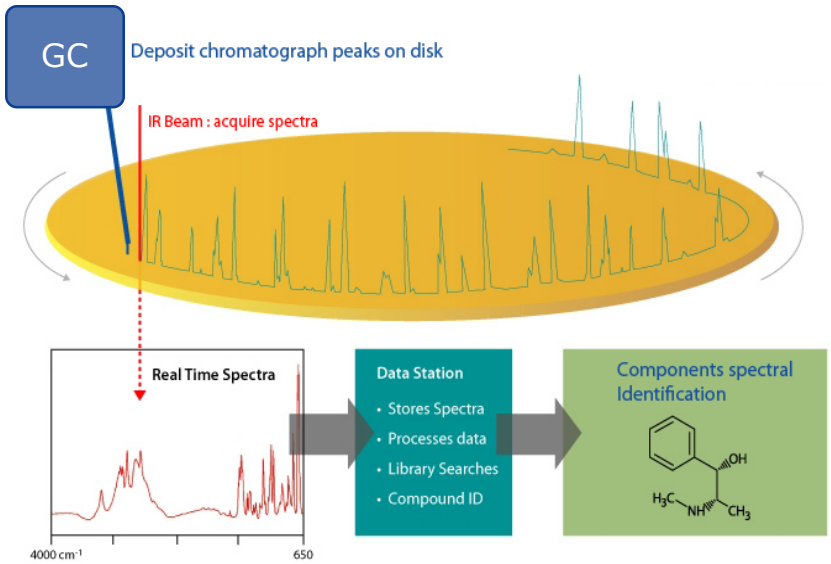
Application Note 35

DiscovIR-GC™

Deposition and Detection System



The DiscovIR-GC is a powerful new tool for materials analysis. When connected to the outlet of a GC column, the DiscovIR deposits GC eluants as a continuous track on an infrared transparent substrate. The built-in interferometer simultaneously captures a set of time-ordered infrared spectra from the deposited track. The result is a map of molecular structure of all sample components.

BATH SALTS ANALYSIS

Sold as "Vanilla Sky" and "Ivory Wave," bath salts have become very popular in recent years for the very "high" they are providing. Packets of bath salts containing mostly white powder can be purchased in truck stops and convenience stores across the United States. This "super coke" is said to contain methylenedioxypyrovalenone (MDPV) and lidocaine. However, the actual contents of bath salts vary widely from "manufacturer to manufacturer" affecting psychotropic effects and safety. For example, chemical additives found in bath salts can also be found in plant fertilizer and/or food. The main active ingredient, MDPV, is a psychoactive drug with very powerful stimulant properties. It provides sensations similar to those experienced through cocaine use, such as euphoria and physical and mental stimulation. However, there is also a wide range of undesirable side effects, such as chest pains, increased blood pressure, increased heart rate, agitation, hallucinations, extreme paranoia, delusions, and fiending.

In the U.S., southern states have begun to ban bath salts due to numerous incidents linked with ingestion, snorting, smoking, or injection. At this time, criminal cases involving the use of bath salts are increasing rapidly. For proper criminal proceedings, identification of active ingredients and other species in the seized drug samples of bath salts must be definite and conclusive. It is imperative that analysis of MDPV, which is often mixed with the isomer 4-fluoromethcathinone (4-FMC) to heighten the drug's potency, be performed and determined accurately.

For conclusive qualitative analysis, The Scientific Work Group for the Analysis of Seized Drugs (SWGDRUG) requires (1) a minimum of two analytical methods to form an acceptable analytical conclusion, and (2) that each method must yield conclusive and definite results (precluding false positives).

Today, new gas chromatography-infrared spectroscopy hyphenated systems meet both requirements set forth by the SWGDRUG committee in one reliable and accurate instrument. Each molecule possesses its own characteristic IR fingerprint[1] offering a clear identification of chemical functions and substructures. In particular, structural and conformational differences become very easily identifiable due to the very different electronic environment of each isomer.

SAI, Inc. has developed a GC-IR method that easily identified all the components of a particular bath salts sample, while minimizing any uncertainty surrounding the identification of particular isomers found in bath salt mixes.

To demonstrate this, a simple organic solution of bath salts called Miracle was prepared and injected (1µl) onto the DiscovIR-GC system.[2] The resulting infrared chromatogram (IR Peaks Chromatogram) showed a number of elution peaks representing 5 small, discrete, synthetic molecules (Figure 1, see back).

The 5 discrete molecules were identified either:

- by using the database searching and matching capabilities of the Spectral ID™ algorithm from GRAMS™ software[3] (The Spectral ID program uses classical algorithm such as PCA and PLS to determine ID match) and a traditional commercial solid phase spectral library, or
- by overlaying a validated reference spectrum against the peak infrared spectrum.

The bath salts mixture contained:

- 4-FMC, a stimulant drug of the cathinone chemical class, a structural isomer of 3-FMC (identified using spectrum overlay) (Figure 2, see back)
- 4-MMC or Mephedrone, a synthetic stimulant of the cathinone class, a structural isomer of 3-MMC (identified using spectrum overlay)
- Lidocaine, a common local anesthetic used to mask a diluted drug mixture (identified using Spectral ID)
- MDPV, a psychoactive drug with stimulant properties (identified using spectrum overlay) (Figure 2, see back)
- JWH-018, an synthetic cannabinoid from the napthoylindole family, producing effects similar to THC, a natural cannabinoid present in cannabis (identified using Spectral ID)

The identity of the different components can also be

confirmed by comparing GC retention time with published references.

The bath salts sample was easily resolved by GC-IR by comparing GC retention time and infrared spectrum of individual adulterants, diluents or API present in the bath salts sample against their references. In particular, unlike GC-MS[4,5], the DiscovIR identified 4-FMC and 4-MMC and distinguished them from their isomers 3-FMC and 3-MMC. Such differentiation and identification were possible for the GC-IR due to the instrument's very high level of specificity in selectively analyzing positional and stereo isomers through infrared spectroscopy.

Spectra Analysis Instruments, Inc. has developed DiscovIR™, the first commercially available hyphenated system with infrared spectroscopy as the detection module and gas or liquid chromatography as the separation module. With the DiscovIR, individual isomer identification is made possible through definitive retention time values as well as individual IR spectrum. When connected to the outlet of a GC column, the DiscovIR deposits GC eluants as a continuous solvent-free track on an infrared transparent substrate. The built-in interferometer simultaneously captures a set of time-ordered infrared spectra from the deposition track. Because of the unparalleled specificity of infrared spectroscopy, the GC-IR hyphenated system enables sharp discrimination among structural isomers and provides conclusive identification of each substance being tested.

To add to this, the DiscovIR automatically generates full IR spectra for each eluant or peak which can be compared to the solid-phase IR libraries. The optional library of 10,000+ compounds can be expanded through access to private or commercially available libraries, and compounds can be added by the user as they are encountered.

Figure 1

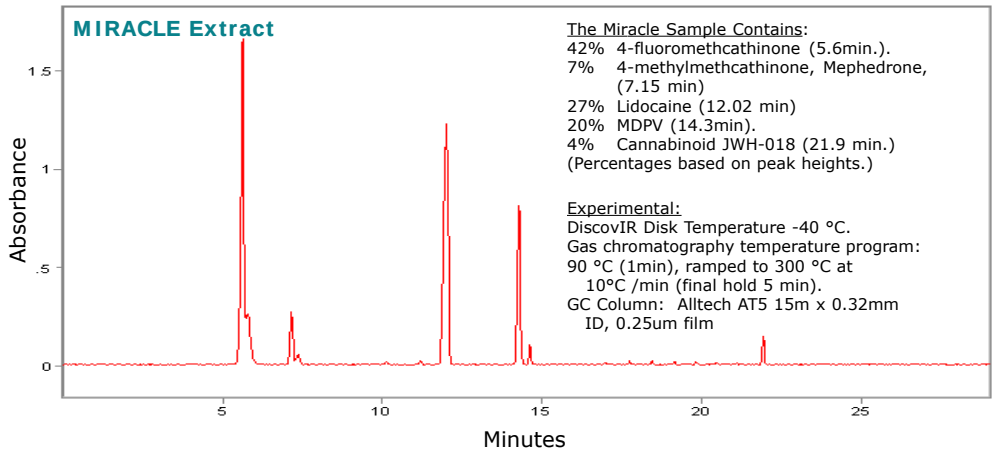
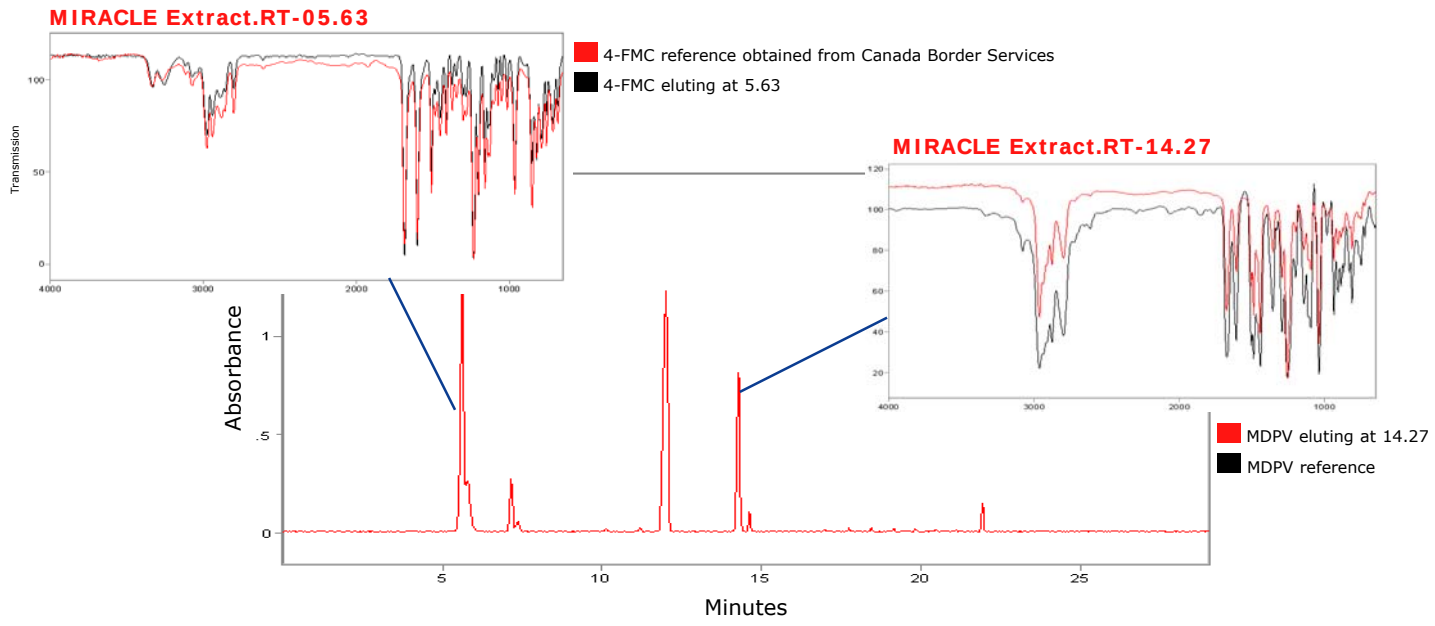


Figure 2



1. *Infrared Spectroscopy is an accepted technique by the United States Pharmacopeia because of its selectivity. See USP General Chapter <197>.*
2. *Bath salts called "Miracle" was first dissolved in water (4.7mg for 1ml of water). The solution was basified with NH4OH (28%, 2 drops). An extraction with $CHCl_3$ (1ml) was performed to recover the organic materials.*
3. *Spectral ID tool and GRAMS software are Trademark of Thermo Scientific.*
4. *Europol-EMCDDA Joint Report on Mephedrone section 3.1 Page 6.*
5. *R.P. Archer, Fluoromethcathinone, a new substance of abuse, Forensic Science International Volume 185, Issues 1-3, 10 March 2009, Pages 10-20.*

Spectra Analysis

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