

## GC/TOFMS analysis of high boiling point compounds ~ cholesterol and Irganox® 1010 ~

### Introduction

As examples of GC/MS analyses of high boiling point compounds, cholesterol and Irganox® 1010, an anti-oxidant additive for polymers, were analyzed. The analyses were performed with electron ionization (EI) and field ionization (FI,) which is a soft ionization method.

### Methods

<u>Samples</u>	cholesterol	1 mg/mL (in methanol)
	Irganox® 1010	1 mg/mL (in methanol)

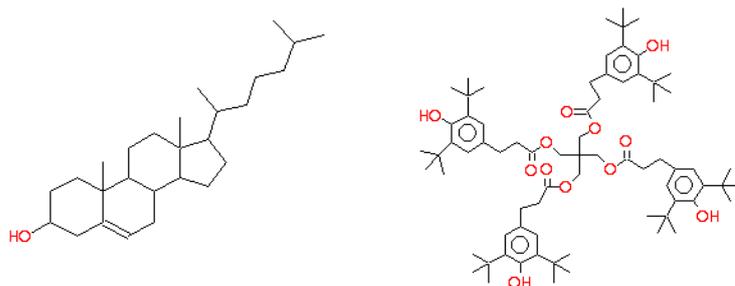


Fig.1 Structural formulas of Cholesterol (left) and Irganox® 1010 (right)

### 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:  $m/z$  35 – 1,400

Spectral recording interval: 0.4 sec

## Results and Discussion

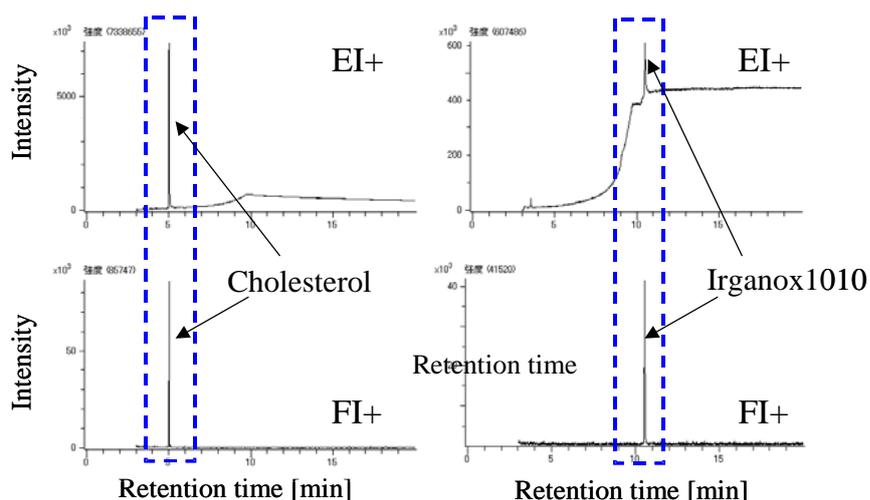


Fig.2 RTICCs of Cholesterol (left) and Irganox® 1010 (right)

Reconstructed total ion current chromatograms are shown on Fig. 2. In GC/EI analyses, ions derived from GC column bleed (e.g.,  $m/z$  207, 281) were strongly observed. As a result, the baseline of RTICC went up as GC oven temperature was programmed up. Since cholesterol eluted at around 240 °C, the base line rise was not so significant and the chromatographic peak was easily seen. Irganox® 1010 peak eluted at 380 °C (i.e., at the final oven temperature,) was on a high baseline.

In GC/FI analyses, the GC column bleed was hardly ionized and the RTICC baseline was flat. Both cholesterol and Irganox® 1010 peaks were easily detectable on RTICCs.

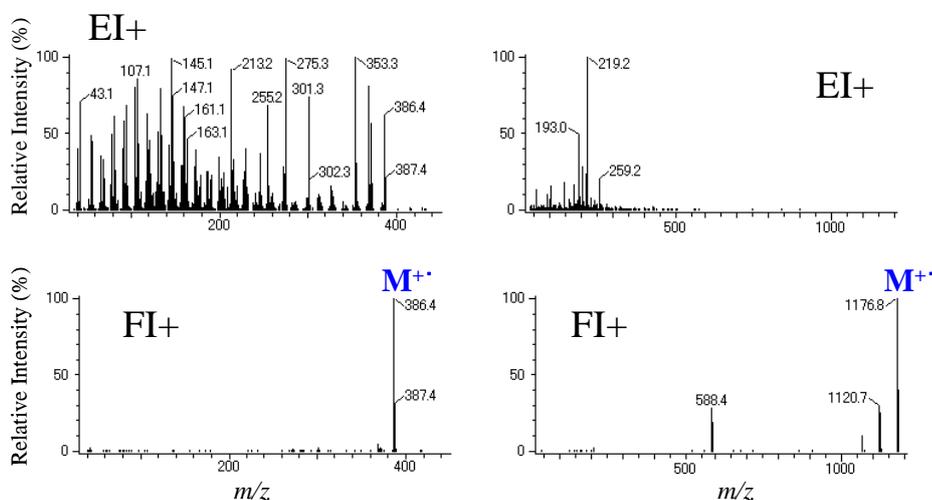


Fig.3 Mass spectra of Cholesterol (left) and Irganox® 1010 (right)

The mass spectra observed are shown on Fig. 3. In EI mass spectra, a lot of fragment ion peaks and no molecular ion peak from Irganox® 1010 were observed. In contrast, there were only a few fragment ion peaks in FI mass spectra and the molecular ion peaks were observed as base peaks for both compounds.

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 chemical ionization (CI.) By combining FI, with which you can observe molecular ion from most analytes, and EI, a hard ionization method, more accurate and confident qualitative analyses become possible. (U – JT)