SINGLE MAGNET MIXER

Reproducibility with repeated SPME extraction

Repeated solid phase microextraction of BTEX from water using a PDMS fibre

RESULTS

After correction of the expected decrease in concentration, the relative standard deviations of the investigated components are between 0.75 and 1.12 percent.

Objective and principle of operation

The single magnet mixer was investigated for its reproducibility in solid phase microextraction (SPME) applications. The system establishes equilibrium by stirring instead of shaking. As a result, the fiber is protected on the carrier. Especially the two-centimeter-long fiber can break off easily if it is immersed in a previously shaken liquid.

The needle is also less mechanically loaded. Under conventional stress, fatigue symptoms can occur after about 60 to 80 injections. The Single Magnet Mixer can therefore increase the mechanical lifetime of the fibers. This is particularly interesting in the case of polydimethylsiloxane fibers (PDMS), whose sorbent basically has a long service life.

Equipment and materials

• Agilent GC 6890 with split/splitless injector, flame ionisation detector and HP5 column (0.32 mm × 30 m)

CTC Combi PAL with Single Magnet Mixer

• 100µm PDMS fiber, 20 ml sample vessels, metal caps, purified water, pre-punched septa and SPME liners

• BTEX standard with 2,000 ppm: benzene, ethylbenzene, toluene and o-, m- and p-xylene

Test parameters

Combi-PAL parameters GC parameters	
Preincubation: 20 min Splitless injection: 1.2 min	
Incubation: 50° C. Flow after 1.2 min: 35.5 ml/min	
SMM: 750 rpm; 60 s on, 2 s from constant flow: 1.8 ml/min	
Depth of penetration into the sample vessel: 22 mm Oven: 40° C. for 3 min	
Extraction: 15 min 8 °C/min to 80 °C; hold 0 min	
Injector penetration depth: 54 mm 30 °C/min to 200 °C; hold for 3 min	
Desorption: 5 min flame ionization detector: 250° C.	
Penetration depth Needle: 30 mm Hydrogen: 33,0 ml/min; Oxygen: 380 ml/min	

Sample preparation and implementation

A sample vessel was filled with purified water in such a way that only 3 to 5 mm of gas space remained above the liquid. Subsequently, 10 ul of the BTEX standard with 2,000 ppm were added. The vessel was sealed with a blue butyl rubber septum with natural Teflon coating and a metal cap suitable for magnetic transport. Several extractions were carried out from the same vessel. With each injection, the sample concentration decreased by the proportion that was established in the fiber.

CHROMATOGRAM

BTEX in water

The chromatogram shows the separation of benzene, ethylbenzene, toluene and the xylenes. The English signal names embedded in the original image are explained below the illustration.

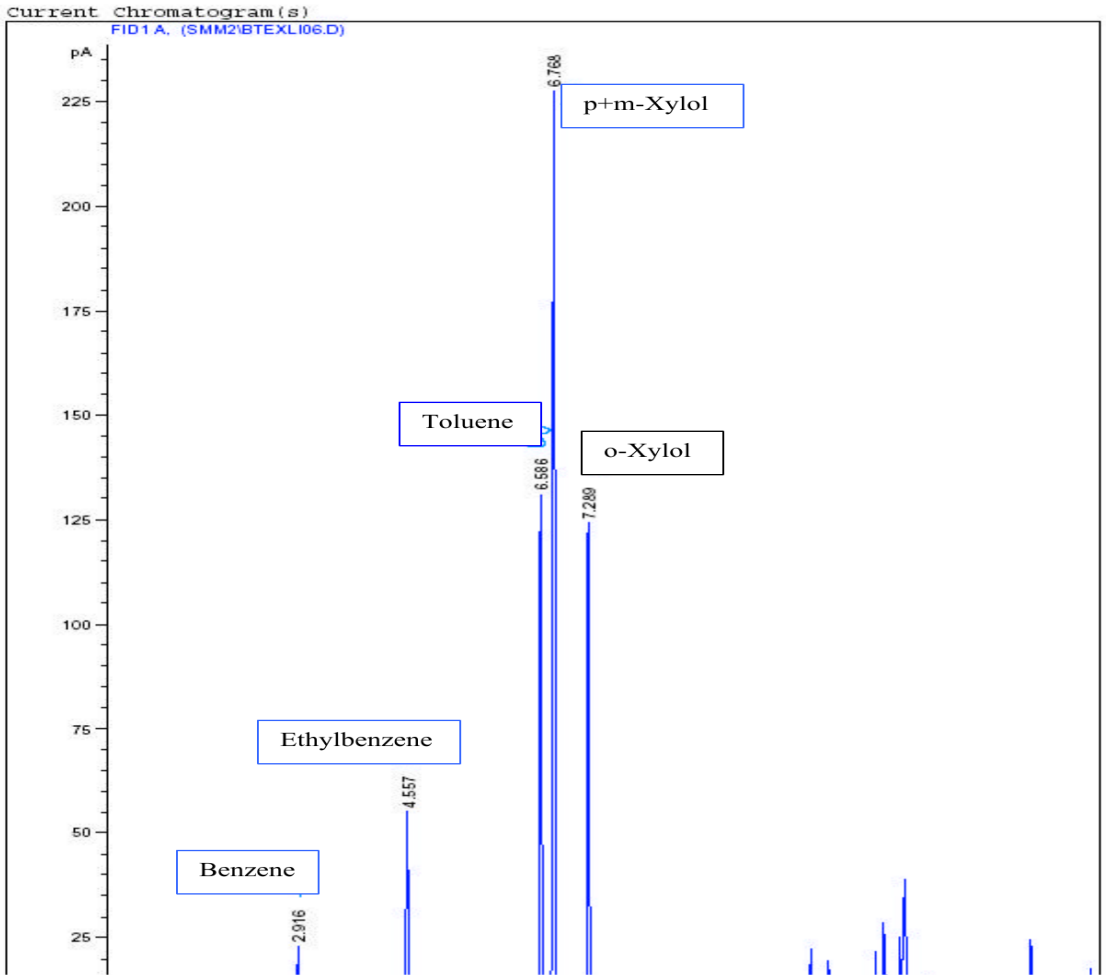


Figure 1: Chromatogram of BTEX components in water.

Signal names: Benzene = benzene; Ethylbenzene = ethylbenzene; Toluene = toluene; p+m-xylene = p+m-xylene; o-xylene = o-xylene.

RESULTS

The measured areas decrease by about 1.2 to 2.7 percent per extraction. After mathematical consideration of this decrease, the repeatability remains very high.

RESULTS

Repeatability after sampling correction

The raw areas show the expected decrease over ten consecutive extractions. The corrected areas are used to evaluate the repeatability.

TABLE 1 Raw areas of the ten extractions

Compound	tR	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10
Benzene	2,9	36,3	36,2	35,7	34,8	35,2	34,6	34,8	33,7	33,3	32,6
Ethylbenzene	4,6	113,3	112,3	110,2	107,7	107,5	104,7	104,6	100,9	100,8	98,4
Toluene	6,6	290,9	282,7	275,5	266,5	261,9	256,4	248,7	238,6	235,9	229,5
p+m-xylene	6,8	569,2	551,7	536,9	518,4	508,8	497,7	482,7	462,3	456,7	443,6
o-Xylene	7,3	258,3	253,0	247,8	241,1	238,4	233,8	228,4	219,9	218,2	212,9

TABLE 2 Relative change in area between extractions

Compound	Δ1	Δ2	Δ3	Δ4	Δ5	Δ6	Δ7	Δ8	Δ9	Resources	Total
Benzene	-0,36 %	-1,27 %	-2,65 %	1,22 %	-1,65 %	0,55 %	-3,18 %	-1,08 %	-2,19 %	-1,18 %	-10,2 %
Ethylbenzene	-0,83 %	-1,87 %	-2,26 %	-0,25 %	-2,54 %	-0,17 %	-3,51 %	-0,12 %	-2,35 %	-1,54 %	-13,1 %
Toluene	-2,80 %	-2,57 %	-3,27 %	-1,70 %	-2,11 %	-3,00 %	-4,04 %	-1,14 %	-2,72 %	-2,60 %	-21,1 %
p+m-xylene	-3,08 %	-2,69 %	-3,44 %	-1,85 %	-2,17 %	-3,02 %	-4,22 %	-1,22 %	-2,87 %	-2,73 %	-22,1 %
o-Xylene	-2,07 %	-2,04 %	-2,69 %	-1,14 %	-1,94 %	-2,31 %	-3,72 %	-0,77 %	-2,42 %	-2,12 %	-17,6 %

Table 3: Corrected areas and relative standard deviation

tR	A1	A2 corr.	A3 corr.	A4 corr.	A5 corr.	A6 corr.	A7 corr.	A8 corr.	A9 corr.	A10 corr.	Resources	s	RSD
2,9	36,3	36,6	36,6	36,0	36,8	36,6	37,3	36,5	36,5	36,1	36,5	0,37	1,01 %
4,6	113,3	114,1	113,6	112,7	114,1	112,8	114,2	111,8	113,2	112,0	113,2	0,85	0,75 %
6,6	290,9	290,1	289,8	287,2	289,1	289,7	287,5	282,1	285,0	283,2	287,5	3,08	1,07 %
6,8	569,2	566,7	566,2	560,8	564,4	565,7	561,8	550,7	556,4	552,6	561,4	6,30	1,12 %
7,3	258,3	258,3	258,3	256,5	258,6	258,6	257,4	252,5	255,2	253,5	256,7	2,25	0,87 %

Assessment

The relative standard deviation of the corrected areas is 1.01 percent for benzene, 0.75 percent for ethylbenzene, 1.07 percent for toluene, 1.12 percent for p+m-xylene and 0.87 percent for o-Xylene. Thus, the Single Magnet Mixer shows very good repeatability in this series of measurements.