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Standard Guide for Microcrystal Testing in Forensic Analysis of Methamphetamine and Amphetamine[1]

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INTRODUCTION

Microcrystal tests are primarily chemical-precipitation tests in which a light microscope is used to observe and distinguish the different types of crystals formed. These tests require skill and expertise on the part of the analyst that can be gained adequately only through appropriate training and experience in their use. These tests should not be attempted by those who are unfamiliar with them for use in the analysis of methamphetamine or amphetamine.

1. Scope

1.1 This guide describes some standard procedures applicable to the analysis of methamphetamine and amphetamine using microcrystal tests (**1-6**).[2]

1.2 These procedures are applicable to methamphetamine and amphetamine, which are present in solid dosage form or an injectable liquid form. These procedures are not typically applicable to the analysis of methamphetamine and amphetamine in biological samples.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard cannot replace knowledge, skill, or ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.*

1.5 *This standard 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 standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E1459 Guide for Physical Evidence Labeling and Related Documentation

E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory

E1732 Terminology Relating to Forensic Science

E2329 Practice for Identification of Seized Drugs

E2548 Guide for Sampling Seized Drugs for Qualitative and Quantitative Analysis

3. Terminology

3.1 For definitions of terms used in this standard, refer to Terminology E1732.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aggregation, n*—the collecting of units or parts into a mass or whole.

3.2.2 *birefringence, n*—property of some crystals, having more than one refraction index; this property will result in interference colors, which are viewed through a polarized light microscope.

3.2.3 *blades, n*—broad, flat, elongated crystals.

3.2.4 *grains, n*—thick tablets having nearly equal width, breadth and thickness.

3.2.5 *habit, n*—the external morphology of the crystal.

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[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[3] 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.

3.2.6 *microdrop, n*—a small drop of liquid that would fit on the end of a standard size, flattened toothpick; the approximate volume of this drop would be 10 to 25 µL.

3.2.7 *needles (acicular), n*—long, thin crystals with pointed ends.

3.2.8 *plates, n*—blades with nearly equal length and breadth and of a thickness substantially less than the width.

3.2.9 *rods, n*—long, thin crystals with squared off ends.

3.2.10 *tablets, n*—plates with appreciable thickness but less than the length or breadth.

4. Summary of the Technique

4.1 A small sample of the material containing the suspected methamphetamine or amphetamine is dissolved in an appropriate acid and the appropriate precipitating reagent is added. The crystals that are formed are observed and distinguished utilizing a light microscope.

4.2 If the proper formation of crystals is inhibited by the presence of diluents, a purification of the sample based on the volatility of methamphetamine and amphetamine may be performed.

5. Significance and Use

5.1 This technique produces a chemical-precipitation reaction between methamphetamine or amphetamine and the precipitating reagent. The habit and the aggregation of the crystals formed may be used to distinguish methamphetamine and amphetamine from other drugs.

6. Interferences

6.1 *Diluents/Adulterants*—Diluents/adulterants present in combination with methamphetamine or amphetamine in the sample to be tested may inhibit crystal formation or result in crystals that are distorted or otherwise rendered unidentifiable. In these instances, it will be necessary to separate the methamphetamine or amphetamine from the diluents or to use other testing methods to analyze the methamphetamine or amphetamine.

7. Apparatus

7.1 A standard light microscope capable of varying magnifications including 100× is needed for viewing the crystals. A polarized light attachment is not essential, but is desirable, because the heavy metal crystals of methamphetamine and amphetamine are birefringent.

8. Reagents and Materials

8.1 *10 % Solution of Hydrochloric Acid.*

8.2 *Concentrated Phosphoric Acid.*

8.3 *1.0 N to 10.0 N Sodium Hydroxide.*

8.4 *Gold Chloride ($HAuCI_4$) Solution,* approximately 5 %, in reagent grade water. Gold chloride in phosphoric acid also is suitable.

8.5 *Platinum Chloride (H_2 $PtC1_6$) Solution,* approximately 5 %, in reagent grade water. Platinum chloride in phosphoric acid also is suitable.

8.6 *Amphetamine Standard.*

8.7 *Methamphetamine Standard.*

9. Sampling, Test Specimens, and Text Units—

9.1 The general handling and tracking of samples should meet or exceed the requirements of Practice E1492 and Guides E1459 and E2548.

10. Calibration and Standardization

10.1 The reagents utilized for these microcrystal tests are to be tested for reliability using amphetamine and methamphetamine standards and negative controls following the prescribed procedure. Only when it is determined that the reagents are producing the expected response may the reagents be used in the testing procedure.

11. Procedure

11.1 *Gold Chloride:*

11.1.1 Place a small sample, a few particles of powder, less than 1 mg, of the suspected methamphetamine or amphetamine on a microscope slide.

11.1.2 Dissolve the sample in a few microdrops of 10 % hydrochloride acid or concentrated phosphoric acid.

NOTE 1—The crystals tend to precipitate faster from the phosphoric acid. There also tends to be less interference when using the concentrated phosphoric acid.

11.1.3 Add a few microdrops of 5 % gold chloride to the edge of the acid solution on the microscope slide.

11.1.4 Observe the formation of the crystals using a properly aligned and adjusted light microscope. This observation can be done between crossed polars if desired. If crossed polars are to be used, orient the polarizer in the east-west direction and the analyzer in the north-south direction, verified by a black background.

11.1.5 The crystals formed will depend on the drug present, as well as, the optical isomer present for the drug. The formations that can be expected for methamphetamine and amphetamine are as follows.

11.1.5.1 *d- or l-Amphetamine,* produces long yellow rods or blades.

11.1.5.2 *d,l-Amphetamine,* produces irregular blades, which have serrated edges and often will grow in groups of three or more.

11.1.5.3 *d- or l-Methamphetamine,* produces long blades and jointed crystals. If these crystals are broken gently by scratching with a probe, they will break apart into smaller rods, which have an appearance of clothespins, which are open on one end and closed on the other. These smaller rods are best viewed between crossed polars.

11.1.5.4 *d,l-Methamphetamine,* produces long blades and jointed crystals similar to those observed for the *d-* or *l-* form. If these crystals are broken gently by scratching with a probe, they will break apart into smaller rods, which are open on both ends, having an appearance of the letter "X."

11.1.6 If a dense cloud of precipitate is formed upon the addition of the precipitating agent, the crystals may not be readily visible. It may be necessary to repeat the test reducing

the concentration of suspected methamphetamine or amphetamine in the acid solution. This is done by either decreasing the sample size or increasing the volume of solvent.

11.1.7 The following procedure to separate the methamphetamine or amphetamine from diluents may be used.

11.1.7.1 Place a small amount of the sample into a spot well. Sample size should be approximately 2 to 3 mg.

11.1.7.2 Add a small volume of 1.0 N to 10.0 N sodium hydroxide, such that the powder is just covered.

11.1.7.3 Place a microdrop of concentrated phosphoric acid, or gold chloride reagent containing phosphoric acid, on a slide and invert the slide over the sample such that the microdrop is over the spot well containing the sample.

11.1.7.4 Any amine present, including methamphetamine and amphetamine, will be converted to the free base form. Many free bases are volatile at room temperature, and as they leave the solution, they will collect in the microdrop of phosphoric acid.

11.1.7.5 After a period of time has elapsed, approximately 1 to 3 min, remove the slide and observe the crystals formed directly if gold chloride is used in the hanging drop, or perform the testing procedure described in 11.1.3-11.1.5 on the microdrop of phosphoric acid. The appropriate period of time is variable and will depend on the concentration of methamphetamine or amphetamine in the sample, the diluents present, as well as the room temperature. It may be necessary to repeat the test for a shorter or longer duration or utilize more sample if adequate recovery of amines is not accomplished during initial attempts.

11.2 *Platinum Chloride:*

11.2.1 Place a small sample, a few particles of powder, less than 1 mg, of the suspected methamphetamine or amphetamine on a microscope slide.

11.2.2 Dissolve the sample in a few microdrops of 10 % hydrochloric acid or concentrated phosphoric acid.

NOTE 2—The crystals tend to precipitate faster from the phosphoric acid. There also tends to be less interference when using the concentrated phosphoric acid.

11.2.3 Add a few microdrops of 5 % platinum chloride to the edge of the acid solution on the microscope slide.

11.2.4 Observe the formation of the crystals using a properly aligned and adjusted light microscope. This observation can be done between crossed polars if desired. If crossed polars are to be used, orient the polarizer in the east-west direction and the analyzer in the north-south direction, verified by a black background.

11.2.5 The crystals formed will depend on the drug present, as well as the optical isomer present for the drug. The formations that can be expected for methamphetamine and amphetamine are as follows.

11.2.5.1 *d- or l-Amphetamine,* produces long needles, which often are bent and which will grow into long rectangular blades.

11.2.5.2 *d,l-Amphetamine,* produces irregular blades and needles, which will grow into plates with irregular arms of blades.

11.2.5.3 *d- or l-Methamphetamine,* produces grains with sharp edges, which aggregate in a skeletal crystal having an appearance of ferns. The ends of the branches of the fern structure tend to bend at a sharp angle to the branches.

11.2.5.4 *d,l-Methamphetamine,* produces grains with sharp edges, which aggregate in a skeletal crystal having an appearance of ferns. The ends of the branches of the fern structure tend to be straight and not bent.

11.2.6 If a dense cloud of precipitate is formed upon the addition of the precipitating agent, the microcrystals may not be readily visible. It may be necessary to repeat the test reducing the concentration of suspected methamphetamine or amphetamine in the acid solution. This reduction is done by either decreasing the sample size or increasing the volume of solvent.

11.2.7 The following procedure may be used to separate the methamphetamine or amphetamine from diluents.

11.2.7.1 Place a small amount of the sample into a spot well. Sample size should be approximately 2 to 3 mg.

11.2.7.2 Add a small volume of 1.0 N to 10.0 N sodium hydroxide, such that the powder is just covered.

11.2.7.3 Place a microdrop of concentrated phosphoric acid, or platinum chloride reagent containing phosphoric acid, on a slide and invert the slide over the sample such that the microdrop is over the spot well containing the sample.

11.2.7.4 Any amine present, including methamphetamine and amphetamine, will be converted to the free base form. The free bases are volatile at room temperature, and as they leave the solution, they will collect in the microdrop of phosphoric acid.

11.2.7.5 After a period of time has elapsed, approximately 1 to 3 min, remove the slide and observe the crystal formed directly if platinum chloride is used in the hanging drop, or perform the testing procedure described in 11.2.3-11.2.5. The appropriate period of time is variable and will depend on the concentration of methamphetamine or amphetamine in the sample, the diluents present, as well as the room temperature. It may be necessary to repeat the test for a shorter or longer duration or utilize more sample if adequate recovery of amines is not accomplished during initial attempts.

12. Interpretation of Results (7)

12.1 The two precipitating reagents utilized in this procedure, that is, gold chloride and platinum chloride, are capable of distinguishing methamphetamine and amphetamine from each other. These tests also can be used to determine whether an optically active form (*d*- or *l*-) or a racemic mixture (*d*, *l*-) of the particular drug is present **(3)**.

12.2 If crystals structurally similar to those formed by methamphetamine or amphetamine standards are formed by both precipitating reagents, the sample *may be* considered positive by this technique for the presence of methamphetamine or amphetamine.

12.3 All observed crystalline precipitates must be documented and included in the analyst's notes for each item analyzed.

12.4 The forensic identification of methamphetamine or amphetamine requires the use of multiple uncorrelated techniques, see Practice E2329.

13. Precision and Bias

13.1 No information is presented about either the precision or bias of this technique.

14. Keywords

14.1 amphetamine; methamphetamine; microcrystalline testing

REFERENCES

(1) Fulton, C., "Modern Microcrystal Tests for Drugs," *Wiley-Interscience*, New York, NY, 1969.

(2) Clarke, E.G.C., "Isolation and Identification of Drugs," *Pharmaceutical Press*, London, England, 1971, pp. 139–141.

(3) Clark, J., "Method for Differentiating Optical Isomers of Amphetamine by the Simple and Direct Examination of Microcrystals in Gold Chloride Solution," *Journal of the A.O.A.C.*, Vol 56, No. 5, 1973, pp. 1065–1068.

(4) Auerbach, L., "Microcrystalline Identification Test of Some Amphetamines and Hydrochlorothiazide: Collaborative Study," *Journal of the A.O.A.C.*, Vol 61, No. 6, 1978, pp. 1435–1440.

(5) Chamot, E. and Mason, C., *Handbook of Chemical Microscopy: Vol I*, John Wiley, New York, NY, 1930.

(6) Chamot, E. and Mason, C., *Handbook of Chemical Microscopy: Vol II*, John Wiley, New York, NY, 1931.

(7) Nichols, R., "Drug Proficiency Test False Positives: A Lack of Critical Thought," *Science and Justice*, Vol 37, No. 3, pp. 191–196, 1997.