

## Draft Recommendations on the Analysis of Clandestine Drug Laboratory Evidence

These recommendations should be used in conjunction with the general requirements for the analysis of seized drugs. This document provides guidance on the chemical analysis of items and samples related to suspected clandestine drug laboratories. It does not address scene attendance or scene processing. This is an upper level recommendation and should not be considered a step-by-step guidance document.

### **1 Introduction**

- 1.1 SWGDRUG considers an understanding of clandestine laboratory analysis and synthesis to be fundamental to the interpretation and reporting of results. This understanding assures that results and conclusions from methods are reliable and analytical schemes are fit for purpose.
- 1.2 The qualitative and quantitative analyses of clandestine laboratory evidence can require different approaches relative to routine seized drug analyses. Analysts shall understand the limitations of the qualitative and quantitative conclusions.
- 1.3 Laboratory management shall ensure that clandestine laboratory synthesis and analysis training be provided through procedures, documentation, scientific literature, and practical experience.
- 1.4 Chemical safety and hygiene plans shall address and mitigate hazards associated with clandestine laboratory evidence.
- 1.5 Laboratory management shall consider customer / local requirements which influence the application of these recommendations.

### **2 Safety**

- 2.1 Many of the items seized at a clandestine laboratory are intrinsically dangerous and of unknown composition. For many components found at clandestine laboratories, the associated hazards are not apparent.
- 2.2 A safety program addressing storage and analysis of items from suspected clandestine laboratories shall be in place that includes:
  - training in safety procedures and in the use of safety and protective equipment for all staff responsible for handling items
  - listings of the hazards (e.g. MSDS) associated with components commonly found at clandestine laboratory sites
  - evidence receipt, storage and disposal requirements designed to mitigate expected dangers (e.g. limited sample size, proper packaging of reactive materials, use of absorbents)

- personal protective equipment such as safety glasses, chemical resistant gloves, laboratory coats, respirators, face masks, and air monitors
  - accident prevention, emergency response procedures, and reporting protocols
- 2.3 Facilities and procedures shall be in place so that all items are safely treated as potentially hazardous materials. Examples include:
- ventilation equipment (e.g. fume hoods) to prevent exposure to harmful fumes and vapors
  - maintenance of a clean, uncluttered workspace
  - emergency washing facilities, particularly for eyes
  - adequate evidence storage facilities (e.g. ventilation, fire protection)
- 2.4 Analysts shall be aware of the unique hazards associated with clandestine laboratories samples. Examples are:
- extracting from strong acids and bases (e.g. hydriodic acid, sodium hydroxide)
  - handling fuming acids and bases (e.g. hydrochloric acid, ammonia)
  - poisonous gasses (e.g. phosphine, chlorine, hydrogen sulfide) and their potential release from evidence during analysis
  - poisonous, carcinogenic, and mutagenic materials (e.g. mercuric chloride, chloroform, potassium cyanide)
  - reactive and air sensitive materials (e.g. white phosphorus, lithium)
  - potential testing incompatibilities (e.g. phosphorus with Raman, color test reagents with cyanide salts, exothermic reactions)
  - radioactive materials (e.g. thorium)
  - volatile and flammable solvents (e.g. acetone, diethyl ether, methylated spirits)
- 2.5 Staff responsible for on-site attendance at suspected clandestine laboratory scenes will require additional training, the details of which are beyond the scope of this recommendation.

### **3 Sample Selection for Analysis**

- 3.1 The primary purpose for analysis is to prove or disprove allegations of clandestine drug syntheses. Accordingly, analysts must test items which relate to the manufacturing process.
- 3.2 Not all items seized at a clandestine laboratory site need be analyzed. It is recommended that information be shared between the analyst and on-scene personnel to aid in sample selection.
- 3.3 Items should be selected for analysis, based on jurisdictional requirements, and which are likely to contain:
- finished product

- intermediates
- precursors
- key reagents
- reaction mixtures

3.4 Some of the following types of samples may be analyzed as they can assist in determining the chemical reaction(s) undertaken and the scope of the clandestine laboratory:

- materials that appear to be waste
- unlabeled materials that appear to be contaminated solvents, acids, or bases
- samples from contaminated equipment

3.5 Items that are readily obtained from local retail stores and are sold from reputable manufacturers/distributors may not need to be identified, particularly if collected from sealed and labeled containers. These include:

- solvents (e.g. toluene, mineral spirits)
- acids (e.g. hydrochloric acid, sulfuric acid)
- bases (e.g. sodium hydroxide, ammonia water)

## 4 Analysis

4.1 Chemicals whose presence are reported or contribute to formulating reported conclusions in the case should be identified using an analytical scheme with discriminating power comparable to those outlined by the guidelines for the analysis of seized drugs.

4.2 The identification of many organic compounds should follow the guidelines for the analysis of seized drugs (see [Part III B](#)).

4.3 The identification of specific commercial products (e.g. camp fuels containing petroleum distillates) involves specific testing beyond the scope of this recommendation.

4.4 The discriminating power of analytical techniques for the identification of inorganic materials depends on the particular analyte to a greater extent than organic chemicals. In each case the analytical scheme shall:

- Have combined sufficient discriminating power to identify the material to the exclusion of others
- Utilize two or more techniques generally falling under different analytical groups described below, and
- Identify both the cation and anion in ionic materials (e.g. salts)

4.5 The following analytical groups are listed in no particular order and are not comprehensive. Analytical techniques must be selected which provide sufficient discriminating power for each analyte. Some techniques may not be useful for particular analytes and each must be individually evaluated to determine suitability.

4.5.1 **Analytical Group 1: Elemental Analysis Techniques** – these techniques provide positive identification of elements present in the sample but typically require additional tests to distinguish forms.

- X-Ray Fluorescence (XRF)
- Energy Dispersive X-Ray Detectors for Scanning Electron Microscopes (SEM-EDX)
- Atomic Absorption Spectroscopy
- Atomic Emission Spectroscopy and Flame Tests (an attached spectrometer significantly increases the discriminating power relative to flame tests)
- Mass Spectrometry (utilizing Inductively Coupled Plasma sources or for elements with unique isotopic abundance patterns)

4.5.2 **Analytical Group 2: Chemical Bonding Techniques** – these techniques may have high discriminating power for polyatomic ions and small molecules such as ammonia.

- Nuclear Magnetic Resonance (NMR)
- Infrared Spectroscopy (IR and FTIR)
- Raman Spectroscopy
- UV-Vis & Fluorescence Spectroscopy
- Mass Spectrometry

4.5.3 **Analytical Group 3: Separation Techniques** – these techniques can be very valuable for mixtures and for distinguishing different forms of an element (e.g. phosphate and phosphite).

- Capillary Electrophoresis
- Ion Chromatography
- Liquid Chromatography
- Gas Chromatography
- Planar Chromatography (e.g. Thin Layer and Paper)

4.5.4 **Analytical Group 4: Chemical Properties** – These techniques involve observations of chemical changes. The discriminating power of these

techniques can often be increased by utilizing several in series or combination.

- Solubility and miscibility tests
- Spot and precipitation tests
- Microcrystalline tests
- pH (of liquids or vapors)
- radioactive decay
- flammability
- reactivity with water, air, or other materials

4.5.5 **Analytical Group 5: Physical Properties** – These techniques involve observations of physical properties and changes. The discriminating power of these techniques depends on the accuracy and precision of the measuring device.

- Physical state or states
- Phase transitions including melting points, boiling points, sublimation temperature and vapor pressure
- Density (relative density and density of mixtures have reduced discriminating power)
- Color
- Viscosity and surface tension
- Refractive index
- Crystal forms measured with polarized light microscopy or x-ray diffraction techniques

4.6 An analytical scheme with a lower discriminating power may be appropriate if only limited or qualified conclusions are required (e.g. basic aqueous layer, non-polar organic solvent, a material containing the element phosphorus).

4.7 Analytical reference materials may not be available for the analysis of intermediates and byproducts. In these cases, samples taken from a test reaction in conjunction with suitable reference literature may be used for comparison purposes. Quantitative analysis of samples may be required by local jurisdiction to address statutory requirements.

4.8 Quantitative measurements of clandestine laboratory samples have an accuracy which is dependent on sampling and, if a liquid, on volume calculations. Accordingly, these measurements and calculations may be based on estimates. Under these conditions, a precise statement of measurement uncertainty for these calculations is often not possible and may best be conveyed by using a qualifier statement on the report. See Section 6.8 for examples.

## 5 Yield and capacity calculations

- 5.1 Yield and capacity calculations can be achieved from a number of approaches and shall be based on relevant case information, suitable literature, laboratory and jurisdictional requirements.
- 5.2 Reported yields and capacities shall be based upon information documented in the laboratory case file.
- 5.3 Potential yields can be calculated as theoretical or practical yields.
- 5.3.1 Theoretical yields are calculated based on the amount of known chemical, the stoichiometry of the reaction used in the clandestine laboratory and the molecular weights of the starting material(s) and product. Theoretical yields are not achievable in practice. As such, SWGDRUG recommends theoretical yields not be reported on their own as they can be misleading.
- 5.3.2 Practical yields are calculated based upon published data or laboratory experimentation.
- 5.4 In calculating practical yields and capacities in clandestine laboratories, many different sources of information can be used. Each case is different and will have a different set of evidence from which to draw information, including, but not limited to:
- amounts of finished products, precursors, or essential chemicals present
  - amount of waste present
  - size of reaction vessels and equipment
  - volume of containers
  - type / quantity of equipment and chemicals used
  - state of equipment and premises (e.g. cleanliness of site and equipment)
  - the apparent skill and laboratory practice of the operator
  - the procedures (i.e. recipe) followed by the operator
- 5.5 In addition to observations about the clandestine laboratory site itself, other pieces of evidence can lead to an understanding of yields and capacities, including, but not limited to:
- length of time the laboratory has been in operation
  - intercepted conversations
  - statements made by the clandestine laboratory operator during an interview/interrogation
  - documents describing purchases of equipment, precursors, or reagents
  - photographs of the clan lab site and other related areas.
  - records kept by the clandestine laboratory operator (e.g. seized recipes or records of previously manufactured quantities)
- 5.6 Whenever possible, use two or more pieces of information (see Section 5.4 and 5.5) to calculate past or future production. All of the available information needs

to be taken into consideration before formulating a conclusion. Any assumptions or conditions should be stated.

- 5.7 When calculating capacity, care should be taken not to combine values which may have been obtained from the same source (e.g. empty blister packs and tablet waste).

## **6 Reports and Conclusions**

- 6.1 Written and verbal conclusions shall be based upon documented information and with clearly stated assumptions and conditions.
- 6.2 There are many facets to a clandestine laboratory investigation, such as:
- the illicit drug being made
  - the synthetic route being utilized
  - the type of equipment found at the site
  - the past/potential production at the site
  - the final form of the illicit drug
  - the batch size at the site
  - whether a tableting / encapsulating operation was underway or possible
- 6.3 Factors to consider in determining what to report include, but are not limited to:
- jurisdictional requirements
  - governing body (agency) requirements
  - customer requests
  - potential exculpatory information
  - samples / analytes which represent the multiple stages in a reaction process
- 6.4 Conclusions shall be made based on the principle of reasonable scientific certainty and shall be clearly stated. Each case shall be based on its own merits.
- 6.5 Written conclusions are to be subject to technical review. The individual reviewing the conclusions must be knowledgeable in the investigation and analysis of clandestine laboratory seizures in order to evaluate the conclusions drawn.
- 6.6 Verbal conclusions shall be subject to technical review whenever possible. It is recognized that responding to queries in court or investigative requirements may present an exception. Laboratories should have policies in place outlining when these exceptions are appropriate.
- 6.7 Where an analytical scheme with a lower discriminating power has been utilized, conclusions shall be qualified or limited in scope (e.g. basic aqueous layer, non-polar organic solvent, a material containing the element phosphorus).

- 6.8 Qualifiers for quantitative analysis and yield/capacity determinations may be necessary to ensure the accuracy of the report (e.g. approximately, not to exceed, no less than).

## 7 Training

- 7.1 Analysis and interpretation of a clandestine laboratory case requires specialized skills. The main objective of clandestine laboratory training programs should be to provide new analysts with a sound education in the fundamental areas of clandestine laboratory evidence analysis. These guidelines assume the student is qualified as a seized drug analyst.
- 7.2 Analysts shall receive additional training which will enable them to safely perform the analysis of clandestine drug laboratory samples.
- 7.3 Analysts shall receive training which will enable them to assist in investigation of clandestine drug syntheses. Critical aspects of this element include:
- advanced techniques for performing chemical separations
  - production estimates
  - study of pertinent drug syntheses by various routes
  - training on intermediates and route specific by-products
  - knowledge of common and alternative sources of chemicals
  - training in inorganic chemistry, analysis techniques, and interpretation
  - common terminology used in organic chemistry and synthesis
  - application of critical thinking and problem solving skills to the evaluation of all case information (e.g. officer and scene reports, recipes, chemical data)
  - the ability to recognize when additional information is required, identify sources for that information (journals, monographs, underground references), critically evaluate the reference and apply that knowledge to case information
- 7.4 Analysts shall receive training which will enable them to interact with the legal community and understand the needs of the respective jurisdictional requirements. Critical aspects of legal training include statutes covering the manufacturing, production, distribution, trafficking of controlled substances. Courtroom testimony training shall include training specific to these legal requirements, including capacity estimates and yield determinations.
- 7.5 Analysts shall stay current in the field of clandestine drug manufacturing and clandestine laboratory investigations. Examples of this element include:
- joining regional, national, and international scientific organizations
  - attending conferences specializing in clandestine drug manufacture
  - receiving training by qualified instructors covering current trends and reviews
  - reading pertinent scientific literature

## 8 Definitions

### **byproduct**

a secondary or incidental product of a manufacturing process.

[Collins English Dictionary - Complete & Unabridged 10th Edition]

### **capacity**

the ability to do or produce.

[Collins English Dictionary - Complete & Unabridged 10th Edition]

### **catalyst**

a substance whose presence initiates or changes the rate of a chemical reaction, but does not itself enter into the reaction.

[ASTM-D6161]

### **clandestine**

secret and concealed, often for illicit reasons.

[Collins English Dictionary - Complete & Unabridged 10th Edition]

### **conspiracy**

an agreement between two or more people to commit an act prohibited by law or to commit a lawful act by means prohibited by law.

[Merriam-Webster's Dictionary of Law, © 1996 Merriam-Webster, Inc.]

### **derivative**

a compound that is formed from, or can be regarded as formed from, a structurally related compound

[Collins English Dictionary - Complete & Unabridged 10th Edition]

### **intermediate**

substance that is manufactured for and consumed in or used for chemical processing to be transformed into another substance.

[ASTM- F2725]

### **finished product**

a manufactured "product", ready for use.

[ASTM-F1709]

### **Precursor**

a chemical that is transformed into another compound, as in the course of a chemical reaction, and therefore precedes that compound in the synthetic pathway

[Random House Dictionary, © Random House, Inc. 2010]

### **reagent**

a chemical used to react with another chemical, often to confirm or deny the presence of the second chemical.

[ASTM-E1605]

**yield**

the quantity of product formed by the interaction of two or more substances, generally expressed as a percentage of the quantity obtained to that theoretically obtainable.

[Random House Dictionary, © Random House, Inc. 2010]

**yield, expected**

the quantity of material or the percentage of theoretical yield anticipated at any appropriate phase of production based on previous laboratory, pilot scale, or manufacturing data.

[ASTM-E2363]

**yield, theoretical**

the quantity that would be produced at any appropriate phase of production based upon the quantity of material to be used, in the absence of any loss or error in actual production.

[ASTM-E2363]