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Standard Guide for Microcrystal Testing in Forensic Analysis of Cocaine[1]

This standard is issued under the fixed designation E1968; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 cocaine.

1. Scope

1.1 This guide describes some standard procedures applicable to the analysis of cocaine using multiple microcrystal tests (**1-5**).[2]

1.2 These procedures are applicable to cocaine, which is present in solid dosage form or an injectable liquid form. They are not typically applicable to the analysis of cocaine 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 refractive index; this property will result in interference colors, which are viewed through a polarized light microscope.

3.2.3 *cocaine, n*—either *d*- or *l*- cocaine; it should be noted that *l*-cocaine is the naturally occurring isomer found in the coca plant.

3.2.4 *dendritic, adj*—multibrachiate or branching crystals, growing in a tree-like manner; each branch of the crystal is contiguous structurally.

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

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.

4. Summary of the Technique

4.1 A small sample of the material containing the suspected cocaine is dissolved in a dilute acid and the appropriate

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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.

precipitating reagent is added. The crystals that are formed are observed and distinguished utilizing a light microscope.

5. Significance and Use

5.1 This technique produces a chemical-precipitation reaction between cocaine and the precipitating reagent. The habit and the aggregation of the crystals formed may be used to distinguish cocaine from other drugs **(6)**.

5.2 This technique can be utilized on cocaine present in either the salt or free base form.

5.3 This technique does not distinguish between the salt and free base forms.

6. Interferences

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

7. Apparatus

7.1 *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 cocaine are birefringent.

8. Reagents and Materials

8.1 *10 % Solution of Acetic Acid.*

8.2 *Cocaine Standard.*

8.3 *5 % Solution of Gold Chloride ($HAuCl_4$),* in reagent grade water.

8.4 *10 % Solution of Hydrochloric Acid.*

8.5 *5 % Solution of Platinum Chloride (H_2PtCl_6),* in reagent grade water.

9. Sampling, Test Specimens, and Test 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 a cocaine standard. Only when it is determined that the reagents are producing the expected response, may the reagents be used in this 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 cocaine on a microscope slide.

11.1.2 Dissolve the sample in a few microdrops of 10 % hydrochloric acid or 10 % acetic 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 Formation of crystals in a habit corresponding to those obtained with standards is indicative of the presence of cocaine. The shape of these crystals may vary slightly depending on the concentration of the cocaine in the acid solution.

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 cocaine in the acid solution. This reduction is done by either decreasing the sample size or increasing the volume of solvent.

11.2 *Platinum Chloride:*

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

11.2.2 Dissolve the sample in a few microdrops of 10 % hydrochloric acid or 10 % acetic 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 Formation of crystals in a habit corresponding to those obtained with standards is indicative of the presence of cocaine. The shape of these crystals may vary slightly depending on the concentration of the cocaine in the acid solution.

11.2.6 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 cocaine in the acid solution. This reduction is done by either decreasing the sample size or increasing the volume of solvent.

12. Interpretation of Results (7)

12.1 Gold chloride is capable of distinguishing cocaine from its diastereoisomers **(8)**.

12.2 If crystals structurally similar to those formed by a cocaine standard are formed by both precipitating reagents, the sample *may be* considered positive by this technique for the presence of cocaine.

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 cocaine 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 analysis; cocaine; microcrystalline testing

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