Using Solid Phase Microextraction (SPME) with the TRIDION™-9 GC-TMS for trace level detection of selected Volatile Organic Chemicals in Air

SPME provides a simple, low cost methodology to collect and introduce samples to an analytical instrument. The low cost and convenience of SPME allows the analytical chemist to use the technique to deploy multiple samplers simultaneously. When coupled with the TRIDION™-9 high speed, high resolution GC-TMS samples covering a wide boiling point range can be analyzed in as little as 3 minutes per run. Methods are easily optimized for detection limits and compounds of interest.

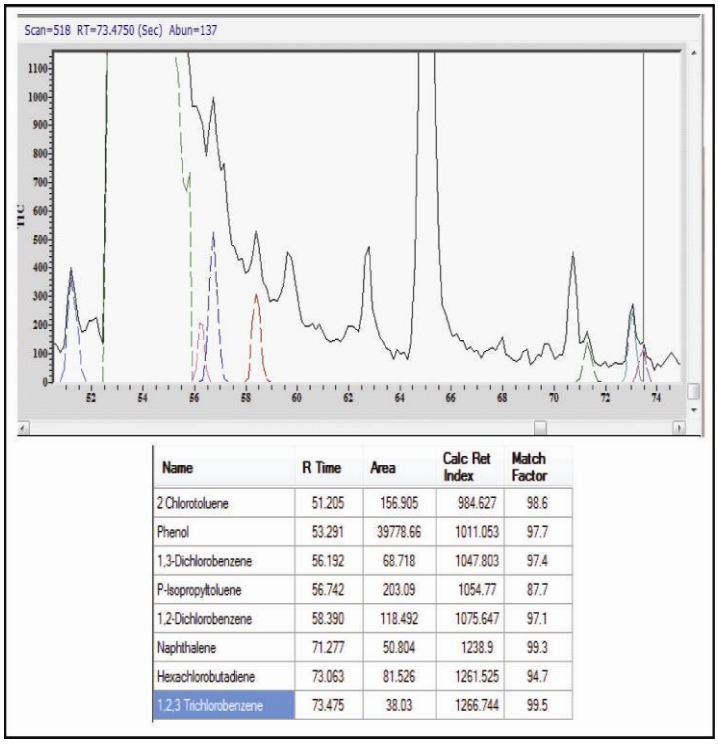
Introduction

Sample concentrations can vary greatly from locations in or near industrial environments to surrounding residential environments that are in close proximity. SPME samplers can be setup to cover a wide area, and sampling times adjusted based on anticipated concentrations. In this example the detection capabilities of the TRIDION-9 and SPME are demonstrated for a number of analytes at approximately 1 part per billion (ppb) v/v in air.

Experimental Conditions

A 10 liter Tedlar™ bag was spiked with a mixture of volatile organic compounds and then a dilution was made to generate a known vapor concentration for the analytes at around 1 ppb. Concentrations were corrected for atmos-pheric pressure and temperature using US EPA method 10-2.4. Calculations for Standard Volume; sample preparation methods are detailed in Table 1. The analytes were extracted from the gas phase at ambient temperature for 60 min using a CUSTODION™ SPME syringe with a 65 µm polydimethylsiloxane/divinylbenzene (PDMS/DVB) fiber.

Following sample extraction, the SPME syringe was inserted into the TRIDION-9 GC-TMS injection port where the target analytes were desorbed into a split-splitless injector (270°C) coupled with a low thermal mass, metal-clad, capillary GC column (MXT-5, 5 m x 0.1 mm, 0.4 µm d_f). After an initial 10 second hold at 50°C, the GC temperature was increased at 2°C/sec to 270°C for a total run time of 1 min and 30 sec. The capillary GC is coupled to a TMS detector having a mass range of 43-500 *m/z*.



Name	R Time	Area	Calc Ret Index	Match Factor
2 Chlorotoluene	51.205	156.905	984.627	98.6
Phenol	53.291	39778.66	1011.053	97.7
1,3-Dichlorobenzene	56.192	68.718	1047.803	97.4
P-Isopropyltoluene	56.742	203.09	1054.77	87.7
1,2-Dichlorobenzene	58.390	118.492	1075.647	97.1
Naphthalene	71.277	50.804	1238.9	99.3
Hexachlorobutadiene	73.063	81.526	1261.525	94.7
1,2,3 Trichlorobenzene	73.475	38.03	1266.744	99.5

Figure 1 *– Total ion chromatogram, extracted ion chromatograms, and results table*

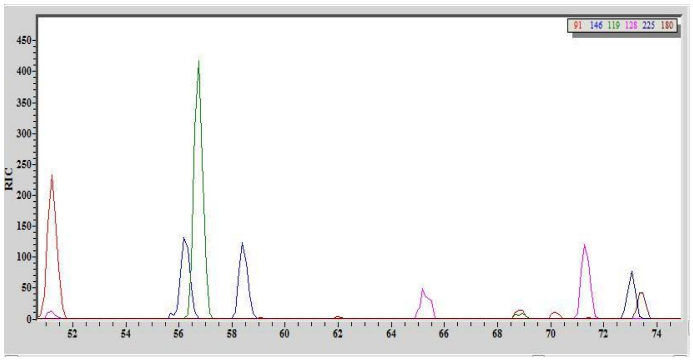


Figure 2 *– Individual extracted ion chromatograms 2-Chlorotoluene M/Z 126, 1,4 and 1,2-Dichlorobenzene M/Z 146, 4-Isopropyltoluene M/Z 119, Naphthalene M/Z 128, Hexachlorobutadiene M/Z 225 and 1,2,3-Trichlorobenzene M/Z 180.*

Results

Figure 1 shows the GC-TMS analysis of the ~ 1 ppb sample. Even with what is supposed to be a relatively clean sample, like one prepared in a Tedlar™ bag, detection and identifi-cation at trace levels can be problematic. Tedlar™ bags contain know contaminates N,N-Dimethylacetamide and Phenol. The target compounds where automatically identi-fied by the deconvolution software in the presence of these large interfering compounds. Figure 1 shows the total ion chromatogram and the individual ion traces for the extracted target compounds.

Conclusions

The CUSTODION SPME syringe and TRIDION-9 GC-TMS are uniquely suited for rapid onsite analysis. The combination of the ease of use, low cost of SPME and the high speed analysis of the TRIDION-9 GC-TMS allows for an unprecedented number of samples to be processed. This high speed analytical capability provides the information when and where it is needed to make in field decisions. Wide concentration ranges can be accommodated with SPME by simply adjusting the sampling time.

Acknowledgements

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