Applications Note

AccuTOF-GCv Series

Analysis of Photo Polymerization Initiator in UV Light Curing Adhesives

Introduction

A UV and visible light curing adhesive is a liquid composed of monomer, oligomer, initiator and additives. These adhesives cure over a short period of time when they are exposed to the appropriate light.

In this report, a photo polymerization initiator found in a UV light curing adhesive was analyzed by electron ionization (EI) and field ionization (FI) using the high resolution and high mass accuracy capabilities of the AccuTOF-GC.

Methods

Sample: UV light curing adhesive

GC condition

GC:	Agilent 6890N
Column:	ZB-5ms、30m×0.25mm I.D., 0.25μm
Oven Temp:	40℃（1min）→10℃/min→280℃(5min)
Injection Temp.:	280℃
Injection mode:	Split (50:1) [for EI+], Splitless [for FI+]
Injection volume:	1.0μL
Carrier gas:	He（1.0mL/min: constant flow mode）

MS conditions

MS:	JMS-T100GC “AccuTOF GC”	
Ionization mode :	EI+ and FI+	
For EI(+):	Ionization voltage:	70V
	Ionization current:	300μA
	Chamber Temp.:	280℃
For FI(+):	Cathode voltage :	-10kV
	Emitter current :	0 mA

Acquired m/z range: *m/z* 35－500

Recording interval: 0.6sec

Results and Discussion

The TIC is shown in Fig.1. Many components are observed in the TIC in both EI+ and FI+. The unreacted photo polymerization initiator (R.T. 27.9 min.) was determined by using accurate mass measurement conditions in EI and FI.

The EI+ and FI+ mass spectra are shown in Fig.2 and Fig.3, respectively. The ions marked in red were selected for elemental composition determination. The results for these elemental composition calculations are shown in Table 1 and Table 2.

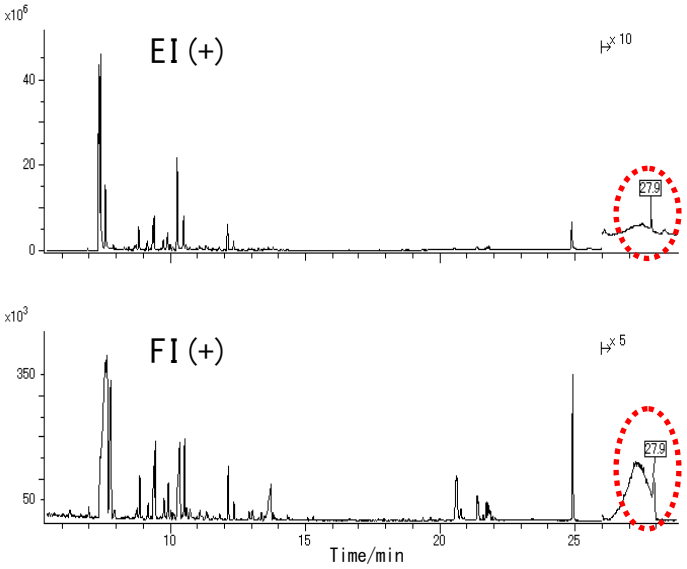


Fig.1 TIC <Upper: EI(+), Lower: FI+>

MS Tips #108

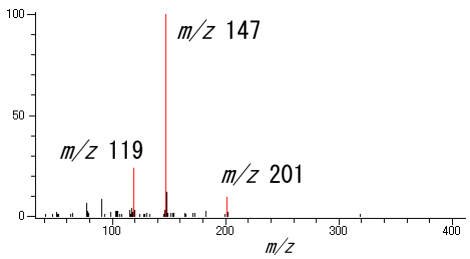


Fig.2 EI+ mass spectrum

Table 1. Elemental composition determination by EI+

Observed m/z	Calcurated m/z	Error (mDa)	Estimated mormula	U.S.
119.08526	119.08608	−0.82	12C9 1H11	4.5
147.08013	147.08099	−0.86	12C10 1H11 16O	5.5
201.04750	201.04693	0.58	12C12 1H10 16O 31P	9.5
	201.04920	−1.70	12C2 1H21 16O2 32P4	−1.5

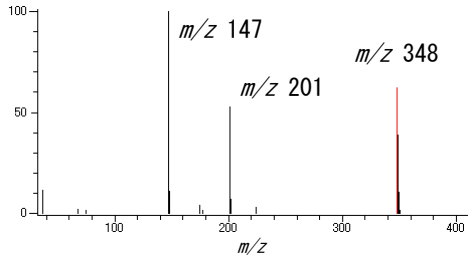


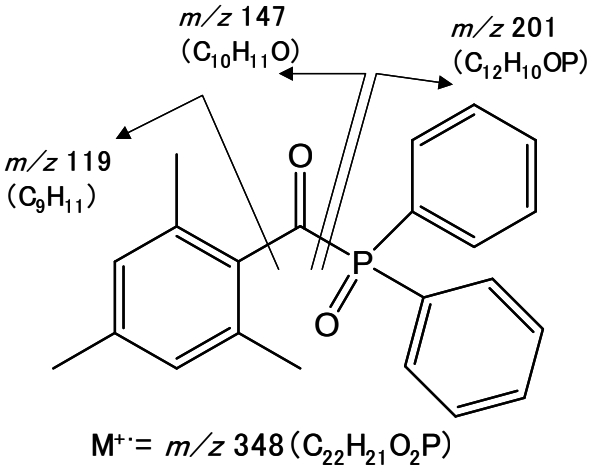
Fig.3 FI+ mass spectrum

Table 2. Elemental composition determination by FI+

Observed m/z	Calcurated m/z	Error (mDa)	Estimated mormula	U.S.
348.12829	348.12792	0.37	12C22 1H21 16O2 31P	14.0
	348.13019	−1.90	12C12 1H32 16O3 31P4	3.0
	348.12555	2.74	12C15 1H26 16O5 31P2	6.0
	348.13256	−4.27	12C19 1H27 32P3	11.0

The *m/z* 119, 147 and 201 ions were the dominant species observed in the EI+ mass spectrum. The results of the elemental composition determination for these ions show that only one ion formula can be estimated for *m/z* 119 and 147. However, for *m/z* 201 there were two different ion formula calculated for this species. The ion formula for *m/z* 201 was assigned as $C_{12}H_{10}OP$ based on the mass difference, the elements present and its number of elements. Also, the ion at *m/z* 201 was a fragment ion because the unsaturation number is a half-integer.

The *m/z* 147, 201 and 348 ions were the dominant species observed in the FI+ mass spectrum. Since the *m/z* 147 and 201 were already assigned using the EI+ data, the *m/z* 348 ion was selected for elemental composition calculations. There were 4 candidates within 5 mDa of the observed exact mass. Despite this, $C_{22}H_{21}O_2P$ was determined to be the best match based on the mass difference and the number of phosphorous and oxygen atoms. The unsaturation number of this formula is an integer so this composition was selected as the best formula for the molecular ion. Since the unsaturation number is 14, it is likely that the structure will contain more than 2 benzene rings present in the structure. The final estimated structure is shown in Fig.4.



Conclusions

The JMS-T100GC "AccuTOF GC" obtains accurate *m/z* values for both EI+ and FI+ analysis. This capability is very helpful for structure analysis of unknown compounds like UV activated initiators.