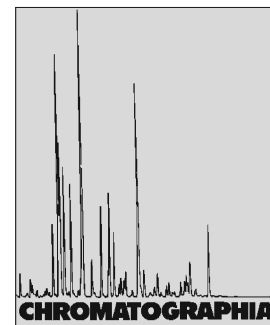


Determination of BTEX Compounds by Dispersive Liquid-Liquid Microextraction with GC-FID



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Abstract

Rapid, inexpensive, and efficient sample-preparation by dispersive liquid-liquid microextraction (DLLME) then gas chromatography with flame ionization detection (GC-FID) have been used for extraction and analysis of BTEX compounds (benzene, toluene, ethylbenzene, and xylenes) in water samples. In this extraction method, a mixture of 25.0 μL carbon disulfide (extraction solvent) and 1.00 mL acetonitrile (disperser solvent) is rapidly injected, by means of a syringe, into a 5.00-mL water sample in a conical test tube. A cloudy solution is formed by dispersion of fine droplets of carbon disulfide in the sample solution. During subsequent centrifugation (5,000 rpm for 2.0 min) the fine droplets of carbon disulfide settle at the bottom of the tube. The effect of several conditions (type and volume of disperser solvent, type of extraction solvent, extraction time, etc.) on the performance of the sample-preparation step was carefully evaluated. Under the optimum conditions the enrichment factors and extraction recoveries were high, and ranged from 122–311 to 24.5–66.7%, respectively. A good linear range (0.2–100 $\mu\text{g L}^{-1}$, i.e., three orders of magnitude; $r^2 = 0.9991$ – 0.9999) and good limits of detection (0.1–0.2 $\mu\text{g L}^{-1}$) were obtained for most of the analytes. Relative standard deviations (RSD, %) for analysis of 5.0 $\mu\text{g L}^{-1}$ BTEX compounds in water were in the range 0.9–6.4% ($n = 5$). Relative recovery from well and wastewater at spiked levels of 5.0 $\mu\text{g L}^{-1}$ was 89–101% and 76–98%, respectively. Finally, the method was successfully used for preconcentration and analysis of BTEX compounds in different real water samples.

Keywords

Gas chromatography
Flame-ionization detection
Dispersive liquid-liquid microextraction
BTEX compounds
Water samples

Introduction

BTEX is the abbreviation for the group of volatile organic compounds (VOCs) benzene, toluene, ethylbenzene, and *o*, *p*, and *m*-xylenes. BTEX are present in the environment because of industrial use of the compounds and the use of petroleum, because of the high concentrations of the compounds in gasoline and other products, for example diesel fuel and lubricating and heating oil. This is a hazard to the environment and to public health, and is the reason why environmental organizations regard these compounds as priority pollutants. To limit the effect of BTEX on the environment, it is necessary to develop accurate, sensitive, and reliable analytical methods to detect their presence. EPA limits for benzene, toluene, ethylbenzene, and the xylenes in drinking water are 5, 1,000, 700, and 10,000 $\mu\text{g L}^{-1}$, respectively [1]. To determine traces of BTEX in water, isolation and preconcentration steps are often necessary before analysis. Such methods include liquid-liquid extraction (LLE) [2, 3], solid-phase extraction [2, 4], solid-phase microextraction (SPME) [5–11], headspace solid-phase microextraction (HS-SPME) [12–14], dynamic solid-phase microextraction [15, 16], solid-phase microextraction coupled with cryo-trapping [17], manual headspace