Automated DMI: Applicability and performance of OPTIC, LINEX, and CDC Station

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Introduction

GC analysis of compounds like pesticides in dirty extracts is traditionally very difficult. The liner and the top of the analytical column easily get dirty and therefore the sensitivity during the analysis decreases fast. This can result in a much more labor intensive sample preparation and/or routine analysis of those dirty samples is difficult. For those samples, there is an other option for sample introduction into the GC and it is called Difficult Matrix Introduction (DMI). The principle of DMI is simple: The dirty extract is injected into a glass micro vial which is placed in the liner. Then the liner is heated up, the (semi-) volatiles are transferred into the column, while non-volatiles residues are retained in the micro vial, which can be deposed after analysis. Analysis using DMI can be automated using a liner exchanger (LINEX) and a capping/decapping station (CDC). It is also possible to use DMI for solid samples for example direct analysis of plastics and washing powder.

Experimental

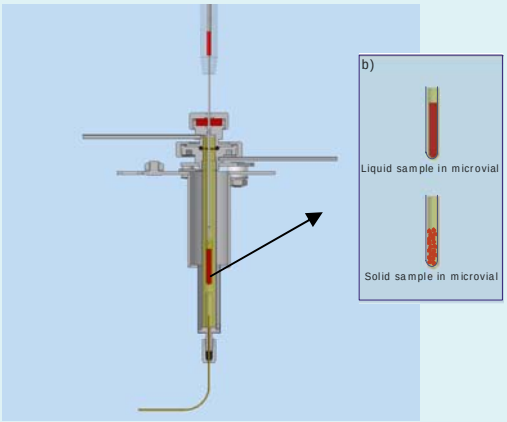


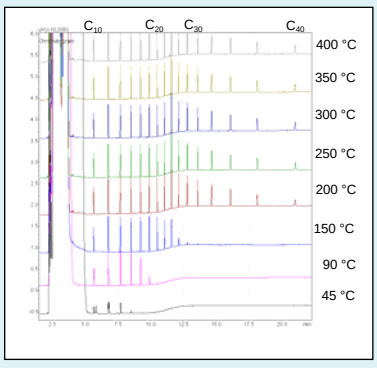
Figure 1: Direct large volume liquid injection of a dirty extract into glass micro vial (b).

Advantages of DMI

- Reduces sample preparation time
- Eliminates potential losses of the volatile compounds during sample preparation
- Volatiles, e.g. solvents, can be removed by venting under controlled conditions
- Non-volatile matrix co-extractives do not contaminate GC-system
- Prolong the life of column and detector (especially MS)
- No contamination of liner
- Minute sample amounts are required (e. g. forensics and life sciences)

Principle of DMI

DMI is based on the principle of selective exclusion. The volatiles and semi-volatiles can be transferred into the column while non/volatile residues are retained in the micro vial.



Automation with LINEX and (de)capping

Effect of open head with GC/MS

With automated liner exchange, it is necessary to open the injector head before the liner is transferred from, or to the injector. When the injector- head is open, air and water can flow into the system. This can be harmful to the GC-column and MS. Therefore a back flush of the injector is necessary. To avoid practical problems, ATAS GL developed a new tool, the T-joint, an practical solution for the use of back flush in GC.

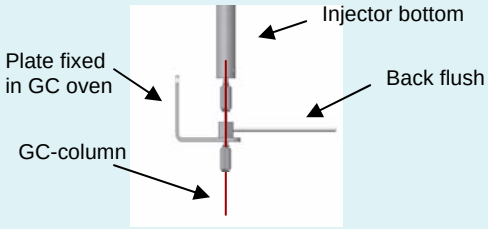
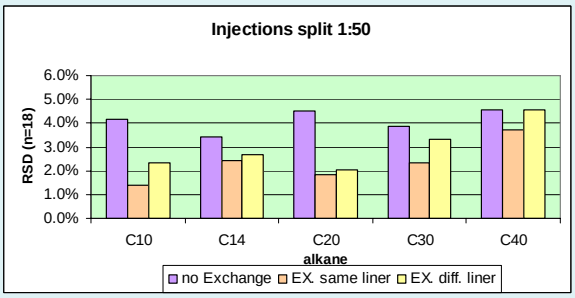


Figure 3: The only difference between normal installation and using the T-joint for back flush is that the column goes through the T-joint to the injector. The original flow path is exactly the same as normal without back-flush. After installation the back-flush is then introduced from the bottom of the injector.

Repeatability of automated DMI analysis



Conditions: alkane mix (12.5 µg/ml); inj.volume 1µl; liner: DMI liner with cup; OPTIC Temp prog.: start 50°C (10 sec.) to 350°C (rate 10°C/s); Flow: start 100 ml/min (before inj.) then split 1:50; column flow: 1ml/min; column: TC5/MS, 0.25mm x 30m (0.25µm film); Oven prog.: 45°C (2 min hold) to 325°C (rate 30°C/min) hold for 11 min.

Conclusions:

-With OPTIC-DMI it is possible to analyze trace compounds in a wide range of different difficult matrices.
-OPTIC-DMI can be used for quantitative analysis of dirty liquid samples or solid samples.
-Exchange of liners doesn't have influence on repeatability.
-LINEX is excellent tool for automation of DMI without losing normal performance analysis.

Reference:

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

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