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Development of a Compendium of Microcrystal Tests for Illicit Drugs

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Microcrystal tests using polarized light microscopy (PLM) can identify most illicit drugs specifically and quickly, and they are inexpensive compared to other methods. "A Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals" [1], recently published online by McCrone Research Institute, includes 19 drugs for which microcrystal tests using various reagents have been previously developed. This compendium describes in detail the microcrystals formed from each test and includes photomicrographs, morphology illustrations, optical properties, notes and infrared spectra of the microcrystals. An example page of the compendium is shown in Figure 1.

For present-day relevance, a survey of crime laboratories was conducted to determine which drugs, reagents and microcrystal tests are currently being used. Because earlier literature and references contain numerous microcrystal tests for each drug, the authors chose only the most commonly used drugs, reagents and test methods for this compendium.

Information about known microcrystal tests and reference material from numerous sources spanning past decades, including textbooks, journal articles and standard operating procedures, were located and evaluated. Many of these are out of print and not easily accessible. Such references typically contain few photomicrographs of microcrystals, and their reagent formulations and procedures may be difficult to interpret. There is also a lack of information regarding potential interferences from other drugs that may be combined with pharmaceuticals or from adulterants found in street drug samples.

The compendium includes the following topics for each drug: reagents and formulation; test methods; sensitivity of the test and limit of detection; time required for crystal formation; evaluation of the tests in the presence of common excipients, diluents and adulterants; and evaluation of the tests for drugs from selected pharmaceutical delivery devices, e.g. tablets, capsules, gels, transdermal patches and oral solutions.

"A Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals" is available as a PDF file free for download on McCrone Research Institute's website, http://www.mcri.org [2].

References:

[1] KM Brinsko, D Golemis, MB King, *et al.* "Modern Compendium of Microcrystal Tests for Illicit Drugs and Diverted Pharmaceuticals," McCrone Research Institute: Chicago, (2015); http://mcri.org/uploads/A_Modern_Compendium_of_Microcrystal_Tests.pdf.
[2] This project was supported by Award No. 2011-DN-BX-K528, awarded by the National Institute of Justice, Office of Justice Programs, U.S. Department of Justice. The opinions, findings, and conclusions or recommendations expressed in this publication/program/exhibition are those of the author(s) and do not necessarily reflect those of the Department of Justice.

Codeine: Fulton's O-2

REAGENT 2: Fulton's O-2 (I-KI)

0.4 mL I-KI solution (0.1 g I_2 and 0.35 g KI in 1 mL H_2O). 1.6 mL H_2O, 2.0 mL glacial HOAc and 2.0 mL concentrated H_3PO_4. This reagent has a shelf life of several weeks to several months. It should be dark brown and free of precipitates.

Test Method

Direct test: Place the sample on a slide and crush it with a pipette tip, glass rod or toothpick. Place a 10 µL drop of reagent on a coverslip; invert the coverslip and place it directly onto the sample.

References

1. Fulton, C.S. *Modern Microcrystal Tests for Drugs*, Wiley-Interscience: New York, 1969.

Limit of Detection

1 PPP (1–10 PPP for some pharmaceutical tablets)

Time Required for Crystal Formation

Immediate

Crystal Morphology and Test Notes

Hatchet blades, hourglasses, triangles, semi-circles, trapezoids and rosettes of these same shapes. Amorphous grains precede crystal formation.

Photomicrograph of Typical Crystals

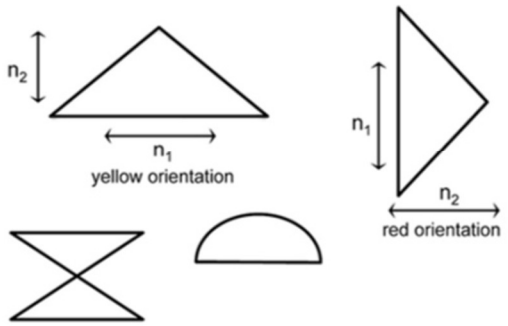
Figure 1. 1 PPP of codeine and 10 µL of reagent. Crystals form rosettes of triangles and hatchet blades.

Pharmaceuticals, Adulterants or Other Drug Interactions

This test was successful on some pharmaceutical tablets. An acid-base extraction may be helpful. Results were not always successful or reproducible, especially when an abundance of starch is present. Immiscible, oily drops preceded crystal formation, however, with some pharmaceuticals, typical crystals never formed. The test was unsuccessful with pharmaceutical oral solutions.

Acid-base extraction procedure: Add a portion of the pharmaceutical tablet to a microcentrifuge tube. Add 200 µL of 10% H_2SO_4 and mix by aspirating with a transfer pipette. Slowly add 200 µL of saturated Na_2CO_3 solution and mix. Add 50 µL of chloroform and mix. Use a micropipette to draw off 10 µL from the chloroform layer (bottom) and place a small drop onto a glass slide. Allow to evaporate, then proceed with microcrystal test.

PLM Optical Properties

Approximate Size Range	5–30 µm; increases over time
Color/Pleochroism	Red-orange. Pleochroic; rusty red-orange to yellow
Refractive Indices (RI)	$n_1 \approx 1.650–1.674$ $n_2 > 1.700$
Morphology Illustration	
How Crystals Were Dried for RI Measurement	Excess liquid was wicked away with lab tissue then washed with chloroform using a tungsten needle.
Estimated Birefringence	High
Extinction	Symmetrical or parallel to one edge of triangle
Sign of Elongation	Not applicable
Crystal Optics and Optic Sign (Interference Figure)	Indeterminable

IR Spectrum

See Figure 13. Download SPC file.

Figure 1. A page from McCrone Research Institute's compendium showing a microcrystal test for codeine.

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