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Needle-type concentrator and its application to the determination of pollutants

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Abstract

A new method for impurity concentration in gas and liquid media using a needle type microconcentrator was developed. The main advantage of this method using microconcentrators is its efficiency and simplicity. Our work was stimulated by Pawliszyn's solid-phase microconcentration method.

Keywords: Sample handling; Instrumentation

Determination of pollutants is one of the main applications of chromatography (e.g., see ref. [1]). In our opinion, the best results in gas chromatography in the last decade were reached in the field of concentration. A simple needle microsorber with a movable sorbent-coated fused-silica fiber as a sorbing element was developed by Pawliszyn and coworkers [2,3]. Hundreds of publications deal with the application and development of this method which is usually called solid-phase microextraction (SPME). Unfortunately, this technique has some limitations. E.g., it is based on the use of delicate moving parts and the sorption capacity of its sorption element is low. Therefore, the aim of the present work is to show the possibility of development of a new method and new unit, that would be free of these limitations.

Two new types of the developed microconcentrators are shown in Fig. 1. In the simplest case (Fig. 1A) the medical needle contains a sorbent layer which sorbs impurities. After sorption in the needle microconcentrator (NM), the sample should be introduced through the self-sealing membrane into a gas chromatographic injector in the high-temperature zone. In this time flow the carrier gas passes through the needle with sorbent for faster desorption of impurities from sorbent.

It was shown that this method enables the determination of impurities at the concentration level ~1–5 mg/l for liquid samples and ~0.5 mg/m^3 for gas samples with reproducibility of results, evaluated by relative standard deviation <0.1%.

To increase the concentrator capacity, a tubular cylindrical microconcentrator (TCMC) was developed: a needle-cylindrical sorber (Fig. 1B) of greater diameter (2 mm) and greater length (100–150 mm). For desorption of concentrated impurities (Fig. 1B and C) the special cylindrical heater (Fig. 1C) was constructed and placed above a standard

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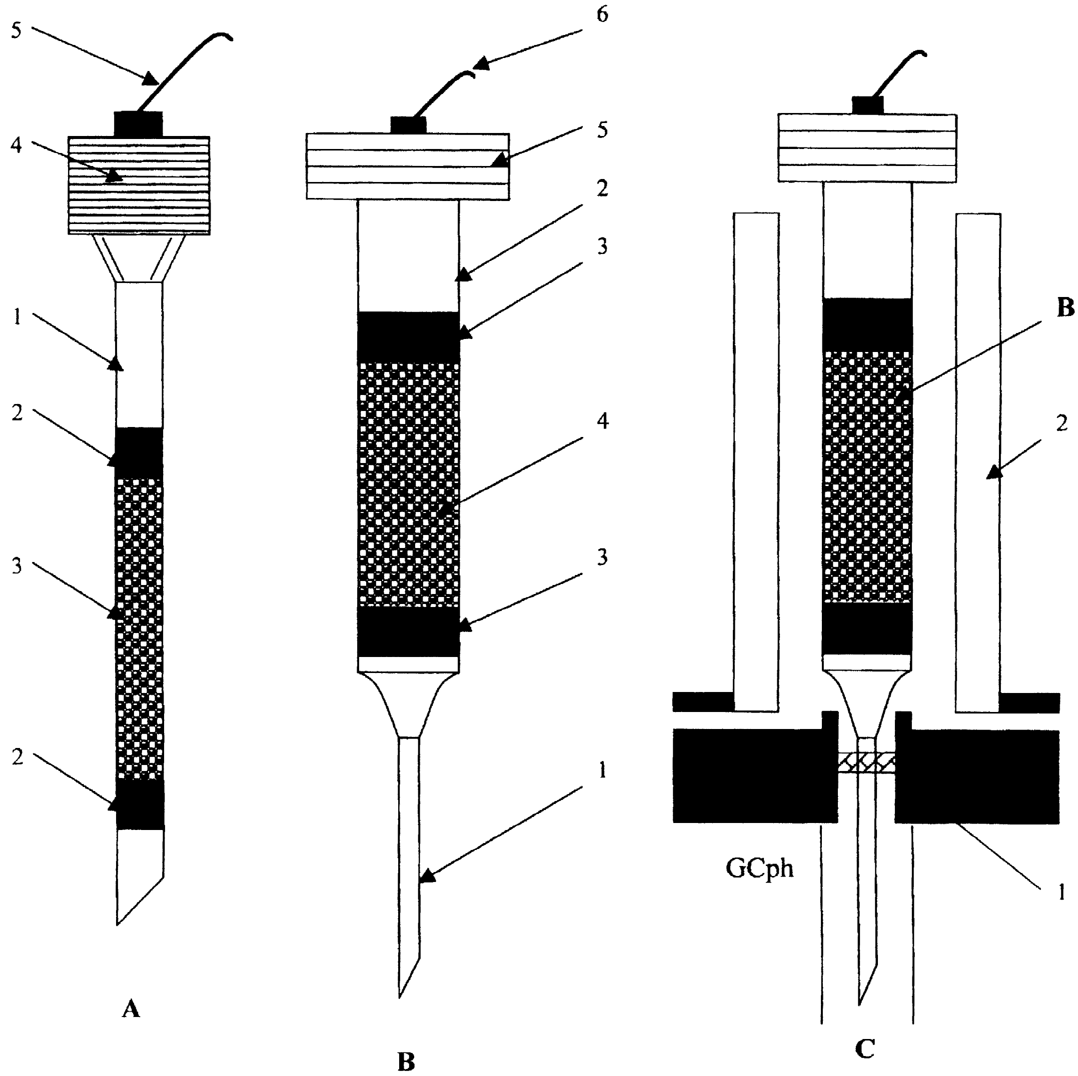

Fig. 1. Needle microconcentrator (A), cylindrical microconcentrator (B) and its use for desorption of concentrated traces in gas chromatograph (C). (A) Needle microconcentrator (NM): 1 = needle stem (0.5 mm I.D. × 0.8 mm O.D.); 2 = plugs, limiting the sorbent layer, 3 = sorbent layer (Tenax); 4 = holder, 5 = capillary line for carrier gas. (B) Cylindrical microconcentrator (TCMC): 1 = needle stem, 2 = tube stem (1 mm I.D. × 2 mm O.D.); 3 = plugs, limiting the sorbent layer, 4 = sorbent layer; 5 = holder, 6 = capillary line for carrier gas. (C) Cylindrical microconcentrator position during desorption process: 1 = elastic seal, 2 = tubular furnace for heating of the microconcentrator with sorbent layer (see B).

sample injection unit of a standard gas chromatograph (GCph). The TCMC needle is immersed into the evaporator cell through a standard elastic seal. Concentrated impurities are desorbed in the carrier gas flow in the sorbent layer by the tubular heater (Fig. 1C).

This TCMC was applied to the practical determination of benzene and toluene in tobacco smoke. As a source of standard tobacco smoke the laboratory-made smoking machine was used. It is similar to the device described previously [4]. Gas flow with tobacco smoke went through the TCMC for concentration of benzene and toluene after selective chemical sorption in chemical subtraction reactor (e.g. described in [1,5]).

The data of aromatic hydrocarbon contents in

Table 1
Determination of benzene and toluene contents in tobacco smoke, mg/cigarette

Hydrocarbon	Exp. 1	Exp. 2	Exp. 3	Exp. 4	Exp. 5	Middle	s	s_r
Cigarette ''Optima''								
Benzene	65.5	72.5	61.8	67.1	65.2	66.4±3.4	3.9	0.06
Toluene	77.5	86.3	68.6	75.1	79.7	77.4±5.7	6.5	0.08
Cigarette ''Soyuz-Apollon''								
Benzene	69.7	78.4	75.5	72.5	72.1	73.6±3.0	3.4	0.05
Toluene	75.9	71.9	74.0	77.2	75.9	75.0±1.8	2.1	0.03

tobacco smoke (mg/cigarette) are listed in Table 1 (where s is standard deviation and s_r is relative standard deviation). ''Optima'' and ''Soyuz-Apollon'' cigarettes were made under control of Philip Morris Products. The experimental results are in good correlation with the earlier published data [6].

In conclusion it should be noted that the results obtained indicates that it is desirable to apply the proposed method of trace concentration in practice and its further development is promising for gas chromatography and other fields of analytical chemistry.

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