Comprehensive LCxGC and LCxGC–TOF MS for the characterisation of edible oils and fats

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INTRODUCTION

The determination of the fatty acid composition of edible oils and fats is one of the most frequently performed analyses in food industry. This because of label claim regulations and health aspects on one hand, and processing and product properties on the other. The problem in the detailed characterisation of oils and fats is the enormous complexity of the tri-acylglyceride (TAG) mixtures. This work shows the results of a feasibility study on on-line automated comprehensive LCxGC of edible oils with a robotic autosampler as the interface between the LC and the GC-ToF MS system. Information from the LC, the GC and the ToF MS are used for detailed characterisation of the compounds at the molecular level.

INSTRUMENTATION

- Focus sample preparation robot (ATAS GL)
- Optic 3 injector (ATAS GL)
- 6890 GC (Agilent)
- Pegasus III ToF MS (LECO)
- Capillary GC column, Ultimetal SimDist 5 m x 0.53 mm x 0.09 µm (Varian)
- Aliance 2690 HPLC system (Waters)
- HPLC column, Ag^+-phase, 25 cm x 2 mm x 5 µm (Varian)

Figure 1:
LCxGC–ToF MS system.
Ag^+-phase column for LC separation (left insert),
Syringe with side entrance used as interface between LC and GC (right insert).

ANALYSIS METHOD

- A solution of 10 mg/ml of the fat/oil is prepared in dichloromethane
- 10 µl is injected on the Ag^+ ion exchange column.
- The LC effluent is transferred into the Focus injection syringe through the side entrance of the syringe (See figure 1).
- When the GC system is ready, the syringe is inserted into the injector. Next the LC pump is started to transfer a 100 µl fraction into the injector. After injection the pump is stopped, the syringe is withdrawn from the injector and the GC analysis is started.
- After completion of the entire LCxGC analysis the GC–ToF MS data of all LC fractions is processed and a bubble is plot constructed.

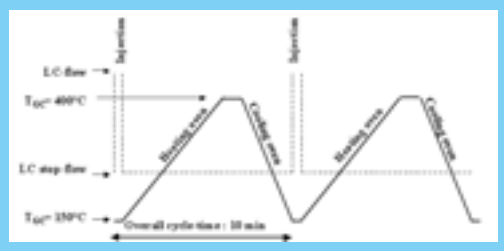

Figure 2: Analysis diagram: Sequence of events.

DIET BUTTER

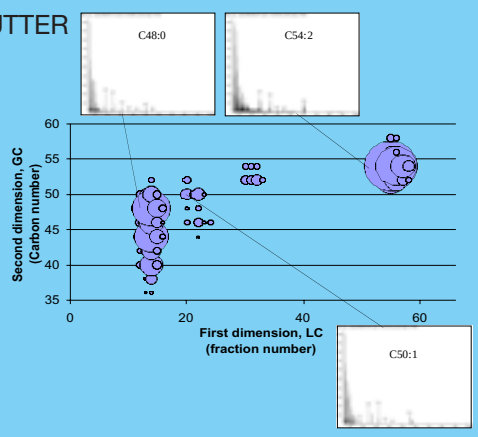

Figure 3A: Contour plot of on-line LCxGC–ToF MS analysis of diet butter sample with mass spectra shown as inserts. Sample: 10 mg/ml, Injection volume: 10 µl, Fraction size: 100 µl, LC flow: 0.2 ml/min, Fraction 1 – 34: eluent A (dichloromethane:acetone=99.5:0.5), Fraction 35-68: eluent B (Acetonitril), GC oven: 150°C/ballistic heating/400°C (1 min), Column flow: 13 ml/min, ToF MS: mass range 50 - 1000, 20 specs/sec.

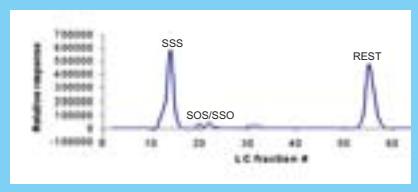

Figure 3B: Reconstructed LC chromatogram of diet butter.

REAL BUTTER

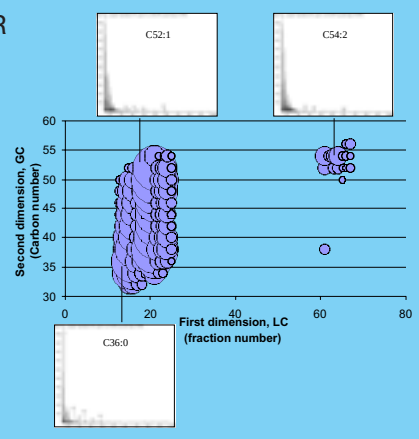

Figure 4A: Contour plot of on-line LCxGC–ToF MS analysis of real butter sample with mass spectra shown as inserts. Sample: 10 mg/ml, Injection volume:. 10 µl, Fraction size: 100 µl, LC flow: 0.2 ml/min, Fraction 1 – 40: eluent A (dichloromethane:acetone=99.5:0.5), Fraction 41-80: eluent B (Acetonitril), GC oven: 150°C/ballistic heating/400°C (1 min), Column flow: 13 ml/min, ToF MS: mass range 50 - 1000, 20 specs/sec.

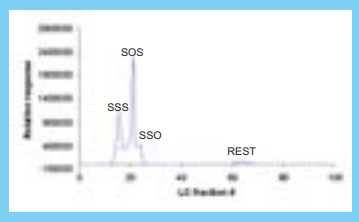

Figure 4B: Reconstructed LC chromatogram of real butter.

CORN OIL

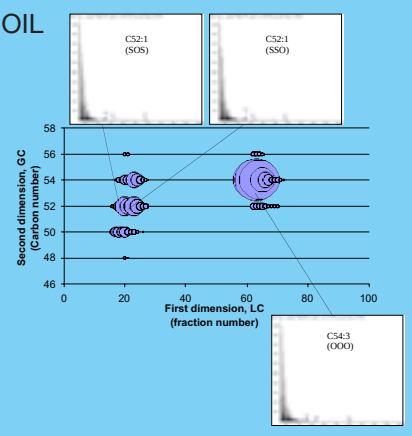

Figure 5A: Contour plot of on-line LCxGC–ToF MS analysis of a corn oil sample with mass spectra shown as inserts. Sample: 10 mg/ml, Injection volume:. 10 µl, Fraction size: 100 µl, LC flow: 0.2 ml/min, Fraction 1 – 40: eluent A (dichloromethane:acetone=99.5:0.5), Fraction 41-80: eluent B (Acetonitrile), GC oven: 150°C/ballistic heating/400°C (1 min), Column flow: 13 ml/min, ToF MS: mass range 50 - 1000, 20 specs/sec.

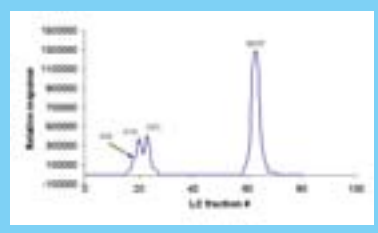

Figure 5B: Reconstructed LC chromatogram of corn oil.

CONCLUSION

A system for on-line comprehensive LCxGC was constructed and succesfully applied to the analysis of TAGs from edible oils and fats. The device uses a standard robotic autosampler as the interface. The advantage of detection by ToF MS is the possibility to produce pure mass spectra that allow a more detailed identification of the TAGs. The system is very flexible. New applications are currently being studied.