Designation: E1968 – 19

An American National Standard

Standard Practice for Microcrystal Testing in Forensic Analysis for 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 practice describes procedures applicable to the analysis of cocaine using multiple microcrystal tests (**1-6**).[2]

1.2 These procedures are applicable to cocaine, which is present in solid 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 *These procedures could generate observations indicating a positive test for cocaine or its enantiomers which could be incorporated into the analytical scheme as defined by the laboratory.*

1.5 *This standard cannot replace knowledge, skills, or abilities acquired through appropriate education, training, and experience (see Practice E2326) and is to be used in conjunction with professional judgment by individuals with such discipline-specific knowledge, skills, and abilities.*

1.6 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

E2326 Practice for Education and Training of Seized-Drug Analysts

E2329 Practice for Identification of Seized Drugs

E2548 Guide for Sampling Seized Drugs for Qualitative and Quantitative Analysis

E2764 Practice for Uncertainty Assessment in the Context of Seized-Drug Analysis (Withdrawn 2020)[4]

3. Terminology

3.1 *Definitions:*

3.1.1 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, those having more than one refractive index; this property will result in interference colors, which are viewed through a polarized light microscope.

[1] This practice is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics.

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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.

[4] The last approved version of this historical standard is referenced on www.astm.org.

3.2.2.1 *birefringent, adj*—material exhibiting birefringence.

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

3.2.5 *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.

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

4. Summary of the Technique

4.1 A small amount of test material containing the suspected cocaine is dissolved in a dilute acid and the appropriate 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 involves a chemical-precipitation reaction between cocaine and the precipitating reagent. The habit and the aggregation of the crystals formed could 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 could inhibit crystal formation or could generate crystals that are distorted or otherwise rendered unidentifiable (**7**). Diluting the sample could reduce the interference. The higher the concentration of the adulterant, the more difficult it will be to observe characteristic crystals. There could be cases where diluting the sample would not work. 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. This is the minimum equipment required. A polarized light attachment is not essential, but is desirable, because the heavy metal crystals of cocaine are birefringent.

7.1.1 *Polarized Light Microscope (PLM),* capable of varying magnifications from 40× to 400×. The following are typical accessories on a PLM and could be useful, but are not required, to conduct microcrystalline testing: specialized rotating stage (360°) and compensator (retardation plate). Cross-polarizers are verified by observing a black background when the polarizer and analyzer are in the optical path at 90 degrees to one another (for example, polarizer is in the east-west direction and the analyzer is in the north-south direction).

7.1.2 The best practice for documenting the crystal formation results is to take a digital photograph. It is advised that the minimum equipment required also has the capability of digital photography.

8. Reagents and Materials

8.1 *10 %–20 % Solution of Acetic Acid* (hereafter, dilute acetic acid).

8.2 *Cocaine Standard.*

8.2.1 *l-Cocaine Standard.*

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

8.4 *10 % or 0.5 N Solution of Hydrochloric Acid* (hereafter, dilute HCl).

8.5 *Platinum Chloride (H_2PtCl_6),* in reagent grade water.

8.6 *10 mg TDTA (+)-O,O'-Di-p-toluoyl-D-tartaric Acid Monohydrate [CAS 32634-68-7]* in 1 mL ethanol, 1 mL glycerin, and 8 mL distilled water (**8**).[5]

8.7 *10 mg TLTA (-)-O,O'-Di-p-toluoyl-L-tartaric Acid Monohydrate [CAS 32634-66-5]* in 1 mL ethanol, 1 mL glycerin, and 8 mL distilled water (**8**).[5]

8.8 *Methanol or Diethyl Ether.*

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. Performance Verification

10.1 Prior to casework, the reagents used for these microcrystal tests shall be tested for reliability using a cocaine standard and negative controls following the prescribed procedure. Only when it is determined that the reagents are producing the expected response could the reagents be used in the testing procedure.

10.2 The microscope should be inspected, adjusted, and aligned to ensure it is in proper working order. This can be confirmed during the testing of the cocaine standard. Perform the analysis of unknown samples and standards under the same microscope operating procedures (for example, use of cross polarizers).

11. Procedure

11.1 *Gold Chloride or Platinum Chloride Tests:*

11.1.1 Place a small amount (approximately 1 mg) of test material on a microscope slide.

11.1.1.1 While the test material can be placed directly onto the slide, it could also be introduced onto the slide from a dilute solution of methanol or diethyl ether and allowing the solvent to dry before continuing with the analysis.

11.1.2 Dissolve the sample in a few microdrops of dilute HCl or dilute acetic acid.

[5] Crystals could form in the reagent after about three months. Prior to use, verify the reagents using the cocaine standard.