



Standard Guide for Microcrystal Testing in the Forensic Analysis of 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 guide describes some standard procedures applicable to the analysis of phencyclidine and its analogues using microcrystal tests.

1.2 These procedures are applicable to phencyclidine 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 *This guide offers an organized collection of information or a series of options and does not recommend a specific course of action. This document cannot replace education or experience and should be used in conjunction with professional judgment. Not all aspects of this guide may be applicable in all circumstances. This ASTM standard is not intended to represent or replace the standard of care by which the adequacy of a given professional service must be judged, nor should this document be applied without consideration of a project's many unique aspects. The word "Standard" in the title of this document means only that the document has been approved through the ASTM consensus process.*

1.4 *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 appro-*

priate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Terminology

2.1 Definitions:

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

2.1.2 *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.

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

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

2.1.5 *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 microliters.

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

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

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

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

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

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

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

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

3. Summary of the Technique

3.1 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.

4. Significance and Use

4.1 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.

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

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

5. Interferences

5.1 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.

6. Apparatus

6.1 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.

7. Reagents and Materials

7.1 10 % v:v acetic acid.

7.2 10 % v:v hydrochloric acid.

7.3 2 % w:v potassium permanganate in 0.5 % v:v phosphoric acid.

7.4 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].

7.5 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].

7.6 Authentic phencyclidine [PCP; 1-(1-phenylcyclohexyl)piperidine].

7.7 Authentic pyrrolidine analogue of phencyclidine [PCPy, PHP, 1-(1-phenylcyclohexyl)pyrrolidine].

7.8 Authentic morpholine analogue of phencyclidine [PCM, 1-(1-phenylcyclohexyl)morpholine].

7.9 Authentic thiophene analogue of phencyclidine [TCP; 1-[1-(2thienyl)cyclohexyl]piperidine].

8. Calibration and Standardization

8.1 The reagents utilized for these microcrystal tests are to be tested for reliability using authenticated 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.

9. Procedure

9.1 Potassium Permanganate

9.1.1 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.

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

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

9.1.4 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.

9.1.5 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:

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

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

9.1.5.3 PCM produces unremarkable crystals.

9.1.5.4 TCP produces unremarkable crystals.

9.1.6 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.

9.2 Gold Bromide in diluted Perchloric and Acetic Acids

9.2.1 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 on a microscope slide.

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

9.2.3 Add a few microdrops of gold bromide in diluted perchloric and acetic acids reagent to the edge of the acid solution on the microscope slide. Cover with a coverslip.

9.2.4 Observe the formation of crystal 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.

9.2.5 The crystal formation will depend on the drug present, if any. The formation that can be expected for phencyclidine and its analogues are as follows:

9.2.5.1 PCP (phencyclidine) produces red-gold colored squares with diagonal markings, often elongated along one axis; birefringent; in high concentrations, colorless nails.

9.2.5.2 PCPy produces aggregates of tiny rods and X-shaped crystals; birefringent.

9.2.5.3 PCM produces amorphous oily drops which slowly aggregate into plates.

9.2.5.4 TCP produces thin X-shaped crystals, often with grossly elongated arms; birefringent.

9.2.6 If a dense cloud 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.

9.3 Gold Chloride in Acetic and Sulfuric Acids

9.3.1 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 on a microscope slide.

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

9.3.3 Add a few microdrops of gold chloride in acetic acid and sulfuric acid reagent to the edge of the acid solution on the microscope slide. Add a coverslip.

9.3.4 Observe the formation of crystal using a properly aligned and adjusted light microscope. The observation can be done between crossed polars, if desired. If crossed polar 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.

9.3.5 The crystal formation will depend on the drug present, if any. The formation that can be expected for phencyclidine and its analogues are as follows:

9.3.5.1 PCP produces unremarkable crystals.

9.3.5.2 PCPy produces yellow X-shaped crystals.

9.3.5.3 PCM produces H-shaped plates with widened ends.

9.3.5.4 TCP produces yellow thick needles, some with short, perpendicular central arms.

9.3.6 If a dense cloud 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.

10. Interpretation of Results

10.1 The three precipitating reagents utilized in this technique, that is, potassium permanganate, gold bromide in diluted perchloric and acetic acids, and gold chloride in acetic and sulfuric acids, are capable of distinguishing PCP (phencyclidine), PCPy, PCM, and TCP from each other.

10.2 If crystals structurally similar to those formed by authenticated phencyclidine and phencyclidine analogue standards are formed by at least two of the precipitating agents, the sample may be considered positive by this technique for the presence of either PCP, PCPy, PCM, or TCP.

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 microcrystalline testing; morpholine analogue of phencyclidine, PCM; phencyclidine, PCP; pyrrolidine analogue of phencyclidine, PCPy; thiophene analogue of phencyclidine, TCP

REFERENCES

- (1) Fulton, Charles C., *Modern Microcrystal Tests for Drugs*, Wiley-Interscience, New York, 1969.
- (2) Clarke, E. G. C., *Isolation and Identification of Drugs*, Pharmaceutical Press, London, 1971, pp. 139-141.
- (3) Chamot, E. and Mason, C., *Handbook of Chemical Microscopy*, Volume 1. John Wiley, New York, 1930.
- (4) Evans, Hiram K., "Microcrystal Tests for the Characterization of Some 1-Arylcyclohexylamines," American Academy of Forensic Sciences, Cincinnati, Ohio, 19 Feb. 1983.
- (5) Harris, H. et al., "Vegetable Matter Coated with a Barbiturate and a Veterinary Anesthetic," *Microgram*, Vol. 10, No. 1, Jan. 1977, pp. 9-10.
- (6) Epstein, R. L. and Lorimer, P., and Sloma, E. J., "Identification of Phencyclidine-Related Drugs," *Microgram*, Vol. 22, No. 1, Jan. 1977, pp. 61-69.
- (7) Koles, J. E., "Some Microcrystal Tests for Drugs" in *Progress in Chemical Toxicology*, Vol. 5, ed. A. Stolman, Academic Press. San Francisco, CA, 1974.
- (8) "Miscellaneous Tests and Procedures," *Microgram*, Vol. 7, No. 2, Feb. 1974, p. 20.
- (9) Nichols, Ronald "Drug Proficiency Test False Positives: A Lack of Critical Thought," *Science and Justice*, Vol. 37, No. 3, pp. 191-196.

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