Released: April 2009

Application Note 037

Workplace air monitoring using pumped sampling onto sorbent tubes and analysis by TD–GC–MS

Summary

This Application Note demonstrates the sensitivity of thermal desorption (TD) and GC–MS analysis for the detection of volatile organic compounds (VOCs) in workplace air, using pumped sampling onto sorbent tubes.

Introduction

Increased knowledge of the long-term effects of exposure to VOCs at work has resulted in the tightening of legislation over the last 15 years[1–4]. At the same time, there has been pressure to reduce occupational exposure limit levels and to ensure that mean workplace air concentrations are well below these limits. Consequently, the demand for more sensitive monitoring methodology has grown dramatically.

Diffusive or pumped sampling onto sorbent tubes followed by TD–GC(–MS) has emerged as one of the most sensitive methods for detecting these VOCs, and is now one of the most popular methods for measuring VOCs in ambient and workplace air.

This has led to many new national and international standard methods for TD-based workplace air monitoring. (for a comprehensive listing, see Application Note 003).

This Application Note shows the sensitivity that can be achieved for VOCs in air using pumped sampling onto sorbent tubes and TD–GC–MS. Two examples are given, showing the relatively high VOC concentrations within a factory, and the ~100-fold lower concentrations at the factory's boundary fence.

Experimental

Sampling:
Five three-bed sorbent tubes from Markes (designed to trap a wide range of organic vapours), were used to monitor both workplace and fenceline air at a Japanese factory producing synthetic rubber.

- Two tubes (A and B) were placed inside the factory building near an emission source, and 100 mL of air at 50 mL/min pumped through them
- Two tubes (C and D) were positioned at the boundary fence, and 12 L of air at 50 mL/min pumped through them
- One tube (E) remained sealed during the entire sampling procedure and was used as a field blank.

The difference in sampled volumes was to compensate for expected concentration differences between the fenceline and workplace.

After sample collection, all five tubes were sealed and sent to the laboratory for TD–GC–MS analysis according to the following conditions.

TD:

Instrument:	UNITY™ (Markes International)
Prepurge:	1 min with split on
Tube desorption:	300 °C for 10 min (no split)
Trap desorption:	300 °C for 3 min (split on)
Trap low:	–10 °C
Trap packing:	30 mm Tenax® TA backed with 30 mm Carbopack™ B
Flow path temp.:	150 °C
Carrier gas:	10 psi
Desorb flow:	30 mL/min
Split flow:	40 mL/min
Split ratio:	20:1
Column flow:	~2 mL/min

GC:

Instrument:	6890 (Agilent Technologies)
Column:	30 m × 0.32 mm i.d. × 1 µm methyl silicone
Temp. program:	40 °C (5 min), then 10 °C/min to 280 °C (2 min)

MS:

Instrument:	5973 MSD (Agilent Technologies)

Results and discussion

Figures 1–3 show the TD–GC–MS analysis of the four sample tubes and the blank tube. Major components are identified.

By analysing all the tubes on a calibrated TD–GC–MS system, it was possible to calculate the mass of every analyte retained on each tube and to use this, together with the volume of air sampled, to derive the atmospheric concentrations.

The factory-sampled Tubes A and B produced almost identical results, with vapour concentrations of *ca.* 0.25 ppm. The fenceline-sampled Tubes C and D likewise produced very similar chromatography, though in this case, vapour concentrations were about 100-fold lower (*ca.* 2 ppb).

In both cases a number of minor compounds were also identified – predominantly chlorinated hydrocarbons and aldehydes.

Repeat desorption of all the tubes produced a clean chromatogram, indicating complete recovery in a single desorption. The repeat desorption of Tube A is shown in Figure 1 as an example.

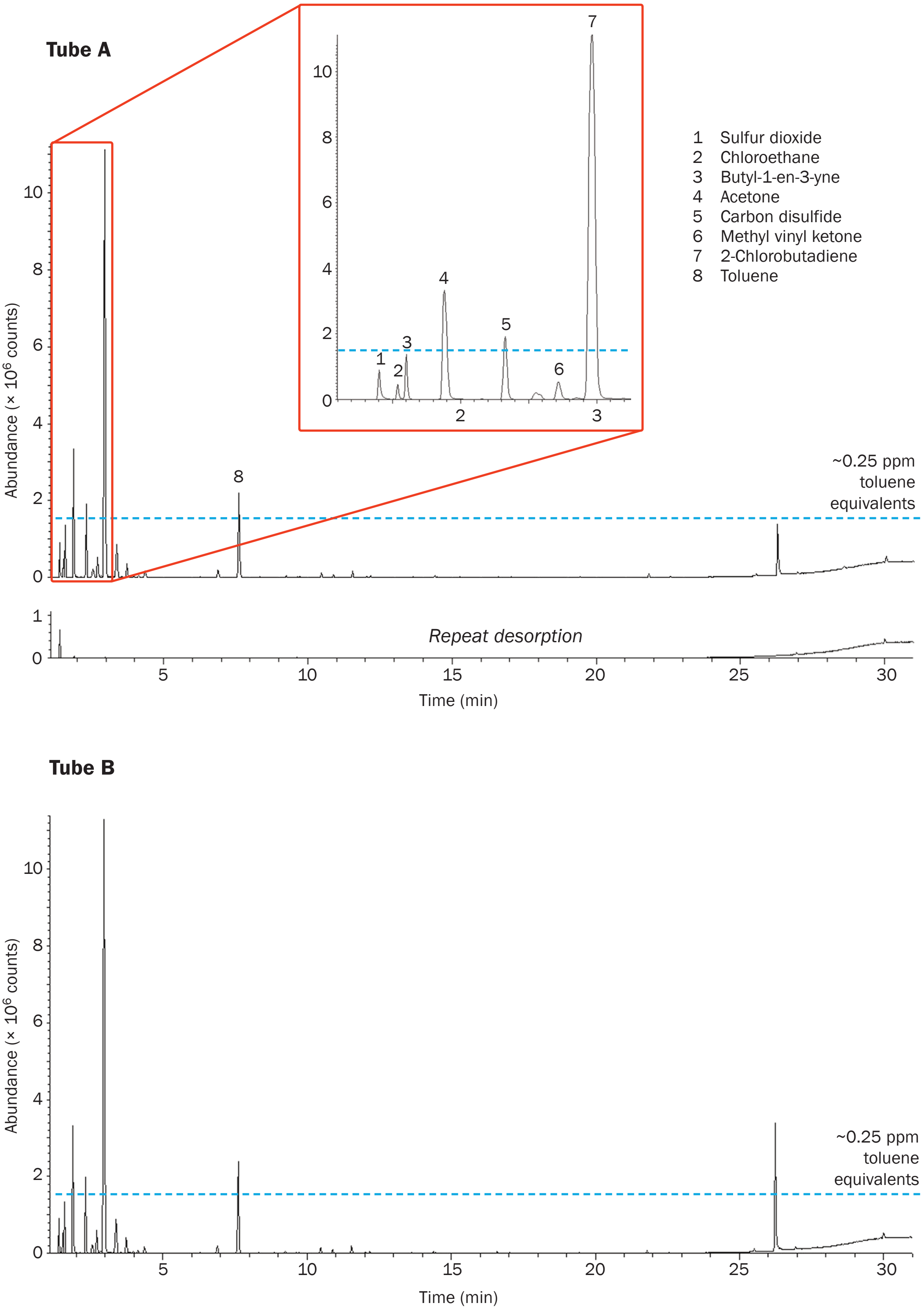

Figure 1: VOC profiles of factory air, sampled onto sorbent tubes, with analysis by TD–GC–MS. Major compounds are identified, and the result of a repeat desorption for Tube A is shown, indicating complete extraction of analytes in the first analysis.

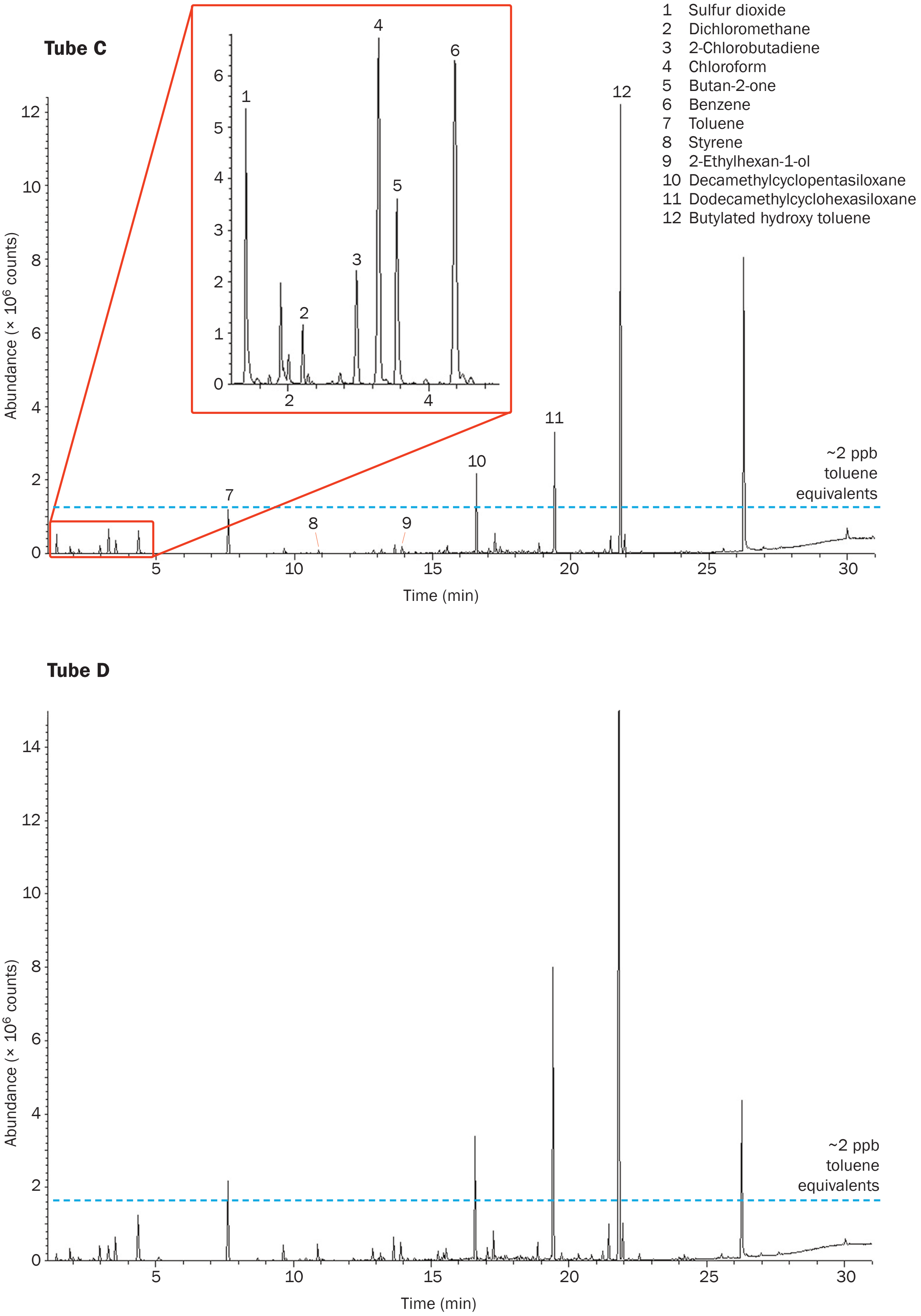

Figure 2: VOC profiles of fenceline air, sampled onto sorbent tubes, with analysis by TD–GC–MS. Major compounds are identified.

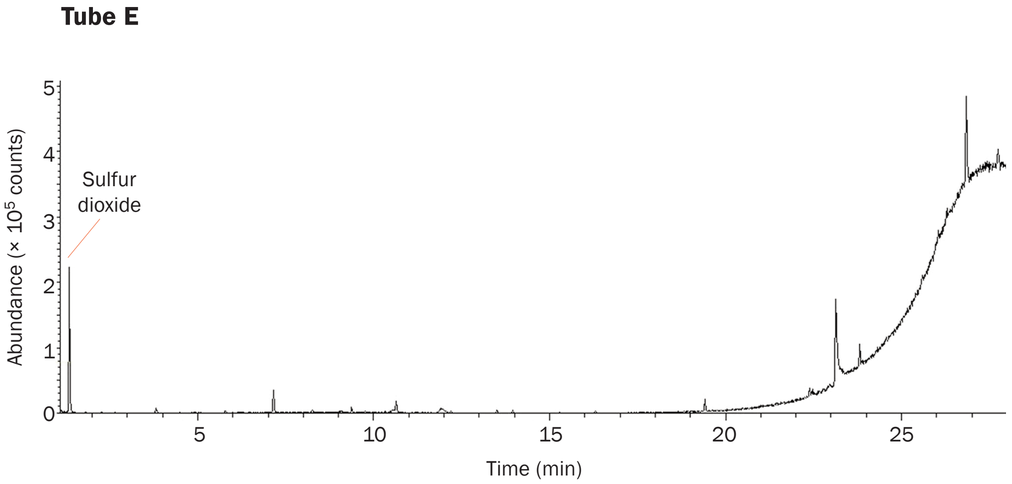

Figure 3: VOC profile of a shipping blank, with analysis by TD–GC–MS.

References

1. Risks related to chemical agents at work (Directive 98/24/EC), European Commission, 2001.
2. European Directive on the REgistration and Authorisation of CHemicals (REACH), EC 1907/2006, European Commission, 2006.
3. US Occupational Safety and Health Standards – Air Contaminants (Standard number 1910.1000), US Department of Labor, 2005.
4. 3rd Indicative Occupational Exposure Limit Value (IOELV) Directive Update, European Commission, 2007.

Trademarks

UNITY™ is a trademark of Markes International.

Carbopack™ is a trademark of Supelco Inc., USA.

Tenax® is a registered trademark of Buchem B.V., The Netherlands.