

Development of a Semi-Automatic System for the Spiking of Thermal Desorption Sorbent Tube with Volatile Organic Compounds for Subsequent Use as Proficiency Test or Reference Material Samples

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Background

Thermal desorption (TD) gas chromatography is a widely used analytical method for measuring airborne concentrations of volatile organic compounds (VOCs) collected using sorbent tube samplers. The technique has greatly improved sensitivity over classical solvent desorption, which allows TD samplers to be used for passive (diffusive) sampling, even in scenarios where concentrations are likely to be very low. The technique is now widely used in the ambient, chamber, fence line, indoor and workplace air monitoring environments.

The Problem

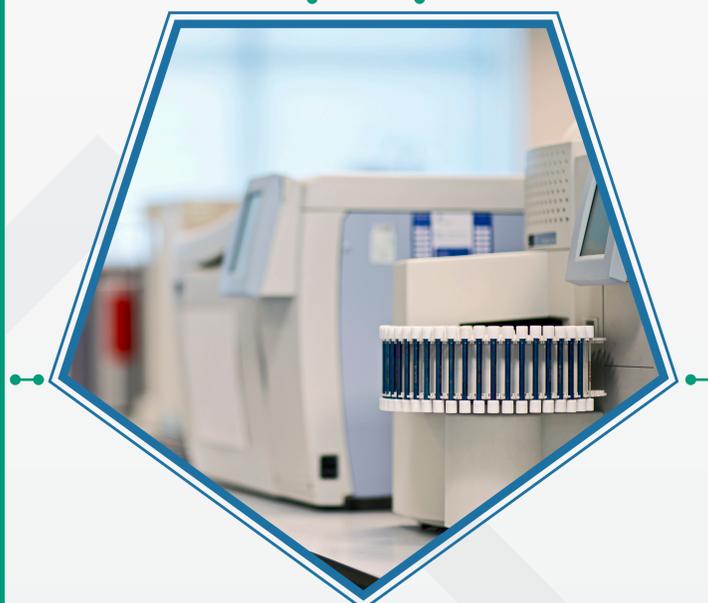
Calibration and internal quality control check samples are generally prepared by end user TD laboratories via spiking individual sorbent tubes with microliter quantities of VOCs in a carrier solvent such as methanol. There are a number of issues with this approach:

- spiking minute quantities can be an error-prone step
- residual carrier solvent may be left on the sorbent if not completely flashed off
- loss of VOCs when flashing off carrier solvent
- sorbent loading from liquid phase does not mimic loading from the gas phase.

What we did

A sorbent tube spiking system was developed consisting of the following components:

- Sample vapour generation system based upon a syringe injection approach codified in ISO 6045 part 4
- n standard atmosphere chamber that can accommodate up to 60 sorbent tubes
- Continuous monitoring of chamber concentrations via FID with feedback control
- Metered quantity of VOC enriched air through each sorbent tube via mass flow controllers
- propriety software system written in Labview™ to control all rig operations and to store run data



Outcome

This new system is now used to prepare spiked VOC on sorbent test samples for proficiency testing exercises and for the preparation of in-house QC samples. Preparation of candidate CRM is also being considered as are other "analyte on sorbent tube" samples such as mercury.

Future enhancements under consideration include the addition of new sample delivery systems e.g. Coriolis-type liquid mass flow controllers and the addition of alternative detector technologies e.g. SIFT-MS for speciated VOC measurements and Zeeman AAS for Hg measurements

Results

A sorbent tube spiking system, based upon requirements set out in ISO 6145 part 4, which is capable of loading up to 60 replicate sorbent tubes simultaneously with a typical within/between lot precision of < 0.5 % based upon flow rate measurements has been developed. All calibration and run data is logged and provided in a spreadsheet format accessible for third party audit purposes.

Manual flow meters used to control diluent gas flows are currently being replaced by more precise mass flow controllers and will be integrated into the system software. The modular design allows other sample delivery systems such as diffusion cells (ISO 6145 part 8) and permeation tubes (ISO 6145 part 10) to be used.

