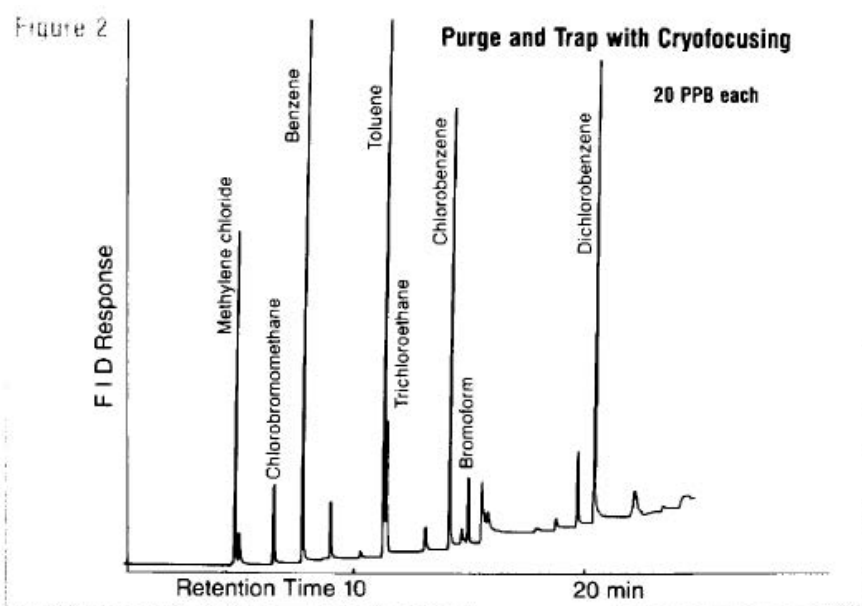
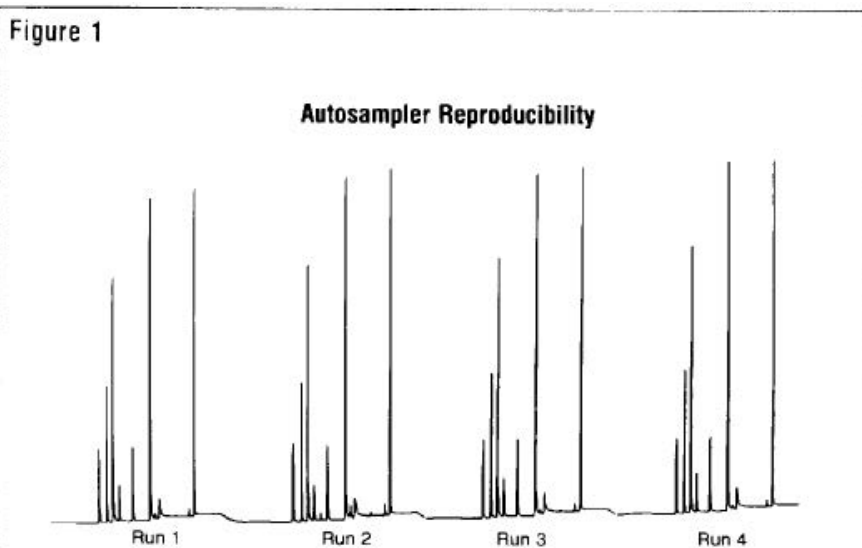


Autosampling in Purge & Trap Analysis

The analysis of volatile organic pollutants in water by purge and trap has been facilitated by advances in capillary columns specific for the technique, and by the development of automated systems for performing the analysis unattended. Analysts may now choose from packed, wide-bore capillary or narrow-bore capillary columns with or without cryogenic refocusing to improve the chromatography.

The CDS Model 380 is a completely automated system for the analysis of priority pollutants in water samples. It includes a 15 vessel autosampler and a sample concentrator which enables the system to purge the pollutants, dry the trap, transfer the analytes to the gas chromatograph, start the GC, empty the water sample and advance to the next bottle. If desired, the GC sample may be cryogenically refocused onto the capillary column.

Two of the greatest concerns in using an automated analysis system are reproducibility and sensitivity. Analysts must know that identical samples will produce the same chromatogram from each vessel of the autosampler (Figure 1). The CDS 380 interfaced to a capillary column and FID has produced chromatograms with an average RSD of <5% for ten runs of the same compounds at the 100PPB level.



Automatic cryofocusing improves the sensitivity by eliminating the need to split the sample at the injection port when using narrow-bore capillaries, and thereby permits operation in the low PPB to parts-per-trillion range. Figure 2 is a chromatogram of a variety of purgables present in a 5ml water sample at 20PPB, using a 0.75mm capillary column. The use of a 0.25mm column with cryofocusing has permitted the determination of aromatics at a concentration of less than 10 parts-per trillion.

EQUIPMENT

CDS 380 Autosampling concentrator
Purge: Helium, 40ml/min for 11 minutes

Trap: Tenax TA, 25cm
Trap Dry: 35°C for 3 minutes
Desorption: 200°C
Cryofocusing: -100°C for 10 minutes, then 200° for 5 minutes at GC Start
GAS CHROMATOGRAPH:
Varian 3700
Program: 35° for 4 min, then 6°/min to 200°
Detector: Flame ionization detector

For more information on this and related topics, we recommend the following readings:

Washall, J. and Wampler, T., "Purge and Trap Analysis of Aqueous Samples with Cryofocusing," *Amer. Lab.*, 20, 7, (1986) 70-74.

Wampler, T., Bowe, W., Higgins, J., and Levy, E., "Systems Approach to Automated Cryofocusing in Purge and Trap, Headspace and Pyrolytic Analysis," *Amer. Lab.*, 17, 8 (1988) 82-87.

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