Automated DMI for scree cosmetic products

GC/MS

Figure 1: The GCMS-QP2010, AOC-5000, Optic 3 injector and LINEX

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Compound	Area RSD %
Diethylene glycol	12.9
Triehtylene glycol	10.9
1.2-Ethanediol, Monoacetate	5.0
Pentaethylene glycol	9.5
Heptaethylene glycol	12.4
Ethylene glycol monododecyl ether	5.7

Table 1: Repeatability of DMI analysis of washing powder

Complex matrices are encountered in many application areas of gas chromatography. Difficult matrices occur for example in environmental analysis, food characterization or in the analysis of home and personal care products. For such complex and difficult matrices the DMI (Difficult Matrix Introduction) technique is a powerful analytical tool.

In the present contribution the DMI technique is applied to the screening of cosmetic products such as lipsticks, lotions, washing powders and shampoos. Pattern recognition, identification of unknown compounds as well as quantification of known ingredients can be done without any sample preparation. If identification of the fragrances in perfumed cosmetic products is necessary, this can be achieved in just one single run during the normal screening since the DMI GC/MS is also coupled to a sniffing port ('PHASER'). At the end of the GC column the carrier gas flow is split into two. One flow is directed to the MS detector whereas the other is sent to the PHASER. Retention times for both detection devices are hence virtually identical.

GC/MS analysis of cosmetic products is generally difficult because the compounds of interest are present at low levels in a complex water and oil-containing emulsion. Transfer of either water or oil to the GC system obviously has a negative influence on the results. A number of labor-intensive sample preparation steps are normally needed for extraction and cleanup if these complex mixtures are to be analyzed using GC/MS.

A different approach to eliminate the matrix is to perform a so-called Difficult Matrix Introduction (DMI). In the DMI technique a small aliquot of the sample is put into a glass vial which is inserted automatically into the injector. The injector is then heated to a temperature just high enough in order to transfer the compounds of interest from the sample onto the chromatographic column. Only the vaporized

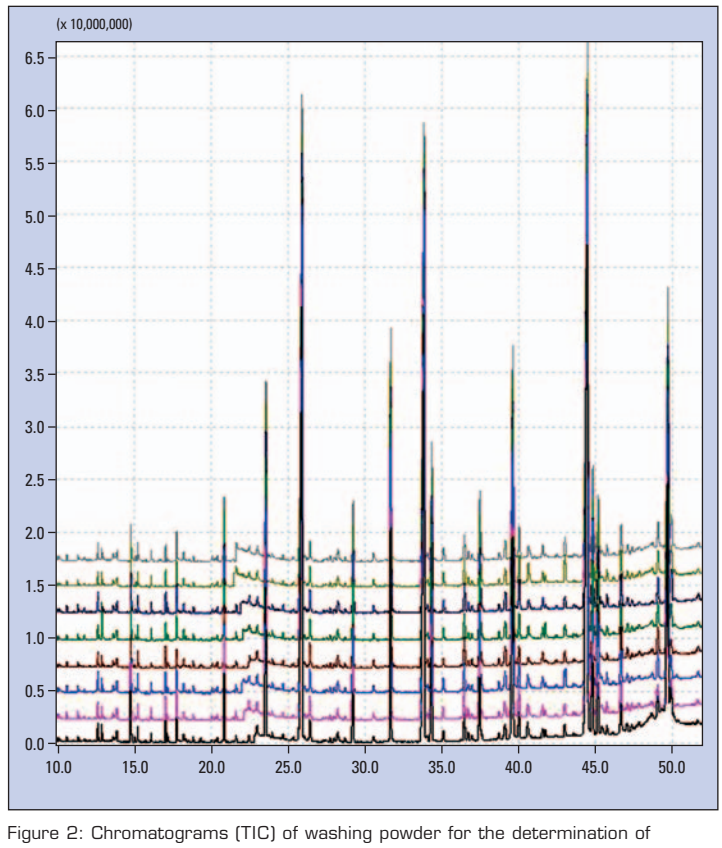

Figure 2: Chromatograms (TIC) of washing powder for the determination of repeatability. GC column: Inertcap wax 0.32 mm x 60 m, film thickness 0.5 µm (GL Sciences); GC temperature program: 40 °C (hold 6.3 min), 15 °C/min to 130 °C, 3 °C/min to 250 °C (hold 25 min); PTV injector: 35 °C to 250 °C rate 5 °C/s.

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compounds are transported from the injector onto the GC column, where they are refocussed at the low starting temperature of the GC program. Because the non-volatile matrix species remain in the vial and the injector liner, no column contamination of the GC system occurs.

Instrumentation

The injector used for this study was an OPTIC 3 injector (ATAS GL Int'l, the Netherlands). It was installed into a GCMS-QP2010 (Shimadzu Corp., Japan). The column effluent flow was split into two separate flows, one going to the PHASER sniffing port (ATAS GL Int'l) and the other to the mass spectrometer. Helium was used as the carrier gas in all experiments.

In order to streamline the analysis, an automated liner exchanger unit (LINEX, ATAS GL Int'l) was used (Figure 1). The sample mass weighed into the DMI micro-vials ranged from 8 mg for detergent powders to 12 mg in the case of shampoos.

Reproducibility

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 reproducibility of DMI analysis for such samples a washing powder was analyzed several times. To minimize inhomogeneity problems, the maximum mass of powder was put into the micro vial. Heating was performed in the split mode at a split ratio of 1 to 40. For desorption the injector was heated to 250 °C at a rate of 5 °C/s. The reproducibility (n = 10) of the retention times and the peak areas for most of the compounds was better than 4 % (Rt) and 10 % (areas) as can be seen from Table 1 or Figure 2.

Screening of shampoos

Perfumes in cosmetic products are one of the most common causes of allergic contact dermatitis. For this reason the quantification of allergens is currently receiving a great deal of attention. With DMI it is possible to identify and quantify these perfume allergens at low levels without any sample preparation. This reduces the cost of analysis but it also eliminates potential losses of the volatile target compounds during sample preparation. In addition to the allergens, other ingredients of shampoo can be also be identified and quantified as demonstrated in Figure 3.

Reference

A. Amirav and S. Dagan, Europ. Mass. Spectrom. 3, 105-111 (1997)

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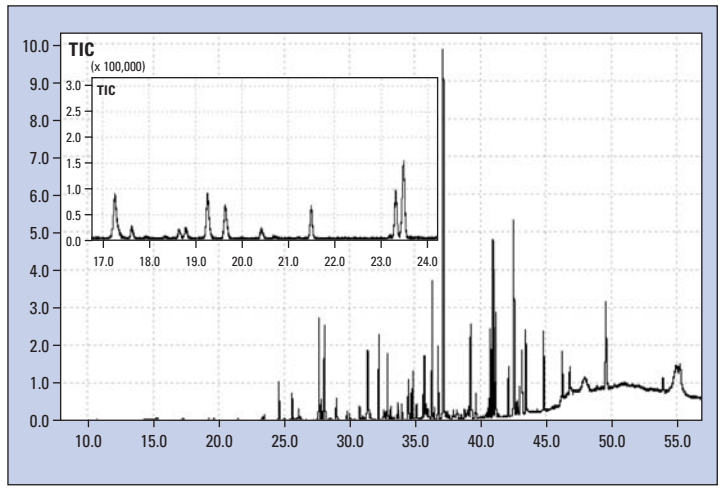

Figure 3: DMI GC/MS chromatogram (TIC) of shampoo for identification of fragrance and allergens. GC column: Inertcap wax 0.32 mm x 60 m, film thickness 0.5 µm (GL Sciences); GC temperature program: 35 °C (hold 8 min), 5 °C/min to 230 °C (hold 10 min); PTV injector: 35 °C to 120 °C, rate 5 °C/s.

01. d-Limonene (Rt 17.1 min)
02. Tetrahydro linalool (Rt 24.6 min)
03. Dihydromyrcenol (Rt 25.6 min)
04. Linalool (Rt 27.7 min)
05. *tert*-Butyl cyclohexyl acetate (Rt 28.1 min)
06. Terpineols (Rt 29.8 min; 31.4 min)
07. Benzyl acetate (Rt 32.2 min)
08. Geraniol (Rt 32.6; 34.7 min)
09. 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)