Methodological developments for analysis of non-intentionally added substances (NIAS)

Gas chromatography combined with mass spectrometry (GC-MS) is a basic technology for the identification and quantification of volatile and semi-volatile compounds. One of its major advantages is the availability of universal libraries of fragmentation patterns of molecules. We present here different methodological developments of the analysis of contaminants in packaging and additives using GC-MS.

1. GC-MS combined with QuEChERS for analysis of agricultural by-products

Gas chromatography combined with mass spectrometry (GC-MS) consists of injecting compounds in gaseous form, which come from the vaporization of a concentrated liquid extract. This mixture of compounds is carried by a carrier gas to a chromatographic column, placed in a thermostatically controlled oven, where a selective separation takes place. The identification of the eluted products is done with a mass spectrometer. **GC-MS combined with QuEChERS allowed us to develop an innovative method for the analysis of NIAS especially contaminants in Deodorizer Distillates (DD) used as co-product in food packaging.**

Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS)

6890 GC-5975 MS (Agilent Technologies)

DB-5ms capillary column (30m × 0.32mm × 0.5µm)

Helium, 1.1 mL/min
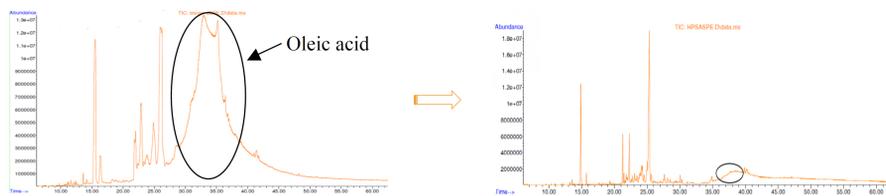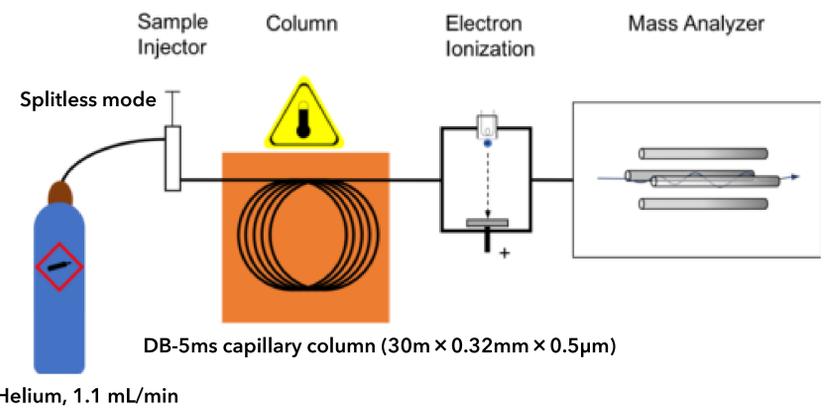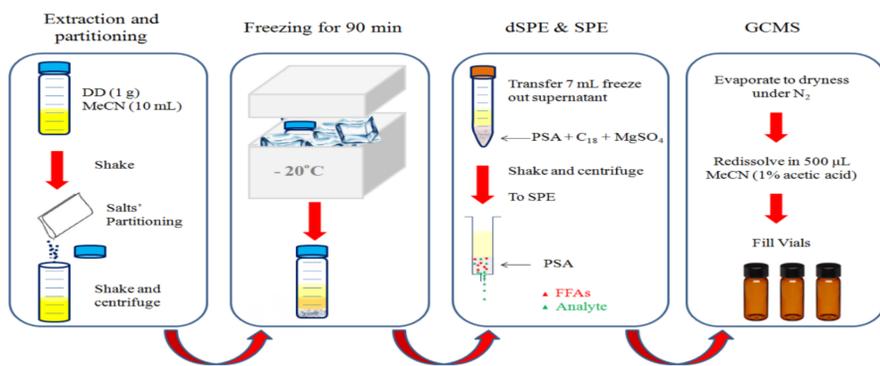
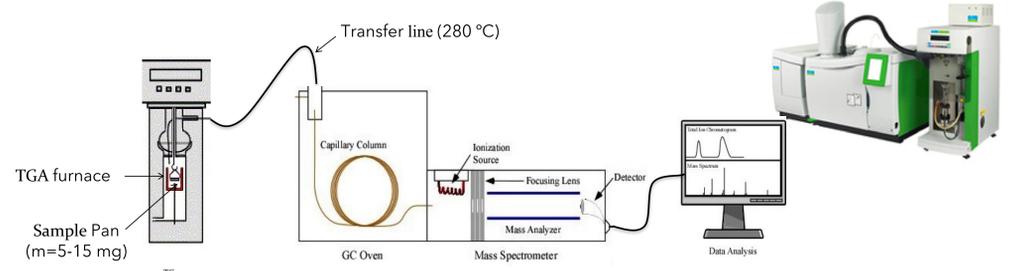**Full scan of colza DD extracted with n-hexane**
Full scan of colza DD extracts obtained using QuEChERS methodology

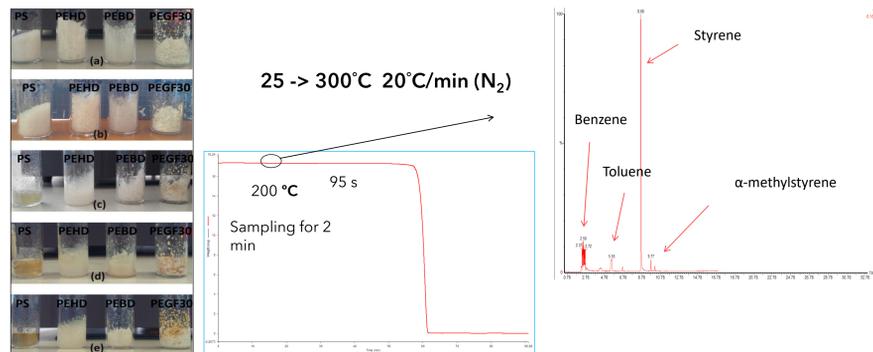| Pesticide	Correlation coefficient	LOD (mg/kg)	LOQ (mg/kg)	Recovery rate (%)
Lindane (HCH)	0.977	0.37	1.11	80 (100 mg/kg) 40.2 (50 mg/kg) 35.3 (10 mg/kg)
Malathion	0.999	5.31	17.69	78 (100 mg/kg)

2. Analysis of thermal degradation products using TGA-GC-MS

Thermogravimetric analysis (TGA) coupled with gas chromatography mass spectrometry (GC-MS) is a powerful tool to chemically identify mass losses, released gases or degradation products during the thermal cycle. In fact, heating a sample by TGA induces the release of volatile compounds initially present in the material or generated during the heating cycle. These volatiles are then transferred to the GC, separated by the chromatographic column and identified by the MS. *Our team uses this equipment to control and analyze NIAS contained in FCMs and to study the thermal degradation products of FCM generated during processing.*
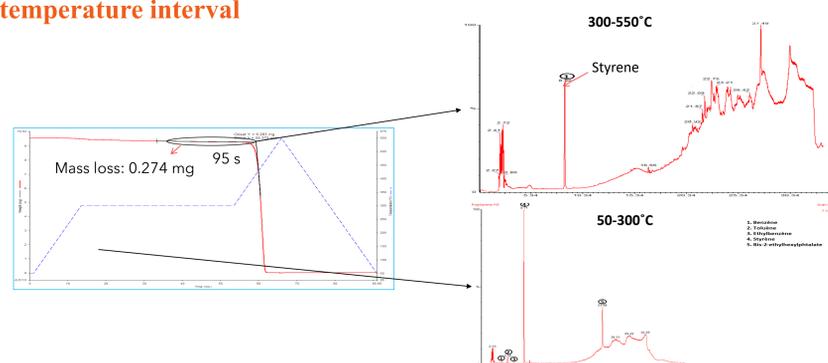
**PerkinElmer® Pyris™ 1 TGA - PerkinElmer Clarus® 680 GC Clarus® SQ 8 MS**
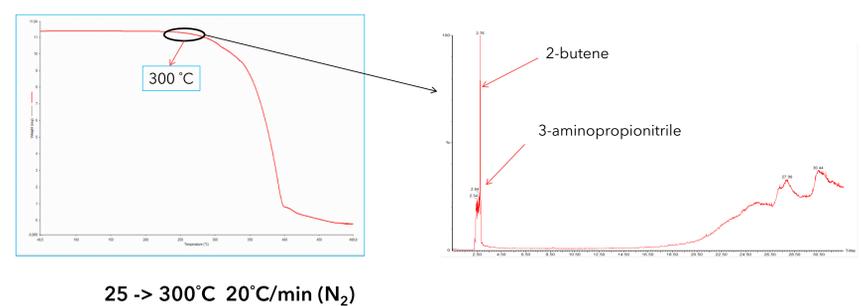

Online measurement - Thermal degradation of PS

$25 \rightarrow 300°C$ $20°C/min$ (N_2)

200 °C 95 s Sampling for 2 min

Styrene
Benzene
Toluene
α-methylstyrene

Online measurement – Desorption of NIAS ⇒ Isothermal test on PET pellets

Sampling for 30 s at 77 °C

Different strategy: flash heating after isotherm and sampling over a large temperature interval

Mass loss: 0.274 mg 95 s

300-550°C
Styrene

50-300°C
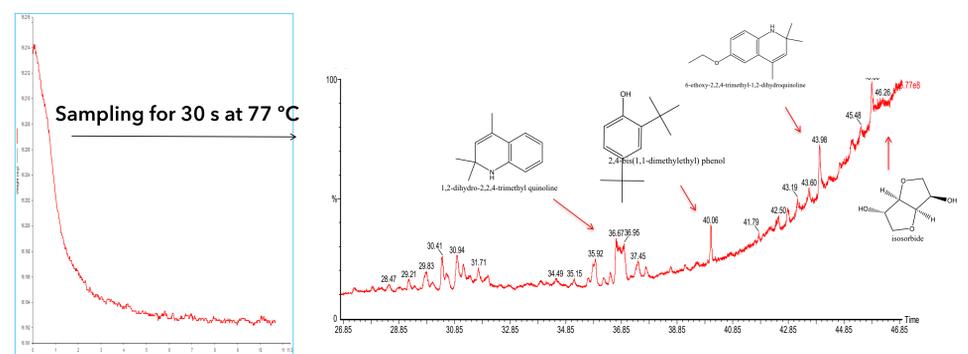
Online measurement – Thermal degradation of Irganox 1076

300 °C

2-butene
3-aminopropionitrile

$25 \rightarrow 300°C$ $20°C/min$ (N_2)

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In-house methodological developments for NIAS analysis

UMT SAFEMAT SAFETY OF PACKAGING