Analysis of volatile organic compounds in air and/or soil gas

Application Note

Introduction

Many volatile organic compounds (VOCs) present health hazards and/or behave as precursors to ozone formation in ambient air. Therefore, it is important to screen for their presence and determine their concentration. Many industrial and waste treatment plants must monitor the air in and around their facilities to avoid excess emissions and to ensure legal limits are not exceeded.

To achieve the method detection limits (sub parts per billion) required in air analysis, it is necessary to concentrate the sample prior to analysis. The system described here is a compact unit with the ability to concentrate the sample, separate it on an appropriate column and detect it with selective detectors. With an optional Stream Selector Valve (SSV) the system has the ability to analyze up to 16 samples, blanks, or calibration mixtures while unattended. The automation allows the operator to designate the 2 mL or 100 mL sample loop.

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System Description

Figure 1 shows a schematic of the system which contains the following:

1. Four automated valves
2. Variable-Temperature Adsorption Trap (VTAT)
3. Analytical column
4. Detectors: PID and ECD or ELCD in series

The Gas Sample valve is equipped with a 100 mL loop to accommodate low concentration samples (ambient air) and with a 2 mL loop to accommodate high concentration samples (soil gas). The second valve provides the surrogate* sample, which is injected with every run. The third valve directs the purge and carrier gas flows to the appropriate paths, while the fourth valve is used to isolate the VTAT during the preheat cycle. The VTAT makes the concentration of VOCs from large volume samples (100 mL) possible.

* Surrogate standard is one or more organics introduced with each sample, blank or standard to evaluate analytical efficiency. These are not expected to occur in environmental samples.

Figure 1. Schematic of the System
In preparation for analysis (during oven cool-down), the sample and surrogate sample loops are filled; upon injection they are flushed to the adsorbent trap (Tenax/Charcoal) which is at 30 °C. After the sample is absorbed on the trap, the trap is isolated and preheated to 250 °C. In the desorb cycle the carrier gas backflushes the hot trap, removing all the VOCs and carrying them to the column for separation. For detection, the PID is used in series with either ECD or ELCD. The combination of these detectors provides excellent sensitivity and selectivity for the VOCs. After desorption the trap is baked out and cooled in preparation for the next analysis.

The sample is trapped on an adsorbent bed at ambient temperature, allowing water vapor to pass through to vent. This is an advantage compared to cryotrapping on glass beads where water is frozen out and must be removed by a semi-permeable membrane dryer.* The latter may allow the loss of polar organics.

Figure 2 shows a chromatogram of a standard mixture containing low boiling compounds such as vinyl chloride, as well as heavier compounds, such as ethylene dibromide. Figure 3 shows a chromatogram obtained analyzing shower room air after taking a shower. Very good repeatability data (retention time <0.1% RSD, area count <5% RSD for the 2 mL loop, and <8% for the 100 mL loop) and low carryover (<0.1%) was observed. The system linearity results are shown in Figure 4. The calibration factors, being constant over the concentration range studied (0.1 to 500 ppb), show the system and detector to be linear.

* Cryogenic trapping on glass beads is possible (VTAT temperature variable from -190 °C to 400 °C)

Recommended capillary columns
Agilent CP-Select 624 CB, 0.32 mm x 30 m, df = 1.8 µm, Part no. CP7414
0.25 mm x 30 m, df = 1.4 µm, Part no. CP7412