JEOL MS Data Sheet

MS Tips

Mass Spectrometry Application Group
Mass Spectrometry Business Unit
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No.101

Analysis of acrylic resin by pyrolysis GC-TOFMS

Pyrolysis gas chromatography / mass spectrometry (PyGC/MS) is widely used for the analysis of synthetic polymers. As the sample is decomposed instantaneously, the thermal decomposition process is reproducible.

We have performed pyrolysis GC/MS analysis with high sensitivity, high resolution, and high mass accuracy by using Frontier Lab's PY2020D pyrolyzer and JMS-T100GC gas chromatograph – time-of-flight mass spectrometer (GC-TOFMS). The sample was a commercially available acrylic resin.

Methods

Mass spectrometer	JMS-T100GC (JEOL)
Pyrolyzer	PY2020D (Frontier Lab.)
Gas chromatograph	6890N (Agilent)
Sample	acrylic resin (0.5 mg)

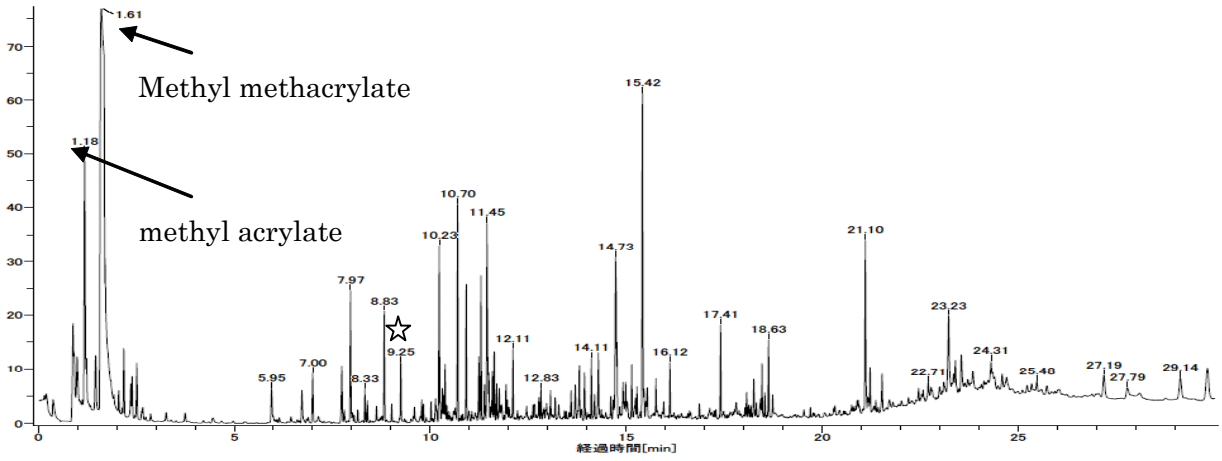
Results and discussion

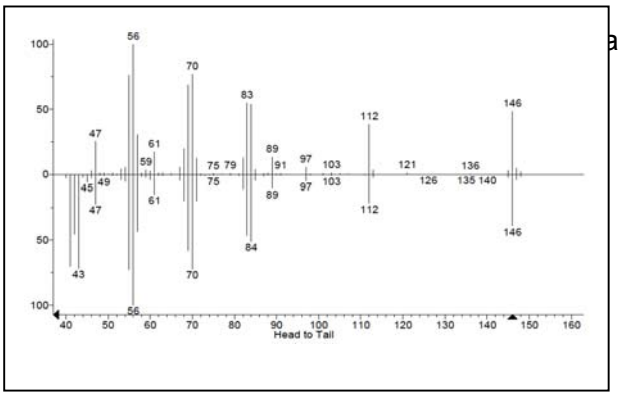
The reconstructed total ion current chromatogram (RTICC) is shown in Fig. 1. Most major peaks on the RTICC were identified by mass spectral library search. Methyl methacrylate, methyl acrylate, and many of their analogs were identified.

The peak at 9.25 min (☆) was identified as 1-ocanethiol by library search. While the result was supported by both Match Factor and Probability, the existence of sulfur was confirmed from measured accurate mass.

Table 1 Analysis Parameters

Pyrolysis GC	
Pyrolysis temp.	550 °C
Interface temp.	300 °C
Carrier gas flow	1.0 mL/min
Split ratio	50:1
GC column	DB-5MS, 30 m x 0.25 mm I.D. film thickness: 0.25μm
GC oven temp.	50 °C – 15 °C/min – 325 °C
MS	
Ion source temp.	250 °C
Interface temp.	320 °C
Detector voltage	2200 V
Acquisition range	*m/z* 45 – 800
Acquisition rate	0.5 s/spectrum





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No.	DB	MF	RMF	Prob.	compound
1	R	923	925R	80.7P	1-Octanethiol
2	M	780	917R	2.54P	Cyclooctane
3	M	780	893R	2.54P	Octane, 1-methoxy-
4	M	764	877R	1.46P	Formic acid, octyl ester
5	M	763	916R	1.41P	cis-1-Butyl-2-methylcyclopropane
6	M	760	895R	1.24P	Cyclopropane, pentyl-
7	R	758	898R	1.14P	Octane, 4-chloro-
8	M	756	910R	1.06P	trans-1-Butyl-2-methylcyclopropane
9	R	754	873R	0.97P	1-Octanol
10	M	750	871R	0.82P	Octane, 3-chloro-

Fig. 2 Library search results (right,) acquired (top left) and library (bottom left) spectra

The elemental compositions of the major peaks in the mass spectrum (marked red in Fig. 3) are determined with errors less than 1 mmu. For the molecular ion at m/z 146, there were two possibilities, $C_8H_{18}S$ and $C_{11}H_{14}$ within 5 mmu tolerance range. Considering the mass accuracy achieved on other peaks and library search result, $C_8H_{18}S$ seems reasonable. The isotopic cluster pattern of the molecular ion in the observed mass spectrum matched well with that calculated from the elemental composition $C_8H_{18}S$. We concluded that the compound detected at 9.25 min is indeed 1-octanethiol based on library search, accurate mass measurement, and isotope cluster pattern.

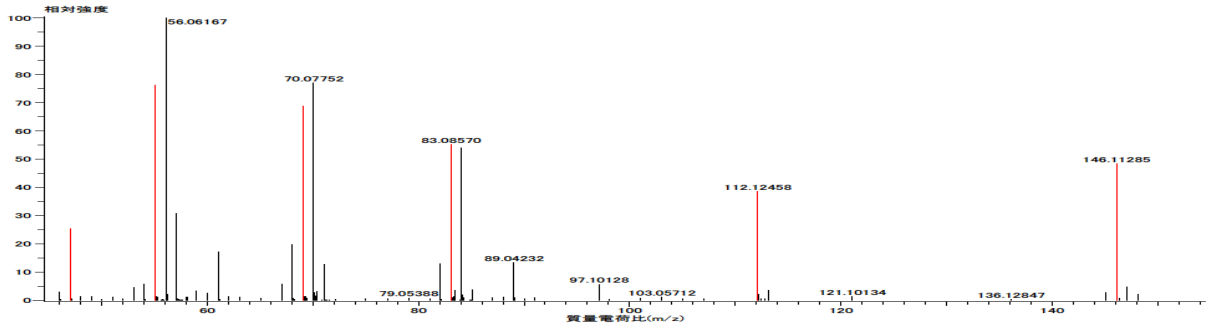

Fig. 3 Observed mass spectrum at 9.25 min and peaks whose elemental compositions were determined

Conclusion

Table 2 Determined elemental compositions

Charge: 1 **Tolerance: 5.00 (mmu)**

Elements: ^{12}C: 0..100, ^{1}H: 0..200, ^{16}O: 0..10, ^{32}S: 0..1

Mass	Intensity	Rel. Int.	Calc. Mass	Error mmu	Formula	Unsat.
46.99473	77718.35	25.18	46.99555	−0.81	$^{12}C_1{}^{1}H_3{}^{32}S_1$	1.5
55.05387	236425.22	76.60	55.05478	−0.90	$^{12}C_4{}^{1}H_7$	1.5
69.06963	214122.77	69.38	69.07043	−0.80	$^{12}C_5{}^{1}H_9$	1.5
83.08569	169120.12	54.80	83.08608	−0.39	$^{12}C_6{}^{1}H_{11}$	1.5
112.12456	117777.66	38.16	112.12520	−0.64	$^{12}C_8{}^{1}H_{16}$	1.0
146.11280	148251.25	48.03	146.11292	−0.12	$^{12}C_8{}^{1}H_{18}{}^{32}S_1$	1.0
			146.10955	3.25	$^{12}C_{11}{}^{1}H_{14}$	5.0

In PyGC/MS analyses, many of the detected compounds are often left unidentified since there are not many mass spectra of pyrolysis products in mass spectral libraries. The JMS-T100GC GC-TOFMS, with its easy accurate mass measurement capability, not only ensures the library search results but also is effective in identifying compounds whose spectra are not in the libraries. Its ability to acquire accurate isotope cluster pattern further aids in identifying unknown molecules.