GL Sciences

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# Automated difficult matrix introduction (DMI) for identification of allergens and fragrances in washing powder with GC-MS-Olfactometry and a liner exchanger (LINEX)

**Key Words:**
**Automated sample preparation**
**Difficult Matrix Introduction (DMI)**
**GC-MS-Olfactory**
**Allergens, fragrances**
**Washing powder**

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## Introduction

For complex and difficult matrices the technique DMI (difficult matrix introduction)* is a powerful analytical tool. In the present contribution the DMI technique is applied for screening of washing powders. Pattern recognition, identification of unknown compounds as well as quantification of known ingredients can be done without any sample preparation. If it is also necessary to identify the fragrances in the perfumed products it can be done just in a single run during the normal screening because 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 flows. One flow is directed to the MS detector whereas the other is sent to the PHASER. Retention times for the two detection devices are hence virtually identical.
For each injection of washing powder the micro-vial in the liner have to be changed to automated this step a LINEX (liner exchanger) is used.

## Results & specifications

- Detection limits of compounds which are thermo stabile and not very polar is approximately 0.1-0.02 ng/micro vial.
- The reproducibility of the analysis of washing powder using DMI is very good. The reproducibility (n=10) of the retention times and the peak areas for most of the compounds was better than 4% (Rt) and 13% (areas) as can be seen from Table 1 or Figure 1.
- Using the parameters of this application it is possible to measure compounds between approximately C11H24 and C44H50. When it is necessary to analyse compounds below C11H24 a cryo-trap can be used.

Table 1: Repeatability (area) of several compounds detected in washing powder (n=10).

| Compound | RSD % |
|---|---|
| diethylene glycol | **12.9** |
| triehtylene glycol | **10.9** |
| 1,2-ethanediol, monoacetate | **5.0** |
| pentaethylene glycol | **9.5** |
| hepta ethylene glycol | **12.4** |
| ethylene glycol monododecyl ether | **5.7** |

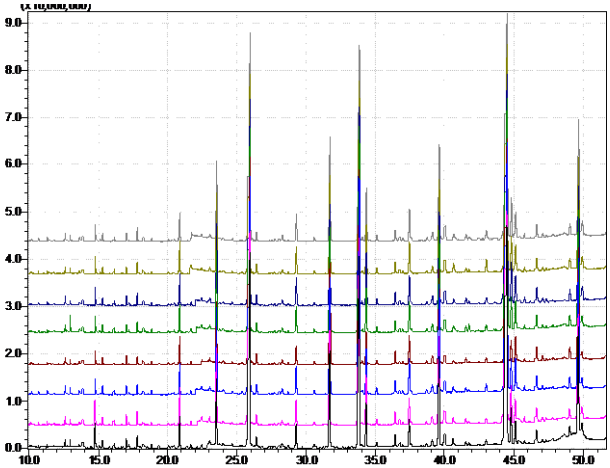


**Figure 1:** Chromatograms (TIC) of washing powder for the determination of the repeatability.

## Discussion

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 minimise in homogeneity problems the maximum mass of powder was put into the micro vial.
Because of the very simple sample preparation it reduces the cost of analysis but also it eliminates potential losses of the volatile target compounds during sample preparation.

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## Experimental:

Sample preparation:

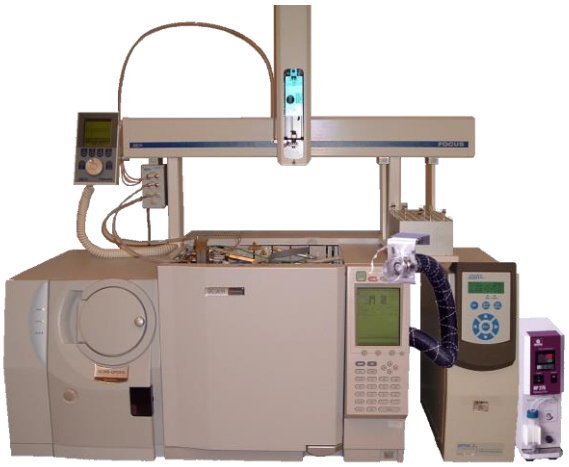
Weight 1-8 mg of washing powder into a micro-vial. Put the micro-vial into a DMI-liner.

Instrumentation:

**Injector:** OPTIC 3 injector (ATAS GL International BV, Veldhoven, the Netherlands).
**GC/MS:** GC-MS-QP2010 (Shimadzu Deutschland GmbH, Germany).
**Sniffing port:** PHASER (ATAS GL International BV)
**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 with micro-vial.



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

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