part of his patient and motivated both by his concern that "*positive heterosexual thoughts were reasserting themselves only slightly*" and the fact that his patient was about to return home due to an expiring visa. It's VERY important to remember that his patient was STILL taking his multiple drug combo at the time of both 'shroom administrations. Stein 1960 mentioned observing no PSOP (4 spots were observed at higher Rf). Interestingly also noting that what appeared to be the main active compound had been isolated and crystallized. They suspected it to be a 4-oxygenated indole but apparently had not characterized it.

P. venenosus was also noted as hallucinogenic (in Japan) but again said to different from *Psilocybe* by Romagnesi 1964.



dried Psilocybe subaeruginosa

Note C (from page 78)

A study published in Beck 1998 suggests that the "*adverse reactions with typical tachycardia*" sometimes reported with *Psilocybe semilanceata* consumption did not appear to be the result of the PSOP present. They proposed that the highly variable phenethylamine content might play a role. See comments on page 231. They reported that the phenethylamine content was more variable than the psilocybin content.



Psilocybe cyanofibrillosa

Note D (from page 81)

Bluing of shrooms and/or psilocin has been studied by: Blaschko & Levine 1960a & 1960b

Bocks 1967 & 1968 [The latter reported that an artificially produced blue compound (psilocin oxidized with laccase) showed UV absorbance at 610 & 400 nm and that this was similar to the natural blue colorant.]

Gartz 1985d

Gilmour & O'Brien 1967

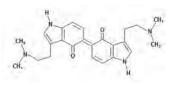
Horita & Weber 1961a

Levine 1967

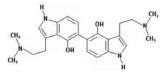
Perkal 1981

Weber & Horita 1963

- The actual blue material in pshrooms has *never* been structurally identified despite repeated study of psilocin's oxidation. (Psilocybin does **not** turn blue.)
- **theobromus** (pers. comm. 2001) has proposed the following two **hypothetical** structures for the blue ("*psindigo*") and colorless ("*leucopsindigo*") forms but structural work for the actual blue compound and the reduced form has **NOT** yet been performed.

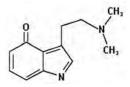


Psindigo

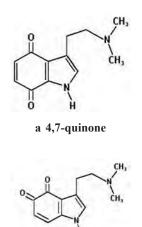


Leucopsindigo

Other proposed hypothetical structures have included:



a keto-imine



or (the more likely of these three) "psilocin-chrome" (a 4,5-quinone)

- or else some dimer or small polymer of one of the above. (Note that theo's proposed structures are both dimers of the 4,5-quinone.)
- Perkal 1981 commented that when the blue pigment was deliberately produced it could go on to form a brown polymer that he suspected was some type of melanin.

Note E (from page 81)

There is an extensive summary in Guzmán *et al.* 2000 (1998) with a current synonym list but it is important to remain aware that the listing of the "active" species, as presented in this article, does not differentiate between:

1) species which have actually had an analysis done,

2) species which still have their analysis pending,

3) species which lack analysis but which have successful human bioassays reported,

4) species which are simply bluing species and the presence of psilocybin/psilocin therefore inferred,

5) species which contain some type or types of active materials other than tryptamines.

For a downloadable .pdf version of this illustrated article see: http://www.museocivico.rovereto.tn.it/MCRPubblicazioni/ Annali/annali-14 htm

Note F (from page 90)

An Erowid-published analysis (contributed by Murple) concerning the brown goo resulting from the oxidation of 4-AcO-DET surprisingly reported that the largest discernable component was 4-Hydroxy-DET with substantial amounts of 4-AcO-DET and minor amounts of unidentifed compounds. Identity of the actual brown pigment (polymeric?) was not attempted.

Note G (from page 68)

Additional Comments on Psilocybin/Psilocin Extraction & Isolation

Comments on various isolation & purification approaches published in the literature can be found under the entries for PSOP and PSOH.

Psilocin and Psilocybin show different solubilities.

- Kysilka & Wurst 1990 reported an increase in yield when attempting to recover one or the other.
- For extracting PSOP used a 10 minute extraction time (homogenized then shaken) in 75% Methanol saturated with potassium nitrate.]

For PSOH they used 75% aqueous-Ethanol & 160 minutes extraction time (homogenized & shaken)

Use of the first approach gave only 8% of the yield of PSOH then the second. (PSOP recoveries were only slightly higher) Gartz on the other hand seemingly claimed rather opposite results (see more details below on page 229). See also comments concerning Stijve & de Meijer 1993

An interesting isolation appearing on the Internet and raved about by a friend:

- "1. Dry mushrooms and turn into powder (a blender works fine).
- 2. Extract the mushroom powder by soaking overnight in 140 proof or 70 percent alcohol.
- 2. Filter the liquid as finely as possible (ideally using $0.2 \ \mu m$ syringe filters or vacuum filtration).
- 3. Add a few drops of hydrochloric acid (aim at pH3).
- 3. Evaporate solution to 1/10 of its volume.
- 4 Remove undesired components (fats & resins) with the following solvents. First use Petroleum ether (twice) [i.e. a solvent which is not miscible with water (cigarette lighter fluid, paint thinner, naphtha etc.).]
- Place the extract in a (tall) vial.
- Just add the solvent to the extract, mix (slowly, not vigorous) and let it stand for a couple of hours.
- The solvent floats on top of the extract and can be removed by syringe or by freezing the water and pouring off the solvent Repeat with fresh solvent.

Next, remove the rest of the gunk by adding acetone to the extract.

Mix slowly and let stand. Again two layers will form.

On the bottom of the vial is a dark layer.

On top of that floats the acetone which is yellowish to greenish.

Remove the top layer by syringe or pipette.

- Add new acetone.
- Mix slowly. Now the dark layer becomes real sticky.

Let stand for a few hours, then remove the acetone.

- Then use 95 percent alcohol (cold).
- 5. Next extract the remaining dark stuff with 95 percent ethanol (boiling) to recover the psilocybin HCl.
- 6. Separate the liquid from the dark granules (using gravity, decanting).
- 7. Cool the ethanol until white crystals form.
- 8. Final step: collect all the dark sticky residue and dry it slowly.
- Large transparent crystals will form.

The more slowly the evaporation the bigger the crystals.

The potency of the mushrooms can now be determined by weighing the crystals.

The magic step probably was in the addition of the few drops of hydrochloric acid to the alcoholic extract.

This produces the psilocybin HCl salt, which is insoluble in acetone and which forms nice big crystals.

It is also important to give each step in the protocol enough time. This is most obvious in the last step, the double acetone wash.

The dark residue crystallizes by itself in the acetone after 12 hours!

I looked into this method to see if the green-blue-dark part of the extract can be separated from the psychoactive part.

In this method that is not possible."

More recently success was reported by the same person with ONLY 70% Ethanol and mushrooms [635 ml: 100 grams dry powder] using a 24 hour soak in a refrigerator, meticulous removal of particulates while still cold & overnight freezing to cause a layer of crystals to form. (This was done 2X) See Yachaj 2003 *The Entheogen Review* 12(3): 82-88. [Yachaj used 70% acetone-denatured ethanol due to availability.]

This process was tested and found to work great using pure ethanol diluted to 70% with water but the separation failed when Bacardi 151 was instead chosen as an alcohol source. [Perhaps from the contained sugars?] However, the resulting liquor from the rum attempt was greatly loved by people who liked both alcohol and mushrooms.

Note H (from page 211)

The Machine works great with DMT or 5-MeO-DMT but is highly recommended for application with isolated *Acacia* or *Mimosa* alkaloid.

For more detailed discussion see Justin Case's article in the 2003 *Entheogen Review*

The steel should be replaced frequently as it will tend to oxidize. Care should be taken not to overheat it as this will produce toxic nitrogen oxides which are bad news for lungs.

We would also suggest **always** throwing away any burned and blackened tryptamine residues rather than trying to smoke them. Degradation products such as neutral indoles are not good for lung tissues. Clean pipes often.



Psilocybe cyanescens (CA, USA)

The following ran in the 13 (2) 2004 *Entheogen Review*. It is reproduced with Pachano's permission.

Extreme-condition hostilis extraction

as told to Mambo Pachano

Several interesting modifications to a normal isolation approach have proved effective in overcoming both the adverse impact of tannins and the emulsion forming nature commonly encountered in this process.

I do not know who to credit as this seems to have resulted from the interactive exploration of at least several people, mostly anonymous, over the course of the last two or three years.

The use of acidic extraction at pH 1 has been questioned as being too extreme and merely overkill but it is worth noting that this approach WORKS and these results have now been replicated by a growing number of people.

Critics can continue to say what they want but any opinions arising from an armchair viewpoint are worth a whole lot less than those resulting from some meaningful wet glove time (IMHO)

Thanks to all involved.

At least one person we know has reproduced the following process but switched methylene chloride for toluene and obtained the same end results. (They still used xylene to defat)

Please understand proper chemical handling & safety protocols for concentrated acids, strong bases and toxic organic solvents before performing this isolation.

Readers should also be aware that in some countries, including the US, the following is considered to be drug manufacturing and can result is serious legal trouble if put into actual practice.

The approach:

1) Prepare the material for extraction.

Starting amount for the following was 1 kilogram.

The bark was first broken into smallish pieces by hand and then run through an industrial blender in small amounts until it was all shredded and/or powdered.

2) Prepare an aqueous acid solution that is strong.

I have seen hydrochloric acid used as a 10% dilution (with more added if needed).

The process below used supersaturated citric acid.

This was prepared by heating 500 ml of water to boiling and adding 125 grams of citric acid with stirring. It was cooled before use.

3) Combine the acid with 3 Liters of Absolute ethanol denatured with isopropanol.

The resulting pH should be around 1.

Add more acid with stirring if needed.

pH2 works OK but the lower the better.

(Our belief is that this extreme acidic condition is degrading the tannins and preventing them from complexing with the alkaloids)

4) Soak material in the dark, at room temperature from overnight to a week.

5) Filter off the acidified alcohol and save it.

Be sure that no particulates came through your filter. Let stand and settle if they did.

Google "noman" for an even simpler approach.

(Repeat steps 2 & 3 another time or two but process them separately and carry the first extraction forward through to its completion as soon as is feasible)

6) Carefully reduce to a solid by evaporating the alcohol.

This will leave an acidic residue

7) Dissolve this residue in warm water

8) Defat this using xylene.

Perform the defatting a total of three times.

9) Basify to pH 14 using a very strong solution of lye (sodium hydroxide)

Lower pH will result in the aqueous phase forming two layers; a dark reddish one and a lower turbid blue-green-greyish one with much solids.

Also an abundant emulsion will form when the basic solution is shaken with a solvent.

Neither of these occurs at high pH.

If either one is an issue, just add more base.

10) Extract with toluene by carefully mixing for an extended period or by shaking.

Let stand until separated then draw off or pipette off the toluene.

11) Perform step10 a total of three times.

12) Evaporate the toluene.

If a rotovap is not available, a stream of air can be used to help but we would suggest using no heat.

The final stage of the evaporation should be in a largish flat bottomed glass dish.

13) When dry scrape up and package. Seeding should not be necessary.

This has reliably produced a yellow waxy-crystalline massive solid that crushed to white powder. It had only a faint floral smell indicating substantial purity and lack of skatole.

Recovery ran around 1% by weight using Brazilian-sourced *Mimosa hostilis* rootbark (GC-MS beow).

Mexican material from Chiapas gave variable but significantly higher yields of 2-3%.



Acacia phlebophylla phyllodes Notice the reddish granular margin

Questions concerning the efficacy of Methanol vs. Optimized Methanol vs. Ethanol when used as extraction solvents

Gartz 1994b: Comparative extraction results. (% by dry weight)

Reported phosphorylase activity in aqueous alcohol extracts and in SOME acid extracts only.

Gartz concluded that the increase in PSOH which had been noted by Kysilka & Wurst 1990 was therefore due to the formation of PSOH from PSOP. Gartz recommends pure methanol for alkaloid profile studies.

1 Acetic acid **2** pure Methanol **3** acueous Methanol and Ethanol systems (*ala* Kysilka & Wurst 1990) Species Baeocystin Baeocystin Baeocystin PSOH PSOH PSOH PSOP PSOP PSOP 2 3 2 3 1 2 3 1 Gymnopilus 0.05 0.01 0.35 0.29 purpuratus 0.01 0.35 0.24 0.34 0.24 Inocybe 0.15 0.05 0.05 0.32 aeruginacens 0.15 0.21 0.32 0.40 **Psilocybe** 0.02 0.21 0.85 0.60 bohemica 0.040.210.60 Psilocybe bohemica 0.04 0.28 0.65 0.93 0.65 (cultivated) 0.02 0.28 Psilocvbe cubensis 0.02 0.25 0.11 0.25 0.45 0.63 0.45 Psilocybe cvanescens 0.02 0.61 0.51 0.61 0.20 0.32 0.20 **Psilocybe** 0.34 0.11 semilanceata 0.11 0.15 0.15 0.97 0.98 0.80

In obvious conflict to this was the earlier report of Kysilka & Wurst 1990:

They claimed to have determined that use of pure methanol had likely caused many previous reports to be lower than reality and had probably produced some false negatives. They did not specify the precise parameters of their methanol extraction; instead citing Stijve & Kuyper 1985 (who soaked overnight in methanol.)

Their modified extractions:

PSOP: 10 minute soak in 75% aqueous methanol (saturated with potassium nitrate)

PSOH: 160 minute soak in 75% aqueous Ethanol (This was found inferior for PSOP) (Only 76% as much PSOP and 8% as much PSOH were recovered in this solvent compared to using pure methanol)

Psilocybe bohemica Šebek

Conventional approach (with MeOH) PSOP (0.932%) & PSOH (0.041%)

Improved approach (with aqueous EtOH & longer extraction) PSOP (1.223%) & (Optimized MeOH) PSOH (0.448%)

Also in apparent conflict with Gartz is the direct comparison by Stijve & de Meijer 1993:

They reported a very substantial increase in PSOH yields (if present) using the optimized systems of Kysilka & Wurst 1990 (For PSOH: 75% aq. MeOH saturated with Potassium nitrate) and also a smaller but significant increase in both PSOP & Baeocystin yields when using 75% ag. EtOH instead of pure MeOH. (Stijve & de Meijer 1993 soaked overnight) Also in contrast to Gartz, Stijve & de Meijer detected NO PSOH in P. semilanceata with the optimized solvent (nor with any other of the species they looked at which normally lacked PSOH).

They reported a very substantial increase in PSOH yields (if present) using the optimized systems of Kysilka & Wurst 1990 (For PSOH: 75% aq. MeOH saturated with Potassium nitrate) and also a smaller but significant increase in both PSOP & Baeocystin yields when using 75% aq. EtOH instead of pure MeOH. (Stijve & de Meijer 1993 soaked overnight) Pertinent results from this paper:

[Yield for Pure MeOH vs. 75% aq. MeOH sat. w/ potassium nitrate for PSOP & 75% EtOH for PSOH]

Inocybe cordyalina var. cordyalina Quélet

0.023% vs. 0.03% PSOP but no PSOH in either system

Inocybe haemacta (Berk. & Cooke) Sacc.

0.023% vs. 0.03% PSOP but no PSOH in either system .

Psilocybe cubensis (Earle) Singer

Labgrown:

0.12% vs. 0.15% PSOP & 0.05% vs 0.50% PSOH (Mexican strain);

0.12% vs. 0.15% PSOP & 0.10% vs. 0.33% PSOH (Amazon: strain)

Psilocybe semilanceata (Fr.) Kummer

0.39% vs. 0.47% PSOP & no PSOH in either system



Photo by FunGal

Psilocybe azurescens (Astoria, WA)

The "*hotknives*" approach will work well for volatile alkaloids, *Cannabis*, hashish or opium.

Our thanks to Aubrey Hempburn for granting us the permission to include the following!!

Hotknives

by Aubrey Hempburn

Please distribute freely, if you care to. No copyright, just good intentions.

Hotknifing is a method of alkaloid volatilisation that is very effective and easily improvised from common household items. It can be used with most smokable Medicines.

Beware, it is often a cause of some regret when one realises which knives one improvised with. Or when someone else realises. *Needful Materials*

Heat source:

Natural or propane gas fires, cookers, blowtorches or camping stoves are the most responsive and best suited to hotknives. Electric heating can be used. Presumably a heater regulated by a thermostat could provide the desired temperature that otherwise must be judged by experience.

Hearth:

A large flat stone or paving slab is a nice option for a hearth, though not very portable. Shovels, saucepans, wire racks from cookers or whatever is heat resistant and to hand. The Earth, perhaps.

Knives:

Preferably stainless steel and relatively thick so they retain the heat for longer. This allows a larger window of opportunity. Fish-knives are fitting. Please ask the owner before destroying their knives.

Plastic handles are not a good idea as they are often forgotten temporarily and overheat.

You may wish to have a mug of cold water or ice handy to cool the metal handles.

Curling tongs have been very popular with some, they allow the hotknife (hot tong in this case) to be delivered single-handedly. It can be more difficult to site the material easily because of the curve of the hot bit requiring a longer fall and likely more bounces. Tongs take more heating but stay hot longer.

Bottle:

A capacity of a half to one litre is usually appropriate. However a four litre demijohn has been used successfully. The choice of bottle composition depends on the type of clumsiness to be expected. Glass may be dropped but plastic may singe. Metal would be best. A large can can be used though the smoke density would be a matter of faith.

The hole in a plastic bottle can be cut with a sharp knife. A good sharp screwdriver for metalwork. Glass bottles are less predictable, of course.

With the right sort of drill the hole can be done very neatly.

It should be just wider and taller than the width of the knives, if possible. The larger the hole the more smoke escapes.

The easiest way to break a hole in the bottom of a glass bottle is to use a knife. Wrap the bottle in some newspaper to catch any flying splinters. Introduce the knife gently through the neck of the bottle. Shake gently up and down the long axis of the bottle. Try to get the knife to strike around the full circle of the edge. Some force may be required to break through, depending on the thickness of the glass, bottles for fizz being much tougher. Just keep increasing the energy smoothly until it breaks. Patience, Grasshopper.

With luck and patience and lots of bottles the same method can be adapted to produce the vastly preferable side-holed style (See illustration on page 205) In this case a small heavy weight such as a ball-bearing is rattled against one side only, attempting to get it to hit the same place on the bottle's inside wall each time. The free-standing and less open side-hole bottle has obvious advantages.

The bottles are then rinsed thoroughly and paranoidly under turbulent water to carry away any splinters.

[Ed.: We would suggest use of a glass cutter or diamond tools with lots of water or the services of a lapidarist]

Bottle-top:

The bottle-top can be used as is or cut down to facilitate quick removal.

A coin can be used to cover the neck of the bottle if the bottletop has been lost but position must be checked before filling the bottle.

Smoking Materials:

Dry calcium carbonate (pure chalk) can be used as a carrier for many alkaloids to prevent them dripping off the knives before heated enough to boil. It can be simply mixed with the alkaloid, with just enough heat to melt it and rub it in if necessary. Too much calcium carbonate will insulate itself, so don't overdo it. The resultant mixture should be wrapped in foil to prevent charring around the outside. These little packets are then lightly perforated with a needle.

Cannabis has been smoked in this manner, though with hashish it is unnecessary to wrap it. Hash oil can be mixed with calcium carbonate to keep it in place.

Procedure

Heat the knives.

The hottest part of the flame is beyond the cold light blue gas. Only leave the knives until they just start glowing.

Switch off the flame if safety (or money) may be an issue.

Allow the knives to just stop glowing when viewed in the shade. This is for general purposes, different materials may need different heats. Some will decompose or burst into flame if overheated. Hold one knife level on the hearth with the non-dominant hand and drop the experimental material on the end.

Try dropping it on again. Repeat until successful.

Insert that end of the knife in the hole in the bottle.

Take the other knife in the dominant hand and press the hot end on the material.

Remove knives and put on the hearth.

Take the bottle-top or coin from the neck and inhale.

Personal preference will determine how the smoke is sipped or slurped.

One can hold the bottle oneself if the compound is not too fastacting. With the glass bottle with no bottom this is the only option as it must be held while filled.

One could have a friend hold it but arrange hand signals beforehand. "Waving all my fingers around meant I wanted another ten seconds, you swine, wasn't it obvious?"

The side-hole style bottle can be either held or allowed to stand while inhaling from it.

It is possible to volatilise huge amounts but repeated small doses are often preferable for both lungs and brain.

Be warned that effects can be overwhelming before you return to your seat



The hotknife is a copyright-free design

Trout's Notes FS-X7: Endnotes

Tolerance & Cross-tolerance

Tolerance

Tolerance, in this instance, refers to the property of a drug to produce progressively less effects each time a given dose is repeated. Despite being a property common to addictive drugs, tolerance has nothing to do with addiction since it occurs in many drugs that lack any type of physical or psychological cravings, dependency or withdrawals upon cessation of drug use (the latter is a requisite feature of an addiction)

Despite having discussed portions of this in the text, it was thought helpful to present a single summary.

Erspamer 1954 noted that both **Tryptamine** and **Serotonin** produce only a short-lived desensitization to themselves with repeated exposure.

Similarly, tolerance to **DMT**, if it truly exists as opposed to similarly being only a brief-lived desensitization, appears to develop quickly (3-5 min) and apparently disappears as rapidly. (Leading researchers to the conclusion that no tolerance develops) Time to recover so that no 'tolerance' is noted is unclear but short. (15 minutes if injected was evaluated in humans by Rick Strassman and this resulted in no tolerance development but this is the only such formal testing).

Justin Case commented that, for him, repeating large doses of DMT immediately after the peak wore off produced intense nausea, headache, darkness and a wretched physical sensation, unlike the marvelous re-entry into DMT-space that would have resulted if waiting only a bit longer. (Case 1995 pers. comm.)

Case's reaction to immediate redosing is not always the case but IS a potential response with larger amounts.

Similarly, smoking a given dose in divided portions over a more protracted period of a few minutes (despite being useful) does not reach the same peak intensity as does smoking it all rapidly.

We do not intend to suggest that the results of smaller repeated administrations are without effect; even if repeated immediately upon regaining awareness of one's surroundings. Sometimes immediate re-entry can be amazing. A euphoric, colorful, mirthful experience is readily possible. This can be repeated, using lower amounts than if smoking a freebase pipe, for a number of repeat administrations before one starts feeling like there is diminishing returns from the amount smoked. This is less a feeling of growing tolerant than it is feeling like one has saturated all available receptors and there is simply nothing left available to stimulate.

A wait of no more than one or two hours has repeatedly been proven to be all that is required for full effectiveness to return from further smoked DMT (or 5-MeO-DMT) after having reached this 'saturation point' via repeated doses.

Gillin *et al.* 1973 was unable to observe tolerance in cats receiving DMT twice a day for 7-15 days and every 2 hours for 24 hours (they instead reported an increasing sensitivity to the drug; something we have also experienced whenever using an intensive dosing schedule).

Kovacic & Domino found mild tolerance to some of the effects after injecting rats with 10 mg/ kg every 2 hours for 21 days. (This would represent 700 mg per injection for a 70 kg human!)



Psilocybe cyanescens (Seattle, WA, USA) Photo by FunGal



Psilocybe cyanescens (CA, USA)



dried Psilocybe azurescens (WA, USA)

Tolerance

Rick Strassman determined that there was **no** development of tolerance to intravenous injections given at 15-minute intervals. He suspects what users think of as DMT-tolerance may instead be an impaired capacity of the lungs to effectively absorb the material after having been previously assaulted by the strongly basic vapors.

It is unclear if tolerance develops to 5-MeO-DMT.

If it does, it must be *extremely* short-lived (a very few minutes) as repeated administrations possess full activity; as does a large dose taken as a rapid series of smaller doses (which can be maintained to stay in the peak.)

Justin Case reported that pyrolyzed doses could be repeated at 3-10 minute intervals for full effect, that this could be done at least 10 times in a row and even after three days of repeating this, no higher dosages were needed to attain the same results.

Instead he found himself MORE sensitive to the effects perdose once well into the evaluation process. At best a mild and very short-lived desensitization appears to occur after many rapidly repeated uses.

This was noticed when smoking 5-MeO-DMT free base immediately after an oral dose of 35 mg (a mild oral dose) had worn off but the response to the smoked 5-MeO was diminished only slightly in peak intensity and otherwise normal. However, repeated administration of smoked 5-MeO-DMT soon after THAT point, were fully effective.

More in depth evaluation produced some interesting results. It was found to be possible to closely repeat DMT and/or 5-MeO-DMT administrations (especially when done concurrently or alternatingly) to such a degree that a sense of complete 'saturation' resulted which rendered subsequent doses trivial. The exact number of repeats needed to reach this point varied from session to session but were always fairly numerous if only the smoked tryptamine was used. (This has been evaluated in literally dozens of such sessions.)

However, if acid was taken first it required relatively few repeats of tryptamine administration (usually less than half a dozen times each) to reach this point of feeling saturated. Mushrooms seemed perfectly effective after that point but this perhaps might have involved their more protracted onset time (compared to smoked free base) since if an hour or two break was taken either of the smoked tryptamines became fully effective again.

The available data and bioassayist pool to draw from is still limited. It is unclear if *everyone* responds this way or if they always respond this way. (It is obvious both that some people DO respond differently than most and that within a single individual there is a huge range of potential responses when ingesting DMT)

Virola snuff users are known to readminister doses but beyond that the issue appears unstudied.

A similar situation is likely true for **Bufotenine** but no data has been located; beyond the fact *Anadenanthera* snuff users do use repeated doses successfully.

In contrast, tolerance to the effects of **PSOP/PSOH** develops rapidly if Pshrooms are re-administered.

> Acacia obtusifolia trunk on an older tree

This is much less noticeable if more pshrooms are consumed DURING the peak of the prior dose with adequate time allowed for effects from the subsequent dose to begin **before** the peak from the preceding dose has begun to fade. So long as each of these subsequent doses is as large or larger than the preceding dose a quite lengthy experience is possible.

This is not the case if the preceding dose is allowed to fade or wear off prior to a subsequent dose taking effect. In this case, a user will soon be unable to achieve the desired results after only 1-3 days of use; even if ingesting huge amounts of fungi. It is possible to ingest enough for some type of discernible effects but by the third day it is generally considered a waste of material to do so as the mental aspects are the most affected.

LSD shows a time course for the development of tolerance similar to *Psilocybe* and mescaline is only a bit slower with effectiveness being reported for up to 3 days of continuous consumption (See *Sacred Cacti*).

A typical & illustrative example, as related by a friend:

"There was one time years ago when I had some particularly strong blotter. The first night I had some [...], it was quite strong.

The following night we each took twice as much, but the trip was very minimal in effect and we were quite disappointed at the waste of good acid."

Tolerance will also return to baseline within several days after the ingestion ceases.

We were unable to find any information concerning DET, DPT, 4-Acetoxy-DET and most other tryptamines & lack enough field experience with them to make any assessment.

Case noted that repeating the administration of smoked DPT immediately after a previous dose wore off was fully effective but did not explore it past that point.



Cross-tolerance

Cross-tolerance is a situation where tolerance developed towards one drug will elicit a similar response towards another drug.

Cross-tolerance between Psilocybin and LSD or Mescaline has been noted by a number of researchers. (Ex.: Abramson *et al.* 1960, Appel & Freedman 1968, Isbell 1959, 1961 & 1962 & Wolbach *et al.* 1962.) Rosenberg *et al.* 1964 reported only slight cross-tolerance between DMT & LSD (in LSD tolerant humans)

It is apparently more pronounced when tolerance to Psilocybin or LSD is first allowed to develop and mescaline is then used; than it is when this order is reversed. (See comments in Sacred Cacti. 2nd ed: p. 265)

Both MLD-41 (active) and LEP-57 (nonhallucinogenic) also showed cross-tolerance with Psilocybin. (The stimulant alkaloid Trichocereine and the inactive/nonhallucinogenic alkaloid DMPEA similarly show a cross-tolerance with mescaline)

Ott 1996 further noted that while LAE-32 (Lysergic acid ethylamide) showed weak cross-tolerance with LSD it had none with Psilocybin (Abramson & Rollo 1967)



Diplopterys cabrerana seedling Photo above by Partrick Noll



Psilocybe cubensis in its natural growth media (Australia) Photo by Anonymous

Drug interactions, potentiation & synergy

DMT combined with harmine or harmaline permits its **oral activation** and significantly prolongs the duration but at the same time it significantly *diminishes* the peak intensity and adds somatic effects that some users find objectionable.

Some users, on the other hand, LIKE to predose with harmine/ harmaline (oral or smoked) and THEN smoke DMT or 5-MeO-DMT. This has been likened to a 'buffering' effect.

Elements of the experience are smoothed out or even obscured but other components are perceived with an enriched robustness.

Smoking DMT following the prior oral or smoked administration of the MAOI has been reported to add an element of buffering reported useful by some shamanic practitioners. (See Ott 2000 & 2001a-c for comments on harmine/harmaline in combination with DMT, Bufotenine and 5-MeO-DMT)

Smoking batches of *Acacia obtusifolia* extract containing significant amounts of *Acacia* betacarboline produced a smoother, less intensely colorful yet quite robust psychospiritual experience that lasted far longer than when smoking isolates from the same species that do not contain this alkaloid or alkaloids. [The factors affecting/effecting its occasional but demonstrable presence are not yet understood. Winter harvesting, inclusion of the roots and stress are all suspected factors.]

Sai-Halasz 1963 reported that human volunteers showed a diminished response to DMT after 4 days of pretreatment with Iproniazid (an MAOI). Moore et al. 1975 found pretreatment of rabbits with Iproniazid caused both a potentiation of DMT's action and a prolongation of its effects. Shah & Hedden 1978 had similar results with mice; observing higher tissue levels of DMT. Lu *et al.* 1974 reported a prolonged half life in rats. The important point to understand about Sai-Halasz's work is the protracted duration of predosing prior to DMT administration. This produces quite different results than does coadministration or MAOI predosing shortly before DMT ingestion.

5-MeO-DMT is active orally in amounts several times or more what would be an effective smoked dose. Effects from oral administration of 35 mg are milder than if even 5 mg had been smoked (albeit longer lasting) but the concurrent addition of or predosing with MAO inhibitors like harmine or harmaline causes a pronounced increase in its effects. (See Ott 2001c's comments on 5-MeO combined with harmaline or harmine.)

The use of LSD (even at barely threshold levels) permits normally trivial amounts of smoked DMT or 5-MeO-DMT to be active and also causes an increase in effects *PER DOSE*.

Some people report a lengthening of the peak for both tryptamines when used with LSD but others do not.

The incredible potentiation of smoked *Salvia divinorum* after predosing with LSD has been commented upon by Case and *MANY* others.

LSD shows a dramatic **synergy** with *Psilocybe* (with an increase in duration); as does harmine.

Meaning that, when used in combination, lower doses can have greater effects.

There are repeated comments appearing in the literature claiming that MAOIs cause a diminished response to orally active hallucinogens such as LSD.

Interactions

This conclusion arises from studies (and reports) of people who had first been predosed daily with irreversible MAOIs for some days to several weeks prior to ingesting the acid. Ameliorated or blocked responses are commonly reported in this situation.

This all also has absolutely NO BEARING on the results of co-administration or ingestion of one closely following the other. As in the examples of incorporating pshrooms in ayahuasca, ingesting *Peganum harmala* seed followed by pshrooms or acid, the smoking of harmine or *Banisteriopsis caapi* leaf/stem during a pshroom peak, combining purified harmine or harmaline with LSD, or ingesting a fully active ayahuasca brew some 15-30 minutes after taking LSD. (It is perhaps noteworthy that of those authors claiming that a diminished response was automatic **none** of them have actually tried the combination outside of ayahuasca and/or pharmahuasca.)

In the case of ayahuasca, it is commonly perceived that the intensity per dose of DMT is significantly lessened or attenuated with a corresponding increase in the duration of the tryptamine's actions.

It might be important to consider that both DMT and 5-MeODMT lack any meaningful oral activity on their own at the dosage levels used in ayahuasca or pharmahuasca.

For the orally active hallucinogens, their interactions with harmine can be quite different.

When either LSD or Pshrooms are taken with harmine or harmaline, a significant increase in effects can be noted, especially in the more electric aspects of the experience, and our suggestion is that the dosage be scaled back. Many people love the combination (and also acid and pshrooms) but despite enjoying the effects, Case reported that these two combinations left him feeling overamped during the event and quite "crisp" or "fried" after the fact.

Unpleasant reactions (not limited to simple excessive dose responses) have also been reported by multiple people.

Examples of symptoms described: Cold sweats, sweating, confusion, vomiting, shallow labored breathing, dementia, loss of ability to articulate, dizziness, hypotension, involuntary spasms. Anecdotal accounts suggest this response may be most common in those fasting prior to ingestion. Caution should be used as a hypertensive crisis or serotonin syndrome are not impossible from some combinations. (Hypertension or hypotension can accompany the latter.)

Fasting often exaggerates the effect per dose. This is appreciated by some people and not by others.

A marked decrease in effects (in some cases even a total lack of effects) is reported by many users of SSRIs (also with dopamine reuptake inhibitors)

Interestingly most people we've interviewed reported only a selective interference with SOME, but not all, entheogens- with a variability depending on *which* antidepressant they were using. This too has resulted in reports of unpleasant or distressing responses. It is stated in the literature that MDMA and SSRIs are a guaranteed fatal combination. While clearly hyperbolic, our suggestion is to avoid the combination of ANY antidepressant with ANY stimulant or hallucinogen. *Psilocybe* with DMT added during the peak produced intense and sometimes disorienting experiences; without any noticeable increase to the duration. (Justin Case; unpublished journal notes). The mixture has been commented on as well loved by some users. A similarly mixed response was found among those users who have smoked DMT after taking mescaline.

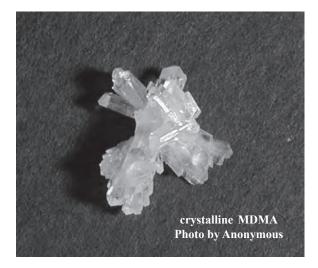
Tao Jones (personal communication) noted an increase in both action and duration for combined *Psilocybe & Salvia divinorum*.

The effects of DMT and Salvinorin A are dramatic when smoked in combination but it is not clear if the time course for either was increased or if the effect was simply additive. (We believe the latter)

It was thought to be better to ingest one and then rapidly follow it with the other rather than mix them together for smoking; due to the widely different boiling temperatures and a tendency to run away from each other in the pipe.. (Perhaps if they were both suspended on a herbal material rather than used in a pipe this would not be an issue?) (Case 1999; pers. comm.)

Piracetam (Nootropil) appears to interact positively with the major hallucinogens and some other psychoactive drugs. Hard data is still lacking but thusfar Justin Case, Toad & others have reported it to substantially increase the potency of the phenethylamines (such as mescaline, MDMA & methamphetamine) and suggest the dosage be halved. Case, in limited trials with several tryptamines, found radically less potentiation than with mescaline (if any) but did comment on an slightly stimulating enrichment of the experience with perceptions of an added robustness to both visual phenomenon & sensory input. More work is needed.

Pharmacodynamics, toxicity and safety of the combination of Piracetam and psychoactives, for the purpose of potentiation, appear to be unknown and unevaluated. It is likely to be fairly innocuous (given the low toxicity of the hallucinogens and the lack of toxicity for Piracetam) BUT we would urge the area be explored cautiously until more data comes in.



Interactions

Use of ketamine with 5-MeO-DMT and also with DPT was found to produce an exceptional experience but neither intensity nor duration appeared to be significantly altered (Case; pers. comm.) (Suggesting a simple additive effect rather than a potentiation.) While ketamine does not seem to potentiate the tryptamine it DOES seem to smooth or 'lubricate' entry into the tryptamine space. Ketamine also has some potential for boundary dissolution making a wondrous shared experience of 'mind puddling' potentially possible.

MANY users like to use ketamine in combination with other tryptamines. Toad has discussed this for several tryptamines, in *The Entheogen Review*, including DPT, 4-Acetoxy-DET and others. (See also D.M. Turner)

Case's best results came when keeping the k dose low and the tryptamine solid but not excessive (As memorable examples: 25 mg of ketamine followed by 165 mg of DMT fumarate IM or 40 mg of DPT and 40 mg of Ketamine snorted as a mixed series of alternating lines ingested in 4 sessions spaced at approximately 2 minute intervals.) Smoking 5-MeO-DMT while snorting small bumps of K is also commonly reported to be a nice combination.

There did seem to be an increase in duration for ketamine when combined with mescaline but we have too few data points to know if this is generally the case.

Ketamine is best be approached with great caution and we suggest it should be viewed as a 'hard drug' possessing a substantial impetus to repeat the experience. It is very clearly an addictive substance for many people who have tried it.

Nitrous oxide inhaled immediately following the inhalation of DMT freebase is also widely commented on both for being amazing and enhancing the perceived duration of the DMT.

Occasionally, mushroom ingestion results in "*adverse reactions with typical tachycardia*". This is believed to not be caused by the psilocybin but is suspected of being linked to the variable presence of phenethylamine.

For example *Psilocybe semilanceata* was shown to contain highly variable amounts of PEA and these levels fluctuated much more wildly than did the PSOP levels.

The highest amount of phenethylamine observed (146 μ g/g fresh weight) was in mushrooms resulting in a hospital admission. (Beck 1998)

Chlorpromazine and similar molecules substantially diminish effects of DMT, LSD and similar molecules. (See Moore *et al.* 1975 or Shah & Hedden 1978)

A similar reduction or abolishment of response has also been noted with 5-HT antagonists such as cyproheptadine but their "side effects.. may limit their clinical utility". Other compounds with similar actions or affinities such as clozapine or risperidone may also have some usefulness in abolishing effects in problem cases but their side-effects may create worse problems.

Benzodiazepines can also be of great usefulness. 0.5-1 mg of Xanax is more often chosen than Valium (diazepam) for this purpose but both can work. Applications include smoothing an agitatedly rough or unpleasant trip or perception of body load, or enabling sleep at the end of a intense session involving stimulant psychedelics or "party" drug combinations.

It is worth considering that within the psychedelic communities there is a widespread belief that to abort a bad trip with mind-numbing drugs can cause serious and lasting psychological problems. This belief is strongly supported by real world observations. Gentle supportive care in the form of a friendly assuring voice and a warm blanket can go much farther towards dealing with bad trips than the sad tendency of medicine to add another drug to the mix to make the responder go numb. Contrary to the appalling track-record of the psychiatric community when dealing with high dose "acid casualties" the use of alternative means such as energy work and other aspects of traditional Chinese medicine has helped many such traumatized people successfully reintegrate. In cases resulting from instances of ingesting dozens to hundreds of **MILLIGRAMS** of LSD-25 recovery has sometimes taken up to several months but there are a surprising number of people who have experienced such high dosages with a complete recovery..

Our belief is that, in normal nonpsychotic people, MOST bad trips producing serious outcomes result from either inappropriate dose coupled with inappropriate setting or else involve the response of nondrug users towards the drug user. When discovered tripping as a young child by her relatives, Maria Sabina was carefully and quietly returned to her home and bed so as to not shock or harm her altered and highly sensitive psyche. As a result she not only experienced no trauma but was enriched by the experience.

This SHOULD be taught to emergency medical personnel as a standard course of response when hallucinogens use is suspected but in reality there are doctors on record stating they deliberately pump stomachs of mushroom trippers, with full knowledge of the ineffectiveness of this stress-inducing action, just to deliberately traumatize the young mushroom eater and *"teach them a lesson"*. Such sadistic psychological abuse is simply neither sound nor ethical in clinical practice.

Infliction of mental or psychological trauma should NEVER be considered acceptable medical treatment and should be considered a form of assault, inappropriate care and/or child abuse. Even if believing psychedelic use is wrong, this does not justify causing demonstrable harm in addition to efforts to address the perceived harm; real or imaginary.



Psilocybe cyanescens above



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Note: The careful reader will notice that our use of some a & b designations for multiple papers by a given author (when published within a given year) are reversed in application from those of other authors. This is due to *their* organization of these being chronological versus our alphabetic arrangement. It seemed arbitrary to preserve some of these in chronological order and not others so we decided to opt for uniformity. (Gasp!) Any incomplete references cited are secondhand.

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Acacia phlebophylla flowering Photo by Anonymous



Acacia maidenii flowers and flower buds

Panaeolus species (Germany) Photo by Patrick Noll



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cebil seedpod

Some Simple Tryptamines 2nd ed.



Psychotria viridis seeds Photo by River's Source

Trout's Notes FS-X7



Psilocybe subaeruginosa (Australia) Photo by R. Kundalini top left

Buddha with a "parasol" upper right

Copelandia cyanescens (NSW, Oz) Photo by Anonymous center right see also page 205







Psilocybe subaeruginosa (Australia) Photos by Snu Voogelbreinder





Anadenanthera colubrina leaflets Photo by Zariat





Psychotria carthaginensis fruiting Photo by Zariat

Amphibian common names

Some of the common names used for the species mentioned in the occurrence lists:

African clawed frog See as Xenopus laevis African toad *See as Bufo regularis* Agile frog See as Rana dalmatina Agile frog See as Rana japonica American leopard frog See as Rana pipiens American toad See as Bufo americanus Angola toad See as Bufo funereus Argentine toad See as Bufo arenarum Arizona toad See as **Bufo microscaphus** Asiatic toad See as **Bufo gargarizans** Asiatic toad See as **Bufo melanostictus** Australian brown treefrog See as Litoria ewingii Australian giant treefrog See as Litoria infrafrenata Australian red-eyed treefrog See as Litoria chloris Australian rocket frog See as Litoria nasuta Australian striped treefrog See as Cyclorana alboguttatus Australian tree-frog See as Litoria caerulea Australian variable treefrog See as Litoria latopalmata Baird's spotted toad See as Bufo punctatus Bella Vista toad See as Bufo fernandezae Berber toad See as **Bufo mauritanicus** Black-backed frog See as Leptodactylus melanonotus Black-soled frog See as Lechriodus fletcheri Black-spectacled toad See as Bufo melanostictus Black-spined toad See as Bufo melanostictus Black-spotted frog See as Rana nigromaculata Black-spotted pond frog See as Rana nigromaculata Bleating tree frog See as Litoria dentata Bleating treefrog See as Litoria dentata Blue frog See as Litoria caerulea Blue Mountains tree frog See as Litoria citropa Blue Mountains treefrog See as Litoria citropa Blue-thighed treefrog See as Litoria raniformis Booroolong frog See as Litoria booroolongensis Boreal toad See as Bufo boreas British toad See as Bufo calamita Broad-palmed frog See as Litoria latopalmata Brown frog See as Rana temporaria Brown tree frog See as Litoria ewingii Bull frog See as Litoria moorei Canadian toad See as **Bufo hemiophrys** Cane toad See as Bufo marinus Cascade treefrog See as Litoria pearsoniana Cedar Creek treefrog See as Litoria pearsoniana Centralian tree frog See as Litoria gilleni Chinese edible frog See as Rana nigromaculata Chusan Island toad See as Bufo gargarizans Clawed frog See as Xenopus laevis Clawed toad See as Xenopus laevis Colorado River toad See as Bufo alvarius Common American toad See as Bufo terrestris Common clawed frog See as Xenopus laevis Common clawed toad See as Xenopus laevis Common cricket frog *See as Acris crepitans*

Common European toad See as Bufo bufo Common frog See as Rana pipiens Common frog See as Rana temporaria Common Indian toad See as Bufo melanostictus Common leopard frog See as Rana pipiens Common lesser toad See as Bufo granulosus Common platanna See as Xenopus laevis Common pond frog See as Rana nigromaculata Common toad See as Bufo bufo Common tree frog See as Hyla arborea Common warty-newt See as Triturus cristatus Common water-frog See as Rana esculenta Common water-holding frog See as Cyclorana platycephalus Confusing toad See as **Bufo perplexus** Crested newt See as Triturus cristatus Cricket frog See as Acris crepitans Dainty green tree frog See as Litoria gracilenta Dainty green treefrog See as Litoria gracilenta Dalmatian frog See as Rana dalmatina Dark-spotted pond frog See as Rana nigromaculata Darwin's frog See as Rhinoderma darwinii Darwin's toad See as Rhinoderma darwinii Desert collared-frog See as Cyclorana cultripes Desert tree frog See as Litoria rubella Desert treefrog See as Litoria rubella Dwarf toad See as Bufo canaliferus Eastern cricket frog See as Acris crepitans Eden Harbour toad *See as Bufo variegatus* Edible frog See as Rana esculenta Emerald-spotted tree frog See as Litoria peronii Emerald-spotted treefrog See as Litoria peronii European common brown frog See as Rana temporaria European common frog See as Rana temporaria European fire salamander See as Salamandra salamandra European green toad See as Bufo viridis European green treefrog See as Hyla arborea European salamader See as Salamandra salamandra European tree frog See as Hyla arborea European treefrog *See as Hyla arborea* Ewing's tree frog See as Litoria ewingii Fire salamander See as Salamandra salamandra Flat-headed frog *See as Cyclorana platycephalus* Fletcher's frog See as Lechriodus fletcheri Fletcher's cannibal frog See as Lechriodus fletcheri Fletcher's forest frog See as Lechriodus fletcheri Florida leopard frog See as Rana sphenocephala Fort Mojave toad See as Bufo microscaphus Fowler's toad See as Bufo fowleri Freycinet's frog See as Litoria freycineti Freycinet's treefrog See as Litoria freycineti Fringe-toed foamfrog See as Leptodactylus melanonotus Giant toad See as Bufo marinus Giant tree frog See as Litoria infrafrenata Girard's toad See as Bufo alvarius Golden frog See as Litoria aurea Golden treefrog See as Litoria aurea

Graceful treefrog See as Litoria gracilenta Granular toad See as Bufo granulosus Grass frog See as Rana palustris Grass frog See as Rana temporaria Great crested newt See as Triturus cristatus Great Plains toad See as **Bufo cognatus** Great water-newt See as Triturus cristatus Green and gold frog *See as Litoria raniformis* Green and golden bell frog See as Litoria aurea Green and golden frog See as Litoria raniformis Green frog See as Rana esculenta Green toad See as Bufo debilis Green tree frog See as Hyla arborea Green tree frog See as Litoria caerulea Green treefrog See as Hyla arborea Green treefrog See as Litoria caerulea Greenstripe frog See as Cyclorana alboguttatus Growling grass frog See as Litoria raniformis Gulf Coast toad See as Bufo valliceps Hammond's spadefoot See as Spea hammondii Hammond's spadefoot toad See as Spea hammondii Hammond's spea See as Spea hammondii House toad *See as Bufo melanostictus* Italian agile frog See as Rana latastei Italian frog See as Rana latastei Japan brown frog See as Rana japonica Japanese wrinkled frog See as Rana rugosa Keferstein's tree frog See as Litoria dentata Kisolo toad See as Bufo kisoloensis Knife-footed frog See as Cyclorana cultripes Lataste's frog See as Rana latastei Leap frog See as Rana dalmatina Leopard frog *See as Rana palustris* Leopard frog See as Rana pipiens Leopard frog See as Rana sphenocephala LeSueur's frog See as Litoria lesueurii LeSueur's treefrog See as Litoria lesueurii Madagascar bullfrog See as Laliostoma labrosum Madagascar jumping frog See as Aglyptodactylus madagascariensis Madang big-eyed treefrog See as Nyctimystes disruptus Maldonado redbelly toad See as Melanophryniscus moreirae Marbled toad See as Bufo marmoreus Marine toad See as Bufo marinus Marsh frog See as Rana palustris Martinique robber frog See as Eleutherodactylus martinicensis Mexican river frog See as Leptodactylus melanonotus Military River frog See as Thoropa militaris Moore's frog See as Litoria moorei Moroccan toad See as Bufo mauritanicus Mouth-breeding frog See as Rhinoderma darwinii Naked treefrog See as Litoria rubella Natterjack toad See as Bufo calamita New Guinea treefrog See as Litoria infrafrenata Nodugl treefrog See as Litoria micromembrena Northern crested newt See as Triturus cristatus Northern cricket frog See as Acris crepitans Northern leopard frog See as Rana pipiens

Northern Sonora toad See as Bufo speciosus Olive water-holding frog See as Cyclorana cultripes Painted frog See as Discoglossus pictus Pantherine toad See as **Bufo mauritanicus** Patagonian toad See as Bufo variegatus Pearson's green tree frog See as Litoria pearsoniana Peeping frog See as Rana pipiens Peron's tree frog See as Litoria peronii Peron's treefrog See as Litoria peronii Peru white-lipped frog See as Leptodactylus rhodonotus Pickerel frog See as Rana palustris Pimocha white-lipped frog See as Leptodactylus labrosus Platanna See as Xenopus laevis Pointedbelly frog See as Leptodactvlus podipinus podipinus Poison bull frog See as Rana palustris Poison bully See as Rana palustris Poison frog See as Rana palustris Rattler See as Acris crepitans Red tree frog See as Litoria rubella Redbelly toad See as Melanophryniscus stelzneri Red-eyed green tree frog See as Litoria chloris Red-eyed tree frog See as Litoria chloris Red-eyed treefrog See as Litoria rothii Red-spotted toad See as Bufo punctatus Rio Parahyba toad See as Bufo pygmaeus Rock river frog See as Thoropa militaris Rocket frog See as Litoria nasuta Roth's tree frog See as Litoria rothii Rust-eyed treefrog See as Litoria rothii Sabinal frog See as Leptodactylus melanonotus San Jose white-lipped frog See as Leptodactylus stenodema Sandy big-eyed treefrog See as Nyctimystes kubori Santa Fe frog See as Leptodactylus laticeps Scottish frog See as Rana temporaria Shad frog See as Rana pipiens Shelkovnikov's tree frog See as Hyla arborea Slender-fingered bladder frog See as Leptodactylus pentadactvlus Smoki jungle frog *See as Leptodactylus pentadactylus* Smoky jungle frog See as Leptodactylus pentadactylus Smooth clawed frog See as Xenopus laevis Somber toad See as Bufo funereus Sonoran desert toad See as Bufo alvarius South American bullfrog See as Leptodactylus pentadactylus Southeast Asian toad See as Bufo melanostictus Southern bell frog See as Litoria raniformis Southern leopard frog See as Rana sphenocephala Southern meadow frog See as Rana sphenocephala Southern orange-eyed treefrog See as Litoria chloris Southern roundgland toad See as Bufo coccifer Southern toad See as Bufo terrestris Southwestern toad See as Bufo microscaphus Spotted salamander See as Salamandra salamandra Spotted-thighed frog See as Litoria cyclorhynchus Spotted-thighed treefrog See as Litoria cyclorhynchus Spring frog See as Rana dalmatina Spring frog See as Rana pipiens Square-marked toad See as Bufo regularis Stony Creek frog See as Litoria lesueurii

- Strait-lipped warty-newt See as Triturus cristatus Stream-lined rocket frog See as Litoria nasuta Striped burrowing frog See as Cyclorana alboguttatus Striped rocketfrog See as Litoria nasuta Striped toad *See as Bufo crucifer* Swamp frog See as Rana palustris Texas toad See as **Bufo speciosus** Three-striped pond frog See as Rana nigromaculata Tiger frog See as Rana palustris Treasury Island treefrog See as Litoria thesaurensis Truando toad *See as Bufo haematiticus* Upland clawed frog See as Xenopus laevis Upland clawed frog See as Xenopus laevis Wallum rocket frog See as Litoria freycineti Wallum rocketfrog See as Litoria freycineti Warty bell frog *See as Litoria raniformis* Warty newt See as Triturus cristatus Warty toad See as Bufo spinulosus
- Water-holding frog See as Cyclorana platycephalus Western cricket frog See as Acris crepitans Western green and golden bell frog See as Litoria moorei Western spadefoot See as Spea hammondii Western spadefoot toad See as Spea hammondii Western toad See as **Bufo boreas** Whistling tree frog See as Litoria ewingii White-lipped tree frog See as Litoria infrafrenata White's treefrog See as Litoria caerulea Wiegmann's toad See as Bufo marmoreus Wood frog See as Rana sylvatica Woodhouse's toad See as Bufo woodhousii Wrinkled frog See as Rana rugosa Yellow Cururu toad See as **Bufo ictericus** Yellow leg frog See as Rana palustris Yellow legs See as Rana palustris Zebra frog See as Rana palustris

The Cultivation of Mushrooms is completely beyond the scope of this book.

While *MANY* other excellent (if not classic) works exist (see JW Allen's CD on this topic), we would suggest that the interested reader who is so inclined consult ALL of the following:

Be certain to check your local laws! Pshroom cultivation is presently considered illegal in some countries.

Recommended reading:

PF TEK (www.fanaticus.com/pf-tek html; use a public computer)

Falconer, William (1891) Mushrooms: how to grow them. A practical treatise on mushroom culture for profit and pleasure. Orange Judd: NY

> Rush Wayne (www.mycomasters.com) Growing Mushrooms the Easy Way, Volume I



Growing Mushrooms the Easy Way, Volume II (Second Edition)

Fungi Perfecti (www fungi.com) *The Mushroom Cultivator* (Paul Stamets & J.S. Chilton) *Growing Gourmet and Medicinal Mushrooms.* 3rd Edition (Paul Stamets)

Yachaj (2001) *The Entheogen Review* 10 (4): 127-139 "Mushroom Cultivation: From Falconer to Fanaticus and Beyond."

Yachaj (2001) *The Entheogen Review* 11 (1): 9-16 "Cooksbooks and Cubensis." "Teonanacatl: A Bibliography of Entheogenic Mushrooms" 2400 references, 1700 annotations, 8000 cross-reference author/date citations More than 700 COLOR images with enlargements \$39.95 plus \$5.95 Priority mail shipping & handling





"Mushroom Pioneers" 200 pages; 75 color photos \$19.95 plus \$5.95 Priority mail shipping & handling

"Psilocybian Mushroom Cultivation: A Brief History" 180 pages; more than 100 color photographs with enlargements \$19.95 Plus \$5.95 Priority mail shipping & handling

"Psychedelic Inspired Art" 1045 images (thumbnails and enlargements), 4 animated movie strips \$19.95 Plus \$5.95 Priority mail shipping & handling





Left-hand images (Top to Bottom) Psilocybe baeocystis Psilocybe semilanceata

"Angkor Sunset" by JW Allen

Send inquiry for price & availability to:

John W. Allen P.O. Box 45164 (Dept. TN) Seattle, Washington 98105, U.S.A.

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Right-hand images (Top to Bottom) Psilocybe azurescens Psilocybe samuiensis Psilocybe cubensis







Acacia maidenii (California)





Acacia maidenii (AUS) (above & left) Photos by Mulga



Acacia obtusifolia (in habitat) Photo by Mulga

Acacia obtusifolia (3 different forms) right and below Photos by Mulga

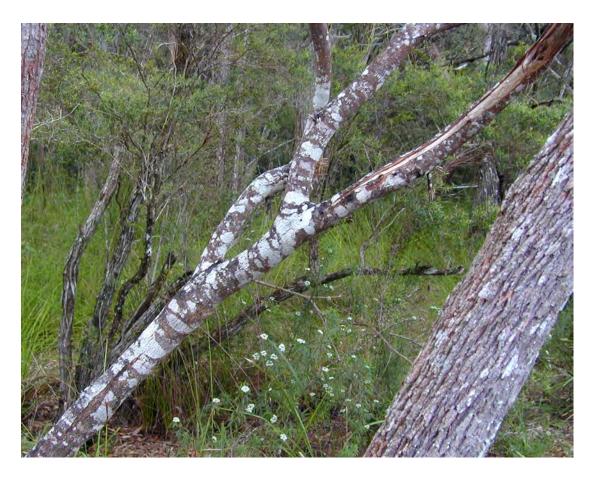
> Anadenanthera colubrina seedling lower right











Acacia obtusifolia in habitat NSW This specimen is growing by an observation point in a National Park Notice the rudely slashed bark that is suggestive of an interrupted harvest by a commercial drug harvester who clearly lacks concern for the species.



Copelandia cyanescens (Maui) Photo by FunGal



Psilocybe subaeruginosa (Australia) Photo by R. Kundalini



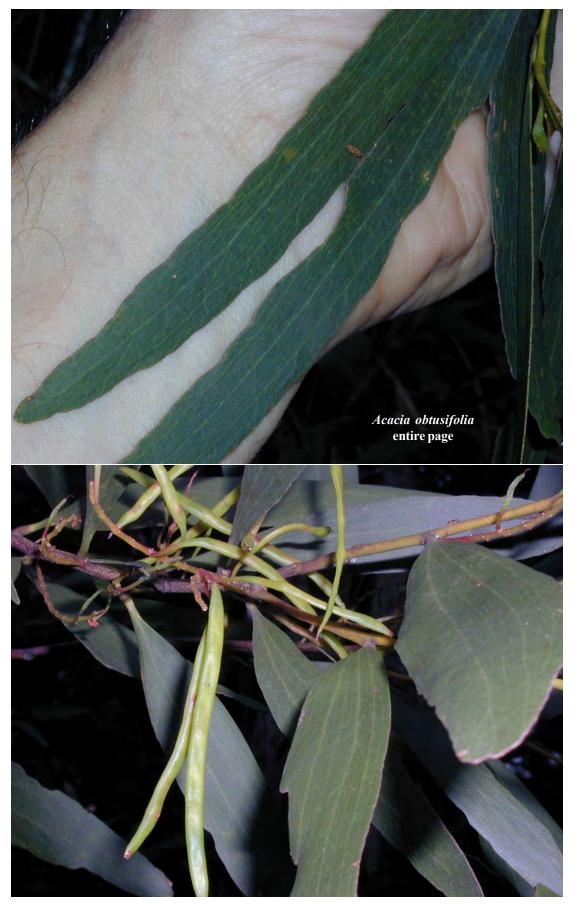
Acacia obtusifolia above & right



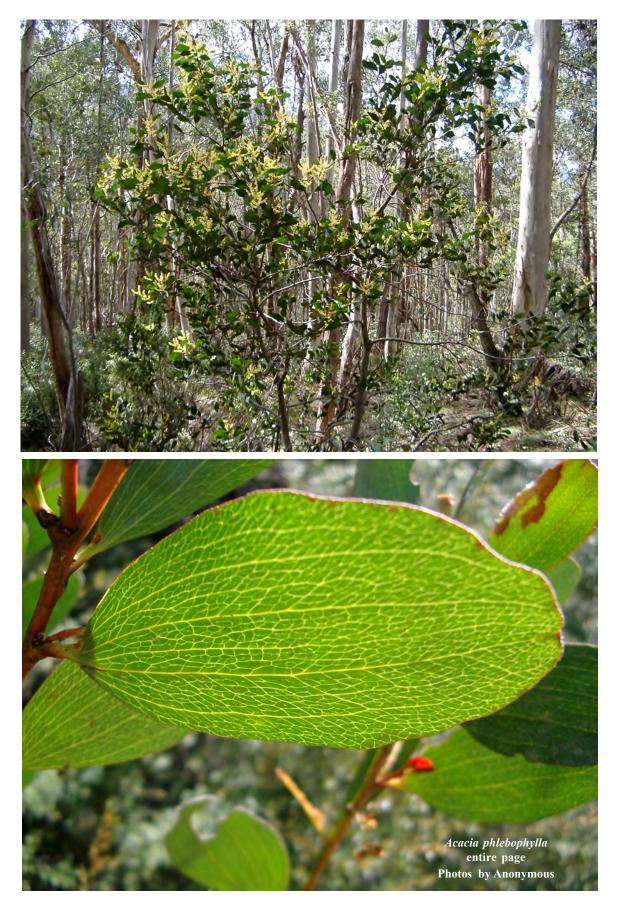
Anadenanthera colubrina var. Cebil

Anadenanthera colubrina





Some Simple Tryptamines 2nd ed.



Valuable Information Resources (Be sure to check their links as well!)

When using the Internet for finding information: *caveat lector*! INSIST ON & USE references; both for fact-checking and learning more detail.

http://www.ars-grin.gov/cgi-bin/npgs/html/index.pl (USDA GRIN) http://plants.usda.gov/ (USDA Plants Database) http://research.amnh.org/herpetology/index html (taxonomic info in Amphibians of the World) www.erowid.org (Erowid's incredible data archives) www ipni.org/ (International plant names index) www lycaeum.org www mobot.org (detailed but somewhat patchy taxonomic info is in W3tropicos) www museocivico rovereto.tn.it www.samorini net www.yage.net

www-sul.stanford.edu/depts/swain/beilstein/bedict1 html#Formula (German-English/ English-German dictionary of chemical terminology designed to accompany Beilstein)



Acacia phlebophylla seeds



Acacia phlebophylla phyllodes Photo by Mulga

Acacia phlebophylla seedling Photo by Mulga

A few words on Acacia phlebophylla

This is an extremely endangered Australia endemic. This is perhaps the world's rarest known entheogen.

It is presently threatened by a number of factors the most serious of which is a pathogen affecting it in its only known native habitat (The vector of distribution is presently not clear for this gall-forming fungus, believed to be an *Uromycladium* species. See Burges *et al.* 1934 & Heinze *et al.* 1998- Thanks Snu!)

Many seemingly healthy seedlings can be evidenced in its native home but very few reach adulthood. The older trees that are barely hanging on are important seed providers without which its long-term survival may not occur.

Many workers are actively trying to determine the cause for the spread of the disease and find an effective solution to ensure the long term future for this beautiful tree. We wish them success in their efforts.

More recently, brush fire has appeared to help suppress the disease and has stimulated seed germination & healthy regrowth. It is premature to make any conclusions beyond the occasional occurrence of fire being a good thing for *Acacia phlebophylla*.

Seeds are commercially available but it appears to be best if they are left in the wild or in the hands of trained conservationists or skilled horticulturalists. Cultivation efforts worldwide have been hampered by a ill-defined sudden death striking 3-5 years after germination.

A truly fascinating feature of this tree is that it sheds its phyllodes and these voluntarily dropped phyllodes have been proven to be fully effective in Australian ayahuasca even when the dried phyllodes were recovered months after dropping.

Acacia phlebophylla Trunk showing the gall forming disease which has threatened its only known population. Photo by Floyd Davis







Phalaris aquatica (probably *stenoptera*) Pt. Reyes National Seashore, California



Phalaris aquatica (probably *stenoptera*) Pt. Reyes National Seashore, California



Acacia auriculiformis cultivated in Austin, Texas



Psychotria viridis (above)



Psychotria carthaginensis



Acacia obtusifolia Photo by Snu Voogelbreinder



Psilocybe cubensis (Australia) Photo by Anonymous



Psilocybe cyanescens (Oakland, CA)



Some Simple Tryptamines 2nd ed.







Trout's Notes FS-X7

Acknowledgements

I would like to offer thanks to John W. Allen, Snu Voogelbreinder, Mulga, James Edmond, Des, FunGal, Nick, T. Greif & S. Czolowski, Patrick Noll, Giorgio Samorini, Floyd Davis, Craig, Maurizio, R.Kundalini, PFE, Kat Harrison, Ringworm, James Arthur, Aleph117 & those friends requesting anonymity for many of the images gracing these pages.

I would also like to thank the sacred plant teachers for their input and guidance (without which this work would not have been created), Sasha & Ann Shulgin for inspiration & feedback over the years, Max Strubel & Steve Kirschner for instilling a joyous love of organic chemistry, Johnny Appleseed for an incredible wealth of TLC assays and feedback. Also a special thanks to all of those amazing beings (whos names are being witheld for their safety) who took the time to introduce me to their dear mother tree and helped me to understand the serendipitous history of its discovery.

Many more people have my gratitude for their research & observations, both in speech or in print although most of those just named also fall into this category. I would additionally like to thank Rob Montgomery, Jonathan Ott, David Aardvark, René, Toad, Reid, Rick Strassman, Jon Hanna, Torsten, Dale Pendell, Dennis McKenna, Eel, Debra, Bob Wallace, Brett Blosser, Jane, friendly, theobromus, AP, DMCD, Gnostic Gardens, Richard Evans Schultes, Bo Holmstedt, John W. Daly, Peter van der Heyden, Tex, Otto Snow, George, Don, & Dennis for variously providing one or more of the following: their written word, be it published, internet posted, emailed or snail mailed, invaluable advice, opinions, input, feedback, suggestions, encouragement, criticism, moral support, observations, photocopies, journal articles, and/or bioassay reports.

I would like to express appreciation to Nick Sand for all of his labors of love in years past.

And also, of course, my gratitude to whoever it was who lost that small bag of DMT-treated herb back in 1972. And I will always be grateful to Andy & yage.net for providing access to a whole new world.

Many more people are owed thanks. Most prefer we NOT name them so we hope that you know who you are.

Last and perhaps most importantly I would like to thank an anonymous friend in Oz, Brook & Geoffrey for believing in both me and this project enough to help support its creation.



Psilocybe cubensis (Thailand) Photo by J.W. Allen



Cover design by by Mulga

THANK-YOU MULGA!



Acacia obtusifolia (Australia) Photo by Floyd Davis Copyright Pagan Love Cult 1998

MydriaticProductions

Known errata, questions & omissions

Known corrections:

Back flap of softcover

The final "s" in "leptolobus" should be in green not black

p 2:

The é in Libertié should be bold

p 21:

"imageson" should read "images on"

p. 74:

"caeruloannulata" should read "caeruleoannulata"

p. 87:

Earlier supposition by Jochen Gartz concerning the structure of aeruginascin proved to be incorrect.

Aeruginascin was determined to be a quaternary ammonium compound: N, N, N-trimethyl-4-phosphoryloxytryptamine. JENSEN *et al.* 2006

p. 92:

Photo of *Psilocybe liniformans* v. *americana* was incorrectly credited to J.W. Allen.

This image was reported to be a photo taken by Paul Stamets. Our thanks to James Edmond for bringing this to our attention and to Paul Stamets both for noticing it and for graciously granting permission to include it in the next edition - correctly attributed.

p. 116:

"Märki et al. 1932" should read "Märki et al. 1961"

p. 262

The comment "[Also appears listed in the literature as "Occurrence of Bufotenine (5-hydroxy-N,N-dimethyltryptamine in Schizophrenic Patients."]" is in reference to an erroneous citation.

The correct citation for the article with that title is Tanimukai, H., *et al.* (1967) Life Sciences 6 (16): 1697-1706. "Occurrence of bufotenin (5-hydroxy-N,N-dimethyltryptamine) in urine of schizophrenic patients." (H. Tanimukai, R. Ginther, J. Spaide, J.R. Bueno & H.E. Himwich)

p 298: "Thanks Snu!" should read "Information thanks to Snu Voogelbreinder"

p 300:

"Photo by Sinbad Vine" should read "Photos by Sinbad Vine"

Known questions:

A dangling question involves a *Psilocybe* growing in Oakland that was believed by their grower to be *Psilocybe cyanescens*. This was propagated using what was believed to be wild mycelium that was collected on woodchip mulch in Tilden Park.

The identification of these photos of woodchip-bed cultivated fungi has been questioned and *Psilocybe azurescens* has been suggested as an alternate identity. All of the images involved (pages 54, 56, 69, 82, 227, 231 & 301) came from a single flush.

Anecdotal accounts of human bioassays also report a low alkaloid content for these carpophores suggesting that it may be neither species. For example, one person had weak but nice results after eating a dozen freshly harvested mushrooms. Which seems rather unlike *azurescens*?

I do not know the answer.

James Edmond sent these 2 images to illustrate the question.



Check for future updates to this list at: www.largelyaccurateinformationmedia.com/addendum.html

Trout's Notes

Known omissions:

Front flap of softcover - Ayahuasca book's URL: http:/erowid.org/library/books_online/ayahuasca_apa/

Under opening comments (p 3)

Banisteriopsis was finally observed being used in the preparation of a traditional snuff. Robin Rodd witnessed and bioassayed highly potent snuff prepared from *Anadenanthera peregrina* seeds pounded to a paste with the fresh shoots of *Banisteriopsis caapi* before being kneaded with ash and heated to dryness. (RODD 2002)

Under N,N-Dimethyltryptamine (p. 29) Anadenanthera falcata (BENTH.) SPEG. DMT 3% of 4.9% dry wt. in seed DMT 0.07% dry wt. in pod SÁVIO NUNES et al. 1982 (via GC-MS)

Under N,N-Dimethyltryptamine (p. 32) *Piptadenia gonoacantha* (MART.) MACBR. DMT 0.48% dry wt. in seed. DMT 0.07% dry wt. in pods. DMT 35% of 0.2% dry wt. in bark. SÁVIO NUNES *et al.* 1982 (via GC-MS)

Under blueing list (pp. 80-81):

Psilocybe aequatoriae Singer Psilocybe naematoliformis Guzmán Psilocybe neocaledonicum Guzmán & Horak Psilocybe neorhombispora Guzmán Guzmán 2004

Under psilocybin (p. 86):

In a controlled double-blind experiment, researchers reported that 61% of participants given a strong dose of psilocybin (as 30 milligrams of psilocybin per 70 kilograms of body weight) had a "full mystical experience," as measured on established psychological scales. Two-thirds of the 36 participants rated the experience as either the single most meaningful experience or among top five most meaningful experiences of their lives. 79% of the participants reported a moderate or greatly increased sense of well-being or life satisfaction two months after taking the drug. None had previously used any hallucinogen and over half were active in church or an-other spiritual community. GRIFFITHS *et al.* 2006

Under bufotenine (p 107):

Acacia obtusifolia has apparently been reported to contain variable amounts of bufotenine in the resin extracted from the stembark. In the four samples where it was reportedly observed, it was as a minor component that appeared to be highest in a winter extract compared to the faint traces in a summer extracts (using gcms.) (TROUT 2005)

This appears to be the first report of bufotenine in an *Acacia* species. It was not corroborated by a second worker's gc-ms but Sasha suggested to me that this was possibly due to a lack of the appropriate column equilibration. The presence of bufotenine was more recently supported in 2005 by Ott using tlc with known reference material according to a friend who was visiting him when this occurred. (ANONYMOUS in personal communication.)

Betacarboline(s) are thought have been observed in the root bark (suggested by fluorescence and bioassay results in summer of 2003) but there has been no attempt at identification.

One stem bark alkaloid was thought to possibly be 1,2-dimethyl -1,2,3,4-tetrahydro- β -carboline based on that second worker's gcms but that was never confirmed. (Trout 2005)

Earlier hplc-ms work by Mulga appears to have also observed a betacarboline in the stem bark but it was not identified.

Mulga did not observe bufotenine. (MULGA 2005)

In all cases mentioned above, DMT was the major alkaloid. The question has been raised whether the N-oxide was observed rather than bufotenine but no one seems to have followed up on it.

Under Bufotenine (p. 107)

Anadenanthera falcata (BENTH.) SPEG. Bufotenine 0.0049% dry wt. in seeds. Bufotenine 0.0056% dry wt. in pods. Bufotenine 0.96% dry wt. in bark. SÁVIO NUNES *et al.* 1982 (via GC-MS)

Under Bufotenine (p. 108)

Piptadenia gonoacantha (MART.) MACBR.

Bufotenine 0.0022% dry wt. in bark.

SÁVIO NUNES et al. 1982 (via GC-MS)

Brosmium acutifolium HUBER subsp. *acutifolium* C.C.BERG [Moracaea]

"takini"

Bufotenine was present in the variety but not in the parent species. White latex was found to contain $0.7 \ \mu g$ of bufotenine per ml. Red latex was found to contain $23.4-25 \ \mu g$ of bufotenine per ml.

Bufotenine is not found to be present in the bark.

Novice shamans drink the latex and smoke the bark but later in life apparently only drink the latex.

Only the frothy red latex is used. The translucent white latex that preceeds it when the tree is tapped is discarded.

Bufotenine is believed to be the active component even though only a total of 12.5 mg was present in the 500 ml portion of red latex consumed. [kt: More work seems to be needed to assess the impact of the role of the smoking of bark by novices.]

The drink produced a strongly sedative component in addition to its hallucinogenic action.

MORETTI et al. 2006

Under Bufotenine (p. 110)

Present in:

Osteocephalus taurinus Osteocephalus oophagus Osteocephalus langsdorffii Costa et al. 2005 (via RP-HPLC, ESI-MS/MS, UV, IR, NMR)

Under 5-Methoxy-N,N-dimethyltryptamine (p. 127)
Anadenanthera falcata (BENTH.) SPEG.
5-MeO-DMT 4.655% dry wt. in seeds.
5-MeO-DMT 0.266% dry wt. in pods.
SÁVIO NUNES et al. 1982 (via GC-MS)

Under 5-Methoxy-N,N-dimethyltryptamine (p. 128)

Piptadenia gonoacantha (MART.) MACBR.

5-MeO-DMT 0.12% dry wt. in seeds. SÁVIO NUNES *et al.* 1982 (via GC-MS)

Under References:

Agurell, S. & J.L. Nilsson (1968) Acta chemica Scandinavica 22 (4): 1210-1218. "Biosynthesis of psilocybin. II. Incorporation of labelled tryptamine derivatives."

Agurell, S. & J.L. Nilsson (1968) *Tetrahedron Letters* 9 :1063-1064. "A biosynthetic sequence from tryptophan to psilocybin."

Agurell, S. *et al.* (1966) *Acta pharmaceutica Suecica* 3 (1): 37-44. "Biosynthesis of psilocybin in submerged culture of *Psilocybe cubensis*. 1. Incorporation of labelled tryptophan and tryptamine." (S. Agurell, S. Blomkvist & P. Catalfomo)

CAM (2000) "Risk assessment report relating to paddos (psilocin and psilocybin)." *Coördinatiepunt Assessment en Monitoring nieuwe drugs (Coordination Centre for the Assessment and Monitoring of new drugs, CAM*) c/o Inspectie voor de Gezondheidszorg (Health Care Inspectorate, IGZ), The Hague, February 2000. 36 pages.

Costa, T.O. *et al.* (2005) *Toxicon* 46 (4): 371-5. "Occurrence of bufotenin in the *Osteocephalus* genus (Anura: Hylidae)." (T.O. Costa, R.A. Morales, J.P. Brito, M. Gordo, A.C. Pinto & C. Bloch Jr.)

Daly, John W. *et al.* (1993) "Amphibian Alkaloids." Chapter Three in G. A. Cordell (ed.) *The Alkaloids*. Vol. 43.

Griffiths, R. R. et al. (2006) Psychopharmacology 142: 41-50. "Psilocybin can occasion mystical-type experiences having substantial and sustained personal meaning and spiritual significance." (R. R. Griffiths, W. A. Richards, U. McCann & R. Jesse) www.hopkinsmedicine.org/Press_releases/2006/GriffithsCommentaries.pdf

Guzmán, Gastón (2004) *Sociedad Mexicana de Micología* 18: 27-29. "Revision of the classification of the genus *Psilocybe*. I. Section Neocaledonicae, a new section in *Psilocybe*."

Jensen, N. *et al.* (2006) *Planta Medica* 72(7): 665-666. "Aeruginascin, a trimethylammonium analogue of psilocybin from the hallucinogenic mushroom *Inocybe aeruginascens*." (N. Jensen, J. Gartz & H. Laatsch)" Märki, F. *et al.* (1961) *Journal of the American Chemical Society* 83 (15): 3341-3342. "Dehydrobufotenine, a novel type of tricyclic serotonin metabolite from *Bufo marinus.*" (F. Märki, A.V. Robertson & B. Witkop)

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Museo dei civico Roverto is now a bad link.

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Phalaris aquatica flowering (in Victoria) Upper left *Acacia obtusifolia* phyllodes (in Victoria) Rest of page

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