Voacanga Extraction Manual

Phase 4: Production and Purification of Ibogaine

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As with the other phases of the manual, it is important to keep a record of the yields in each part of this phase, as well as note any anomalies in the appearance or analysis of the intermediates or product both for the sake of quality control and to anticipate problems with the quality of ingredients or with implementation of the procedure. These records will also facilitate improvement of the procedure and associated quality control systems, not to mention financial accounting. A photograph of each intermediate or product collected for each batch would also allow any discrepancies noticed during quality control later in the process to be better investigated. If the quality of any intermediate seems questionable, a sample of 20 milligrams should be sufficient to allow later analysis by HPLC. It is strongly recommended that each completed data sheet be entered into a central database or spreadsheet for ongoing analysis before being archived. The expected yield of each intermediate based on historical records should be printed on the data collection sheets to allow any variation from it to be discovered immediately and investigated. These historical yields should be updated for accuracy and whenever there is a significant change in the materials or procedure.

Overview of Part A: Conversion of Voacangine into Ibogaine

Of the entire process of preparing ibogaine HCl from *Voacanga* bark, the first part of this phase is the only one that involves a chemical reaction – the decarboxylation of voacangine to produce the deprotonated carboxylic acid derivative of ibogaine, followed by further conversion to ibogaine by loss of carbon dioxide:

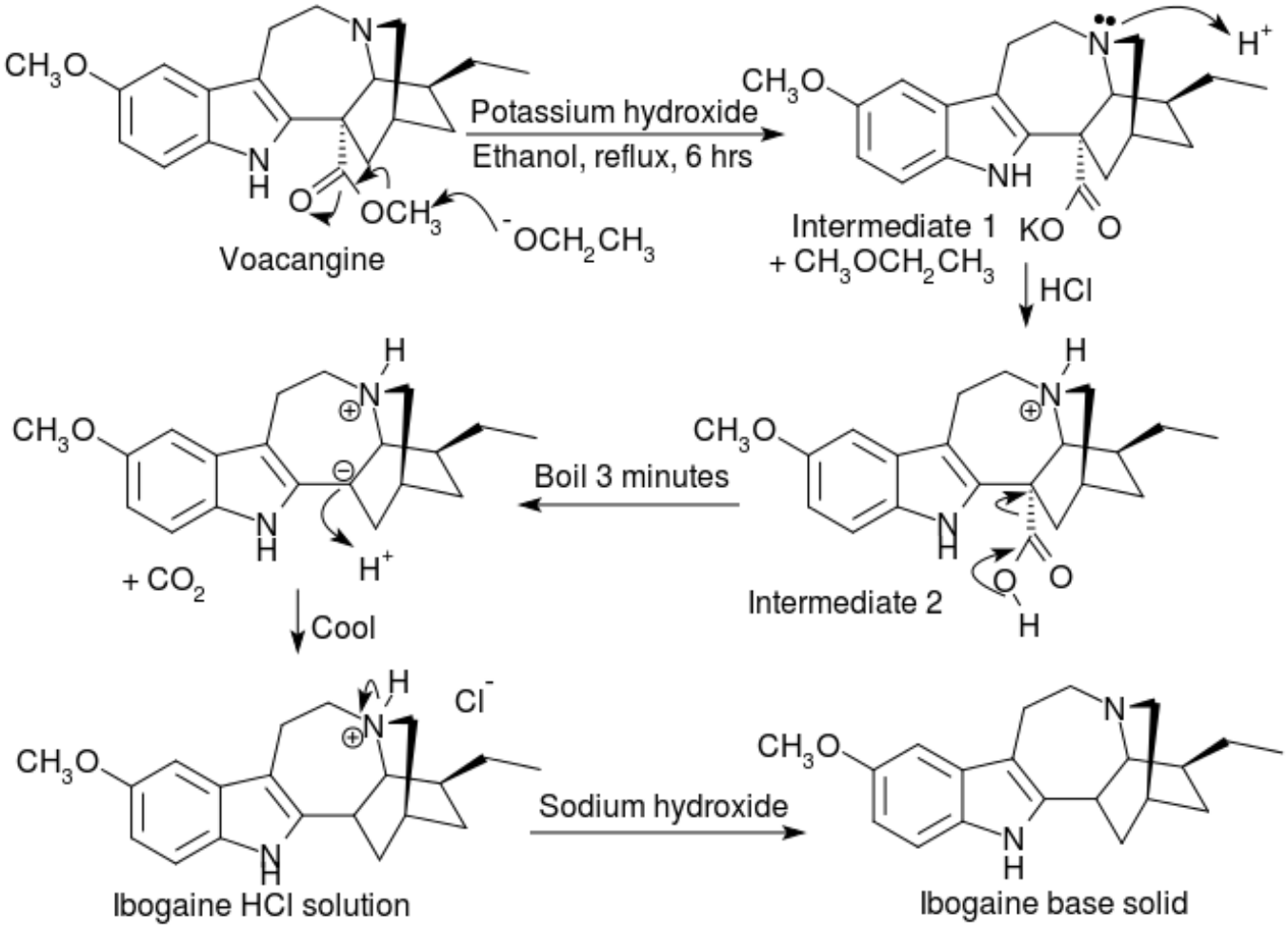

Figure 1: Conversion of Voacangine into Ibogaine

So the reaction happens in two parts. In the first part, once the voacangine dissolves in the boiling ethanol, it reacts with potassium hydroxide, losing a methyl group and going from being a methyl ester to the potassium salt of a carboxylic acid (Intermediate 1). This is the white paste left over after evaporation of the methanol, but it is neither voacangine nor ibogaine. Just as a hydrochloride salt dissolves in water, the potassium salt of this carboxylic acid derivative dissolves even better, which is why it is possible to dissolve the white paste in only half a liter of water when several liters would be needed to dissolve this much hydrochloride. Note that if any voacangine or ibogaine were present at this point they would not be expected to dissolve in the strongly basic solution, as that is how they are separated from water throughout the rest of the manual. The methyl group of the methyl ester in voacangine combines with the ethanol to form methyl ethyl ether ($CH_3OCH_2CH_3$) which later evaporates. Hot hydroxide solution slowly dissolves glass, so eventually the flask used for this reflux will become thin on the bottom and need to be replaced. Also, the ground glass joint between the flask and the reflux condenser should be wrapped with a single layer of thin teflon tape to prevent spattering solution from causing it to fuse together. A nitrogen atmosphere to reduce darkening of the reaction mixture in Step 2 is suggested because boiling ibogaine hydrochloride in ethanol was observed to darken unless oxygen is excluded. Cooling the solution of ibogaine hydrochloride in Part A Step 9 is intended to reduce the time it can spend darkening.

In the second part of the reaction, the basic solution of intermediate carboxylate salt is poured into excess boiling hydrochloric acid. Immediately the carboxylate salt is converted into the corresponding carboxylic acid (Intermediate 2), and the base form becomes the hydrochloride. Over the next three minutes, the unstable carboxylic acid decomposes due to the high temperature of the boiling solution, losing carbon dioxide and forming ibogaine hydrochloride in solution. The reason such a large volume of acid solution is used, as mentioned, is to ensure that there will be sufficient volume to keep the ibogaine hydrochloride in solution long enough to cool it and precipitate the base. Unfortunately the carbon dioxide leads to more vigorous boiling when it is released, increasing the risk of a full flask overflowing. The three minutes of boiling were verified to be sufficient by reacidifying and reheating the waste from this process an additional hour with no additional ibogaine produced. But it is very important to make sure the acid boils at least three minutes because any carboxylic acid intermediate which does not have time to decompose will remain dissolved when the ibogaine is precipitated and filtered and will be discarded in the filtrate waste. It is also important that there be more than enough acid to neutralize all the potassium hydroxide, and the pH of the boiling solution should be verified to be below 2 if there is any doubt.

As with the other phases of this manual, the scale of the process should be adjusted as needed, especially to match the amounts of materials available. Note that the maximum possible product which could be produced in Part A at the scale presented is less than the mass of starting material proposed for Part B because each part has been roughly optimized for scale to make the best use of the equipment available on the date this manual was first written. The chemist should not feel constrained to keep the proposed scales and should calculate the relative amounts of ingredients, volumes of solvents and washes, expected yields of product and glassware sizes based on the most convenient scale at the time. However, reaction and other times should not be modified on the basis of scale.

When no chemical reaction takes place, such as in a recrystallization, the yield is simply 100% times the weight of the product, divided by the weight of the limiting ingredient. In the case of this reaction, the maximum possible (theoretical) yield of ibogaine from voacangine depends also on the proportion of molecular weight lost in the conversion. Voacangine ($C_{22}H_{28}N_2O_3$) has a molecular weight of 368.478 while ibogaine ($C_{20}H_{26}N_2O$) has a molecular weight of 310.441, so the theoretical yield of ibogaine from 50.00 grams of voacangine is:

$$50\ grams\ voacangine * \left(\frac{1\ mole\ voacangine}{368.478\ grams\ voacangine}\right)\left(\frac{1\ mole\ ibogaine}{1\ mole\ voacangine}\right)\left(\frac{310.441\ grams\ ibogiane}{1\ mole\ ibogaine}\right) * 100\%$$
$$= 42.12\ grams\ ibogaine$$

If the actual amount of crude ibogaine obtained were 39.87 grams, then the actual yield would be:

$$\left(\frac{39.87\ grams\ ibogaine}{42.12\ grams\ ibogaine\ possible}\right) * 100\% = 94.66\%$$

Part A Procedure: Conversion of Voacangine into Ibogaine

1. Place a stir bar and 50 grams of first crop voacangine (Illustration 1) into a 500 mL flat bottom round flask (Illustration 2) and add 300 mL of ethanol (Illustration 3) and 73.9 grams of potassium hydroxide (Illustration 4, Illustration 5, Illustration 6). The ethanol can be used to rinse the flask used for recrystallization in Phase III (Illustration 3).

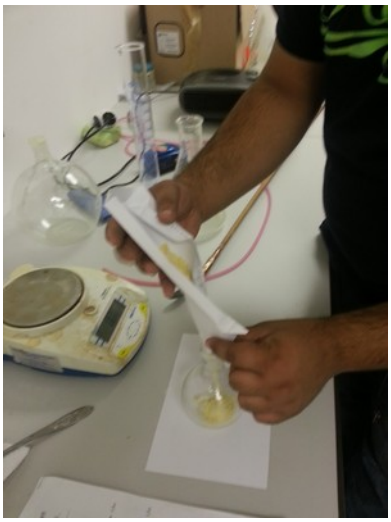

Illustration 1: Pour the weighed voacangine into the flask

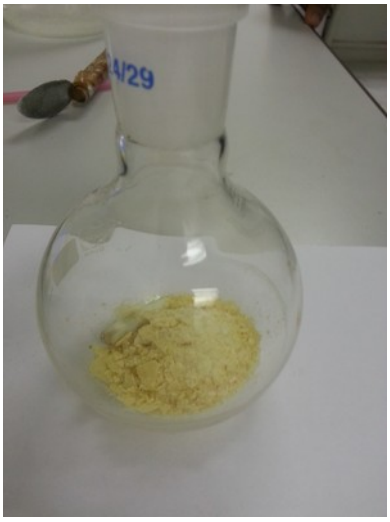

Illustration 2: The voacangine in the flask

Illustration 3: The flask from recrystallizing the voacangine can be rinsed with the ethanol

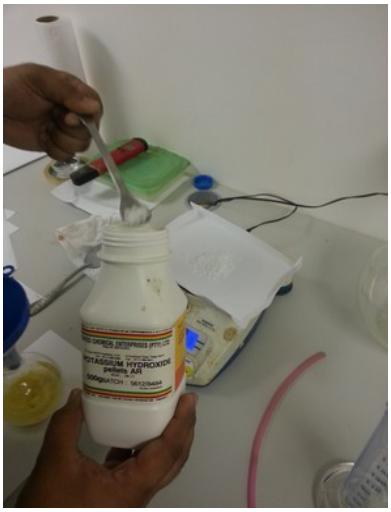

Illustration 4: Weigh out the potassium hydroxide.

Illustration 5: When finished weighing, pour the potassium hydroxide into the flask.

Illustration 6: Voacangine with ingredients, ready to react.

2. Attach the flask to a water-cooled reflux condenser (Illustration 7) with the joint protected by teflon tape (Illustration 8). If a source of nitrogen is available, attach it to the top of the reflux condenser to reduce the darkening of the ibogaine (not shown). Once the equipment is ready, begin heating and stirring (Illustration 9).

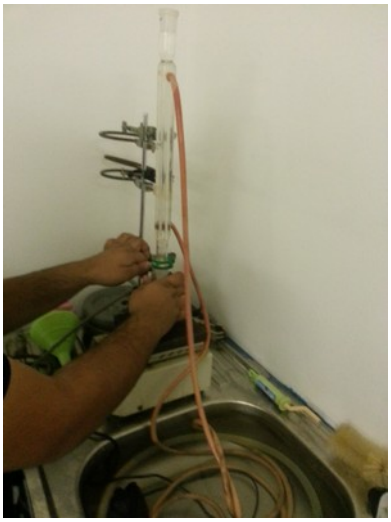

Illustration 7: Attach a reflux condenser to the flask.

Illustration 8: Seal the flask to the condenser with teflon tape and a Keck clamp,

Illustration 9: Voacangine ready to react.

3. Heat the stirring flask on high until it reaches boiling (Illustration 10). Record the time and lower the heat to a setting of 7 (Illustration 11). Let the mixture boil for six hours. Within the first hour all the solid should dissolve to leave a tea-colored solution (Illustration 12).

Illustration 10: Heat the stirring mixture to boiling.

Illustration 11: Continue boiling the mixture on a heat setting of 7 (your setting may vary).

Illustration 12: After about an hour the solid should dissolve to give a clear tea-colored solution.

4. Pour the hot solution and stir bar into a glass casserole dish (Illustration 13) in front of a fan and let the stirring ethanol evaporate overnight (Illustration 14), leaving a white paste but no liquid (Illustration 15). Set the heat so low that the hot plate is no more than warm without the dish.

Illustration 13: Pour the hot solution and stir bar into a glass casserole dish in front of a fan.

Illustration 14: Let the stirring ethanol evaporate overnight

Illustration 15: All the liquid should evaporate to leave a tan or light-yellow paste.

5. Add enough water (Illustration 16) to the stirring casserole dish to dissolve all the solid (Illustration 17), but do not heat it. This will require about half a liter. Once the flask from step A3 has dried, dissolve the residue in the flask in water and add the solution to the casserole dish (Illustration 18).

Illustration 16: Add half a liter of water, or more if necessary to eventually dissolve off the solid.

Illustration 17: Eventually all the solid should dissolve to give a light yellow or yellow solution.

Illustration 18: Rinse the residue from the reaction flask into the casserole dish.

6. Add four liters of water (Illustration 19) and 240 mL of concentrated hydrochloric acid (Illustration 20) to a 5 liter Erlenmeyer flask with stirring (Illustration 21). Heat the flask of dilute acid to boiling with magnetic stirring on a hot plate.

Illustration 19: Measure four liters of water and add it to a five liter Erlenmeyer flask containing a stir bar.

Illustration 20: Measure out 240 mL of concentrated hydrochloric acid

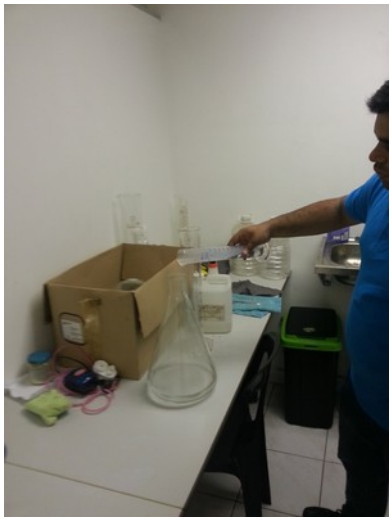

Illustration 21: Add the hydrochloric acid to the water in the flask.

7. Pour the liquid from the casserole dish into a closed separatory funnel through a funnel (Illustration 22), rinsing the dish with a spray of water. Slowly add the solution from the separatory funnel to the stirring, boiling acid in portions, taking care that the foaming does not cause the contents of the Erlenmeyer flask to overflow (Illustration 23). Rinse the separatory funnel into the acid using a spray of water (Illustration 24). Keep the Erlenmeyer flask purged with a stream of nitrogen to prevent darkening of the ibogaine if nitrogen is available. After the addition the yellow solution should still be strongly acidic (pH < 2) if enough hydrochloric acid was present.

Illustration 22: Add the solution from the casserole dish to the separatory funnel.

Illustration 23: Add the solution from the separatory funnel to the boiling acid slowly enough that it does not overflow.

Illustration 24: Rinse the separatory funnel into the acid with a water spray.

8. Heat the stirring Erlenmeyer flask until the contents have boiled for three minutes (Illustration 25), then turn off the heat and wait for the boiling to stop (Illustration 26).

Illustration 25: Boil the solution for three minutes.

Illustration 26: Then let it cool until the boiling stops.

9. Submerge the Erlenmeyer flask in room temperature water and stir until the contents have reached room temperature (Illustration 27).

Illustration 27: Swirl the flask in cool water until it is cool.

10. Refrigerate the Erlenmeyer flask (Illustration 28) for six hours to ensure that the ibogaine will precipitate as a solid in step A12.

Illustration 28: Refrigerate the yellow solution for six hours.

11. Dissolve 60 grams of sodium hydroxide (Illustration 29) in enough water to reach a final volume of 120 mL (Illustration 30) in a 250 mL Erlenmeyer flask and let the solution (Illustration 31) return to room temperature. This is a 50% solution of sodium hydroxide.

Illustration 29: Weigh 60 grams of sodium hydroxide into the flask.

Illustration 30: Add enough water to bring the volume to 120 mL.

Illustration 31: Swirl the flask until the sodium hydroxide has dissolved.

12. Add the sodium hydroxide solution slowly to the stirring Erlenmeyer flask of ibogaine HCl solution (Illustration 32) and let the suspension of ibogaine base (Illustration 33) stir for ten minutes. The mixture should be strongly basic (pH > 10) if enough sodium hydroxide was added (Illustration 34). If an oil separates (Illustration 35) instead of a powder, add a tiny amount of solid ibogaine base to the flask to initiate solidification of the ibogaine and place the flask in the refrigerator overnight.

Illustration 32: The yellow solution is clear before the sodium hydroxide solution is added.

Illustration 33: A fluffy precipitate of ibogaine base appears as the sodium hydroxide is added.

Illustration 34: The mixture should be strongly basic (> pH 10) if enough sodium hydroxide was added.

Illustration 35: If the mixture continues to look milky, add some solid ibogaine base from another batch to induce solidification.

13. Filter the ibogaine (Illustration 36) through a labelled, pre-weighed paper, rinse it with water (Illustration 37) and let it dry in a stream of air (Illustration 38). As the ibogaine dries, spread it (Illustration 39) and chop it up with a knife (Illustration 40) to facilitate drying. Weigh the crude ibogaine when it is dry (Illustration 41). The theoretical yield of ibogaine is 42.12 grams, or 84.24% of the initial weight of voacangine. If the product weighs more than this it probably means it is not finished drying or was not sufficiently rinsed with water. The expected yield is close to the theoretical.

Illustration 36: Filter the ibogaine from the yellow solution.

Illustration 37: Rinse the ibogaine with a spray of water.

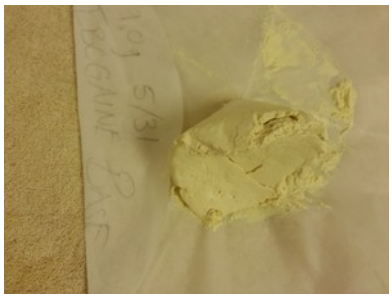

Illustration 38: Once the rinse has drained, set the filter paper on a dry towel.

Illustration 39: Use a butter knife to spread out the ibogaine so it dries faster.

Illustration 40: As the chunks of ibogaine become more firm, chop them into pieces with the knife.

Illustration 41: Record the weight of the crude ibogaine when it loses no further weight when drying.

Overview of Part B: Recrystallization of Ibogaine

Any improvements found for this part should be considered for Part C of Phase 3 because the recrystallizations in both parts apply the same concepts. The purpose of recrystallization is to increase the proportion the most abundant component of a mixture represents. Ideally the most abundant component, ibogaine in this case, would become completely pure and none of it would end up in the separated impurities. In practice neither of these conditions are met: the recrystallized ibogaine is still not perfectly pure and much ibogaine is left behind with the impurities to be recovered.

Recrystallization usually takes advantage of the increased solubility of substances in hot solvent compared with cold solvent, along with the general tendency of the most concentrated component (ibogaine) to leave solution as crystals as the solution cools before the more dilute impurities do. Recrystallization is an art and also requires a great deal of experimentation to optimize. Usually the boiling point is the best temperature to dissolve the substance to be recrystallized unless the substance melts below the solvent boiling point. However, the colder the solution is then made to induce further crystallization the more impurity tends to deposit, and this tendency needs to be assessed experimentally to find the best final temperature. Allowing the solution to sit and even partially evaporate over a long time may afford better crystal yields, but solutions of ibogaine decompose over time due to exposure to oxygen and especially light. Nevertheless the process should not be rushed because the purity of the ibogaine crystals is greater when they are allowed to form more slowly by allowing cooling to be gradual. For this reason the crystallizing mixture should not be disturbed since that increases the rate of nucleation, the number of crystals and thus the rate of their formation.

The second crop of ibogaine crystals is less pure than the first crop. The recycling of the second crop of ibogaine crystals into the crude ibogaine of the subsequent run is intended to recover the ibogaine they contain while attempting to remove the impurities with the residual ibogaine. It may be worthwhile attempting to obtain a third crop of ibogaine crystals by further concentrating the mother liquor, especially as the scale of this process increases. The residual ibogaine may have to be purified by chromatography such as preparative HPLC, silica flash chromatography using ethyl acetate or basic alumina flash chromatography using toluene. Any unreacted voacangine is expected to end up in this fraction and should be recovered to be recycled in Phase 3.

Once this process has been repeated many times so that the expected yield, product quality, labor requirements and potential complications are well known, other solvents should be substituted for methanol to see if any of these might lead to an improved procedure. The most important improvement to look for is a higher yield of first and second crop ibogaine, with a corresponding reduction in the yield of residual ibogaine which must be reprocessed. Another important improvement is an increase in the purity of the first crop ibogaine crystals without loss of yield. Ethanol has been used successfully in this step and will require about half the volume of methanol specified. Other appropriate solvents to consider, in order of promise, include isopropanol, toluene, petroleum naphtha, ethyl acetate, acetone, butanone, cyclohexane, chloroform, tetrahydrofuran, acetonitrile or mixtures thereof.

Part B Procedure: Recrystallization of Ibogaine

1. Put about 100 grams of the weighed crude ibogaine (Illustration 42) plus any second crop ibogaine from step B11 of the previous batch (Illustration 43) and a stir bar into a five liter Erlenmeyer flask (Illustration 44).

Illustration 42: Put a weighed amount of crude ibogaine into the flask.

Illustration 43: Any second-crop ibogaine available can be used to supplement the crude ibogaine.

Illustration 44: The carefully weighed second crop ibogaine is also added to the flask.

2. Use two liters of hot methanol (Illustration 45) to rinse the filter paper (Illustration 46) from the ibogaine in step A13 and put the rinse into the flask (Illustration 47).

Illustration 45: Heat the methanol to boiling.

Illustration 46: Rinse the filter paper with methanol.

Illustration 47: Let all the rinse drain into the flask.

3. Heat the stirring contents of the flask to simmering (Illustration 48) and add additional methanol as needed to dissolve the ibogaine (Illustration 49). About two more liters will be required. Some solid which is not ibogaine will refuse to dissolve and the addition of methanol should be stopped when all solid with the original consistency is gone (Illustration 50). If nitrogen is available, a stream should be used to purge the flask to prevent darkening of the ibogaine.

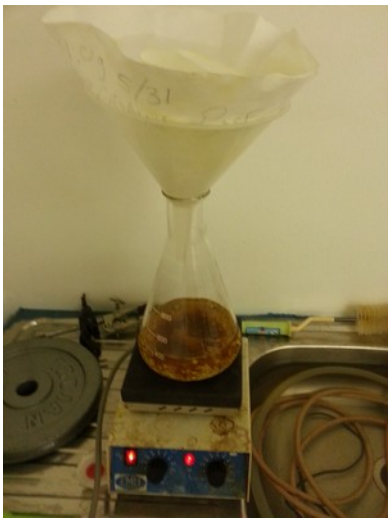

Illustration 48: Heat the stirring mixture to simmering.

Illustration 49: Add portions of methanol as needed to dissolve most of the solid.

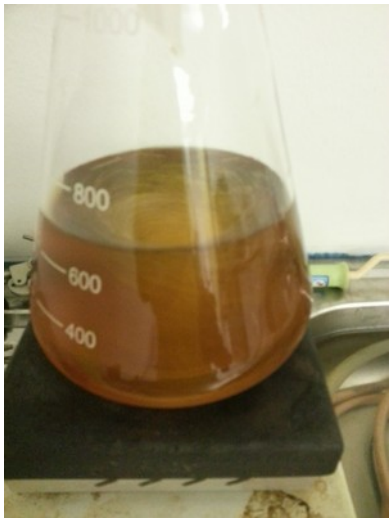

Illustration 50: If the chunks or globs are gone and only a bit of fine powder remains, stop adding methanol.

4. Decant or filter the hot ibogaine solution into a second 5 liter Erlenmeyer flask to remove any undissolved solid.

5. Let the flask cool as slowly as possible to room temperature (Illustration 51) and let it sit for at least eight hours after the ibogaine begins to crystallize.

Illustration 51: The slowly cooling flask before crystallization.

6. Refrigerate the flask (Illustration 52) for at least another eight hours (Illustration 53).

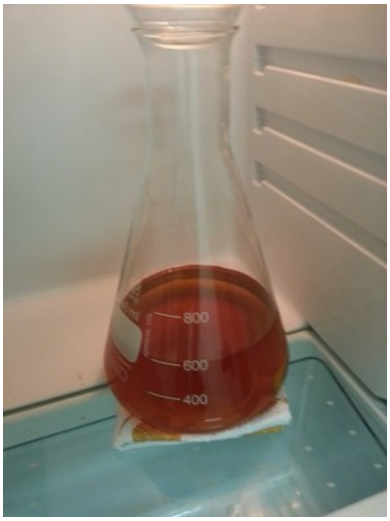

Illustration 52: The flask before refrigeration.

Illustration 53: The flask after refrigeration.

7. Thoroughly decant the mother liquor from the ibogaine base crystals into a 5 liter flat bottom round flask containing a stir bar (Illustration 54) and dry the ibogaine base crystals with a stream of air from an aquarium pump (Illustration 56). The expected yield of this first crop of ibogaine crystals (Illustration 56) is about 50% of the weight of the crude ibogaine.

Illustration 54: Decant the liquid to leave the crystals behind.

Illustration 55: Warm the flask while using a stream of air from an aquarium pump to dry the ibogaine crystals.

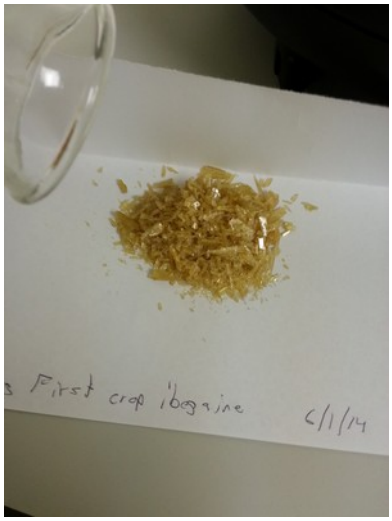

Illustration 56: Use a spoon to scrape the dry crystals from the flask to weigh.

8. Distill (Illustration 57) about 75% of the methanol away (Illustration 58) and save the distilled methanol for reuse (Illustration 59).

Illustration 57: Assemble the still as described previously in this documentation.

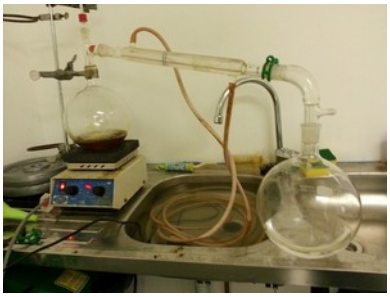

Illustration 58: Continue the distillation until the volume remaining is about 25% of the original.

Illustration 59: Save the distilled methanol for reuse in the next recrystallization.

9. Label the round flask and let it cool to room temperature (Illustration 60) and sit for at least six hours after crystallization of the second crop of ibogaine has started (Illustration 61).

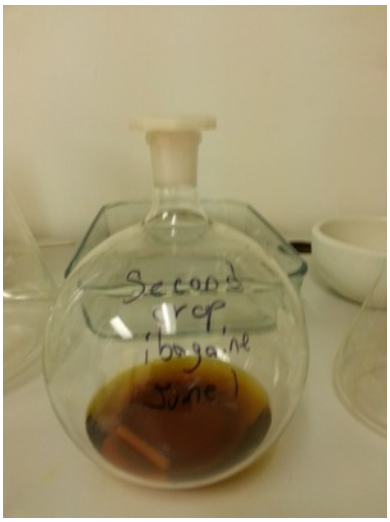

Illustration 60: Concentrated mother liquor before cooling.

Illustration 61: Second crop ibogaine crystals after sitting.

10. Refrigerate the round flask for at least six hours.

11. Decant the mother liquor from the second crop of ibogaine crystals into a casserole dish and dry the crystals with a stream of air from an aquarium pump. This crop of crystals can be added to the crude ibogaine in step B1 the next time this procedure is run. The expected yield is about 25% of the weight of the crude ibogaine.

12. Evaporate the mother liquor in the casserole dish with a fan until it becomes hard.

13. Scrape the residue out of the casserole dish with a razor blade and store it for recovery of the ibogaine using chromatography. The expected yield of residual ibogaine is about 25% of the weight of the crude ibogaine.

Overview of Part C: Precipitation of Ibogaine HCl

The use of acetone for precipitation of ibogaine HCl in Part C is based on both the good results obtained with precipitation of PTA HCl from iboga TA and with keeping voacangine, the main expected impurity in this case, from precipitating. The theory behind this is that the ester group that voacangine has and ibogaine lacks makes voacangine less basic than ibogaine. With a limited amount of hydrochloric acid present, the more basic ibogaine will preferentially combine with it to form the insoluble hydrochloride. In addition, acetone seems to have a nice balance of solvent properties allowing it to keep much of the remaining impurity dissolved while keeping only a little of the ibogaine, while conveniently mixing with the HCl solution added to it. It is also cheap, has very low toxicity and is easy to distill.

Hydrochloric acid fumes are irritating to the eyes and lungs, and ventilation by fumehood or window fan is recommended during its addition, along with eye protection and gloves. Acetone is highly flammable but mixes with water, so a stream of water can be used to extinguish it if it ignites. As with flammable solvents in general, sufficient acetone vapor may become explosive when mixed with air.

During precipitation the acetone has been noted to acquire a light green color. This may have been due to the dye from pH paper making its way back into the mixture from the glass rod used to apply it. Cleaning the glass rod between sample collections would test this hypothesis.

The acetone recovered by distillation in Step 6 is unstable because of the trace of hydrochloric acid it contains, and it will slowly react with itself over months in storage to form a smelly, higher boiling polymer. This can be prevented by adding a few grams of sodium hydroxide pellets to the acetone and redistilling it with rapid stirring to remove the acid. This gives acetone which is stable but is contaminated with water and therefore may not be suitable for use in this procedure. This wet acetone should be clearly labeled as such and used for general cleaning purposes.

A review of the acid volumes added and the final pH in this procedure in comparison with the yields may reveal that more or less acid is conducive to better yield or product purity.

Further purity of the product can be obtained by recrystallization of the ibogaine HCl from methanol or ethanol. Unless oxygen is excluded from the hot solution it will darken and this will frustrate the attempt to improve the apparent purity of the product. Also, the product may appear darker even if it is purer because the larger resulting crystals scatter less light and transmit more than the original powder, making what impurity is left much more visible.

The purity of the recovered impure ibogaine in step 8 has not been assessed, but may exceed that of the residual ibogaine in Part B Step 13, making it suitable to be combined with the crude ibogaine used in Part B Step 1.

Part C Procedure: Precipitation of Ibogaine HCl

1. Dissolve 50 grams of first crop ibogaine base (Illustration 62) in 500 mL of acetone (Illustration 63) in a 1 L Erlenmeyer flask containing a stir bar. Filter if necessary.

Illustration 62: Add the ibogaine base to the Erlenmeyer flask containing a stir bar.

Illustration 63: Add the acetone to the flask.

2. Add 10 mL of concentrated hydrochloric acid dropwise to the stirring Erlenmeyer flask.

Illustration 64: The ibogaine solution is clear yellow before the acid is added.

Illustration 65: During the addition of acid the solution will become very cloudy.

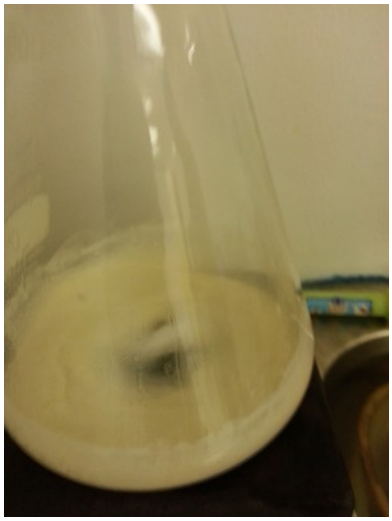

Illustration 66: After the acid is added the mixture should be thick with precipitated solid.

3. Add concentrated hydrochloric acid dropwise in 0.5 mL portions to the stirring flask (Illustration 67) until the pH of the liquid in the flask drops to pH 3 (Illustration 68). This will require about 5 mL more acid. It is the liquid, not the solid, which is being tested for pH. Apply it quickly to the pH test paper using a glass rod before the acetone can evaporate. Use the color that the paper first turns to indicate the pH, as the color may change over time.

Illustration 67: Add HCl in small portions, testing the pH of the mixture each time.

Illustration 68: Stop adding acid when the pH reaches 3. The pHs shown here are about 9, 7 and 6 from bottom to top.

4. Refrigerate the flask for at least six hours to ensure that as much as possible precipitates from the acetone (Illustration 69).

Illustration 69: Refrigerate the product.

5. Filter (Illustration 70, Illustration 71), rinse (Illustration 72, Illustration 73), dry (Illustration 74, Illustration 75) and weigh the finished ibogaine HCl from the suspension in the flask. The expected yield is close to theoretical.

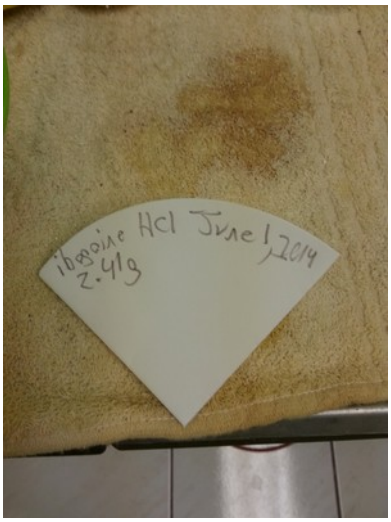

Illustration 70: Write the product, date and paper weight on a filter paper in pencil.

Illustration 71: Filter the ibogaine HCl product from the acetone.

Illustration 72: Rinse the stir bar and flask with acetone, and use this to rinse the product in the filter.

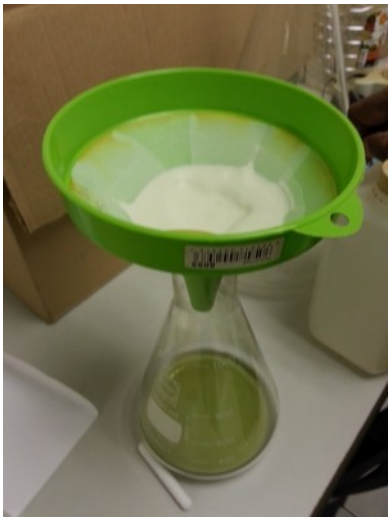

Illustration 73: Let all the acetone drain from the filter.

Illustration 74: Set the funnel on its side to let the acetone evaporate slowly.

Illustration 75: Move the paper onto the towel to allow the last of the acetone to evaporate.

6. Distill 90% of the acetone from the filtrate and pour what remains in a casserole dish (Illustration 76) to evaporate (Illustration 77).

Illustration 76: Pour the concentrated acetone solution into a casserole dish.

Illustration 77: Let the acetone solution evaporate in a well ventillated area.

7. Dissolve the residue from the evaporated acetone in two liters of water and add 20 mL of concentrated ammonia.

8. Filter, dry and weigh the recovered impure ibogaine and add it to the residue in step B13. The yield of recovered ibogaine is expected to be very low.

Ibogaine Production Data Collection Sheet

Last revised October 16, 2015

Chemist name: _____ Starting date: _____

A1. Weight of voacangine: _____ g Voacangine batch number: _____

A1. Volume of ethanol: _____ mL Weight of potassium hydroxide: _____ g

A3. Time reflux started: _____ Duration of reflux: _____ Nitrogen used (Y/N)

A5. Volume of water used to dissolve residue: _____ mL

A6. Volume of water _____ and concentrated HCl _____ used for decarboxylation

A7-8. pH after pouring reaction product into HCl: _____ Duration of boiling: _____

A11-12. Weight of sodium hydroxide for basification: _____ g pH after basification: _____

A13. Weight of crude ibogaine base: _____ g Yield: _____ %

B1. Weight of any second crop ibogaine added before recrystallization: _____ g

B2-3. Volume of methanol used for recrystallization: _____ mL

B7. Weight of first crop of ibogaine base: _____ g Yield: _____ %

B8. Estimated volume of mother liquor after concentration: _____ mL

B11. Weight of second crop of ibogaine base: _____ g Yield: _____ %

B13. Weight of residual ibogaine base from evaporated mother liquor: _____ g

C1. Volume of acetone used: _____ mL

C2-3. Total volume of HCl used to titrate the ibogaine: _____ mL Final pH:_____

C4. Duration of refrigeration: _____

C5. Weight of final ibogaine HCl: _____ g Yield: ____ % Batch number assigned: _____

Composition of ibogaine HCl: _____ % ibogaine _____ % voacangine Color: _____

C7. Volume of water used: _____ mL Volume of ammonia: _____ mL

C8. Weight of recovered impure ibogaine: _____ g