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## Microcrystal Analysis of Cocaine Hydrochloride and Added Adulterants

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### Abstract:

*Microcrystalline tests were performed on samples of cocaine with 10, 20, and 50% adulterant. The tests were first performed with aqueous solutions of the reagents. The microcrystal tests were then done on powders to more closely simulate actual drug testing. Specific trends in the changes of the crystal morphology could be identified, making it possible to determine both the identity and the concentration of the adulterant.*

### Introduction:

Cocaine submitted into evidence at trials is usually between 60-70% pure. Caffeine, sugar, lidocaine, and procaine are commonly added to increase the bulk and the street value (1). Levamisole, a cancer drug and also a pig dewormer, is one of the most recent adulterants seen in cocaine submitted to crime labs. Because the identity of the adulterant is generally unknown, extraction for analysis in the crime lab can be difficult and time consuming. Without prior purification, microcrystal tests are complicated by alteration of the crystal formation due to the reaction of adulterants with the reagents (2). The objective of this project was to see if the trends in the changes of the crystal morphology could be linked to particular adulterants and their concentrations in the cocaine samples.

Microcrystal tests are highly developed chemical precipitation tests that use specific reagents and a polarizing light microscope to document the characteristic crystal formation of an unknown substance (3,4). These tests are specific and sensitive enough to detect even small quantities of a substance such as cocaine and eliminate false positive results that may result with simple color tests. In the analysis of controlled substances, microcrystal tests are a presumptive test used for screening unknown powders quickly. A positive microcrystal test for cocaine is followed by two confirmatory tests, generally, gas chromatography and mass spectroscopy.

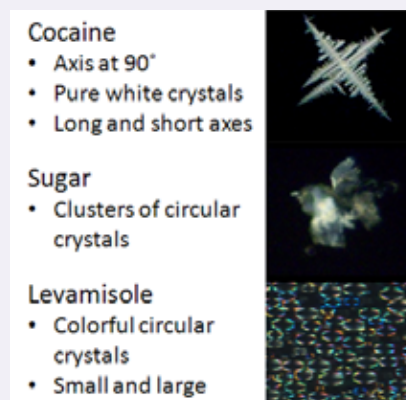
One presumptive test for controlled substances uses gold chloride as a reagent to form a distinctive crystal morphology specific to the drug in question. Cocaine and gold chloride form cocaine hydroaurate characterized by cross-shaped crystals with branches perpendicular to the axes (2). However, the microcrystalline test depends on having a relatively pure sample of the illicit drug. The presence of impurities will affect the shape or morphology of the crystal making a definite identification difficult.

A study done by Swaitko *et al.* used three color tests (Wagner's, marquis, and cobalt thiocyanate) and two microcrystal tests to determine specificity for cocaine. They tested 20 drugs using the color tests. The color tests could not differentiate between cocaine and nine other compounds. With the gold chloride the microcrystal test, there were no false positives for any of the compounds examined (4).

A second study by Weilbo and Tebbett combined the microcrystal test and Fourier transformed infrared (FTIR). They used 19 reagents and determined that gold chloride was sensitive enough to detect micrograms of cocaine and is suitable for FTIR. The microcrystal test is quick, sensitive to a specific drug, and excellent for screening use (2).

### Materials and Methods:

Stock solutions (2.50 mg/ml) were made for cocaine, sugar, and levamisole. Pure samples of each drug were then tested by adding 10  $\mu$ L of the stock solution to a glass microscope slide followed by 10  $\mu$ L of 5% gold chloride and 10  $\mu$ L 20% acetic acid. The above procedure was then used on cocaine/adulterant mixtures with 10%, 20%, or 50% of sugar or levamisole added. For the preparation of 10% samples, 1  $\mu$ L of adulterant/ 9  $\mu$ L cocaine, for 20% samples, 2  $\mu$ L adulterant /8  $\mu$ L cocaine, and for 50% samples, 5  $\mu$ L adulterant /5  $\mu$ L cocaine. Each sample was repeated ten times.



*Figure 1. Crystal habit of cocaine, sugar, and levamisole*

The microcrystal tests were then performed on solid samples with the adulterant in the same concentrations of 10, 20 and 50% concentrations. The mixtures were ground with a mortar and pestle. Then 10  $\mu$ L of 5% gold chloride and 10  $\mu$ L 20% acetic acid was added to a 2-4 mg of the solid mixture and crystal formation was observed.

### Results:

The crystals formed from pure solutions of cocaine, sugar, and levamisole are shown in Figure 1. The characteristic crystal morphology of cocaine is two perpendicular axes with branching. The sugar crystals are circular, and the levamisole forms

small circular crystals with an X in the center.

**10 % Sugar**

- Enlarged long axis and short middle axis
- Some crystals with protruding branches
- Some with little or no branching
- Sphere shaped crystals
- U and Y shaped middle axes

**20 % Sugar**

- Sphere shaped clusters
- Decreased length of middle axis
- Y shaped middle or long axis

**50 % Sugar**

- Small
- Enlarged long axis with a short branching middle axis
- Sphere and X shaped crystals




Figure 2. Crystal habit of cocaine with 10%, 20%, and 50% Sugar

The first adulterant studied was sugar (Figure 2). As increasing amounts of sugar were added to cocaine, the resulting crystals began to have increased branching on the long and middle axes. With 10% sugar, one axis was elongated, the branching became three dimensional, and the middle axis was deformed into either a Y or U shape. At 20%, small number of sphere shaped crystals appeared and the long axis began to deform. At 50%, the same trends were observed and the crystals were smaller.

At 10% levamisole, the cocaine crystals had varied shapes composed of multiple branching of different lengths. The middle axis took on an X or Y shape. Most of the crystals were spherical or shaped like an asterisk. With 20% levamisole, the branching increased on one or both axes. Sphere and asterisk shapes formed with protruding branches (Figure 3).

**10 % Levamisole**

- Many different shaped crystals
- Mostly sphere and asterisk shape
- X and Y shape
- Different length branching
- Protruding in different directions

**20 % Levamisole**

- Sphere, asterisk, X and Y shaped crystals
- Many protruding branches
- Long and short axes with some branching
- Some crystals, on one axis with branching

**50 % Levamisole**

- Small sphere and asterisk shaped crystals
- A few X and Y shaped crystals
- Protruding branches

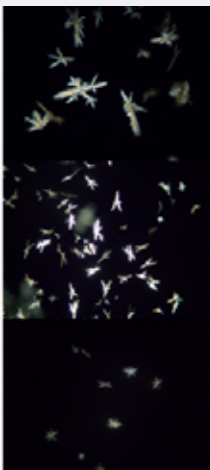


Figure 3. Crystal habit of cocaine with 10%, 20%, and 50% Levamisole

While the initial tests were performed on aqueous solutions of the cocaine and adulterants, in a forensic crime lab, the microcrystal tests are performed on street drugs that are in powder form. The microcrystal tests were repeated with powder samples to more closely emulate the actual tests done in the lab. Similar

changes were observed in the powder samples (Figure 4).

**Sugar**      **Levamisole**

10 %

20 %

50 %

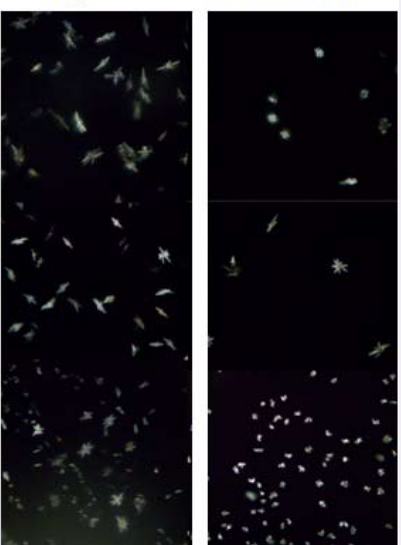


Figure 4. Crystal habit of powder samples of cocaine with levamisole and sugar

### Conclusions:

The results indicate that the cocaine crystal morphology changes if an adulterant is present and the characteristics of the changes can be correlated to the type and amount of adulterant present in the sample. Sugar, as the adulterant, can be identified by X and Y-shaped middle axis and the concentration by the amount of branching on both axes, size, and spheres. Levamisole as the adulterant can be identified by variation of the crystal morphology and the concentration by degradation of the crystals and the amount of sphere, asterisk, and X-shaped crystals.

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