Thermal Desorption Technical Support

Note 37: Workplace Air Monitoring using Pumped Sampling onto Sorbent Tubes and Thermal Desorption

Keywords:

International standard methods, factory fence-line, carbon disulphide (CS_2), chlorinated hydrocarbons, aldehydes

Introduction

Diffusive or pumped sampling onto sorbent tubes followed by thermal desorption (TD) - GC(/MS) analysis is one of the most versatile and well accepted methods for measuring vapour phase organic compounds (VOCs) in ambient and workplace air.

Increased knowledge of the long term effects of exposure to VOCs at work has resulted in the tightening of legislation over recent years[1-4]. Concurrently, there has been continuous pressure to reduce occupational exposure limit levels and to control mean workplace air concentrations well below these limits to minimize risk. As workplace air concentrations have fallen (see TDTS 38), traditional workplace air monitoring methodology - charcoal tube with CS_2 extraction – no longer offers the detection limits required, and demand for more sensitive monitoring methodology has grown.

Thermal desorption offers 1000 times more sensitivity than traditional charcoal/CS_2 methods and overcomes many other inherent limitations – CS_2 toxicity, variable extraction efficiency, solvent interference, *etc*. (see TDTS 46). This has led to the recent development of many new national and international standard methods for TD-based workplace air monitoring. Key examples include:

- EN ISO 16017 parts 1 and 2
- ASTM D 6196-03
- EN 838 and EN 1076
- NIOSH 2549
- UK HSL Methods for the Determination of Hazardous Substances (MDHS) 72 & 80

For a more comprehensive listing of TD standard methods for workplace air monitoring see TDTS 3.
This application describes the sampling and analysis of a workplace environment using pumped sorbent tubes combined with method-compliant TD-GC/MS analysis.

Method

Five 3-bed “universal” sorbent tubes from Markes International Ltd., designed to trap a wide range of organic vapours, were used to monitor both workplace and fence-line air at a synthetic rubber producing factory. Two of the tubes (labeled PW1 and PW2) were placed inside the workplace near an emission source and two additional tubes labeled (AT1 and AT2), were positioned at the boundary fence. 12 L of air was pumped through tubes AT1 and AT2 (at the boundary fence) at a rate of 50 ml/min and 100 ml of air was pumped through tubes PW1 and PW2 (workplace) also at a rate of 50 ml/min. The difference in sampled volumes was to compensate for expected concentration differences between the fenceline and workplace. A fifth 3 bed sorbent tube remained sealed during the entire sampling procedure and was used as a field blank.

After sample collection, all five tubes were sealed and sent to the laboratory for TD-GCMS analysis using a Markes UNITY™ thermal desorber linked to an Agilent 6890 GC and 5973 MS. The analytical conditions are given below.

UNITY conditions

Prepurge Time: 1 min with split on
Tube desorption: 300°C for 10 mins - no split
Trap desorption: 300°C for 3 mins - split on
Trap low: -10°C
Trap packing: 30 mm Tenax TA™ backed with 30 mm Carbopack B™
Flow path temperature: 150°C
Carrier gas pressure: 10 psi
Desorb flow: 30 ml/min
Split flow: 40 ml/min
Split ratio: 20:1
Column flow: ~2 ml/min

GC conditions

Start Temperature: 40°C for 5 mins
End temperature: 280°C for 2 mins
Ramp rate: 10°C/min
Column: 30 m x 0.32 mm i.d. with 1 µm methyl silicone phase

Results

Figures 1-4 show the TD-GCMS analysis of each of the four sample tubes and figure 5 shows the TD-GC/MS analysis of the blank tubeMajor components have been identified in each case.

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 respective atmospheric concentration.

PW1 and PW2 (workplace) produced almost identical results with vapour concentrations averaging in the order of 0.25 ppm. Tubes AT1 and AT2 (fence-line) produced very similar chromatography, though in this case, given the larger sample volume collected, vapour concentrations averaged around the 2 ppb level.

A number of minor compounds were also identified – predominantly chlorinated hydrocarbons or aldehydes.
Repeat desorption of all the tubes produced a clean chromatogram indicating complete recovery in a single desorption. The repeat desorption of tube PW1 is shown in Figure 6 as an example.

References

1. European Chemical Agents Directive, 98/24/EC (2001)
2. European Directive on the REgistration and Authorisation of CHemicals (REACH), EC 1907/2006 (2006)
3. US, Occupational Safety and Health Standards – Air Contaminants, DOL 29 CFR 1910.1000 (2005)
4. 3rd Indicative Occupational Exposure Limit Value (IOELV) Directive Update, European Commission, 2007

Trademarks

UNITY ™ is a trademark of Markes International Ltd, UK.
Tenax TA® is a registered trademark of Buchem B.V., Netherlands.

Carbopack B™ is a trademark of Sigma-Aldrich Co., USA.

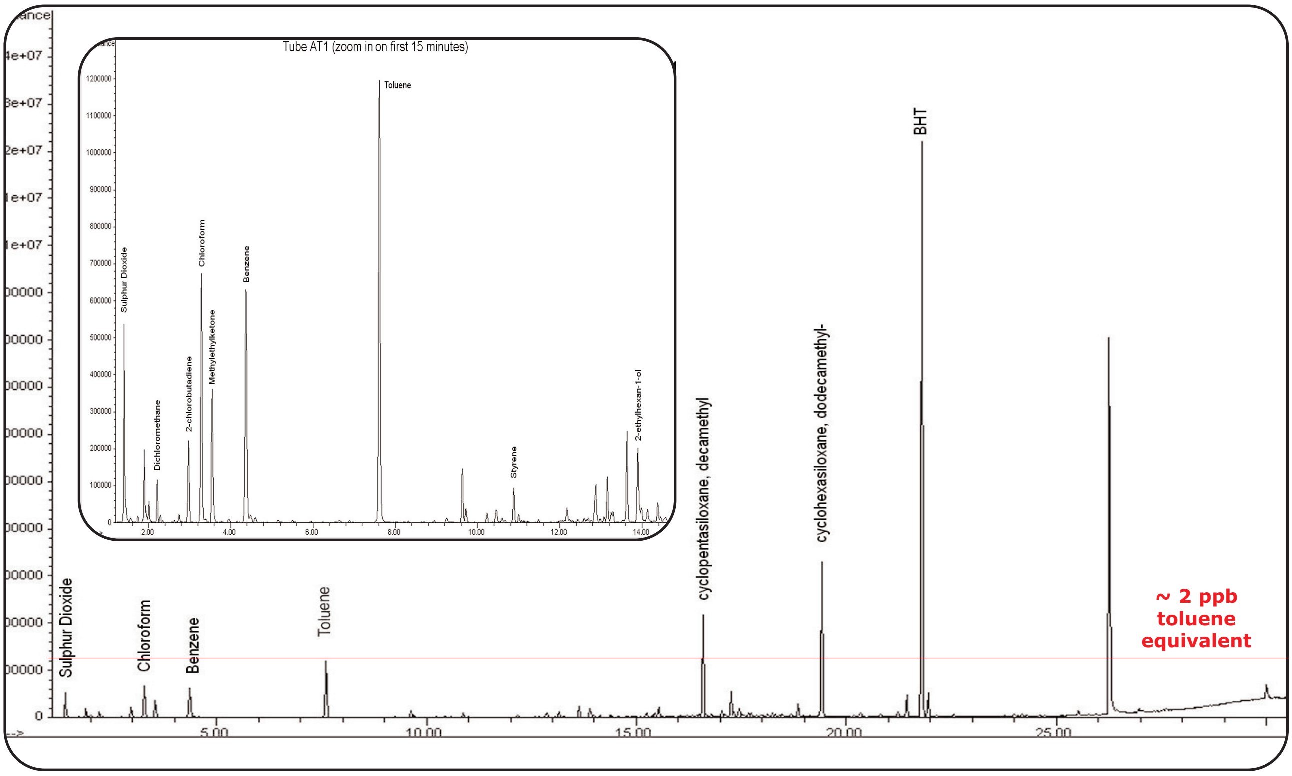

Figure 1: Desorption of tube AT1 (Boundary fence air). N.B. insert shows a close up of the results obtained over the first 14 minutes of the GC run

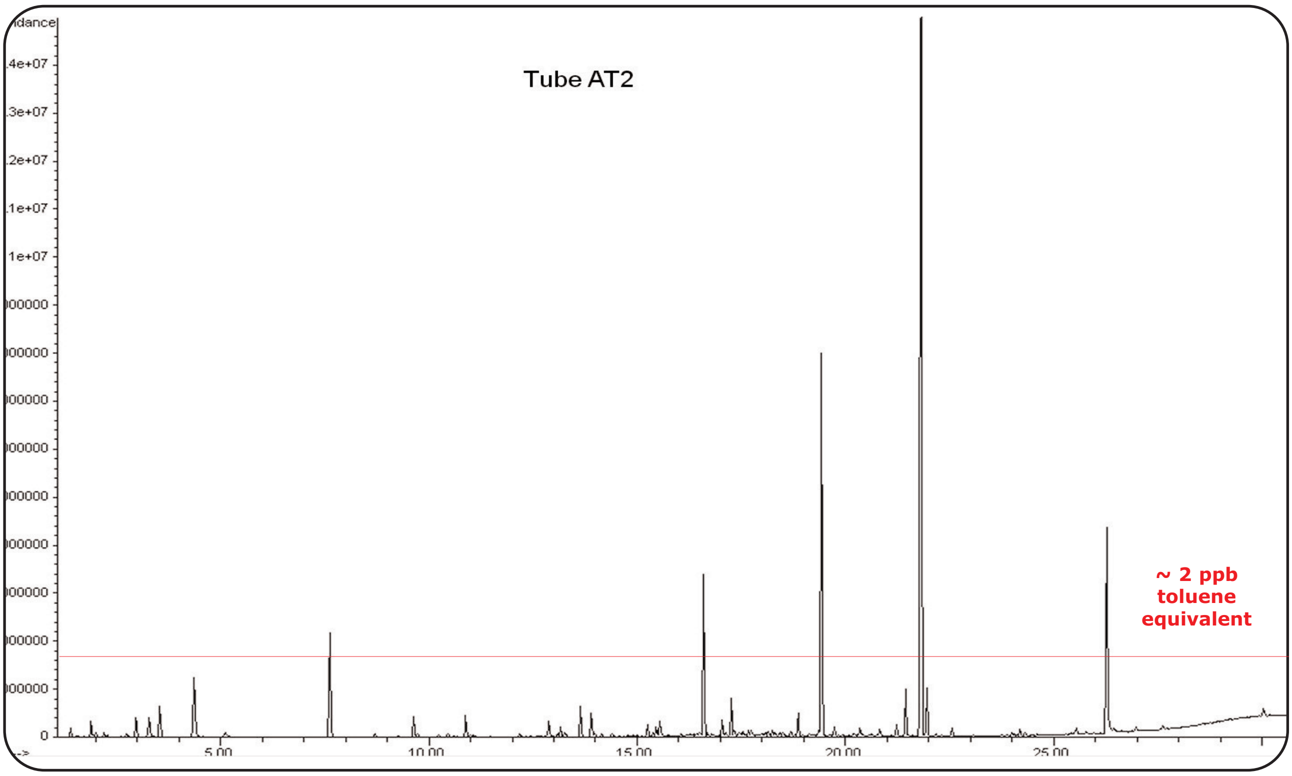

Figure 2: Analysis of tube AT2 (Boundary fence air)

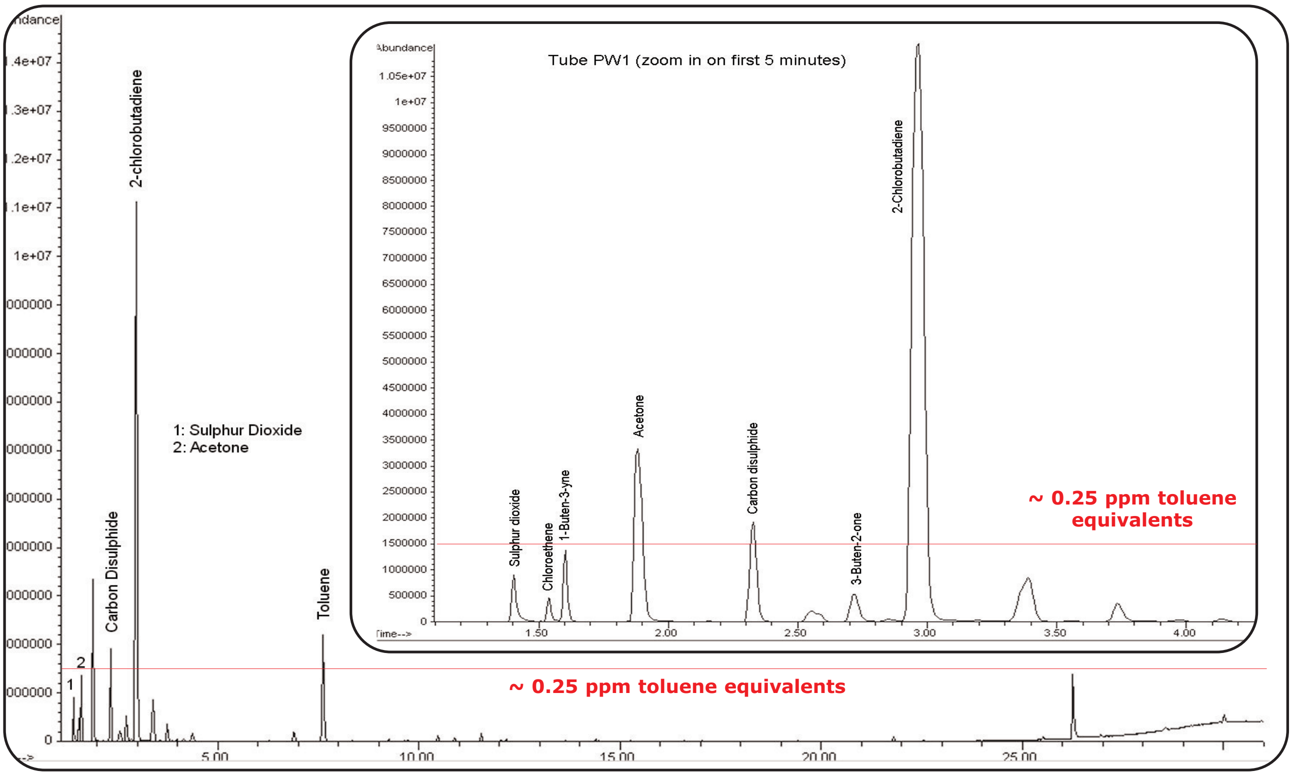

Figure 3: Analysis of tube PW1 (factory air). N.B. insert shows a close up of first 4 minutes of the GC run

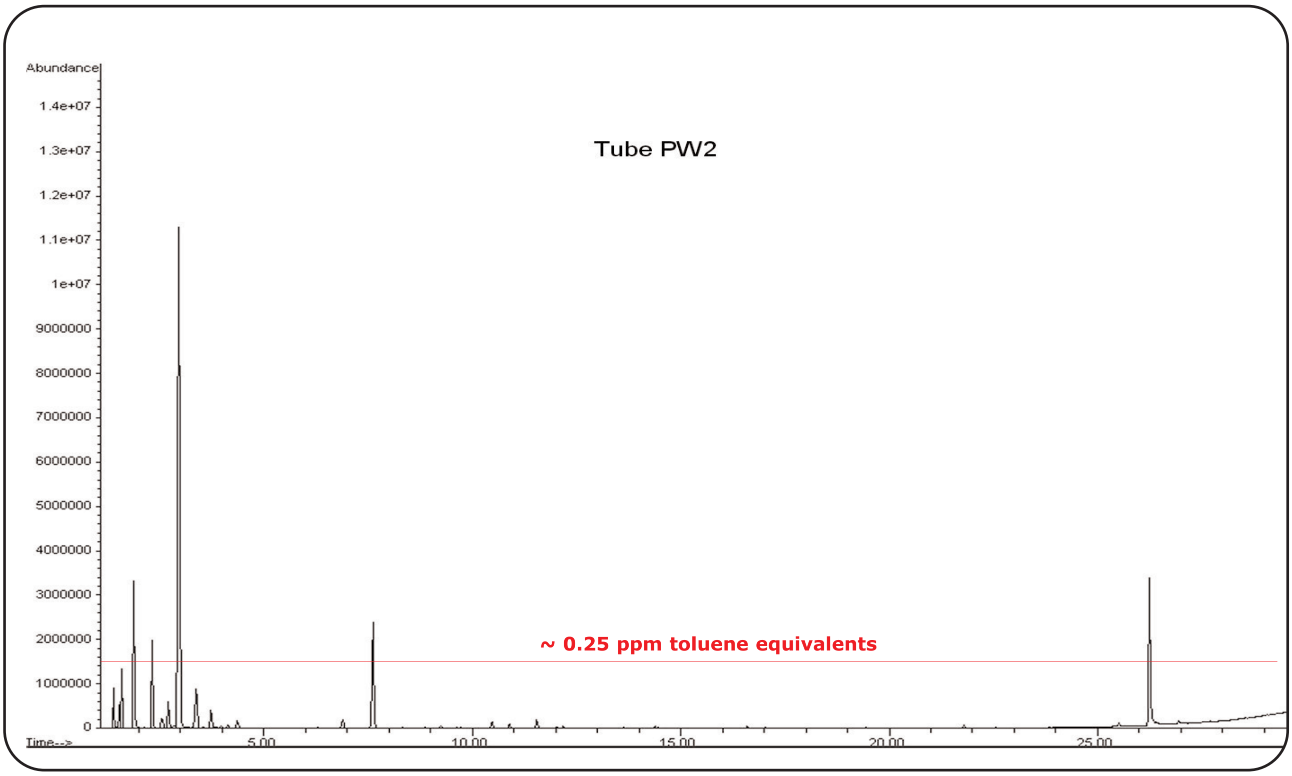

Figure 4: Analysis of tube PW2 (factory air)

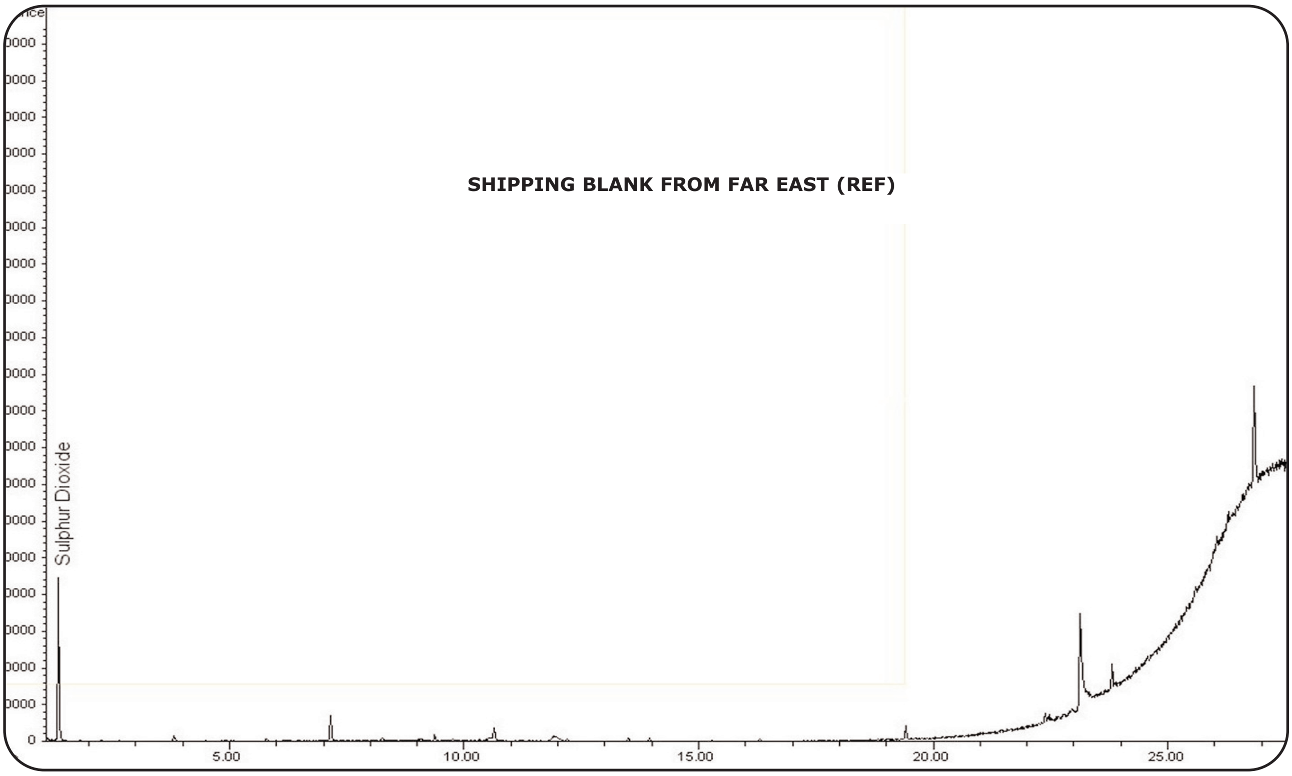

Figure 5: Shipping blank supplied with four sample tubes

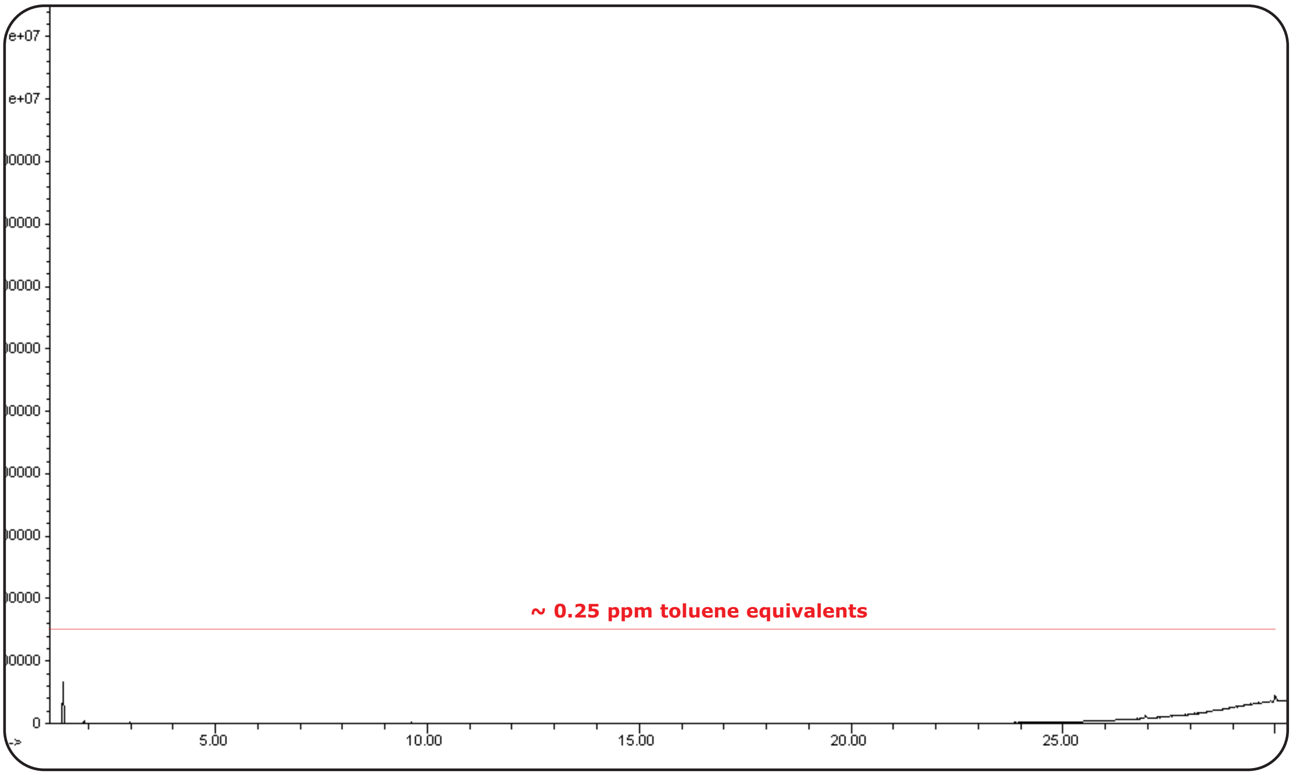

Figure 6: Repeat analysis of tube PW1 indicating complete extraction of sample from the tube during analysis