Theory

Part A takes advantage of the discovery that when a solution of voacangine in acetone was acidified with concentrated hydrochloric acid, in an attempt to precipitate the hydrochloride as is done for ibogaine, what actually happens is that everything but the voacangine precipitates. Maybe this has to do with the weakly basic nature of voacangine. But in any case, it provides a means to purify the voacangine (PVTA) from phase II by precipitating the impurities. The product is called VRA (Voacanga recovered alkaloids) to be analogous to the RA (Recovered Alkaloids) obtained using the same procedure with iboga. The purity of voacangine by this point should be around 80%.

Part B simply takes advantage of the slight solubility of voacangine in hot petroleum which much of the impurity in VRA apparently lacks. A novelty of this procedure is the distribution of the VRA throughout a mass of diatomaceous earth. The purpose of the diatomaceous earth is to provide a large surface area that the VRA can dissolve from while being itself inert, composed of silica. Fine sand might have been used instead. Being a uniform, small particle size, diatomaceous earth has the advantage of filtering easily and is used as an aid in filtering. If the VRA were heated with the naphtha without being mixed with the diatomaceous earth, it would fuse into a blob.

Part C is a standard recrystallization. This step could be repeated to increase the purity of the voacangine at the expense of labor but this doesn't seem necessary.

As with all phases on this manual, note that the amounts in the procedure can be scaled to the amounts on hand, and the size of the equipment adjusted accordingly. The times and temperatures should not be scaled.

Instructions for Phase III of the process for producing ibogaine from Voacanga:
Final purification of voacangine
Last updated August 11, 2015

Part A. PVTA to VRA:

1. Place 100 grams of PVTA (Illustration 1) in a 3 liter Erlenmeyer flask (Illustration 2) with a stir bar and add 1000 mL of acetone (Illustration 3).

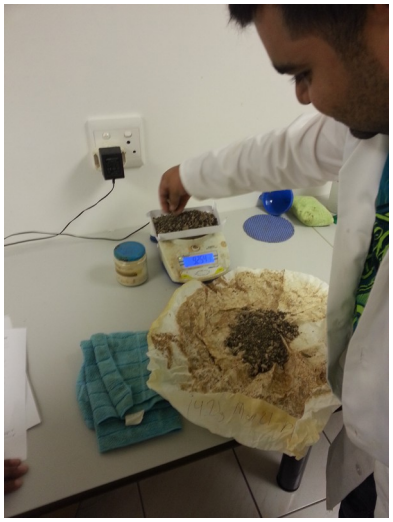

Illustration 1: Weigh the PVTA

Illustration 2: Pour the PVTA into an Erlenmeyer flask

Illustration 3: Add one liter of acetone to the flask

2. Stir for one hour or until all clumps of PVTA are gone.

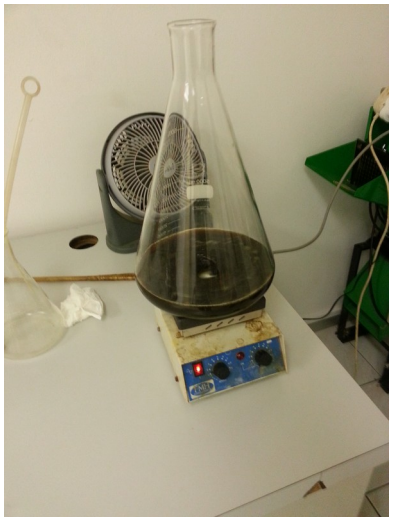

Illustration 4: Stir for an hour

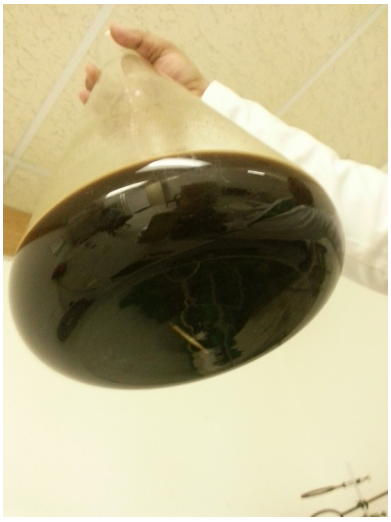

Illustration 5: Until the clumps are gone

3. Weigh (Illustration 6) and label (Illustration 7) a 18 or 24 cm filter paper. It is especially important to use pencil when filtering acetone since most ink dissolves in acetone. Be sure to fold the paper so the writing is visible on the outside of the filter cone (Illustration 15). Filter into a second 3 liter Erlenmeyer flask containing a stir bar to remove the fine chocolate-brown solid (Illustration 8). Cover the funnel to keep the acetone from evaporating (Illustration 9).

Illustration 6: Weigh a filter paper

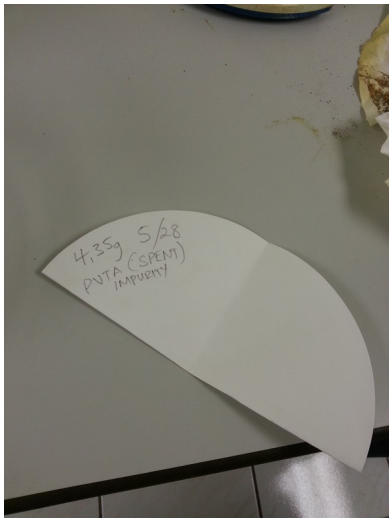

Illustration 7: Label the paper with the date, voacangine batch number and "PVTA impurity" in pencil

Illustration 8: Filter the impurity from the solution

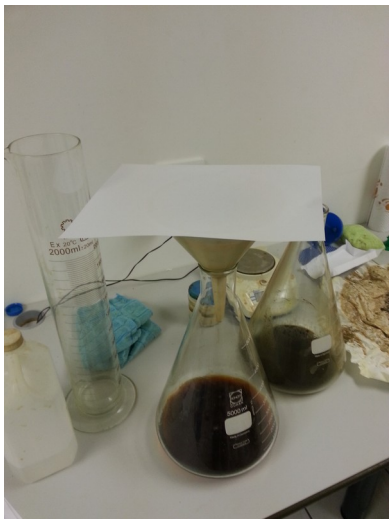

Illustration 9: Cover the funnel to prevent the acetone from evaporating

4. Rinse the flask (Illustration 10) and the filter (Illustration 11) with 600 mL more acetone. Stir the solid carefully to keep from tearing the paper (Illustration 12), and let the filter drain (Illustration 13). Dropping the filter onto the flask from about an inch helps the solid to settle and drain additional liquid.

Illustration 10: Use the acetone to rince the flask

Illustration 11: Rinse the filter with acetone

Illustration 12: Stir the solid so it is all exposed to the acetone

Illustration 13: Let the acetone drain

5. Dry the filter paper (Illustration 14, Illustration 15, Illustration 16), weigh the spent PVTA and recycle it with the Voacanga bark.

Illustration 14: Remove the drained filter paper

Illustration 15: Which should be properly labeled

Illustration 16: And let it dry

6. Add 15 mL of concentrated hydrochloric acid (Illustration 17) dropwise (Illustration 18), until precipitation is heavy (Illustration 19), to the stirring acetone extract.

Illustration 17: Measure out the HCl

Illustration 18: Add HCl dropwise to let precipitation start gradually

Illustration 19: Once the precipitation starts, the rest of the HCl can be added more quickly

7. Add concentrated hydrochloric acid dropwise in 1 mL portions (Illustration 20), measuring the pH after each portion (a stir bar retriever is being used for sampling, Illustration 21), until the pH of the acetone solution drops to 3 (Illustration 22, Illustration 23). This should take about six portions to accomplish. The result should be a fine solid (Illustration 24) that settles nicely (Illustration 25).

Illustration 20: Add HCl in 1 mL portions

Illustration 21: Sampling the acetone

Illustration 22: Apply the sample to pH test paper

Illustration 23: The pH should be approximately 3.

Illustration 24: After adding the acid there is a fine solid

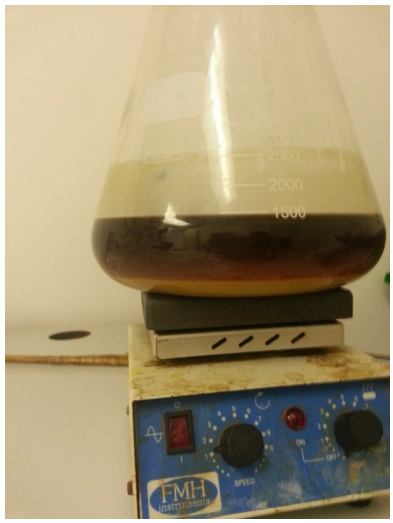

Illustration 25: When the stirring stops the solid settles nicely

8. Please the flask in a refrigerator for at least six hours to maximize the precipitation of solid.

9. Weight (Illustration 26) and label an 18 or 24 cm filter paper with its weight, the date, the voacangine batch number and “PVTA HCl” in pencil (Illustration 27). Remove the flask from the refrigerator (Illustration 28) and filter the light tan PVTA HCl from the orange or brown mixture into a 3 liter Erlenmeyer flask (Illustration 29) or, ideally, directly into a 2 liter flat-bottom round flask containing a stir bar (Illustration 30). Rinse the flask (Illustration 31) and the solid (Illustration 32) with about another 100 mL of acetone. Let the solid dry (Illustration 33), weigh it and submit it to be recycled with the Voacanga bark. The PVTA HCl normally darkens on drying and weighs about 36 grams. The dried residue of PVTA HCl in the Erlenmeyer flask can be dissolved with water and added to Voacanga bark extract before precipitation (Illustration 34).

Illustration 26: Weigh a filter paper

Illustration 27: Label the paper in pencil

Illustration 28: Remove the refrigerated flask

Illustration 29: Filter the PVTA HCl from the acetone solution of VRA

Illustration 30: Pour the filtrate into a distilling flask or, better, filter into it directly

Illustration 31: Add about 100 mL of acetone to the flask to rinse it

Illustration 32: Rinse the PVTA HCl with acetone

Illustration 33: Let the PVTA HCl dry

Illustration 34: Rinse the PVTA HCl adhering to the flask into bark extract with water

10. Distill the acetone from the filtrate (Illustration 35) until about 300 mL of solution is left (Illustration 36), then carefully dismantle the still (Illustration 37). The distilled acetone contains some water and also hydrochloric acid and will polymerize if stored. It can be stored by distilling it again from sodium hydroxide, and then used for cleaning purposes. Reusing the recycled acetone in this procedure is not ideal because it still contains some water.

Illustration 35: Distill the acetone from the VRA solution

Illustration 36: Stop the distillation when approximately 300 mL remain

Illustration 37: Carefully remove the receiving flask and the boiling flask from the condenser

11. Pour the concentrated acetone solution of VRA into a glass casserole dish (Illustration 38) and set in a breeze (Illustration 39) in a ventilated area until the acetone has evaporated. The smell of acetone should be gone and a light brown syrup should remain (Illustration 40).

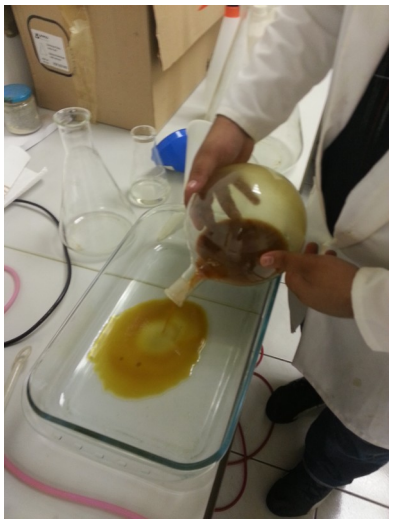

Illustration 38: Pour the contents of the distilling flask into a casserole dish

Illustration 39: Put the casserole dish in a breeze so the acetone evaporates

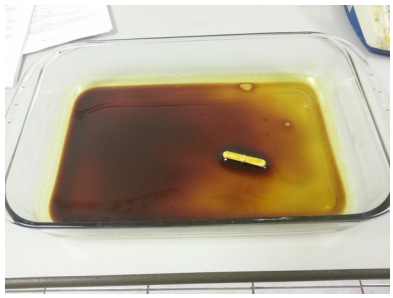

Illustration 40: When the smell of acetone is gone, there should be a clear, very think and sticky liquid remaining

12. Add two liters of water to the casserole dish (Illustration 41) and stir with a spoon (Illustration 42) until all the syrup has dissolved (Illustration 43). Rinse the dried residue from the round bottom distilling flask into the casserole dish with water.

Illustration 41: Add two liters of water to the casserole dish

Illustration 42: Use a spoon to stir the paste in the casserole dish

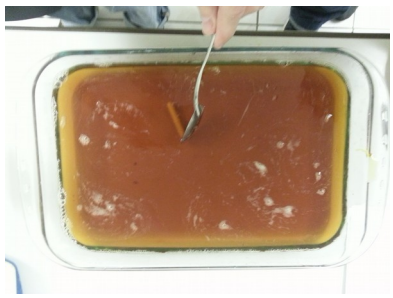

Illustration 43: Until it has all dissolved

13. Add 75 mL of 25% ammonia to the dark yellow solution in the casserole dish (Illustration 44) and mix thoroughly with the spoon (Illustration 45) until the creamy yellow mixture is homogeneous (Illustration 46).

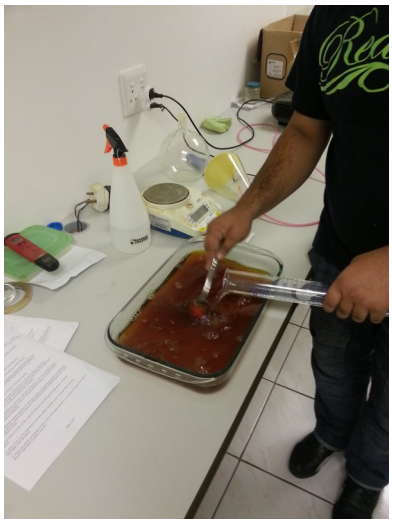

Illustration 44: Add ammonia to the VRA solution

Illustration 45: Stir with a spoon

Illustration 46: Until completely homogeneous

14. Weigh and label a 24 cm filter paper with its weight, the date, the voacangine batch number and "VRA" with pencil. Filter the yellow precipitate of VRA (Illustration 47), rinse with water (Illustration 48) and set it in a stream of warm air to dry (Illustration 49). It should weigh about 52 grams once dry.

Illustration 47: Filter the VRA

Illustration 48: Rinse the dish and filter with water

Illustration 49: Dry the VRA in a stream of air

Part B. Leaching of VRA with petroleum:

1. Grind 100 grams of VRA (Illustration 50) with 100 grams (Illustration 51) of diatomaceous earth (Illustration 52) in a coffee grinder (Illustration 53) until it is a fine, homogeneous powder (Illustration 54, Illustration 55).

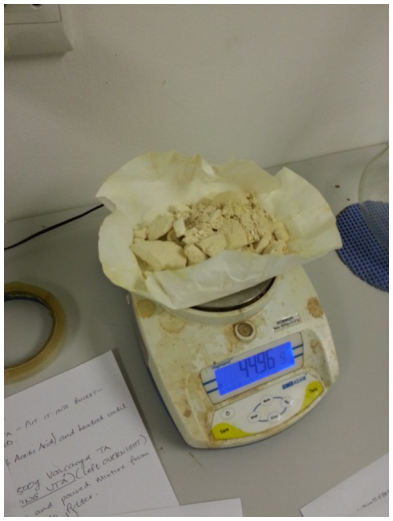

Illustration 50: Accurately weigh out the VRA

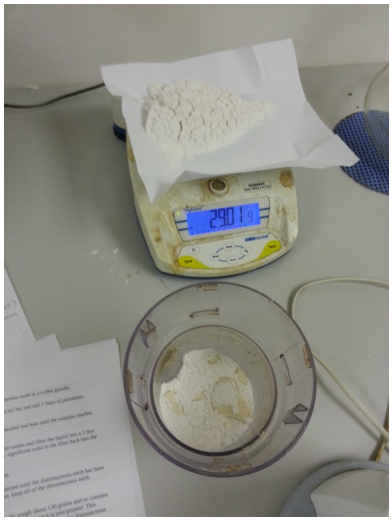

Illustration 51: Weigh out an equal amount of diatomaceous earth and put it with the VRA in the grinder

Illustration 52: This diatomaceous earth was intended to filter swimming pool water

Illustration 53: Grind the equal amounts of VRA and diatomaceous earth until all the VRA is a fine powder

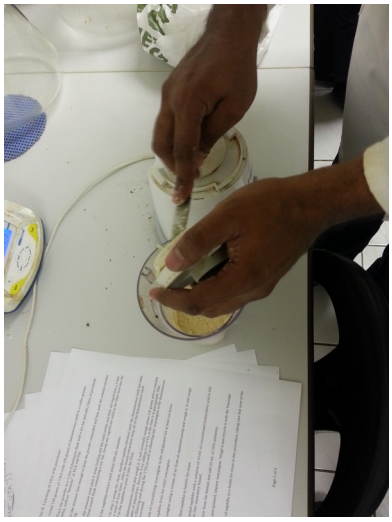

Illustration 54: What doesn't scrape off can be rinsed onto filter papers to be recycled with acetone

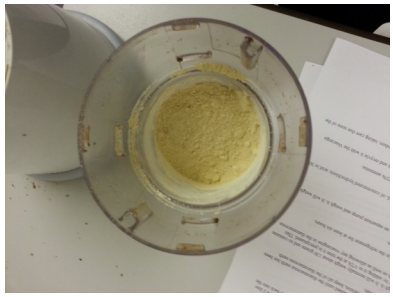

Illustration 55: The final mixture should be a fine yellow powder

2. Place the VRA/diatomaceous earth powder into a 3 liter Erlenmeyer flask with a stir bar (Illustration 56) and add 2 liters of petroleum naphtha (Illustration 57), sold as “benzine” at Pick and Pay in South Africa (Illustration 58). The boiling point of this product is around 80°C.

Illustration 56: Add the VRA/diatomaceous earth to an Erlenmeyer flask

Illustration 57: Add petroleum naphtha to the flask

Illustration 58: "Benzine" on the shelf at Pick 'n' Pay.

3. Stir the mixture fast enough to keep the powder suspended (Illustration 59) and heat until the solution reaches boiling (Illustration 60).

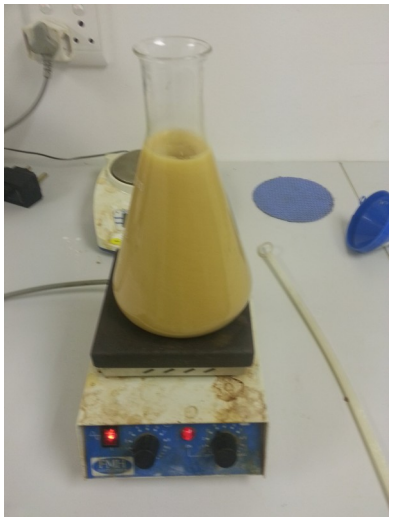

Illustration 59: Stir the mixture so the solid is suspended in the liquid

Illustration 60: Heat the mixture to boiling but do not let much liquid boil away

4. Turn off the heat and let the mixture stir until it stops boiling. If this is the first cycle for these steps, weigh a 18 or 24 cm filter paper and write the weight, the date, the voacangine batch number and “Spent diatomaceous earth” on it in pencil. Filter the hot mixture into an accurately weighed 2-liter flat-bottom round flask containing a stir bar (Illustration 61, Illustration 62). Scoop any significant solid in the filter back into the flask (Illustration 63).

Illustration 61: Filter the hot petroleum naphtha extract of the VRA

Illustration 62: Wait until the naphtha finishes draining from the filter

Illustration 63: Return the diatomaceous earth that gets into the filter to the flask

5. Place the round flask containing the naphtha on a hot plate/stirrer and attach it to a thermometer adapter bearing a thermometer (Illustration 64). Clamp the thermometer to a West condenser with a Keck clamp and supply a flow of cool water to the jacket of the condenser (Illustration 65). Attach a vacuum adapter to the other (male) end of the condenser and attach an empty round flask to the adapter (Illustration 66). Distill off most of the naphtha (Illustration 67) until about 200 mL remains in the original flask (Illustration 68). Turn off the heat and, once distillation stops, carefully dismantle the still (Illustration 69).

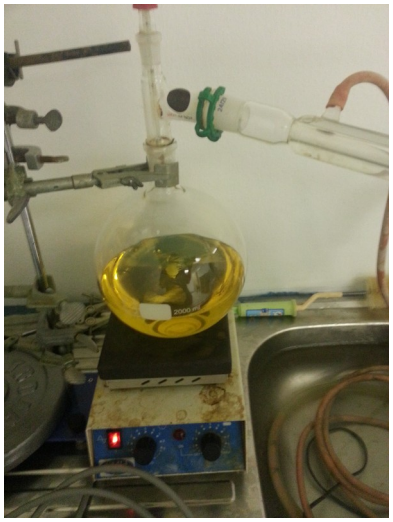

Illustration 64: The round flask with the naphtha extract

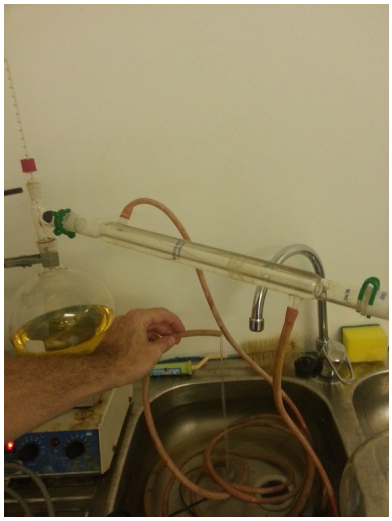

Illustration 65: A condenser to recover the naphtha as it boils away

Illustration 66: An empty round flask to receive the distilled naphtha

Illustration 67: Distill most of the naphtha

Illustration 68: Stop distilling when about 200 mL of VRA extract remains.

Illustration 69: Carefully dismantle the still

6. Return to step 2 using the clean, distilled naphtha obtained (Illustration 70) (adding fresh naphtha to match the original volume) until the diatomaceous earth has been extracted a total of four times. After the fourth extraction, keep all of the diatomaceous earth suspended in the naphtha to pour it into the filter (Illustration 71). After the fourth distillation return the distilled naphtha to the naphtha containers (Illustration 72).

Illustration 70: Extract the diatomaceous earth again with the distilled naphtha

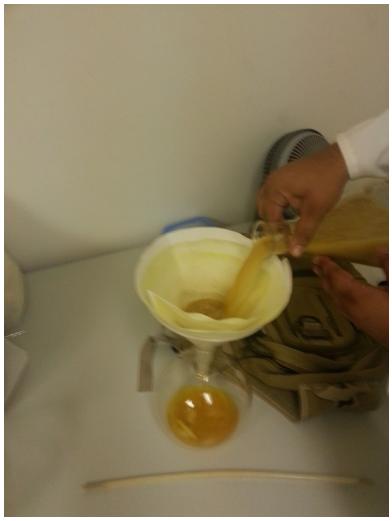

Illustration 71: After boiling, filter through the same paper used for the diatomaceous earth previously

Illustration 72: When the cycle of extractions is complete, store the naphtha in the original bottles

7. After all the liquid has drained (Illustration 73), dry the diatomaceous earth (Illustration 74), weigh it (Illustration 75) and record the weight (Illustration 76). It will normally weigh about 130 grams and so contains about 30 grams of impurities. Recycle it (Illustration 77) by adding it to VTA at the time it is precipitated. This should aid in filtering and drying the VTA as well as allowing any voacangine in the diatomaceous earth to be recovered. Extract the used filter paper with the next batch of *Voacanga* bark to recover any voacangine in it (Illustration 78).

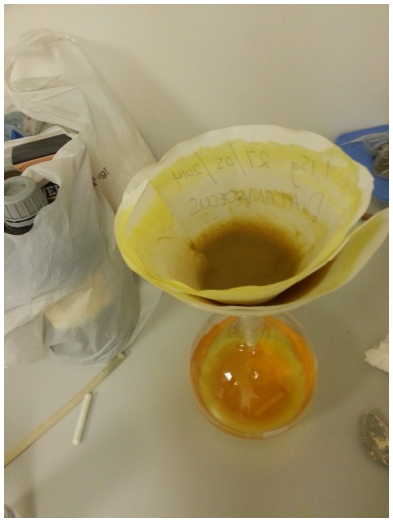

Illustration 73: Drain all the VTA extract

Illustration 74: Let the diatomaceous earth dry

Illustration 75: Weigh the used diatomaceous earth

Illustration 76: Record the weight of the used diatomaceous earth

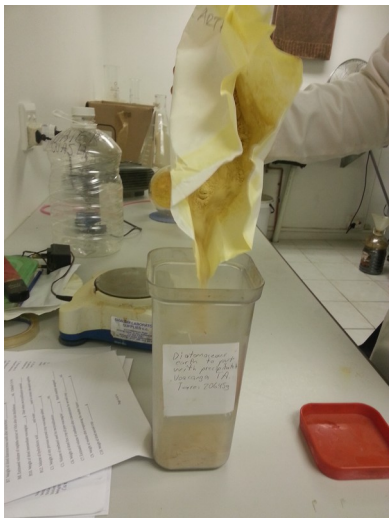

Illustration 77: Store the used diatomaceous earth until it can be added to precipitated VTA

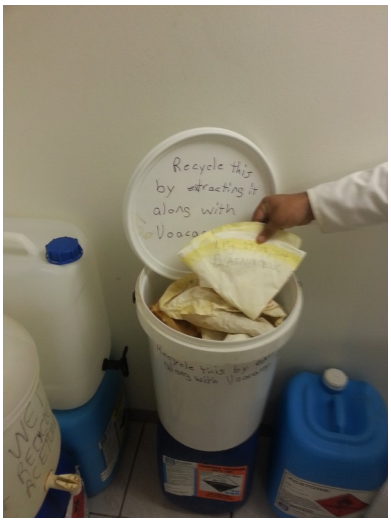

Illustration 78: Save the used filter paper to be extracted with the next batch of Voacanga bark

8. Remove the stir bar from the hot flask containing the VRA extract and stopper it (Illustration 79). Put the flask in a refrigerator (Illustration 80) for at least six hours until the voacangine has finished crystallizing (Illustration 81).

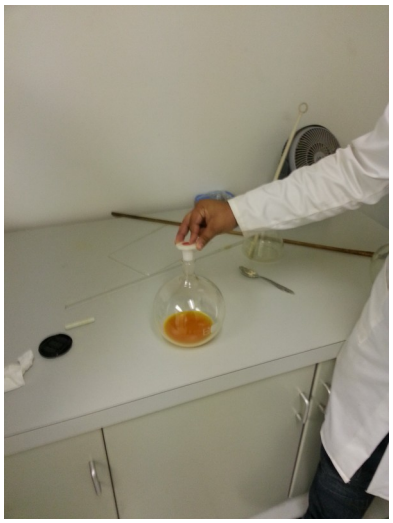

Illustration 79: Remove the stir bar and stopper the flask

Illustration 80: Place the flask in a refrigerator for at least six hours

Illustration 81: Voacangine finished crystallizing

9. Decant the naphtha from the solid voacangine into a one liter separatory funnel while it is still cold (Illustration 82). Rinse the solid in the flask with a small amount (~20 mL) of clean naphtha (Illustration 83) and add the rinse to the separatory funnel (Illustration 84).

Illustration 82: Decant the mother liquor into a separatory funnel

Illustration 83: Rinse the crystals of voacangine with a bit of clean naphtha

Illustration 84: Decant the rinse from the crystals into the separatory funnel

10. Dry the voacangine using a stream of air from an aquarium pump until it is dry (Illustration 85). Gently break up the mass of voacangine crystals using a soft rod such as a plastic stir bar retriever (Illustration 86) and continue the drying. The weight of the crystals can be determined by subtracting the weight of the flask now from what it weighed in step 4 when it was empty, or the solid can be removed for further drying and storage if not proceeding to part C. If the voacangine is removed from the flask, the residue in the flask (Illustration 87) can be weighed and the flask reused for this par. The voacangine will weigh about 70 grams.

Illustration 85: Dry the voacangine with a stream of air from an aquarium pump and gentle warming from a heater

Illustration 86: Use a rod to gently break up the mass of crystans and scrape them from the glass to facilitate drying or to remove them from the flask

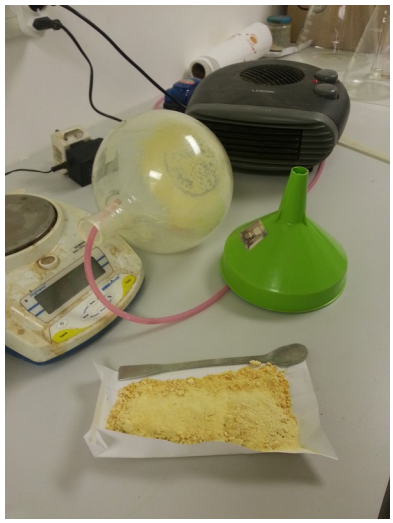

Illustration 87: If the voacangine is removed from the flask, let it dry completely before recording its weight

11. Place the distilled naphtha in bottles for reuse.

12. Extract the decanted naphtha with a solution of 10 mL of concentrated hydrochloric acid in 500 mL of water (Illustration 88) in the one liter separatory funnel (Illustration 89) to recover the voacangine from the naphtha. Let the layers separate (Illustration 90).

Illustration 88: Add the dilute HCl to the naphtha mother liquor

Illustration 89: Shake the stoppered separatory funnel thoroughly

Illustration 90: Wait for the layers to separate

13. Remove the stopper and drain the yellow acid layer from the bottom, keeping the colorless naphtha layer in the separatory funnel (Illustration 91). Add 20 mL of 25% ammonia (Illustration 92) and mix thoroughly (Illustration 93).

Illustration 91: Remove the stopper and drain the lower yellow layer

Illustration 92: Add ammonia to the yellow aqueous layer to make it basic

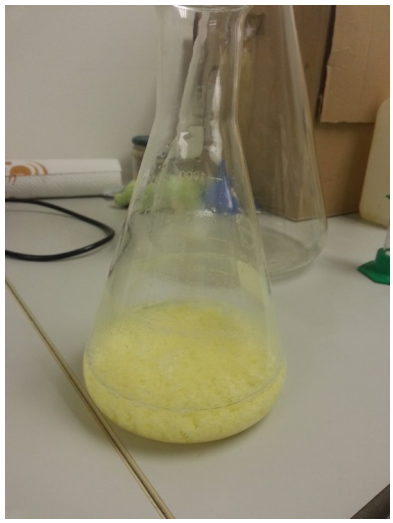

Illustration 93: Mix thoroughly to get a fluffy yellow solid containing recovered voacangine

14. Filter (Illustration 94, Illustration 95) and dry (Illustration 96) the precipitated, impure voacangine. Weigh it and recycle it with the Voacanga bark to be extracted.

Illustration 94: Filter the recovered voacangine through a weighed paper

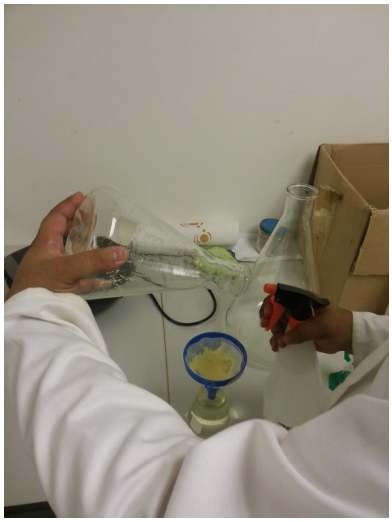

Illustration 95: Rinse any remaining solid into the filter with a spray of water

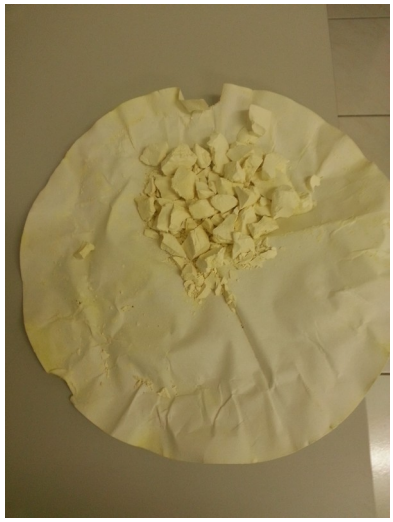

Illustration 96: Weigh and recycle the recovered voacangine

15. Put the upper layer of naphtha from step B12 in a bottle for reuse in this procedure (Illustration 97), taking care that none of the water gets in.

Illustration 97: Recycle the naphtha

Part C. Recrystallization:

1. To 100 grams of voacangine from part B combined with any second crop voacangine from step C10 add 420 mL of methanol. It is convenient to leave the voacangine in the flat bottom round flask with the stir bar from part B.

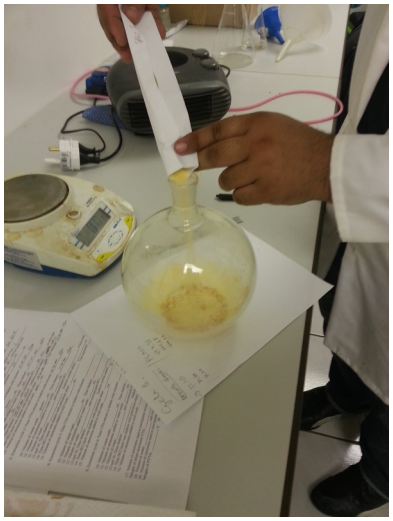

Illustration 98: Put a carefully weighed quantity of voacangine in a flat-bottom round flask

Illustration 99: Add the measured amount of methanol to the flask

Illustration 100: The voacangine can be weighed in the flask in Part B without removing it

2. Heat the mixture to boiling and let it stir until no more solid dissolves. Some of the voacangine or an impurity may not dissolve.

Illustration 101: Stir the mixture and heat it

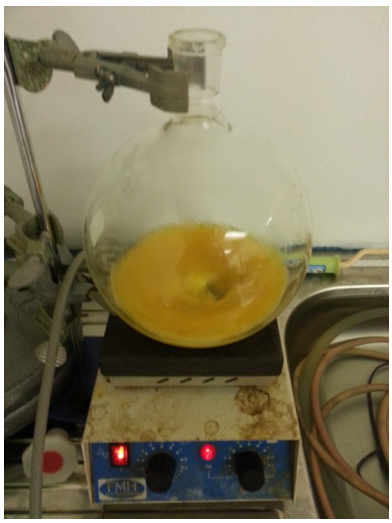

Illustration 102: Heat until the methanol boils and no more solid appears to dissolve.

3. Decant the liquid into a 1 liter Erlenmeyer flask. If there is a fine solid present which does not look the same as the voacangine crystals, decant the liquid through a filter (Illustration 103). Retain any undissolved voacangine in the flat bottom round flask (Illustration 104). Heat the Erlenmeyer flask to keep the voacangine in solution until the filtration is finished (Illustration 105).

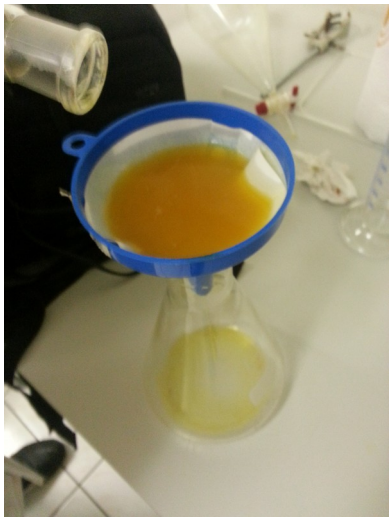

Illustration 103: Filter the fine solid impurity out of the hot solution

Illustration 104: Keep undissolved voacangine in the round flask for the next step

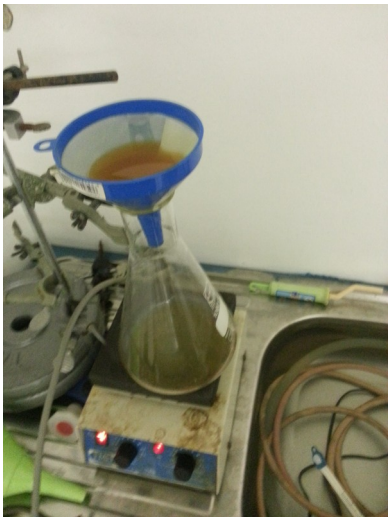

Illustration 105: Keep the filtrate hot but don't let it boil away

4. Use approximately another 350 mL of methanol in portions (Illustration 106) to dissolve the remaining voacangine and to rinse the filter, bringing it to boiling and stirring for ten minutes (Illustration 107) before decanting. Use only as much methanol as needed to dissolve the voacangine and rinse the filter. Cover the filter to keep the solution hot and prevent evaporation of the methanol (Illustration 108). Submit the voacangine-encrusted filter paper for recycling with the next Voacanga bark to be extracted.

Illustration 106: Add ~50 portions of methanol and heat the flask to dissolve the remaining voacangine

Illustration 107: Heat each portion of methanol to simmering but don't let it boil away

Illustration 108: Keep the methanol in the filter hot by covering it, and heat the flask but don't let it boil away

5. Let the covered Erlenmeyer flask containing the voacangine solution cool as slowly as possible without disturbance to maximize the size and purity of the voacangine crystals that form (Illustration 109). Once it has been at room temperature for at least eight hours, crystals should be forming. Refrigerate the flask for at least another eight hours (Illustration 110) to increase the yield of crystals (Illustration 111).

Illustration 109: Let the covered flask cool slowly and sit at room temperature for eight hours

Illustration 110: Refrigerate the flask to increase the yield of crystals

Illustration 111: Crystals of voacangine after refrigeration

6. Thoroughly decant the cold mother liquor from the pure, first crop voacangine crystals into a 2-liter flat bottom round flask and dry the crystals using a stream of air from an aquarium pump (Illustration 112) before scraping them from the flask (Illustration 113) and recording their weight (Illustration 114). This crop of voacangine can be converted into ibogaine. The expected yield is around 50 grams.

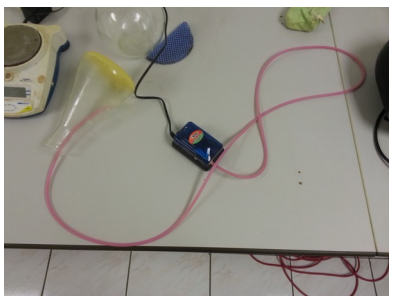

Illustration 112: Dry the voacangine with a stream of air from an aquarium pump

Illustration 113: Gently scrape the voacangine crystals from the flask with a scoop or spoon

Illustration 114: Dry voacangine on a weighed paper

7. Distill the mother liquor from the round flask until its volume is approximately one-quarter of the original (~200 mL) (Illustration 115, Illustration 116). Save the distilled methanol for reuse (Illustration 117).

Illustration 115: Distill about 75% of the original volume – this picture is less than full scale.

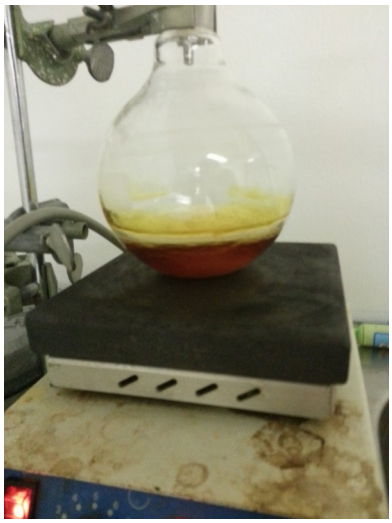

Illustration 116: At the end of the distillation there should not yet be solid in the concentrated mother liquor

Illustration 117: Put the distilled methanol in the original container to save it for reuse

8. Let the flask cool to room temperature (Illustration 118) and then refrigerate it at least eight hours (Illustration 119) until no more crystals form (Illustration 120).

Illustration 118: Let the flask cool to room temperature

Illustration 119: Refrigerate the flask at least eight hours

Illustration 120: Second crop voacangine after refrigeration

9. Decant the mother liquor in portions onto a weighed wad of paper towels sufficiently large to absorb all the liquid (Illustration 121). Dry the paper in front of a fan until dry (Illustration 122) and record the weight increase. Submit the paper to be recycled with Voacanga bark to be extracted in the future (Illustration 123).

Illustration 121: Decant the mother liquor from the second crop onto a wad of paper towels

Illustration 122: Let the paper towels dry in a stream of warm air

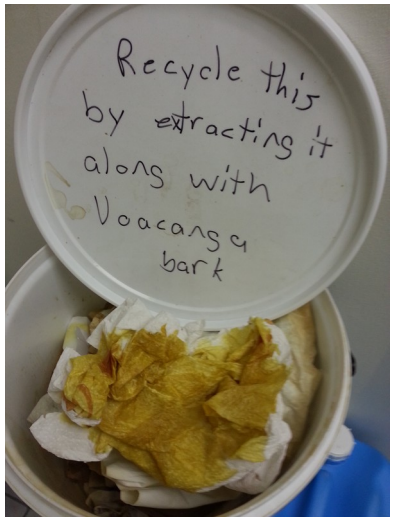

Illustration 123: Put the dried paper in a bucket of recyclables

10. Dry the second crop of voacangine crystals using a stream of air from an aquarium pump (Illustration 124), scrape them from the flask (Illustration 125) and weigh them (Illustration 126). This crop of crystals can be added to the voacangine isolated from naphtha the next time this procedure is run. The residue in the flask can be dissolved in a little hot acetone or methanol, evaporated on a paper towel and recycled as in step C9.

Illustration 124: Dry the crystals with a stream of air from an aquarium pump

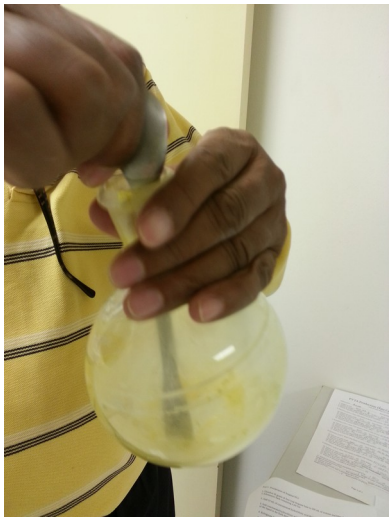

Illustration 125: Carefully scrape the voacangine from the flask with a scoop or spoon

Illustration 126: Weigh the second crop of voacangine crystals

Voacangine Production Data Collection Sheet

Last revised May 27, 2014

Chemist name: _____ Starting date: _____

A1. Weight of PVTA: _____ g PVTA batch number: _____

A1-4. Acetone volume used for extraction: _____ mL Acetone rinse: _____ mL

A3. Weight of the dried brown impurity filtered from the acetone: _____ g

A6-7. Volume of hydrochloric acid used to reach pH of 3: _____ mL Acid strength: _____ %

A9. Weight of dried PVTA HCl filtered from the acidified acetone: _____ g Chilled (Y/N)

A13. Volume of ammonia used to precipitate VRA: _____ mL Ammonia strength: _____ %

A14. Weight of dry VRA: _____ g Date when VRA purification started: _____

B1. Weight of diatomaceous earth added to the VRA: _____ g

B2. Volume of boiling petroleum naphtha used to extract the VRA: _____ mL

B2-6. Number of times the VRA/diatomaceous earth is extracted: _____

B7. Weight of dried diatomaceous earth after extraction: _____ g

B8. Estimated volume of naphtha extract of VRA after last distillation: _____ mL Chilled? (Y/N)

B10. Weight of dried intermediate voacangine: _____ g Date when recrystallization started: _____

B12. Volume of hydrochloric acid _____ and water _____ used to extract decanted naphtha.

C1. Weight of any previous second crop voacangine added: _____ g

C1. Volume of methanol used for recrystallization: _____ mL

C6. Weight of dried first crop of pure voacangine: _____ g Batch number assigned: _____

C7. Estimated volume of methanol after concentration for second crop: _____ mL

C9. Weight of residue after evaporating the mother liquor: _____ g

C10. Weight of dried second crop: _____ g