

# New fully automated device for sa

## Polar airborne organic compounds in air



Figure 1a: System for automated SPME operation

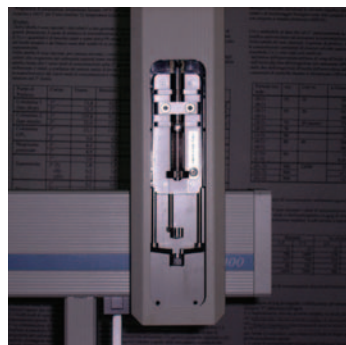


Figure 1b: Holder with plunger/magnetic system

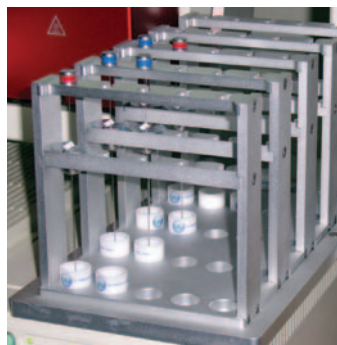


Figure 1c: 25-position tray

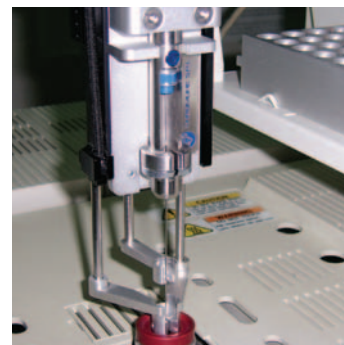


Figure 1d: SPME injection

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The authors present a method employing sequential mixed mode by automated Solid-Phase Micro Extraction (SPME) Multifiber System Fast GCMS, for sampling and analysis of polar airborne organic compounds after derivatization, thereby enabling the development of new sampling devices for the rapid analysis of air.

### Conditions

GCMS analyses were performed using a Shimadzu GCMS-QP2010 with GCMSSolution software and an SLB5-MS column (5 m x 0.1 mm ID x 0.4  $\mu$ m film thickness, Supelco, Sigma-Aldrich) in EI (Electron Ionization) mode. Oven settings were 45 °C hold for 1 min, with a ramp of 150 °C/min up to 100 °C and 50 °C/min up to 300 °C, maintained for 2 min. Full automation of the procedure was achieved using an AOC-5000 autosampler (Shimadzu) equipped with an SPME Multifiber System developed by ChromLine (Alfatech SpA).

### SPME Multifiber System

With the SPME Multifiber System, the multifiber analysis sequence works in a simple way (Figures 1b-d): the fibers are transported between the 25-position tray and injector by means of the new holder equipped with a plunger/magnetic system. At the end of the analysis the desorbed fiber is moved back to the tray and the cycle is repeated.

Moreover, the autosampler parameters used for all derivatization reagents (by sample tray rotation or magnetic stirrer) allowed completion of the analysis cycle by conditioning and sealing in the Teflon septum inserted into the tray.

### SPME Multifiber System with Fast GCMS

In the present contribution the SPME Multifiber System Fast GCMS is applied to the SPME technique proposed as a passive sampler in the field and personal samplers<sup>(1,2,3,4,5,6)</sup>, both as "rapid SPME" (a completely exposed fiber for short samplings) and as "TWA SPME" (time-weighted average SPME) – the fiber is retracted inside the needle to a defined distance Z.

### Air analysis with SPME Fast GCMS

This article explores the usage of SPME Fast GCMS for the quantitative analysis of aldehydes, ketones and carboxylic acid in air by rapid and TWA sampling mode under non-equilibrium conditions with liquid and porous fibers. The method includes, prior to sampling, the

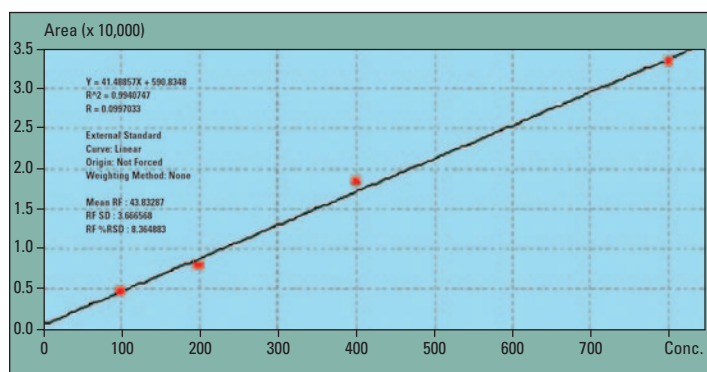


Figure 2: Calibration curve of acetaldehyde

# Sampling and analysis

adsorption of O-(2,3,4,5,6-pentafluorobenzyl)hydroxylamine on the fiber (polydimethylsiloxane-divinylbenzene) for analysis of aldehydes and ketones and the absorption of 1-pyrenildiazomethane on the fiber (polyacrylate) for analysis of carboxylic acid.

## Results

The suitability of SPME as a method for rapid and TWA-sampling of polar airborne organic compounds under non-equilibrium conditions was investigated. For TWA sampling, statistical analysis of the laboratory validations demonstrated that temperature (15 and 35 °C), relative humidity (0 and 85 %) and air velocity (0.5 and 13.9 cm/sec) did not influence the absorption efficiencies ( $p < 0.05$ ). The theoretical and experimental SR values at 25 °C correlated well. The reproducibility ( $n = 5$ ) of the retention times and the peak areas for most of the compounds was better than 6 % of retention times and 10 % of areas (Figure 2).

## Applications

Starting by the theoretical considerations which determine the passive adsorption of the analytes in a sampling SPME system, the authors of the present work have developed a system capable of analyzing great quantities of samples in a very short time, while reducing the costs of the monitoring campaigns. Aim of this system is to extend its use to all working structures, such as public and private laboratories needing quick determinations. SPME Multifiber System with AOC-5000 autosampler, designed to analyze up to 100 fibers, could be more readily accepted in industrial hygiene applications for rapid and time-weighted average determinations with sampling times ranging from 1 to 600 minutes.

It provides a simple, sensitive, fast- and reusable-, screening tool. Sampling by SPME requires no pumps and avoids organic solvents – that pollute the environment, thus cutting down time and sampling costs. Fast GCMS provides rapid separation and maintains an excellent quality of analysis (Figure 3). Further research suggests that Multifiber System Fast GCMS could also be applied to analyze other volatile organic compounds.

## References

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Compounds	Range of calibration (µg/mc)
(1) Formaldehyde	150 – 1200
(2) Acetaldehyde	200 – 1600
(5) Chloroacetaldehyde	224 – 1800
(6) Crotonaldehyde	350 – 2800
(3) Acrolein	174 – 1400
(9) Glutaraldehyde	100 – 800
(7) Valeraldehyde	160 – 1280
(4) Propionaldehyde	160 – 1280
(8) Furfural	160 – 1280

Table 1: Ranges of calibration

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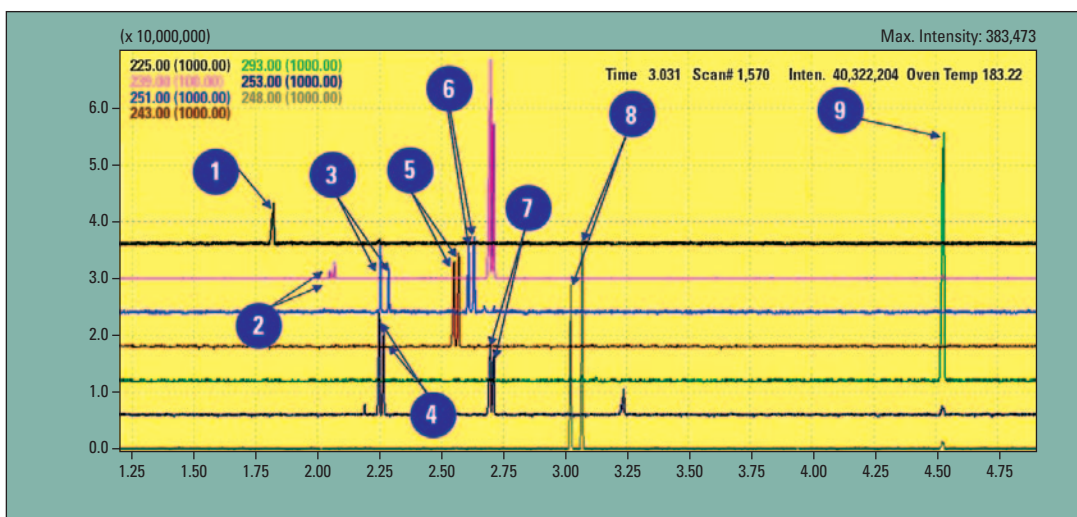


Figure 3: "Rapid" SPME Fast GCMS application with 60 sec sampling time