Purge and Trap/GC/MS Analysis of Volatile Organic Compounds: Advances in Sample Throughput

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Purge and trap cycle time is the limiting factor in volatile organics analysis by gas chromatography/mass spectrometry. To overcome these time limitations, two purge and trap units, each with its own autosampler unit, can be connected to a single GC/MS. With this system, and a chromatography column designed to separate compounds that share common ions, it is possible to analyze 80 samples in 24 h.

Demand for increased productivity in volatile organics analysis (VOA) by gas chromatography/mass spectrometry (GC/MS) has resulted in the creation of automated water and soil autosamplers that enable environmental laboratories to operate purge and trap systems around the clock. Still, laboratories seek faster turn-around-time, for better customer service and return on capital equipment investments. This need has resulted in a push to develop columns that drastically reduce analysis time and instruments that accommodate short cycle times.

Currently, the limiting factor in VOA is the purge and trap cycle time: it includes an 11-min purge time followed by a 6-12 min bake-out time. A modern GC, on the other hand, can acquire a sample in 10 min or less. To overcome the time limitations of the purge and trap cycle, two purge and trap units, each with its own autosampler unit, can be connected to a single GC/MS operating system (1). The concentrators are synchronized so that while one is desorbing a sample onto the column for analysis, the other completes the bake cycle and begins to purge the next sample. A Duet® interface (Tekmar-Dohrmann, Mason, Ohio) gates the signals between the two concentrators, to prevent a faster concentrator from overtaking a slightly slower one. Calibration curves and quality control samples (QC, MS, MSD) must be run for each concentrator. A tracer compound is added to samples from one of the concentrators, to eliminate any question as to which purge and trap system provided a particular sample.

Using this system and a computer-designed chromatography column, tuned specifically for separating compounds that share common ions and typically coelute on columns intended for VOA, it is possible to analyze 80 samples in 24 h, a significant increase in output from a single GC/MS instrument. Figure 1 shows an analysis on an Rtx®-VMS column according to US EPA Method 8260B, including the specified internal standards and surrogates.

For additional information about VOA procedures, refer to (1) and to technical guide *Optimizing the Analysis of Volatile Organic Compounds* published by Restek Corporation (2).

Compounds in 5 mL of RO water.

Concentrator:	Tekmar LSC-3100 Purge and Trap
Trap:	Vocarb® 3000 (type K)
Purge:	11 min @ 40 mL/min @ ambient temperature
Dry purge:	1 min @ 40 mL/min
Desorb preheat:	245 °C
Desorb:	250 °C for 2 min, Flow 40 mL/min
Bake:	260 °C for 8 min
Interface:	0.53 mm i.d. Silcosteel® transfer line 1:40 split at injection port. 1 mm i.d. liner
Oven temp.:	50 °C (hold 4 min) to 100 °C @ 18 °C/min (hold 0 min) to 230 °C @ 40 °C/min (hold 3 min)
Carrier gas:	Helium @ ~1.0 mL/min constant flow Adjust dichlorodifluoromethane (1) to a retention time of 1.03 min @ 50 °C
Detector:	Agilent 5973 MSD
Scan range:	35-300 amu

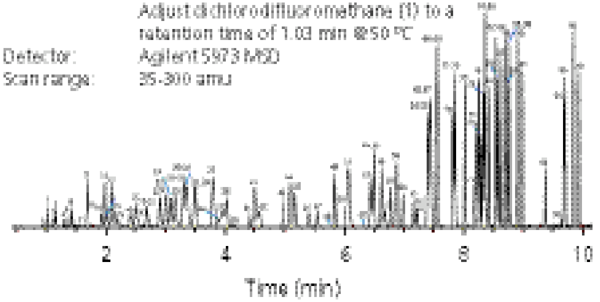

Figure 1: Volatile organics by U.S. EPA Method 8260B. Column: Rtx®-VMS (Restek Corp., cat. # 49914), 20 m, 0.18 mm i.d., 1.00 µm film; conditions listed on figure. Peaks: 30 = dibromofluoromethane (SMC), 36 = pentafluorobenzene (IS), 42 = 1,4-difluorobenzene (SMC), 50 = toluene-d8 (SMC), 64 = chlorobenzene-d5 (IS), 74 = 4-bromo-1-fluorobenzene (SMC), 88 = 1,4-dichlorobenzene-d4 (IS).

References

(1) A.L. Hilling and G. Smith, *Environmental Testing & Analysis* **10**(3), 15-19 (2001).
(2) *Optimizing the Analysis of Volatile Organic Compounds,* lit. cat. # 59887A (Restek Corp., Bellefonte, PA, 2003).

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