Journal of Chromatography, 509 (1990) 111–113
Elsevier Science Publishers B.V., Amsterdam

CHROM. 22 195

Note

Simple device for the determination of volatile chlorinated hydrocarbons in water by gas chromatography

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In trace analysis, methods for concentrating analyte compounds from the sample are often necessary. For this purpose, the dynamic headspace method is

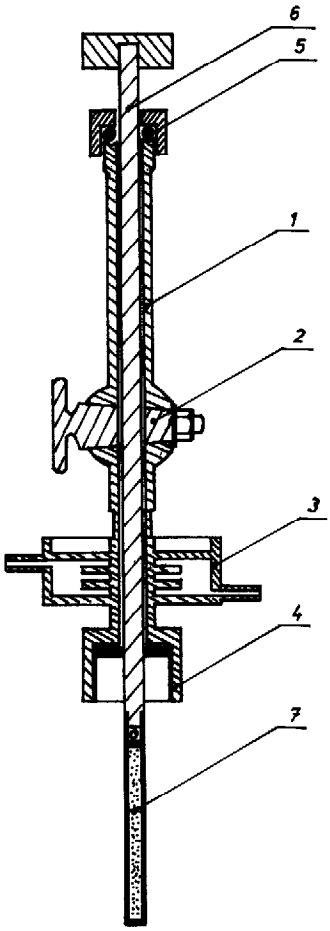

Fig. 1. Device for insertion of concentration microcartridge into the injector of the gas chromatograph.

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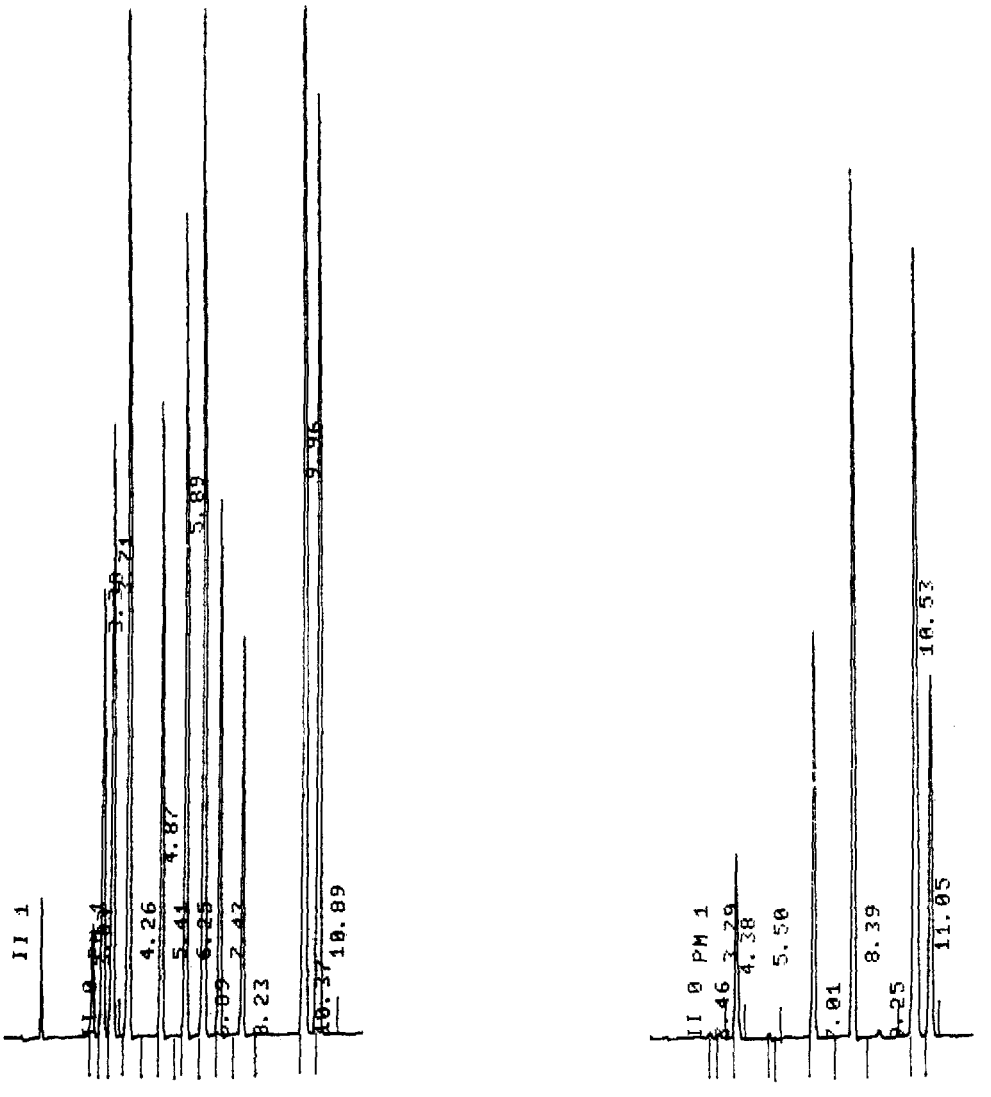

Fig. 2. Chromatogram of a calibration mixture for the determination of volatile aromatic and chlorinated hydrocarbons in water. Peaks: retention time 3.03 min = chloroform; 3.38 min = 1,2-dichloroethane; 3.71 min = benzene; 4.26 min = trichloroethylene; 4.87 min = toluene; 5.89 min = perchloroethylene; 6.89 min = chlorobenzene; 7.43 min = xylene; 8.23 min = *n*-nonane; 10.37 min = 1,4-dichlorobenzene; 10.89 min = 1,2-dichlorobenzene.

Fig. 3. Chromatogram of a real water sample. Peaks: retention time 3.46 min = 1,2-dichloroethane; 3.79 min = benzene; 4.38 min = trichloroethylene; 5.50 min = toluene; 7.01 min = chlorobenzene; 8.39 min = *n*-nonane; 10.53 min = 1,4-dichlorobenzene; 11.05 min = 1,2-dichlorobenzene.

suitable[1–3] but requires a special accessory, *i.e.*, a furnace with a controlling unit for thermal desorption of the analyte compounds.

We have designed a simple device (Fig. 1) for collecting the analyte compounds

in a preconcentration microcartridge, which can be inserted into the injector of any gas chromatograph with a sufficiently wide injector without interruption of the carrier gas flow[4].

The apparatus consists of a tube (1) with a tap (2), and is surrounded by a cooling rib (3) and a coil (4) for attachment to the injector of the gas chromatograph on one side, and a nut with a washer on the other side. A piston (6) with connected microcartridge (7) is inserted through this nut.

The chromatographic conditions adopted were as follows: gas chromatograph, Chrom 5 (Laboratory Instruments, Prague, Czechoslovakia); column, high-efficiency packed column (2.5 m × 3 mm I.D.) of 5% OV-101–Inerton Super (0.125–0.16 mm) (Lachema, Brno, Czechoslovakia); number of plates (80°C, isothermal run), 14 000 for octane and 19 000 for 1,4-dichlorobenzene; column temperature, 40°C for 2 min, then increased to 65°C, and finally at 7.5°C/min to 190°C; carrier gas, nitrogen at 19 ml/min; flame ionization detector, electrometer attenuation 1/128; integrator, Spectra-Physics 4200; and chart speed, 0.5 cm/min.

This method was applied to the determination of volatile chlorinated hydrocarbons in water. The analyte compounds were isolated from a 50-ml water sample by the dynamic headspace method by concentration on the micocartridge and inserted into the injector of the gas chromatograph.

The minimum concentration of chloroform, 1,2-dichloroethane, perchloroethylene and trichloroethylene that can be determined is 0.2 $\mu g\ l^{-1}$, of chlorobenzene, 1,2- and 1,4-dichlorobenzene 0.1 $\mu g\ l^{-1}$ and of benzene and toluene 0.05 $\mu g\ l^{-1}$.

The utilization of flame ionization detection (FID) for routine water quality control is advantageous with regard to the possibility of the simultaneous determination of halogenated hydroarbons and other volatile compounds. Compared with electron-capture detector, FID is not as sensitive to different interferences (*e.g.*, carrier gas purity, effects of water vapour). The determination of volatile hydrocarbons and chlorinated hydrocarbons is illustrated by the separation of a calibration mixture in Fig. 2 and of a river-water sample in Fig. 3.

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