



Determination of trace volatile organic compounds in air with an integrated preconcentrator/GC system

Application Note

Environmental

Authors

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Introduction

Many organic compounds that occur in ambient air are the result of emissions from mobile, industrial, or natural sources as well as landfills and hazardous waste sites. The levels of these compounds that are detrimental to human health over long time intervals are very low or at the low part per billion level and are generally regulated by national or local government agencies. The measurement of these trace levels often requires the use of a preconcentration device to trap enough material from an air sample to allow detection with chromatographic detectors.

Such preconcentration devices are often stand alone units that are interfaced to a gas chromatograph pneumatically with either metallic or fused silica transfer lines or electronically with status input and output cables. They are usually controlled from a separate PC or an external controller and therefore occupy a lot of space.

The Single Valve Air GC, described in this note, consists of a GC with a built-in sample preconcentrator trap (SPT) and a single multipart rotary valve. Fully integrated into the GC, the SPT has several major advantages over the standalone trapping systems: 1) a very short interface connection between the preconcentrator and the GC column, 2) a small footprint, and 3) total control from the GC or a PC Workstation.



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Experimental

The Single Valve Air GC preconcentrator (Figure 1) consists of the following major components: 1) the SPT, 2) 10-port valve and air actuator, 3) surrogate/internal standard option, 4) an Electronic Mass Flow controller and 5) a stainless steel diaphragm pump. A simplified version using a 6-port valve without surrogate/internal standard is possible. A wide variety of trap packing materials are available.

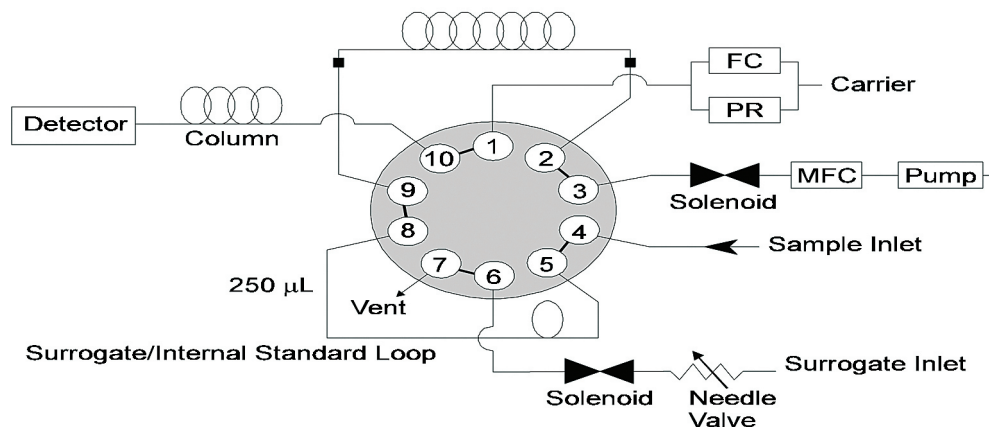


Figure 1. Single Valve Air GC Valving Diagram

An air sample is drawn from a stainless steel canister, a Tedlar bag, or directly from the source, be it the indoor air, outdoor air, or a conduit to a stack. The air sample is pulled through the SPT adsorbent or cryogenic trap for several minutes with the valve in the counterclockwise position. The valve is then switched to the clockwise position, the SPT is heated to the desorb temperature, and the sample is simultaneously backflushed to the capillary column for chromatographic separation of the trapped constituents.

Results

Several applications using the Single Valve Air GC are described. Benzene, toluene, ethyl benzene and xylenes (BTEX's) are the most commonly determined volatile organics in ambient and source gases. They are trapped on Tenax TA or carbonaceous adsorbents at room temperature and easily desorbed at moderate temperatures. One hundred milliliters of a BTEX standard was trapped on a 30 cm adsorbent trap and desorbed to a DB-1 column giving the chromatogram shown in Figure 2. The meta and para xylenes could be resolved using a DB-WAX or equivalent polar liquid phase.

Peak identification

1. benzene*
2. toluene**
3. chlorobenzene
4. ethyl benzene
5. m/p-xylene
6. o-xylene
7. styrene

* 65 µg/M³

** 76.5 µg/M³

100 mL, 20 PPBV each

SPT: Tenax TA (60/80), 35 °C Trap, 200 °C Desorb

Column: 100% PDMS, 0.32 mm x 60 m, 1.0 µm

Agilent Equivalent: CP-Sil 5 CB, CP8870 film 35 °C/5 min,

10 °C/min to 200 °C/hold

Detector: FID

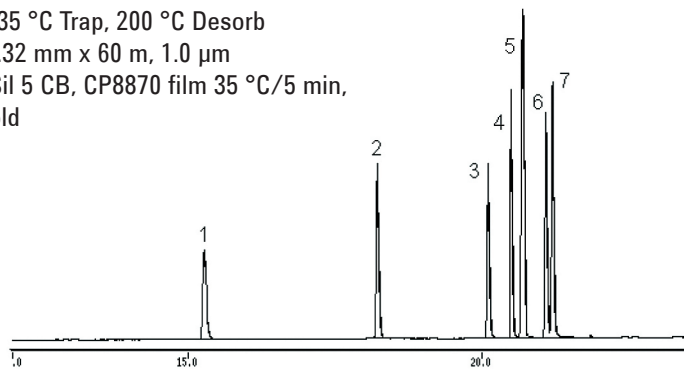


Figure 2. Aromatics with Single Valve Air GC

Hydrocarbons are measured in ambient air and automobile exhaust gases to monitor their contribution as precursors to the formation of ozone. Figure 3 shows the heavier ends of the precursors normally measured. While Tenax TA was also used in this case, carbonaceous adsorbents such as Carbotrap Band Carbosieve S-III may be used in multi-adsorbent traps with Tenax TA to trap and determine C₂ to C₁₃ hydrocarbons.

Peak identification

1. C₆
2. benzene
3. C₇
4. i-C₈
5. toluene
6. C₈
7. p-xylene
8. o-xylene
9. C₉
10. trimethylbenzene
11. C₁₀
12. C₁₁
13. C₁₂
14. C₁₃

100 mL, 40 PPBV each

SPT: Tenax TA (60/80), 35 °C Trap, 200 °C Desorb

Column: 100% PDMS, 0.32 mm x 60 m, 1.0 µm film,

Agilent Equivalent: CP-Sil 5 CB, CP8870

35 °C/5 min, 10 °C/min to 200 °C /hold

Detector: FID

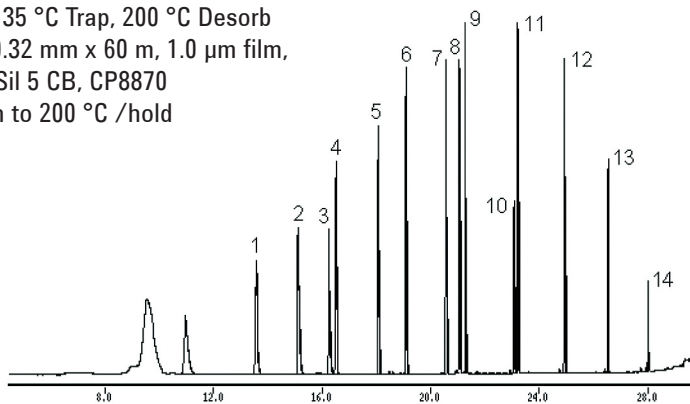


Figure 3. C₆ to C₁₃ Hydrocarbons

Ozone precursors are emitted primarily from automobile exhaust while air toxics are emitted mostly from industrial sources. These source emissions are coming under increased restriction as the US Clean Air Act amendments of 1990 (Titles III and V) and increased government scrutiny in other parts of the world take effect. The EPA method T0-14 targets 41 air toxics while the future method T0-15 raises this figure to 97 volatile organic compounds VOCs. Figure 4 shows a chromatogram of the T0-14 target list while Figure 5 displays a subset of six air toxics that serve as a test standard covering the full range of volatiles. While the previous VOC examples were concentrated on adsorbents, liquid nitrogen cryogenic trapping on glass beads was used for the T0-14 list (Figure 4).

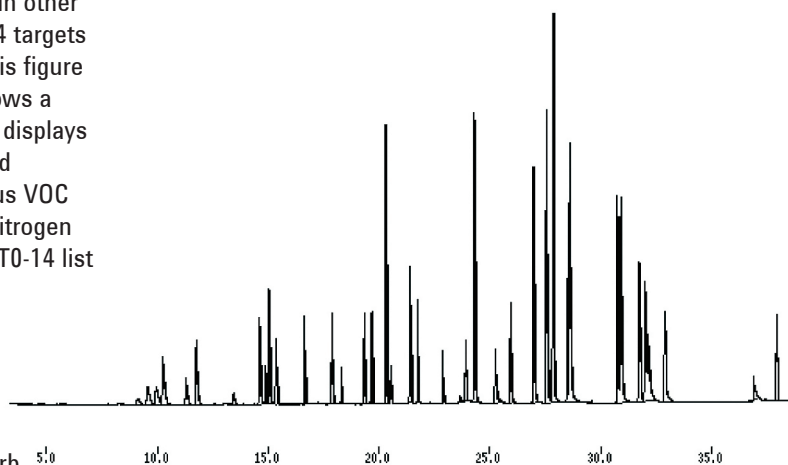


Figure 4. T0-14 Air Toxics

100 mL, 20 PPBV each
 SPT: Glass beads (60/80), -150 °C Trap, 200 °C Desorb
 Column: 100 PDMS, 0.32 mm x 60 m, 1.0 µm
 Agilent Equivalent: CP-Sil 5 CB, CP8870
 0 °C/5 min, 6 °C/min to 200 °C/2 min

100 mL, 20 PPBV each
 SPT: Tenax TA (60/80), 35 °C Trap, 200 °C Desorb
 Column: Rtx-1, 0.33 µm x 60 m, 1.0 µm film,
 35 °C/5 min, 10 °C/min to 200 °C/hold
 Detector: FID

Peak identification

1. vinylchloride
2. 1,1,1-trichloroethane
3. benzene
4. BFB
5. dichlorobenzene
6. hexachlorobutadiene

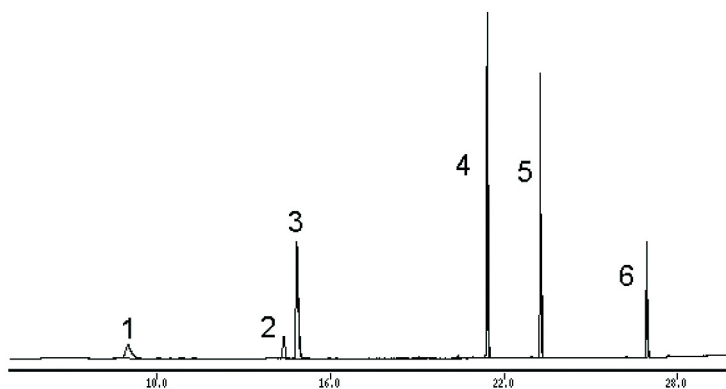


Figure 5. Air Toxics Test Sample

Conclusions

A gas chromatograph with a built-in compact preconcentration device is capable of multiple air monitoring applications ranging from ozone precursors to air toxics. Air sampling is possible from containers or directly from sources with preconcentration on either adsorbents of choice or glass beads at subambient temperature.

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