Oil of Iron

The Procedure for making the
O I L o f I R O N
In accordance with the Precepts of Alchemy

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Since it is difficult, in fact almost impossible to obtain pure metallic Iron, it is preferable to commence the procedure by using C. P. (Chemically Pure) Ferrous Sulphate Crystals (Fe SO_4 $7H_2O$), the Vitriol Salts of this metal. (Use Baker's Analyzed C. P. Ferrous Sulphate obtainable from any Chemical Reagent Supply House). These Salts have already been purified by several successive washings and re-crystallization.

Using a Number 3 Porcelain Coors Crucible (glazed inside and outside) level full with the Ferrous Sulphate Crystals (five) calcine the Salts in the electric muffle furnace. Place the crucible containing the SALTS in the *cold* furnace and then raise the temperature of the furnace to 1750 C. Continue the calcination until all fuming ceases and the iron is brought completely to a state of oxidation. This usually takes about two hours. A good supply of air should have access to the furnace. Remove the crucible and allow to cool to nearly room temperature. Transfer the oxide sponge to a glass mortar and triturate with a glass pestle to a fine powder.

Weigh out 14 grams (approximately one half ounce avoirdupois) of the powdered iron oxide and transfer to a Pyrex Beaker of about 800 ml. capacity and digest on the hot plate with 120 ml. of 6 normal Hydrochloric Acid until all the Iron oxide is in solution, adding more acid if necessary to accomplish solution. Add 20 ml. of C. P. Nitric Acid (Specific Gravity 1.42) and boil for five minutes to bring all the Iron to the

Ferric State. (Note: It cannot be assumed that the Iron Oxide product of the Calcination is in a uniform condition of oxidation). Cool for a few minutes and filter the solution through a fast filter paper with distilled water and allowing the washings to drain through this filter paper into the Beaker containing the main portion of the filtrate. Discard the filter paper and any residue remaining in it.

Make up the volume of the Solution in the Pyrex Beaker to 1,000 ml. Neutralize the Solution with 6 normal Sodium Hydroxide Solution (To make: Add to 250 gm. of Na OH, purified by alcohol, enough distilled water to make the volume 1000 ml.) thus precipitate the Iron as Ferric Hydroxide, then add a slight excess of the 6 normal Sodium Hydroxide Solution. The solution containing the Iron should be stirred vigorously with a glass stirring rod during the preceeding operation. Still stirring vigorously, add 6 normal Hydrochloric Acid cautiously, using a dropper until the Solution is neutral or to the barest trace of acid reaction to litmus paper.

Make up the volume of the contents of the Pyrex Beaker to about 1,900 ml. with distilled water. Stir well and then allow the Ferric Hydroxide precipitate to settle for about an hour. Decant the clear Supernatant Solution. Again bring the volume of the contents of the Beaker to about 1,900 ml., stirring well as the distilled water is added, and allow to settle. Repeat this washing by decantation three or four times. (See Note 1.)

Heat the Solution, containing the washed Ferric Hydroxide precipitate, after the last decantation and without further addition of distilled water, to near boiling and filter through a *hardened* filter paper (Whatman No. 52 or No. 54) using vacum with a Buchner Vacum Filter Funnel and a Vacum Filter Flask and finally washing any Ferric Hydroxide precipitate adhering to the wall of the Beaker into the Buchner Filter Funnel by means of a fine stream of distilled water from the wash bottle.

After the filtrate has been sucked through the Ferric Hydroxide cake into the Filter Flask, wash the Ferric Hydroxide cake in the Buchner with three consecutive 100 ml. portions of hot distilled water, taking great care to seal up with the end of a glass stirring rod, all cracks which may develop in the Ferric Hydroxide cake, *as they appear*.

Maintain the vacum in the Filter Flask after the last washing until the Ferric Hydroxide cake is free of excess water and is firm and solid. Remove the cake and filter paper by inserting the blade of a small spatula between the cake and the porcelain wall of the Buchner and running it around the periphery of the cake. Detach the Buchner Funnel from the Vacum Filter Flask and invert the Buchner over a porcelain dish of sufficient diameter to receive the cake and filter paper and then jar these from the Buchner into the porcelain dish. Peal off and remove the hardened filter paper (this may be washed and used again) from the cake. Remove carefully any Ferric Hydroxide still adhering to the Buchner Funnel and add it to the main portion of the cake in the porcelain dish. Discard the filtrate and wash waters from the vacum filtration.

Place the porcelain dish, containing the Ferric Hydroxide cake in the drying oven and dry at 225 F. for about three hours. At intervals, as the drying proceeds, break up the cake in the dish with spatula or knife blade into smaller and smaller pieces until, finally, it has been chopped up into small pieces about the size of a match head. This will ensure proper drying. When the precipitate is finally quite dry, remove from the drying oven and allow to cool. Then transfer the dried material to a glass mortar and triturate it to a fine powder with the glass pestle.

Transfer the fine powder to a Pyrex Beaker of 400 ml. capacity. Add sufficient fuming C. P. Hydrochloric Acid (Specific gravity 1.18), to dissolve the powdered iron oxide (approximately 50 ml. should be sufficient). Heat on the hot plate until all the solid material is in Solution. Pour the Hydrochloric Acid Solution, containing the Iron as Ferric Chloride, into a glazed porcelain evaporating dish (deep type) large enough to contain it and yet small enough to provide as small a surface area of the Solution as possible in the course of the evaporation to follow. (Use a Coors Slayed Porcelain Dish 120 mm. Diameter. Height 50mm.) *No water* may be used to wash any of the Ferric Chloride Solution adhering to the Beaker into the dish. (See Note 4.) Evaporate the acid Ferric Chloride Solution slowly on the sand bath, care being taken to see that the temperature of the sand bath is not sufficiently high to cause burning or undue drying. *Do not* use the water bath or the steam bath for this evaporation.

As the evaporation proceeds work down into the body of the thickening solution any Ferric Chloride gel which may form and adhere to the evaporating dish around and above the solution level. Use a rubber policeman on a small stirring rod to accomplish this. The solution will finally thicken to a gel, and the evaporation *must not* be carried out to the point at which the gel commences to dry. The gel must remain moist with a slight excess of Hydrochloric Acid.

As soon as bulk of the acid has evaporated and the mass consists of a gel still moist with Hydrochloric Acid, remove from the sand bath and, after thoroughly cooling the dish and contents on cold water and then carefully drying the bottom of the dish with a cloth, making sure that no trace of water is left around the lip of the dish nor has reached the interiour of it, transfer the Ferric Chloride Gel to a wide mouthed glass bottle of about 250 ml. capacity, and having a ground glass stopper. Accomplish this by working the gel over the decanting lip of the dish with the rubber policeman. Use *no* water. Pour a few ml. of ether (See Note 2.) into the dish after making sure that no bare flame, nor electric element or sparking equipment is near, and work any gel residue remaining in the dish out with the ether into the glass bottle by means of the rubber policeman on the stirring rod. Ignore any dried rim of Ferric Chloride there may be in the evaporating dish.

Add, *at once*, a 100 ml. portion of ether (have this ready at hand) to the Ferric Chloride gel in the glass bottle and insert the glass stopper. (It is well to have the glass bottle standing in a dish of cold water the level of which is not sufficiently high to tend to float the bottle). Shake the bottle containing the gel and the ether vigorously for a few minutes, cooling the lower half of the bottle frequently under the cold water faucet. Allow to stand a minute, then remove the stopper, first being very careful to dry off thoroughly any drops of water on the bottle and especially around the rim of the stopper orfice, and decant the now brown ethereal solution into an Erlenmeyer Flask of 300 ml. capacity and stopper the Flask with a rubber stopper.

Add another fresh 100 ml. portion of either to the contents of the glass shaking bottle and repeat the ether extraction by shaking once more for a few minutes, cooling as before under the cold water faucet.

Dry the bottle and allow it to remain stoppered. Both the glass bottle and the Erlenmeyer Flask containing the first portion of ether should be kept in a cool place until required or, if this is not available, stand them in shallow running cold water keeping the stoppers dry and protected from water.

Set up a distillation train consisting of a Pyrex distillation flask or retort of 300 ml. capacity, the stopper neck of which is fitted with a bored rubber stopper through which is inserted a 3 inch immersion mercury Thermometer reading from 0 C. to 200 C., so that when the rubber stopper and thermometer are placed in position in the distillation flask or retort the thermometer bulb clears the bottom of the distillation flask or retort by three or four millimeters--a condensor, water jacketed so that cold water may be passed continually through the jacket, filled to the delivery arm of the distillation flask or retort on one end, and to a receiver of sufficient capacity, (about 500 ml.) at the other end.

Having removed the Thermometer and stopper from the distillation flask or retort, arrange a 60 degree Pyrex Filter Funnel (having a diameter at the top of about 2½ inches) in a ring support or clamp on the retort stand and with the stem lowered into the distillation flask or retort so that the end of the stem is well below the level of the opening into the delivery arm of the flask or retort. Place a fast filter paper in the funnel and fix in place by moistening with a little fresh Ether. Using first the Ether extract contained in the Erlenmeyer Flask, filter this through the filter paper in the funnel. Then replace the filter paper in the funnel with a fresh filter paper and introduce into the funnel the Ether extract contained in the glass bottle together with the gross body of the Iron, decanting and filtering first the bulk of the clear brown Ether Solution, with the last remaining 20 ml. or so, swirling the gross body of the Iron into suspension with the remaining Ether Solution, decant the whole into the filter funnel. Allow the ethereal solution to filter through into the distillation flask or retort, then wash out the glass bottle with about 10 ml. of fresh Ether and pour this over the residual gross body of the Iron in the filter paper. Allow these washings to pass through into the distillation flask or retort, then

remove the filter funnel and discard the filter paper and contents.

Moisten the rubber stopper fitted on the Thermometer, with a drop or two of absolute alcohol (Ethyl) and insert the rubber stopper and Thermometer in the distillation flask or retort. (The alcohol facilitates the proper placement of the rubber stopper). As a precaution arrange a Burette clamp on the stand so that one claw of the clamp bears down, when the clamp arm is screwed in position on the rod of the stand, on the top of the rubber stopper carrying the thermometer, and just to one side of the Thermometer.

Using a Precision Electric Heater equipped with Rheostat, *not a bare flame*, and arranged so that the bottom of the distillation flask or retort is about 2½ inches from the heater element, gradually raise the temperature of the contents of the distillation flask or retort until the Ether is distilling at about 40 C., as shown on the thermometer Scale.

Continue the distillation until the volume of the contents of the flask or retort is about 125 ml. Ignore for the time being any suspended solid matter or cloudiness which may have appeared in the solution.

Until the first small portion, about 3 ml., of distillate appears in the Receiver, the Receiver should be left loose at the nexk where it fits on to the discharge end of the Condenser. When about 3 ml. have distilled over, the temperature in the distillation flask or retort will have risen to more than 35 C. The Receiver may then be fitted up tightly to the end of the condenser; but until this is done and while the neck of the Receiver is loose, a wet strip of cloth should be wound around the open joint to prevent the escape of any vapour. When the neck of the receiver is closed up with the discharge end of the condenser this cloth may be dispensed with.

Then the contents of the distillation flask or retort approximate 125 ml. in volume, turn off the electric current to the Heater and by loosening the Heater clamp drop the Heater on the stand away from the bottom of the distillation flask or retort and place a sand bath or some such insulation over the element of the Heater to prevent the heat from rising.

Cool the bottom of the distillation flask or retort with a wet cloth. Remove the rubber stopper carrying the Thermometer, and add to the contents of the flask or retort about 100 ml. of absolute Ethyl Alcohol (See Note 3.) Replace the stopper and Thermometer.

Raise the Precision Electric Heater to its original position on the stand and continue the distillation at 45 C. to 50 C., until 40 ml. or 50 ml. have distilled over, and observe the same precautions as before with respect to the Receiver Flask.

At this point any portion of the gross body of the iron which may have passed into solution in the excess of Hydrochloric Acid present with the Ferric Chloride Gel when the Ether Extraction was made, will have been thrown down out of solution (See Note 5), and the excess of Hydrochloric Acid will have combined with the Ether in the first distillation and alcohol in the subsequent distillation to pass over as Ethyl Oxychloride and Ethyl Chloride respectively.

Again disconnect and drop the Electric Heater on the stand. Cool the distillation flask or retort as before and remove from the train. (Note. Whenever the distillation train is to be broken at any point after heat has been applied to the distillation flask or retort, great care must be exercised for the reason that if pressure within the apparatus has not attained equilibrium with the outside pressure, violent restoration of equilibrium will take place if the difference in pressure is excessive, and this may completely ruin the operation. Always break the train or remove the rubber stopper from the distillation flask or retort only after sufficient cooling has taken place, and then very carefully and by degrees, thus allowing gradual re-adjustment of pressure.)

Decant, carefully, the contents of the distillation flask or retort into a Pyrex Beaker of 400 ml. capacity. Wash out the distillation flask or retort thoroughly with a little absolute Ethyl Alcohol, adding these washing to the main portion in the Beaker.

Replace the distillation flask or retort in position in the train and, again using the Pyrex Filter Funnel fitted with a fast filter paper, filter the Solution contained in the Beaker back into the distillation flask or retort, washing the filter paper and contents, after the main

body of the solution has passed through the filter, with 5 ml. of absolute Ethyl Alcohol, but first having washed out the Beaker with a like portion of alcohol, and having passed these washings through the filter. When the last washings of the filter paper and contents have passed throuth into the distillation flask, remove the filter funnel and discard the filter paper and residual contents.

Replace the rubber stopper and Thermometer in the distillation flask or retort, raise the electric heater, so that the heater element is, this time, about one inch from the bottom of the distillation flask or retort. Raise the temperature of the contents of the flask or retort gradually (observing the same precautions as before with respect to the Receiver Flask) until a temperature of about 85 C. is reached, and continue the distillation until the volume of the contents of the distillation flask or retort is about 50 ml.

Disconnect the Electric Heater once more and drop it away from the bottom of the flask or retort. Cover the element with a sand bath as before, to prevent the heat from rising. Cool the flask or retort to about 40 C. Remove the rubber stopper and Thermometer carefully to allow readjustment of pressure. Add 75 ml. of absolute Ethyl Alcohol. Re-insert stopper and Thermometer. Raise the Electric Heater into position, so that one inch separates the bottom of the flask or retort anq the heater element, and continue the distillation, observing the same precautions as before, with respect to the Receiver Flask.

Allow the temperature of the contents of the distillation flask or retort to mount a little more rapidly this time until a temperature of 90 C. is reached, and maintain this temperature until the volume of the contents of the flask or retort is about 65 ml. Then allow the temperature to mount gradually until there is a sudden, short and moderately violent ebullition of the Solution in the flask or retort. Allow the temperature to continue to mount until the contents of the flask or retort appear wine red by transmitted light. The volume of the Solution at this point should be about 35 ml. The fluid in the flask or retort is now the true Oil of Iron, of which five, six or seven drops in half a tumbler full of water may be taken two or three times a day before meals with great advantage and without any fear of ill effects, for the general health

and especially in cases of anaemia and other ailments as stated in Frater Archibald Cockran's great work entitled "Alchemy Rediscovered and Restored". The writer wishes here to express his deep gratitude and thanks to Frater Archibald Cockran for the very great amount of help he has received through his study of this book, and to Imperator Ralph M. Lewis and others who made the book available to and brought it to the attention of the membership of the A.M.O.R.C.

Disconnect the Electric Heater and drop it away from the bottom of the distillation flask or retort. Cool the flask or retort and remove it from the train observing the usual precautions with respect to pressure readjustment. Decant the Oil of Iron into a dropper bottle made preferably of brown glass.

If, at the conclusion of the decanting operation a thin red-brown film of sediment is found adhering to the bottom of the flask or retort, pay no attention to it. It is not caused by the presence of any of the gross body of the Iron. It is due to local overheating of the oil and is caused by there having been too high a heat on the electric heater element. This, however, should be avoided. After decanting the Oil of Iron from the flask or retort, and washing the flask or retort out with a little distilled water (not to be added to the Oil) without disturbing this film, and then, upon the addition of a further portion of distilled water, about 25 ml., rubbing off the film with a rubber policeman and agitating it with the water, it will be found that it dissolves in the water to form a bright red solution. Warming the Solution helps to accomplish this.