

SAMPLE CONCENTRATION AND SEPARATION ON MICROCHIPS

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Abstract

Microfabricated devices integrating solid phase extraction and chromatographic separation with solvent programming were demonstrated. Preconcentration and separation were performed on channels 5 μm deep and 25 μm wide coated with a C18 phase, and elution was achieved under isocratic, step, or linear gradient conditions. For the solid phase extraction, signal enhancement factors of 400 over a standard injection of 1.0 s were observed for a 320 s injection.

Keywords: Solid phase extraction, solvent programming, electrochromatography

1. Introduction

Sample concentration and separation are necessary in instances where trace analysis of complex mixtures is desired, and the detection sensitivity is too low to reliably detect and quantify the analytes. In such cases, routinely used methods in liquid phase analysis are solid phase extraction (SPE) and liquid chromatography. In this work microchip solid phase extraction¹ and open channel electrochromatography (OCEC) with solvent programming² are coupled together for the analysis of polycyclic aromatic hydrocarbons (PAHs).

2. Experimental

Quartz microchips containing separation and solvent programming elements (Figure 1) were prepared and treated by selectively coating the analysis and sample waste channels with an octadecyltrimethoxysilane/toluene solution.

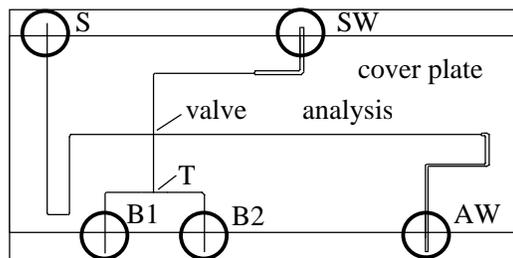


Figure 1. Chip schematic. Reservoirs: (B1) Buffer with 20% acetonitrile, (B2) Buffer with 60% acetonitrile, (S) Sample, (SW) Sample waste, (AW) Analysis waste. (not to scale).

The gated injection method was used to load the sample onto the 5 μm deep, 25 μm wide, 30 mm long, and C18 coated analysis channel (Figure 1). Computer controlled power supplies allowed precise mixing of the buffer solutions from B1 and B2 containing 20% and 60% acetonitrile, respectively. To perform SPE and OCEC, the sample was concentrated on the analysis channel for the desired time, e.g., 160 s, at low

organic concentration and separated at high organic concentration under isocratic or gradient conditions.

3. Results and discussion

Figure 2 shows a comparison of a standard 1 s injection of 900 nM pyrene eluted in high organic concentration with a 320 s injection under SPE conditions. The concentration increased linearly with time, and an enhancement factor up to 400 was observed for the 320 s injection. Solid phase extraction was then coupled to electrochromatographic separations. In Figure 3, four PAHs were concentrated for 160 s and separated using a step gradient. Detection limits of 3.1, 1.0, 8.1, and 17 nM were obtained for anthracene, pyrene, 1,2-benzofluorene, and benzo(a)pyrene, respectively.

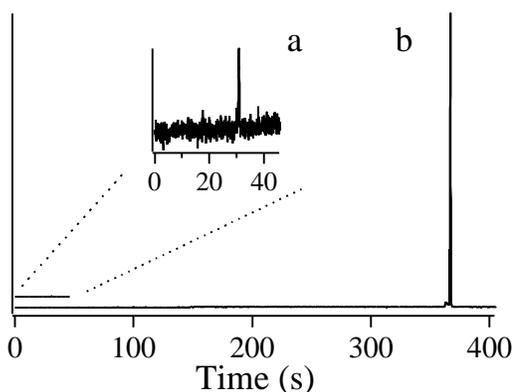


Figure 2. 900 nM pyrene injections. (a) 1 s injection and elution with 56% acetonitrile without concentration. (b) 320 s concentration and elution. (a) is offset from (b) for clarity. Y axis is detector response (V).

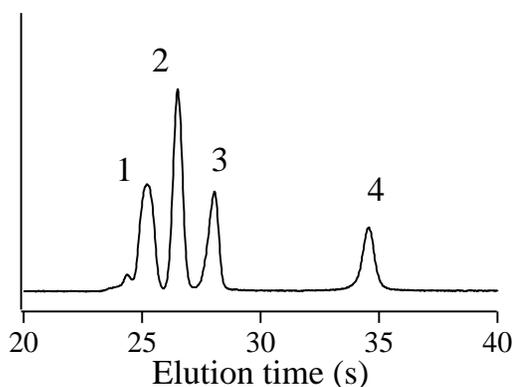


Figure 3. Separation using a step gradient that started at 52% acetonitrile, held for 10 s, and switched to 56% acetonitrile for the remaining elution time. 160 s concentration time. 1) Anthracene- 2.8 μM ; 2) Pyrene- 0.9 μM ; 3) 1,2 Benzofluorene- 5.8 μM ; 4) Benzo(a)pyrene- 5.0 μM . Y axis is detector response (V).

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References

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