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Simple Extraction and Analysis of Cocaine Freebase Vapors from Cocaine-HCl, Using SPME/IMS

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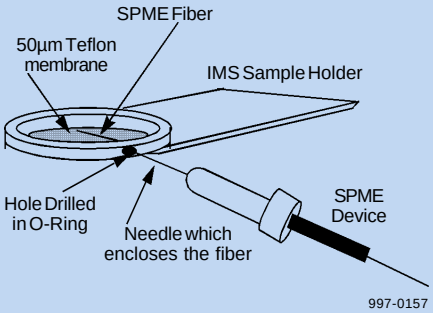
Success in combining solid phase extraction (SPE) with an ion mobility spectrometer (IMS), together with the emergence of solid phase microextraction* (SPME), led investigators at Old Dominion University to examine the possibility of combining SPME with IMS to detect cocaine freebase (pure cocaine) vapors from cocaine-hydrochloride (HCl). This method may be of particular interest to customs agents, coast guards, and other drug-interdiction specialists who are concerned whether there is detectable cocaine vapor produced from cocaine-HCl that is stored in confined spaces (e.g., cargo containers).

IMS has advantages over GC and HPLC when quantitative results are not critical. The IMS is a compact detection instrument that separates compounds by measuring their ion mobility through a drift tube. Compounds with a high proton or electron affinity are most easily detected, and analysis is completed within seconds. The IMS is very sensitive (can detect 1ng or lower, depending on the compound) and requires little maintenance — it is available in a variety of configurations, including hand-held models for field detection.

To use the SPME device with an IMS, a hole was drilled into the O-ring of the IMS sample ticket holder. This hole serves as the SPME/IMS interface, where the SPME needle is inserted and the fiber exposed for thermal desorption of the analytes (Figure A). A Teflon® membrane is used in the sample holder to prevent contamination of the desorber plate.

An 85µm polyacrylate SPME fiber was chosen for IMS screening because it is more resistant to higher temperatures than the other fiber suitable for this analysis, a Carbowax® fiber. (It is estimated that the temperature of the fiber, while in the IMS for desorption, reached 200°C).

Figure A. SPME/IMS Integrated Sample Holder

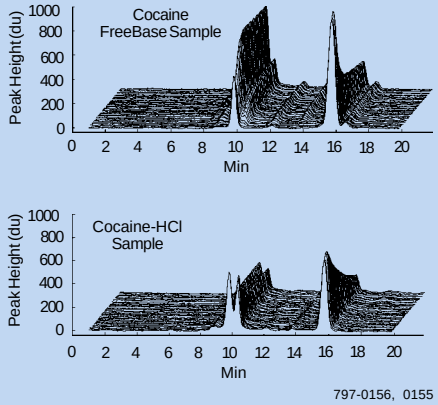


As cocaine-HCl, a salt, converts to cocaine freebase, a semivolatile solid, SPME extracts the cocaine freebase from the vapors. The polyacrylate fibers were exposed separately to the headspaces of a cocaine freebase standard sample and a cocaine-HCl standard sample. The samples were sealed and the fibers were exposed overnight, at ambient temperature (heating the sample during adsorption will decrease the necessary exposure time). Little difference can be seen in the cocaine peak intensities of the two samples (Figure B). The three-dimensional plasmagrams shown in Figure B represent scans over time.

The low intensity peaks (at close to 14ms) represent background from the SPME fibers. The fiber used in the cocaine freebase extraction had been used extensively prior to this analysis, and the background peak is noticeable. A relatively new SPME fiber was used in the cocaine-HCl extraction, therefore, the background peak decreased appreciably. The peaks near 12ms may be due to a decomposition product, methyl ecgonidine, often found in cocaine IMS plasmagrams, and the larger peaks at 10ms are calibrant peaks, which are common in IMS plasmagrams.

Figure B. Semiquantitative Analysis of Cocaine Vapors by SPME/IMS

Sample: 10µL
SPME Fiber: **polyacrylate, 85µm**
Cat. No.: **57304**
Extraction: headspace, 19 hours
Det.: IMS



Sampling times for SPME extractions typically are only minutes. For this analysis, sampling time can be decreased by increasing the temperature during extraction. Shorter sampling times at ambient temperature result in less pronounced cocaine peaks. Of more importance may be the ability of SPME to discretely sample concealed packages, and the field-portability, high-sensitivity, and simplicity of this method.

Researchers also performed a number of experiments, with headspace SPME/IMS, that easily followed the course of cocaine and cocaine-HCl reactions. The use of SPME/IMS resulted in semiquantitative analysis, and unequivocal identification of cocaine and its reaction products.

Ordering Information:

Description	Cat. No.
85µm Polyacrylate SPME Fiber, pk. of 3	
Manual sampling	**57304**
Autosampler	**57305**
SPME Holder**	
Manual sampling	**57330-U**
Autosampler	**57331**
Standards, 1g, 5g, or 10g, neat▲	
Cocaine freebase	**C8912**
Cocaine hydrochloride	**C5776**

This article is adapted from the manuscript by G. Orzechowska, E. J. Poziomek, and V. Tersol entitled *Use of Solid Phase Microextraction (SPME) with Ion Mobility Spectrometry,* published in *Analytical Letters,* **30:7** (1997).

* Solid phase microextraction technology licensed exclusively to Supelco. US patent #5,691,206; European patent #0523092.

** Initially you must order both holder and fiber assembly. Holder is reusable indefinitely. Use Cat. No. 57331 with Varian 8100/8200 AutoSampler (requires Varian SPME upgrade kit, available from Varian), or with Supelco™ SPME/HPLC interface.

▲These standards cannot be obtained from Supelco. Please order from Sigma Chemical Co. (phone 800-325-3010).

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