#023

Effect of Operating Parameters on Purge & Trap Efficiency

The accepted technique for the analysis of volatile organic pollutants is purge and trap. This technique makes use of a helium carrier gas which is bubbled up through the sample releasing the organics and collecting them onto an absorbant trap which is then backflushed to the gas chromatograph. The mechanism governing the release of the organics from the water is dependent on the solubility of organics in water and their relative volatility. Research and method development must first optimize the operating parameters in order to achieve efficient, accurate results. Purge time must be long enough to ensure total release of the VOC's from the water and the trap desorption temperature should be hot enough to desorb the organics but not cause thermal breakdown.

The purge time was varied from 1 minute to 10 minutes with a trap desorption temperature of 180°C for 10 minutes. Figures 1 and 2 show the chromatograms of benzene, toluene, o-xylene and ethyl benzene at 1 and 10 minutes represents 200 PPB of each component; after reducing the purge time to 1 minute the recoveries ranged from 11.5% for benzene to 35.9% for o-xylene. A graphic representation of this data can be seen in Figure 2.

In terms of trap desorption temperature, the temperature

Figure 1 Purge and Trap Analysis of Aromatics

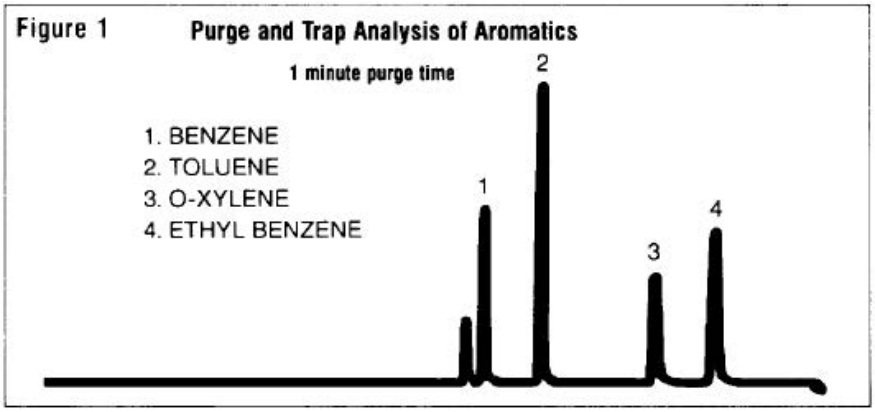


Figure 2 Purge and Trap Analysis of Aromatics

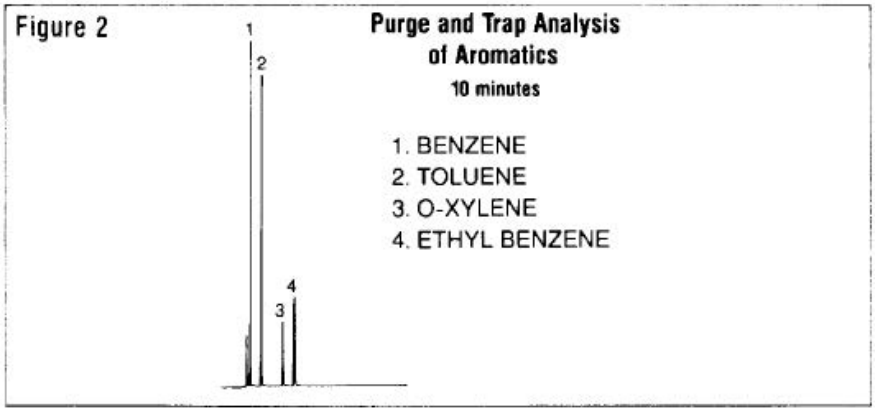
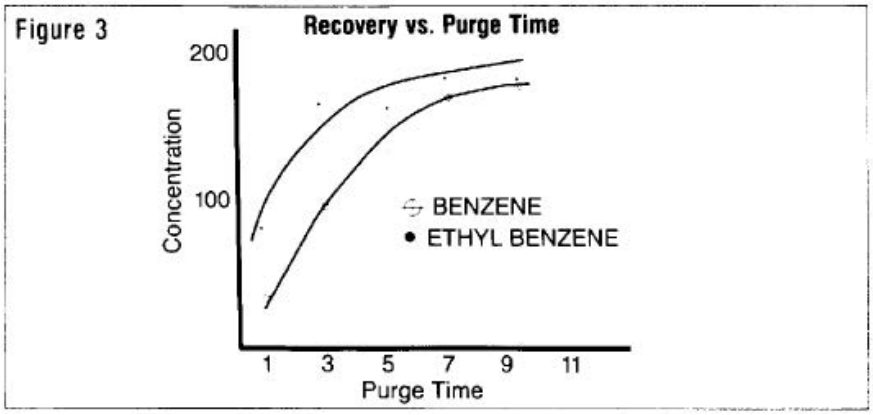


Figure 3 Recovery vs. Purge Time



was varied from 180°C to 60°C in 30°C increments. No change was observed in organic concentrations until the trap desorption temperature was reduced to 120°C. When the trap temperature was lowered to 60°C, 60% of benzene was recovered while only 17% of ethyl benzene was recovered.

These data reveal the necessity for careful choice of purge and trap parameters in order to obtain accurate and meaningful results.

EQUIPMENT

PURGE AND TRAP

330 Sample Concentrator,
purge temperature: ambient
Internal trap: Tenax TA

GAS CHROMATOGRAPHY

Varian 3700 equipped with a flame ionization detector
Column: 50m x 0.25mm SE-54 capillary
Program: 40°C for 2 min., then 6°C/min. to 100°c
Carrier: Helium

For more information on this and related applications, we sug- gest the following readings:

Washall J. W. and Wampler, TP., "Purge and Trap Analysis of Aqueous Samples with Cryofocusing," *Am. Lab.* July 70-74, (1985).

Wampler, T P., Bowe, W. A., Higgins, J. and Levy, E. J., "Systems Approach to Automatic Cryofocusing in Purge and Trap, Headspace, and Pyrolytic Analysis," *Am. Lab.,* August (1985).

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