Analysis of Triglycerides by Liquid Chromatography / Mass Spectrometry Application

Food

Author

Hiroki Kumagai

Abstract

Triglycerides were readily analyzed using liquid chromatography / mass spectrometry with atmospheric pressure chemical ionization in positive ion mode.

Background

Oil is a primary component of many foods, such as cooking oils and dairy products. Consequently, it is desirable to conduct compositional analyses of oil-based fatty acid components in food. Such analyses may use either gas chromatography (GC) or high-performance liquid chromatography (HPLC), but the GC methods have problems with the complexity of sample preprocessing, which requires saponification.

With HPLC a direct analysis is possible; however, these methods are low in sensitivity because the compounds of interest do not absorb ultraviolet and separation is barely adequate.

In this study, six triglycerides (Table 1) with identical fatty acid compositions were analyzed using liquid chromatography/mass spectrometry (LC/MS) and atmospheric pressure chemical ionization (APCI) as the ionization mode.

Table 1. Chemical Structure of Triglycerides

$CH_2 - OCO - R_1$
$CH - OCO - R_2$
$CH_2 - OCO - R_3$

Triglyceride	$R_1=R_2=R_3$
Trilaurin	C_{11}
Trimyristin	C_{13}
Tripalmitin	C_{15}
Tristearin	C_{18}
Triolein	$C_{18:1}$
Trilinolein	$C_{18:2}$

Method

- Instrument: Agilent 1100 LC/MS with APCI in positive mode

  Mass range: 100 to 1000 *m/z*

  Drying gas: N_2 4 L/min, 350 °C

  Nebulizer: N_2 50 psi

  Fragmentor: 160 V

  Vaporizer: 400 °C

- LC Conditions:

  Mobile phase: $(CH_3)2CO/H_2O$ (98/2)

  Flow rate: 1.0 mL/min

  Oven temperature: 40 °C

  Injection volume: 15 µL

- Column: Develosil ODS-UG3, 4.6 mm id × 75 mm long

Results

The following figures show total ion chromatogram (TIC) and selected ion mode (SIM) chromatograms, and mass spectra for the selected triglyceride standards.

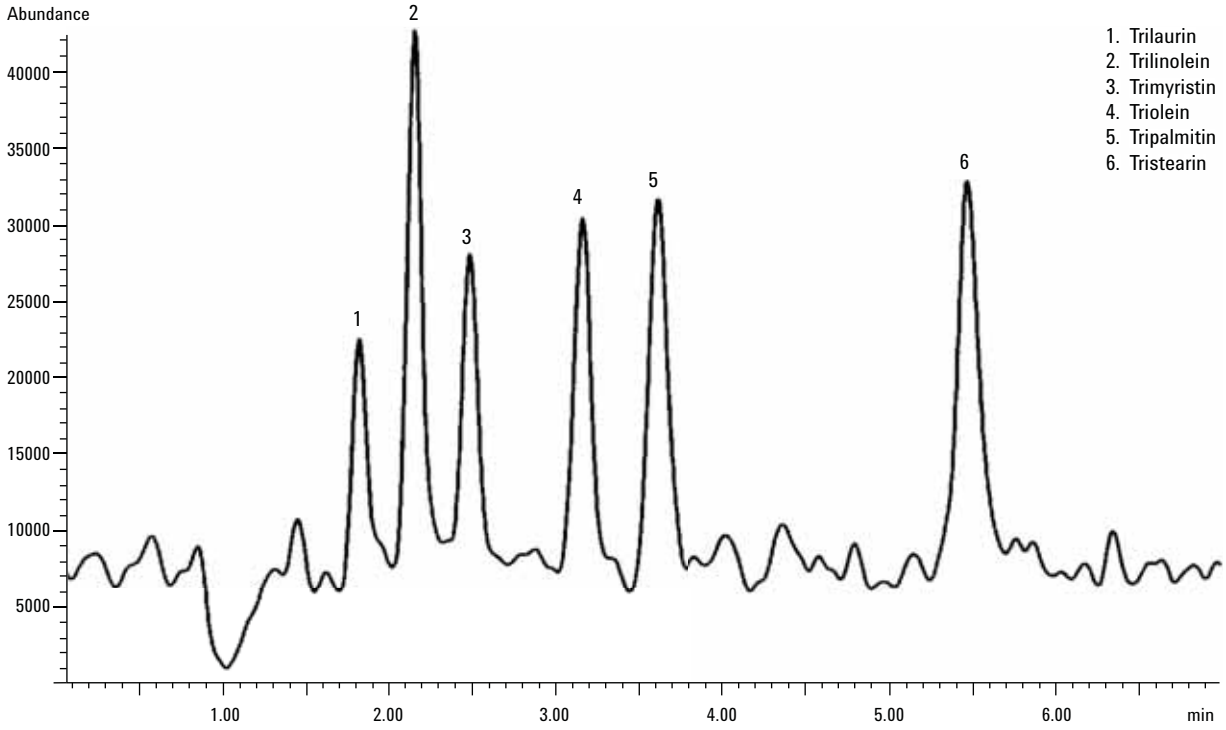

Figure 1. TIC of triglyceride standards, each at 0.2 ppm.

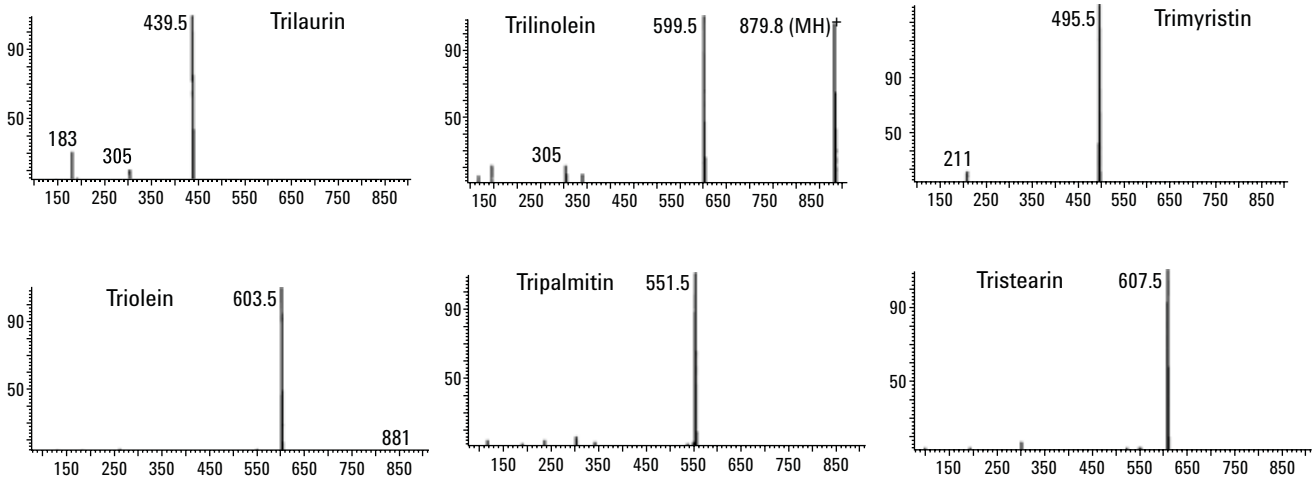

Figure 2. Mass spectra of individual triglyceride standards at 0.2 ppm.

The tallest peak in each case represents the $[MH\text{-}HOOCR]^+$ ion created after the loss of a fatty acid group from the protonated psuedomolecular ion. With the exception of Trilinolein, the protonated psuedomolecular ion was not observed.

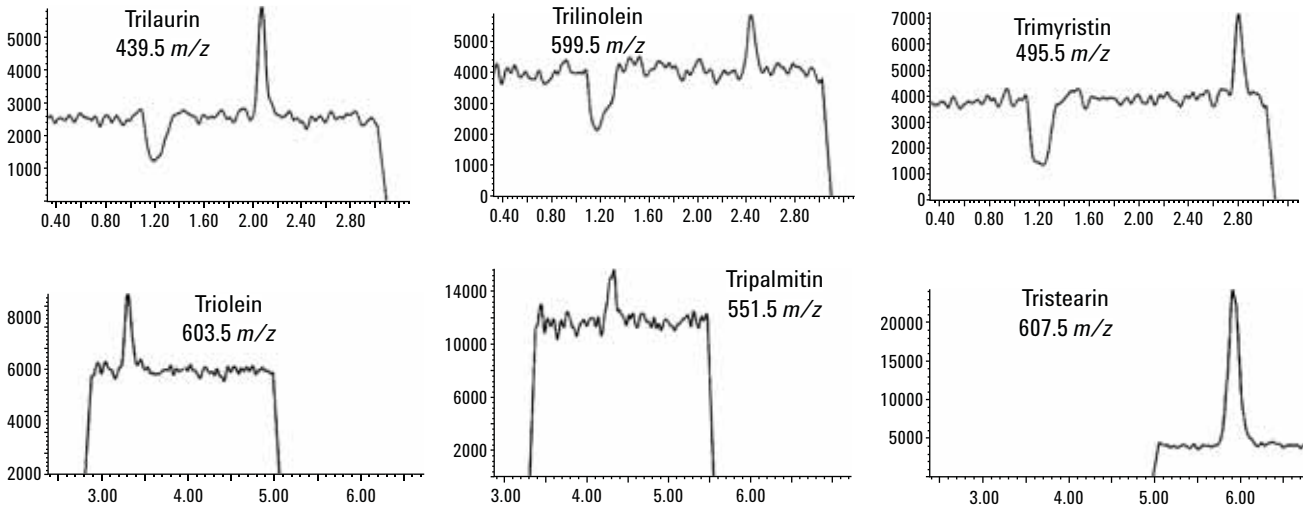

Figure 3. SIM chromatograms of individual triglyceride standards at 1 ppb.

The protonated molecular ions were rarely observed—trilinolein in the scan mode is the exception—and the base peaks consisted of fragmented ions from which fatty acid had been detached.

Sensitivity was favorable, extending down to 0.2 ppm in TIC mode. In the SIM mode, measurements at 1 ppb were possible by selecting the base peak in the mass spectrum of each composition of monitored ions. With this technique, it is possible to measure fat-soluble substances such as triglycerides with a high degree of sensitivity.

Conclusions

LC/MS, with APCI in positive ion mode, readily detected selected triglyceride standards with high sensitivity.

The analytical peaks were the positvely charged fragmented ions from which fatty acid had been detached.

SIM mode yielded sensitivity to 1 ppb.

Hiroki Kumagai is an application chemist at Agilent/Yokogawa Analytical Systems, Tokyo, Japan.

www.agilent.com

www.agilent.com/chem

Printed in the USA
August 22, 2006
5988-4235EN

Agilent Technologies