DETERMINATION OF MOSH AND MOAH BY GC×GC-TOFMS.

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Abstract

Nowadays across the European Union food contamination by mineral oils from production processes and packaging is becoming a serious problem for public health institutions and governments. In this study comprehensive two-dimensional gas chromatography (GC×GC) in combination with time-of-flight mass spectrometry was evaluated, in order to find a robust one run analytical method.

LECO GC×GC system takes advantage of a dual-stage, quad-jet thermal modulator positioned between the two columns and a secondary oven allows independent temperature control of the second dimension column, combined with high acquisition rate, full range TOF mass spectra.

The combination of two different polarity columns led to effective separations between compound families, identifications within families were easily reached by high acquisition rates TOFMS systems and ChromaTOF software classification capabilities defined chromatogram regions to locate clearly each compound family.

Introduction

A wide range of derived from petroleum distillation compounds are contained in mineral oil. MOSH, mineral oil saturated hydrocarbons, are linear and branched alkanes and cyclic compounds such as naphthenes. MOAH, mineral oil aromatic hydrocarbons, contains the aromatic fraction of mineral oil usually alkylated.

Contamination is possible by several ways (environment, heating fluids, packaging…) but nowadays inks present in paper or recycled paper to product cardboard are the heart of concern of the issue. Mass range contained in inks is between C13- to C25, and paperboard can be contaminated by up to C45. Contamination involves both the MOSH and MOAH fractions, but no clear criteria about daily intake and LOQ are on use in Europe and different opinions are published year by year by different authorities on food safety.

In regard to analytical methods two choices have been proposed, off-line method (sold phase extraction, LC preparative, and GC) and the most popular on-line heart-cutting LC-GC-FID. In this study sample extraction from a LC-GC interface are injected onto the PEGASUS® BT 4D the LECO´s GC×GC-TOFMS system.

Sample Treatment

Curcuma samples the were pre-separated in MOSH and MOAH fractions by a using a commercial LC-GC interface. The pre-separated fractions have been enriched (±350 uL per fraction after evaporation of the solvent) and Injected onto the GC×GC system.

Experimental Conditions

INLET	1ml/min constant flow splitless
	330ºC entire run
COLUMNS	Rxi-17SILMS 12m, 250µm, 0.25µm
	Rxi-1HT 1.05m, 250µm, 0.1µm
PRIMARY OVEN	40ºC (1min); 5ºC/min 360ºC (10 min)
SECONDAY OVEN OFFSET	+7ºC
MODULATOR OFFSET	+15ºC
TRANSFER LINE	340ºC
MODULATOR	Hot pulse 1.65s (until 2498.35s)
	Hot pulse 1.80s (until end of the run)
ACQUISITION RATE	200 spectra/s
MASS RANGE	40-700 um
ION SOURCE TEMPERATURE	280ºC
ELECTRON ENERGY	70eV

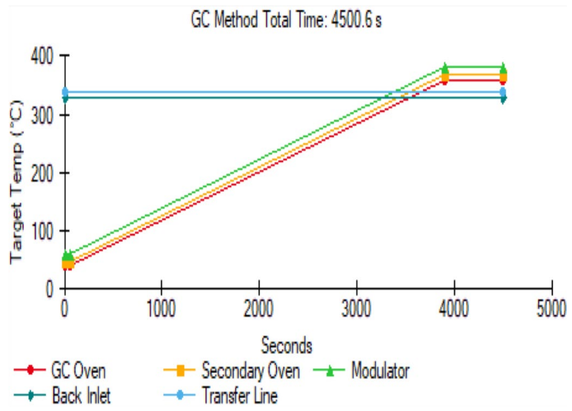


Figure 1: Temperature plots

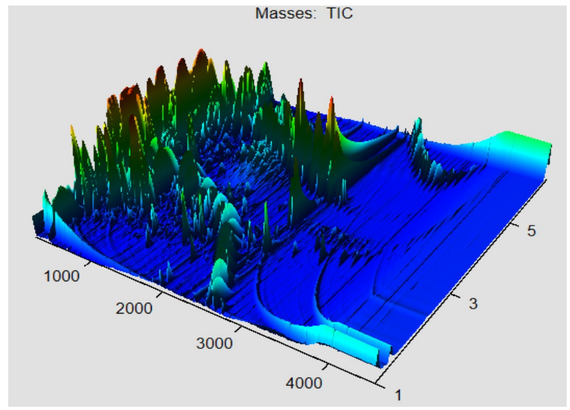
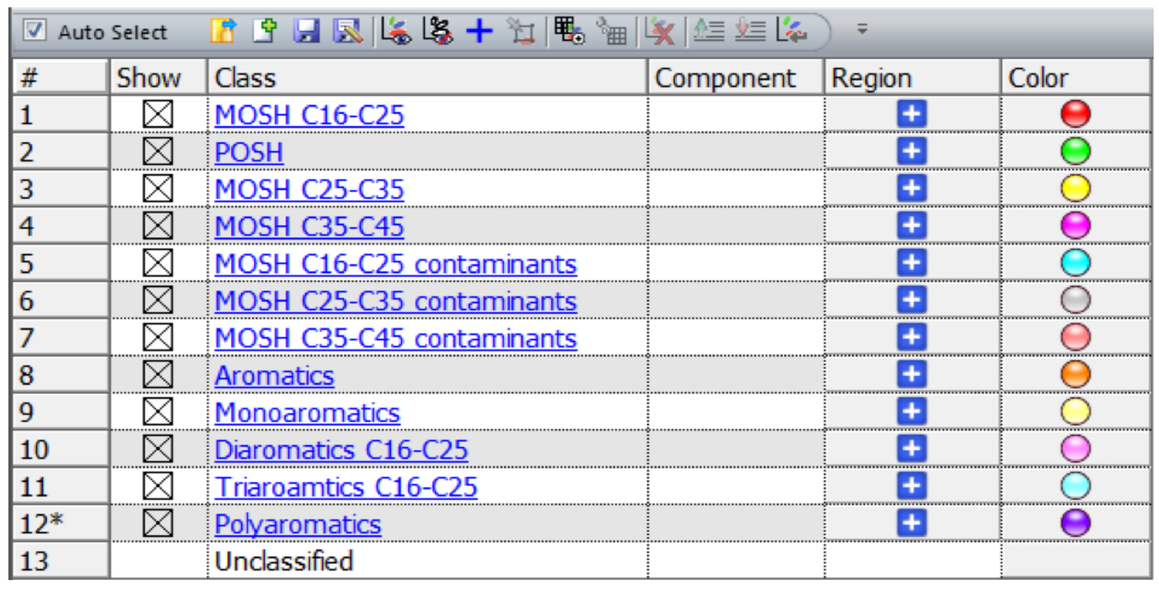


Figure 2: Surface plot chromatogram



#	Show	Class	Component	Region	Color
1	☒	MOSH C16-C25		+	
2	☒	POSH		+	
3	☒	MOSH C25-C35		+	
4	☒	MOSH C35-C45		+	
5	☒	MOSH C16-C25 contaminants		+	
6	☒	MOSH C25-C35 contaminants		+	
7	☒	MOSH C35-C45 contaminants		+	
8	☒	Aromatics		+	
9	☒	Monoaromatics		+	
10	☒	Diaromatics C16-C25		+	
11	☒	Triaroamtics C16-C25		+	
12*	☒	Polyaromatics		+	
13		Unclassified			

Figure 3: Classification summary applied

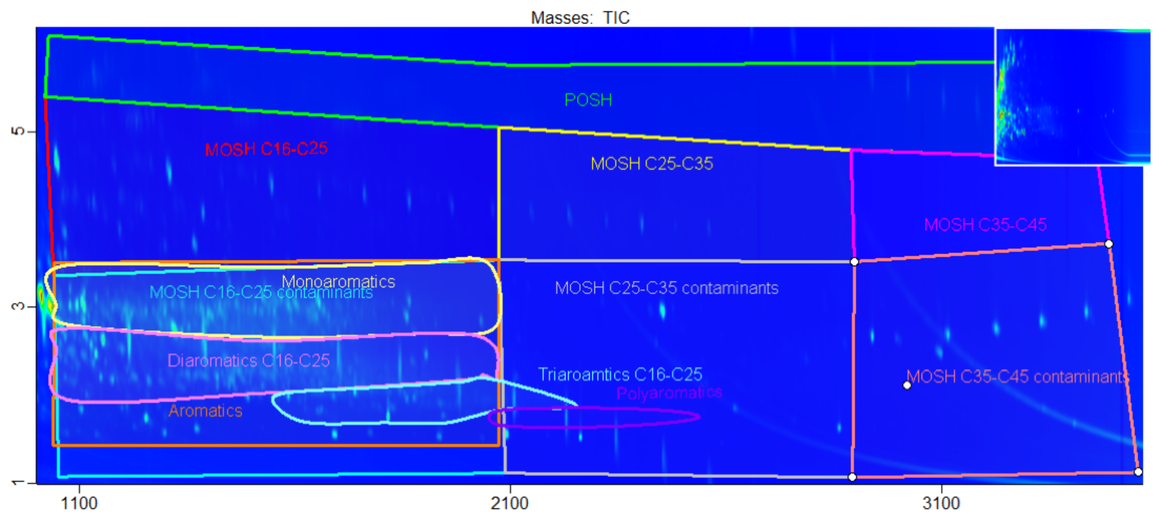


Figure 4: Enlarged chromatogram contour plot. Chromatographic regions of obtained once the classification data processing from method is applied to the sample.

Conclusions

This study demonstrates the benefits of LECO's *Pegasus* BT4D GC×GC-TOFMS and ChromaTof software capabilities in MOSH/MOAH determination. Basically, are the following: Increased resolution compared 1D systems, confirmation of MOSH/MOAH contamination, difference between MOSH and polymers synthetic polymers (PP, PE) difference between MOAH and biogenic substances (terpenoids) due to the obtention of high-quality mass spectra, finding the source of contamination (Figure 5: *Pegasus* BT 4D markers), prevention of errors in MOAH identification.

Figure 5: Pegasus BT 4D

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