Designation: E2125 - 19

Standard Practice for Microcrystal Testing in Forensic Analysis for Phencyclidine and Its Analogues¹

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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 practice describes procedures applicable to the analysis of phencyclidine and its analogues using microcrystal tests (1-8).²
- 1.2 These procedures are applicable to phencyclidine and its analogues which are present in solid 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 These procedures could generate observations indicating a positive test for phencyclidine and its analogues 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 sound 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 This practice is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics. Current edition approved Nov. 1, 2019. Published December 2019. Originally

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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 Ouantitative Analysis

E2764 Practice for Uncertainty Assessment in the Context of Seized-Drug Analysis

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.

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.

- 3.2.2 *birefringence*, *n*—property of some crystals, those having more than one refractive index; this will result in interference colors which are viewed through a polarized light microscope.
 - 3.2.2.1 *birefringent, adj*—material exhibiting birefringence.
- 3.2.3 *grains*, *n*—thick tablets having nearly equal width, breadth, and thickness.
 - 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 μ L.
- 3.2.6 *nails*, *n*—a skeleton of some kinds of triangles, elongated, usually pointed with a short head usually thicker or broader.
- 3.2.7 *needles (acicular), n*—long, thin crystals with pointed ends.
- 3.2.8 *nuggets*, *n*—irregularly formed grains without sharp faces or edges.
- 3.2.9 *pliers*, *n*—crystals resembling pliers, generally X-shaped.
- 3.2.10 *razor blades*, *n*—thin oblong crystals with length about twice the width, resembling a safety razor blade.
- 3.2.11 *sheaves, n*—elongated crystals form two opposite fans from the same joining point.
- 3.2.12 *skeletal crystal*, *n*—a crystal in which all of the spaces in the crystal lattice are not occupied.
- 3.2.13 *spindles*, *n*—shorter than course needles, but more substantial cross-section.

4. Summary of the Technique

4.1 A small amount of test 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 This technique involves a chemical-precipitation reaction between the phencyclidine or its analogues and the precipitating reagent. The habit and the aggregation of the crystals formed could be used to distinguish phencyclidine or its analogues from other drugs.
- 5.2 This technique can be utilized on phencyclidine or its analogues present in either the salt or free base form.
- 5.3 This 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 could inhibit crystal formation or could generate crystals that are distorted or otherwise rendered unidentifiable. Diluting the sample could reduce the interference. The higher the concentration of the adulterant, the more difficult it will be to observe

characteristic crystals. 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. This is the minimum equipment required. A polarized light attachment is not essential, but is desirable because crystals resulting from the precipitation reaction 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 % Solution of Acetic Acid (hereafter, dilute acetic acid).
- 8.2 10 % Solution of Hydrochloric Acid (hereafter, dilute hydrochloric acid).
- 8.3 2 % Potassium Permanganate in 0.5 % Phosphoric Acid.
- 8.4 Gold Bromide ($HAuBr_4$) in Diluted Perchloric and Acetic Acids [0.55 g HAuBr₄, 42 mL distilled water, 37 mL concentrated perchloric acid, 21 mL glacial acetic acid].
- 8.5 Gold Chloride ($HAuCl_4$) in Acetic and Sulfuric Acids [2 g $HAuCl_4$, 20 mL glacial acetic acid, 40 mL concentrated sulfuric acid, 40 mL distilled 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 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 use in casework, the reagents used for these microcrystal tests shall be tested for reliability using phencyclidine or its analogues and negative controls following the