B58 The Development of an Infrared Microspectroscopy Method for Microcrystals

Michael Cain, Jr., West Chester University of PA, 750 S Church Street, Schmucker Science S, West Chester, PA 19383; and Monica Joshi, PhD, West Chester University, Dept of Chemistry, Schmucker Science, S, 750 S Church Street, West Chester, PA 19383*

After attending this presentation, attendees will better understand the combined role of infrared microspectroscopy and microcrystalline tests for the analysis of drug evidence.

This presentation will impact the forensic science community by creating a deeper understanding of the reliability and validity of an infrared microspectroscopy method for the analysis of drug microcrystals. This presentation describes the challenges and solutions associated with growing microcrystals on substrates suitable for transmission and reflectance infrared analysis.

Microcrystalline tests have long been used by forensic chemists for the analysis of drugs, but there is continued debate regarding the extent of their discriminatory power. The debate lies in the fact that the chemistry of the microcrystals is not completely understood, although characteristic crystals are obtained for some substances. Lack of tangible instrumental data to confirm the microcrystal identity has steered analysis schemes away from these tests; however, microcrystalline tests are advantageous for several reasons. They are easy to perform, require virtually no sample preparation, use negligible amounts of solvents, require microgram quantities of test samples, are cost-effective, very sensitive, are non-destructive and, most importantly, very rapid. Analytical techniques that can combine the simplicity of the microcrystalline tests and molecular structure information would be very useful.

Infrared spectroscopy is widely used in forensic laboratories for the analysis of drug evidence and is classified as a category A technique in the Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) analytical techniques, affording the highest discriminating ability. Infrared spectroscopy is a valuable tool for differentiating between isomers based on spectral differences. Combining infrared microspectroscopy with drug microcrystals will provide unambiguous confirmation of the crystal identity, thereby removing the subjectivity often seen as a weakness of microcrystalline tests.

Infrared microspectroscopy has been previously demonstrated for the analysis of drug microcrystals; however, there are challenges associated with obtaining quality spectra with this technique.[1,2] Three modes of Fourier Transform Infrared Spectroscopy (FTIR) are commonly used: transmission, reflectance, and Attenuated Total Reflectance (ATR). ATR is destructive to the crystals and spatial resolution is poor. The microcrystalline reagents are often acidic and are incompatible with the traditional infrared substrates used for transmission and reflectance studies. A detailed study to improve the understanding of this technique will help analysts adopt this technique in their analytical schemes for drug evidence.

This presentation describes the challenges associated with analyzing microcrystals in the different infrared spectroscopy modes. This presentation compares the quality of spectra obtained with gold-coated slides to the MirrIR low-e microscope slides in the reflectance mode. In the transmission mode, barium fluoride windows are compared to the novel Amorphous Material Transmitting Infrared Radiation (AMTIR) windows. Differences in the vibrational bands of the drug-reagent microcrystal are observed when compared to the drug-only spectra. These differences are documented and quantified. In addition, procedures to reduce interference from the reagent in solution around the microcrystal are presented. These steps improve the quality of spectra obtained for the drug-

reagent microcrystals. This presentation discusses the validity and reliability of the infrared microspectroscopy method for microcrystals analyzed in different modes.

Reference(s):

1. Wielbo D., Tebett I.R., The use of microcrystal tests in conjunction with fourier transform infrared spectroscopy for the rapid identification of street drugs. *Journal of Forensic Sciences.* 1992, 37 (4), 1134-1148.
2. McCrone Research Institute. *A Modern Compendium of Microcrystal Tests for Illicit-Drugs and Diverted Pharmaceuticals.* 2015.

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