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Standard Guide for Microcrystal Testing in Forensic Analysis of Phencyclidine and Its Analogues[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 adequately gained 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 phencyclidine and its analogues.

1. Scope

1.1 This guide describes some standard procedures applicable to the analysis of phencyclidine and its analogues using microcrystal tests (**1-8**).[2]

1.2 These procedures are applicable to phencyclidme and its analogues which are present in solid dosage form or in a liquid form. They are not typically applicable to the analysis of phencyclidine and its analogues 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:*

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

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

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

3.2.4 *habit*—the external morphology of the crystal.

3.2.5 *microdrop*—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 *nails*—a skeleton of some kinds of triangles, elongated, usually pointed with a short head usually thicker or broader.

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

3.2.8 *nuggets*—irregularly formed grains without sharp faces or edges.

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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.

3.2.9 *pliers*—crystals resembling pliers, generally X-shaped.

3.2.10 *razor blades*—thin oblong crystals with length about twice the width, resembling a safety razor blade.

3.2.11 *sheaves*—elongated crystals form two opposite fans from the same joining point.

3.2.12 *skeletal crystal*—a crystal in which all of the spaces in the crystal lattice are not occupied.

3.2.13 *spindles*—shorter than course needles, but more substantial cross-section.

4. Summary of the Technique

4.1 A small sample of the material containing the suspected phencyclidine or its analogues 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 The technique produces a chemical-precipitation reaction between the phencyclidine or its analogues and the precipitating reagent. The habit and the aggregation of the crystals formed may be used to distinguish phencyclidine or its analogues from other drugs.

5.2 The technique can be utilized on phencyclidine or its analogues present in either the salt or free base form.

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

6. Interferences

6.1 Diluents/adulterants present in combination with phencyclidine or its analogues in the sample to be tested may result in crystals that are distorted or otherwise rendered unidentifiable. In these instances, it will be necessary to separate the phencyclidine or its analogues from the diluents/adulterants or to use other testing methods to analyze for phencyclidine or its analogues.

7. Apparatus

7.1 Standard light microscope capable of varying magnifications including 100× is needed for viewing the crystals. Polarized light attachment is not essential, but is desirable because crystals resulting from the precipitation reaction are birefringent.

8. Reagents and Materials

8.1 10 % v:v acetic acid.

8.2 10 % v:v hydrochloric acid.

8.3 2 % w:v potassium permanganate in 0.5 % v:v phosphoric acid.

8.4 Gold bromide ($HAuBr_4$) in diluted perchloric and acetic acids [0.55 g $HAuBr_4$, 42 mL water, 37 mL concentrated perchloric acid, 21 mL glacial acetic acid].

8.5 Gold chloride ($HAuCl_4$) in acetic and sulfuric acids [$HAuCl_4$ in HOAc-4(1+1)H_2SO_4 ; 2 g $HAuCl_4$, 20 mL glacial acetic acid, 40 mL concentrated sulfuric acid, 40 mL water].

8.6 Phencyclidine [PCP; 1-(1-phenylcyclohexyl)piperidine] standard.

8.7 Pyrrolidine analogue of phencyclidine [PCPy, PHP, 1-(1-phenylcyclohexyl)pyrrolidine] standard.

8.8 Morpholine analogue of phencyclidine [PCM, 1-(1-phenylcyclohexyl)morpholine] standard.

8.9 Thiophene analogue of phencyclidine [TCP; 1-[l-(2thienyl)cyclohexyl]piperidine] 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 phencyclidine or its analogues 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 *Potassium Permanganate:*

11.1.1 Place a small sample (a few particles of powder, less than one (1) milligram (mg) or a small drop of liquid, allowed to dry) of the suspected phencyclidine or its analogue on a microscope slide.

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

11.1.3 Add a few microdrops of 2 % acidified potassium permanganate to the edge of the acid solution on the microscope slide. Add a coverslip.

11.1.4 Observe the formation of crystals using a properly aligned and adjusted light microscope. The observation can be done between crossed polars, if desired. If crossed polars are used, care should be used to 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 crystal formed will depend on the drug present, if any. The formation that can be expected for phencyclidine and its analogues are as follows:

11.1.5.1 PCP (phencyclidine) produces purple razor blade crystals. In high concentrations, the crystals resemble needles impaling small spheres.

11.1.5.2 PCPy produces purple plier-shaped crystals with embedded nuggets and irregular forms.

11.1.5.3 PCM produces unremarkable crystals.

11.1.5.4 TCP produces unremarkable crystals.

11.1.6 If a dense cloud of precipitate is formed upon 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 phencyclidine or its analogue. This is done by either decreasing the sample size or increasing the volume of solvent.

11.2 *Gold Bromide in Diluted Perchloric and Acetic Acids:*