A new prototype with dual detection GC-IMS/MS for olive oil sample classification

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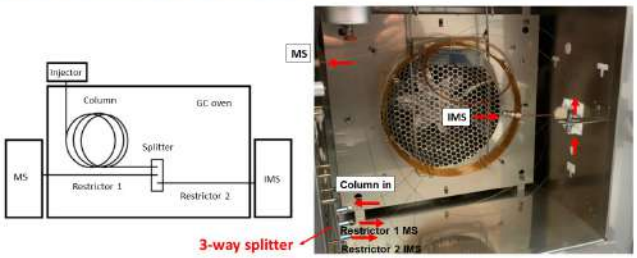
INTRODUCTION

The coupling of **gas chromatography with ion mobility spectrometry** (GC-IMS) has proven to be an effective tool in the classification of olive oils of the three categories (extra virgin, virgin or lampante) due to its robustness, low cost and easy system configuration. Within the framework of the **Innovative Public Procurement Agreement (IPC) "Innolivar"**, the company **Ingeniería Analítica** has developed a new prototype with dual detection ion mobility spectrometry/mass spectrometry (IMS/MS) both coupled to a GC. The prototype must be able to classify olive oil samples in routine laboratories in a simple and fast way.

The **objective** of this research has been to evaluate the prototype configuration proposed by the company and the possibilities of the dual detection IMS/MS in the analysis and classification of olive oil samples.

DIAGRAM OF THE INSTRUMENT



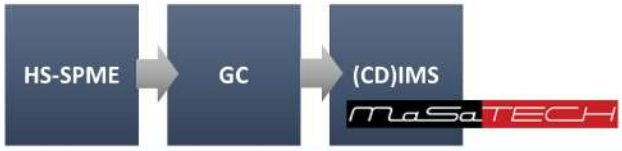
At the end of the chromatographic column, the gas flow is divided by means of a 3-way splitter and a restrictor system to send approximately 50% of the sample to each detector (MS and IMS).

The user can easily remove or adapt the restrictor to work with:

- GC-IMS
- GC-MS
- Dual detection GC-IMS/MS

Firt version of the Prototype

The company Ingeniería Analítica initially presented a prototype based on an IMS analyzer with corona discharge (CD) ionisation source.

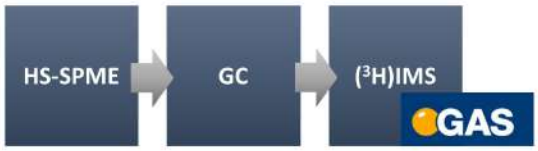


Problems observed with the prototype:

- High nitrogen consumption (700 mL/min). The (CD)IMS analyzer requires N_2 generator.
- High maintenance cost (N_2 generator, gas purification system with moisture and hydrocarbon filters...).
- Unstable RIP signal.

Second version of the Prototype

(CD)IMS was changed by an IMS analyzer based on tritium ionisation source (^{3}H)IMS.



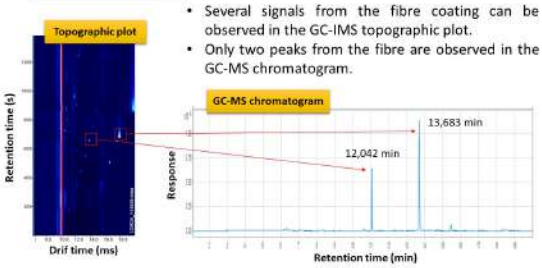
Challenge:

Study of the compatibility of (^{3}H)IMS and MS analysers considering the difference in the sensitivities of both detectors:

- MS detector requires the use of HS-SPME.
- Some ghost signal from the fibre were observed in the topographic plots when HS-SPME was used.
- By working with both detectors, the column flow is divided, and less concentration reaches each detector. Therefore, the signals of the compounds present at low concentration in the olive oil are lost in the MS detector.

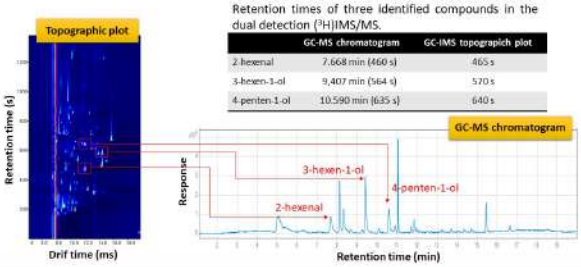
Evaluation of the dual detection (^{3}H)IMS/MS

Blank analysis:



- Several signals from the fibre coating can be observed in the GC-IMS topographic plot.
- Only two peaks from the fibre are observed in the GC-MS chromatogram.

Analysis of an EVOO sample:



Retention times of three identified compounds in the dual detection (^{3}H)IMS/MS.

	GC-MS chromatogram	GC-IMS topographich plot
2-hexenal	7.668 min (460 s)	465 s
3-hexen-1-ol	9.407 min (564 s)	570 s
4-penten-1-ol	10.590 min (635 s)	640 s

- High correlation in the retention times between signals appearing in GC-IMS topographic plot and peaks in GC-MS chromatogram.
- Only compounds at high concentration in olive oil samples can be identify with this dual detection (^{3}H)IMS/MS.

Classification results

	GAS HS-GC-(^{3}H)IMS (PCA-LDA / KNN)		MaSaTECH HS-SPME-GC-(CD)IMS (Random Forest)		HS-SPME-GC-MS (OPLS-DA)
	130 samples*	320 samples	130 samples*	320 samples	150 samples
Calibration success	80 %	96 %	81 %	80 %	81 %
Validation success	90 %	85 %	95 %	91 %	74 %

*Samples evaluated by two sensory panels.
(Prelimiary result, number of samples will be increased)

CONCLUSIONS

- The methodology based on (CD)IMS analyzer provides the best validation rates. However, the high maintenance cost and the lack of robustness of the detector make the implementation of this methodology in routine laboratories unfeasible.
- The methodology based on (^{3}H)IMS analyzer provides validation rates higher than 85%. Moreover, validation results are improved with the use of samples evaluated by two sensory panels, since the transfer of the sensory panel error to the chemometric model is avoided. This instrument is also robust and easy to maintain and to use in a routine laboratory.
- The high sensitivity of (^{3}H)IMS makes compatibility with SPME and the dual detection IMS/MS difficult. Since column flow is split 50% to each detector, only the compounds at high concentrations in the olive oil can be detected and identified in MS detector. Moreover, several signal from the fiber coating can be observed in the topographic plots.
- SPME-GC-MS provides promising calibration and validation rates, although these data are still preliminary.

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