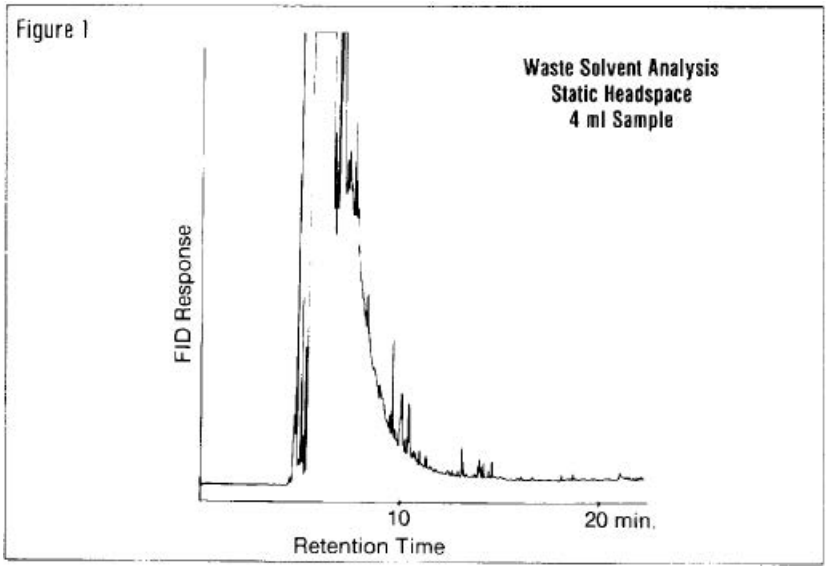
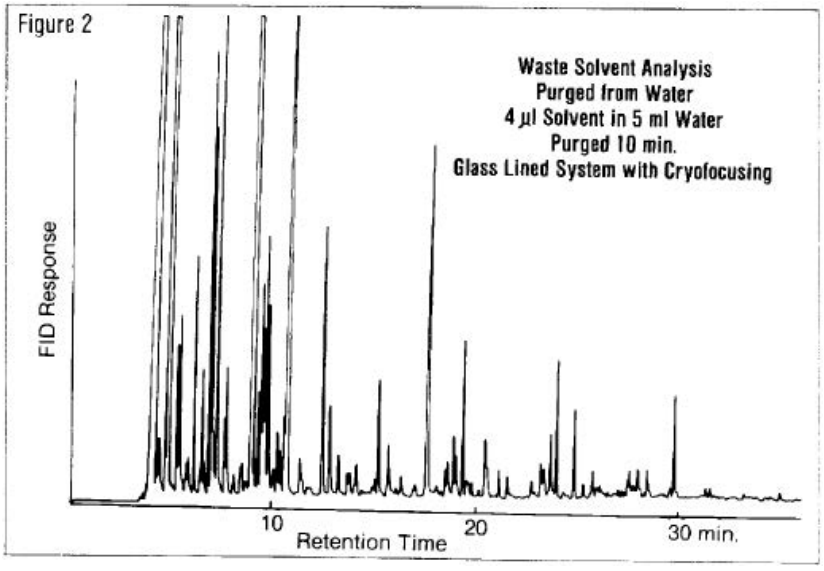
#003

Analysis of Waste Solvent by Purge & Trap

The purge and trap technique was originally developed for the analysis of organic volatiles in water samples. The process involves passing a stream of inert gas through the sample to carry away the organics, which are then trapped from the gas stream onto the surface of a trapping material, generally Tenax. Since that time, the principles involved in purge and trap analysis have been applied to a wide variety of samples including foods, polymers, pharmaceuticals, soils and arson debris.

The accompanying chromatograms concern the analysis of waste solvent. The solvent was an alcohol which was used to clean and remove other solvents, especially halogenated organics, from manufactured parts. It was of interest to determine the amounts of halogenated materials in the alcohol before disposal. Static headspace produced the chromatogram in Figure 1, for which a 10ml sample of the solvent was heated to 70°C for 30 minutes, after which a 4ml sample of the enclosed headspace was injected onto the gas chromatograph. Because the alcohol is present in such large concentration relative to the other solvents, the alcohol





peak obscured most of the compounds of interest.

Much better results may be obtained by taking advantage of the alcohol's solubility in water. For the chromatogram in Figure 2, 4)...ll of the alcohol was dissolved into 5ml of water, and then the water was purged with helium for 10 minutes. Because the halogenated solvents were less water soluble, their recovery was enhanced relative to the alcohol, most of which was left behind in the water. The organics were trapped onto Tenax, then backflushed to the gas chromatograph, where they were refocused directly onto the capillary column using liquid nitrogen. This cryofocusing provides sharp resolution of the early eluting peaks, while still permitting splitless operation for maximum sensitivity.

Equipment

SAMPLE CONCENTRATOR

COS 330 Sample Concentrator with ambient trap A (Tenax filled), cryogenic trap B, and glass lined tubing.

Purge flow: Helium, 30ml/min for 10 minutes

Ory flow: 30ml/min for 4 minutes

Trap A desorption: 250° C for 10 minutes

Cryogenic focusing: -100°c for 10 minutes

Trap B desorption: 200° C for 5 minutes

Valve oven: 250° C

Transfer line: 250° C

GAS CHROMATOGRAPH

Varian 3700 equipped with flame ionization detector

Column: 50m x 0.25mm SE-54 fused silica capillary

Program: 50°C for 2 minutes, then 6° C/min to 200° C

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

T. Bellar, and J. Lichtenberg, J. Am. Water Works Assn., 66(12): 739.

T. Wampler, W. Bowe, J. Higgins, and E. Levy, "Systems Approach to Automatic Cryofocusing in Purge and Trap, Headspace and Pyrolytic Analyses," Am. Lab., 17, 8 (1985), 82.

Grote, J., "Apparatus for Concentration of Volatile Organic Pollutants in Water," Am. Lab., 7(2): 47, 1975.

Headquarters
JSB International
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 251 47 53
F +31 (0) 40 251 47 58

Zoex Europe
Tramstraat 15
5611 CM Eindhoven
T +31 (0) 40 257 39 72
F +31 (0) 40 251 47 58

Sales and Service

Netherlands
Apolloweg 2B
8239 DA Lelystad
T +31 (0) 320 87 00 18
F +31 (0) 320 87 00 19

Belgium
Grensstraat 7
Box 3 1831 Diegem
T +32 (0) 2 721 92 11
F +32 (0) 2 720 76 22

Germany
Max-Planck-Strasse 4
D-47475 Kamp-Lintfort
T +49 (0) 28 42 9280 799
F +49 (0) 28 42 9732 638

UK & Ireland
Cedar Court,
Grove Park Business Est.
White Waltham, Maidenhead
Berks, SL6 3LW
T +44 (0) 16 288 220 48
F +44 (0) 70 394 006 78

info@go-jsb.com
www.go-jsb.com