Application Note No. 103 (April 2006)

Automated difficult matrix introduction (DMI) for identification of residues of washing powder on clothing with GC-MS and a LINer EXchanger (LINEX)

Key Words:

Automated Difficult Matrix Introduction (DMI)*
GC-MS
Allergens, fragrances
Washing powder

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Introduction

Sample preparation techniques are almost always necessary in each analytical procedure. The preparation of the sample is a important step in the whole analytical procedure because only good sample pre-treatment give valid results. However, in analytical chemistry laboratories just this step in the whole procedure is labour-intensive and thus economically unattractive. The basics of sample preparation is to convert a real matrix into a sample which can be analyzed by a analytical technique. Difficult Matrix Introduction (DMI) is such a sample preparation technique only almost without manual sample handling and thus less expensive. DMI is based on difference between the evaporation temperature of the matrix and the compounds of interested. When the real matrix is put into the liner and heated only the volatile compounds are transported to the analytical column and the matrix remains in the vial. To demonstrate that DMI is a really powerful technique in case of a low/non volatile matrices the analyse of residues of washing powder ingredients on clothes is carry out.

Results & specifications

A piece of cotton was washed in water including washing powder and afterwards flushed with normal tape water and dried. After drying, small pieces of cotton were analyzed using DMI-GC-MS. The chromatograms of the washed cotton are compared with the chromatograms of the pure washing powder, which were also analyzed using the same DMI method. The results show that there are a lot of compounds found on the treated pieces of cotton, which are also present in the washing powder, for example linalool (tetrahydro), citronellol, butyl glycol, hexadecanol, butylalcohol and α –isomethyl ionone (see figure 1). The relative amount of residue of the washing powder found on the cotton is very low and is different for each single compound. The repeatability of DMI-analysis of pure washing powder is for retention times 4% and for peak areas 13% as shown in application note no. 102.

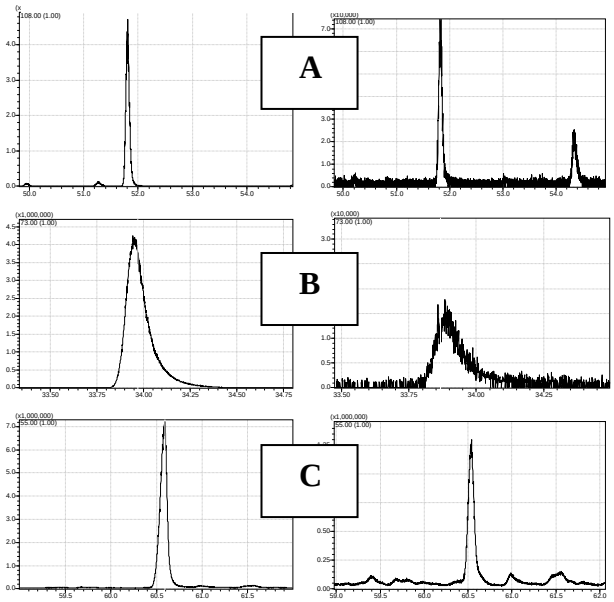

Figure 1: Peaks of typical ingredients of washing powder (left column) and the residue on clothing after washing and flushing (right column).
A Benzylalcohol (m/z 79) **B** Linalool, tetra hydro (m/z 73) **C** hexadecanol (m/z 55).

Discussion

With DMI-GC-MS it is possible to identify residue on clothes of ingredients of washing powder at low levels almost without any manual sample preparation. This reduces the cost of analysis but also it eliminates potential losses of the volatile target compounds during sample preparation. DMI can be a good alternative for commonly used sample preparation techniques especially for identification of compounds at low levels and/or screening purposes.

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Experimental:

Sample preparation of the clothes (cotton):

The cotton is cut into pieces of a width of 3 cm and a height of 1 cm.
The pieces of cotton are put into a mixture of water and dissolved washing powder.
The cotton is moved around in the mixture and it stayed there for 1 hour (this is normally the length of time a washing machine run takes) at room temperature.
After washing the cotton was flushed with streaming water (normal tape water) , 3 times.
Than the cotton was placed on a piece of paper until dryness.

Sample preparation for DMI-analysis:

A little piece of the washed cotton is cut (a weight of 5-10 mg) and placed into a DMI micro-vial.
The micro vial is placed into in a CLEANED DMI-Liner.

Instrumentation:

Injector: OPTIC 3 injector (ATAS GL International BV, Veldhoven, the Netherlands).
GC/MS: GC-MS-QP2010 (Shimadzu Deutschland GmbH, Germany).
Autosampler: FOCUS (ATAS GL International BV)
LINEX (ATAS GL International BV)

DMI-GC-MS conditions for shampoo:

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)
Carrier gas: Helium
PTV-injector: 35°C to 250°C rate 5°C/sec.
Column flow: 1,0 ml/min (without sniffing)
5,0 ml/min (split MS/sniffing)
Split flow: Start 1.5 min. 150 ml/min (flush liner)
During heating: 1:40
During analysis: 1:40
Liner: DMI-liner

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

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