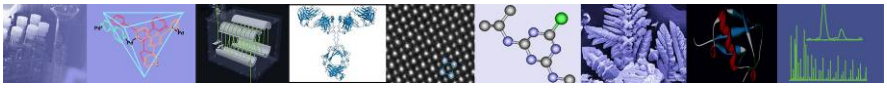

Applications Note

AccuTOF-GCv Series

The Qualitative Analysis of an Antioxidant Additive Using the Full Capabilities of the EI/FI/FD Combination Ion Source

Introduction

JEOL has developed a unique EI/FI/FD combination ion source for the "AccuTOF GCv 4G", a high-resolution GC-time-of-flight (TOF) MS system. This unique ion source provides the capabilities of GC/EI, GC/FI and FD measurements without having to break vacuum in order to switch between each ionization mode. Additionally, this combination is particularly powerful in that it provides library searchable fragmentation information by using EI and high mass accuracy molecular ion information by using FI and FD. In this work, we measured an antioxidant additive by using each ionization mode available on the AccuTOF GCv 4G combination ion source (EI/FI/FD).

Experimental

Sample information and measurement condition are shown in Table 1.

Results

The GC/EI and GC/FI total ion chromatograms (TICs) for the antioxidant sample are shown in Figure 1. Both chromatograms showed the presence of 8 components in the sample. The corresponding EI and FI mass spectra for each component are shown in Figure 2 and Figure 3.

The FI mass spectra for each of the 8 components showed very simple mass spectra that were dominated by their molecular ions. Additionally, the exact masses measured for these compounds showed that there were several isomers present in the antioxidant additive—(A) one at *m/z* 225, (B) three at *m/z* 281, (C) two at *m/z* 337, and (D) two at 393. The accurate mass and calculated elemental composition results are shown in Table 2. The ions generally showed good mass accuracy with less than 1 mDa for both EI and FI mode.

Condition	Measurement		
	GC/EI	GC/FI	FD
Sample	Antioxidant additive		
Concentration	100 ng/uL		10 ug/uL
GC-TOFMS system	AccuTOF GCv 4G (JEOL)		
Ion source	EI/FI/FD combination ion source		
Ionization mode	EI+	FI+	FD+
Ionization condition	70 eV, 300 uA	−10 kV, 45 mA (30 msec refresh between every stored spectrum)	−10 kV, 0 mA → 51.2 mA/min → 45mA
m/z range	*m/z* 35-800		*m/z* 35-1600
GC column	DB-5ms, 30 m x 0.25 mm, 1.0 um		
Inlet mode	Splitless		
Oven temp.	35 C(2min) → 10 C/min → 300 C(22 min)		

Table 1. Measurement condition.

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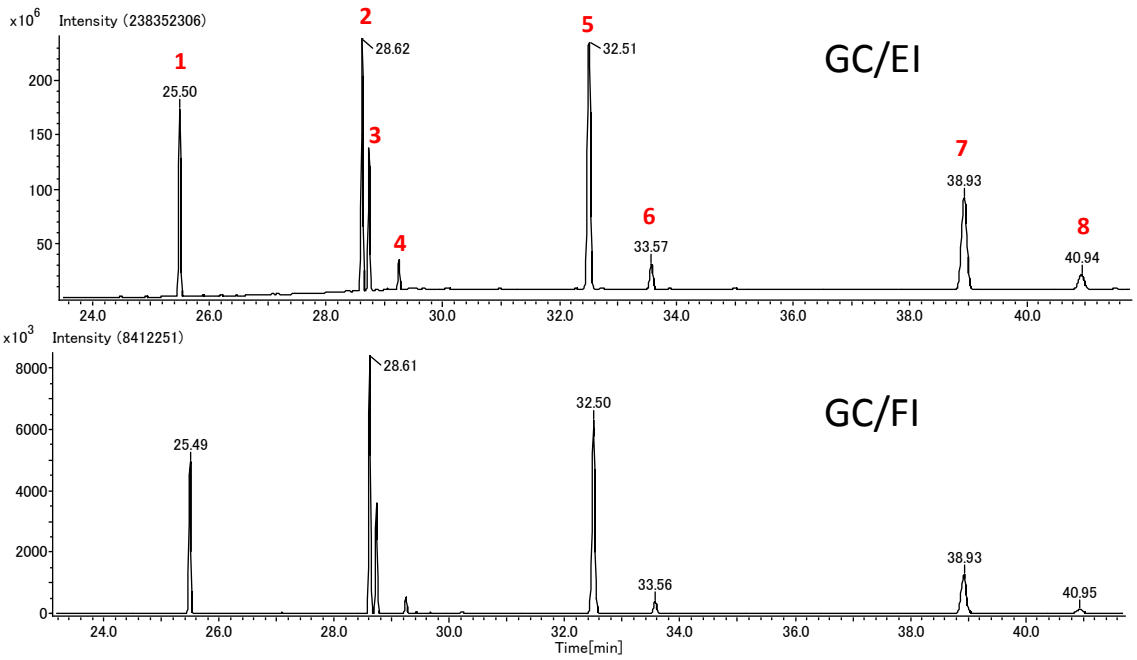


Figure 1. The TICs for GC/EI and GC/FI

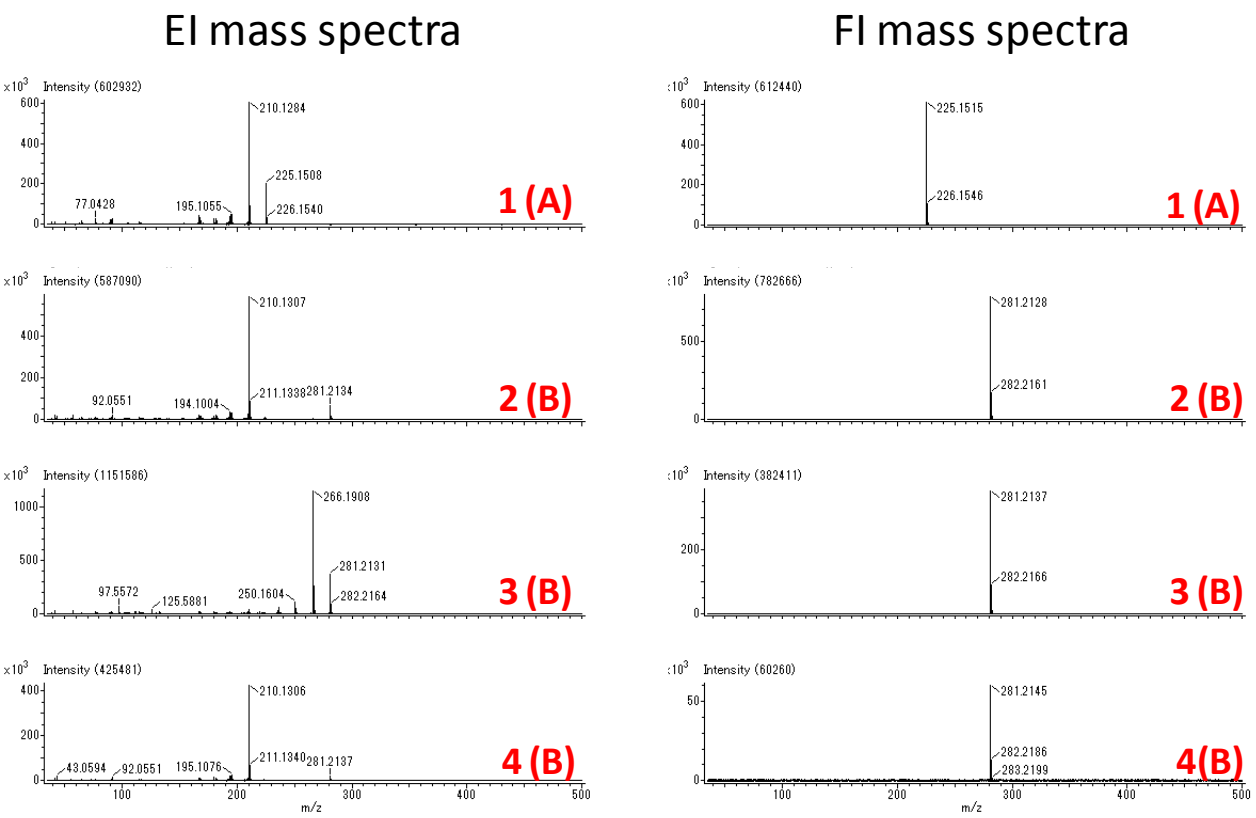


Figure 2. Mass spectra of component1-4, left: EI mass spectra, right: FI mass spectra (A,B: Isomer group)

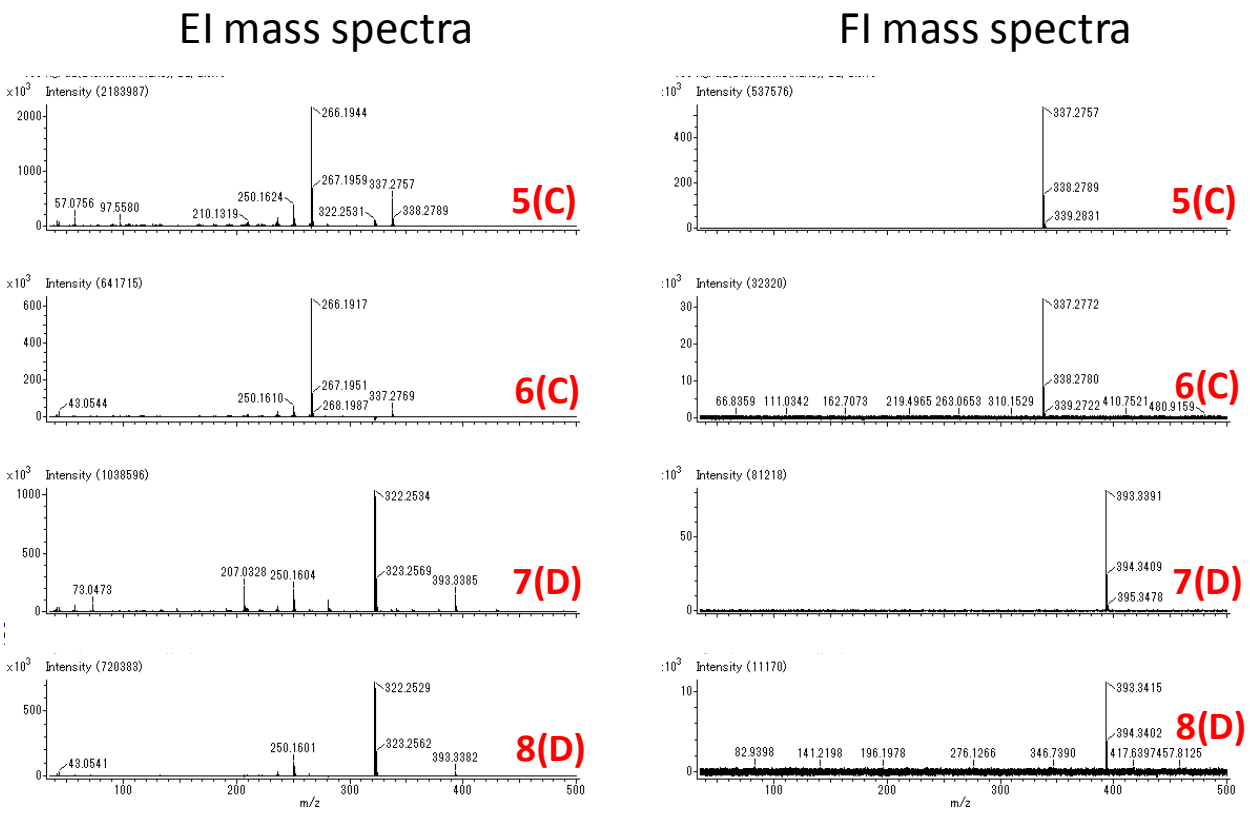


Figure 3. Mass spectra of component5-8, left: EI mass spectra, right: FI mass spectra (C,D: Isomer group)

Component (Isomer group)	Ionization mode	Obs. *m/z*	Theo. *m/z*	Error (mDa)	Fomula
1(A)	EI	210.1284	210.1283	0.1	$C_{15}H_{16}N$
		225.1508	225.1518	−1.0	$C_{16}H_{19}N$
	FI	225.1515	225.1518	−0.3	$C_{16}H_{19}N$
2(B)	EI	266.1908	266.1909	−0.1	$C_{19}H_{24}N$
		281.2131	281.2144	−1.3	$C_{20}H_{27}N$
	FI	281.2137	281.2144	−0.6	$C_{20}H_{27}N$
5(C)	EI	266.1917	266.1909	0.8	$C_{19}H_{24}N$
		337.2769	337.2770	−0.1	$C_{24}H_{35}N$
	FI	337.2772	337.2770	0.3	$C_{24}H_{35}N$
7(D)	EI	322.2534	322.2535	−0.1	$C_{23}H_{32}N$
		393.3385	393.3396	−1.1	$C_{28}H_{43}N$
	FI	393.3396	393.3396	−0.4	$C_{28}H_{43}N$

Table 2. Accurate mass measurement results

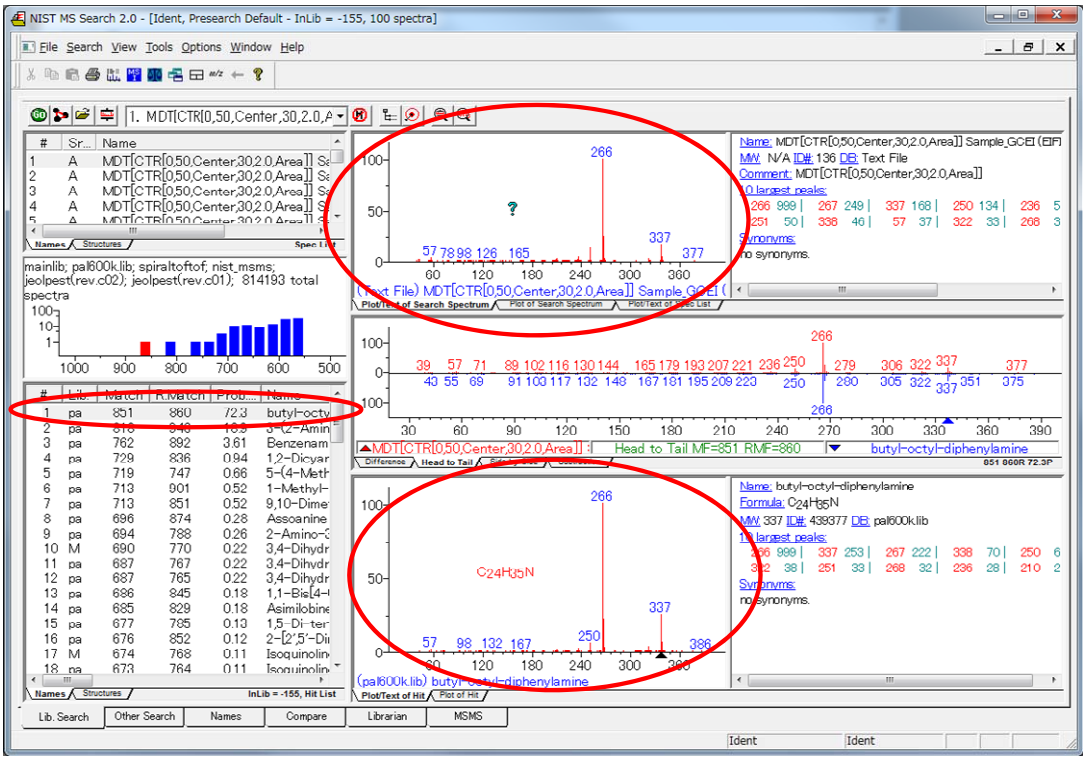


Figure 4. NIST search for component 5.

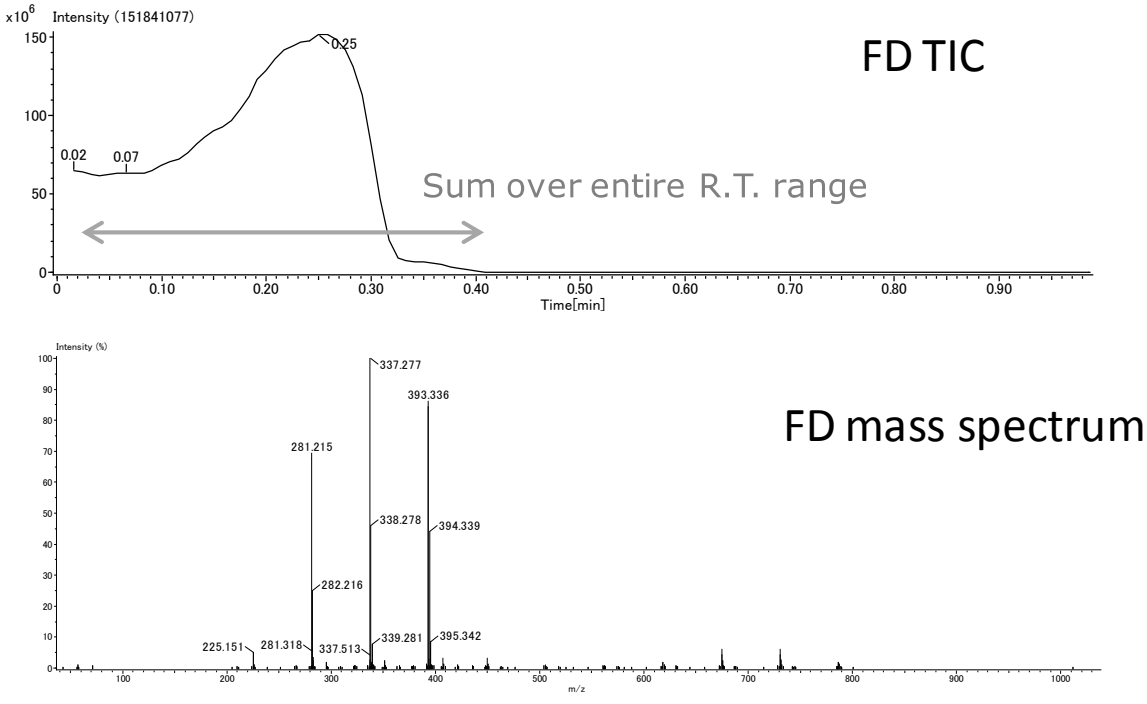
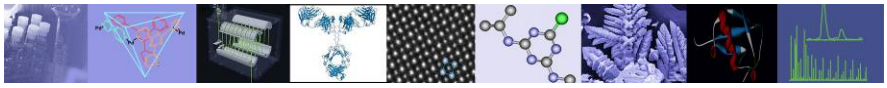


Figure 5. FD measurement result



As an example of how the EI data is library searchable, the EI spectrum for component 5 was exported to the NIST database which in turn showed that the top candidate for this EI fragmentation pattern is butyl-octyl-diphenylamine (Figure 4). To further support this match, the elemental composition of this compound ($C_{24}H_{35}N$) exactly matches the composition identified through the EI and FI accurate mass measurements. Moreover, butyl-octyl-diphenylamine is an antioxidant which further supports this identification for component 5.

Next, the same antioxidant additive mixture was measured using FD mode, in which the sample is loaded directly onto the emitter probe. Figure 5 shows both the TIC and mass spectrum for this analysis. The measurement was completed within 1 minute and confirmed that the same four compositions were observed in this experiment (*m/z* 225.2, 281.2, 337.3 and 393.3) as were observed in the GC/EI and GC/FI analyses. Additionally, the dimers for several of these ions were also observed in the mass spectrum. While FD is not able to determine the presence of multiple isomers (like the chromatography techniques), the analysis speed (less than 1min) is very useful for quickly evaluating the types of constituents that are present in a given sample.

Conclusion

In this work, we showed a brief study for an antioxidant additive using each ionization mode available on the AccuTOF GCv 4G EI/FI/FD combination ion source. Furthermore, each technique was accessed without changing out the ion source or breaking vacuum. The EI/FI/FD combination ion source used in conjunction with the high resolution capabilities of the AccuTOF GCv 4G is a powerful tool for doing chemical qualitative analysis.

JEOL USA ♦ 11 Dearborn Road ♦ Peabody MA 01960 ♦ 978-535-5900 ♦ www.jeolusa.com

