INFICON

APPLICATION NOTE

Solid Phase Microextraction (SPME) and HAPSITE ER: Detection of Analytes in Mixtures Using SPME Survey

INTRODUCTION

Rapid, accurate identification is imperative when determining unknowns in time critical situations. The HAPSITE® ER is a portable Gas Chromatograph/Mass Spectrometer (GC/MS) designed to detect volatile organic compounds (VOCs) and low boiling point semi-volatile organic compounds (SVOCs). HAPSITE ER, when used with the Solid Phase Microextraction (SPME) Sampling System and SPME Survey method, allows the user to perform a quick analysis for unknown compounds. The SPME Sampling System expands the capabilities of HAPSITE ER to include a wider range of SVOCs.

Analytes either adsorb or absorb onto the SPME fiber based on the type of fiber selected, and may desorb at slightly different times based on the physical properties of each analyte. This allows for rudimentary separation of mixtures, and identification of analytes based on mass spectra, without utilizing the GC column. Utilizing SPME Survey, a mass spectrometer only method, the user will see identification of unknowns displayed on the front panel in minutes. SPME Survey is a useful tool for quick, tentative unknowns identification, to aid in optimum fiber selection, and to evaluate sampling parameters and sample exposure times.

For this study, a mixture of toluene and methyl salicylate was chosen to demonstrate the separation ability of the SPME Survey methodology.

EXPERIMENTAL

A 100 µm fiber coated with polydimethylsiloxane, or PDMS (Red fiber), was chosen for this analysis due to its ability for absorbing VOCs and SVOCs. The SPME fiber, contained in a fiber holder, was conditioned in the SPME Sampling System at 250 ºC for two minutes prior to sampling with the SPME Survey method SPME_Survey_Red. The sample was a mixture of one drop (~1 µL) of neat methyl salicylate and one-half drop (~0.5 µL) of neat toluene in a 40 mL vial, sealed with a PTFE septum lined cap. The fiber holder containing the Red fiber was inserted through the septum of the vial and the fiber exposed to the headspace above the sample for a very short time (~1 second). The fiber was then retracted and removed from the vial.

SPME Survey Method SPME_Survey_Red was selected. The desorption chamber temperature in the SPME Sampling System for this method was set to 200 ºC, and the HAPSITE ER was configured with high-temperature set points of 110 ºC for the valve oven and GC heated lines, and 120 ºC for the membrane for optimum analysis. After a stable baseline was established, the SPME holder was then inserted into the heated SPME Sampling System desorption chamber and the fiber was exposed. The fiber was allowed to remain in the desorption chamber until the survey indicator bar on the front panel approached the yellow range, indicating the analytes were close to saturating the detector. The fiber was quickly retracted and the SPME fiber holder removed from the SPME Sampling System. Shown below are screenshots of the analysis from the front panel.

Figure 1: Front Panel Toluene Identification

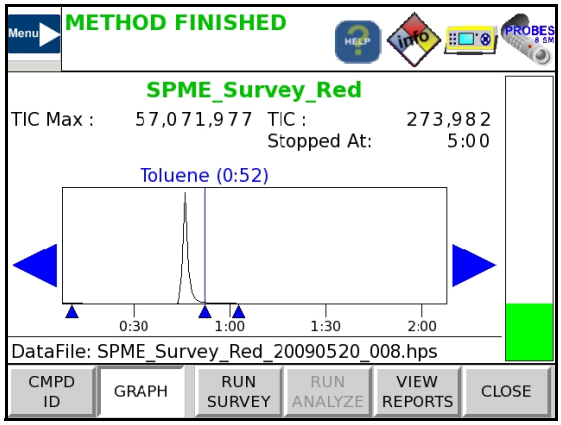


Figure 2: Front Panel Methyl Salicylate Identification

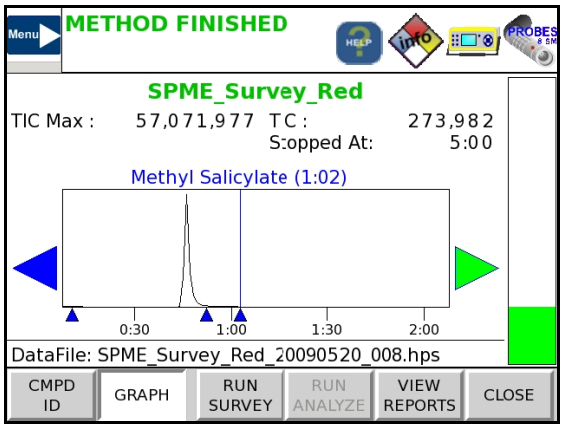
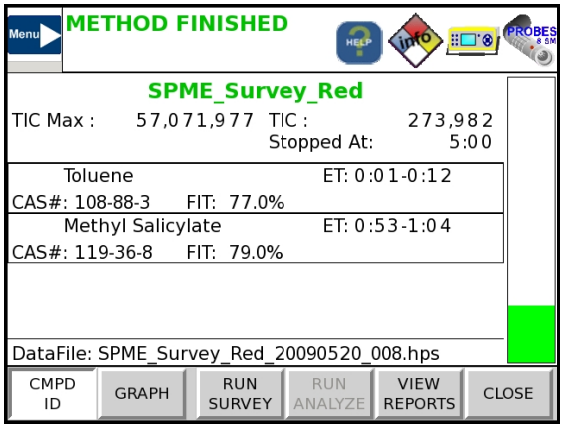


Figure 3: Front Panel Compound ID



CONCLUSION

HAPSITE ER, used with the SPME survey methodology, was able to separate and accurately identify the analytes in this mixture. Figures 1 and 2 show the AMDIS identification of these compounds directly on the front panel in real-time, giving the user the ability to make decisions based on the analytes found, sampling parameters, and fiber selection in preparation for a confirmatory GC analysis. Figure 3 shows the Compound ID.

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