

Short communication

Liquid phase separator with chromatographic column

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Received 13 August 2004; received in revised form 10 October 2004; accepted 12 October 2004

Abstract

Presented liquid phase separator with integrated chromatographic column enables the separation and treatment of a solvent extract layer after the extraction of organic compounds from water in one simple run in a single piece of glassware. The separator has been successfully applied for the determination of hydrocarbon oil index in waters.

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Keywords: Liquid phase separator; Water analysis; Hydrocarbon oil index; Chromatographic column

Current and proposed environmental regulations require analyses of a broad spectrum of organic compounds in water using gas chromatographic methods. Among the variety of techniques in sample preparation, liquid–liquid extraction is still in frequent use.

To eliminate the drawbacks of separatory funnel, a solvent thin layer separator for microextraction of water was developed [1]. In the international standard for the determination of hydrocarbon oil index in waters ISO 9377-2, another type of microseparator is presented [2].

It is often necessary to remove undesirable substances from the solvent extracts, e.g. coextracts, which contaminate the injection port of a gas chromatograph or which introduce interferences to the chromatographic analysis. In many preparation procedures, the extracts are fractionated via column chromatography. These procedures are time-consuming, because the extract has to be withdrawn from the separator, transferred into and run through the preconditioned chromatographic column. During this procedure, the extract is

diluted with extracting-agent phase and some pollution of the working environment with solvent vapours can occur. To speed up and simplify this procedure, the liquid phase separator with chromatographic column (Fig. 1), has been developed [3].

The separator consists of two necks and a joint. The top neck with a sintered glass bottom operates as a chromatographic column, and the side neck is used for addition of water. After the completion of extraction, the separator is connected to the extraction flask and through its side arm pure water is added. The added water causes the solvent layer to rise through the packing of the chromatographic column and the treated extract from the top of the column can be concentrated and analysed directly.

Advantage of this separator is that the separation of liquid phase and the chromatographic process are accomplished in one run in a single piece of glassware which significantly reduces time consumption and air pollution with solvent vapours. The extract enters the dry column packing from the bottom, by which means problems of air bubble formation and extract dilution are avoided. The separation and treatment of the extract can be completed within a few minutes.

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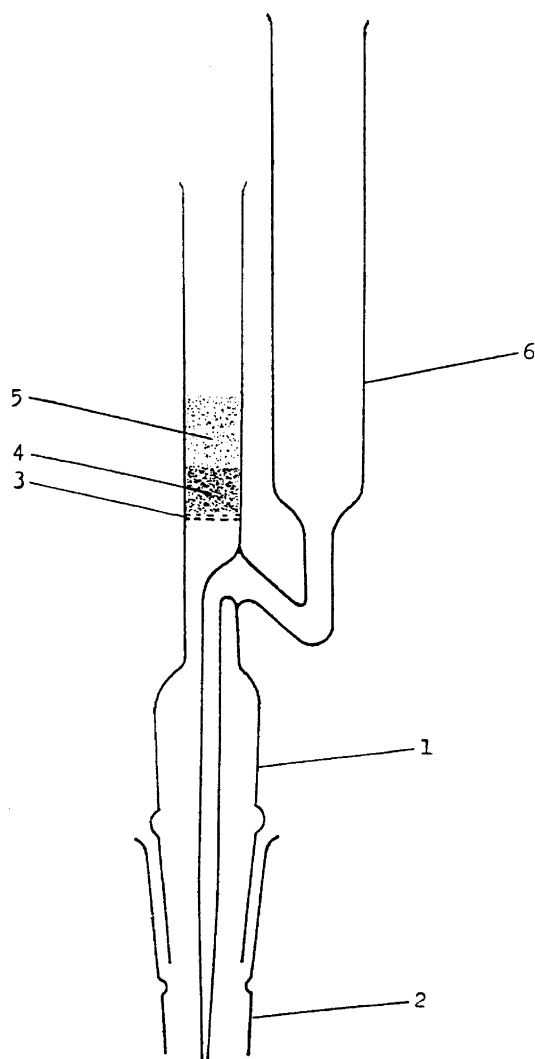


Fig. 1. Liquid phase separator with chromatographic column (1); neck of extraction flask (2); sintered glass (3); sodium sulfate (4); Florisil (5); side arm for water (6).

The liquid phase separator with chromatographic column has been used for the determination of hydrocarbon oil index in waters according to the standard ISO 9377-2 [2]. Fig. 2 shows a chromatogram from the analysis of tap water fortified with a mixture of equal amounts of diesel fuel and motor oil with a total concentration of 1 mg/900 ml. Aqueous standard solution (900 ml) was acidified to pH 2 by adding hydrochloric acid and extracted by shaking (5 min) with 50 ml of hexane (with the addition of 2 mg/l each of *n*-decane and *n*-tetracontane). The solvent layer was separated using the liquid phase separator with chromatographic column filled with 2 g of anhydrous sodium sulfate and covered with a layer of 2 g of Florisil. Then 40 ml of the treated extract was concentrated to 1 ml using a rotary evaporator and an aliquot of the final extract was analysed by gas chromatography/mass spectrometry. The average recovery ($n=3$) of the spiked hydrocarbon mixture was 90%.

References

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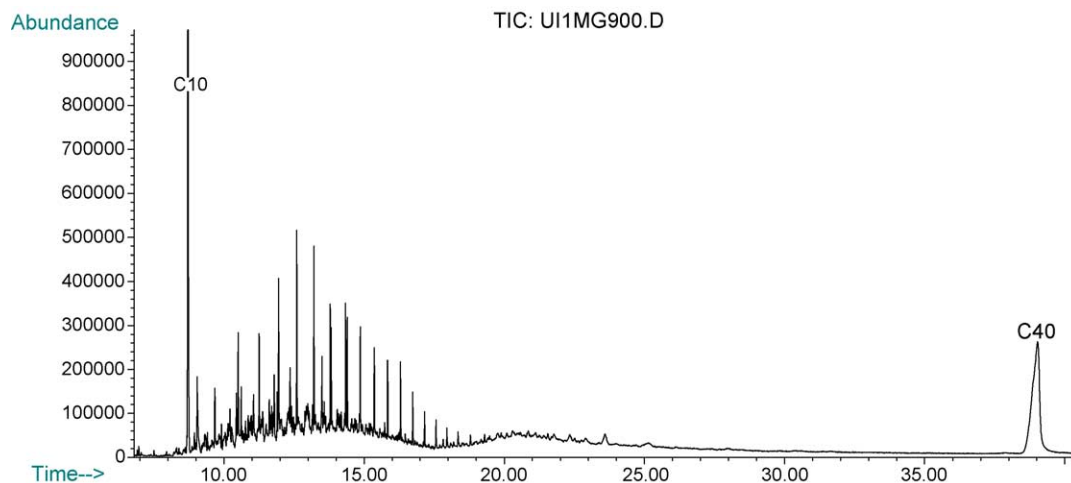


Fig. 2. Total ion chromatogram from the GC–MS analysis of tap water fortified by mixture of equal amounts of diesel fuel and motor oil with total concentration of 1 mg/900 ml.