Application Note 028

Optimising analytical performance and extending the application range of thermal desorption for monitoring air indoors and inside vehicle cabins

Summary

This Application Note describes considerations relating to analyte retention efficiency, desorption efficiency, artefacts, band broadening and analyte/system stability during the development and validation of thermal desorption procedures. Examples of optimum method performance in terms of precision, linearity and sensitivity are presented.

1. Introduction

Thermal desorption (TD) is a readily automated gas extraction technology based on standard gas chromatography parameters that provides an efficient, high-sensitivity alternative to conventional solvent extraction.

The process of thermal desorption involves the extraction of volatile or semi-volatile organic compounds from a sorbent or material by heating the sample in a flow of inert gas. The extracted analytes are then transferred in the flow of carrier gas to the analyser (typically GC or GC–MS) as a small, discrete and concentrated volume of vapour. In effect the thermal desorber becomes a multi-purpose, stand-alone GC injector. Concentration factors as high as 10^5–10^6 can be achieved using modern systems, with analytes collected from several tens, even hundreds, of litres of air being delivered to the analyser in as little as 200 µL of gas.

Though inherently simple, many factors contribute to the performance and efficiency of the thermal desorption process, which in turn determines the ultimate sensitivity and reliability of a TD-based analytical method. These factors include retention efficiency (during sampling/focusing), desorption efficiency, artefacts, band broadening and analyte/system stability. This Application Note describes optimisation of all of these parameters during the development and validation of thermal desorption procedures. Examples of optimum method performance in terms of precision, linearity and sensitivity are presented. A novel approach to overcoming the traditional one-shot limitation of thermal desorption is also described.

Examples are presented that illustrate the range of indoor air related applications for optimised TD–GC(–MS). New specialist sampling apparatus and alternative real-time detection systems, which can be combined with thermal desorption to extend its utility for indoor air research, are also described.

2. The process of analytical thermal desorption

Analytical thermal desorption (TD) is essentially a gas-phase introduction technique for vapour-phase analytical systems such as GC and GC–MS. It combines many pre-analytical procedures - sample preparation/collection, selective concentration, analyte extraction and injection – into one labour-saving automated operation. It is invariably used for the measurement of trace VOCs. However, the range of sample types and applications is very diverse:

- Air/gas streams can be sampled and analysed on-line on a semi-continuous basis for real-time studies – diurnal variation of indoor air pollution, real-time tracer gas studies, *etc.*
- The volatile content of solid and liquid samples (paints, textiles, *etc.*) can be measured by weighing the material into empty sampling tubes for extraction and quantitative analysis by direct TD–GC.
- In other cases, vapour phase samples can be collected off-line into containers (canisters, bags, *etc.*) or sorbent tubes. Relevant applications for this latter approach include exhaust from material emissions tests, indoor air pollution profiling, studies of building ventilation using tracer gases and odour characterisation.

Whichever primary sampling device is used, volatiles are ultimately swept by carrier gas into a secondary focusing device inside the thermal desorber where target analytes are selectively retained. Once all the compounds of interest have been transferred to the focusing device and all unwanted volatiles (*e.g.* water) have been swept to vent, the focusing device is thermally desorbed extremely fast in a reverse flow of inert gas. This process 'injects' target compounds into the analyser in a tiny, concentrated 'slug' of vapour.

3. Sampling options

3.1 Containers – canisters and bags

Containers, such as passivated canisters or Tedlar® bags, are the best air sampling option for ultra-volatile chemicals such as C_2 hydrocarbons. Though expensive, evacuated canisters also provide the simplest of all air sampling options, with 'grab' sample collection by release of a single valve. However, Tedlar bags offer limited storage stability (<24 hours) for many common VOCs[1]. Canisters are similarly prone to poor recovery of less volatile or more polar species[2] and when used for

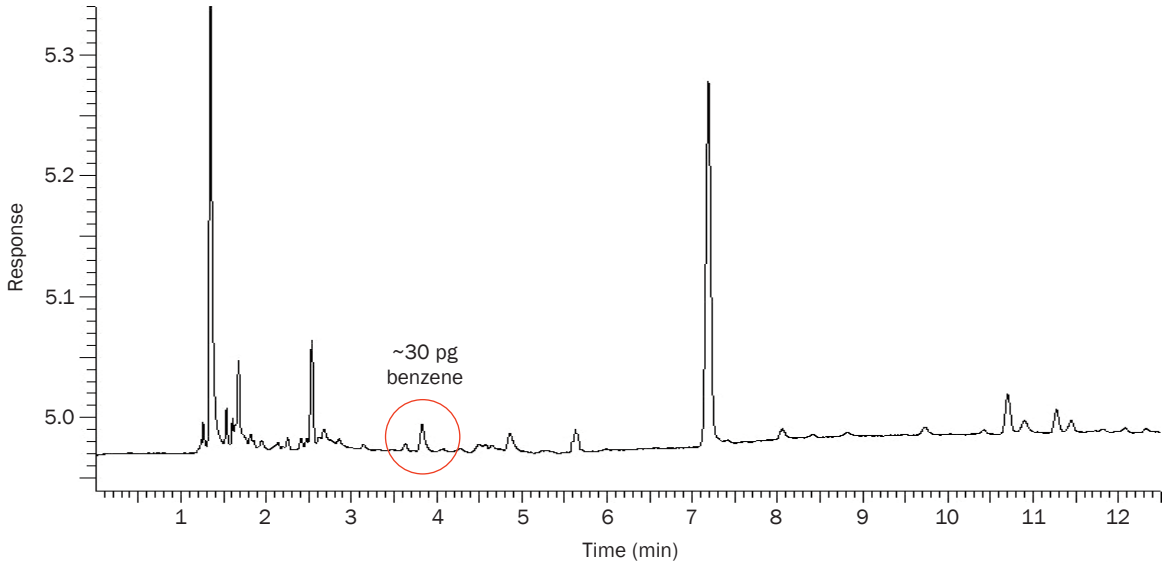

Figure 1: TD–GC–FID analysis of 30 pg of benzene from a sorbent tube, equivalent to 10 ppt in 1 L of air.

higher-concentration atmospheres (many polar or less volatile species tend to stick to the internal walls of containers and cannot be quantitatively recovered). Humidity can also cause problems – if liquid water condenses inside the container, organic compounds, particularly the more polar species, will partition between the aqueous and vapour phases, giving non-reproducible results. Tedlar bags are typically one-use only. Canisters can be reused indefinitely but require stringent cleaning, involving repeated evacuation and purging, between uses.

3.2 Vapour sampling onto sorbent tubes

While no single sampling method suits all indoor applications, thermal desorption sample tubes perhaps provide the most versatile option. They can be used either empty, for desorbing volatiles from materials (see below), or packed with sorbent for retaining vapour-phase organics. They are compatible with all but the most volatile species, *e.g.* acetylene, ethylene and some freons. Typically a known volume of air is pulled through the tube at around 50 mL/min using a standard pump. Alternatively, industry-standard steel or coated steel tubes, which have a well-defined, fixed air gap between the end of the tube and the sorbent sampling surface, can also be used as diffusive (passive) samplers. In this mode, tubes are simply left open at the sampling end allowing analytes to migrate from the air to the sorbent at a rate controlled by the diffusion gradient according to Fick's first law.

Many of the limiting factors dictating the performance of sorbent tube based air sampling methods (pumped or diffusive) are related to sorbent selection and preparation.

4. Optimising analytical performance for methods based on sorbent tubes

4.1 Sorbent selection

- **Sorbent strength** – Analytical sensitivity and precision are largely determined by sampling efficiency, desorption efficiency and the level of interferences (see bullet-point on artefacts below). The sorbent or sorbents selected must be sufficiently strong to retain target analytes during sampling/concentration, but weak enough to release them efficiently during the thermal desorption phase.
- **Concentration enhancement potential** – Depending on the retentive strength (breakthrough volume) of the sorbent tube selected for the analytes in question, as much as 100–200 L of air can be pumped onto a sorbent tube during sampling. During primary (tube) desorption the compounds of interest can be eluted in as little as 100–200 mL of carrier gas and transferred to the focusing trap. Using new, state-of-the-art, fast desorption traps, analytes can subsequently be desorbed from the trap and transferred to the analyser, splitless, in as little as 200 mL of gas. This produces an overall concentration enhancement of 10^6.

  Exceptional detection limits can be achieved, even with conventional FID detection (Figure 1).

A wide range of sorbents are now available commercially, and these can be classified generally as weak, medium or strong. Less volatile analytes should be trapped on weaker sorbents and the more volatile on stronger sorbents. If a wide volatility range of compounds is to be monitored, it is often necessary to pack the tube with more than one sorbent material, arranged in order of increasing strength from the sampling end. At ambient temperatures, sorbents can be used to quantitatively retain compounds as volatile as vinyl chloride and propane. Electrically-cooled sorbent focusing traps can concentrate components as volatile as acetylene and the most volatile freons without additional cooling from liquid cryogen.

Other key considerations when selecting sorbents include:

- **Inertness** – Some sorbents are contaminated by chemically active materials such as trace metals from the production process. This is especially true of carbon blacks, many of which derive originally from natural charcoals.
- **Hydrophobicity** – Most common weak and medium strength sorbents are very hydrophobic, thus their sorbent strength is not compromised even when sampling at high (>90%) humidity. However, most strong sorbents comprise some sort of carbonised molecular sieve and their sorbent strength can be reduced by as much as a factor of 10 at very high humidity. If a large amount of water is retained on the tube this can also adversely affect the analysis, though most modern desorption systems incorporate one or more methods for selectively eliminating water.
- **Artefacts** – Sorbents vary significantly with respect to inherent artefact levels. Some porous polymers such as the Chromosorb® Century series and PoraPak™ sorbents have relatively high artefacts, with several peaks at 5–10 ng levels. Tenax® TA is better with minimum levels between 0.1 and 1 ng for well conditioned materials. Both carbon blacks and carbonised molecular sieves are excellent with respect to inherent artefacts – between 0.01 and 0.1 ng if well conditioned. However, the carbonised molecular sieves require extended conditioning at steadily increasing temperatures and can continue to show a high background of inorganic gases for several days when new.
- **Temperature stability** – Most sorbents, including Tenax TA, are stable up to 350°C, and many of the carbon sorbents can be taken much higher. Care must be taken with most of the other porous polymers (Chromosorbs and PoraPaks), which typically have temperature limits at or below 225°C. This means that they cannot be packed into mixed-bed tubes with more stable sorbents. If this was done, it would not be possible to adequately condition the higher-temperature materials without overheating the polymers.
- **Mechanical strength** – Carbon blacks are extremely friable and prone to the formation of fines. Care should be taken not to over-compress these sorbents during tube packing and to avoid sharp knocks to the tubes once they are packed. As the carbon packing ages, the formation of fines may increase tube impedance beyond the limit of some pumps. Most other sorbents are mechanically strong, though Tenax TA can have a high percentage of fines when new and may require sieving before use. Generally speaking, recommended mesh sizes for sorbents in standard 4–5 mm bore sampling tubes range from 30–80 mesh. For further information on sorbent characteristics, please refer to Application Note 005.

4.2 Pumped monitoring/active sampling through sorbent tubes

Pumped monitoring is the most versatile sampling option for packed tubes, being compatible with both single-bed and multi-bed sorbents. It is specified by a number of international standard methods relating to indoor air. These include prENV 13419 and ISO DIS 16000 for testing emissions from building materials and US EPA Method TO-17, ISO 16017 Part 1 and ASTM D6196-97 for general monitoring of VOCs in indoor air. Industry-standard 3½″ tubes with ¼″ o.d. can be sampled efficiently at rates ranging from 10 to 200 mL/min, with the optimum being 50 mL/min [3].

'Universal' tubes – There is, of course, no such thing as a truly universal tube. However, perhaps the most useful combination of sorbents that can be packed into a single tube for pumped monitoring of uncharacterised atmospheres is Tenax TA backed up by Carbopack 1TD backed up in turn by UniCarb™ or Carboxen™ 1000. The main limitation of this sorbent combination is that the middle-strength carbon black sorbent is not completely inert, and may cause degradation of labile analytes such as nitrogen- or sulfur-containing compounds and some monoterpenes. The ultimate 'train' of tubes for monitoring uncharacterised areas comprises three inert inert-coated tubes connected together in series using inert, non-emitting fittings – the front tube packed with Tenax TA, the middle one packed with Chromosorb 106 and the back one packed with UniCarb. Whenever using these or any multi-sorbent combination of tubes, sampling must always take place through the weaker sorbent first. Higher-boiling analytes are thus retained by (and desorbed from) the weak sorbent without coming into contact with the stronger sorbents behind.

4.3 Diffusive sampling

- **Axial samplers** – Single-bed, ¼″ o.d., 5 mm i.d. stainless steel or inert-coated stainless steel sorbent tubes with a 15 mm air gap between the surface of the sorbent and the sampling surface are used as standard for pumped monitoring (see above). However, these tubes were also designed, as long ago as 1979[4] to be used as axial-format diffusive samplers. The uptake rate of many common solvents is already well validated for these tubes. Diffusive sampling eliminates the expense and relative complexity of sampling pumps and facilitates large-scale air monitoring campaigns at affordable cost. The diffusive sampling rate is a constant function of atmospheric concentration, as predicted by Fick's law (Figure 2), provided the concentration at the surface of the sampler remains at zero. For use as diffusive samplers, the tubes are capped with special diffusion caps at the sampling end and kept sealed at the non-sampling end. The diffusion caps simply have a gauze surface to stop macro-particles from entering the tube and masking the sorptive surface.

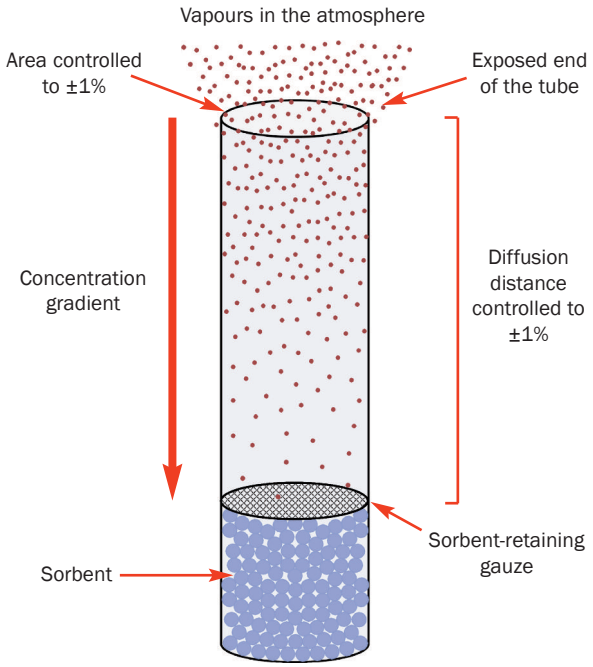

Figure 2: Axial diffusive sampling onto sorbent tubes.

Typical sampling rates on axial diffusive tubes are quoted at around 2 ng ppm^{-1} min^{-1} (2 pg ppb^{-1} min^{-1}), which is equivalent to a pumped flow of 0.5–1 mL/min. Standard sorbent tubes may be used in diffusive mode for both short-term monitoring (1–8 hours) in high-concentration areas and long-term (3 days to 4 weeks) monitoring of 'typical' indoor and ambient air.

- **Radial diffusive samplers** – Radial diffusive samplers comprise a sorbent sampling cartridge housed in a porous polymer body that allows sampling along and around the whole cylindrical surface of the sampler (Figure 3). These devices sample at a rate equivalent to 50–100 mL/min but saturate quickly. They can only be used for short-term (0.5–6 hour) air monitoring and at ambient/indoor (low ppb) levels. They are thus a useful complement to the axial diffusive tubes. After sampling, the sorbent cartridge is simply slipped from its porous polymer housing and into an empty 'carrier' tube for analysis by TD–GC–MS. The sampling cartridge is an impedance fit inside the middle of the carrier tube to ensure gas passes through the body of the cartridge during desorption. The cartridge can be reused as many times as a standard sorbent tube.

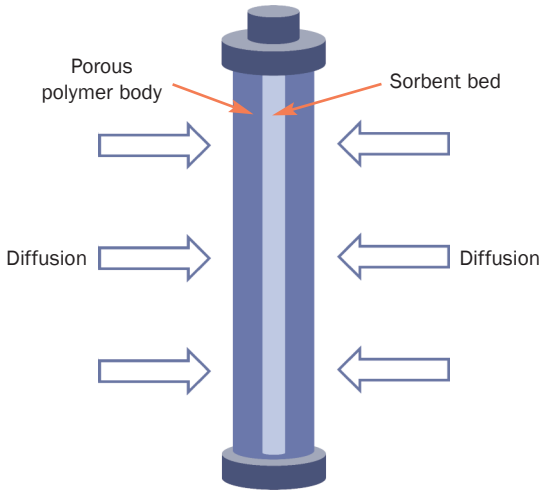

Figure 3: Radial diffusive sampling.

Diffusive sampling is specified by a number of international standard methods relating to indoor air. These include prEN 13528 and ISO 16017 Part 2 for general monitoring of VOCs in indoor air, and the Dutch standard for ventilation testing using perfluorocarbon tracer gases[5].

4.4 Conditioning and storage of sorbent tubes

Sorbents invariably require stringent conditioning at high temperatures in a flow of inert gas to clean them before use. Many of the porous polymer-type sorbents also require preconditioning in bulk before they are used to pack tubes. This is because as much as 10–15% of sorbent mass may be lost during the first conditioning cycle. It is rare for any form of solvent washing to be required, but conditions used for tube cleaning should be more stringent in terms of flow and temperature than those to be used subsequently for analytical thermal desorption.

Conditioned and sampled tubes should be stored using long-term ¼" screw caps fitted with combined PTFE ferrules. Tubes capped and stored in this way are reported to be stable for up to 27 months[6] provided the compounds concerned are not chemically active.

5. Desorption and analysis

Advice on optimising and validating the thermal desorption and GC(–MS) analysis process has been described in the literature[7] and is outlined in many of the international standard methods listed above for pumped and diffusive sampling. In summary, analytical desorption temperatures should be kept below those used for tube conditioning with a carrier gas flow of 20–30 mL/min. All air should be purged from the tubes before heat is applied and system flow paths should be kept short, inert and uniformly heated. Thermal desorption methods should easily permit recoveries better than 95% in a single desorption.

The linearity of TD–GC(–MS) methods should be the same as can be achieved using GC(–MS) with conventional liquid inlets. The precision of TD methods is typically limited to 1–2% by the manual introduction of standards to sorbent tubes during calibration. However, this is insignificant relative to the overall variability of the complete sampling and analysis procedure for indoor or ambient air. Most standards quote this at 15–30%.

As demanded by international standard methods, thermal desorption instrumentation should feature automatic leak testing and tubes that are sealed before and after analysis to maintain data integrity.

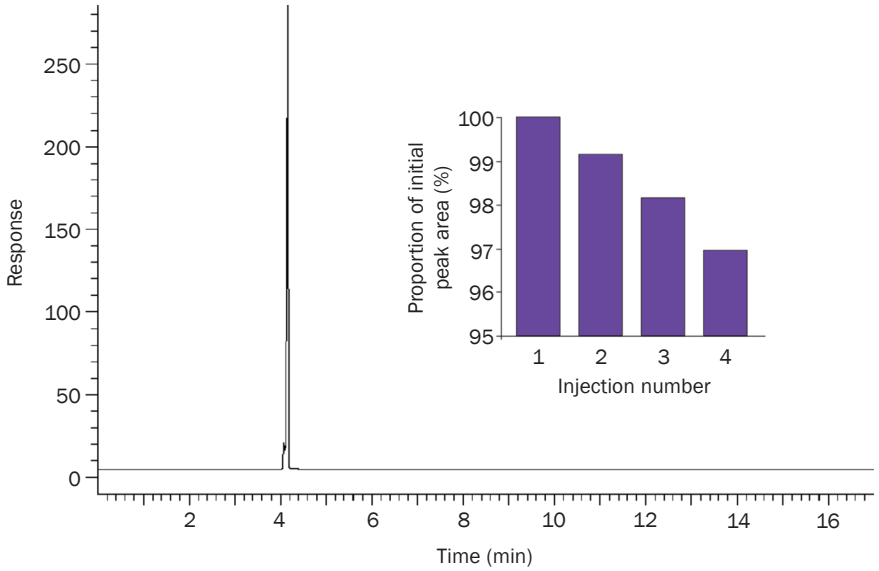

Figure 4: Re-collection and repeat analysis of desorbed toluene.

6. Repeat analysis for thermal desorption

Given its enormous advantages over solvent extraction in terms of sensitivity, reusable tubes, reproducibility and environmental acceptability, it is sometimes surprising that TD hasn't universally replaced solvent extraction for all air monitoring applications. The reason that solvent extraction is still used, especially for some industrial applications, is that more than one analysis can be carried out on each extracted sample.

Thermal desorption, on the other hand, has traditionally been a one-shot technique, that is, once a sample has been thermally desorbed – heated in a flow of inert gas – it is gone. No sample remains for repeat analysis.

However, commercial thermal desorption technology can now overcome this limitation by allowing quantative re-collection of the split effluent for repeat analysis. In all but the lowest (ppt) level applications, a small split can be used, and in some cases, for example when determining the solvent content of coatings or adhesives, a large split is often necessary for optimum analytical performance. The latest commercial thermal desorption apparatus facilitates quantitative split re-collection for repeat analysis. In these systems, the normal charcoal filter is designed to be the same size as a standard sorbent tube and is easily interchanged. The flowpath leading up to the split point is also uniformly heated and inert. For sample re-collection, the charcoal tube is simply replaced with a conditioned sorbent tube. A steep temperature gradient along the first portion of the re-collection tube ensures good retention efficiency.

The re-collected sample can be archived for analysis by a third-party lab or re-analysed immediately under the same or different analytical conditions as required. Excellent precision can be obtained (Figure 4).

7. The Field and Laboratory Emissions Cell (FLEC)

The FLEC® (Figure 5) is a specialist piece of sampling apparatus for testing VOC emissions from construction products and other materials used indoors. It is a simple, portable material emissions chamber shaped a little like the mouth of a trumpet. It is placed onto the surface of a planar material and air is passed uniformly over the surface. Exhaust gases are collected onto one or two sorbent tubes and then analysed using standard TD–GC–MS techniques.

Figure 5: The Field and Laboratory Emission Cell (FLEC) for material emissions testing.

The FLEC complies with Section 2 of the European standard for testing VOC emissions – prENV 13419. FLEC applications include emissions testing from applied wall coverings, floor coverings, adhesives, sealant materials, paints and coatings.

8. Breath sampling as a tool for investigating biological exposure

The Bio-VOC™ breath sampler (Figure 6) is a disposable device used, firstly, to collect a 100 mL sample of end-tidal air and then to transfer it to a sorbent tube[8]. Several breath samples from the same person can be loaded onto the same sample tube if required. Analysis is typically by conventional TD–GC–MS.

Figure 6: Collecting alveolar air using the Bio-VOC breath sampler.

Indoor-air-related applications for the Bio-VOC include chronic personal exposure monitoring – particularly for people living near local emission sources (*e.g.* above a dry cleaning shop) or those potentially exposed to skin-absorbed compounds, for example in areas with highly chlorinated water. Breath sampling could also be used for clinical and lifestyle studies – for example research into passive and active smoking.

9. Direct desorption

Direct thermal desorption provides a convenient and high-sensitivity alternative to purge-and-trap or solvent extraction for some relatively homogeneous materials. Samples such as powders, film, fibres, granules, resin or even droplets of liquid can be weighed directly into empty tubes or special PTFE tube liners.

The sample is then heated in a stream of inert carrier gas, stripping volatiles from the sample and transferring them to the focusing trap. In this mode, thermal desorption becomes a dynamic headspace procedure. It is used in two key ways:

- For complete and quantitative extraction of residual volatiles to determine the solvent or volatile content of a sample. Examples of materials suitable for this approach include printed polymers/papers, water-based paint, wood varnish and adhesives.
- For desorption of a representative fingerprint or profile of volatiles for characterising odour or fragrance, for example, domestic cleaning products.

For many quality control and troubleshooting applications, direct thermal desorption eliminates hours of conventional sample preparation. In many cases a three-way separation can be achieved: solids are left in the sample tube and prevented from contaminating the analytical system, and unwanted interferents such as water or ethanol may be eliminated during the focusing process. This leaves only the target organics to be collected in the focusing trap and ultimately transferred to the analyser.

Samples may be weighed directly into empty tubes supported by two glass wool plugs, or placed into inert PTFE tube liners to prevent contamination of the inner surface of the tube. If quantitative extraction is required, samples should have a relatively high surface area to mass ratio – for example film, powder, granules, liquid droplets or fibres. Resins should be smeared inside PTFE inserts or onto glass fibre 'boats' to prevent contamination of the inside of the tube.

Similarly, liquids can be introduced as droplets on glass wool plugs inside PTFE liners. The two key issues to be aware of are: (a) that the sample does not block the sample tube, because this will stop the gas flow and prevent complete thermal desorption; and (b) that the sample is not taken above its decomposition temperature. An example of paint analysis by direct TD–GC–MS is shown in Figure 7.

1 2-Butoxyethanol
2 2-Ethylhexanol
3 3,5,5-Trimethylhexanoic acid
4 Ethoxymethyl ethyl ether
5 Glycol ether
6 2,4,7,9-Tetramethyldec-5-yn-4,7-diol

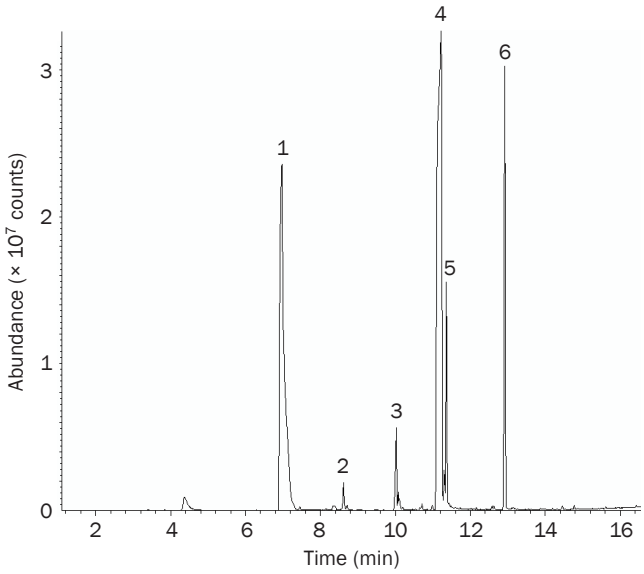

Figure 7: Direct desorption of (semi-)volatile organics from paint.

10. Out of the laboratory

In some cases, laboratory equipment and measurements do not provide the speed and turn-around required by indoor applications. GC(–MS) is predominantly a laboratory-based technology and, while it provides the most comprehensive information about a sample, it does take a significant time to run each sample and typically requires skilled technicians to operate and validate results. In a few cases the sheer quantity

of data provided by TD–GC–MS can even complicate the analysis – this is particularly true with respect to fragrance monitoring, where key olfactory components can be masked by more concentrated constituents such as alcohol.

In these situations, real-time systems – combining on-line sampling of gas or air streams with direct read-out instrumentation (process mass spectrometers, MS-sensors, 'e-nose' sensors, *etc.*) may be more suitable. On-line thermal desorption can significantly enhance the performance of real-time detectors by enhancing sensitivity, and selectively eliminating high-concentration interferents such as water or solvents.

An example is shown in Figure 8, which shows analysis of the breath of five people, two smokers and three non-smokers, collected using the Bio-VOC and transferred directly into the focusing trap of the desorber before analysis by process MS. This was tuned to monitor benzene, substituted aromatics and isoprene, the latter being a key marker for human breath. Levels of benzene and substituted aromatics are in the order of 20 ppb for smokers and noticeably higher than in the breath of non-smokers.

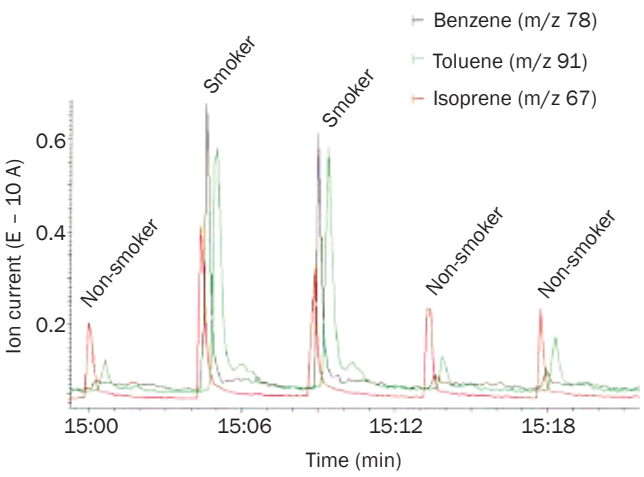

Figure 8: Analysis of the breath of smokers and non-smokers using TD and process MS, monitoring for benzene, toluene and isoprene.

Note that real-time technology usually works best for well-characterised samples, where the number and nature of possible interferents is known. This makes them suitable for routine monitoring of critical locations in many industrial or specific indoor atmospheres, but precludes their use for general-purpose environmental screening of uncharacterised atmospheres.

If applicable, the benefits of real-time systems typically include low-ppb measurements as often as once every 5 minutes and unattended (or remote) operation.

As mentioned above, the latest on-line thermal desorption technology facilitates transfer two or even three measurement systems in parallel. Thus data may be collected for a single sample on both a real-time detector and conventional GC–MS. This speeds up the development and validation of real-time measurement methods.

11. Summary of applications for thermal desorption in indoor air monitoring

Thermal desorption methods have been demonstrated to provide a high-sensitivity and robust analytical option for a wide range of applications related to indoor air monitoring. Factors affecting method optimisation have been described, and many key applications relating to indoor air have been referenced. A summary of these and other relevant TD applications is listed below with key references:

- Testing VOC emissions from building materials and furnishings[9,10].
- Indoor air profiling[11].
- Ventilation testing[5].
- VOC content of materials[12].
- Identifying indoor mould/fungal contamination via VOC profiling[13].
- Biological exposure testing[14].
- Real-time air profiling for diurnal variation studies[15].
- Vapours from burnt materials[16].
- Monitoring vapour-phase concentrations of high-boiling contaminants in semiconductor clean-room fabrication facilities[17].

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Trademarks

Applications were performed under the stated analytical conditions. Operation under different conditions, or with incompatible sample matrices, may impact the performance shown.

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