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Standard Guide for Microcrystal Testing in the Forensic Analysis of Cocaine[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 cocaine.

1. Scope

1.1 This guide describes some standard procedures applicable to the analysis of cocaine using multiple microcrystal tests.

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.

2. Terminology

2.1 *Definitions of Terms Specific to This Standard:*

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

2.1.2 *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.

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

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

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

2.1.6 *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.

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

3. Summary of the Technique

3.1 A small sample of the 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.

4. Significance and Use

4.1 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.

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

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

5. Interferences

5.1 *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.

6. Apparatus

6.1 *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.

7. Reagents and Materials

7.1 *10 % Solution of Acetic Acid.*

7.2 *Authenticated Cocaine Standard.*

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7.3 *5 % Solution of Gold Chloride ($HAuCl_4$),* in reagent grade water.

7.4 *10 % Solution of Hydrochloric Acid.*

7.5 *5 % Solution of Platinum Chloride (H_2PtCl_6),* in reagent grade water.

8. Calibration and Standardization

8.1 The reagents utilized for these microcrystal tests are to be tested for reliability using an authenticated cocaine standard. Only when it is determined that the reagents are producing the expected response, may the reagents be used in this procedure.

9. Procedure

9.1 *Gold Chloride*:

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

9.1.2 Dissolve the sample in a few microdrops of 10 % hydrochloric acid or 10 % acetic acid.

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

9.1.4 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.

9.1.5 Formation of crystals in a habit corresponding to those obtained with authenticated 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.

9.1.6 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.

9.2 *Platinum Chloride*:

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

9.2.2 Dissolve the sample in a few microdrops of 10 % hydrochloric acid or 10 % acetic acid.

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

9.2.4 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.

9.2.5 Formation of crystals in a habit corresponding to those obtained with authenticated 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.

9.2.6 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.

10. Interpretation of Results

10.1 Gold chloride is capable of distinguishing cocaine from its diastereoisomers.

10.2 If crystals structurally similar to those formed by an authenticated cocaine standard are formed by both precipitating reagents, the test results *may be* considered positive for the presence of cocaine.

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

11. Precision and Bias

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

12. Keywords

12.1 cocaine; microcrystalline testing

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