SHIMADZU

Application Data Sheet

No.35

GCMS

Gas Chromatograph Mass Spectrometer

Analysis of Detergent Residues on Clothing Using DMI-GC/MS

Sample pretreatment is an important step in the analytical process that can determine the validity of analytical results. However, pretreatment is time consuming and can increase analysis costs.
DMI (difficult matrix introduction) is a method for analyzing volatile components by placing micro vials containing actual samples into a liner and heating them in the injection port. By controlling the injection port temperature, this method enables analysis of only the volatile compounds, without introducing any contaminants into the column, which can simplify the pretreatment process.
This datasheet investigates the effectiveness of using GC/MS to analyze non-volatile matrices, by using DMI-GC/MS to analyze detergent residues on clothing.

Experiment

Preparation of Cotton Fabric

Cotton fabric was cut into 3 × 1 cm squares and placed in a solution of detergent dissolved in water. The fabric squares were stirred thoroughly in the detergent solution, then left at room temperature for one hour (equivalent to the washing time in a washing machine). After washing, the fabric squares were rinsed in water (normally public tap water) three times and dried on paper.

Treatment of Cotton Fabric

Cotton fabric was cut into small pieces (5 to 10 mg) and placed in DMI micro vials. Next, the micro vials were placed in DMI liners.

Table 1: Analysis Conditions

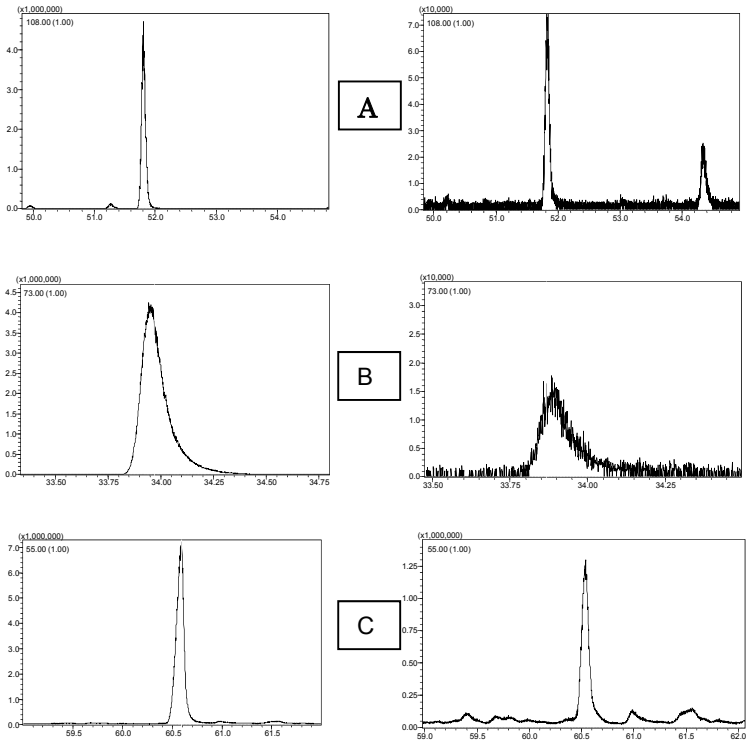
Instrument
- Inlet : OPTIC 4 injector (ATAS GL International BV)
- Liner : L100011, DMI liner with taper (ATAS GL International BV)
- GC-MS : GCMS-QP2010 Ultra (Shimadzu)
- Auto Sampler : AOC-5000 Plus (Shimadzu)
  LINEX (ATAS GL International BV)
- Column : InertCap WAX (60m x 0.32 mm I.D. , df=0.5 µm (GL Science)

[OPTIC-4]
PTV Vaporization chamber temperature:
35℃ → (5℃/ min) → 250℃
Carrier Gas : Helium
Column flow rate : 1.0 ml/min
Split flow rate : Start-1.5min 150 ml/min （Flush Liner）
Heating 1:40
Analyzing 1:40

[GC]
Column oven temperature:
40 ℃（6.3 min）→（15 ℃/min）→130 ℃
→（3 ℃/min）→250 ℃（25min）

[MS]
- Interface temperature : 230℃
- Ion source temperature: 200℃
- Solvent elution time : 9.9 min
- Data sampling time : 10 – 77min
- Measurement mode : Scan
- Mass range : *m/z* 40-350
- Detector voltage : +0 kV (relative value)

Results and Discussion



Fabric samples that were only washed and fabric samples that were washed and rinsed, were measured by DMI-GC/MS. The resulting chromatograms are shown in Figure 1. Many of the same components that were detected in the washed-only fabric were also detected in the washed and rinsed fabric. For example, tetrahydrolinalool, citronellol, butyl glycol, hexadecanol, butylalcohol, and alpha-isomethylionone, among others, were detected. Trace amounts of detergent residues were detected in the rinsed fabric, but the relative amounts differed from the un-rinsed fabric. The %RSD repeatability of retention time and peak area for the un-rinsed fabric using DMI-GC/MS was 4 % and 13 %, respectively.

Fig. 1: Cotton Fabric with Detergent Added (Left) and after Rinsing (Right)
A Benzylalcohol (*m/z* 79); B Tetrahydrolinalool(*m/z* 73); C Hexadecanol (*m/z* 55)

Summary

Using the DMI-GC/MS method enabled identifying trace levels of detergent remaining in washed and rinsed cotton fabric, with almost no sample pretreatment. This method not only reduced analysis costs, but also reduced the risk of volatile component loss during sample pretreatment.
DMI-GC/MS is an excellent method for detecting or screening low-concentration compounds that offers the possibility of simplifying typical sample pretreatment methods

This Application Datasheet was prepared in cooperation with Erwin Kaal and Iwan Horsting at ATAS GL International B.V.

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