Training Procedure for Clandestine Laboratories

1.0 Purpose - This procedure provides an introduction to the synthesis and analysis of clandestine laboratories for Forensic Scientists.

2.0 Scope - This procedure applies to trainees in Drug Chemistry at the Raleigh, Triad and Western locations of the State Crime Laboratory.

3.0 Definitions

- **Clandestine Laboratory -** an illicit operation consisting of a sufficient combination of apparatus and chemicals that either has been or could be used in the manufacture or synthesis of controlled substances.

4.0 Procedure

4.1 Objectives

4.1.1 Be familiar with the major types of clandestine labs found in the State of North Carolina.

4.1.2 Be able to list the ingredients necessary to manufacture methamphetamine based on the primary methods utilized in North Carolina.

4.1.3 Be able to identify dangerous gases, reactants, and products found in a methamphetamine lab.

4.1.4 Be able to calculate a theoretical yield of methamphetamine based on the number of (pseudo)ephedrine tablets found at a scene.

4.1.5 Be able to describe an anhydrous ammonia condenser.

4.1.6 Be familiar with the reactants and reactions that occur at clandestine labs with respect to the production of methamphetamine.

4.1.7 Be able to indicate where a certain chemical will be used during the production of methamphetamine.

4.1.8 Be familiar with air monitoring equipment and the basic safety equipment necessary to process a clandestine drug lab.

4.1.9 Be familiar with evidence packaging and analysis.

4.1.10 Review the Clandestine Labs section of the Drug Chemistry worksheet in FA with the Clandestine Laboratory Training Assistant (or his/her designee).

4.1.11 Successfully complete a written exam.

4.2 Types of Methamphetamine Labs

4.2.1 Red Phosphorus

4.2.1.1 (pseudo)ephedrine – extracted from cold and allergy preparations using methanol ("Heet").

4.2.1.2 Red phosphorus – removed from match book striker plates using rubbing alcohol or acetone.

4.2.1.3 Iodine crystals – precipitated by combining hydrogen peroxide (usually 3%) with tincture of iodine (usually 7%) and adding acid.

4.2.1.4 Synthesis

4.2.1.4.1 Extract (pseudo)ephedrine from tablets.

4.2.1.4.2 Combine (pseudo)ephedrine, red phosphorus, iodine crystals, and water.

4.2.1.4.3 Cook (heat) for about 3.5 hours.

4.2.1.4.4 Use sodium hydroxide ("Rooto Drain Opener") to make the solution basic.

4.2.1.4.5 Add a solvent (must not be miscible with aqueous layer, e.g., ether or tetrachloroethylene).

4.2.1.4.6 Salt out the methamphetamine using a hydrogen chloride gas generator.

4.2.1.5 The cold cook method can also be used due to this reaction being quite exothermic. All steps are the same with the exception of using heat, causing the method to not be as efficient as when a heat source is used.

4.2.2 Anhydrous Ammonia

4.2.2.1 (pseudo)ephedrine – extracted from cold and allergy preparations using methanol ("Heet").

4.2.2.2 Anhydrous ammonia – used in agriculture and stolen from large tanks or precipitated using an anhydrous ammonia condenser.

4.2.2.3 Lithium or sodium metal – lithium usually collected from lithium batteries.

4.2.2.4 Anhydrous ammonia production

4.2.2.4.1 Some form of fertilizer is reacted with water and a base such as sodium hydroxide ("Rooto Drain Opener") inside of a container affixed with plastic tubing.

4.2.2.4.2 The plastic tubing is coiled, typically around a piece of larger diameter pipe, and placed inside a water cooler.

4.2.2.4.3 Dry ice and acetone, or propane, is placed in the bottom of the cooler.

4.2.2.4.4 As the gas produced cools, it liquefies and is collected.

4.2.2.5 Synthesis

4.2.2.5.1 Extract (pseudo)ephedrine from tablets.

4.2.2.5.2 Combine (pseudo)ephedrine, lithium (or sodium), and anhydrous ammonia.

4.2.2.5.3 Allow the reaction to occur for approximately 45 minutes.

4.2.2.5.4 Add water slowly to react with any un-reacted lithium.

4.2.2.5.5 Add a solvent (must not be miscible with aqueous layer, e.g., ether or tetrachloroethylene).

4.2.2.5.6 Remove the solvent and salt out the methamphetamine using a hydrogen chloride gas generator.

4.2.3 One Pot

4.2.3.1 (pseudo)ephedrine – typically whole tablets or crushed tablets are added to one-pot reaction vessel.

4.2.3.2 Ammonia – some form of fertilizer containing nitrogen, commonly through Instant Cold packs

4.2.3.3 Lithium or sodium metal – lithium usually is collected from lithium batteries.

4.2.3.4 Base – usually a drain opener containing sodium hydroxide.

4.2.3.5 Organic solvent – (must not be miscible with aqueous layer, e.g., ether or tetrachloroethylene).

4.2.3.6 Synthesis

4.2.3.6.1 Optional: Crush tablets containing (pseudo)ephedrine.

4.2.3.6.2 Combine (pseudo)ephedrine, lithium (or sodium), ammonia, base, and organic solvent.

4.2.3.6.3 Allow the reaction to occur for approximately 45 minutes. (Note the presence of “gold beads” produced during the cooking process)

4.2.3.6.4 Add water slowly to react with any unreacted lithium.

4.2.3.6.5 Remove the solvent layer and salt out the methamphetamine using a hydrogen chloride gas generator.

4.3 Other Clandestine Laboratories

4.3.1 DMT

4.3.1.1 Mimosa hostilis root bark or Acacia confuse root bark (can be ordered off the internet)

4.3.1.2 Synthesis

4.3.1.2.1 Put water into a jar, add lye. (Pickle jars are normally used)

4.3.1.2.2 Add bark to the jar, close lid and let sit. (1 hour to 24 hours)

4.3.1.2.3 Add solvent (e.g. Naptha). Gently mix and let layers separate.

4.3.1.2.4 Remove top clear layer and put into a Mason jar.

4.3.1.2.5 Put Mason jar with clear liquid in freezer and leave overnight.

4.3.1.2.6 Filter the solution in the Mason jar and collect the crystalline form.

4.3.2 MDMA

4.3.2.1 Safrole (Sassafras oil, yellow camphor oil) – Natural/Essential Oil Distributor

4.3.2.2 p-Benzoquinone (Quinone, Benzoquinone, Hydroquinone) – Photo Shop or Chemical Supply company

4.3.2.3 Palladium Chloride ($PdCl_2$) – Photo Shop or Chemical Supply company

4.3.2.4 Mercury salt ($HgCl_2$) – Chemical Supply company

4.3.2.5 Ammonium Chloride (NH_4Cl) – Photo Shop or Chemical Supply company

4.3.2.6 Paraformaldehyde – Hardware store (Damp-Rid)

4.3.2.7 Synthesis (see erowid article for synthesis)

4.3.2.7.1 Distillation: of Natural Oil to obtain pure Safrole.

4.3.2.7.2 Rxn: Formaldehyde + Ammonium Chloride -> MethylAmine.HCl

4.3.2.7.3 Rxn: Safrole - (Wacker Oxidation ($PdCl_2$+Benzoquinone)) -> MDP_2P

4.3.2.7.4 Distillation: of Reaction contents to yield pure MDP_2P

4.3.2.7.5 Rxn: MDP_2P -(Al/Hg Amalgam (MeAm.HCl) -> MDMA oil

4.3.2.7.6 Crystallization (MDMA oil + HCl in IPA/Xylene)

4.3.3 Methcathinone

4.3.3.1 (pseudo)ephedrine - extracted from cold and allergy preparations

4.3.3.2 Potassium Permanganate ($KMnO_4$) – oxidizer that treat water. Found at most stores (e.g. Wal-Mart, Lowes Home Improvement, etc).

4.3.3.3 Synthesis

4.3.3.3.1 Extract (pseudo)ephedrine from tablets. Keep (pseudo)ephedrine solution in the fridge.

4.3.3.3.2 Add water and potassium permanganate. Keep solution in the fridge.

4.3.3.3.3 Mix the two solutions together and stir. Place in the fridge overnight.

4.3.3.3.4 Take out of fridge and let sit for 2 hours, filter.

4.3.3.3.5 Add acid and dry.

4.3.4 Tableting

4.3.4.1 Tablet binders (such as cellulose, starch, etc.).

4.3.4.2 Controlled substances in bulk powder form (e.g., alprazolam and fentanyl).

4.3.4.3 Pill press (available online).

4.3.4.4 Synthesis

4.3.4.4.1 Mix tablet binders and controlled substances together.

4.3.4.4.2 Put into pill press and make counterfeit or pharmaceutically marked tablets.

(Note: There are other methods of manufacturing the above listed controlled substances. The ways listed are those that have been observed here in NC.)

4.4 Scene Processing Familiarization

4.4.1 Items are removed from the crime scene.

4.4.1.1 Examples:

- Tablet soakings (usually liquids with solid on the bottom)
- Tablet binders (usually dense white powder in coffee filters)
- Iodine crystals
- Finished product
- Bi-layered liquids
- Liquids
- Sludge material (usually in coffee filters or plastic bottles)

- Coffee filters with methamphetamine (usually residual)
- Precipitated iodine crystals
- Red phosphorus removed from matchbook striker plates
- Grinders containing (pseudo)ephedrine tablets or residue.
- Reaction vessels and/or the residue associated with them can also be seized if necessary
- Any other chemical, glassware, or equipment that is clan lab related must also be removed.

4.4.2 Iodine crystals are placed in a glass vial with a Teflon lid.

4.4.3 Liquids are checked for acid/base characteristics and aqueous/organic characteristics. Volume measurements are also taken, recorded and photographed in order to determine the mass of the liquid sample.

4.4.4 Individual layers of bi-layered liquids are sampled, photographed and volume measurements are taken of each layer in order to determine the mass of the liquid sample.

4.5 Evidence Analysis

4.5.1 Short form Technical Field Assistance (TFA) and Request for Examination of Physical Evidence Form are completed and submitted to the Evidence Control Unit of the State Crime Laboratory.

4.5.2 Long form Technical Field Assistance report is written.

4.5.2.1 All items to be disposed of associated with a clandestine drug lab are listed. Items in this category would include, but are not limited to, the following:

4.5.2.1.1 Labeled solvent bottles (e.g., “Heet” - methanol, starting fluid - ether, camp fuel - hydrocarbons, acetone)

4.5.2.1.2 Labeled acid containers (e.g., muriatic acid - hydrochloric acid, drain cleaner - sulfuric acid)

4.5.2.1.3 Labeled base containers (e.g., “Rooto Drain Opener” – sodium hydroxide)

4.5.2.1.4 Methylsulfonylmethane (MSM) (a cutting agent)

4.5.2.1.5 Butane torches

4.5.2.1.6 Labeled sources of ammonia (e.g., instant cold packs)

4.5.2.1.7 Safety equipment (e.g., gloves, face mask)

4.5.2.2 A qualifying statement about each item and its regulatory status shall also be included.

4.5.2.3 Items seized for Laboratory analysis shall be labeled with the Laboratory item numbers.

4.5.3 Calculations shall be used to identify the approximate Methamphetamine yield of the lab based on the number of (pseudo)ephedrine tablets present.

Molecular weight (pseudo)ephedrine HCl = 201.7 g/mol
Molecular weight methamphetamine HCl = 185.7 g/mol
1 mol (pseudo)ephedrine HCl = 1 mol methamphetamine HCl

$$\frac{\text{201.7 g/mol (pseudo)ephedrine HCl}}{\text{\# g (pseudo)ephedrine HCl at scene}} = \frac{\text{185.7 g/mol methamphetamine HCl}}{\text{X grams of meth HCl}}$$

$$\text{X g of meth HCl} = \frac{(\text{\# g (pseudo)ephedrine HCl at scene})(\text{185.7 g/mol meth HCl})}{\text{201.7 g/mol (pseudo)ephedrine HCl}}$$

The theoretical yield is easily calculated since nearly all of the (pseudo)ephedrine hydrochloride is converted to methamphetamine hydrochloride in a one-to-one molar relationship. This formula calculates a 100 % theoretical yield.

Molecular weight (pseudo)ephedrine sulfate = 428.5 g/mol
Molecular weight methamphetamine HCl = 185.7 g/mol
1 mol (pseudo)ephedrine SO_4 = 2 mol methamphetamine HCl

$$\frac{\text{428.5 g/mol PSE } SO_4}{\text{\# g PSE } SO_4 \text{ at Site}} = \frac{\text{2 mol meth HCl}}{\text{1 mol PSE } SO_4} \times \frac{\text{185.7 g/mol meth HCl}}{\text{X g of meth HCl}}$$

$$\text{Xg of meth HCl} = \frac{(\text{\#g (pseudo) } SO_4 \text{ at site})(\text{2 mol meth HCl/1 mol (PSE } SO_4)(\text{185.7 g/mol Meth HCl})}{\text{428.5g/mol (pseudo)} SO_4}$$

The theoretical yield is calculated based on a one-to-two molar relationship between (pseudo)ephedrine sulfate and methamphetamine hydrochloride. This formula calculates a 100 % theoretical yield.

4.5.4 Evidence Analysis

4.5.4.1 Solids shall be analyzed as typical drug cases along with the identification of precursors (e.g., (pseudo)ephedrine). Residues should also be considered as good evidence for precursor materials (e.g., coffee grinders, pill crushers).

4.5.4.2 Red phosphorus and iodine samples shall be turned over to the Trace Evidence Section for X-Ray Fluorescence analysis (XRF).

4.5.4.3 Bi-layered liquids shall be analyzed. Each layer shall be checked for acid/base characteristics and aqueous/organic characteristics. Each layer shall also be weighed using a graduated cylinder to determine mass of the sample. Chlorinated solvents (tetrachloroethylene) will sink while non-chlorinated solvents (ether) will rise. Care shall be taken when drying basic liquids since methamphetamine base is volatile when heated.

4.5.4.4 Liquids may be analyzed. The liquid shall be checked for acid/base characteristics and aqueous/organic characteristics. The liquid will also be weighed using a graduated cylinder to determine mass of the sample.

4.5.4.5 In most cases, unless otherwise specifically stated, the items that are

considered to be of the best evidentiary value to support manufacturing (ex: liquids from one-pots, sludge) will be analyzed.

4.5.4.6 Disposition of evidence – Items shall be labeled as acids, bases, and solvents in order to aid the hazardous waste contractor in properly disposing of clandestine laboratory chemicals. Finished product, paraphernalia and all other hazardous material, such as liquids, sludge, iodine, and red phosphorous shall be dispositioned as defined in the Drug Chemistry Technical Procedure for Clandestine Laboratory Analysis.

5.0 Scene Safety Familiarization

5.1 Scene Assessment

5.1.1 Types of air monitoring equipment

5.1.1.1 Gastech - useful for flammable atmosphere, toxic atmosphere, oxygen concentration.

5.1.1.2 Draeger - useful to identify class and concentration of toxic chemical present.

5.1.1.3 Four Gas Monitor - useful for phosphine concentration, ammonia concentration, oxygen concentration, flammable/explosive atmospheres.

5.2 Hazard Assessment

5.2.1 Using air monitoring equipment along with necessary protective equipment to determine toxicity or explosiveness of environment.

5.2.2 Photographing and removing hazardous materials/open containers from enclosed areas.

5.2.3 Ventilating the house/building by opening windows/doors and installing intrinsically safe fans.

5.3 Role of Site Safety Officer

5.3.1 Maintain control of the clandestine laboratory site.

5.3.2 Keep Forensic Scientists safe.

5.3.3 Provide equipment for Forensic Scientists.

5.3.4 Forensic Scientists and the Site Safety Officer work together. However, the Site Safety Officer's decisions shall be followed by all Laboratory employees.

5.3.5 Coordinate other responders (e.g., fire, EMS, etc.).

5.3.6 Sampling of evidence for chemical analysis unless a Forensic Scientist is called to assist, then the Forensic Scientist will sample.

5.3.7 Fill out submission form. State location found, acid/base, organic/aqueous and total volume of liquids at the scene.

5.4 Hazards

5.4.1 Red Phosphorus Method

5.4.1.1 Red phosphorus –a solid that converts to white phosphorous (flammable) when it overheats if vessel goes to dryness.

5.4.1.2 Phosphine gas – a poisonous and flammable gas, highly toxic, and may be fatal if absorbed or inhaled. Generated from the conversion of red phosphorus to white phosphorus during cooking or if the reaction vessel becomes dry.

5.4.1.3 Sodium Hydroxide - causes severe eye and skin burns, respiratory irritant, and corrosive. Usually found as "Rooto Drain Opener." This is added during one of the last steps of cooking in order to make the solution basic.

5.4.1.4 Hydrogen chloride gas - highly toxic, causes severe eye and skin burns, may be fatal if inhaled. Created from a combination of sulfuric acid and rock salt, or hydrochloric acid and aluminum foil.

5.4.1.5 Iodine crystals – toxic. Created from combining tincture of iodine with hydrogen peroxide and acid.

5.4.2 Anhydrous Ammonia and One Pot Methods

5.4.2.1 Anhydrous Ammonia - corrosive and toxic gas, irritant to eyes, nose, and throat; can cause frostbite and burn skin; may be fatal if inhaled. Anhydrous ammonia may be found stored in propane cylinders which will be unstable due to incompatibility with this strong base. Visual clue to presence of anhydrous ammonia stored in propane cylinders is a blue discoloration of the copper valve.

5.4.2.2 Sodium and Lithium Metal - water and air reactive; corrosive, flammable solid. These reactive metals must be stored under a solvent to prevent reaction with moisture in the air. Excess sodium/lithium metal can be found in reaction vessels or as strips that have been removed from peeled lithium batteries.

5.4.3 General

5.4.3.1 Strong acids and bases along with their incompatibilities.

5.4.3.2 Chemical solvents.

5.4.3.3 Biological hazards such as syringes and bodily fluids.

5.4.3.4 Booby traps.

6.0 Study Questions

6.1 Define the following terms:

6.1.1 Precursor

6.1.2 Byproduct

6.1.3 Catalyst

6.1.4 Limiting reagent

6.2 Read Chapter 90 of the *NC General Statutes* and list the charges related to methamphetamine possession and manufacture.

6.3 Which precursor chemicals listed in NC G. S. Chapter 90 are commonly found at clan labs?

6.4 What does Chapter 90 of the *NC General Statutes* say about possessing or distributing immediate precursor chemicals?

6.5 List chemicals and starting materials which would indicate the various syntheses of methamphetamine. What byproducts would be expected from these syntheses and why?

6.6 What types of evidence would be important to sample?

6.7 How do clandestine meth cooks learn to cook methamphetamine?

6.8 How does taking the pH at the scene reduce the amount of evidence that you need to take back to the Laboratory for full analysis?

6.9 Explain how a Forensic Scientist shall sample a liquid with two layers.

6.10 Which items of evidence shall be transferred to the Trace Evidence Section?

6.11 Explain several different types of hazards that make clandestine laboratories dangerous.

6.12 What is the purpose of the Methamphetamine Lab Prevention Act of 2005 and Combat Methamphetamine Epidemic Act of 2005?

6.13 Explain smurfing. Explain Smurfing group.

6.14 If a chlorinated solvent was used, in which layer would you expect to find the meth?

6.15 Why is meth converted from the base to the salt form?

6.16 Discuss the importance of working closely with prosecutors and officers to decide the amount of analyses necessary.

6.17 Once analysis is complete, what shall the disposition of evidence be on the Laboratory report?

6.18 How does (Pseudo) ephedrine Sulfate differ from (Pseudo)ephedrine HCl in theoretical yield calculations?

6.19 Name two reasons why propane tanks are present at clandestine laboratories.

6.20 How are cold packs used?

6.21 Typically basic liquids are of the most interest. When would a Forensic Scientist consider sampling an acidic layer for evidence?

6.22 When clandestine laboratory evidence is submitted to the laboratory, what must be put on the submission form?

6.23 What must be present to give the laboratory permission to destroy items of evidence seized from a clandestine laboratory?

6.24 When a liquid is received and analyzed, what should be documented in your case file?

7.0 Practical Exercises

7.1 Observe a forensic scientist analyzing a case that has been submitted to the crime laboratory as a clandestine laboratory submission.

8.0 References

Clandestine Lab Basic Guide. Presented at the 12th Annual Clandestine Laboratory Investigating Chemists Training Seminar: 2002.

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9.0 Records

- Drug Chemistry Training Checklist
- Section Completion Summary

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original Document
12/06/2013	2	Added issuing authority to header; removed SBI-5 reference
10/19/2015	3	**Header –** Revised issuing authority **4.1.2** and **4.1.6** Removed reference to two methods of manufacture **4.2.2.4.1-4.2.2.4.3** Elaborated on the production of anhydrous ammonia **4.2.2.5.6** and **4.2.3.6.5** Updated to include removal of solvent layer **4.2.3.1** Reworded **4.2.3.2** Added example of ammonia source **4.2.3.6.1** Added "Option" to line item **4.2.3.6.3** Inserted note about gold beads **4.3.1.1** Updated list to include additional criteria; removal of duplicate items; formatting adjustments **4.2.3.4** Clarified sampling criteria for bi-layered liquids **4.4.2.1.6** Inserted additional criteria **4.4.3** Added calculation for conversion of (pseudo)ephedrine sulfate to methamphetamine hydrochloride; formatting corrections **4.4.4**, **5.3, and 5.4.2.2** Inserted additional criteria **5.4.1.1** Clarified description of Red Phosphorous **5.4.2.1** Formatting correction **Original 5.5** Removed **Original 6.3** Removed reference to Drug Chemistry Section Training Procedure for Origins and Extractions **7.0** Inserted practical exercise
08/17/2018	4	**4.2** Added the word Methamphetamine. **4.3** Other laboratories added **4.1.10, 4.1.11** – added **4.4.1.1** Added more items that can be removed from crime scene. **4.4.3 and 4.4.4** Added that liquids shall be photographed at the scene. **4.5.2.1.7** Added Safety equipment. **4.5.4.1** Added residue. **4.5.4.3** Added what should be checked when analyzing a bi-layer liquid and how it is to be measured. **4.5.4.4** Added Liquids **5.3.6** Took out assist.

		5.3.7 Added filled out submission form. **6.22** Added **6.23** Added **6.24** Added **8.0** Added references