SINGLE MAGNET MIXER

# Single Magnet Mixer Compared to Agitator

Solid phase microextraction of a BTEX-added beer sample

**RESULTS**

**For volatile and heavier-volatile components from C8 onwards, the Single Magnet Mixer improves reproducibility and signal. The average signal gain is 15 percent.**

## Objective and principle of operation

The performance of the single magnet mixer was compared directly with a classic agitator in a SPME application with beer. In the single magnet mixer, the equilibrium is established by stirring instead of by shaking. This reduces the mechanical loading of the fiber on the carrier. Especially two centimeters long fibers can break off easily when they are immersed in a previously shaken liquid.

## 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, pre-punched septa and SPME liners

• Beer sample and BTEX standard with 2,000 ppm as internal addition

## Test parameters

| Combi-PAL parameters GC parameters | |
|---|---|
| Preincubation: 20 min Splitless injection: 1.2 min | |
| Incubation: 35° 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 | |
| Agitator: 500 rpm; 5 s on, 2 s from oven: 40° C. for 3 min | |
| Depth of penetration into the sample vessel: 22 mm 8 °C/min at 80°C; hold for 0 min | |
| Extraction: 15 min 30 °C/min to 200 °C; hold for 3 min | |
| Depth of penetration Injector: 54 mm Flame ionisation detector: 250 °C | |
| Desorption: 5 min Hydrogen: 33.0 ml/min; oxygen: 380 ml/min | |
| Penetration depth needle: 30 mm | |

## Sample preparation and implementation

Four sample vessels were prepared. The beer bottle was shaken and then opened to allow some of the carbon dioxide it contained to escape. The beer was filled into the vessels in such a way that 3 to 5 mm of gas space remained above the liquid. Each sample received 10 ul of BTEX standard with 2,000 ppm. The vessels were sealed with blue butyl rubber septene with natural Teflon coating and metal caps for magnetic transport. From each vessel, several extractions were carried out using Single Magnet Mixer and Agitator.

CHROMATOGRAM

# Beer sample with internal BTEX addition

The chromatogram shows ethanol, benzene, ethylbenzene, toluene, p+m-xylene and o-xylene as well as three unidentified components. The English signal names embedded in the original image are explained below the illustration.

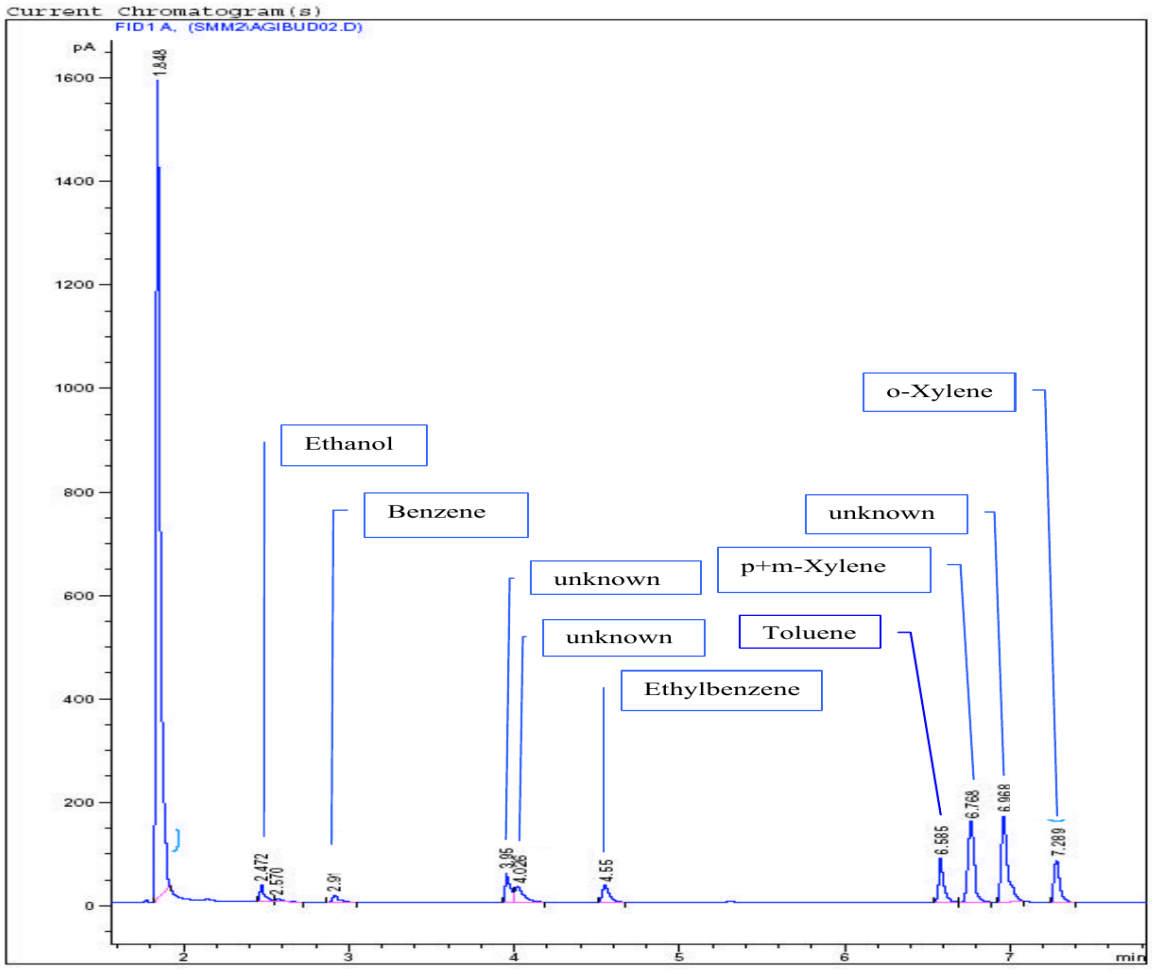



Figure 1: Chromatogram of the beer sample with internal addition of BTEX.

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

RESULTS

**For low-boiling components up to C6, the reproducibility is comparable. For components from C8 onwards, the single magnet mixer predominantly shows lower relative standard deviations and higher signals.**

RESULTS

# Direct comparison of single magnet mixer and agitator

The table compares retention time, mean value, standard deviation and relative standard deviation. The signal difference refers to the single magnet mixer compared to the agitator.

**TABLE 1 Measurements of the beer sample**

| Component SMM tR | SMM means | SMM s | SMM RSD | Agitator tR | Agitator means | Agitator RSD | Signal Difference | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Ethanol | 1,8 | 2524,6 | 53,9 | 2,13 % | 1,8 | 2549,0 | 40,3 | 1,58 % | -1,0 % | |
| Benzene | 2,9 | 35,9 | 2,9 | 7,96 % | 2,9 | 34,3 | 2,0 | 5,90 % | 4,5 % | |
| Unknown compound | 4,0 | 141,0 | 2,2 | 1,55 % | 4,0 | 133,2 | 5,4 | 4,03 % | 5,5 % | |
| Unknown compound | 4,0 | 100,9 | 4,1 | 4,08 % | 4,0 | 111,7 | 4,7 | 4,23 % | -10,7 % | |
| Ethylbenzene | 4,6 | 91,9 | 1,8 | 1,94 % | 4,6 | 75,7 | 9,1 | 12,04 % | 17,7 % | |
| Toluene | 6,6 | 185,5 | 11,2 | 6,02 % | 6,6 | 155,7 | 24,5 | 15,75 % | 16,1 % | |
| p+m-xylene | 6,8 | 357,1 | 22,2 | 6,20 % | 6,8 | 301,0 | 46,5 | 15,46 % | 15,7 % | |
| Unknown compound | 7,0 | 339,9 | 3,3 | 0,98 % | 7,0* | 361,2 | 7,5 | 2,07 % | | |
| o-Xylene | 7,3 | 170,3 | 7,8 | 4,60 % | 7,3 | 147,3 | 18,4 | 12,49 % | 13,5 % | |

* In the case of the agitator, two peaks were observed at a retention time of 7.0 minutes.

## Assessment

For the low-boiling components C2 to C6, the reproducibility of Agitator and Single Magnet Mixer is comparable. For volatile and less volatile components from C8 onwards, the Single Magnet Mixer delivers more favorable values for reproducibility and signal. The average signal gain is 15 percent. For most components, the repeatability is significantly improved. The increased relative standard deviation of the internal standard is attributed to a manual error in the original test.

The difference is particularly clear for ethylbenzene, toluene and xylenes: here the relative standard deviation decreases with the Single Magnet Mixer, while the signal increases by 13.5 to 17.7 percent.