



Review

Sorbent-based sampling methods for volatile and semi-volatile organic compounds in air. Part 2. Sorbent selection and other aspects of optimizing air monitoring methods

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ARTICLE INFO

Article history:

Available online 14 January 2010

Keywords:

Air monitoring
Sorbent tubes
Sorbent traps
VOCs
SVOCs
Volatile organic chemicals
Semi-volatile organic chemicals
Thermal desorption

ABSTRACT

Sorbent tubes/traps are widely used in combination with gas chromatographic (GC) analytical methods to monitor the vapour-phase fraction of organic compounds in air. Applications range from atmospheric research and ambient air monitoring (indoor and outdoor) to occupational hygiene (personal exposure assessment) and measuring chemical emission levels. Part 1 of this paper reviewed the main sorbent-based air sampling strategies including active (pumped) tube monitoring, diffusive (passive) sampling onto sorbent tubes/cartridges plus sorbent trapping/focusing of whole air samples that are either collected in containers (such as canisters or bags) or monitored online. Options for subsequent extraction and transfer to GC(MS) analysis were also summarised and the trend to thermal desorption (TD)-based methods and away from solvent extraction was explained. As a result of this trend, demand for TD-compatible sorbents (alternatives to traditional charcoal) is growing. Part 2 of this paper therefore continues with a summary of TD-compatible sorbents, their respective advantages and limitations and considerations for sorbent selection. Other analytical considerations for optimizing sorbent-based air monitoring methods are also discussed together with recent technical developments and sampling accessories which have extended the application range of sorbent trapping technology generally.

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1. Introduction

For reasons explained in part 1 of this paper (sensitivity, automation, repeatability, etc.), thermal desorption (TD) methods are gradually superseding solvent extraction procedures for analysis of air samples collected on sorbent tubes/traps. Whole air monitoring (online or using containers) is already almost exclusively carried out using thermal desorption to extract the target organics from the sorbent focusing traps and transfer them to the GC(MS) analytical system. TD is also the method of choice for most ambient air monitoring and atmospheric research studies because of the 1000-fold sensitivity enhancement it offers when compared with solvent extraction. However, even higher level air monitoring applications such as routine industrial hygiene or fugitive emissions testing, are beginning to transfer to TD methods. The driver in this case is not usually sensitivity but rather automation and the elimination of solvents such as CS₂ which present both a health & safety hazard to operators and interfere with the subsequent GC(MS) analysis.

It is important to point out that solvent extraction can be carried out using standard GC(MS) instrumentation and that it offers some advantages versus basic TD technology in that it allows repeat analysis – e.g. for confirmation of results or repeat analysis under different conditions. However, suitable manual and automated thermal desorbers are now available from multiple commercial sources and are usually compatible with any make of GC(MS). The latest TD technology also allows repeat analysis, overcoming the one-shot limitation of older systems (see below). Moreover, lower running costs versus solvent extraction usually mean that the additional capital investment required for TD is recovered relatively quickly.

One consequence of the trend away from charcoal/CS₂ and towards thermal desorption is the demand for TD-compatible alternative sorbents—charcoal is too 'strong' and too active to allow reliable thermal desorption of all but the most volatile and stable organic compounds. A summary of the most common sorbents used for thermal desorption and the factors to consider when selecting which sorbent to use for a particular application are reviewed below.

2. Selection of TD-compatible sorbents – factors to consider

Sorbent-packed tubes and focusing traps that are compatible with thermal desorption typically contain between 1 and 4 sorbents arranged in order of increasing sorbent strength from the sampling end. There are a range of factors to consider when selecting suitable sorbents or sorbent combinations including – the strength of the sorbent–sorbate interaction, artefacts, hydrophobicity, inertness and mechanical strength (friability) [1].

2.1. Sorbent 'strength'

Analytical sensitivity and precision are largely determined by sampling efficiency, desorption efficiency and the level of interferences (see Section 2.4). 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. As described in part 1, sorbent strength is usually measured in terms of retention or breakthrough volumes. Standard air monitoring methods [2,3] are a good source of validated retention and breakthrough volume information for a wide range of common sorbent/sorbate combinations and describe how these values can be determined experimentally. Such standards may also list 'Safe Sampling Volumes' (SSVs) – derived either by halving the chromatographically determined retention volume or by reducing the experimentally determined breakthrough volume by a factor of 2/3.

Reported retention volumes are susceptible to temperature and are typically quoted at 20 °C. As a (very) approximate rule, retention volumes halve for every 10 °C rise in temperature. The performance (retention characteristics) of strong sorbents such as carbonized molecular sieves are adversely affected by high relative humidity (>80%) as recorded in standard methods. The retention volumes of hydrophobic sorbents such as carbon blacks, Tenax® TA and other porous polymers are much less sensitive to atmospheric humidity with negligible impact reported even up to 90% RH. The competitive effect of other organic vapours is also reported as negligible at levels up to 100 ppm [36].

Flow rate has been shown to have a negligible impact on sorbent strength (i.e., analyte retention volumes) provided minimum and maximum rates are observed, e.g. 10–200 ml/min for std 6.4 mm (1/4-in.) O.D. stainless steel tubes with 5 mm I.D. (see part 1 of this paper for more information).

It is important to select the appropriate sorbent or series of sorbents for the target analytes in question. If the selected sorbent is too weak (for example Tenax® TA for n-pentane or acetone) there will be a temptation to use bigger tubes/traps and larger quantities of sorbent. Oversized tubes or traps can significantly compromise air monitoring methods. In the case of whole air sampling (e.g. online or canister/bag methods) the larger the cooled focusing trap, the longer it takes to desorb resulting in slower, less efficient transfer to the analytical system. This leads to broader peaks and a consequent reduction in sensitivity and resolution. Using larger masses of sorbent for pumped or diffusive air sampling tubes makes it difficult to get good blanks – wide bore tubes (e.g. 6–10 mm I.D.) are notoriously difficult to condition stringently and are also more difficult to purge leading to increased risk of analyte and/or sorbent oxidation. If such tubes are packed with sorbent close to the sampling end, they are also prone to error due to high diffusive uptake.

Selection of sorbents of appropriate strength allows quantitative retention and release of compounds ranging from C₂ hydrocarbons and freons to semi-volatiles such as PCBs, phthalates and PAHs without exceeding optimized tube/trap dimensions and without requiring liquid cryogen coolant (see part 1 of this paper).

A wide range of weak, medium and strong commercial sorbents are now available for air monitoring (Table 1). Generally speaking vapour-phase organics should be sampled using the weakest compatible sorbent, i.e. one that offers a practical/useful retention volume and quick, quantitative recovery during desorption and analysis.

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