

AccuTOF-GCv Series

Analysis of High Boiling Point Compounds

Cholesterol and Irganox® 1010

Introduction

The high boiling point compounds cholesterol and Irganox® 1010, an anti-oxidant additive for polymers, were analyzed by GC/MS. The analyses were performed using electron ionization (EI) and field ionization (FI), which is a soft ionization method, to show the difference in mass spectral patterns that result from these two ionization techniques.

Methods

Samples

Cholesterol	1 mg/mL (in methanol)
Irganox® 1010	1 mg/mL (in methanol)

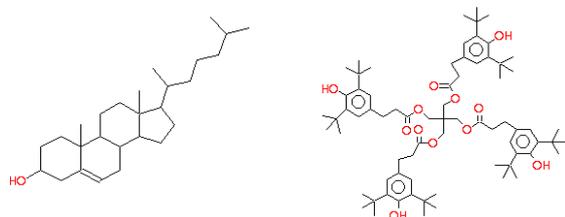


Fig. 1 Structural formulas of Cholesterol (left) and Irganox® 1010 (right)

GC conditions

Column:	DB-1HT, 7 m x 0.25 mm, 0.10 µm
Injector:	380 °C, 2 mL/min (constant flow mode)
Injection mode:	EI: split (80:1), FI: split (10:1)
Oven:	120 °C (1 min) → 30 °C/min → 380 °C (10.33 min)

MS conditions

Mass spectrometer:	JMS-T100GC "AccuTOF GC"
Ionization mode:	EI: Electron energy: 70 eV Ionization current: 300 µA Ion source temperature: 300 °C
	FI: Cathode potential: -10 kV Emitter current: 35 mA for 30 msec between spectra Ion source temperature: 250 °C
GC interface temperature:	350 °C
Acquired mass range:	<i>m/z</i> 35 – 1,400
Spectral recording interval:	0.4 sec

Results

Reconstructed total ion current chromatograms (RTICCs) are shown in Fig. 2. During the GC/EI analyses, the GC column bleed ions (e.g., *m/z* 207, 281) were strongly observed which resulted in the RTICC baseline to rise significantly as the GC oven temperature increased. Since cholesterol eluted at around 240°C, the baseline rise was not as significant which resulted in an easily identifiable chromatographic peak. Conversely, the Irganox® 1010 peak eluted at 380 °C, which was the final oven temperature, so the baseline was much higher than for cholesterol.

As for the GC/FI analyses, the GC column bleed was minimally ionized which resulted in a flat RTICC baseline. Consequently, both the cholesterol and Irganox® 1010 peaks were easily observed in the RTICCs.

The mass spectra observed for each technique are shown in Fig. 3. The EI mass spectra showed a number of fragment ion peaks for both analytes with no molecular ion peak observed for the Irganox® 1010. In contrast, there were only a few fragment ion peaks observed in the FI mass spectra. Furthermore, the molecular ion peaks were the base peaks for both compounds in the FI spectra.

Conclusions

The JMS-T100GC "AccuTOF GC" is capable of performing GC/MS analyses of very high boiling point compounds, such as Irganox® 1010. The ionization method can be chosen from EI, FI, and also chemical ionization (CI). By combining FI, which provides molecular ion information for most analytes, and EI, which provides fragment information, a more accurate and confident qualitative analyses becomes possible.

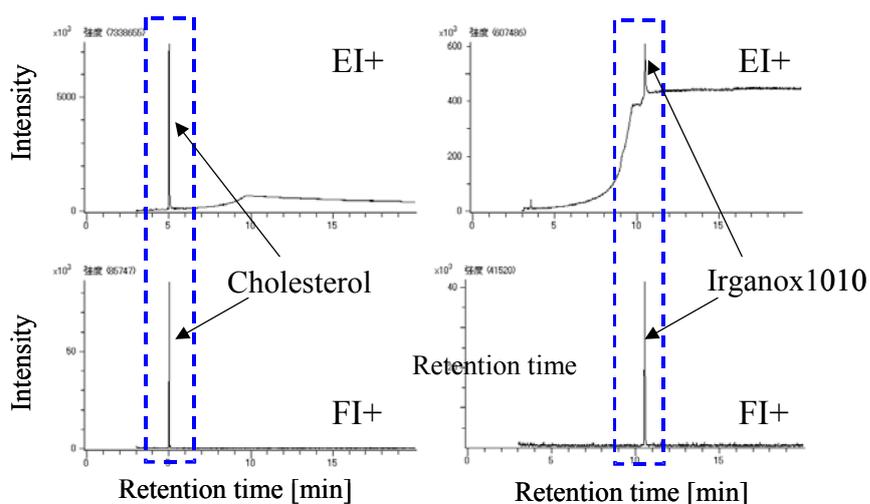


Fig. 2 RTICs of Cholesterol (left) and Irganox® 1010 (right)

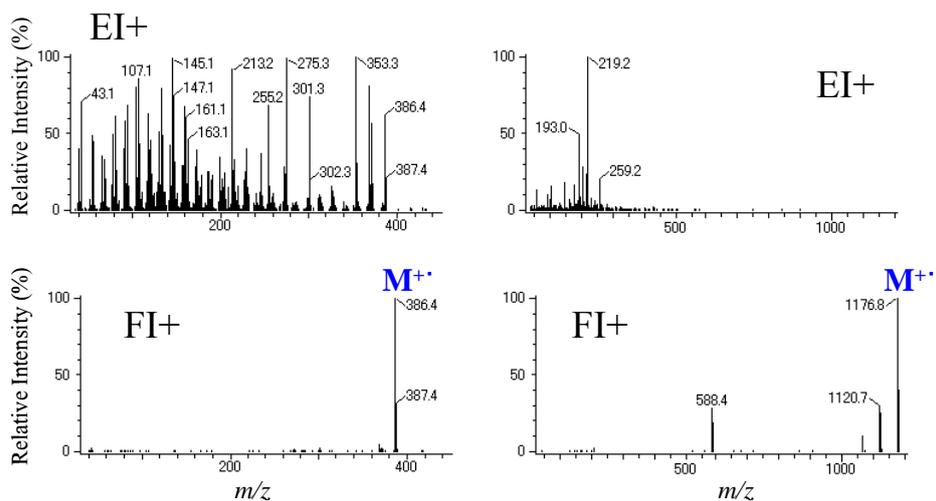


Fig.3 Mass spectra of Cholesterol (left) and Irganox® 1010