Automated difficult matrix introduction (DMI) for screening of washing powder with GC-MS/olfactometry

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Introduction

Complex matrices are encountered in numerous application areas of gas chromatography. Difficult matrices occur for example in environmental analysis, food characterisation or in the analysis of home and personal care products. For such complex and difficult matrices the technique DMI (difficult matrix introduction) is a powerful analytical tool. During the normal screening of these complex samples it is also possible to identify fragrances in perfumed products in the same run because the DMI-GC-MS is coupled to a sniffing port (PHASER). In this contribution the DMI technique is applied for screening of washing powders and shampoo. To automate the analysis a LINEX was used.

Experimental

Figure 1: Shimadzu GCMS QP2010, ATAS GL Phaser, Optic 3 and LINEX (LINer EXchanger).

Repeatability of DMI

One of the advantages of the DMI method is that only minute sample quantities are required. For inhomogeneous samples such as washing powders, however, this could also be a potential drawback. To determine the repeatability of DMI analysis for such samples a washing powder was analysed multiple times (n=10). The repeatability of the analysis is for peak areas was between 4% and 13%.

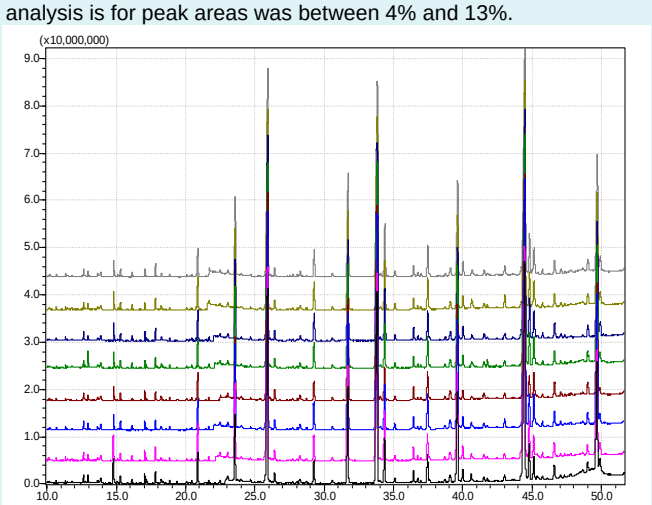

Figure 2: GC-MS chromatogram of washing powder for the determination of the repeatabilty of DMI. GC-column: Inertcap Wax 0.32 mm X 60 m, film thickness 0.5 µm (GL Sciences). GC program: 40°C (hold 6.3 min), 15 °C/min to 130 °C, 3°C/min to 250°C (hold 25 min). Optic program: 35°C, 5°C/sec to 250°C, split 1:40.

DMI of Shampoo

Automated DMI-GC-MS-Olfactometry is very a powerful technique for screening of shampoos as it is demonstrated at figure 3. With the combination of olfactometry and mass spectrometry the smelly compounds of the shampoo can be determined by the nose and identified by MS at the same time.

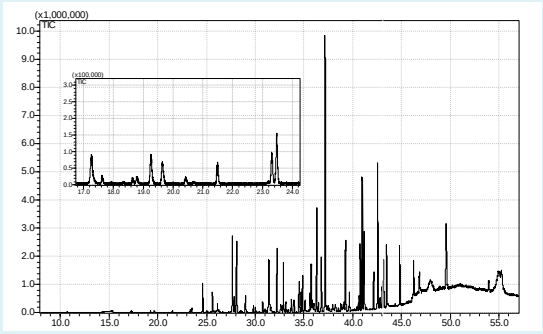

Figure 3: Typical GC-MS chromatogram of shampoo using DMI-GC/MS. GC-column: Inertcap Wax 0.32 mm X 60 m, film thickness 0.5 µm (GL Sciences). GC program: 35°C (hold 8 min), 5 °C/min to 230 °C (hold 10 min). Optic program: 35°C, 5°C/sec to 120°C, splitless.

1. d-limonene (Rt 17.1 min)
2. tetrahydro linalool (Rt 24.6 min)
3. dihydromyrcenol (Rt 25.6 min)
4. linalool (Rt 27.7 min)
5. *tert*-butyl cyclohexyl acetate (Rt 28.1 min)
6. terpineols (Rt 29.8 min; 31.4 min)
7. benzyl acetate (Rt 32.2 min)
8. geraniol (Rt 32.6; 34.7 min)
9. citronellol (Rt 32.9 min)
10. nerol (Rt 33.7 min)
11. α-isomethyl ionone (Rt 34.8 min)
12. β-ionone (Rt 36.8 min)
13. 2-(4-*tert*-butylbenzyl)propionaldehyde (38.9 min)
14. n-hexyl salicylate (Rt 40.0 min)
15. piperonal (Rt 42.8 min)
16. cinnamal (Rt 43.0 min)

Conclusions:

- The repeatabilty of DMI analysis of washing powder is acceptable.
- Identification of unknown smelly compounds is possible using the combination of DMI-GC-MS/olfactometry.
- Automated DMI-GC-MS injections is possible using a liner exchanger.

Reference:

H. Jing, A. Amirav, Anal. Chem 1997,69,1426-1434

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