SGE Analytical Science

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INDUSTRY APPLICATION

Food

eVol® Use in Sample Preparation for GC-C-IRMS Reduces Interference Caused by Solvents and Plastic Pipette Tips

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http://www.favv.be/home-en/

Introduction

The Belgian Federal Agency for the Safety of the Food Chain (FASFC) is a federal executive agency that sets the operational standards applicable to businesses and integrates all official control and inspection services for the food chain. The FASFC is responsible for setting, implementing and enforcing measures related to the analysis and the management of risks that may affect the health of consumers.

FASFC has five different laboratories in Belgium. The ISORA department of the FASFC laboratory in Gentbrugge is developing methods using IRMS for the assessment of hormones in matrices of animal origin.

Description

In native and natural animal steroids there is a stable ratio between the C12 and C13 isotopes. When an animal receives treatment with hormones, the ratio is modified as synthetic molecules contain less of C13 isotope.

An accurate measurement of this ratio is essential to identify samples containing synthetic molecules. This is so animals that have been treated and deemed unhealthy will be removed from the food chain.

GC-C-IRMS is the technique used by the ISORA department to assess the C13/C12 ratio. Fractions eluted from the GC are combusted in a reactor at 940 °C before being transferred to the IRMS system; a specific MS system able to identify the C Isotope ratio.

Method

The laboratory uses a GC coupled to an MS system and an IRMS through a combustion oven.

8 µL of prepared sample is injected into a 5 % phenyl phase column, 30 m length x 0.25 mm ID x 0.25 µm film thickness.

10 % of the eluted fraction goes to the MS detector and is used to monitor the purity of the sample, while 90 % is directed to the combustion oven and the IRMS. The IRMS is based on the detection of carbon isotopes in CO_2. All fractions are combusted and transformed into CO_2 so it is essential they are free of any contaminants. For this reason, samples have to be prepared with a high level of purity in order to avoid any risk of ghost peaks.

The sample preparation is the critical and most time consuming step of the method.

Samples in methanol are transferred into a GC vial and evaporated, then derivatized with pyridine and acetic anhydride. After the derivatization step, samples are evaporated and re-dissolved in iso-octane and analyzed in the GC-C-IRMS chain.

Previously all steps were completed using manual syringes, classical micro-pipettes and multi-pipettes for multi dispenses. However, it was observed that the solvents used in the methods reacted with the polymers from the pipetting tools creating interfering ghosts peaks and polluting the analysis. ISORA evaluated the use of eVol® digital analytical syringe to improve results.

Results

Today the ISORA department uses eVol for all steps from the sample preparation through to the samples being dispensed into GC Autosampler Vials.

eVol as a positive displacement device solves the issue of solvent volatility and delivers improved accuracy compared with the air displacement mechanism of pipettes.

eVol utilizes SGE's Diamond syringe technology in which samples and solvents are not in contact with polymers, the only contact is with the PTFE plunger tip. Subsequent to eVol being used, all ghosts peaks disappeared, significantly improving the analysis.

The several modes available using eVol (dispense, repeat dispense) allows work to proceed more rapidly thereby increasing laboratory workflow.

Summary

By amending methodology to utilize eVol for sample preparation processes for GC-C-IRMS C isotope ratio analysis, the ISORA department has:

- improved sample preparation conditions and the GC-C-IRMS analysis results by removing the risk of ghost peaks, and increased their workflow,
- been able to work more rapidly while maintaining ergonomic practice for laboratory staff,
- increased liquid handling accuracy.

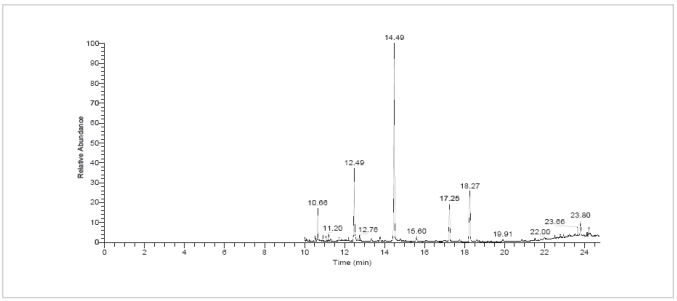

Figure 1. Total Ion Chromatogram when using plastic pipette tips, showing ghost peak at 12.49 minutes.

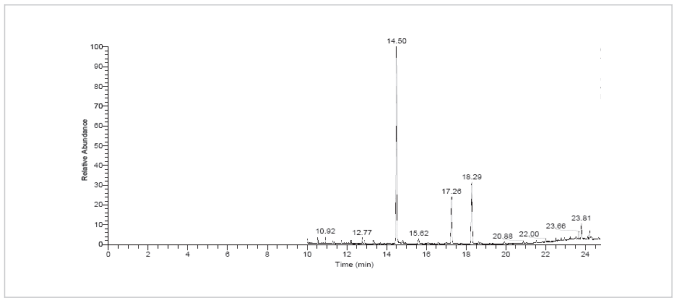

Figure 2. Total Ion Chromatogram when using eVol, showing no ghost peak at 12.49 minutes.

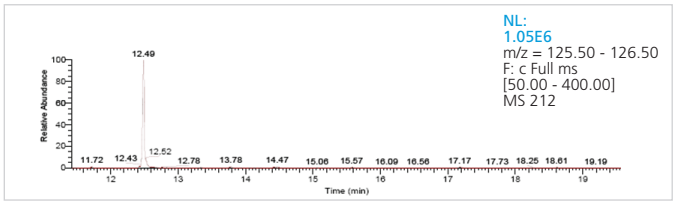

Figure 3. Selected Ion Chromatogram (m/z=125.50-126.50) using plastic pipette tips, showing ghost peak at 12.49 minutes.

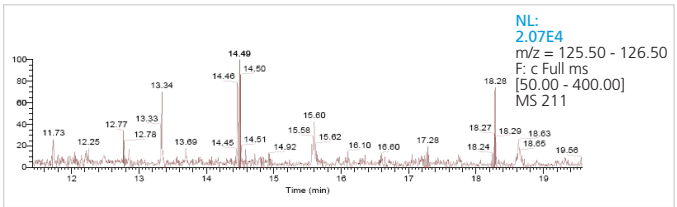

Figure 4. Selected Ion Chromatogram (m/z=125.50-126.50) when using eVol, showing no ghost peak at 12.49 minutes.

Please note the difference in intensity between Figures 3 and 4.

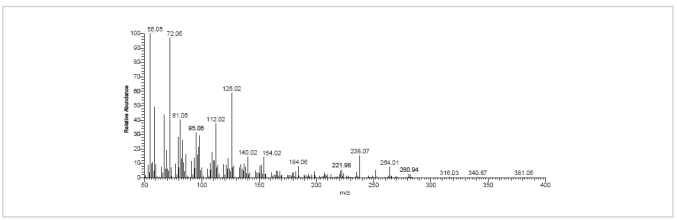

Figure 5. Full Scan (m/z=50.00-400.00) using plastic pipette tips at 12.46-12.50 minutes.

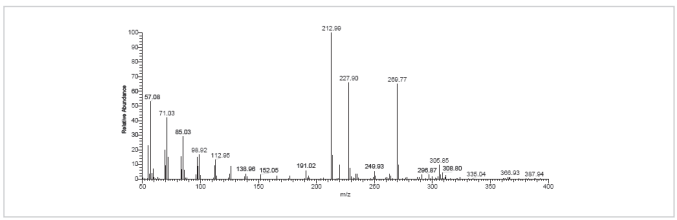

Figure 6. Full Scan (m/z=50.00-400.00) using eVol at 12.46-12.50 minutes.