JEOL MS Data Sheet

MS Tips

Mass Spectrometry Application Group
Mass Spectrometry Business Unit
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No.108

Analysis of photo polymerization initiator in UV light curing adhesives by GC/TOFMS

A UV and visible light curing adhesive is a liquid composed of monomer, oligomer, initiator and additives. It cures layers in a short period of time under the influence of light.
In this report, a photo polymerization initiator in a UV light curing adhesive was analyzed by electron ionization (EI) and field ionization (FI).

<Sample and measurement conditions>

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

<Result>

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.

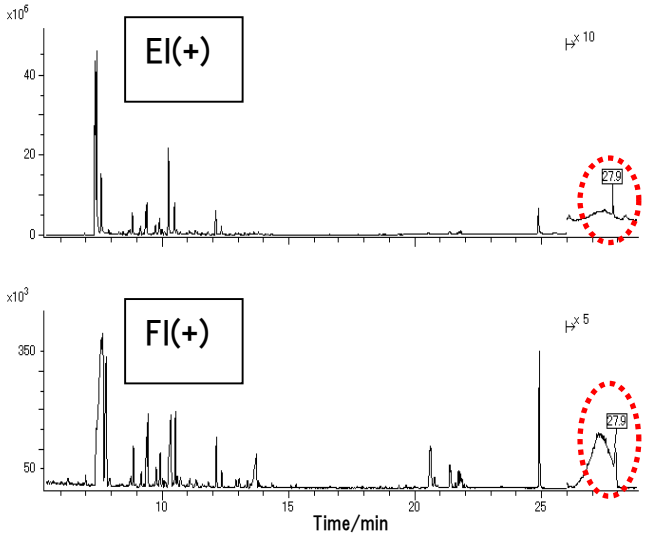

Fig.1 TIC <Upper: EI(+), Lower: FI+>

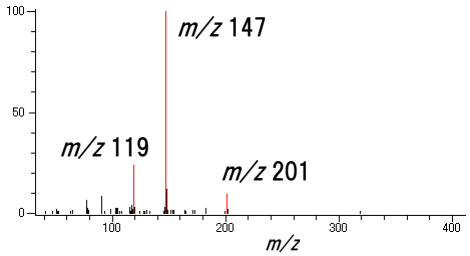

Fig.2 EI+ mass spectrum

Table1 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

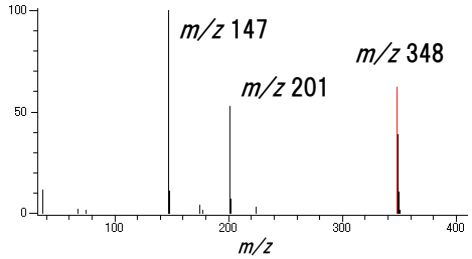

Fig.3 FI+ mass spectrum

Table2 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

An EI mass spectrum and a FI+ mass spectrum are shown in Fig.2 and Fig.3. The indicated ions (red) are selected for elemental composition determination. The results for each spectrum are shown in Table 1 and Table 2.

Ions with m/z 119, 147 and 201 are mainly observed by EI+. The result of the elemental composition determination for these ions, shows that only one ion formula can be estimated for *m/z* 119 and 147. On the other hand, 2 different ion formula are estimated for *m/z* 201. Finally, ion formula for m/z 201 could be estimated as $C_{12}H_{10}OP$ according to 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 half-integer.

Ions at m/z 147, 201 and 348 are mainly observed in FI+. Since ions at m/z 147 and 201 are the same in EI+, an elemental composition for ion at m/z 348 was estimated. There are 4 candidates within 5 mDa of mass difference but $C_{22}H_{21}O_2P$ was estimated, based on the mass difference and the number of phosphor and oxygen atoms. The unsaturation number of this formula is integer so, this ion is determined as molecular ion. Since the unsaturation number is 14, it's possible to consider more than 2 benzene rings are included in the structure. The final estimated structure is shown in Fig.4.

JMS-T100GC "AccuTOF GC" obtains accurate m/z values very easily in EI+ and also in FI+. This capability is very helpful for structure analysis for unknown compounds.

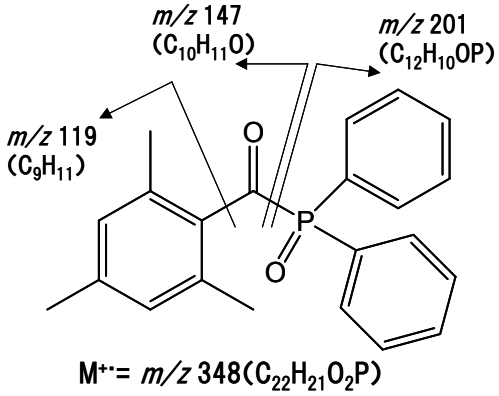

Fig.4 Estimated structure