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# Standard Guide for Analysis of Clandestine Drug Laboratory Evidence[1]

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## 1. Scope

1.1 This guide is intended to be used in conjunction with the general requirements for the analysis of seized drugs (Practices E2326, E2327, E2329, and E2549; Guides E2548 and E2329). This guide 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 document provides general guidance for the analysis of clandestine laboratory evidence and is not a substitute for detailed and validated laboratory policies and technical procedures.

1.2 This guide does not replace knowledge, skill, ability, experience, education, or training and should be used in conjunction with professional judgment.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D6161 Terminology Used for Microfiltration, Ultrafiltration, Nanofiltration and Reverse Osmosis Membrane Processes

E1605 Terminology Relating to Lead in Buildings

E2326 Practice for Education and Training of Seized-Drug Analysts

E2327 Practice for Quality Assurance of Laboratories Performing Seized-Drug Analysis

E2329 Practice for Identification of Seized Drugs

E2363 Terminology Relating to Process Analytical Technology in the Pharmaceutical Industry

E2548 Guide for Sampling Seized Drugs for Qualitative and Quantitative Analysis

E2549 Practice for Validation of Seized-Drug Analytical Methods

F2725 Guide for European Union's Registration, Evaluation, and Authorization of Chemicals (REACH) Supply Chain Information Exchange

## 3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *capacity*—the amount of finished product that could be produced, either in one batch or over a defined period of time, and given a set list of variables. **SWGDRUG**[3]

3.1.2 *catalyst*—a substance whose presence initiates or changes the rate of a chemical reaction, but does not itself enter into the reaction. **D6161**

3.1.3 *finished product*—a manufactured product ready for use. **SWGDRUG**[3]

3.1.4 *intermediate*—ubstance that is manufactured for and consumed in or used for chemical processing to be transformed into another substance. **F2725**

3.1.5 *reagent*—a chemical used to react with another chemical, often to confirm or deny the presence of the second chemical. **E1605**

3.1.6 *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. **E2363**

3.1.7 *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. **E2363**

## 4. Significance and Use

4.1 An understanding of clandestine laboratory synthetic routes and the techniques used in the analysis of related samples is considered 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.

4.2 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 procedures used in their qualitative and quantitative analyses. These include such factors as method selectivity, uncertainty, and the basis for inferences from a sample(s) to a population.

4.3 Laboratory management shall ensure that clandestine laboratory synthesis and analysis training be provided through

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from the Scientific Working Group for the Analysis of Seized Drugs, http://www.swgdrug.org.

relevant procedures, literature, and practical experience. Practical experience typically includes production, sampling and analysis of clandestine laboratory training samples.

4.4 Laboratory management shall ensure that chemical safety and hygiene plans address and mitigate hazards associated with clandestine laboratory evidence.

4.5 Laboratory management shall consider customer/local requirements which influence the application of these recommendations.

## 5. Safety

5.1 This guide does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this guide to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

5.2 Many items seized at clandestine laboratories may be intrinsically dangerous. These may include items of unknown composition and chemicals that have not been fully characterized and whose specific hazards are not known. Therefore, caution must be exercised and routine safety protocols may not be sufficient.

5.3 The following are required in addition to the routine laboratory safety program in place for the analysis of seized drugs (see Practice E2327, Health and Safety):

5.3.1 Safety procedures and the use of safety and protective equipment for all staff responsible for handling items;

5.3.2 Protective breathing equipment;

5.3.3 Listings of the relevant hazards (for example, MSDS) associated with components commonly found at clandestine laboratory sites and knowing what they mean; and

5.3.4 Accident prevention, emergency response procedures, and incident reporting protocols.

5.4 The handling, analysis, and storage of items seized from clandestine laboratories require additional procedures, facilities and equipment (see Practice E2327, Physical Plant). Examples are:

5.4.1 Specialized ventilation equipment (for example, fume hoods) to prevent exposure to harmful fumes and vapors;

5.4.2 Provision of personal protective equipment such as safety glasses, chemical resistant gloves, laboratory coats, respirators, face masks, and air monitors;

5.4.3 Maintenance of a clean, uncluttered workspace;

5.4.4 Specialized emergency equipment stations;

5.4.5 Chemical disposal and destruction facilities and procedures; and

5.4.6 Specialized evidence receipt, storage and disposal requirements designed to mitigate expected dangers (for example, limited sample size, proper packaging of reactive materials, use of absorbents, properly ventilated storage).

5.5 Analysts shall be aware of the hazards associated with clandestine laboratories samples. Examples are:

5.5.1 Extracting from strong acids and bases (for example, hydriodic acid, sodium hydroxide);

5.5.2 Handling fuming acids and bases (for example, hydrochloric acid, ammonia);

5.5.3 Poisonous gases (for example, phosphine, chlorine, hydrogen sulfide) and their potential release from evidence during analysis;

5.5.4 Poisonous, carcinogenic, and mutagenic materials (for example, mercuric chloride, chloroform, potassium cyanide);

5.5.5 Reactive and air sensitive materials (for example, white phosphorus, lithium);

5.5.6 Potential testing incompatibilities (for example, phosphorus with Raman, color test reagents with cyanide salts, exothermic reactions);

5.5.7 Radioactive materials (for example, thorium); and

5.5.8 Volatile and flammable solvents (for example, acetone, diethyl ether, methylated spirits).

## 6. Sample Section for Analysis

6.1 The primary purpose of analysis is to prove or disprove allegations of clandestine drug syntheses. Accordingly, analysts must select items which relate to the manufacturing process.

6.2 Not all items seized at a clandestine laboratory site may need to be analyzed. It is recommended that information be shared between the analyst and on-scene personnel to aid in sample selection.

6.3 Items should be selected for analysis, based on jurisdictional requirements, and which are likely to contain:

6.3.1 Finished product,

6.3.2 Intermediates,

6.3.3 Precursors,

6.3.4 Key reagents, and

6.3.5 Reaction mixtures.

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

6.4.1 Materials that appear to be waste;

6.4.2 Unlabeled materials that appear to be contaminated solvents, acids, or bases; and

6.4.3 Samples from contaminated equipment.

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

6.5.1 Solvents (for example, toluene, mineral spirits),

6.5.2 Acids (for example, hydrochloric acid, sulfuric acid), and

6.5.3 Bases (for example, sodium hydroxide, ammonia water).

## 7. Analysis

7.1 Substances whose presence are reported or contribute to formulating reported conclusions shall be identified with an adequate analytical scheme.

7.2 Where possible, the identification of organic compounds shall follow the guidelines for the analysis of seized drugs (see Practice E2329).

7.3 The discriminating power of analytical techniques for the identification of inorganic materials depends on the particular analyte. In each case the analytical scheme shall:

7.3.1 Have sufficient discriminating power to identify the material to the exclusion of others (for example, identification of both the cation and anion in salts), and

7.3.2 Utilize two or more techniques, preferably from different analytical groups described below.

7.4 The following list of analytical groups and techniques are in no particular order and are not exhaustive. 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 evaluated to determine suitability.

7.4.1 *Analytical Group 1: Elemental Analysis Techniques*—These techniques may provide positive results for elements present in a sample but typically require additional tests to distinguish forms (for example, oxidation state).

7.4.1.1 Atomic absorption spectroscopy,

7.4.1.2 Atomic emission spectroscopy and flame tests (an attached spectrometer significantly increases the discriminating power relative to flame tests),

7.4.1.3 Energy dispersive X-ray detectors for scanning electron microscopes,

7.4.1.4 Mass spectrometry (utilizing inductively coupled plasma sources or for elements with unique isotopic abundance patterns), and

7.4.1.5 X-ray fluorescence.

7.4.2 *Analytical Group 2: Structural Elucidation Techniques*—These techniques may have high discriminating power for polyatomic analytes.

7.4.2.1 Infrared spectroscopy,

7.4.2.2 Mass spectrometry,

7.4.2.3 Nuclear magnetic resonance,

7.4.2.4 Raman spectroscopy,

7.4.2.5 UV-vis and fluorescence spectroscopy, and

7.4.2.6 X-ray diffractometry.

7.4.3 *Analytical Group 3: Separation Techniques*—These techniques can be valuable for mixtures and for distinguishing different forms of an element (for example, phosphate and phosphite).

7.4.3.1 Capillary electrophoresis,

7.4.3.2 Gas chromatography,

7.4.3.3 Ion Chromatography

7.4.3.4 Liquid chromatography, and

7.4.3.5 Thin layer chromatography.

7.4.4 *Analytical Group 4: Chemical Properties*—These techniques involve observations of chemical changes. Utilizing several of these techniques, in series or combination, can often increase discriminating power.

7.4.4.1 Flammability;

7.4.4.2 Microcrystalline tests;

7.4.4.3 pH (of liquids or vapors);

7.4.4.4 Radioactive decay;

7.4.4.5 Reactivity with water, air, or other materials;

7.4.4.6 Solubility and miscibility tests; and

7.4.4.7 Spot and precipitation tests.

7.4.5 *Analytical Group 5: Physical Properties*—These techniques involve observations of physical properties. The discriminating power of these techniques depends on the measuring device.

7.4.5.1 Color;

7.4.5.2 Crystal forms measured with polarized light microscopy;

7.4.5.3 Density (relative density and density of mixtures have reduced discriminating power);

7.4.5.4 Phase transitions including melting points, boiling points, sublimation temperature, and vapor pressure;

7.4.5.5 Physical state or states;

7.4.5.6 Refractive index; and

7.4.5.7 Viscosity and surface tension.

7.5 If limited or qualified conclusions are sufficient (for example, basic aqueous layer, non-polar organic solvent, a material containing the element phosphorus), tests of limited discriminating power may be utilized within an analytical scheme.

7.6 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.

7.7 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 rigorous calculation of measurement uncertainty is often not possible or necessary and the uncertainty may best be conveyed by using a qualifier statement on the report (for example, approximately, not to exceed, no less than).

## 8. Yield and Capacity Calculations

8.1 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.

8.2 Reported yields and capacities shall be based upon information documented in the laboratory case file.

8.3 Calculated yields can be expressed as theoretical or expected.

8.3.1 It is recommended that reported yields be accompanied with an explanation clarifying the limitations or considerations.

8.3.1.1 Theoretical yields are calculated based on the amount of known chemical, the stoichiometry of the reaction used in the clandestine laboratory and the product. Theoretical yields are not achievable in practice and their reporting can be misinterpreted.

8.3.1.2 Expected yields are calculated based upon published data, experience, or practical experimentation. Expected yields can be highly variable based upon the factors listed below.

8.4 In calculating expected 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:

8.4.1 Amounts of finished products, precursors, or essential chemicals present;

8.4.2 Amount of waste present;
8.4.3 Size of reaction vessels and equipment;
8.4.4 Volume and quantity of containers;
8.4.5 Type/quantity of equipment and chemicals used;
8.4.6 State of equipment and premises (for example, cleanliness of site and equipment);
8.4.7 The apparent skill and laboratory practice of the operator; and
8.4.8 The procedures (that is, recipe) followed by the operator.

8.5 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:
8.5.1 Length of time the laboratory has been in operation;
8.5.2 Intercepted conversations;
8.5.3 Statements made by the clandestine laboratory operator during an interview/interrogation;
8.5.4 Documents describing purchases of equipment, precursors, or reagents;
8.5.5 Photographs of the clandestine laboratory site and other related areas; and
8.5.6 Records kept by the clandestine laboratory operator (for example, seized recipes or records of previously manufactured quantities).

8.6 When calculating capacity, ensure that the values were not obtained from the same source (for example, empty blister packs and tablet waste).

## 9. Reports and Conclusions

9.1 Communications and reports, either written or verbal, shall be based upon all of the available and relevant information and with clearly stated assumptions and conditions.

9.2 There are many facets to a clandestine laboratory investigation, such as:
9.2.1 The illicit drug being made,
9.2.2 The synthetic route being utilized,
9.2.3 The type of equipment found at the site,
9.2.4 The past/potential production at the site,
9.2.5 The final form of the illicit drug,
9.2.6 The batch size at the site, and
9.2.7 Whether a tableting/encapsulating operation was present.

9.3 Factors to consider in determining what to report include, but are not limited to:
9.3.1 Jurisdictional requirements,
9.3.2 Governing body (agency) requirements,
9.3.3 Customer requests,
9.3.4 Potential exculpatory information, and
9.3.5 Samples/analytes which represent the multiple stages in a reaction process.

9.4 Laboratories should have documented policies establishing protocols for reviewing verbal information and conclusions should be subject to technical review whenever possible. It is acknowledged that responding to queries in court or investigative needs may present an exception.

9.5 When technical reviews are conducted, the individual reviewing the conclusions must be knowledgeable in the processing, analysis, and reporting of clandestine laboratory seizures.

## 10. Training

10.1 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.

10.2 Analysts shall receive training which will enable them to safely perform the analysis of clandestine drug laboratory samples.

10.3 Analysts shall receive training which will enable them to assist in investigation of clandestine drug syntheses. Aspects of this training may include:
10.3.1 Chemical separation techniques (for example, acid/base extractions, ion pair extractions, precipitation);
10.3.2 Production estimates;
10.3.3 Study of pertinent drug syntheses by various routes;
10.3.4 Training on intermediates and route specific by-products;
10.3.5 Knowledge of common and alternative sources of chemicals;
10.3.6 Training in inorganic chemistry, analysis techniques, and interpretation;
10.3.7 Common terminology used in organic chemistry and synthesis; and
10.3.8 Application of critical thinking and problem solving skills to the evaluation of all case information (for example, officer and scene reports, recipes, chemical data).

## 11. Keywords

11.1 analysis of clandestine laboratory evidence; chemical analysis; chemical properties; clandestine drug laboratories; elemental analysis; physical properties; separation; structural elucidation