



# The Journal of Gemmology



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## Cover Picture

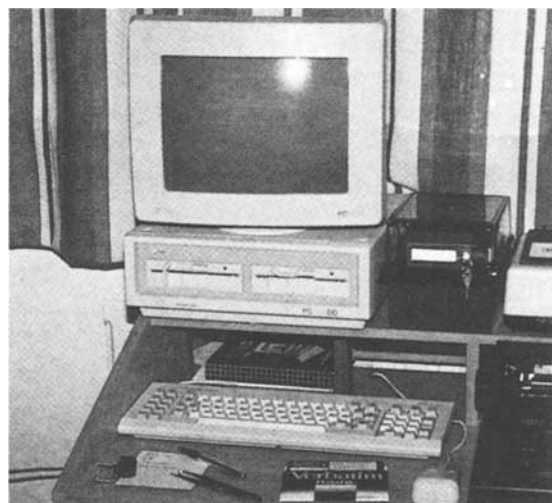
Hat ornament exhibiting the sumptuous principal stone, the  
Dresden Green, natural size. (See 'The Dresden Green', p.351.)

*Photograph by Robert Bosshart*

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## Notes from the Laboratory – 14

*Kenneth Scarratt, FGA*

The Gem Testing Laboratory of Great Britain, 27 Greville Street, London, EC1N 8SU.

For many years the Laboratory knew that at some stage it would have to obtain an instrument which 'described' the absorbance and transmittance of 'light' in a graphic form. Whilst the hand-held spectroscope had, and still does serve us extremely well in the identification of many gem species, in a laboratory it does have its limitations.

Even B.W. Anderson, the father of gemmological spectroscopy and a lover of the hand-held spectroscope, in explaining his observations had at times to resort to somewhat imaginative descriptions for some characteristic spectra, e.g. the 'organ pipe' spectrum seen in spinels.

Observations with the hand spectroscope apart from fine lines, in general, have to be described in vague terms such as, 'an area of absorption centred at approximately...' and these are more than sufficient in the identification of most gem species such as sapphire, almandine garnet, peridot, diamond, etc. One must add at this point though, that when Anderson published the wavelength of a certain absorption feature having measured it with one and confirmed it with another of the laboratory's excellent hand-held wavelength measuring spectroscopes, in general the accuracy of that measurement has proved, following checks with modern spectrophotometers, to be remarkably good.

In terms of absorbance and transmittance, many gemmologists have also tried to describe the ultraviolet 'spectrum' of certain gemstones, such as ruby, emerald and diamond, by immersion contact photography and using a short-wave ultraviolet lamp. The objective being the separation of natural from synthetic and in the case of diamond to 'type' the stone as an aid to identifying artificial coloration.

In the modern gemmological laboratory though, the problems are such with modern synthetic gems, and treatments to otherwise natural stones, that every piece of information that one can coax out of a test is needed and if we can have such a thing as a 'degree' of accuracy, then that degree must be the highest. An example of this would be the description of those two absorption lines which

may possibly be observed either side of the blue/green border (say 500nm) in some natural or treated brown green or yellow diamonds. These two lines are reported by Anderson to be situated at 504 and 497nm. The actual measurement for the '504', which is the zero-phonon\* line of the H3 centre, is 503.2nm, and that of the '497', which is the zero-phonon line of the H4 centre, is at 496nm. At first glance these small errors in measurement may seem unimportant but as the 496nm line may only rarely be observed in a naturally coloured diamond and far more often observed in an artificially irradiated and subsequently annealed diamond, the advantages of having an ability to be absolutely sure about your observations is obvious. This becomes most apparent when one realises that just to the short wave side of the '496' is what can at times be a relatively strong absorption line which is the peak of the first phonon sideband of the H3 centre. Therefore, when using only a hand spectroscope at room temperature for a diamond that has a strong H3 line at 503.2nm, one could mistake the sideband of this centre for the 496nm line. There are examples concerning other gem materials, such as ruby and emerald, where one benefits from being able to 'read' an absorption curve rather than observing dark or bright lines crossing a narrow ribbon of colours down a tube.

The laboratory's first response to the problem in the late 1970s was to install a cooling unit for the examination of coloured diamonds. The unit reduced the temperature at which the diamond was observed to 120K, at which temperature the absorption bands became sharper to the eye and were therefore easier to observe. These observations were made with the hand-held spectroscope and therefore we still had no concept of the 'shape' of the spectrum. With the problems increasing all the time it was decided that to keep ahead in the field we had to obtain a recording spectrophotometer. Having decided this and knowing the limitations governing the financing of such a

\*A phonon is defined as a quantum of sound or elastic vibrations.



Fig. 1. Nicolet model 510 FT-IR spectrometer with 620 work station, plotter and added 8" floppy drive in use at the Gem Testing Laboratory of Great Britain.

purchase, we set about researching the market. Looking towards the future we hoped to be able to obtain an instrument at a reasonable cost that would record the spectra of faceted gemstones, without resorting to destructive testing, for the ultraviolet, the visible and the infrared regions. Spectrophotometers are not built taking into account a possible requirement of having to record the spectra of faceted gemstones and therefore the search for the correct instrument was not an easy one. In fact when the choice was made we had to accept that the infrared region was going to have to be a thing for the future, as not only could we not obtain an instrument that gave us all three regions, but even the recording of infrared spectra from whole faceted gemstones with a dedicated instrument was unsatisfactory. The instrument obtained, in the end through subscription and in the memory of B.W. Anderson,\* was and still is one of the best of its type for obtaining spectra from gemstones, the Pye Unicam PU 8800 UV/visible spectrophotometer.

For some years we faced the fact that the infrared area was going to be out of our scope until a new form of instrument was developed that was more 'in tune' with the requirements of gemmology. Then problems in which the answers were held

in the infrared became more frequent. New types of hydrothermal synthetic emerald, plastic impregnated opal, synthetic diamond and the answer to those treated yellow diamonds which had been annealed to higher temperatures to remove the characteristic 595nm line are a few examples.

The Research Department of the Gemological Institute of America obtained a Nicolet Fourier Transform Infrared spectrometer and published some excellent data, and with new hope we began the search for an FT-IR unit that would be most suitable for our needs. In the end, having chosen the instrument, we were fortunate enough that one of our members came forward and bought it for us – with no strings attached. We now have a Nicolet model 510 Fourier Transform Infrared Unit (Figure 1) which will give us all the information we need in the range previously not covered.

Increasingly the author is told by gemmologists throughout the world to 'keep it simple' so that 'ordinary gemmologists' can understand what is going on. Unfortunately sometimes the black or grey boxes are the only answer to some of the problems we all face. For the past few years when describing spectra in 'Notes from the Laboratory' and elsewhere, I have reproduced absorption curves rather than trying to attempt drawing what might be seen in the hand spectroscope. There has

\*Opening of Appeal Fund, *Journal of Gemmology*, XIX, 5, 445. – Ed.

been some criticism over this change and people have told me that they cannot understand, but it really is not that difficult. If one looks down the left or right hand of the side of the curve it will be seen whether the curve was recorded in absorbance or transmittance; if it is in absorbance the sharp peaks are where dark lines will be seen and the wide bands are where areas of general absorption may be seen, in the hand-held spectroscope. Along the base of the curve are the units of measurement, which for the visible region are normally in nanometres. Apart from displaying a spectrum accurately one should not forget the obvious advantage in choosing to reproduce a curve, one can easily convert a curve to what might be seen in the hand spectroscope in the mind but it is not possible the other way around.

The move towards the black boxes in some cases may make a few gemmologists 'uncomfortable' but this latest update to the laboratory's equipment has already proved itself many times over with such problems as synthetic amethyst, synthetic alexandrite and amber. A few recent examples are detailed in the following paragraphs.

### The De Beers and Sumitomo synthetic gem diamonds

Both the Sumitomo and the De Beers synthetic gem diamonds have been reported upon in the English speaking journals, although information on the latter has not as yet been published in these pages. An excellent descriptive paper was published in *Gems & Gemology* at the end of 1987 (Shigley *et al*, 1987).

During 1988 we were given the opportunity to examine ten of the De Beers synthetic diamonds and, whilst I refer the reader to the aforementioned paper for the expanded detail, with which our information and examination mostly concurs, we shall briefly note the characteristics one might observe with the aid of a microscope or an ultraviolet lamp, before seeing how the infrared spectra of these samples help in their identification.

The samples examined ranged in size from a 0.27 ct yellow emerald-cut to a crystal of 5.24 ct and the colours were either deep brownish-yellow, yellow or greenish-yellow (Figure 2). Under the long-wave ultraviolet lamp both the fluorescence



Fig. 5. A metallic needle-like inclusion in a De Beers synthetic diamond.



Fig. 6. A colourless cross formation in an uncut De Beers synthetic diamond.

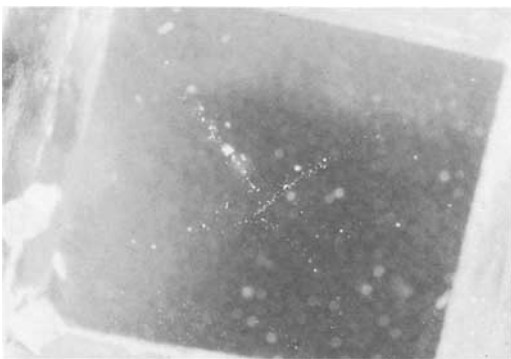


Fig. 7. Fine dust-like inclusions associated with the cross formation of the De Beers synthetic diamond of Figure 6. (Dark field).



Fig. 8. The De Beers synthetic diamond of Figure 6 between crossed polarized light.

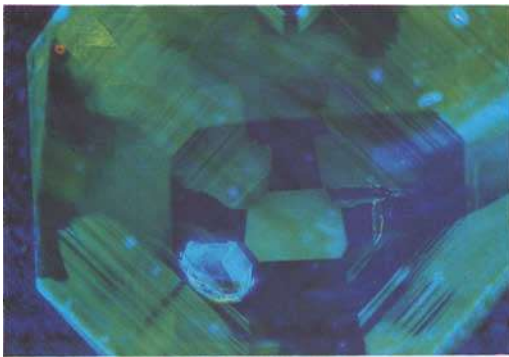


Fig. 2 (top). Faceted and uncut De Beers synthetic diamonds.

Fig. 3 (above, left). The fluorescence of a De Beers synthetic diamond when exposed to short-wave ultraviolet rays.

Fig. 4 (above, right). Fine dust-like inclusions clouding the interior of a De Beers synthetic diamond.

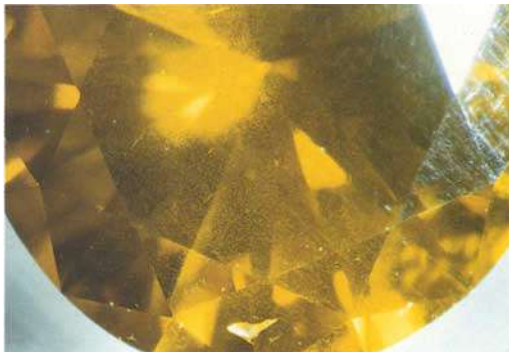


Fig. 9 (left). A natural type Ib diamond.



RED - NATURAL 'TYPE Ib' YELLOW DIAMOND ( SEE FIG.9 )

BLUE - DE BEERS SYNTHETIC TYPE Ib YELLOW DIAMOND

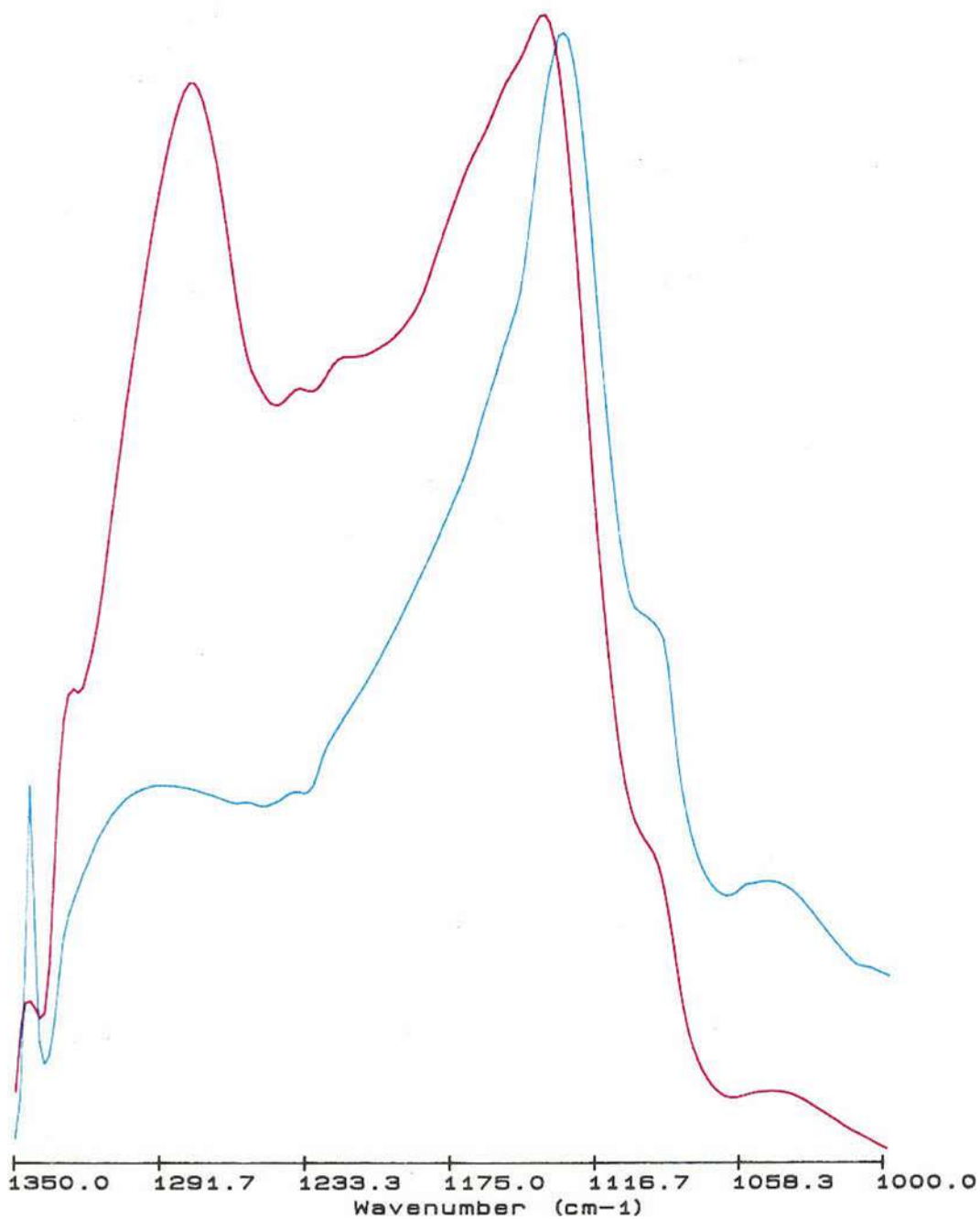


Fig. 10. A comparison between the infrared spectra of a natural 'type Ib' yellow diamond and a De Beers synthetic type Ib yellow diamond. Both curves are recorded in absorbance.

and phosphorescence varied from sample to sample. The more brownish stones were inert to this wavelength, the yellow stones tended to have a weak orange glow, with one stone being inert, and a clear phosphorescence (apart from one stone), and the greenish-yellow stones each displayed a moderate to clear phosphorescence whilst the fluorescence varied from almost inert to weak. Again under the short-wave ultraviolet lamp the response was varied. The brownish stones tended to have an orange background with distinct yellow zones which corresponded with the cubic growth sectors, (Figure 3), whilst the yellow stones were either a moderate orange with no phosphorescence or a distinctly zoned strong yellow with a strong phosphorescence. The greenish-yellow stones were either a zoned yellow or a moderate orange, but in both cases the phosphorescence was strong.

Material observed within the stones included dust-like particles (Figure 4) and metallic inclusions (Figure 5). Yellow/colourless zoning within these synthetics (Figure 6) relates to the nitrogen content in various growth sectors. When seen this zoning may serve as a first warning of a possible synthetic origin and when observed a little more closely as in Figures 6 and 7, it may be seen that tiny dust-like inclusions are orientated with this zonal structure. When observed between cross polarized light a distinctive cross-like pattern may be seen (Figure 8 [which relates to Figures 6 and 7]).

The infrared spectra observed for all of the De Beers synthetic stones were distinctive in that they revealed the characteristic peak for dispersed nitrogen (type Ib diamond) and there were no signs of the peaks associated with aggregated nitrogen (common to IaA and IaB diamonds). In natural Ib diamond it seems that almost invariably the peaks due to aggregated nitrogen are found to be present along with the dispersed nitrogen peak. This is clear when one looks at the example in Figure 9, which is a natural type Ib (canary) diamond with a visible spectrum similar to that of a synthetic yellow diamond, i.e. one of continuing and gradual absorbance towards the shorter wavelengths. The infrared curve of this stone shows a clear peak due to aggregated nitrogen (Figure 10) whilst a De Beers synthetic stone of a similar colour, as compared in Figure 10, shows only the dispersed nitrogen peak.

#### Coated amber

On two occasions recently we have been asked to identify material which has turned out to be coated amber, and infrared spectroscopy has helped in its identification. On the first occasion apart from a

single row of coated amber beads, a pair of interesting amber earclips were also submitted.

As these were not coated with an 'artificial' substance, we shall deal with the amber in the earclips first. They were a rounded cabochon shape, the metal earclip was attached to the flat side, and the colour was approaching orange. The metal fitting, being attached with an adhesive, prevented an SG determination and as there were no inconspicuous areas where an olfactory test could be facilitated this too was initially ruled out. An estimation of the refractive index by the distant vision method put this constant in the region of 1.54. The material was 'cloudy' but contained no inclusions which would assist in identification.

On the back of one of the cabochons it was noticed that a small sliver of the material had parted from the main body but was being held, in place by, and under, the metal fitting. As the sliver was not 'attached' to the main body of the 'amber', with the careful use of a fine pair of tweezers we were able to remove it. In doing so we discovered that the colour was concentrated at the surface, whilst the centre was colourless (Figure 11). The colour was not a coating of an artificial substance but was actually a part of the surface. This is a feature which we have noted before in heat treated amber (Scarratt, 1986). In order to confirm its identity a small scraping was taken from the inside colourless part of the sliver and an infrared spectrum was taken from this. The resulting spectrum was characteristic of amber. The process from taking the scraping to obtaining the spectrum (see Figure 12 (1)) took no more than five minutes.

The artificial coating on the surface of the beads in the necklace was quite obvious when examined with either a 10x lense or a microscope (Figure 13), but identifying the material underneath would have been difficult if we were only to conduct the normal gemmological tests.

The beads appeared to be an orange colour but in a number of places, around the ends of the drill holes, the coating had come away to reveal that the material beneath was coloured yellow (Figure 13). A scraping was taken from the bead underneath the coating and as revealed in Figure 12 (2) the infrared spectrum produced from this was found to be that of amber. Interestingly, upon taking this scraping we discovered that the yellow colour of the bead was also a 'surface only' colour. The bead was colourless underneath, it had a yellow surface probably due to heat treatment, and on top of this was an artificial coating which was orange in colour.

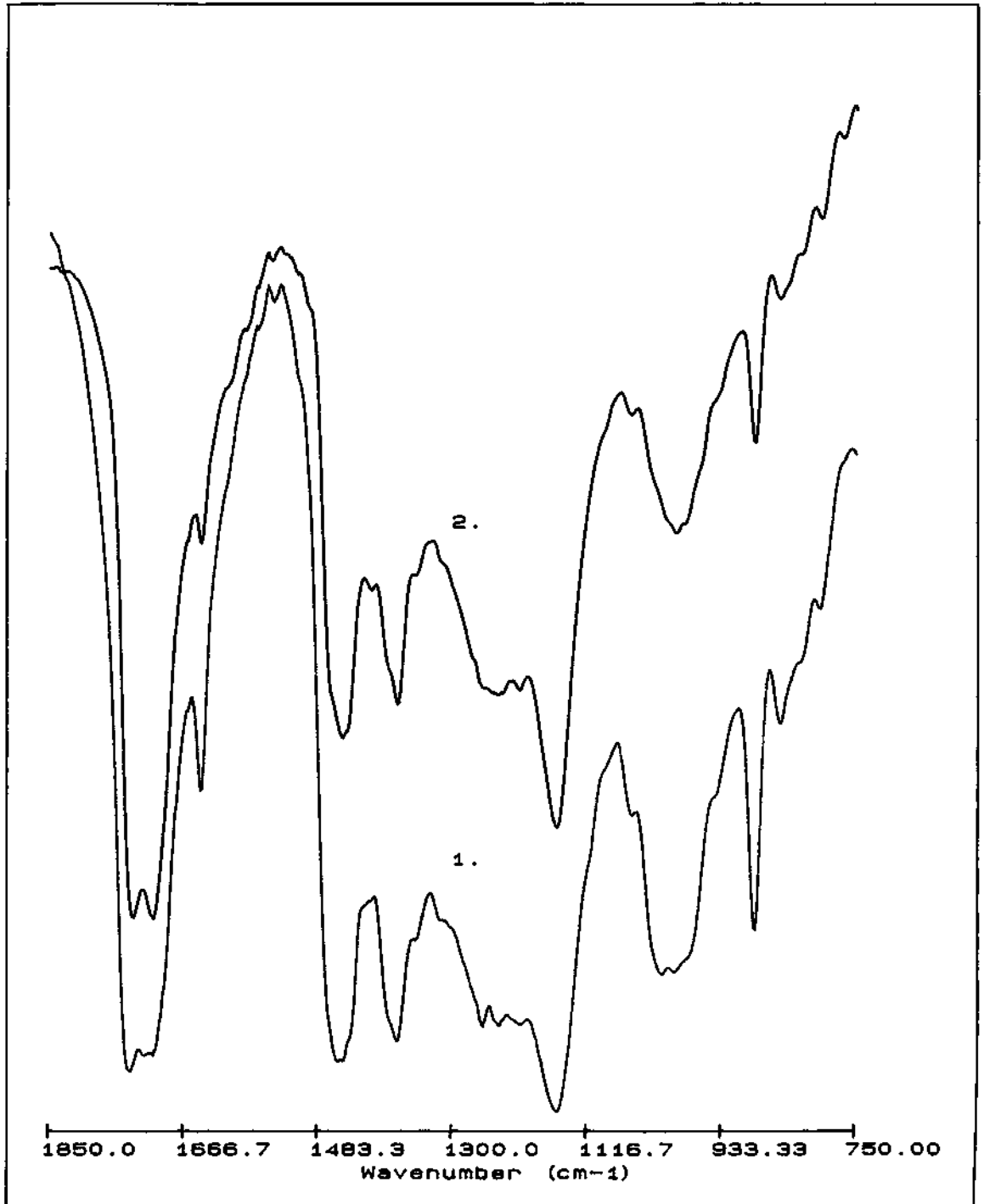


Fig. 12. The infrared spectra of two pieces of amber both recorded in transmittance. (1). An amber earclip, and (2) an amber bead which had an artificial coating (the scraping being taken from the bead below the coating).

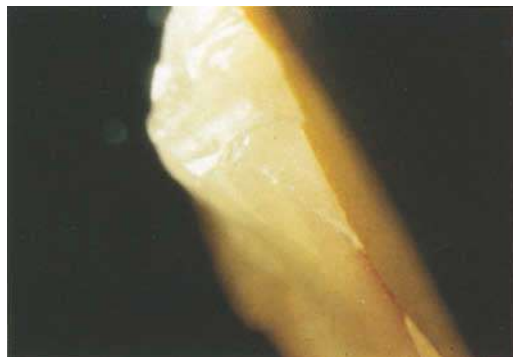


Fig. 11. A small sliver of amber removed from an earclip showing the colourless interior and coloured exterior.



Fig. 13. A coated amber bead showing an area where the coating has come away.

#### Naturally coloured green Type IIb diamond

We mostly think of type IIb diamonds as being blue in colour although colourless and colourless/brown zoned stones have been reported. A short time ago an octagonally shaped faceted diamond weighing 5.80 ct was submitted for examination (Figure 14). The colour of the stone was a little difficult to describe, however: in a standard diamond grading light box grey, brown and green colours could be seen; under a bench lamp fitted with a tungsten bulb just green and brown could be seen; and in the normal laboratory lighting, i.e. daylight fluorescent tubes, both grey and green could be seen. The predominant colour appeared to be green in most instances but in some circumstances brown predominated.

Apart from making observations on the colour and examining the stone with the aid of a microscope, the first test carried out on the stone was the recording of its visible spectrum. The type of spectrum we were expecting, judging from the part



Fig. 14. A green/brown (colour depending upon lighting) type IIb diamond. 5.80 ct.

brown colour, was either that typical of a brown type Ia stone or that of a Ib stone. The result was a little like a type Ib curve, with increasing absorption for the shorter wavelengths from about 520nm but with a wide absorption 'hump' stretching from 520 to 460nm and peaking at 490nm. The area of least absorption was in the green, as might be expected, but then unlike Ib stones the absorbance level begins to rise again for the longer wavelengths from 620nm. The level of absorbance at 750nm being slightly more than half that at 390nm.

The spectrum, in which there were no sharp peaks, did not fall into a convenient category, but the next test gave us a clue to the type of stone we were dealing with, and it was far from what we were expecting. Under both long-wave and short-wave ultraviolet light the stone appeared to be inert but when the short-wave lamp was switched off the stone displayed a strong blue-green phosphorescence. This being a typical characteristic of type IIb blue diamonds, we then tested the stone for electro-conductivity. Apart from confirming that the stone was a conductor of electricity, the test proved to be quite dramatic with blue flashes coming from within the stone almost from the instant that contact was made.

The infrared spectrum was then taken and compared with other type IIb spectra filed in the computer. The spectrum of this stone was the same as those recorded previously for type IIb blue stones.

#### References

- Scarratt, K., 1986. Notes from the Laboratory - 6. *Journal of Gemmology*, 20, 2, 95.  
 Shigley, J.E., et al., 1987. The gemological properties of the De Beers gem-quality synthetic diamond. *Gems & Gemology*, 23, 4, 187-206.

[Manuscript received 25 January 1989.]

# Proceedings of the First International Amber Symposium

*Helen Fraquet, FGA*

London



Fig. 1. Natural freeform amber necklace with pressed amber rods, pendant and bead.

## Summary

Sixty participants from Europe, USSR, Africa and USA, were invited to the first International Amber Symposium, hosted by the Museum of the Earth, Warsaw, in October 1988.

Twenty-seven papers were offered, covering all aspects of the sciences and arts in relation to this fossil resin, in four working languages – German, Russian, English and Polish.

## Introduction

This was in fact the sixth meeting on Amber and Amber-bearing sediments to be held in Poland, but the first of an international nature. Accordingly, much of the Symposium explored areas of future mutual exchange of samples and scientific papers. The first two papers – ‘The present status of research of amber and other fossil resins’ (Saw-

kiewicz, S., Leningrad) and 'The significance of museum collections of amber and other fossil resins' (Kosmowska-Ceranowicz, B., Warsaw), explored the possibilities opened up by the global political thaw of the late 1980s, especially with regard to re-examining existing collections, many of which had been transferred across Europe during the progress of the two World Wars.

By cross-referencing experiences it became obvious that an extensive catalogue of the amber contained in various national collections would be a great step forward for both those working as art-historians and in the sciences. This is especially so, as many former great collections are now split between various museums. For example, that of the Albert University in Königsberg (Kaliningrad) which once comprised 70 thousand specimens, is thought to be split between the University Museum in Goettingen, the Natural History Museum of Humboldt University in Berlin, and the British Museum of Natural History in London. During the course of the conference it was learned from Dr Katinas, that his nation's Lithuanian Amber Museum at Palanga would be opened to the West for the first time.

#### New finds

Several new sources of amber were reported during the conference. Kosmowska-Ceranowicz and Krumbiegel, G. (Halle), presented a joint paper on Bitterfeld amber, a Miocene deposit (22 million years old) mined near Leipzig in East Germany, of which a proportion goes into the production of jewellery. The amber shows the usual colour range, clear and cloudy, etc. Sokolowa, T. (Leningrad), and Rasnitsyn, A. (Moscow), described four types of Cretaceous amber recovered from field trips to Northern Siberia and the Arctic Sea in the 1970s. Some of the pieces appear to be similar to gedanite, and all are too fragile for decorative purposes, although the inclusions are naturally of great interest to paleo-entomologists and botanists. A third find of Upper Cretaceous amber was also reported from Alberta in Canada.

Regional updates were offered from Romania, Sicily and the Dominican Republic, as well as from areas less familiar to the gemmologist such as Bulgaria and Nigeria. This last is an Eocene resin with a chemical structure shown by IR spectra to be a new species. It has been given the name Amekit. Perhaps the most obscure piece of amber to be discussed was a single nodule found at a depth of 2,302 m below sea level during the course of deep drilling in the Persian Gulf. Its infrared spectrum is identical to that of Rumanite from Romania. Several of the core samples contained this resin, but only one sample was analysed.

Schluter, T. (Dar es Salaam), gave a comprehen-

sive paper about East African copal – and confirmed the author's view that very little copal has ever been used as jewellery.

Migaszewski, Z. (Kielce), showed current investigation into black resins, jet and the new appearance on the market of black amber jewellery. The latter is being produced by a Co-operative south of Warsaw with a source material of processed succinite. The streak mark left by this manufactured substance is black, the lustre is good, and under a microscope there are to be seen two distinct materials – dark grey particles cemented by a very fine grained light grey substance.

The Museum of the Earth in Warsaw has two samples of stantienite collected from the Palmnicken deposit in 1880. These have a velvet black colour, a brown to light brown streak, and under the microscope a structureless groundmass of dark grey containing 'yellow-orange and blood red inner reflections.'

Also in the collection are two samples of soft fossil resin from Lower Miocene deposits of the Ukraine and Bitterfeld. The microscope reveals a structureless, porous cracked lustreless resin of black colour yielding a dark greyish-brown streak. The samples have the same reflections as above with the addition of scattered fragments of crushed plant tissue.

A third type of black resin is represented by a sample from Bitterfeld and another from Bytow in Poland, whilst there are at least three petrographic varieties of jet assigned to the Lower Jurassic from Odrowaz in Poland.

Black resins were thought in the nineteenth century to originate from fire in the forest at the time of formation. However, the authors of this 1988 paper consider lightning to be a more likely theory.

#### Other scientific papers

The majority of those present at the Symposium represented the sciences of geology, mineralogy and paleontology. However, many of the papers proved interesting across traditional boundaries. To this end gemmologists should find useful 'Comparative studies on micro-elements in amber' from Koziorowska, L. (Warsaw), which included data such as the presence of silver amongst Baltic amber, but not that from Sicily or the Dominican Republic. Also 'Gas chromatography – an effective tool for chemical characterization of fossil resins', Vavra, N. (Vienna). This may prove to be a rapid method of investigation, which already differentiates samples from within the Dominican Republic. 'The presence of vertebrates and their remains in amber', Poinar, G. (Berkeley), should also be required reading for all those faced with abnormally large inclusions brought in by members of the public for verification.



Fig. 2(a)



Fig. 2(b)

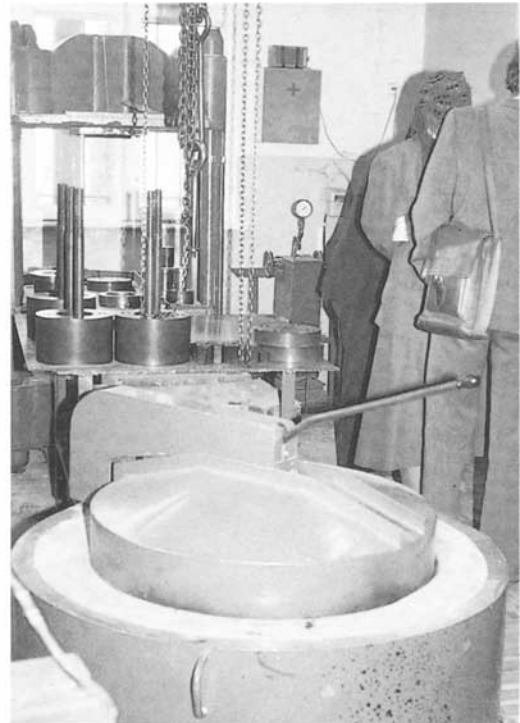


Fig. 2(c)

Fig. 2 (a, b and c) Interior of the Bursztyny amber co-operative, Gdansk, Poland: (a) visiting the grinding and polishing room; rods of pressed amber awaiting despatch; (c) the production area for heating and pressing amber into rods (the same equipment is used for producing amber with stress-induced inclusions).

### Field Expedition to Gdansk and Malbork

The two days of presentations were followed by a weekend field trip to the coast. In Gdansk the party was able to join those working to extract amber from marshland with the aid of hydraulic hoses. The amount of amber washed out from the reed-bed deposit was surprisingly high, and paid the wages of ten people on that site alone. The land was owned by a nearby factory and had been leased by an amber processing co-operative in the town of Gdansk (Figure 2).

The co-operative was open to our detailed questioning – the operatives making round and faceted beads on simple rotating laps had been thus employed for up to 22 years, and coloured rods of pressed amber had been manufactured in black, green, red, sand, etc., as well as the usual two-tone swirled syrup since 1981. Although the author's overall feeling was one of depression, at least the larger, and purer specimens of amber were sorted out, after an initial clean, and reserved on one side not to be pressed but to be used without further processing. Although not seen, the factory apparently produced sun-spangled amber from time to time, using different time and pressure ratios.

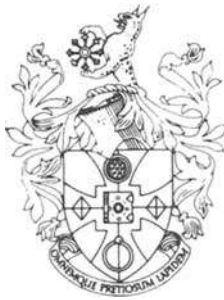
There is much amber jewellery for sale in Gdansk and in the larger international hotels. It is expensive, and disappointingly unexciting. Very little is polished freeform (as is the necklace illustrated in Figure 1), most being non-graduated or graduated

round or oval beads. The major colours represented are bright yellow (natural) or syrup/brown (pressed). There are some animal carvings of elephants, tortoises, etc. One exception to this general dull conformity was the use of thin-slab amber with silver in tie-clips, and brooches by a single shop in Gdansk. The lack of general flair is understandable given the isolated nature of the country. However, one is left with the impression that Poland is rebuilding, despite the system.

The hosts of this meeting in Warsaw only started collecting amber in 1951, yet today house one of the largest hoards of natural resin in Europe. The Archeological Museum in Gdansk overflows with amber finds made in the last few years, and the National Collection of Amber Artefacts at Malbork Castle already occupies a whole gallery. There is an infuriating lack of pin-point dating at the latter two institutions, and one or two gross errors – for example a whole display of phenol formaldehyde plastic beads, described as Sicilian Amber, but the academic enthusiasm is contagious.

Thanks must be given to all those who made this Symposium possible. Summary papers are available from the author, and it is hoped that before too many years elapse Britain will herself come forward to host such an event.

[Manuscript received 26 January 1989.]



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# The Dresden Green

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## Introduction

*Once upon a time there lived a king. He was tall, strong, and immeasurably wealthy since his ancestors were already in possession of productive silver mines. He did not waste his treasures on foolish wars. On the contrary, he accumulated riches in the safe vaults of his castle of such vastness that his kingdom became renowned and was admired far and wide.*

This is not the beginning of a legend but a sketch of what happened in the seventeenth and eighteenth centuries prior to and during the reign of the Saxon Elector August II, also named *August the Strong* (with the exception of the wars). His residential cities were Dresden and, after his coronation as King Frederick August I of Poland, also Warsaw. The accumulated riches of gold, silver, jewels, curiosities, and in particular gemstones, were arranged and stored below his residential palace in vaults painted green. These rooms soon came to be known as the *Green Vaults* (Grünes Gewölbe). They served as strong-rooms and, in anticipation of a coming era, also as a treasury museum, fitted as an elegant French-style mirror cabinet (1723-1724). In the following years, the copper-plate engraving cabinet, the gallery of paintings, the collection of porcelain and the collection of natural sciences were founded and exhibited in the pavilions of the Zwinger, stronghold of the former fortress. (Menzhausen, 1987a, 1988.) For good reasons, Dresden was known as the Florence of the Elbe River.

In 1741, the son of August the Strong, Elector August III of Saxony (since 1733 King Frederick August II of Poland), bought one of the rarest diamonds from a Jewish dealer. Together with the Diamond Suite (Copeland, 1966, 29), the green gem was registered as No. 50 in the inventory of 1733 (cf transcription below Figure 1). From then on, the diamond was known by the name of 'Dresden Green Diamond' or shorter 'Dresden Green' and 'Green Brilliant' (Table 1). In 1742 this brilliant-cut stone was mounted into the Order of the Golden

Fleece by the Dresden court jeweller J. F. Dinglinger. However, in 1746 it was reset 'à jour' by the Geneva jeweller A. J. Pallard at the court of Vienna (open-back bezel setting as in Figure 3) and in 1768 finally mounted by Diessbach into the hat brooch known to the present day (Figure 2).

The Dresden Green remained in the Green Vaults for over two centuries until, due to the Second World War, all Art Collections of Dresden were moved up to the Königstein (King's Rock), the imposing grey mountain fortress at the entrance to the Elbsandsteingebirge (Sandstone Sierra of the Elbe River region). From there the Soviet Trophy Organisation (Copeland, 1966, 25) transported the Collections into the subterranean safes of the USSR Ministry of Finance at Moscow (Menzhausen, 1987a) shortly after the war. They were handed out to the German Democratic Republic in 1958. Since then the Collections have been conserved and displayed mostly at the Albertinum and in the Zwinger. The State Art Collections of Dresden as much in their totality as also in their parts, e.g. the Saxon-Polish Crown Jewels of the Green Vaults or the Meissen Porcelain, are unique worldwide, but surprisingly little known, even in Europe.

It has come to be considered the destiny of many famous diamonds that their histories remain unknown or fragmentary at most. In the case of the Dresden Green this concerns its trade route before 1741 and the sojourn from 1945 till 1958. It is assumed that the stone originated in India rather than in Brazil.

The rarity of the Dresden Green is based not so much on its weight, which is over 40 ct, but rather on its green colour. In reality however, it is the coincidence of both properties that makes the diamond so exceptionally rare. Up to the present day, it has remained the largest cut diamond of natural green colour on record. Over the centuries it has not lost anything of its celebrity status. But on the contrary, it has become of increasing cultural interest and, recently, also of great scientific value.

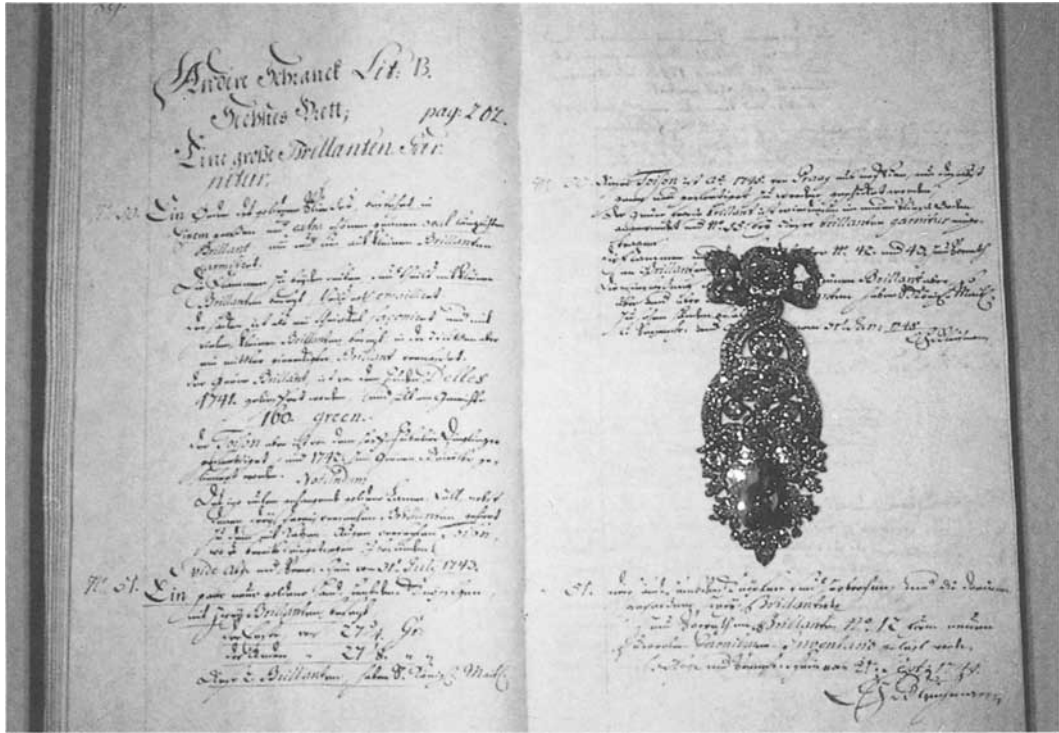


Fig. 1. Hat ornament (brooch) belonging to the Diamond Suite of the Elector August III of Saxony, displayed on the original volume of the 1733 Inventory of the Green Vaults.

Transcription of the Registration No. 50 in the Inventory (written in elderly chancery language, Pfister, 1988) [Seite] 89. Andere Schranck Lit. B.

Sechstes Brett: pag: 202.  
Eine große Brillanten Gar.  
nitur.

- Nº. 50 Ein Orden des goldenen Vlieses bestehet in einem großen und extra schönen grünen oval länglichten Brillant um und um mit kleinen Brillanten carmisiret. [umrandet, eingefasst] Die Flammen zu beyden Seiten sind theils mit kleinen Brillanten besetzt, theils roth emailirt.  
Der Haken ist als ein Schnirkel façoniret und mit vielen kleinen Brillanten besetzt. In der Mitten aber ein mittler viereckigter Brillant verfaßet.  
Der grüne Brillant, ist von dem Juden Delles 1741. geliefert worden, und hält am Gewichte 160. green. [von engl. grain?, ca 40 ct]  
Der Toison aber ist von dem Hoff-Jubelier Dinglinger gefertigt und 1742. zum grünen Gewölbe geliefert worden [Watzdorf 1962]

#### Notandum

Das izo unten anhangende goldene Lamm-Fell, nebst denen drey darin versetzten Brillanten gehört zu dem mit Katzen-Augen versetzten Toison, wo es bereits eingetragen zu befinden. vide abg. und Verw. Schein vom 31. July 1743. [Seite] 90.

- Nº. 50 Dieser Toison ist aº. 1745. von Praag aus nach Wien, um daselbst ganz neu gefertigt zu werden geschicket worden, Der grüne große brillant ist wiederum im neuen Vließ Orden angewendet und [als] Nº. 55 bey dieser brillanten garnitur eingetragten. [Neufassung durch den Genfer J. Pallard, 1746] Die Flammen und der Schnirkel [Schnörkel] aber Nº. 42 und 43. zum Vorrath an Brillanten geleyet worden. Die Einfassung um den großen grünen Brillant aber, so über und über mit kleinen brillanten, haben S. Königl. Mait. zu hohen Händen behalten.  
C. Verwahr. und abg. Schein vom 31. Jan: 1748.

C. G. Cleichmann. [?]

- Nº. 51 Ein paar goldene Hand-Hembden-Knöpfgen mit zwey Brillanten besetzt...

**Table 1. Physical Properties of the Dresden Green Diamond**

CUT	Pear-shape with rounded tip (almond-shape), historic brilliant-cut style (pear-shaped old-cut brilliant)		
Measurements of diamond	Length $\times$ Width $\times$ Height approx. 30.30 $\times$ 20.35 $\times$ 10.288mm (TESA dial calipers and micrometer)		
	Table T	approx. 16.30 $\times$ 10.15 mm (SSEF)	
	Culet C	approx. 3.25 $\times$ 1.65 mm table gauge)	
Proportions	Table width/Width	49.9%	
	Culet width/Width	8.1%	
	Height/Width	50.6%	
	Crown height/Width	approx. 17% (estimated)	
	Pavilion height/Width	approx. 33% (estimated)	
	Girdle width/Width	approx. 0.5% (where visible), i.e. very thin to sharp-edged	
	L / W	approx. 3 / 2	
	H / W	approx. 1 / 2	
	TW / W	approx. 1 / 2	
	TW / TL	approx. 1 / 1.6	
	CW / CL	approx. 1 / 2	
	Cr / Pv	approx. 1 / 2	
Symmetry	Good to very good: no large remnants of crystal faces ('naturals') or extra facets present, very sharp facet corners; bezel and pavilion (upper and lower main) facets in part slightly distorted, girdle slightly wavy		
Polish	Good to very good: table facet perfectly plane; some light wheelmarks		
Measurements of bezel	Gold bezel	L $\times$ W	approx. 30.90 $\times$ 20.60 mm
		Height	1.90 to 2.40 mm (without prongs)
	Silver bezel (pavilion)	Height	3.30 to 3.80 mm
		Thickness	0.48 to 0.51 mm
Condition	Girdle of diamond mediocre: numerous small breakages, girdle projecting over the gold bezel on almost half of the circumference (from right to top) and extremely thin.		
	Bezel setting good: excepting a fine-grained layer on the gold bezel at top right, on right side and bottom left (Figure 3, remainders of tin solder rather than oxidized silver solder; no patina)		
WEIGHTS of the Dresden Green	A1	approximately 41.1 ct (author's calculation by means of similarly proportioned pear-shaped old-cut brilliants as length $\times$ width $\times$ height $\times$ 0.0065)	
	A2	approximately 43 ct (too high; calculation on the basis of the conversion factor 0.00678 for a loose pear-shaped old-cut of 13.24 ct of the Green Vaults)	

	B1	41 ct (Green Brilliant, Menzhausen 1987a)
	B2	Dresden Green, identical with Green Brilliant, i.e. 41 ct (Menzhausen, 1987b)
	C1	41.00 ct (Dresden Green, De Beers, 1983, 60)
	C2	40.00 ct (Green Brilliant, De Beers, 1983, 60), citation of erroneous indications in the Anglo-Saxon literature (Copeland, etc.)
	D1	48½ ct (Bauer, 1896, 157), contradiction to D2
	D2	40 ct (Bauer, 1896, 287: 'not 31¼ or 48 carat')
	E	160 green (Inventory volume 1733), [green = engl. grain (?), between 40 and 41 ct]
Weight including bezel setting		11.182 g (55.91 ct) (Owa laboratory balance 160 g max., e = 0.01 g)
<b>PURITY</b>		
External characteristics		One small extra facet at the tip of the pavilion, one very small natural near the girdle on the right of the crown side
Blemishes		Numerous small to tiny breakages along the girdle on the crown and pavilion side, one short curved scratch on the left in the table, some slightly abraded facet edges on the crown and pavilion side (Hänni and Bosshart, 1987)
Internal characteristics		One small, concentrically ribbed tension fissure of a whitish appearance and similar to a healing crack and one shallow and short fissure with green specks (Figure 7), both on the left of crown near the girdle; very short tension fissures with green spots on the right of crown at the girdle, one group of tiny, brown reflecting opaque crystal inclusions (chromian spinels?) near a lower girdle facet on the right (approx. 0.15 mm deep)
Growth characteristics		Weak to distinctive, lamellar zoning in triangular octahedral arrangement (three-point diamond)
Strain birefringence		Fine, cross-hatched and pronounced, lamellar extinction pattern in cross-polarized light ('Tatami' type, Orlov, 1977, 116), no interference colours
Damages		One small tension fissure emanating from a girdle chip on the right crown side, one tiny percussion mark in the bezel at the tip
Purity grade		SI (small inclusions, CIBJO Rules 1986), theoretical possibility of improvement of the purity and girdle by a professional repolishing operation.
<b>COLOUR</b>		
Description		Transparent, non-fluorescent slightly bluish-green of weak saturation
Colour grade		Fancy colour

Classification	After the Colour Atlas DIN 6164 (Biesalski, 1957) as colour index triplet «hue : saturation : greyness» body colour K (in transmitted light) $\approx 21 : \frac{1}{2} : 1$ reflection colour R (in reflected light) $\approx 21\frac{1}{2} : 3 \pm 2 : 3$ reflection colour R (Rösch, 1969) $\approx 22 : 2-3 : 3$																																													
Colour distribution	Apparently homogeneous body coloration (no colour zoning, mottled or surface colour)																																													
Colour type	Natural coloration (Menzhausen, 1987b)																																													
Cause of coloration	Light absorption (GR 1-8) at a limited number of crystal lattice defects, generated by a continued natural radioactive irradiation in the diamond deposit; annealing in the parent rock at a maximum of 350°C																																													
Colour stability	Possibility of fading of the green coloration by thermal healing of the GR 1-8 lattice defects starting at about 500°C (Woods and Collins, 1986)																																													
UV Fluorescence	Weak, impure yellowish-green, homogeneous (VEB Quarzlampe markkleeberg UA 150.1, 365nm/140W/10cm)																																													
ABSORPTION	<table border="0"> <tr> <td>UV/VIS</td> <td>GR 1</td> <td>very weak absorbance band from 750 to 530 nm, with medium strong zero-phonon doublet (Collins, 1982) at 741.0 and 744.3 nm</td> </tr> <tr> <td></td> <td>667</td> <td>very weak line at 666.6 nm (after Davies, 1977 stable below 700K/430°C)</td> </tr> <tr> <td></td> <td>594</td> <td>extremely weak band at 594 nm (appears above 275°C only, Woods and Collins, 1986)</td> </tr> <tr> <td></td> <td>495</td> <td>very weak band at 495 nm</td> </tr> <tr> <td></td> <td>473</td> <td>very weak band at 473 nm</td> </tr> <tr> <td></td> <td>TR 12</td> <td>very weak line at 470.1 nm</td> </tr> <tr> <td></td> <td>GR2-8</td> <td>weak to extremely weak lines at 430.4, 429.5, 419.1, 413.2 nm, etc.</td> </tr> <tr> <td></td> <td>R 10</td> <td>very weak line at 393.5 nm</td> </tr> <tr> <td></td> <td>R 11</td> <td>weak line at 310.8 nm</td> </tr> <tr> <td></td> <td>306</td> <td>very weak line at 306 nm</td> </tr> <tr> <td></td> <td>303</td> <td>very weak line at 303 nm</td> </tr> <tr> <td></td> <td>289</td> <td>very weak line at 289 nm</td> </tr> <tr> <td></td> <td>279</td> <td>extremely weak band at 279 nm</td> </tr> <tr> <td></td> <td>225</td> <td>steep and high absorption edge at 225 nm</td> </tr> <tr> <td></td> <td>MIR</td> <td>No specific absorption bands in the middle infra-red region (4000 to 400 <math>\text{cm}^{-1}</math> / 2.5 to 25 <math>\mu\text{m}</math>) except the strong lattice absorption present in all diamonds between 2650 and 1400 <math>\text{cm}^{-1}</math> (Davies, 1977)          → TYPE IIa</td> </tr> </table>	UV/VIS	GR 1	very weak absorbance band from 750 to 530 nm, with medium strong zero-phonon doublet (Collins, 1982) at 741.0 and 744.3 nm		667	very weak line at 666.6 nm (after Davies, 1977 stable below 700K/430°C)		594	extremely weak band at 594 nm (appears above 275°C only, Woods and Collins, 1986)		495	very weak band at 495 nm		473	very weak band at 473 nm		TR 12	very weak line at 470.1 nm		GR2-8	weak to extremely weak lines at 430.4, 429.5, 419.1, 413.2 nm, etc.		R 10	very weak line at 393.5 nm		R 11	weak line at 310.8 nm		306	very weak line at 306 nm		303	very weak line at 303 nm		289	very weak line at 289 nm		279	extremely weak band at 279 nm		225	steep and high absorption edge at 225 nm		MIR	No specific absorption bands in the middle infra-red region (4000 to 400 $\text{cm}^{-1}$ / 2.5 to 25 $\mu\text{m}$ ) except the strong lattice absorption present in all diamonds between 2650 and 1400 $\text{cm}^{-1}$ (Davies, 1977) → TYPE IIa
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RADIOACTIVITY	No residual $\beta/\gamma$ -activity observed (background 0.2 counts/sec on Geiger counter EMA GZ25/VA-Z125)																																													
CONDUCTIVITY	Thermal conductor (EICKHORST Thermolyzer II), electrical insulator (voltmeter)																																													
DIAMOND TYPE	IIa																																													



Fig. 2. Hat ornament exhibiting the sumptuous principal stone, the Dresden Green. Magnification 0.8x

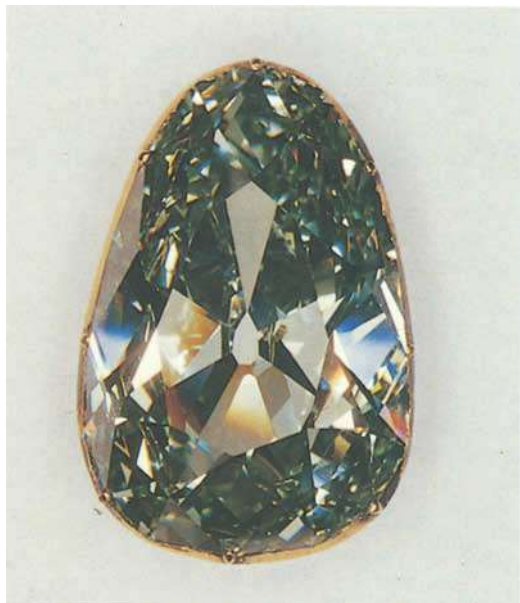


Fig. 3. The Dresden Green set in an eighteenth century gold (and silver) bezel and scintillating in all spectral colours. Magnification 3x.

### Modern investigations of the Dresden Green diamond and interpretation of the results

Motivated by the contributions of Collins (1982) and Orlov (1977) on the one side and by Bauer (1896) and other writers of the past century on the other, the author applied for permission two years ago, to investigate the Dresden Green extensively by modern, non-destructive gemmological and analytical methods. A unique authorization was granted from 22 until 24 November 1988 to him as well as to R. E. Kane and S. F. McClure. These colleagues of the Gemological Institute of America jointly and shortly will report on the history and gemmological properties of the diamond and will publish professional colour reproductions.

#### Colour Testing

In addition to the urgently needed characterization of such a prominent diamond individual by weight, measurements, proportions, etc., the intention was to find out whether the Dresden Green belonged to the 'relatively rare, uniformly bottle-green' diamonds (according to Orlov) or whether it possessed, as Bauer put it, a 'very nice, light apple-green' colour (or even an 'oil-green, yellow-green, pale, leek, asparagus, pistachio, olive, siskin, emerald, bluish or greyish-green colour?') The question is what sort of bottles and apples they meant. According to the Identity card of the Diamond (Table 1), the colour hue is definitely a bluish-green.

Collins (1982) confirmed that uniformly green (by this he did not mean surface-coloured but body-coloured green) diamonds are so rare that up to now no corresponding absorption spectra existed in literature. According to Collins, however, these spectra would be of scientific importance to solid state physicists for the clarification of the natural causes of green coloration (e.g. irradiation), as a possible precursor of yellow and brown colorations (annealing during or subsequent to irradiation).

In addition it would be a breakthrough of commercial significance if specialized gemmological laboratories henceforth would be in a position to differentiate between diamonds of natural and artificially caused green colour. At present few laboratories succeed in reliably identifying these colorations and then only in exceptional cases. For these reasons the author has pursued his goal for over three years now (outside the daily routine analysis of gemstones) to trace cut diamonds of guaranteed naturally green colour - this proved to be unusually difficult - and to measure them accurately. The research work will come to a close soon and will then be published (Bosshart, 1989).

#### Absorption Analysis

In some of the recognized gemmological laboratories it has been standard practice for more than

ten years now to record the absorption spectra of coloured diamonds applying refrigeration by liquid nitrogen (boiling temperature of  $\text{LN}_2$  at atmospheric pressure:  $-196^\circ\text{C}/77\text{K}$ , where Kelvin (K) = Celsius ( $^\circ\text{C}$ ) + 273.16). The advantages of this procedure are absorption diagrams which give much increased evidence compared to the spectra registered at ambient temperature (Collins, 1982), as is proved by Figure 4:

**Survey spectra:**

upper blue curve – medium refrigeration

lower blue curve – weak refrigeration

red curve – ambient temperature

(horizontal red line: baseline of spectrophotometer and cryogenic cell)

Figure 5 presents the well established cooling cell of the bath type developed by the author. In contrast to the cryogenic flow type devices of other laboratories cooling the diamond with nitrogen gas crossing the spectrometer sample beam at right angles (Scarratt, 1979, and Hofer and Manson, 1981), the Dresden Green was mounted in the  $\text{N}_2$ -vapour atmosphere of the illustrated highly-transparent cooling cell (i.e. above the liquid nitrogen bath) in such a manner that the diamond could be traversed by the light beam on a linear optical path.

It was and is a favourable and rather unusual circumstance that the Dresden Green, still mounted in its bezel setting, possesses a large culet facet parallel to the table facet. The optical path length between table and culet, however, was the absolutely shortest of the stone, corresponding to the weakest possible absorbance (cf Figures 3 and 6). On the other hand, the light traverse in the long or diagonal directions of the diamond was rendered practically impossible by the silver bezel hiding the pavilion of the diamond just below the girdle.

The 'Dresden Green Project' was consented to only after the author's party had furnished physical evidence that the necessary refrigeration (and subsequent warming up) could cause damage neither to the diamond nor its green coloration nor the bezel setting (Rosenfeld and Bosshart, 1987, etc.). Despite the author's many years of experience in refrigerating large and small, mounted and loose diamonds, there remained a residual risk which needed a little courage to overcome since doubts about the innocuousness of the project for the famous large diamond had been expressed in some gemmological circles. Although the present investigation has been completed successfully and without detriment to the diamond and its setting, this still must not be interpreted as an encouragement to deep-freeze gemstones unscrupulously as certain significant conditions have to be fulfilled.

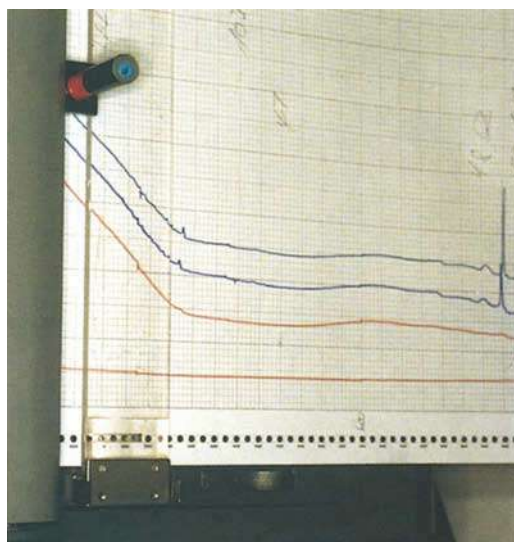


Fig. 4. The first three absorption spectra of the Dresden Green Diamond showing lesser spectral information in the red curve registered at ambient temperature than the spectra run at freezing temperatures (blue curves). Baseline of spectrophotometer and cryogenic cell (horizontal, red) and absorption curves vertically displaced for better understanding.



Fig. 5. The Dresden Green mounted in the  $\text{N}_2$  vapour atmosphere of the highly transparent cooling cell and adjusted in the sample beam of the Pye Unicam SP8-100 UV/VIS spectrophotometer prior to the first absorption measurement in refrigeration.

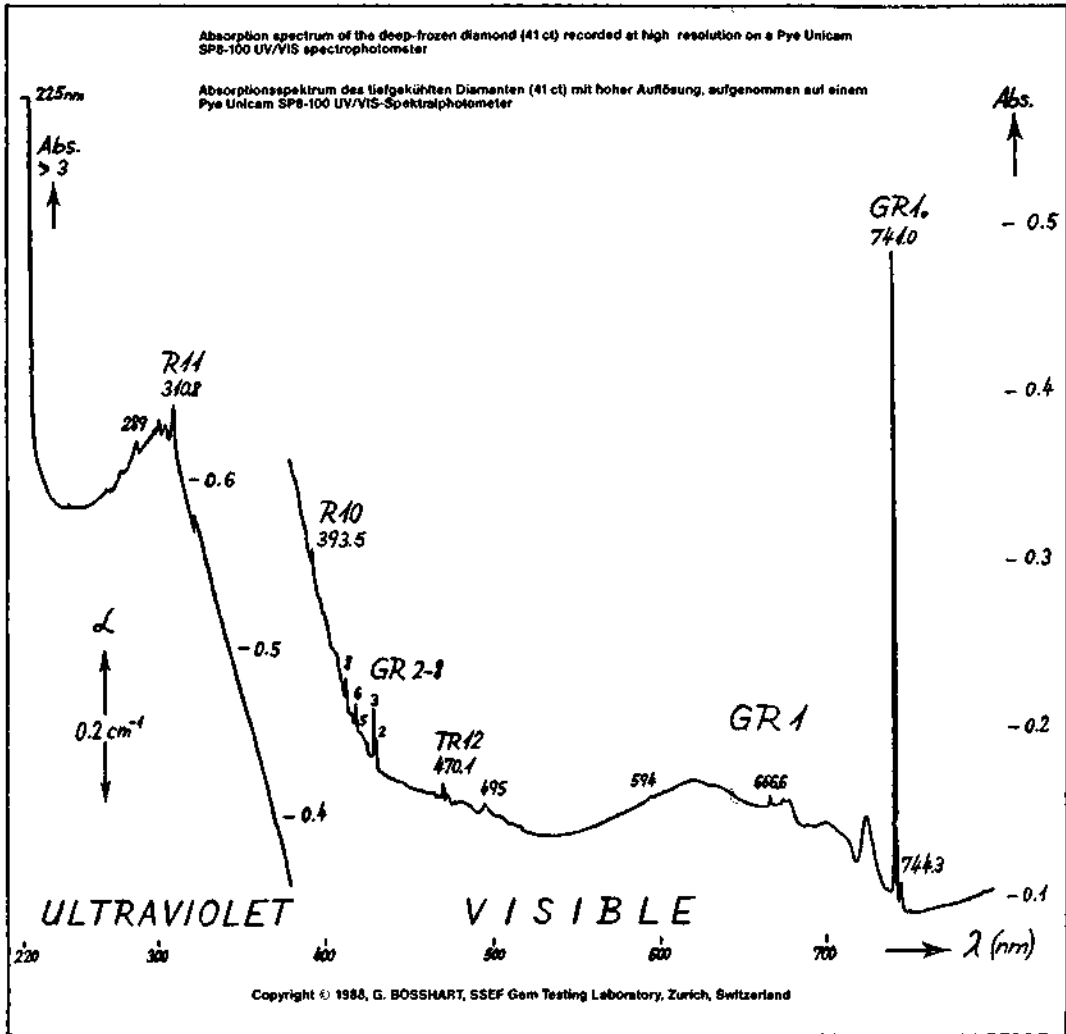


Fig. 6. High-resolution absorption spectrum of the refrigerated Dresden Green Diamond recorded on a Pye Unicam SP8-100 UV/VIS spectrometer at four-fold gain and slow wavelength scanning and chart advance speed. Optical path length from table to culet 10.288 ( $\pm 0.005$ ) mm. Classical absorption behaviour of an irradiated type IIa diamond.

Main spectral characteristics are the bands of the GR 1 absorption system (750 to 530 nm, present in all irradiated diamonds) and the GR 2-8 system. The wide absorption band between 440 and 250 nm is characteristic of irradiated type IIa diamonds, the chemically nearly pure form of natural and synthetic diamond. The absorption line R 11 occurs only in type IIa diamonds (Davies, 1977). The exceptionally steep and high absorption edge of the Dresden Green is situated immediately below 225 nm in the short-wave ultraviolet spectral region.

The absolutely lowest absorbance of the Dresden Green Diamond in the region of the spectral colours (visible electromagnetic waves between 700 to 400 nm) is found

at 534 ( $\pm 2$ ) nm. A green hue corresponds to this wavelength (after DIN 6164 hue no. 22½). The human eye, however, senses the hue no. 21½, i.e. a slightly bluish-green because the absorption in the blue is even lower than in the orange portion. Due to the fairly weak GR 1 absorption in the red and orange regions (right half of Figure 4), the colour saturation of the Dresden Green is relatively moderate. As a result of this, virtually no body colour is visible through the culet (Figure 3). This observation was confirmed by comparison with the DIN Colour Atlas charts (Tab. 1: degree of saturation of the body colour K only about 0.5 units, i.e. very pale).

Fortunately numerous vividly green internal reflections are created by the interaction of the exceptional old-cut and the GR 1 absorption (Tab. 1: degree of saturation of the reflection colour R around 3 units, i.e. a green of medium saturation).

*This reflection colour has made the Dresden Green into the famous diamond admired for centuries.*



The absorption diagram of the Green Diamond of Dresden in the ultra-violet and visible portions (Figure 6) now represents one of the first if not the first published absorption spectrum for a naturally green, cut diamond (thus being body-coloured) which had been missed by Orlov (1977) and Collins (1982) so far. After the author had already measured other bluish-green old-cut brilliants of natural coloration identified as type IaA diamonds (containing considerable amounts of nitrogen, mainly as pairs named A), it came as a great surprise in the Green Vaults when it became evident that the Dresden Green unambiguously belonged to the rarer, almost nitrogen-free type IIa. This observation was clearly confirmed by the absence of absorption bands below  $1600\text{ cm}^{-1}$  and above  $2650\text{ cm}^{-1}$  in the recorded mid-infrared transmission diagram. Artificially coloured diamonds of type IIa hitherto encountered displayed indeed a blue colour but almost identical absorption spectra to that of the Green Diamond.

Virtually all diamonds showing a non-fluorescent green colour component owe this to the influence of natural or artificial high-energy radiation. In both cases it generates defects in the carbon lattice of diamond, the so-called vacancies, themselves causing the same kind of selective light absorption in all four known types of diamond (Ia, Ib, IIa, IIb), i.e. in the first place the *GR I (General Radiation) absorption system* presented in Figure 6.

At the beginning of this century, W. Crookes executed the first intentional irradiations of diamond by applying radium salts. The green coloration of diamonds can therefore only be accepted, for the intended research study, as guaranteed natural if they spent the last one hundred years under credible supervision (e.g. in a museum) as stones known to always have been green. Considering that industrial routine irradiation of diamond had already begun by approximately 1950, it must be attributed to fortunate circumstances that this can be proved for the Dresden Green even for its absence from Dresden.

- firstly, the colour of the diamond had been described as pale green (Bauer, 1896) long before the stone was stored away on the Königstein, a colour it still shows;
- secondly, the diamond retained several small and shallow fissures with diffuse green specks (Figure 7 and Table 1) encountered so far only as products of natural radiation emanating from radioactive groundwaters.

Undoubtedly, however, these few irradiation spots do not account for the homogeneous green coloration of the Dresden Green. A natural body colour must be present.

#### *Weight Estimation*

The author pointed out in the early stages of

negotiation with the director of the Green Vaults, Dr J. Menzhausen, that the Dresden Green Diamond should be removed from its mounting and weighed precisely, for the literature (Bauer, 1896, De Beers, 1983, etc.) which until now contains the most incredible weight figures (cf. Table 1). Regrettably no permission could be procured for taking the diamond out of its setting. At that point, the author unfortunately did not know that the girdle projects over the top edge of the bezel on almost half of its circumference.

In order to be able to check the weight figure of 41 ct cited in most instances, two approaches of weight estimation were chosen:

- the weight determination of the diamond in the bezel setting did not permit a satisfactory approximation in spite of the precise measurement of the metal parts because their fineness was not known;
- calculation by means of the factor for the conversion of dimensions into weight of a loose pear-shaped old-cut brilliant of no less than 13 ct proved unsuccessful because the proportions deviated too strongly from those of the Dresden Green.

Based on data gathered in the laboratory, the author reached the conclusion that a conversion factor of 0.0065 would be the most supportable. Calculation with this factor resulted in a weight of 41.1 ct and thus confirmed Menzhausen's value (1987a). If the accurate weight differed, it would be slightly inferior (using the factor of 0.0064, near 40.5 ct).

Diamonds of type IIa reveal a clear tendency to be sizeable as well as nearly to completely colourless (Collins, 1982, translator's footnote p. 164) if not irradiated like the Dresden Green.

#### *Examination of the Cut*

The Green Diamond of Dresden was cut more than 240 years ago. Whoever examines this historic cut will be astonished by the quality of its symmetry, polish and proportions:

- according to prevailing grading regulations (CIBJO Rules 1986), the quality would have to be classified as good or even very good. The table facet of a diamond of 41 ct although being expansive is optically plane. The common corners of joining neighbour facets are perfectly pointed. Facets of one kind have an identical size and are not distorted. Table and culet are exactly concentric. The outline is pleasingly rounded and symmetrical. The quality of this cut cannot easily be rivalled by more recent old-cuts. And to beat it all, this remarkably cut diamond exhibits a unique series of ratios of length to width, etc., in whole numbers (cf. Table 1).



Fig. 7. Shallow fissure in one of the upper girdle facets showing diffuse green spots on the fissure walls, very probably generated by radioactive waters having circulated on the diamond deposit. Gold bezel (top) and silver bezel (bottom) visible below the fissure. Dark-field illumination. Length of longer fissure approx. 0.4mm.

The question shall be admissible whether such a perfection of cut was conceivable already in the early eighteenth century. Tillander (1988) considers this possible if the diamond had been cut in London, and Guichon (1988) as well if a top cutter executed the opus. In those times already a lap (scaife) of porous cast iron was employed. Yet it did not turn at 3000 but at less than 1000 rpm. The diamond was almost completely coated with lead, and the lead dop was mounted on a thin and flexible copper stick for easy adjustment of the facet angles in relation to the grinding wheel. Because also less oil than boart was applied the cut remained 'cold' but took months for a large facet to be completed (Guichon, 1988).

### Conclusions

The secret why a diamond of this size possesses such an outstanding cut and still displays its original coloration is that it was cut slowly and with little

pressure only. In that manner neither the diamond nor the lead could heat up over a certain critical temperature.

Without cutters of the past centuries having realized it, green diamonds were protected by the 'thermostat effect' of the lead/tin alloy. Pb/Sn solders melt at low temperatures, varying with composition (CRC 1978):

Pb70/Sn30 at 255°C maximum

Pb60/Sn40 at 238°C standard

Pb40/Sn60 at 190°C minimum

The green radiation colours of diamonds are sensitive to heat, without exception. Hänni (1987 and 1988) checked this fact on an experimental basis on a naturally green-skinned diamond crystal and on an artificially greened brilliant. Many a diamond cutter of today involuntarily has had this experience with his own cutting goods (and many a diamond setter also during his jewellery work).

The discoloration of green diamonds resulting from overheating is a continuous process of healing up the GR 1-8 radiation damage in the crystal lattice. This fading process starts already at 500°C, e.g. when diamonds are brought to red-heat which is performed easily on a modern 3000 rpm lap. In type Ia diamonds the discoloration of green comes to an end at about 800°C (Woods and Collins, 1986), in type IIa at higher temperatures (Davies, 1977). The process takes place quicker the higher the effective heat is. In general, uncontrolled annealing leads to unpleasant brown colour changes of incorrecable stability.

Thanks to the thermostat reaction of the lead dop after the ancient cutting technique, the discoloration of the Dresden Green could therefore not start at all. Thus, if a larger number of historical green old-cut diamonds is unknown world-wide, this can only mean that these body-coloured diamonds are extremely rare, much rarer than the relatively abundant green-skinned rough diamond crystals. The latter lose their surface and sub-surface coloration entirely during cutting most of the time. Occasionally however, individual green specks survive the cutting process, more frequently on remnants of crystal faces ('naturals') than in natural fissures, e.g. like those in the Dresden Green (Figure 7). The causes of this rarity of green body coloration will be pursued in the planned study.

Two further consequences of the low melting point of the lead dop are noteworthy:

no burn marks can be observed on old-cuts after the lead dop technique was applied. As the author pointed out earlier, these surface corrosion marks develop only at 600°C (Hänni and Bosshart, 1987). This temperature can be exceeded easily with modern cutting techniques. According to what has been said so far, the production of burn marks under the prongs of the mechanical dop of

today forcibly involves the destruction of green body and skin-colours of diamond too;

the Green Diamond of Dresden cannot have exceeded the temperature of 260°C when it was cut more than 240 years ago. This means that the absorption band at 594 nm (in Figure 6 present in extremely weak intensity) cannot be an artefact caused by elevated cutting temperatures but represents a natural property of the Dresden Green. The absorption band at 594 nm only develops above 275°C (Woods and Collins, 1986) while the line at 667 nm is destroyed above 430°C (Davies, 1977). Hence it follows that the Dresden Green experienced a temperature of not more than 350 ( $\pm 80$ )°C in the earth's crust.

### Summary

It can be stated that the Dresden Green Diamond:

1. belongs to the rare and chemically pure type IIa and as such is typically big and free of macroscopic inclusions;
2. possesses internal strain of normal strength ('Tatami' type);
3. as an historical old-cut brilliant exhibits an extraordinarily good cutting quality and noteworthy proportions;
4. was cut at slow speed, with little pressure and, mounted in a lead dop, at less than 260°C and then was carefully set;
5. is damaged along the girdle (its purity and girdle theoretically being improvable by a professional repolish);
6. very nearly weighs 41ct;
7. should be weighed at least to the hundredth of the carat and be set anew;
8. experienced a moderate irradiation by radioactive ground-waters;
9. does not show residual radioactivity;
10. reveals a non-fluorescent bluish-green colour hue and a weak saturation (due to 8);
11. evinces a rare heat-sensitive body colour (due to 1 and 8);
12. displays a brilliant reflection colour (thanks to 3 and 8);
13. was heated in the earth's crust to about 350°C;
14. has retained its green coloration (owing to 4 and 13) but could lose it on the occasion of a repolishing operation on a rapid lap;
15. however, endured multiple deep freezing in a nitrogen gas atmosphere without any problems;
16. differs little in absorption behaviour from type IIa diamonds artificially coloured blue; and
17. will not reveal its place of origin in the near future, based on the inclusions present and on the criteria enumerated above.

### Appreciation

The author wishes to express his satisfaction concerning the availability of such a unique gemstone as the Dresden Green for investigation, across all borders. The successful outcome of the 'Dresden Green Project' creates the hope that *in future an increasing number of projects of particular scientific or cultural significance could be realized between East and West.*

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## 'Aqua Aura' enhanced quartz specimens

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### Abstract

Singly and doubly terminated euhedral quartz crystals and crystal clusters with a coating producing an apparent blue colour and strong iridescence are being sold under the trade name 'Aqua Aura'. The authors report on their examination of a specimen which confirmed that the coating consists of gold.

### Introduction

In the Fall of 1988 one of the authors (RCK) was shown an interesting quartz specimen by Mr. Peter Flusser of Overland Gems in Los Angeles, California. The crystal was singly-terminated, weighed 47.50 ct. and measured 52 millimetres in length. It had an unusual light blue to greenish blue body colour (Figure 1a) and strong iridescent colours on all surfaces (Figure 1b). This author was informed that the specimen had been obtained from the firm Bob Jackson Minerals in Renton, Washington, and was being marketed as 'Aqua Aura'. The authors subsequently saw additional specimens at the annual Pasadena Gem and Mineral Show in Pasadena, California, and Dr. Emmanuel Fritsch of GIA's Research Department later provided a crystal cluster of this coated quartz given to him directly by Bob Jackson for visual examination (Figure 2).

According to a small slip of promotional literature obtained at the Pasadena Show, 'Aqua Aura' specimens are 'Natural quartz crystals coated with pure gold'. Following is some additional text provided with the crystals that relate to the coating process used: 'Quartz crystals are naturally surrounded by an electrical charge. Aqua Aura is created by allowing pure gold molecules to adhere to that charge. The extremely thin gold coating breaks light into its spectral colours, causing the beautiful colours of Aqua Aura.' With respect to durability, the promotional piece states that 'Aqua Aura requires no special care to keep its beauty. The gold coating cannot be rubbed or scraped off'.

### Gemmological examination

The examination began with the testing of the quartz crystal to determine its gemmological properties. The crystal had a colour that varied from greenish-blue to blue, with overlying iridescent colours of violet, blue, green and yellow. Of the iridescent colours, the violet was most prominent. Overall, the crystal had a high degree of transparency. As the crystal faces of the specimen were well-formed with reasonably good surface lustre, flat facet refractive indices readings were taken



Figs. 1a & b. 'Aqua Aura' coated quartz crystal (GIA Collection No.16667) showing (left) the blue colour (right) the iridescent colours provided by the coating. Oblique fibre optic illumination. Photos by William Videto.



Fig. 2 (centre). 'Aqua Aura' quartz crystal cluster. Oblique fibre optic illumination. *Photo by William Videto.*

Fig. 3 (top, left). 'Aqua Aura' coated quartz crystal showing areas devoid of the coating and colour concentrated along the edge of the crystal face. 40x. *Photo by John I. Koivula.*

Fig. 4 (top, right). Surface pits on an 'Aqua Aura' quartz crystal in which little or no coating was observed. 30x. *Photo by John I. Koivula.*

Fig. 5 (right). Iridescent surface layer on an 'Aqua Aura' quartz crystal. 10x. *Photo by John I. Koivula.*



using a GIA GEM Instruments Duplex II refractometer and near-monochromatic light. Somewhat vague readings of 1.54-1.55 were obtained. In a field of polarized light a typical doubly refractive reaction was noted.

The specimen was also examined for its reaction to ultraviolet radiation and proved to be inert to both long- and short-wavelengths. No visible absorption features were noted when the crystal was examined with both diffraction-grating and prism type hand held spectrosopes, using both transmission and external reflection lighting methods.

Specific gravity was determined by immersing the crystal in a mixture of methylene iodide and benzyl benzoate calibrated to 2.67. When placed in this liquid the crystal floated very slowly – at essentially the same rate as an untreated rock crystal quartz. Specific gravity was thus estimated at 2.66.

Under magnification the coating revealed a number of interesting features. First, it appeared to vary in thickness on some crystal faces, with some small areas showing no coating at all (Figure 3). It also seemed not to have deposited as well in surface pits as it had on flat surfaces (Figure 4). The colour also looked to be concentrated near crystal edges (Figures 3 and 4) and appeared as if it may have been deposited directionally.

#### Durability testing

A number of additional tests were performed to determine the relative durability of the coating. A hardness pencil of Mohs hardness 6 failed to scratch the crystal or produce any visible mark on the coating, while one of Mohs hardness 7 produced a scratch. No visible reaction was noted when the surface was tested with a thermal reaction tester heated to a red heat (approximately 800 degrees Centigrade). The crystal was then placed on an electrically-activated 'Kanthal' bridge and heated to a temperature of approximately 1000-1100 degrees Centigrade (gold melts at 1064 degrees Centigrade). This also appeared to have no effect on the coating. Finally, one crystal face was rubbed vigorously with a cotton swab dipped in acetone. No discoloration appeared on the swab and no visible effect was noted on the crystal face.

#### Chemical analysis

The crystal was next examined by means of energy dispersive X-ray fluorescence (ED-XRF) to determine its elemental characteristics. The major elements detected were silicon, gold and titanium. The silicon was almost certainly from the quartz itself. It is believed that the titanium detected may have been from sub-microscopic rutile inclusions

(TiO<sub>2</sub>) in the quartz crystal or more probably from titanium in solid solution, because it is well known that titanium is one of the secondary elements commonly found as a solid solution constituent in natural quartz (Fronde, 1962). The other major element detected, gold, supports the information from the vendor that the coating used is pure gold. Dr. Fritsch also noted that, when tested for electrical conductivity on a conductometer, in some areas the gold coating showed excellent conductivity.

**Table 1: Summary of gemmological properties**

Colour:	Greenish-blue to blue
Iridescent colours:	Violet, blue, green and yellow; violet most prominent
Diaphaneity:	Transparent
Refractive indices:	o – 1.54; e – 1.55
Polariscope reaction:	Doubly refractive
UV fluorescence	
Long-wave:	inert
Short-wave:	inert
Absorption spectrum:	none
Specific gravity:	2.66 (heavy liquid method)
Hardness:	6½-7
Reaction to heat:	None observed to 1000-1100 degrees C.
Reaction to acetone:	None

#### Discussion

The blue colour shown by 'Aqua Aura' quartz is not an interference colour. Gold in thin film is bluish-green in transmitted light. Thus the green to blue colour exhibited by 'Aqua Aura' quartz is the true absorption-caused colour of gold (Nassau, 1983).

In addition to the thin film effects observed internally as inclusions in some gems (Koivula, 1980), thin film coatings, similar to the iridescent surfaces (Figure 5) shown by these artificially gold-coated 'Aqua Aura' quartz crystals, are known to occur in nature. The common oil slick on water



is probably the best single example (Ribbe, 1972) but some types of sun-oxidised glass, certain minerals such as cuprite, and the gems faceted from them, will also often show iridescent, metallic-appearing superficial thin films. The iridescence colours superimposed on the surface of 'Aqua Aura' quartz are related to the thickness of the gold film, probably of the same order of magnitude as the visible wavelength.

If we think of the already existing technology in relation to thin film applications of elemental gold, then the appearance of 'Aqua Aura' in the gem and mineral trade is not too surprising. Many of today's modern synthetics came to gemmology from other fields of science such as laser research and the computer industry. Now, as an apparent spin-off from the thin film gold-coating technology that gives us the sun visors on astronauts' helmets and the vacuum deposited conductive coatings for scanning electron microscopy, we have 'Aqua Aura'.

### Conclusion

Based on the examination of this 'Aqua Aura' coated quartz crystal (GIA Collection No. 16667) as summarised in Table 1, it appears that the coating does not affect the normal quartz properties. The vendor's claim that the coating is 'pure gold' is substantiated through energy dispersive X-ray fluorescence testing. Furthermore, in view of the rigorous durability testing, statements relating to the relatively durable nature of the coating

also appear to be accurate. At this point in time we have only seen single quartz crystals and crystal clusters given the 'Aqua Aura' treatment. But, knowing the history of other gemstone treatments, can faceted 'Aqua Aura' gems be far off?

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# Methods for the distinction of natural and synthetic citrine and prasiolite

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## Abstract

Different types of natural and synthetic citrine are characterized by a combination of absorption spectroscopy in the visible and ultraviolet range with microscopic investigations of structural properties such as twinning, growth structures, colour zoning as well as orientated channel-like inclusions or orientated striations.

Both methods reveal a number of typical features which indicate artificial irradiation and/or heat treatment. The colour of natural and synthetic citrine is caused by traces of iron and/or cobalt, as well as by radiation-induced colour centres. According to colour causes and treatment histories, natural and synthetic citrines are classified into different categories as indicated below:

- (a) yellow to orange-brown heat-treated natural citrine, originally amethyst: Fe-containing particles;
- (b) green heat-treated natural prasiolite, originally amethyst: Fe-containing particles and  $\text{Fe}^{2+}$  ( $I_6$ );
- (c) light yellow, non-artificially-irradiated, non-heat-treated natural citrine; radiation-induced colour centres;
- (d) light yellow, non-artificially-irradiated, heat-treated natural citrine: originally morion: radiation-induced colour centres;
- (e) greenish-yellow artificially-irradiated, non-heat-treated natural citrine, originally colourless quartz: radiation-induced colour centres;
- (f) greenish-yellow artificially-irradiated, heat-treated natural citrine, originally colourless quartz: radiation-induced colour centres;
- (g) yellow to orange-brown synthetic citrine: Fe-containing particles;
- (h) yellow to orange-brown synthetic citrine: Fe-containing particles and  $\text{Fe}^{3+}$  ( $I_4$ );
- (i) green synthetic prasiolite: Fe-containing particles,  $\text{Fe}^{3+}$  ( $I_4$ ) and  $\text{Fe}^{2+}$  ( $I_6$ );
- (j) yellow to orange-brown synthetic citrine: Fe-containing particles and  $\text{Co}^{3+}$  ( $I_6$ );
- (k) greenish-yellow artificially-irradiated, non-heat-treated synthetic citrine, originally colourless synthetic quartz: radiation-induced colour centres;
- (l) greenish-yellow artificially-irradiated, heat-treated synthetic citrine, originally colourless synthetic quartz: radiation-induced colour centres.

A number of specific properties was found which are useful for the determination of growth and/or treatment histories of samples of unknown origin and for a

distinction of natural versus synthetic citrine. Most natural citrines used in the trade are heat-treated natural amethysts. However, non-artificially-irradiated, non-heat-treated or heat-treated as well as irradiated and possibly heat-treated natural quartz is also found. Most synthetic citrines of the trade are different types of Fe-bearing as well as Fe- and Co-containing quartz crystals, which are grown hydrothermally from  $\text{K}_2\text{CO}_3$ -bearing solutions with or without additional oxidizers. Trace element analyses of synthetic citrine can indicate growth media and oxidizers used in the working solution.

## 1 Introduction

In the past years great efforts were made and many investigations were undertaken for the characterization of the properties of natural and synthetic amethyst in order to develop practicable methods for the distinction of natural and synthetic samples. In the papers published so far emphasis is laid on infrared spectroscopy and microscopy as the two dominant methods to recognize features useful for diagnostic purposes (e.g. Schmetzer and Bank, 1980; Zecchini and Mérioux, 1980; Lind and Schmetzer, 1982, 1983; Schneider and Dröschel, 1983; Lind *et al.*, 1983, 1985; Schmetzer, 1985, 1986; Lind and Schmetzer, 1985, 1987; Crowningshield *et al.*, 1986). Although in some of the papers mentioned above, some hints are given which are useful for the distinction of natural and synthetic citrine, no comprehensive publication exists which enables the practical gemmologist to decide whether a yellow or brown quartz of unknown origin is a natural or man-made, treated or untreated stone (cf. O'Donoghue, 1983).

In the present publication a combination of absorption spectroscopy in the visible and ultraviolet areas with microscopic investigations is suggested as a routine method for a distinction between natural and synthetic members of the quartz family with yellow, greenish-yellow, green, orange-brown or brown coloration. However, before describing the characteristic features of natural and synthetic quartz varieties, it is necessary to give a short review of the scientific literature dealing with colour and colour causes of natural citrine types as well as with

the production of synthetic yellow, yellowish-green, green, orange-brown and brown quartz. With the information given in the literature, most of the properties of all different types of natural and synthetic citrine, which are found in the gem trade, can be understood. In addition, a short overview of the literature should help to avoid further confusion between the different types of natural and synthetic citrines described so far, as well as between different applications of closely related terms such as citrine, honey quartz and greenish-yellow quartz for samples of identical origin or with similar growth and/or treatment histories (cf. Nassau, 1977).

According to the personal opinion of the present author, for practical reasons the terms natural citrine and synthetic citrine should be used for all yellow, greenish-yellow, orange-brown and brown members of the quartz group, as well as the terms natural prasiolite and synthetic prasiolite for green samples. The main argument for this suggestion is that the determination of the exact colour-causing trace element or colour-causing defect (colour centre) is not practicable in the gem trade for each sample. However, a general decision of international nomenclature commissions, e.g. a decision of the Commission on Gem Materials of the International Mineralogical Association or a decision of the CIBJO Commission, for the use of the term citrine are needed in order to clarify and avoid all possibilities of misunderstanding and misinterpretation.

## II Review of the literature dealing with yellow, greenish-yellow, green, orange-brown or brown quartz varieties

A general survey of natural and synthetic quartz varieties, growth conditions and colour causes is given by various authors; these papers are suggested for additional reading (Gordienko *et al.*, 1969; Lehmann and Bambauer, 1973; Hutton, 1974; Lehmann, 1978a, b; Balakirev *et al.*, 1979; Elwell, 1979; Nassau, 1980; Balitsky, 1981; Sunagawa, 1982).

### II.1 HEAT-TREATED NATURAL AMETHYST

Most yellow, yellowish-brown, orange-brown and brown natural citrines of the trade are natural amethysts which were converted to citrine by heat treatment at temperatures between approximately 400 and 600°C. The absorption spectrum of this type of citrine consists of an increasing absorption from the red area to the violet end of the visible region, which is due to an absorption band centred in the ultraviolet area and extending into the visible range with its low energy tail. According to EPR-investigations, this absorption is caused by small iron-containing particles, presumably Fe<sub>2</sub>O<sub>3</sub> precipitates, which are submicroscopically dispersed in the host crystal (Lehmann and Moore, 1966; Hutton

and Troup, 1966; Lehmann, 1967, 1971a, b; Wild, 1968; Lehmann and Bambauer, 1973; Stock and Lehmann, 1977; Neumann and Schmetzer, 1984a, b). The colour of heat-treated natural amethyst crystals, which turn green during the annealing process (the so-called prasiolite of the trade) is due to interstitial Fe<sup>2+</sup> on distorted octahedral sites, designated Fe<sup>2+</sup> (I<sub>6</sub>). The absorption band of Fe<sup>2+</sup> (I<sub>6</sub>) in quartz in the visible region is centred in the red area at about 13,500 cm<sup>-1</sup> (approx. 741 nm), a second broad absorption band is observed at 10,500 cm<sup>-1</sup> (approx. 950 nm) in the near infrared (Lehmann, 1967; Lehmann and Bambauer, 1973; Neumann and Schmetzer, 1984a,b).

### II.2 GREENISH-YELLOW, IRRADIATION-TREATED NATURAL QUARTZ (ORIGINALLY COLOURLESS OR LIGHT YELLOW)

Colourless natural quartz crystals of peculiar occurrences turn smoky or greenish-yellow by irradiation treatment with gamma rays. After subsequent heat treatment of irradiated samples at lower temperatures (e.g. about 150 – 250°C), smoky-quartz may turn greenish-yellow or colourless. Greenish-yellow quartz, which is produced by heat treatment of smoky quartz or directly without annealing by gamma irradiation, becomes colourless again by heat treatment at higher temperatures (e.g. about 250 – 350°C). Two different descriptions and interpretations of the absorption spectra of irradiated greenish-yellow quartz and irradiated smoky quartz, which turned greenish-yellow after subsequent heat-treatment, are found in the literature.

The first group of authors measured absorption spectra with two distinct maxima, one of them between 24,400 and 25,600 cm<sup>-1</sup> (410 and 390 nm), the second one between about 15,600 and 16,100 cm<sup>-1</sup> (640 and 620 nm). Both absorption bands are superimposed by an absorption in the ultraviolet area, the low energy tail of which extends to the visible region (Samoilovich *et al.*, 1969b; Bukanov and Markova, 1969; Lehmann, 1971a, b; Lehmann and Bambauer, 1973; Samoilovich *et al.*, 1976; cf. also the paper dealing with neutron irradiated quartz by Maschmeyer and Lehmann, 1984). Most characteristic for all these publications is the description of an absorption maximum in the 24,400 to 25,600 cm<sup>-1</sup> area (410 to 390 nm), the position of which is distinctly shifted to the short wavelength region compared with the position of the ordinary maximum of the smoky colour centre in quartz. The maximum of this Al-related colour centre is found at approximately 23,400 to 22,200 cm<sup>-1</sup> (427 to 450 nm).

The second group of papers dealing with irradiation-induced colour of greenish-yellow quartz describes the absorption spectra of all types of irradiation-

ted smoky and greenish-yellow quartz crystals to be due to a superposition of an absorption maximum in the ultraviolet, the low energy tail of which extends to the visible area (so-called greenish-yellow absorption) with several distinct absorption maxima at  $15,000\text{ cm}^{-1}$  (designated  $A_1$ ),  $20,650\text{ cm}^{-1}$  (designated  $A_2$ ),  $23,400\text{ cm}^{-1}$  (designated  $A_3$ , smoky colour centre) and  $32,000\text{ cm}^{-1}$  (designated B) (Nassau and Prescott, 1975, 1977a).

In the first group of publications, besides an absorption maximum in the ultraviolet causing an increasing absorption from red to violet in the visible region, two different types of colour centres are assumed to be additionally present in the visible part of the absorption spectra of quartz crystals with smoky brown, yellowish-grey, and greenish-yellow colours. In these varieties of irradiated quartz crystals, the ordinary smoky centre is thought to be responsible for the absorption band at about  $22,200\text{ cm}^{-1}$  (450 nm), and a similar, but not identical colour centre is thought to cause the absorption band at about  $25,000\text{ cm}^{-1}$  (400 nm). The various colour shades of irradiation-treated samples with or without subsequent annealing are explained by combinations of the ultraviolet absorption maximum with the two absorption bands in the blue and violet, all three of them having different intensity ratios as well as different bleaching characteristics.

In the second group of publications all different smoky to greenish-yellow colour shades of irradiated quartz crystals are explained by combinations of two absorption bands only, i.e. by a superposition of the greenish-yellow absorption in the ultraviolet and the ordinary smoky absorption band. The presence of a different third absorption band, the maximum of which is shifted from  $22,200\text{ cm}^{-1}$  to the short wavelength region is not assumed in those papers (cf. also the results of Sawyer, 1974, 1976).

In general,  $\text{Al}^{3+}$  on  $\text{Si}^{4+}$  sites with charge compensating alkali ions is thought to be responsible for the formation of smoky colour centres in quartz, and  $\text{Al}^{3+}$  on  $\text{Si}^{4+}$  sites with charge compensating hydrogen and lithium ions is assumed to cause greenish-yellow colours. However, not all details of irradiation-induced smoky and greenish-yellow colour centres are understood at present.

### II.3 NATURAL YELLOW TO BROWN QUARTZ AND HEAT-TREATED MORION

Natural yellow and light brown quartz, which is not produced from amethyst by heat treatment, is long known in the gem trade. Part of the material used for cutting purposes is of natural colour, i.e. these stones are neither heat-treated nor artificially-irradiated. In some instances, natural smoky quartz or morion is able to be converted by heat treatment at about  $300^\circ\text{C}$  to yellow or light brown citrine (cf. Wild, 1968; Bank, 1976). The first papers, in which

the independent nature of this third type of citrine coloration is recognized, were published by Samoilovich *et al.* (1976) and Lehmann (1977, 1978a). Further information about this type of citrine coloration is found in the publications of Nassau and Prescott (1977a), Steshchin (1979a,b), Maschmeyer *et al.* (1980), Lehmann (1981) and Maschmeyer and Lehmann (1983).

The absorption spectrum of this third type of citrine is similar to the spectrum of heat-treated amethyst, i.e. the spectrum consists of a strong absorption in the ultraviolet area, the low energy tail of which extends to the visible region. Instead of the coloration of heat-treated amethyst, natural untreated citrine of this third type is slightly pleochroic. The difference between artificially irradiated greenish-yellow quartz, i.e. the second type of citrine, and this third type of citrine coloration is the absence of a distinct absorption band at about  $25,000\text{ cm}^{-1}$  (400 nm) as well as an improved thermal stability of the colour centres up to about  $500^\circ\text{C}$ . A model for two paramagnetic centres observed in two crystals of this third type of citrine from Madagascar was worked out by Maschmeyer *et al.* (1980) and Lehmann (1981), and new investigations by Maschmeyer and Lehmann (1983) using natural citrines from Madagascar revealed the presence of at least seven different hole centres. Most of them are related to substitutional Al on Si sites; however, the exact nature of additional defect structures which cause their difference to smoky quartz centres are still unknown.

The unique green quartz described by Nassau and Prescott (1977b) is possibly a citrine of this third type having an additional weak absorption band at about  $16,100\text{ cm}^{-1}$  (620 nm), the origin of which is unknown at present.

### II.4 SYNTHETIC Fe-CONTAINING YELLOW, GREEN, ORANGE-BROWN AND BROWN QUARTZ

The first samples of synthetic brown and green quartz were described by Tsinober *et al.* (1959), Tsinober and Chentsova (1959) and Ballmann (1961). The crystals were grown on basal seeds in aqueous  $\text{K}_2\text{CO}_3$ -containing solutions by hydrothermal techniques. Brown crystals containing mainly  $\text{Fe}^{3+}$  were obtained from lower concentrations of  $\text{K}_2\text{CO}_3$  and green crystals containing  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  were grown from solutions with very high  $\text{K}_2\text{CO}_3$  concentrations. Heat treatment at  $400 - 500^\circ\text{C}$  turned brown crystals green by reducing part of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$ .

The growth of synthetic yellow quartz, so-called synthetic citrine, is described comprehensively by Chadschi and Reschetova (1975) and Khadzhi and Reshetova (1977). The crystals were grown on quartz seeding plates orientated parallel to the basal pinacoid (0001) or with plates inclined at an angle

**Table 1.** Absorption bands assigned to trivalent iron in interstitial sites of distorted tetrahedral symmetry in the quartz lattice (in  $\text{cm}^{-1}$ )

Lehmann, 1971a	Khaliov <i>et al.</i> , 1979	Transition
18,700	18,000	${}^6A_1 \rightarrow {}^4T_1$
20,200	20,300	$\rightarrow {}^4T_2$
22,500	21,800 22,700	$\rightarrow {}^4A_1, {}^4E$
24,800	24,800	$\rightarrow {}^4T_2$
27,300	26,000	$\rightarrow {}^4E$
29,100	28,700	$\rightarrow {}^4T_1$

within the range of up to  $20^\circ$  to the basal plane in aqueous solutions of  $\text{K}_2\text{CO}_3$ . Furthermore, substances for the oxidation of iron such as nitrites and nitrates of alkali metals or potassium permanganate (e.g.  $\text{LiNO}_2$ ,  $\text{NaNO}_2$ ,  $\text{KNO}_2$ ,  $\text{LiNO}_3$ ,  $\text{NaNO}_3$ ,  $\text{KNO}_3$ ,  $\text{KMnO}_4$ ) are added to the solution. The presence of  $\text{Li}^+$  in the working solution of  $\text{K}_2\text{CO}_3$  is responsible for purification from undesired aluminium impurities due to the formation of hardly soluble impurities such as eucryptite,  $\text{LiAlSiO}_4$ . Without the presence of lithium ions, capillary channels parallel to the optical axis of the quartz crystals are formed by Al impurities, disturbing the homogeneity and colour quality of synthetic citrine.

A third method for the production of iron-bearing yellow to orange-brown quartz is recently described by Hosaka and Taki (1983). The hydrothermal growth was performed using NaCl- and KCl-containing solutions with X-cut (11 $\bar{2}$ 0) and Y-bar seeds.

Various publications deal with colour and colour causes in synthetic Fe-containing yellow, brown and green quartz (Lehmann and Moore, 1966; Lehmann, 1967, 1971a,b; Samoilovich *et al.*, 1969a; Gordienko *et al.*, 1969; Lehmann and Bambauer, 1973; Samoilovich *et al.*, 1976; Zakharov *et al.*, 1978; Khaliov *et al.*, 1979; Balakirev *et al.*, 1979; Balitsky and Balitskaya, 1985, 1986). Some of the results can be summarized as follows: in synthetic yellow, orange-brown, brown and green quartz three different colour causes were found to exist. These are iron-containing colloidal particles, most probably  $\text{Fe}_2\text{O}_3$ , ferric iron on interstitial sites of distorted tetrahedral co-ordination, designated  $\text{Fe}^{3+}(\text{I}_4)$ , and ferrous iron on interstitial sites of distorted octahedral co-ordination, designated  $\text{Fe}^{2+}(\text{I}_6)$ . According to the details of crystal growth of an individual sample, the three colour-causing parti-

cles or ions are present in various amounts, and the colour is due to a superposition of three basic types of absorption spectra.

The spectrum of iron-containing colloidal particles consists of a strong absorption in the ultraviolet, the low energy tail of which extends to the visible area. A continuous absorption results increasing from red to violet. In the absorption spectrum of  $\text{Fe}^{3+}(\text{I}_4)$  numerous weak absorption bands are found in the visible and ultraviolet region (Table 1). The spectrum of  $\text{Fe}^{2+}(\text{I}_6)$  is dominated by two strong broad absorption bands with maxima at about 13,500 and 10,500  $\text{cm}^{-1}$  (741 and 950 nm).

In yellow to orange-brown quartz grown in  $\text{K}_2\text{CO}_3$  solutions with the presences of oxidizing phases, iron-containing particles are dominant. In brown quartz from  $\text{K}_2\text{CO}_3$  solutions without the presence of oxidizers, iron-containing particles and  $\text{Fe}^{3+}(\text{I}_4)$  are observed. In green quartz, additional amounts of  $\text{Fe}^{2+}(\text{I}_6)$  are found. All colours of iron-containing synthetic quartz can be explained by a combination of all three colour-causing particles or ions in various concentrations. Upon heat treatment, brown samples of iron-containing synthetic quartz are converted to green prasiolite. This colour change is explained by a reduction of  $\text{Fe}^{3+}$  to  $\text{Fe}^{2+}$  accompanied by a migration from tetrahedral to octahedral interstitial sites, i.e.  $\text{Fe}^{3+}(\text{I}_4) \rightarrow \text{Fe}^{2+}(\text{I}_6)$  (Tsinober *et al.*, 1959; Samoilovich *et al.*, 1969a; Lehmann and Bambauer, 1973).

## II.5 YELLOW, GREENISH-BROWN AND YELLOW-BROWN, IRRADIATION-TREATED SYNTHETIC QUARTZ (ORIGINALLY COLOURLESS)

Irradiation-induced colour changes of synthetic quartz are slightly different from colour changes of irradiation-treated natural samples. In synthetic quartz, smoky and greenish-yellow colours are

obtainable after irradiation (Gordienko *et al.*, 1969; Sawyer, 1974, 1976; Samoilovich *et al.*, 1976; Nassau and Prescott, 1977a). Subsequent heat treatment turns smoky quartz blue, blue-green, green, greenish-yellow or colourless, whereas greenish-yellow quartz is only bleached by annealing processes (Nassau and Prescott, 1977a). In some papers, a correlation between trace element contents and irradiation-induced colour centres is discussed, e.g. for Al- and Ge-containing synthetic quartz with smoky coloration after gamma irradiation (Chentsova and Butuzov, 1962; Haven *et al.*, 1966; Tsinober *et al.*, 1967; Gordienko *et al.*, 1969) as well as for Ga-containing synthetic quartz with yellow-brown coloration after irradiation treatment (Balitskii *et al.*, 1970). The influence of charge compensating hydrogen and alkali ions (sodium and lithium) is also questionable (cf. Sawyer, 1974, 1976). However, not all details of irradiation-induced colour changes of synthetic quartz are understood at present.

For jewellery purposes, synthetic quartz crystals with irradiation-induced yellow, greenish-yellow or yellow-brown coloration are not suitable due to the low thermal stability of this type of citrine colour centres (Nassau and Prescott, 1977a), which are already bleaching in daylight (Balitsky, 1981).

## II.6 SYNTHETIC CO-CONTAINING YELLOW, BROWN AND BLUE QUARTZ

Co-bearing synthetic quartz is grown from  $\text{Na}_2\text{CO}_3$ - and  $\text{K}_2\text{CO}_3$ -containing solutions on basal seed plates. The colour of the samples obtained are blue from  $\text{Na}_2\text{CO}_3$ - and yellow to brown from  $\text{K}_2\text{CO}_3$ -containing environments, respectively. The absorption spectra of blue samples are assigned to bivalent cobalt in interstitial positions of distorted tetrahedral symmetry, designated  $\text{Co}^{2+} (\text{I}_4)$ , whereas the spectra of brown crystals are explained by the presence of trivalent cobalt in interstitial positions of distorted octahedral symmetry, designated  $\text{Co}^{3+} (\text{I}_6)$ . Brown samples turn blue after heat treatment at approximately 450°C. This colour change is due to a reduction of cobalt from trivalent to bivalent state accompanied by a displacement from distorted octahedral to tetrahedral interstitial positions, i.e.  $\text{Co}^{3+} (\text{I}_6) \rightarrow \text{Co}^{2+} (\text{I}_4)$  (Wood and Ballman, 1966; Samoilovich *et al.*, 1969a; Gordienko *et al.*, 1969; Lehmann, 1969, 1971a; Lehmann and Bambauer, 1973). In addition to the presence of  $\text{Co}^{3+} (\text{I}_6)$ , synthetic samples may also contain distinct amounts of  $\text{Fe}^{3+}$ , most presumably as colloidal particles (Gordienko *et al.*, 1969; Lehmann, 1969; Balakirev *et al.*, 1979). At present, no natural counterparts of synthetic Co-bearing blue, yellow or brown quartzes are known.

## III Investigations of commercially available, and some non-commercially, natural and synthetic citrines including yellow, yellowish-green, green, orange-brown and brown quartz varieties

### III.1 MATERIALS AND METHODS

Only few references are found in literature which contain specific indications on techniques applicable for the distinction of natural and synthetic citrine (cf. O'Donoghue, 1983). In the present research project, the two main methods which were suggested for the distinction of natural and synthetic amethyst, i.e. infrared spectroscopy and microscopic determination of twinning, growth structures and inclusions, were also tested for the determination of citrine. According to Zecchini and Mérioux (1980), infrared spectroscopy in the 4000-3000  $\text{cm}^{-1}$  range is useful for the distinction of natural and synthetic citrine. However, experiments performed by Th. Lind at Heidelberg University in 1982 and 1983 (unpublished) using heat-treated amethysts and Fe-containing synthetic citrines were not successful and failed to establish infrared spectroscopy as a routine method for the determination of cut citrines of unknown origin.

According to microscopic investigations, the recognition of the presence of polysynthetic twinning on the Brazil law is useful for the determination of natural citrine which is produced from natural amethyst by heat treatment (Schmetzer, 1985, 1986). This result was worked out using a horizontal microscope with immersion liquids and an especially developed sample holder. By the application of a simplified technique for the recognition of polysynthetic twinning in natural amethyst without microscopic investigations, an identical behaviour of natural amethyst and citrine produced by heat treatment of natural amethyst is described (Crowningshield *et al.*, 1986). This statement, however, was found to be partly incorrect and thus will be discussed below in detail.

In summary, it was assumed at the beginning of the present investigation, that the determination of twinning and other structural properties can be a useful method for the characterization of citrines of unknown origin whereas the application of infrared spectroscopy for the study of citrines, especially for faceted samples, was neglected in this project. However, it is evident from literature (cf. sections II.1-II.6), that different natural and synthetic varieties of citrine reveal various colour causes. Thus, the investigation of absorption spectra in the visible and ultraviolet region was assumed to be an adequate method to characterize the samples. In addition, it was suspected that possibly some spectroscopic properties of natural and synthetic citrines are not common for all types of samples. These considerations led to systematic investigations by a combination of microscopy and absorp-

tion spectroscopy in the visible and ultraviolet.

The samples investigated were selected in order to be typical and representative for the material used in the gem industry of Idar-Oberstein. For most natural samples, the exact localities were known. In contrast, no synthetic samples were found in the trade, for which an appropriate characterization of growth conditions was available and, therefore, the assignment of the samples investigated to certain categories of synthetic citrine as well as to specific growth conditions had to be worked out experimentally.

of this particular type of citrine. The absorption maximum in the ultraviolet extending to the visible is assigned to iron-containing colloidal particles in literature, which are formed during the annealing process. The weak absorption band at  $21,000\text{ cm}^{-1}$  was not mentioned previously.

The absorption spectra of heat-treated natural amethysts which turned green during the annealing process, the so-called prasiolite of the trade (Figure 2), show an additional weak absorption band at about  $13,500\text{ cm}^{-1}$  (741 nm), which was assigned to bivalent iron on interstitial sites of distorted



Fig. 1. Colour sequence of natural citrines; five samples represent heat-treated natural amethysts from Brazil, the right stone represents natural untreated citrine from Goyaz, Brazil. Diameter of the sample approx. 10 mm. Photo by O. Medenbach, Bochum, FRG.

### III.2 RESULTS

#### III.2.1 Heat-treated natural amethyst (citrine and prasiolite)

Yellow to orange-brown citrine and green prasiolite which was commercially produced by annealing of natural amethyst was available from different localities in Brazil, from Uruguay and Namibia. The designations Bahia, Palmeira and Madeira citrine are sometimes used in the trade in order to characterize the colour intensity of samples in the yellow to orange-brown sequence (Figure 1), greened amethyst is often called prasiolite.

Absorption spectra of this type of natural citrine (heat-treated amethyst) reveal a continuously increasing absorption from red to violet in all samples with different colour shades and intensities (Figure 2). The increasing absorption is caused by a strong band with maximum in the ultraviolet, the low energy tail of which extends to the whole visible area. In addition, a weak absorption band at about  $21,000\text{ cm}^{-1}$  (476 nm) was measured in all samples

octahedral symmetry,  $\text{Fe}^{2+}$  ( $I_6$ ). The colour of heat-treated amethyst is dependent only of the intensity ratios of the green and yellow to brown absorption, i.e. the colour after annealing is due to the relative amounts of iron-containing precipitates and  $\text{Fe}^{2+}$  ( $I_6$ ) produced by heat treatment. However, the detailed mechanisms of the development of both types of iron in various ratios are not completely understood at present (cf. Neumann and Schmetzer, 1984a,b).

Polysynthetic twinning on the Brazil law is thought to be one of the most important properties of natural amethyst, the presence of which can be used for recognition of an unknown sample to be of natural origin. Unfortunately, the possibility for direct observation of polysynthetic twinning on the Brazil law in natural amethyst and heat-treated natural amethyst (citrine and prasiolite) is limited to a small number of specimens only (cf. Schmetzer, 1987). However, the presence of polysynthetic twinning is clearly indicated by the observation of

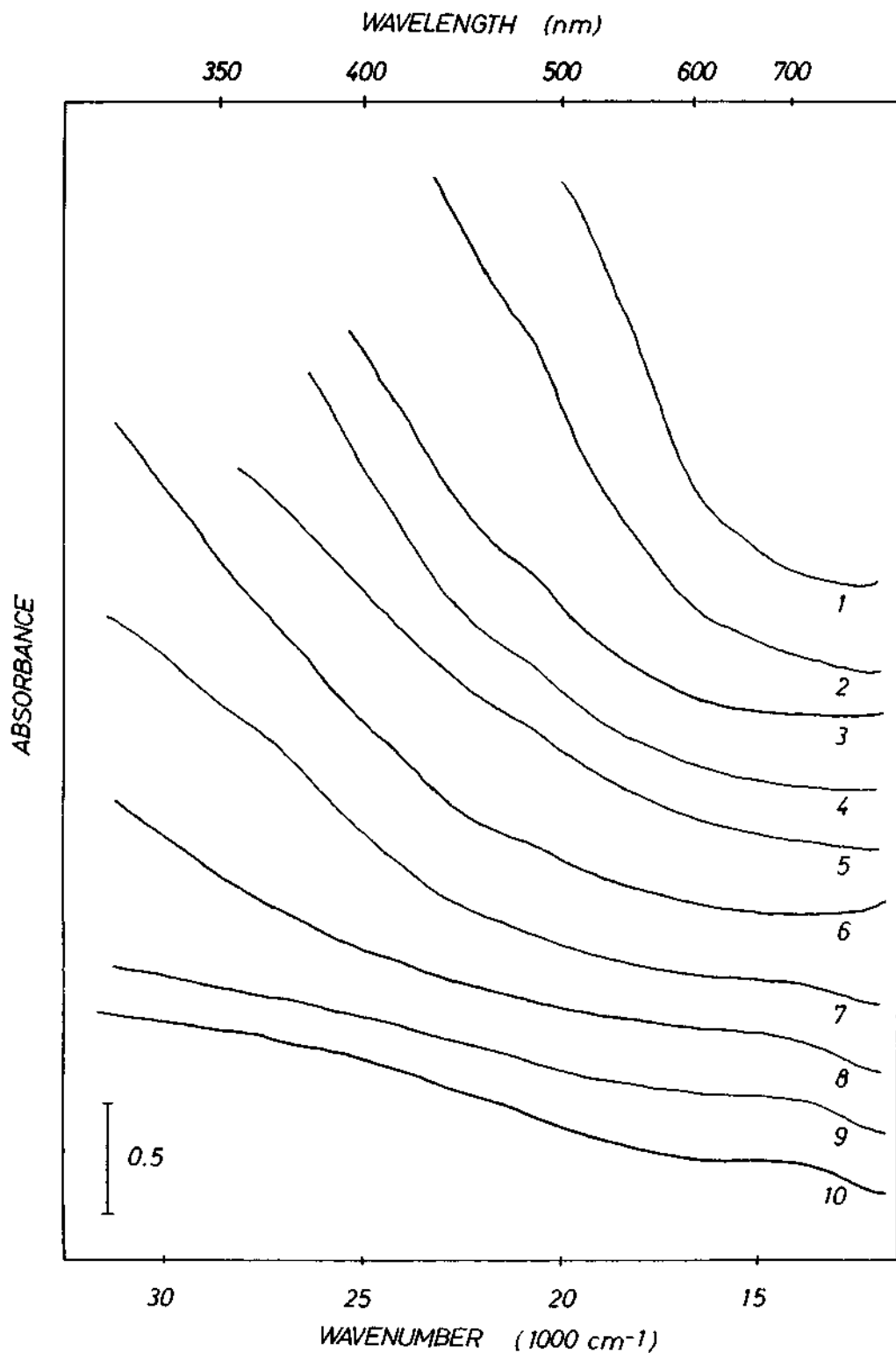


Fig. 2. Absorption spectra of natural citrines and natural prasiolites (heat-treated natural amethysts); citrines: 1 Brazil, 2 Brazil, 3 Brazil, 4 Uruguay, 5 Brazil, 6 Maramba, Brazil, 7 Bahia, Brazil; prasiolites: 8 Jakovina, Brazil, 9 Montezuma, Brazil, 10 Zambia.



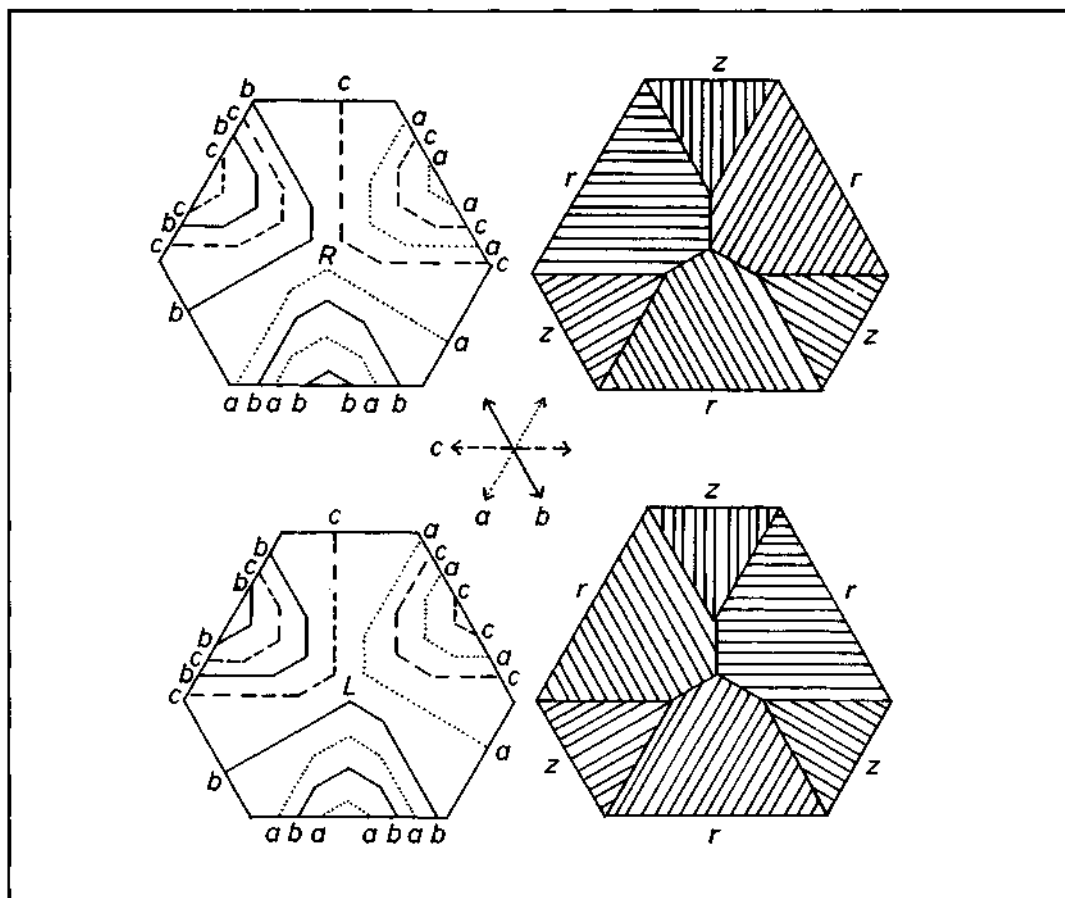


Fig. 3. Idealized drawings of natural citrine (heat-treated natural amethyst) in a view parallel to the  $c$ -axis showing the model of polysynthetic twinning on the Brazil law after McLaren and Pitkethly (1982) and the orientation of striations in growth sectors confined to the major and minor rhombohedron  $r$  and  $z$ .

Left: quartz crystals with right-handed (R) or left-handed (L) quartz in the centre. Brewster fringes (extinction bands) in growth sectors confined to the major rhombohedral faces  $r$  ( $10\bar{1}1$ ) separate lamellae of right-handed and left-handed quartz, a single twin boundary enters each growth sector confined to the minor rhombohedron  $z$  ( $01\bar{1}1$ ).

Centre: the directions of the double-headed arrows (which are represented by the symbols  $a$ ,  $b$  and  $c$ ) correspond to traces of the predominant twin boundaries; in subsequent Brewster fringes, alternating directions of predominant twin boundaries are observed.

Right: directions of orientated striations in growth sectors confined to the major and minor rhombohedron  $r$  and  $z$  in crystals with right-handed and left-handed quartz in the centre.

extinction bands (so-called Brewster fringes) in polarized light under crossed polarizers provided that the optic axis of the amethyst is arranged parallel to the line of sight. The investigation is possible in the horizontal microscope in combination with an immersion cell and immersion liquids. For convenient handling, the use of an improved sample holder with horizontal and vertical rotation axes is suggested (Schmetzer, 1985, 1986).

A somewhat simplified method was described by Crowningshield *et al.* (1986), using a standard gemmological polariscope with an immersion cell.

According to the experience of the present author, both versions of the method yield similar or identical results for large cut stones. However, for smaller stones in the range of about 0.5 to 2.0 carats in weight, the application of the microscope may be necessary. In addition, samples from distinct localities, e.g. samples from Pará, Brazil, reveal an extremely complicated twinning structure. For the recognition of such samples as well as for a distinction from that particular type of twinning, which is found in synthetic amethyst (cf. Lind and Schmetzer, 1985, 1987), the author feels that the application

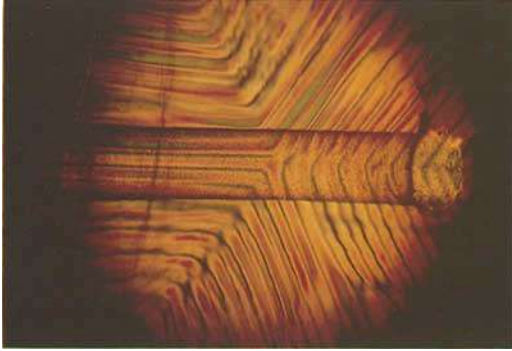


Fig. 4

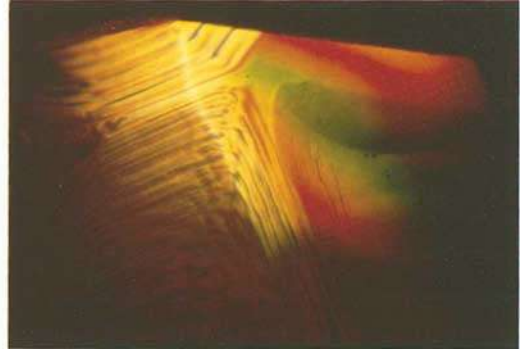


Fig. 5

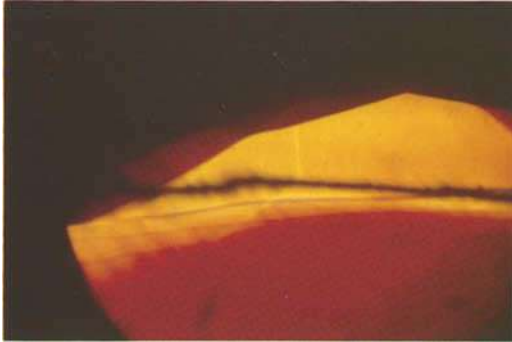


Fig. 6

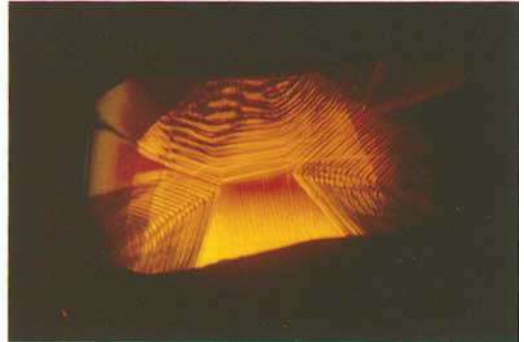


Fig. 7



Fig. 8



Fig. 9

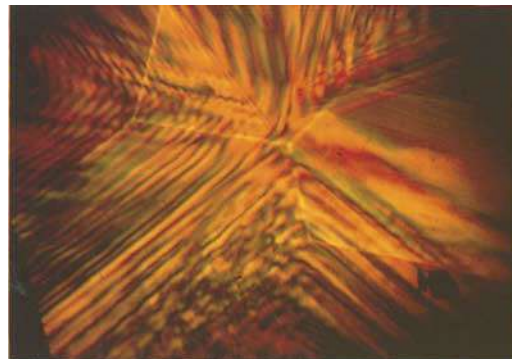


Fig. 10

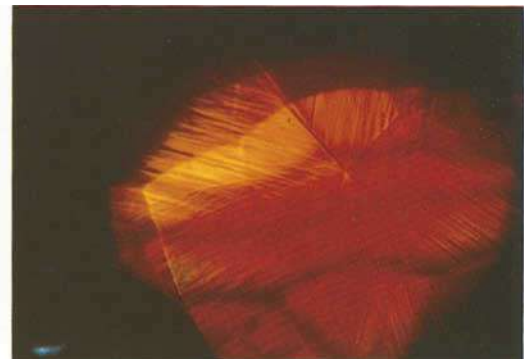


Fig. 11

of the gem microscope yields more reliable results. Furthermore, diagnostic structures in heat-treated amethyst may be extremely complicated thus favouring the investigation by use of the microscope.

In heat-treated natural amethyst two basic types of diagnostic structures and one group of samples with an intermediate type of characteristics are observable. In the first group of samples, the twinning structures of untreated amethyst are completely preserved during the annealing process. The growth sectors of the stones, which are confined to the major rhombohedron  $r$   $\{10\bar{1}1\}$ , consist of polysynthetically twinned lamellae of alternating right-handed and left-handed quartz. The growth sectors confined to the minor rhombohedron  $z$   $\{01\bar{1}1\}$  do not show this type of twinning on the Brazil law; only one single twin boundary entering each  $z$  growth sector is observed (Figure 3, for a more detailed description the reader is referred to the papers of Schlössin and Lang, 1965; McLaren and Pitkethly, 1982; Schmetzer, 1985, 1986, 1987 as well as Crowningshield *et al.*, 1986). Typical microphotographs of interference figures of this group of samples are pictured in Figures 4-6.

The second important group of heat-treated amethyst does not show interference figures which are related to polysynthetic twinning on the Brazil law. In other words, investigations of this group of samples under crossed polarizers yield interference figures which are comparable to the interference patterns of quartz crystals, which do not consist of

polysynthetic lamellae of right-handed and left-handed quartz. However, growth sectors confined to the major and minor rhombohedron  $r$  and  $z$  are still observable in the gem microscope. They are frequently recognizable due to different colour intensities with a more intense brown or yellow hue in growth sectors confined to the major rhombohedron  $r$  and a less intense brownish or yellow hue in sectors confined to the minor rhombohedron  $z$  (cf. colour zoning in heat-treated bicolour amethyst-citrine quartz; Nassau, 1981). In addition, boundaries between different growth sectors are distinctly outlined and orientated striations of dark brown colour are found in different directions confined to different growth sectors  $r$  and  $z$ , respectively (Figures 11, 12). In growth sectors confined to the minor rhombohedron  $z$   $\{01\bar{1}1\}$ , these brown striations are orientated perpendicular to the three prism faces  $\{0110\}$ . In growth sectors confined to the major rhombohedron  $r$   $\{10\bar{1}1\}$ , two different orientations of the brown striations exist, which are dependent on the nature of the centre of the untreated crystal, which may consist of right-handed or left-handed quartz. Schematic line drawings of the two possibilities mentioned are given in Figure 3. At present, no description or interpretation of this particular type of orientated striations are known to the present author from literature. Most probably, the disappearance of polysynthetic twinning on the Brazil law as well as the development of orientated brown striations is due to the annealing of natural samples. However, additional

Fig. 4. Natural citrine (heat-treated natural amethyst) from Brazil; interference figure showing polysynthetic twinning in three growth sectors confined to the major rhombohedron  $r$ ; a drill-hole is running through the centre of the bead. Crossed polarizers. 20x.

Fig. 5. Natural citrine (heat-treated natural amethyst) from Maramba, Brazil; interference figure showing polysynthetic twinning in two growth sectors confined to the major rhombohedron  $r$  and not in growth sectors confined to the minor rhombohedron  $z$ . Crossed polarizers. 16x.

Fig. 6. Natural citrine (heat-treated natural amethyst) from Brazil; interference figure showing a single boundary entering one growth sector confined to the minor rhombohedron  $z$ . Crossed polarizers. 30x.

Fig. 7. Natural citrine (heat-treated natural amethyst) from Brazil; distorted interference figure showing polysynthetic twinning in three growth sectors confined to the major rhombohedron  $r$  and not in growth sectors confined to the minor rhombohedron  $z$ ; a single twin boundary enters one growth sector confined to the minor rhombohedron  $z$ ; striations orientated according to the growth sector they are confined to. Crossed polarizers. 14x.

Fig. 8. Natural citrine (heat-treated natural amethyst) from Brazil; distorted interference figure showing polysynthetic twinning in three growth sectors confined to

the major rhombohedron  $r$  and not in growth sectors confined to the minor rhombohedron  $z$ ; a single twin boundary enters one growth sector confined to the minor rhombohedron  $z$ ; striations orientated according to the growth sector they are confined to. Crossed polarizers. 26x.

Fig. 9. Natural citrine (heat-treated natural amethyst) from Brazil; highly distorted interference figure showing polysynthetic twinning in three growth sectors confined to the major rhombohedron  $r$  and not in growth sectors confined to the minor rhombohedron  $z$ ; striations orientated according to the growth sector they are confined to. Crossed polarizers. 20x.

Fig. 10. Natural citrine (heat-treated natural amethyst) from Uruguay; highly distorted interference figure showing polysynthetic twinning in three growth sectors confined to the major rhombohedron  $r$  and not in growth sectors confined to the minor rhombohedron  $z$ ; striations orientated according to the growth sector they are confined to. Crossed polarizers. 20x.

Fig. 11. Natural citrine (heat-treated natural amethyst) from Brazil; interference figure showing no polysynthetic twinning on growth sectors confined to the major and minor rhombohedra  $r$  and  $z$ ; striations orientated according to the growth sector they are confined to. Crossed polarizers. 40x.

investigations are necessary in order to clarify details of the process. Using a simplified technique without microscopic examination as described by Crowningshield *et al.* (1986), this second group of natural citrine originating from heat-treated amethyst yields the interference figure of untwinned quartz under the polariscope. Consequently, this type of heat-treated amethyst does not behave in the polariscope in the same manner as natural amethyst.

The third intermediate group of samples of heat-treated natural amethyst shows more or less distorted interference figures as well as more or less intense orientated brown striations (Figures 7-10). These observations are explainable by a decrease of polysynthetic twinning on the Brazil law parallel to a development of orientated striations, the directions of which are dependent on the respective growth sector which they are confined to.

Lamellar growth structures, parallel to the major and minor rhombohedron, which are frequently connected with colour zoning, are most common in natural amethyst. These zonal growth structures are still observable in all types of heat-treated natural amethyst. In most samples, sharp lamellar structures parallel to several rhombohedral faces are found, incidentally growth structures parallel to the hexagonal prism faces were also present. In a high percentage of samples, families of straight parallel growth planes parallel to the major and minor rhombohedron were observed, which form two characteristic angles of  $94^\circ$  (Figure 13, formed by the two faces  $r$  and  $r'$  or  $z$  and  $z'$ ),  $134^\circ$  (Figure 14; formed by two adjacent rhombohedral faces  $r$  and  $z$ ) as well as  $76^\circ$  (Figure 15; formed by two opposite rhombohedral faces  $r$  and  $z$ ). Angular growth structures between prism and rhombohedral faces, forming an angle of  $142^\circ$  were found to be extremely rare.

According to information from the trade, only internally flawless material of natural amethyst without healing feathers consisting of liquid and two-phase inclusions are selected for heat treatment due to the formation of fissures and cracks in impure samples. Thus, the absence of healing feathers in commercially heat-treated amethyst is explained, which was confirmed by microscopic investigations of numerous samples. Only incidentally, the presence of black or reddish-brown solid state inclusions of iron oxides or hydroxides was observed (Figure 16).

### III.2.2 NON-ARTIFICIALLY-IRRADIATED NATURAL CITRINE

Natural citrines in the colour sequence light yellow, yellow, brownish-yellow and greyish-yellow were available from localities in Bahia and Goyaz, Brazil, from Salamanca, Spain and Madagascar

(Figure 1). According to the informations given by the suppliers, these samples were not artificially heat-treated. However, samples of this type of citrine with more intense smoky coloration are known for a long time in the trade. In samples of this particular type (natural smoky quartz), smoky coloration is commercially removed by heat-treatment.

The samples available for the present investigation were not altered by heat treatment up to about  $375^\circ\text{C}$ . At  $400^\circ\text{C}$  the yellow coloration became lighter and was completely bleached at temperatures between  $450$  and  $550^\circ\text{C}$ . Exposition of heat-treated (colourless) and non-heat-treated (yellow) samples to gamma rays (dose rate about  $30$  Mrads) or neutrons (integrated fluence rate about  $3 \times 10^{15}$  n/cm<sup>2</sup>) turned all samples an intense smoky coloration, which was bleached at temperatures between about  $225$  and  $275^\circ\text{C}$ . The resulting yellow or greenish-yellow coloration was stable up to  $450$ - $550^\circ\text{C}$  in samples, which were not bleached artificially before and up to  $325^\circ\text{C}$  in artificially bleached samples. These results indicate, that an almost identical yellow or greenish-yellow coloration which is caused by colour centres with lower stability was induced in colourless samples or superimposed on the natural yellow coloration in unbleached samples by exposure to gamma rays and neutrons. This type of artificially-induced coloration will be described in the following chapter, more experimental details have been published elsewhere (Schmetzer, 1988).

The absorption spectra of all samples of non-artificially-irradiated citrine from different localities (Figure 17) consist of an absorption band in the ultraviolet, the low energy tail of which extends to the visible region causing a continuously increasing absorption from red to violet. In some samples an additional weak absorption band at about  $15,500$  cm<sup>-1</sup> ( $645$  nm) was measured (Figure 17).

Microscopic examinations of this type of natural citrine reveal the presence of diagnostic properties only in part of the samples. Some rough or cut stones were internally flawless and did not show growth structures or twinning on the microscope scale, i.e. no interference figures of polysynthetically twinned quartz were observed (Figure 18). Incidentally, in stones from Brazil and Madagascar thin plates or bodies were found to be inserted in the crystals (Figure 19). Most probably, the samples are macroscopically twinned on the Brazil law, as described comprehensively by various authors, e.g. Frondel (1962).

In samples from Brazil and Spain, growth structures were sometimes observed in the form of single growth planes which are orientated parallel to the prism faces  $m$   $\{1120\}$  as well as parallel to the rhombohedral faces  $r$   $\{1011\}$  and  $z$   $\{0111\}$

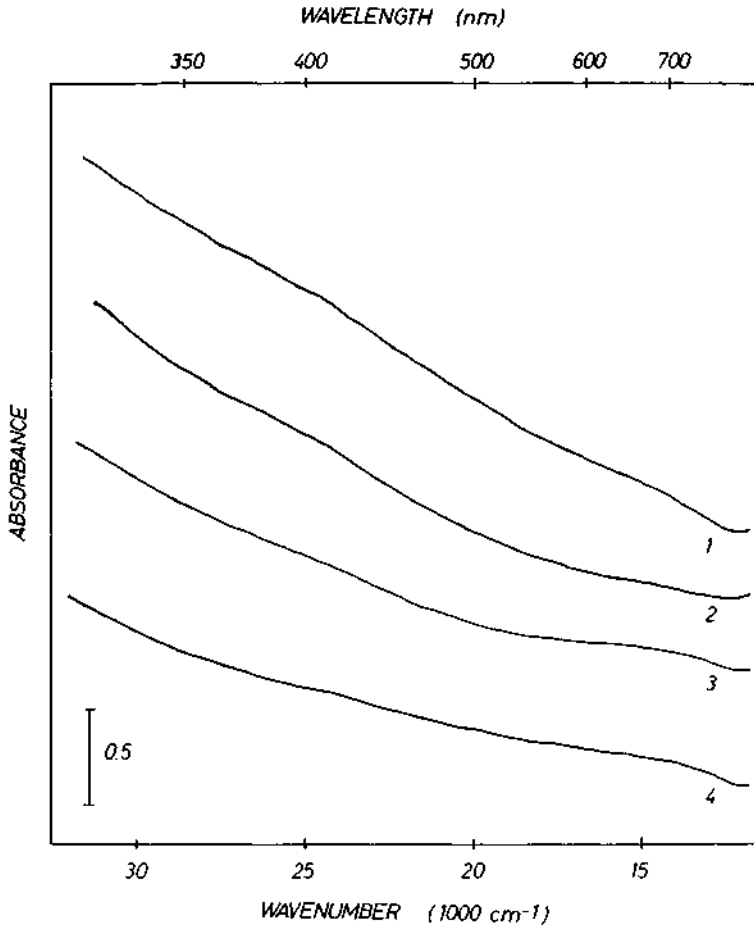


Fig. 17. Absorption spectra of natural citrines; 1 Salamanca, Spain, 2 Goyaz, Brazil, 3 Madagascar, 4 Bahia, Brazil.

of the crystals (Figures 20, 21). Occasionally, in citrines from Goyaz, Brazil, double refracting mineral inclusions were found to be confined to the growth boundaries parallel to prism and rhombohedral faces (Figure 21). These mineral inclusions were not yet determined exactly. In citrine crystals from all localities investigated, healing feathers consisting of liquid and two-phase inclusions were also present incidentally (Figure 22).

### III.2.3 ARTIFICIALLY-IRRADIATED NATURAL CITRINE (ORIGINALLY COLOURLESS)

Some samples of artificially-irradiated greenish-yellow citrine from unknown localities were made available for investigation. The colour was completely bleached at 325 or 350°C. After irradiation with gamma rays (dose rate about 30 Mrads), the stones turned an intense greyish-black (smoky) or a

lighter yellowish-grey colour. Subsequent heat treatment at 200°C bleached part of the irradiation induced smoky colour centres. After annealing of the samples at 225, 250 or 275°C, the smoky colour centres were completely bleached and the colour of the samples was identical to the as-received form. Subsequent heat treatment to 325 or 350°C again bleached the greenish-yellow colour.

Absorption spectra of one typical sample are presented in Figure 23. In the as-received greenish-yellow coloration, the absorption spectrum of the quartz crystal is composed of an absorption band in the ultraviolet, the low energy tail of which extends to the visible region. This increasing absorption from red to violet is superimposed by two absorption bands with maxima at about 15,500 and 26,000  $\text{cm}^{-1}$  (645 and 385 nm). After irradiation, the

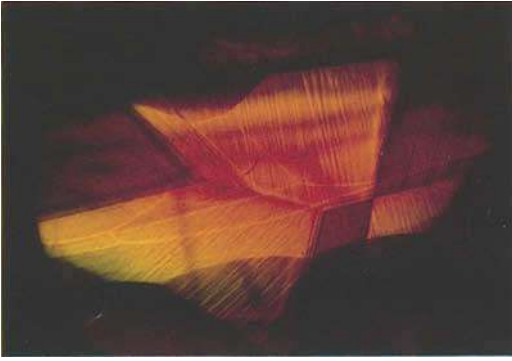


Fig. 12. Natural citrine (heat-treated natural amethyst) from Brazil; interference figure showing no polysynthetic twinning on growth sectors confined to the major and minor rhombohedra  $r$  and  $z$ ; striations orientated according to the growth sector they are confined to. Crossed polarizers. 30x.

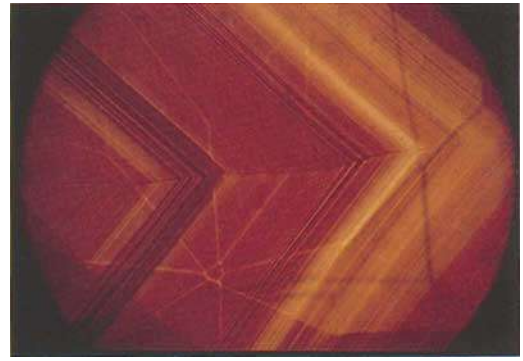


Fig. 13. Natural citrine (heat-treated natural amethyst) from Brazil; families of straight parallel growth planes parallel to the major rhombohedron  $r$  and  $r'$   $\{1011\}$  forming an angle of  $94^\circ$ . 30x.



Fig. 14. Natural citrine (heat-treated natural amethyst) from Brazil; families of straight parallel growth planes parallel to the major rhombohedron  $r$   $\{1011\}$  and parallel to the minor rhombohedron  $z$   $\{0111\}$  forming an angle of  $134^\circ$ , different colour intensities are observable in the growth sector confined to  $r$  (dark orange-brown) and the growth sector confined to  $z$  (light orange-brown). 30x.



Fig. 15. Natural citrine (heat-treated natural amethyst) from Brazil; families of straight parallel growth planes parallel to the major rhombohedron  $r$   $\{1011\}$  and parallel to the minor rhombohedron  $z$   $\{0111\}$  forming an angle of  $76^\circ$ , different colour intensities are observable in the growth sector confined to  $r$  (dark orange-brown) and the growth sector confined to  $z$  (light orange-brown). 26x.

additional absorption bands of smoky colour centres with absorption maxima at about  $22,200\text{ cm}^{-1}$  ( $450\text{ nm}$ ) are observable. Heat treatment between  $200$  and  $325^\circ\text{C}$  in intervals of  $25^\circ\text{C}$  each (annealing time  $0.5\text{ h}$  at each step) caused first a bleaching of these additional smoky colour centres and subsequently a bleaching of the group of three absorption bands at  $15,500$  and  $26,000\text{ cm}^{-1}$  and in the ultraviolet (Figure 23).

These results indicate that the greenish-yellow quartz crystals received from the trade are artificially irradiated samples, the smoky coloration of which were bleached by heat treatment at about  $250^\circ\text{C}$ . After complete bleaching, the smoky and greenish-yellow colour centres are restored by re-irradiation.

By microscopic investigations, the presence of small healing feathers in part of the citrines of this type was observed.

#### III.2.4 HEAT-TREATED SYNTHETIC AMETHYST

Synthetic amethysts commercially grown in Japan and USSR were annealed at various temperatures. The amethyst colour centres bleached at about  $460^\circ\text{C}$  and the samples turned colourless. No yellow or brown coloration was developed at annealing temperatures and times up to  $600^\circ\text{C}$  and  $24\text{ h}$ , respectively. However, the violet amethyst coloration was completely restored after subsequent gamma irradiation. This unexpected result indicates a stabilization of both precursor centres of the amethyst colour centre, i.e. trivalent iron on substituent

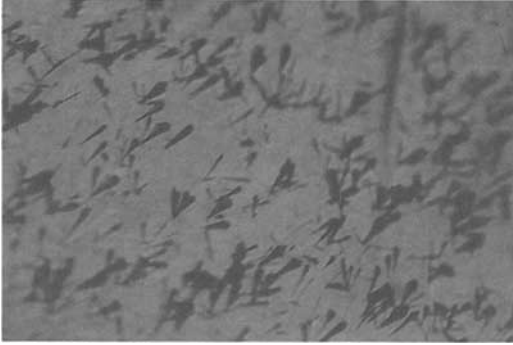


Fig. 16. Natural citrine (heat-treated natural amethyst) from Namibia; black mineral inclusions (Fe-oxides or hydroxides). 65x.

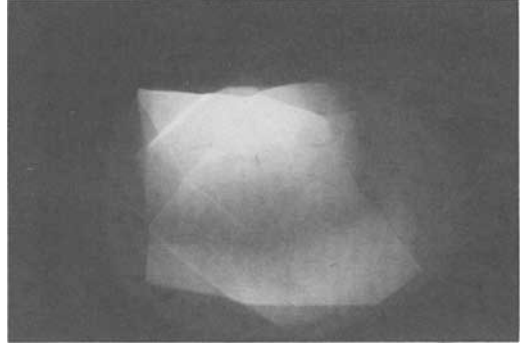


Fig. 18. Natural citrine from Goyaz, Brazil; interference figure of an optically untwinned crystal. 20x.

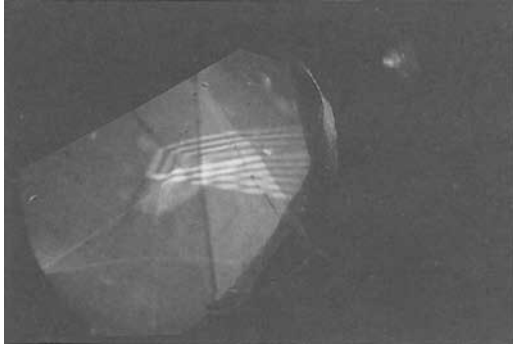


Fig. 19. Natural citrine from Brazil; isolated Brazil twin within an optically untwinned crystal showing interference colours and interference striations at the twin boundaries. Crossed polarizers. 16x.



Fig. 20. Natural citrine from Brazil; growth planes parallel to the major rhombohedron  $r(10\bar{1}1)$  and parallel to the minor rhombohedron  $z(01\bar{1}1)$  forming an angle of  $76^\circ$ . 34x.

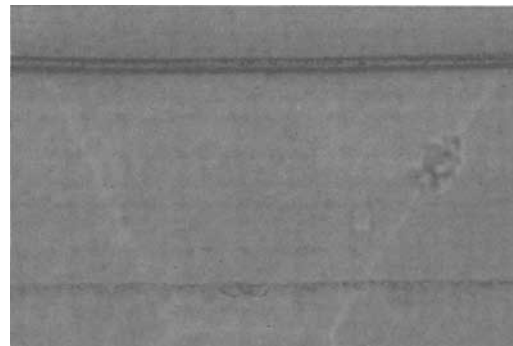


Fig. 21. Natural citrine from Brazil; growth planes parallel to the hexagonal prism  $(10\bar{1}0)$ , double refracting mineral inclusions are confined to one growth plane. 45x.

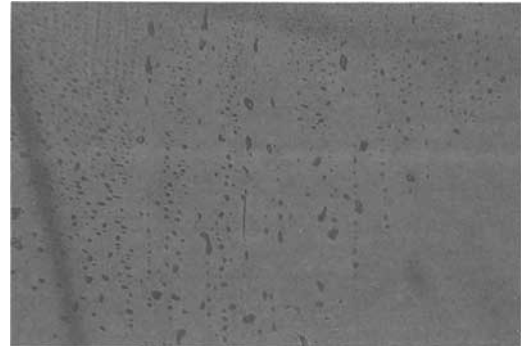


Fig. 22. Natural citrine from Goyaz, Brazil; healing feather consisting of liquid and two phase inclusions. 45x.

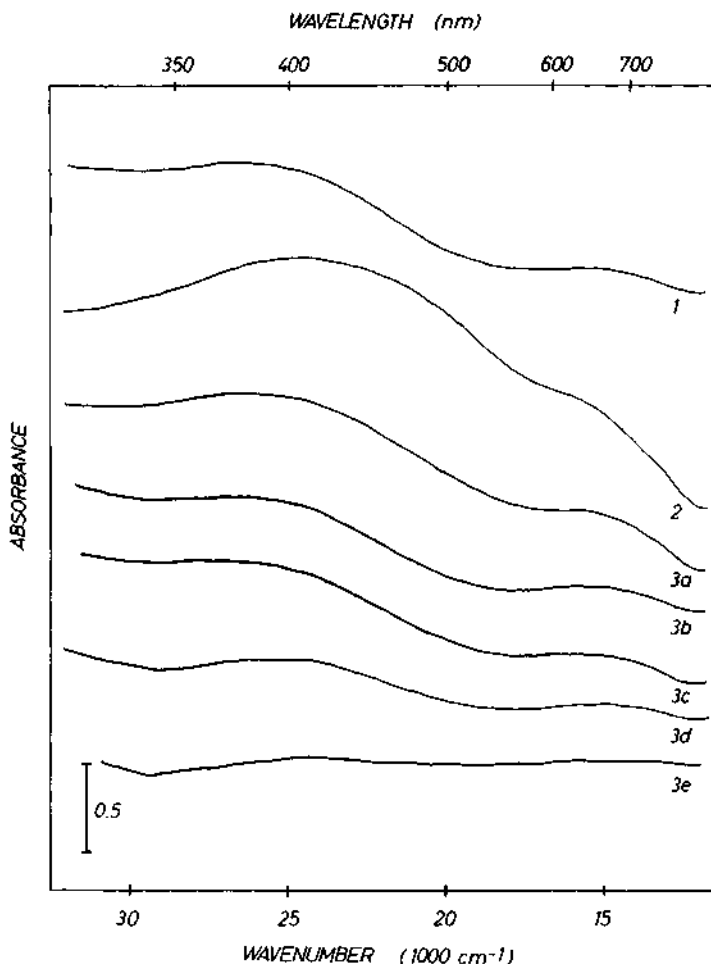


Fig. 23. Absorption spectra of artificially-irradiated natural citrine (originally colourless); 1 sample in the as-received form (greenish-yellow); 2 sample after heat treatment at 325°C (colourless) and subsequent gamma irradiation (yellowish-grey); 3a-e irradiation-treated sample after subsequent heat treatment at 200°C (3a, greyish-yellow), 225°C (3b, greenish-yellow), 250°C (3c, greenish-yellow), 275°C (3d, light greenish-yellow), 300°C (3e, very light yellowish), 325°C (not pictured, colourless).

tional silicon sites and on interstitial sites of distorted tetrahedral symmetry, which is different from the stabilization of the amethyst precursor centres in most natural samples.

### III.2.5 SYNTHETIC FE-CONTAINING YELLOW, GREEN AND BROWN QUARTZ (INCLUDING FE- AND CO-BEARING SAMPLES)

Most samples of synthetic Fe-bearing quartz with yellow, green and brown coloration, which were made available for the present study, were supplied from various cutting factories or gem collectors in Idar-Oberstein (Figure 24). In all cases, the country of production was the only

information which was given by the trade. According to their diagnostic features, the samples were subdivided into several groups with identical or similar properties. In some cases, it was evident that a distinct type of synthetic Fe-containing citrine was grown by different producers using closely related or almost identical growth conditions. In other cases, it may be possible that samples from different producers or different producing countries were mixed with or without intention. Therefore, the present author feels that a subdivision and description of the samples according to their diagnostic properties is more suitable than a description according to the producer or producing country.



In addition, it has to be pointed out that, according to diagnostic properties, only part of the samples are easily assigned to a distinct type of synthesis as described in Sections II.4 and II.6. In other cases, some of the observed properties, e.g. absorption bands in the visible, are not known to the present author from literature. Thus, in order to work out at least some details of the production processes, trace element analyses were carried out using atomic absorption spectroscopy. A general survey of the samples investigated and some of their diagnostic properties is given in Table 2, analytical data are presented in Table 3, and absorption spectra are pictured in Figures 25-29.

The absorption spectra of all samples investigated consist of a strong absorption maximum in the ultraviolet, the low energy tail of which extends to the visible area. In all samples with the exception of KCl-NaCl-grown synthetic citrine (type 5, Fi-

gure 27), the continuously increasing absorption from red to violet is superimposed by several weak and strong absorption bands. These bands are assigned to  $\text{Co}^{3+}$  ( $I_6$ ) in the case of intense yellow to intense yellow-orange citrines (type 6, Figure 28). This assignment is confirmed by analytical data (Table 3). In addition, it was observed that the colour of  $\text{Co}^{3+}$ - and Fe-containing synthetic citrines turned blue by heat treatment at 600°C, which is explained by a conversion of  $\text{Co}^{3+}$  ( $I_6$ ) to  $\text{Co}^{2+}$  ( $I_4$ ) (Figure 28). In green samples of type 7 a strong absorption band of  $\text{Fe}^{2+}$  ( $I_6$ ) at 13,500  $\text{cm}^{-1}$  is observed, which is consistent with literature data (Figure 29).

Several weak absorption bands measured in the spectra of brown, orange-brown, yellow-orange and green samples (Table 2, types 1, 2, 7, Figures 25, 29) are in good agreement with literature data (Lehmann, 1971a; Khaliov *et al.*, 1979; cf. Table 1).

Table 2. General survey of synthetic Fe-containing yellow, green and brown quartz investigated, including Fe- and Co-bearing samples

designation	type 1	type 2	type 3	type 4	type 5	type 6	type 7
country of production	USA, USSR	Japan, USSR	Japan, USSR	Japan, USSR	Japan	USA, USSR	USSR
colour	intense brown or intense orange-brown	intense yellow-orange	intense orange-brown	intense orange-brown	intense yellow-orange	intense yellow or intense yellow-orange	light green or intense green
working solution	$\text{K}_2\text{CO}_3$	$\text{K}_2\text{CO}_3$	$\text{K}_2\text{CO}_3$	$\text{K}_2\text{CO}_3$	KCl	$\text{K}_2\text{CO}_3$	$\text{K}_2\text{CO}_3$
absorption maxima ( $\text{cm}^{-1}$ )	18,700 20,200 21,500  22,500  24,800 26,500	20,200 21,500	22,000  23,500 24,500	22,000  23,500 24,500		26,800 broad, strong	13,500 broad, strong  20,200 21,500  24,800 26,500  29,000
optical twinning	not observed	not observed	not observed	not observed	not observed	not observed	not observed
growth inhomogeneities and/or seed plane	(0001)	(0001)	(0001)	(0001) channels    c distorted in-figure	not observed X-cut and Y-bar seeds were used*	(0001)	not observed

\*Hosaka and Taki (1983)

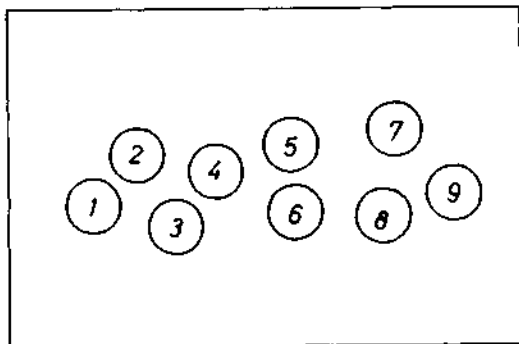


Fig. 24. Colour sequence of synthetic citrines: sample 1: type 1, produced in USSR; sample 2: type 4, produced in Japan; sample 3: type 4, produced in Japan; sample 4: type 3, produced in USSR; sample 5: type 3, produced in USSR; sample 6: type 4, produced in Japan (heat-treated); sample 7: type 6, produced in USA; sample 8: type 3, produced in Japan; sample 9: type 6, produced in USSR. Size of sample 2 approx.  $11 \times 14$  mm. Photo by O. Medenbach, Bochum, FRG.

They are assigned to spin-forbidden transitions of trivalent iron in interstitial sites of distorted tetrahedral symmetry,  $Fe^{3+} (I_4)$ . In contrast, the positions of several weak absorption maxima in the spectra of orange-brown synthetic citrines (Table 2, types 3 and 4, Figure 26) are distinctly shifted and thus, an assignment of these absorption maxima is rather uncertain. The different spectroscopic properties of various samples are certainly caused by differing growth conditions. However, without an exact knowledge of the compositions of the working solutions (e.g. concentrations of  $K_2CO_3$  and  $Fe_2O_3$ ), a more detailed interpretation of varying spectroscopic properties is impossible. An influence of different oxidizers such as  $LiNO_2$ ,  $LiNO_3$ ,  $KMnO_4$  is also assumable (cf. manganese concentrations in samples of these particular types, Table 3).

All samples investigated were optically single crystals (Figure 30) and did not consist of macroscopically twinned or polysynthetically twinned individuals on the Brazil law (cf. Figures 4-12). Only in samples of type 4, an extremely distorted interference figure is observed when the stones are viewed parallel to the optical axis (Figures 31-32). An examination in a direction perpendicular to the optical axis reveals the presence of fibrous structures, the fibre axes of which are running parallel to the optical axis (Figure 33). Incidentally, liquid and two-phase inclusions are trapped within those channels. Similar fibrous or channel-like structures parallel to the  $c$ -axis [0001] were mentioned by Chadschi and Reschetowa (1975) and Khadzhi and Reshetova (1977). These capillary channels penetrating into the citrine crystals are due to the absence

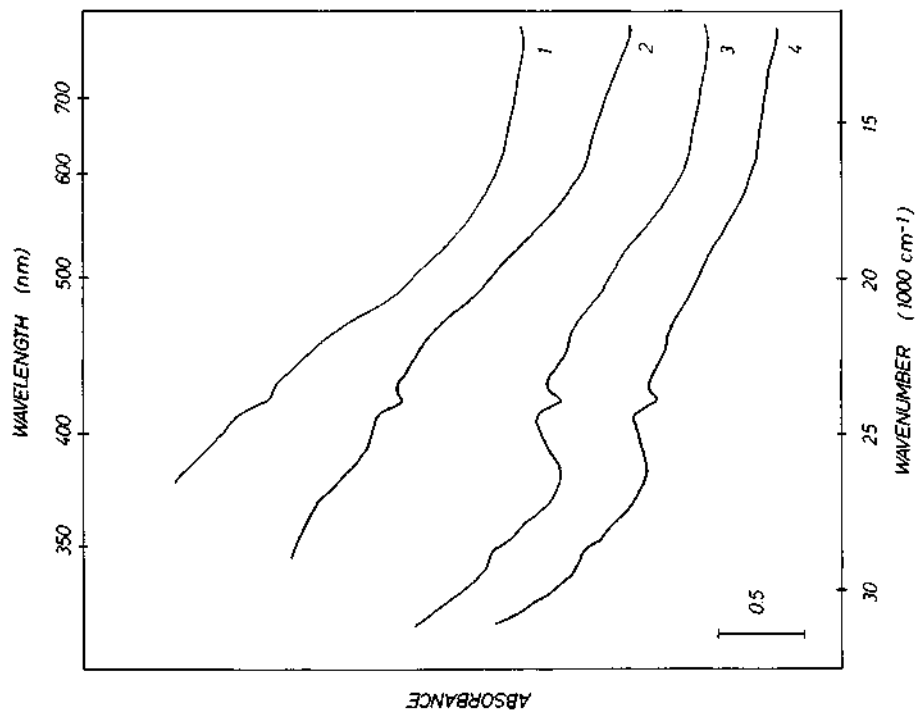


Fig. 26. Absorption spectra of synthetic citrines of types 3 and 4; type 4: 1 produced in USSR, 2 produced in Japan, type 3: 3 produced in Japan, 4 produced in USSR.

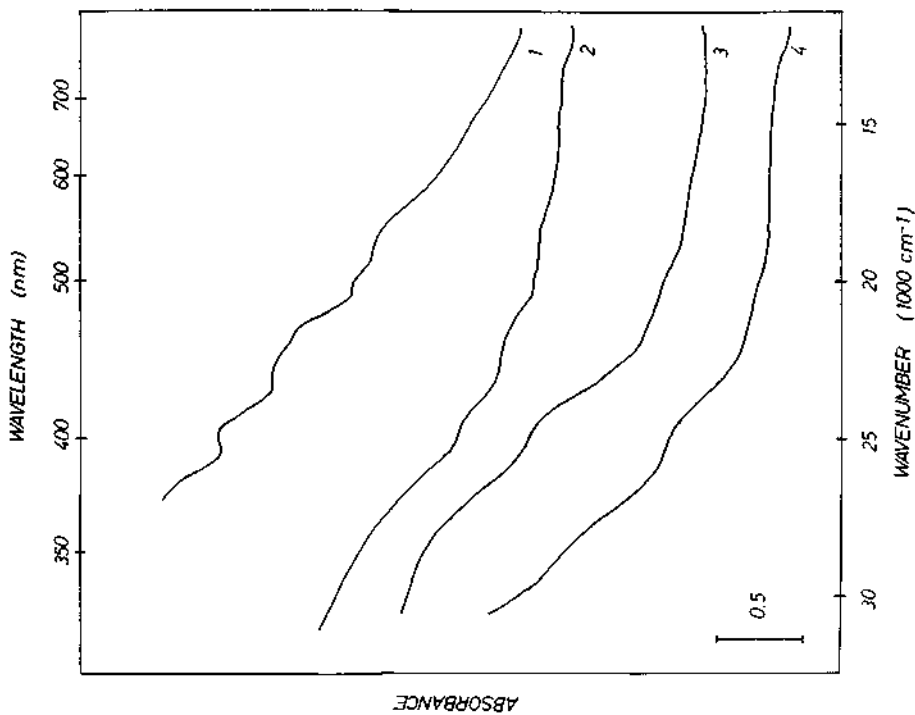


Fig. 25. Absorption spectra of synthetic citrines of types 1 and 2; type 1: 1, 2 produced in USSR; type 2: 3 produced in Japan, 4 produced in USSR.

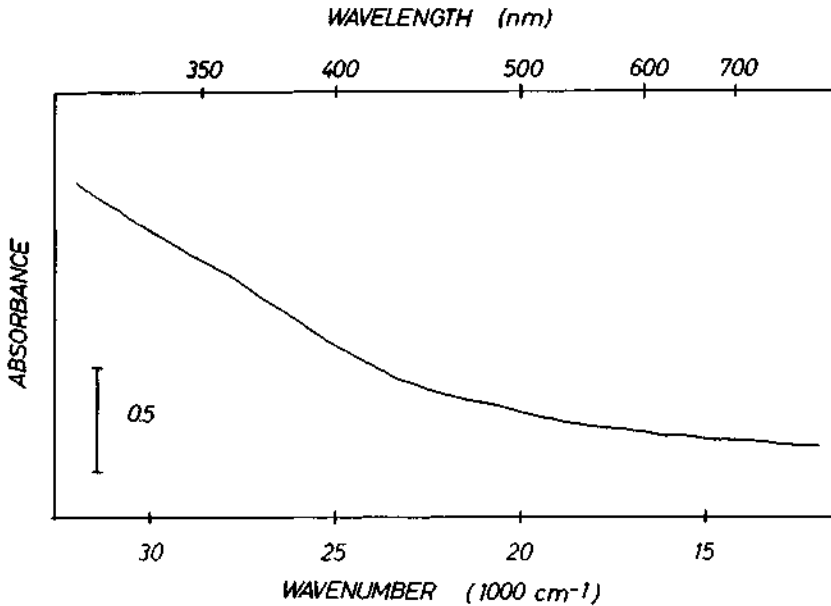


Fig. 27. Absorption spectra of synthetic citrine of type 5 produced in Japan.

of lithium nitrates or nitrites in growth media, which form hardly soluble silicates, e.g. eucryptite, and purify the solution from aluminium. Due to the paper of Sato (1986), the channel-like structures parallel to the optic axis are also observable using laser tomography.

According to microscopic examinations all samples of synthetic Fe-bearing citrine with the exception of type 5 and possibly some samples of type 7 are grown from basal seeds and thus reveal more or less pronounced growth structures and colour zoning parallel to the basal plane (0001) (Figure 34). Growth structures parallel to the basal pinacoid are not observed in natural citrine and natural heat-treated amethyst and, thus, basal growth structures and colour zoning unequivocally indicate synthetic

citrines. However, it has to be mentioned, that some faceted synthetic citrines were cut from samples which were obviously grown under homogeneous conditions and, therefore, the samples do not display any growth inhomogeneities in the gem microscope. These observations are consistent with the literature dealing with crystal growth of synthetic citrines (cf. Sections II.4 and II.6).

Only occasionally, solid state inclusions in the form of the so-called breadcrumb inclusions are observable in synthetic citrine (Figure 35). In stones of type 6, needle-like inclusions which are orientated parallel to the optical axis are rarely found. Feathers consisting of liquid and two-phase inclusions are also very rare.

Table 3. Chemical data of synthetic Fe-containing yellow and brown quartz investigated, including Fe- and Co-bearing samples (in ppm)

designation	type 1	type 2	type 3		type 4		type 5	type 6	
country of production	USA	Japan	USSR		Japan	USSR	Japan	USA	USSR
colour	intense brown	intense yellow-orange	intense orange-brown	intense orange-brown	intense orange-brown	intense orange-brown	intense yellow-orange	intense yellow-orange	intense yellow-orange
Na	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
K	354	777	376	460	1045	694	150	1120	935
Fe	1850	1350	455	580	870	1160	2830	960	880
Co	< 10	< 10	< 10	< 10	< 10	< 10	< 10	130	60
Mn	16	17	36	33	64	87	29	5	5

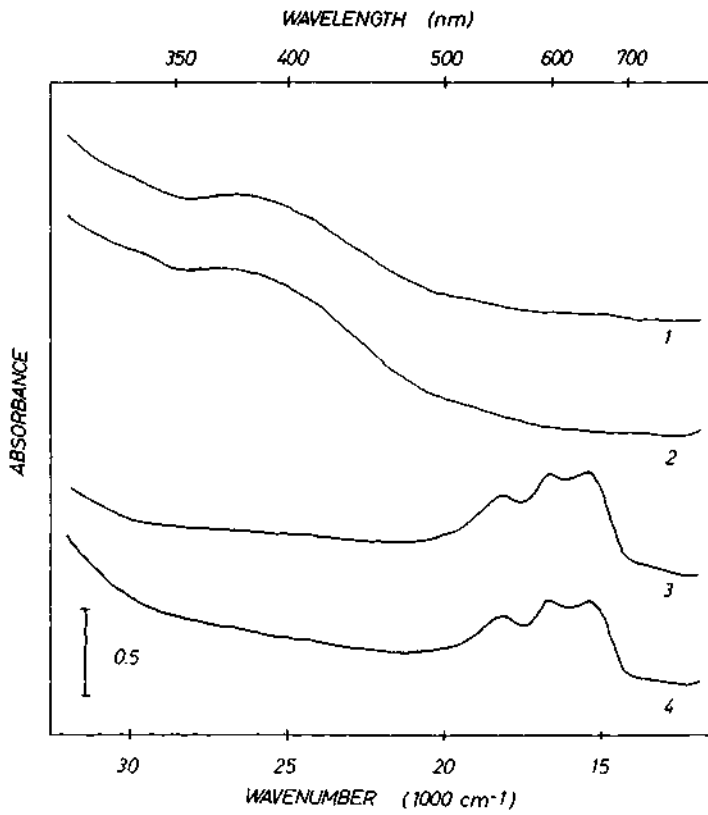


Fig. 28. Absorption spectra of synthetic citrines of type 6; samples in the as-received form: 1 produced in USSR, 2 produced in USA; samples converted to a blue colour by heat treatment: 3 originally sample 1, 4 originally sample 2.

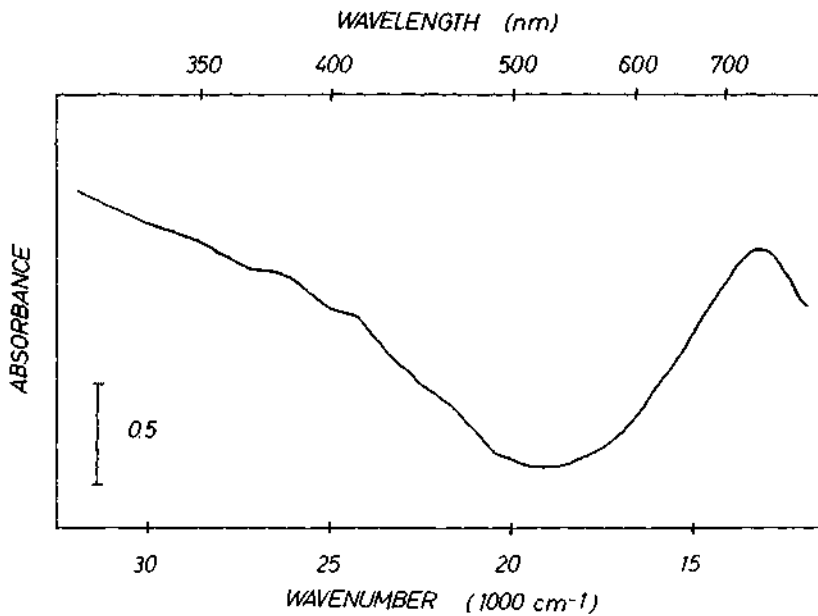


Fig. 29. Absorption spectrum of synthetic prasiolite of type 7 produced in USSR.

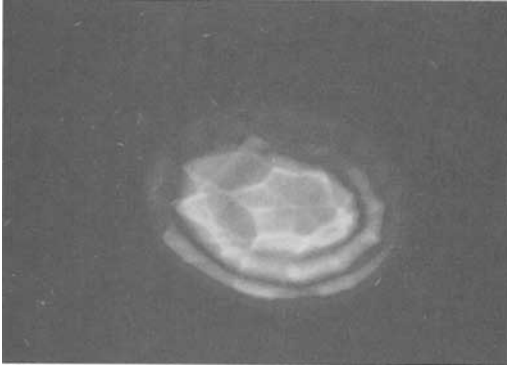


Fig. 30. Synthetic citrine of type 3 produced in Japan; interference figure of an optically untwinned crystal. Crossed polarizers. 14x.



Fig. 31. Synthetic citrine of type 4 produced in Japan; highly distorted interference figure. Crossed polarizers. 18x.

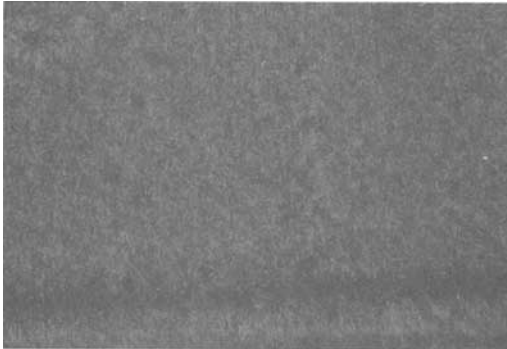


Fig. 32. Synthetic citrine of type 4 produced in Japan; highly distorted interference figure. Crossed polarizers. 35x.

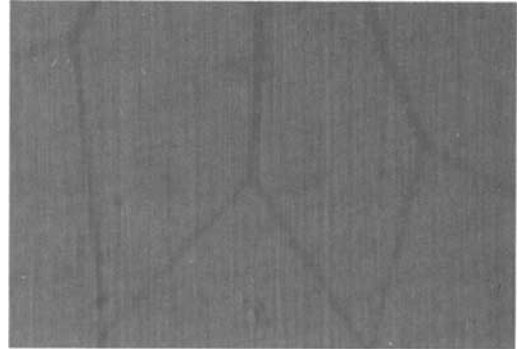


Fig. 33. Synthetic citrine of type 4 produced in Japan; fibrous structures running parallel to the optic axis. 16x.



Fig. 34. Synthetic citrine of type 3 produced in USSR; growth planes parallel to the basal pinacoid. 22x.

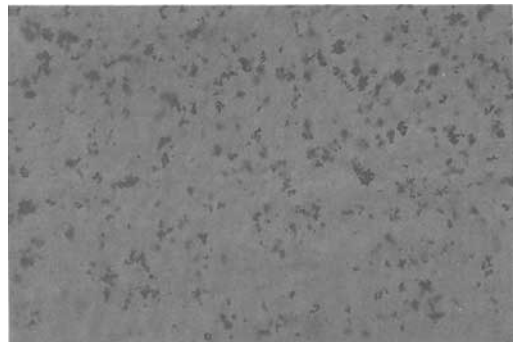


Fig. 35. Synthetic citrine, country of production unknown; breadcrumb inclusions. 35x.

### III.2.6 ARTIFICIALLY-IRRADIATED SYNTHETIC CITRINE (ORIGINALLY COLOURLESS)

Though distinct types of colourless synthetic quartz turn yellow or greenish-yellow by irradiation treatment (cf. Section II.5), no irradiation-treated synthetic citrine of known origin was commercially available. However, two samples submitted for investigation were possibly of this type. The greenish-yellow faceted stones, 63.7 and 88.8 carats in weight, were internally flawless and without any optical twinning or growth structure of diagnostic value. The absorption spectra consisted of an absorption maximum in the ultraviolet, the low energy tail of which extended to the visible. This increasing absorption was superimposed by two absorption bands at about 15,000 and 26,000  $\text{cm}^{-1}$  (667 and 385 nm). All these data are assumed to be found in both, natural and synthetic irradiation-treated yellowish-green citrines. By chemical investigations using energy-dispersive X-ray fluorescence analysis, the presence of distinct amounts of Ge was proved. Though not unequivocal, this result can be assumed as an important hint towards Ge-containing synthetic quartz, which was irradiation-treated (and possibly annealed subsequently) in order to produce a greenish-yellow coloration (cf. Section II.5).

## IV Discussion

All types of natural and synthetic citrine, which are found on the gem market, reveal a number of characteristic features which are useful for diagnostic purposes. Combinations of spectroscopy in the visible and ultraviolet and microscopic investigations are suggested for the determination of citrines of unknown origin.

Natural iron-bearing citrines and prasiolites, which are produced from natural amethyst by heat treatment, are polysynthetically twinned on the Brazil law or display a distinct pattern of parallel striations, the orientation of which corresponds to the respective growth sector which they are confined to. Colour zoning and growth structures are dominant on faces parallel to the major and minor rhombohedron,  $r$  and  $z$ , forming characteristic angles of 94°, 134° and 76°. Absorption spectra consist of one strong maximum in the ultraviolet, the low energy tail of which extends to the visible, and one single weak absorption band at 21,000  $\text{cm}^{-1}$  (476 nm). In prasiolites, an additional band at about 13,500  $\text{cm}^{-1}$  (741 nm) is found.

All types of synthetic iron-containing citrine and green quartz are characterized by the absence of polysynthetic twinning on the Brazil law and the lack of orientated striations. Frequently, dominant growth structures parallel to the seed plane (0001) are observable. In one distinct type of synthetic

iron-containing citrine, channel-like structures parallel to the optic axis are found. In addition, these samples reveal highly distorted interference figures.

The absorption spectra of all types of iron-bearing synthetic citrine consist of an absorption maximum in the ultraviolet, the low energy tail of which causes an increasing absorption from red to violet in the visible range. The spectra of all five types of synthetic iron-bearing citrine and synthetic green quartz, which are grown from  $\text{K}_2\text{CO}_3$ -containing solutions with or without additional oxidizers (e.g.  $\text{LiNO}_2$ ,  $\text{LiNO}_3$ ,  $\text{KMnO}_4$ ) are superimposed by a number of weak absorption bands, which are not found in natural Fe-bearing citrines. Fe- and Co-containing synthetic citrines have a strong absorption band at about 26,800  $\text{cm}^{-1}$  (373 nm). Only the absorption spectra of synthetic citrines, which are grown from KCl-NaCl-bearing solutions are identical with the spectra of heat-treated natural amethyst. Samples of this type of synthetic citrine, which are not yet grown commercially, however, do not display the microscopic characteristics of heat-treated natural amethyst.

Natural non-heat-treated, non-artificially-irradiated citrines, which are also used in the gem trade, are often macroscopically twinned and have growth structures parallel to the major and minor rhombohedron as well as parallel to the hexagonal prism. The absorption spectra consist only of an intense maximum in the ultraviolet, the low energy tail of which extends to the visible. The citrine colour centre is completely destroyed by heat treatment up to 450-550°C and cannot be restored by subsequent gamma or neutron irradiation. At present, this type of citrine has not yet been grown synthetically.

The thermal stability of artificially-irradiated (and in some cases subsequently heat-treated) natural quartz is lower than the thermal stability of artificially untreated citrine. A complete bleaching of this type of greenish-yellow citrine colour centres is observed up to 350°C or, in most samples, even at lower temperatures, and the colour centres are restored by subsequent irradiation. The absorption spectra of this third type of citrine are characterized by an absorption in the ultraviolet, the low energy tail of which extends to the visible. This absorption is superimposed by two bands at about 15,500 and 26,000  $\text{cm}^{-1}$  (645 and 385 nm).

In the literature this type of citrine coloration is also described for synthetic quartz, which was artificially irradiated. However, it was not confirmed experimentally that samples of this type are found in the trade and no information about this is available to the present author. Due to the fact that a large amount of low costing natural quartz, e.g. from Brazil, is converted to a greenish-yellow

coloration by irradiation, no necessity exists to use more expensive synthetic colourless quartz samples for the production of greenish-yellow quartz by irradiation treatment.

In all types of natural or synthetic citrine, the presence of typical solid state inclusions, e.g. Fe-oxides or Fe-hydroxides in heat-treated natural amethyst, mineral inclusions in natural untreated citrine and breadcrumb inclusions in synthetic citrine may give additional information about the nature of the sample. The presence or absence of healing fissures or fingerprint patterns of liquid or two-phase inclusions, however, is of no diagnostic value.

### Acknowledgements

Numerous samples of natural and synthetic citrine, which were investigated in this paper, were kindly supplied by different firms in the gem trade as well as by private collectors. The author is grateful to the following persons or firms including Prof. Dr. H. Bank, Gebr. Bank, R. Dröschel, E. Georg, W. Haag, Gebr. Leyser, Th. Lind, J. Ph. Wild, all of Idar-Oberstein, FRG, Dr. H. A. Hänni of Basel, Switzerland, and G. Bosshart of Zürich, Switzerland. Gem materials which were not available in the gem trade were kindly given by Prof. Dr. G. Lehmann of Münster, FRG, and Prof. Dr. S. Taki, Dr. M. Hosaka of Kofu, Japan. Mrs. M. Vysocanski translated the original literature from Russian language. Financial support was given by grants of the Wirtschaftsministerium des Landes Rheinland-Pfalz, FRG.

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# Proceedings of the Gemmological Association of Great Britain and Association Notices

## OBITUARY

**Mr Syed Vaqar Ahmad, FGA (D.1970)**, London, died on 23 January 1989.

**Mr Cecil A. Bond, FGA (D.1964)**, Ufford, Woodbridge, died on 4 January 1989 after a long illness. Cecil Bond was the third generation of retail jewellers known as A.A. Bond & Sons Ltd with shops at Hoddesdon and Waltham Abbey. He retired in 1983 because of ill health. As well as being a Fellow of the Gemmological Association, he was a craft member of the British Horological Institute and an active member of the National Association of Goldsmiths. He leaves a widow, Joan, two sons, a daughter and seven grandchildren.

**Mr Ronald C. Stevens, FGA (D.1970)**, Howick, Auckland, New Zealand, died on 25 December 1988.

## GIFTS TO THE ASSOCIATION

The Council of the Association is indebted to the following for their gifts:

**Mr B.A. Handana** of Indonesia for a copy of the book *Batu Permata* by Rahasia.

**Mr E. Fontana** and **Mr W.L. Kunsch** of Sao Bernardo do Campo, Brazil, for a copy of their book entitled *Gems of Brazil*.

**Dr Dietmar Schwarz** of Ouro Preto, Brazil, for a copy of his book entitled *Esmeraldas - Inclusões em nas*.

## NEWS OF FELLOWS

On 23 November 1988 Mr Peter Read gave two illustrated talks at a Gala Buffet Supper arranged by the Gemmological Association of South Africa in the Sunnyside Park Hotel, Johannesburg. The subjects of the two talks were the laser Brewster-angle refractometer developed by Peter Read, and his GEMDATA computer program for gem identification. An experimental Brewster-angle refractometer was demonstrated after the first talk, and an IBM PC-compatible computer was made available to show the various features of the GEMDATA program. After his talks, Peter Read presented a

copy of the 2nd edition of his *Dictionary of Gemmology* to the Association's Chairman, Mr Arthur E. Thomas, FGA.

## MEMBERS' MEETINGS

### London

On 29 March 1989 at the Flett Theatre, Geological Museum, Exhibition Road, South Kensington, London SW7, Mr E.A. Jobbins gave an illustrated lecture using the extensive collection of colour transparencies taken by him during his five months stay in Brazil in 1973 and his one month trip in 1987. As well as a description of the gemstones found in Brazil, Alan Jobbins explained some of the reasons for their distribution.

### Midlands Branch

On 20 January 1989 at the Society of Friends, Dr Johnson House, Colmore Circus, Birmingham, Mr C.R. Burch, Senior Lecturer in Gemmology and Geology at the Sunderland Polytechnic, gave an illustrated talk on inclusions in natural and synthetic corundum.

On 17 February 1989 at the Society of Friends Mr John R. Bugg gave a talk entitled 'The law and the gemmologist'.

On 17 March 1989 at the Society of Friends, a talk was given by Mr Peter Read entitled 'The Brewster Angle refractometer'. Mr Read also demonstrated the new GEMDATA computer program.

### North West Branch

On 18 January 1989 at Church House, Hanover Street, Liverpool 1, Mr John Pyke displayed and gave a talk on his collection of rare and interesting gemstones.

On 15 February 1989 Dr J.W. Harris, Senior Lecturer of Strathclyde University, gave a talk on diamonds and diamond mining around the world.

## COUNCIL MEETING

At a meeting of the Council of the Association held on 28 February 1989 at the Royal Automobile Club, 89 Pall Mall, London SW1, the business

transacted included the following: (1) the appointment of Mrs Evelyn Stern, FGA, as an Examiner; (2) the election to membership of the following:

#### **Fellowship**

Bruggeman, Renate F., Almelo, The Netherlands. 1986

Chan, Yuk C., Hong Kong. 1988

Chan Cheng, Rosalia Y.K., Honolulu, HI, USA. 1988

Collins, Steven J.C., Letchworth. 1988

Dabell, Louise C., Nottingham. 1988

Dunn, Wendy S., London. 1987

Franco da Fonseca, Ana L., Sao Paulo, Brazil. 1988

Girod, Daniel F., Amsterdam, The Netherlands. 1988

Haufe, Wilfred, Toronto, Ont., Canada. 1988

Hergel, James E., Toronto, Ont., Canada. 1988

Kuwahara, Chizu, Yokosuka City, Japan. 1988

Lewis, David T.K., Nottingham. 1968

Lewis, Rachel, Stockport. 1988

McKearney, Michael C., London. 1984

Maharaj, Rajendra K., Toronto, Ont., Canada. 1988

Muff-Somma, Walter C., Muri, Switzerland. 1988

Nakamura, Midori, Ichikawa City, Japan. 1983

Ng, Siu K., Hong Kong. 1987

Potts, James D., Telford. 1988

Sumar, Nizarali P., Nairobi, Kenya. 1988

#### **Ordinary Membership**

Aoki, Kazuo, Fujisawa City, Japan.

Candela, Conchita, Bari, Italy.

Chang, Kuei Y., Taipei, Taiwan.

Chowdhary, Kawaljit, Wembley.

Christofi, Christiana, Limassol, Cyprus.

Costa, L.R., London.

Desor, Manmohan, Leicester.

Fernando, Joseph S., Colombo, Sri Lanka.

Fogarty, Jolyan L., Jackson, Wyo., USA.

Girardi, Josephine C., Hong Kong.

Grigoriadis, Giorgios D., Athens, Greece.

Hainovitz, Jonathan, Jerusalem, Israel.

Hare, Rebecca, London.

Hayakawa, Taketoshi, Chigasaki City, Japan.

Hill, Linda S., Jakarta, Indonesia.

Holt, Dorothy O., London.

Hughes, David J., Cardiff.

Itoyama, Mariko, Nagasaki City, Japan.

Jhaveri, Devang, London.

Johns, Alan T., Perranporth.

Kammerling, Robert C., Santa Monica, Calif., USA.

Kapur, Sharad, New Delhi, India.

Knudson, Eric, London.

Komai, Rie, Kawaguchi City, Japan.

Kuinose, Masafumi, Wakayama City, Japan.

Leadbetter, Bibette, Skelmersdale.

Lilly, Gregory R., Taranaki, New Zealand.

Lockie, Debra L., Auckland, New Zealand.

McDowell, Barry M., Wellington, New Zealand.

Matoba, Hiroshi, Nara City, Japan.

Murata, Michiko, Tokyo, Japan.

Naitoh, Yukihiro, Shimizu City, Japan.

Nakane, Tomoko, Tokyo, Japan.

Nercessian, Armine, New York, NY, USA.

Noyori, Kazuhiko, Fukuoka City, Japan.

O'Reilly, Stan J., Galway, Eire.

Ozaki, Keiko, Kawanishi City, Japan.

Palmer, Guen, Ditchling.

Pimantip, Nin, London.

Pine, Irving C., Delray Beach, Fla., USA.

Ravindran, Mahaluxmy, Bangkok, Thailand.

Richards, Keith, Epsom.

Safwat, Hanafi, London.

Sarin, R., Arlington, Va., USA.

Sarmiento, Luis, Madrid, Spain.

Satoh, Mariko, Yokohama City, Japan.

Seymour, Nicola, Hove.

Shimoda, Tetsuhiro, Fukuoka City, Japan.

Stanley, Sylvia, Clearwater Bay, Hong Kong.

Tachibana, Hirogi, Osaka, Japan.

Tanaka, Norihiro, Tokyo, Japan.

Tanzawa, Megumi, Nakacoma-Gun, Japan.

Tay, Thye S., Singapore.

Themelis, Ted., Clearwater, Fla., USA.

Wedekind, Kenneth J., Wakefield.

Wilson, Mathew D., Broadstairs.

Woods, Janina, Warwick.

Yasuda, Jun, Tokyo, Japan.

Yutani, Atsuko, Osaka, Japan.

#### **GEMMOLOGICAL ASSOCIATION OF ISRAEL**

The formation of this new organization has just been announced. The address is 1 Japotinsky Street, Ramat-Gan, 52520 Israel.

#### **FORTHCOMING MEETINGS**

The 1989 Annual General Meeting of the Association is to be held on 12 July 1989 at the Flett Theatre, Geological Museum, Exhibition Road, South Kensington, London SW7. This will be followed by a Gemmological Forum similar to those held in previous years that have proved so successful.

## Letters to the Editor

*From Rudolf Voll*

Dear Sir,

As a gem tester, Mr Kenneth Scarratt fails to divulge the cause of double-nucleated cultured pearls from the *Pinctada martensii* oyster (Notes from the Laboratory – 13, *Journal of Gemmology*, 21, 5, 294); let me advise readers that there was no attempt whatsoever to create 'twins', but that, decades ago, the technique that prevented the undesired joining of large nuclei inside the gonad was insufficiently developed.

But today it is well-nigh impossible to find one twin among many 'kan' (kame) of cultured pearls. Kan is an ancient Chinese weight unit of 3.75kg or 1000 momme, which was outlawed for general use in Japan in 1960 and is, by special licence, only permitted to be used by the pearl industry.

'Twins' still occur today in the 2-3 mm nuclei but they are usually cut apart and used as pearls for 'trimming' or 'paving' purposes, having a practical near-half-pearl shape after separating.

The 'hollow' cultured pearls are a phenomenon which happened mainly in the freshwater mollusc *Hyriopsis schlegeli* in Lake Biwa. Because of the pearl-'unlike' weight and especially the usually 'dried prune' surface, cultivation has been discontinued.

My daughter Aloha, a 1981 winner of the Anderson Medal and student of Mrs Anne Paul's class, lives now in Bangkok, but I read the *Journal* which still arrives in Hong Kong, and I believe that you do not object to my co-operation on her behalf, believing to be competent enough as the only active non-Japanese member at the monthly Tokyo pearl auctions. I have specialized in cultured pearls since 1938 in Tokyo, but spend two weeks of every month in Hong Kong.

Yours etc.,  
Rudolf Voll

14 February 1989  
C-6, 15 Fl., Hankow Centre, 1 Middle Road,  
Kowloon, Hong Kong.

*From Kenneth Scarratt, FGA*

Dear Sir,

RE: LETTER FROM MR RUDOLF VOLL

I have often written items into 'Notes from the Laboratory' in the hope that someone with a close knowledge of that particular area of our subject will write to the Editor with additional information. This, however, and somewhat disappointingly, very seldom occurs. I was therefore very pleased to read Mr Voll's comments on the double-nucleated cultured pearls.

Mr Voll is correct in assuming that his contribution would be well received; one only wishes that there were more people in the gemstone industry who would put pen to paper and let us read of their experiences within these pages.

Yours etc.,  
Kenneth Scarratt

24 February 1989  
The Gem Testing Laboratory of Great Britain,  
27 Greville Street, London EC1N 8SU.

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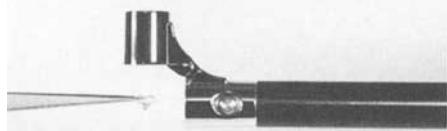
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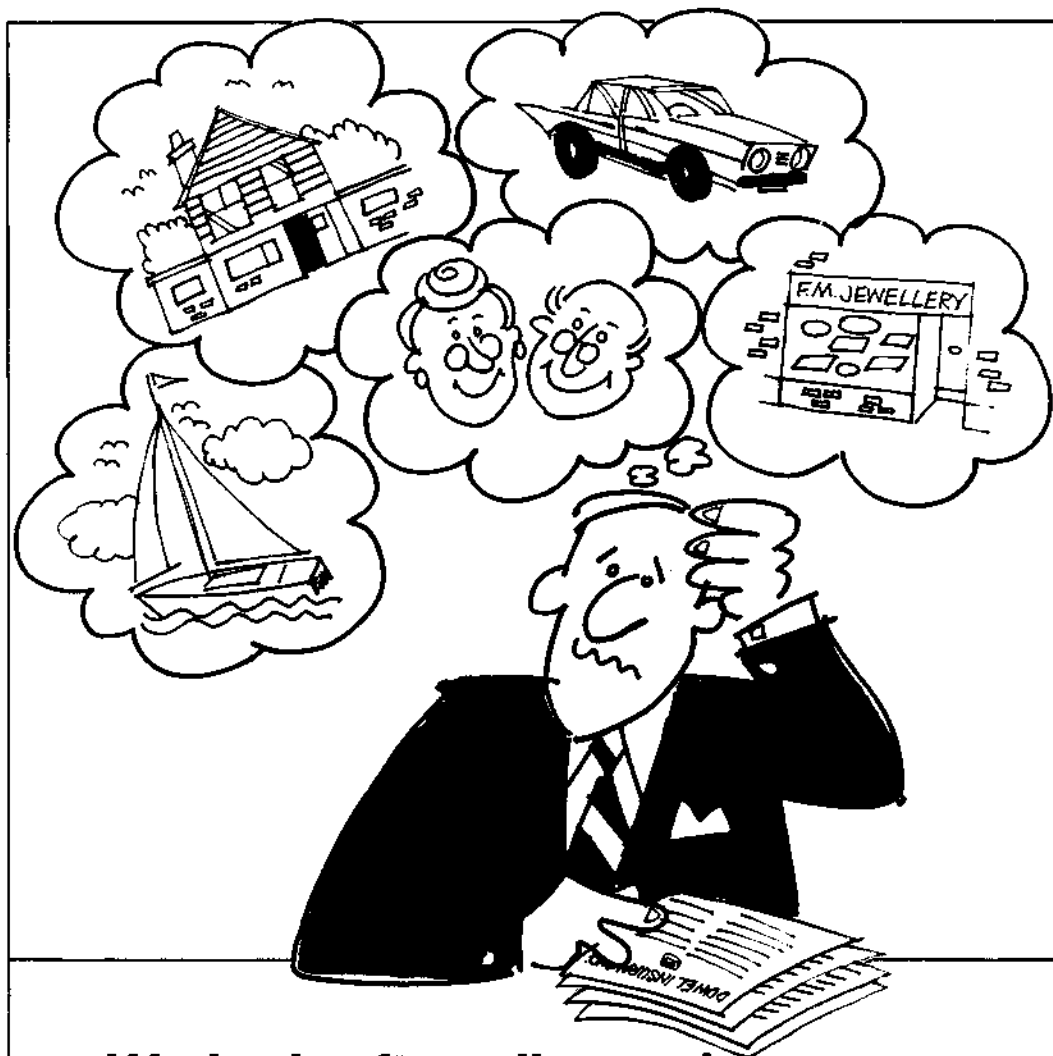
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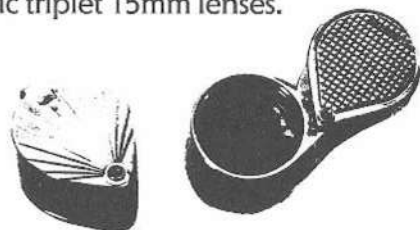
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# GEMMOLOGICAL ASSOCIATION OF GREAT BRITAIN

The Arms and Crest of the Association, conferred by a grant of Arms made by the Kings of Arms under royal authority. The cross is a variation of that in the Arms of the National Association of Goldsmiths of Great Britain and Ireland. In the middle is a gold jewelled book representing the study of gemmology and the examination work of the Association. Above it is a top plan of a rose-cut diamond inside a ring, suggesting the scrutiny of gems by magnification under a lens. The lozenges represent uncut



octahedra and the gem-set ring indicates the use of gems in ornamentation. The lynx of the crest at the top was credited, in ancient times, with being able to see through opaque substances. He represents the lapidary and the student scrutinizing every aspect of gemmology. In the paws is one of the oldest heraldic emblems, an escarbuncle, to represent a very brilliant jewel, usually a ruby. The radiating arms suggest light diffused by the escarbuncle and their tips are shown as jewels representing the colours of the spectrum.

## Historical Note

The Gemmological Association of Great Britain was originally founded in 1908 as the Education Committee of the National Association of Goldsmiths and reconstituted in 1931 as the Gemmological Association. Its name was extended to Gemmological Association of Great Britain in 1938, and finally in 1944 it was incorporated in that name under the Companies Acts as a company limited by guarantee (registered in England, no. 433063).

Affiliated Associations are the Gemmological Association of Australia, the

Canadian Gemmological Association, the Gem and Mineral Society of Zimbabwe, the Gemmological Association of Hong Kong, the Gemmological Association of South Africa and the Singapore Gemologist Society.

The *Journal of Gemmology* was first published by the Association in 1947. It is a quarterly, published in January, April, July, and October each year, and is issued free to Fellows and Members of the Association. Opinions expressed by authors are not necessarily endorsed by the Association.

## Notes for Contributors

The Editors are glad to consider original articles shedding new light on subjects of gemmological interest for publication in the *Journal*. Articles are not normally accepted which have already been published elsewhere in English, and an article is accepted only on the understanding that (1) full information as to any previous publication (whether in English or another language) has been given, (2) it is not under consideration for publication elsewhere and (3) it will not be published elsewhere without the consent of the Editors.

Papers should be submitted in duplicate on A4 paper. They should be typed with double line spacing with ample margins of at least 25mm all round. The title should be as brief as

is consistent with clear indication of the content of the paper. It should be followed by the names (with initials) of the authors and by their addresses. A short abstract of 50–100 words should be provided. Papers may be of any length, but long papers of more than 10 000 words (unless capable of division into parts or of exceptional importance) are unlikely to be acceptable, whereas a short paper of 400–500 words may achieve early publication.

Twenty five copies of individual papers are provided on request free of charge; additional copies may be supplied, but they must be ordered at first proof stage or earlier.

The Journal of  
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