



B125 Microcrystalline Tests in Conjunction With Vibrational Spectroscopy for the Analysis of Illicit Drugs and Their Metabolites

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After attending this presentation, attendees will understand that the coupling of microcrystalline tests with vibrational spectroscopy (either infrared or Raman) is an excellent analytical scheme for drug identification because it is rapid, reliable, and creates a reviewable record of the analysis.

This presentation will impact the forensic science community by presenting a method of drug analysis that pairs microcrystalline tests with vibrational spectroscopy, thus creating an analytical scheme that satisfies the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) standards and can be easily incorporated into a criminalist's drug-testing protocol.

The analysis and identification of illicit drugs is a critical component of the field of forensic science as it constitutes a majority of casework performed in most forensic laboratories. Any analytical method for the identification of drugs must be rapid, reliable, and create a reviewable record in order to satisfy legal standards as well as manage the large number of submissions to a forensic laboratory. SWGDRUG has established minimum standards for the forensic identification of illicit drugs.¹ The SWGDRUG guidelines classify techniques into three categories which are based on discriminating power, with A being the most and C the least. One proposed analytical scheme is using two techniques, one of which must be from category A (infrared spectroscopy, Raman spectroscopy, mass spectrometry, nuclear magnetic resonance spectrometry, or X-ray diffractometry). This research proposed the coupling of microcrystalline tests (a Category B technique) with vibrational spectroscopy (either infrared or Raman) as a fast, efficient, inexpensive, and non-destructive drug analysis and identification analytical scheme that satisfies SWGDRUG standards and can be easily executed by criminalists.

Microcrystalline tests are highly developed precipitation tests that use specific reagents to create crystals with particular morphologies that can be used to determine the presence of many chemicals, including both controlled substances and other related compounds. An advantage of microcrystalline testing is that there are a plethora of crystal habits that show morphological differences, and this enables closely related analogs to be readily differentiated. In addition, these tests work with sample mixtures and, in effect act, as a method of separation because the reagent will form characteristic microcrystals only with specific drugs. Although in the past microcrystalline tests were considered to be confirmatory techniques, today they are used as preliminary or presumptive testing methods to indicate what the drug might be, which is why instrumentation is needed to confirm the results. Infrared and Raman spectroscopy are common forms of instrumentation that are used in forensic laboratories to identify illicit drugs. Although both types of vibrational spectroscopy are considered to generate the highest discriminating capabilities, the discrimination power is considered to be diminished in mixtures because what is produced is a combined spectrum that is more difficult to interpret. Thus, a method of separation is often employed to make a pure sample capable of spectroscopic analysis.

The proposed method uses microcrystalline tests both to identify the drug by its morphology and also as a means for isolating the drug for vibrational microspectral analysis. Prior research published by Wielbo and Tebbett coupled infrared microspectroscopy with microcrystalline tests of seven drugs; however, the microcrystalline samples were allowed to dry at room temperature prior to being analyzed by infrared spectroscopy.² The current research improves on this method by using a diamond-Attenuated Total Reflection (d-ATR) infrared microprobe, thus enabling analysis of the microcrystals in solution.³ Research presented at the 2007 American Academy of Forensic Sciences (AAFS) Annual Scientific Meeting by Cullinan and Bell used both Raman and infrared microspectroscopy to analyze drug microcrystals of phenylethylamines (amphetamine, methamphetamine, phentermine, and ephedrine).⁴ This research has extended the application of this technique to the identification and confirmation of cocaine and its metabolite ecognine, morphine, codeine, as well as additional phenylethylamines, such as common bath salts (methylenedioxypropylone or MDPV and ethylone) and 3-4-methylenedioxymethamphetamine (MDMA).



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The coupling of microcrystalline tests with vibrational spectroscopy is a rapid testing method which requires minimal reagents, combines two reliable techniques, produces reviewable records of the results (in the form of photomicrographs and spectra), is non-destructive (test compounds are recoverable from the test slide), uses instrumentation that is currently available in most forensic laboratories, and satisfies SWGDRUG guidelines for the identification and confirmation of illicit drugs. This research demonstrated that pairing microcrystalline tests with either infrared or Raman microspectroscopy is a valuable technique for drug identification within the ever-growing field of forensic drug analysis.

Reference(s):

1. United States Department of Justice, Drug Enforcement Administration. Scientific working group for the analysis of seized drugs (SWGDRUG) recommendations. Executive office of the president's office of national drug control policy counterdrug technology assessment center. 2013.
2. Wielbo D, Tebbett I.R. The Use of Microcrystal Tests in Conjunction with Fourier Transform Infra Red Spectroscopy for the Rapid Identification of Street Drugs, *J Forensic Sci.* 1992;37(4):1134-48.
3. Reffner J.A. Rapid Illicit Drug Analysis Using the Infrared Microprobe. *Proceedings of the American Academy of Forensic Sciences*, 58th Annual Scientific Meeting, Seattle, WA. 2006.
4. Cullinan R.M., Bell S.C. Comprehensive and Definitive Characterization of Drug Microcrystals. *Proceedings of the American Academy of Forensic Sciences*, 59th Annual Scientific Meeting, San Antonio, TX. 2007.

Drugs, Microcrystalline Tests, Spectroscopy