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IMPROVED LIQUID PHASE SEPARATOR WITH CLEAN-UP COLUMN FOR WATER ANALYSIS

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Abstract

A simple modification of liquid phase separator with integrated clean-up column for water analysis is presented. The modification enables treatment of strong emulsions and speeds up the sample preparation. The use of separator is demonstrated in the analysis of polycyclic aromatic hydrocarbons in water sample.

Key words: liquid phase separator; water analysis; sample preparation

1. Introduction

Among the variety of techniques intended for preconcentration and isolation of organic substances from water matrix, liquid-liquid extraction (LLE) is still in frequent use. LLE comprises a mixing and a separation step. After the mixing step the two immiscible liquids with different specific gravities are commonly separated using appropriate separator [1-3]. Due to the fact that the resulting solvent extract phase contains some amount of water and in the case of treatment of dirty samples it also contains undesirable interfering substances; it is necessary to include a drying and clean-up step before the chromatographic analysis.

Recently, for the separation and treatment of solvent extract layer after the extraction of organic compounds from water, the liquid phase separator with chromatographic column has been developed [4]. This separator speeds up and simplifies the sample preparation procedure because both the separation and treatment steps are accomplished in one simple run in a single peace of glassware. The disadvantage of this separator arises in the case of formation of strong emulsions. To perform well also in treating strong emulsions, the separator was simply modified.

2. Results and discussion

In Figs. 1 and 2 the improved liquid phase separator with integrated clean-up column is illustrated. The separator consists of a top neck with a sintered glass bottom (operates as a clean-up column), filling tube for water and male joint. After the completion of extraction, the separator is connected to the extraction flask and through the filling tube attached via flexible tubing to a water reservoir, pure water is added. The added water causes the solvent layer to rise through the packing of the clean-up column and the treated extract from the top of the column can be concentrated and analyzed directly.

The main difference between the previous design and the new one is that the side arm for water from the former is replaced by flexible tubing connected to the larger water reservoir in the latter. This modification results in substantial increase of hydrostatic pressure. Greater water pressure makes the solvent extract layer easier to overcome the resistance to flow in the clean-up column. This speeds up the treatment and separation procedure and is of great importance when strong emulsions are treated and large amounts (up to 30 g) of anhydrous sodium sulphate are used. In the case of normal and emulsion forming samples, separators with smaller (e.g. 10 cm long, 1.7 cm I.D.) and larger (e.g.

10 cm long, 2.7 cm I.D.) internal diameter clean-up columns can be used (for application of different amounts of treatment material).

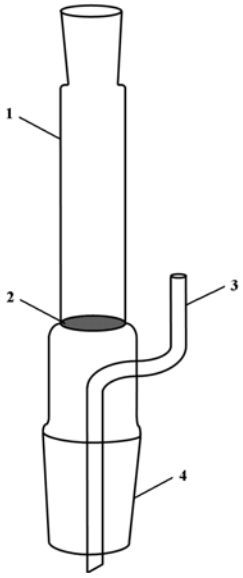

Figure 1. Liquid phase separator with clean-up column: (1) clean-up column, (2) sintered glass frit, (3) filling tube for water, (4) male joint.

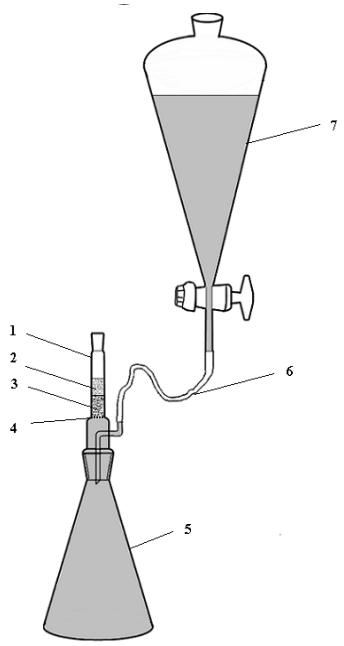

Figure 2. Separator in operation: (1) liquid phase separator, (2) separated extract, (3) anhydrous sodium sulphate, (4) sintered glass frit, (5) extraction flask, (6) flexible tubing, (7) water reservoir.

The modified separator is a very helpful tool in trace analysis of organic contaminants in water samples when LLE is performed with solvents lighter than water. The separation and treatment of the solvent extract is fast and can be completed within a few minutes. The simple design of the separator makes its cleaning easier, which also speeds up the work.

In our laboratory, the liquid phase separator is used in sample treatment for the determination of polycyclic aromatic hydrocarbons (PAHs), phthalates, alkyl phenols, polychlorinated biphenyls, organochlorinated pesticides or hydrocarbon oil index. Fig. 3 shows an example of chromatograms from the determination of PAHs (A) and phthalates (B) in surface water sample. 1 L of water sample was transferred into a 1-L Erlenmeyer flask and 5 g of sodium chloride and 5 mL of 50% (v/v) sulphuric acid aqueous solution were added.

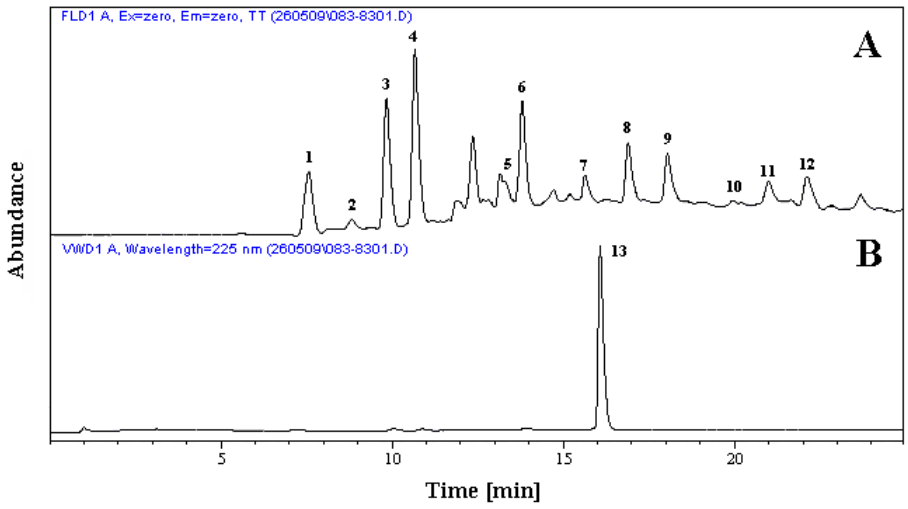

Figure 3. Chromatograms from the HPLC determination of PAHs and phthalates in surface water sample using (**A**) fluorescence and (**B**) UV detection. Peaks: (1) phenanthrene, (2) anthracene, (3) fluoranthene, (4) pyrene, (5) benzo[a]anthracene, (6) chrysene, (7) benzo[b]fluoranthene, 8) benzo[k]fluoranthene, (9) benzo[a]pyrene, (10) dibenzo[a,h]anthracene, (11) benzo[g,h,i]perylene, (12) indeno[1,2,3-cd]pyrene, (13) bis(2-ethylhexyl) phthalate.

Then, after addition of a stirring bar and 10 mL of hexane, the flask was closed with a ground stopper and thoroughly mixed using a magnetic stirrer at maximum setting for 2 hours. The solvent layer was separated and dried using the liquid phase separator with clean-up column filled with 10 g of anhydrous sodium sulphate. Then the treated extract was evaporated to dryness, redissolved in 200 µl of acetonitrile and 15 µl aliquot of the final extract was analysed by HPLC using both fluorescence (for PAHs) and UV (for phthalates) detection.

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