Applications Note

AccuTOF-GCv Series

Analysis of Acrylic Resin by Pyrolysis GC-TOFMS

Introduction
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 provides reproducible results that are related to the structure of the analytes.

In this work, we performed a pyrolysis GC/MS analysis with high sensitivity, high resolution, and high mass accuracy by using a Frontier Lab's PY2020D pyrolyzer interfaced to a JMS-T100GC gas chromatograph – time-of-flight mass spectrometer (GC-TOFMS). The sample tested 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)

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

Table 1. Analysis Parameters

Results and Discussion
The reconstructed total ion current chromatogram (RTICC) is shown in Fig. 1. Most of the major peaks on the RTICC were identified through a mass spectral library search (Fig 2). 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 by the measured accurate mass.

The elemental compositions of the major peaks in the mass spectrum (marked red in Fig. 3) are shown in Table 2 and had composition 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 for the other peaks and the 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.

Conclusions
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 accurate mass measurement capability, not only ensures the library search results but also is effective for identifying compounds with spectra that are not in the libraries. Furthermore, this system's ability to acquire accurate isotope cluster patterns further aids in identifying unknown molecules.

Frontier Laboratories' pyrolyzer system is provided and supported through Frontier Laboratories' sales and support network and may not be available in your territory. Contact your local JEOL representative for detail.

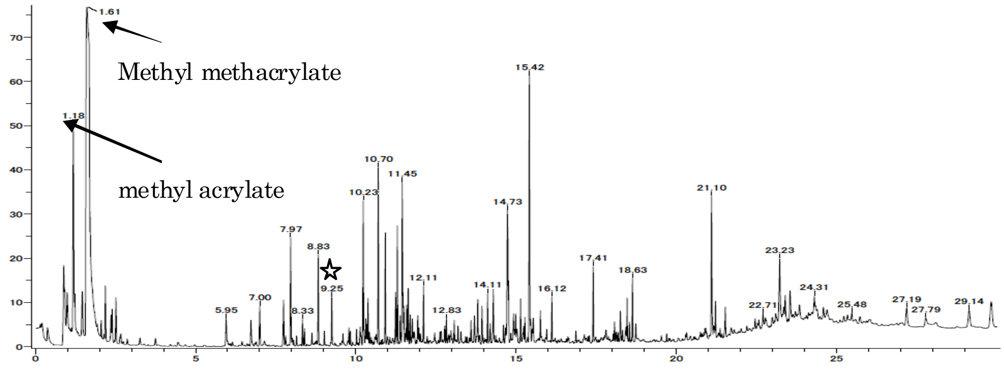
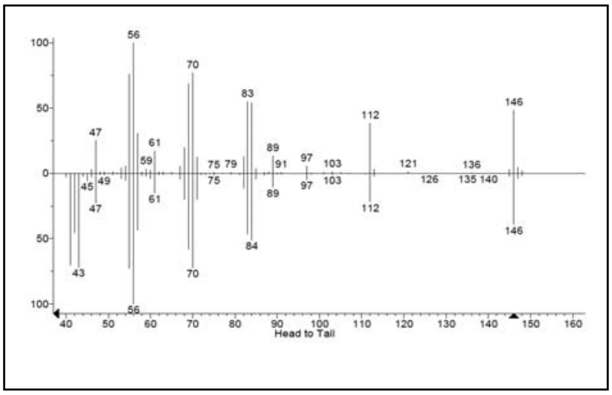


Fig. 1 Reconstructed TIC chromatogram of PyGC/TOFMS analysis of acrylic resin



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 Acquired spectra (top left), library spectra (bottom left), and library search results (right)

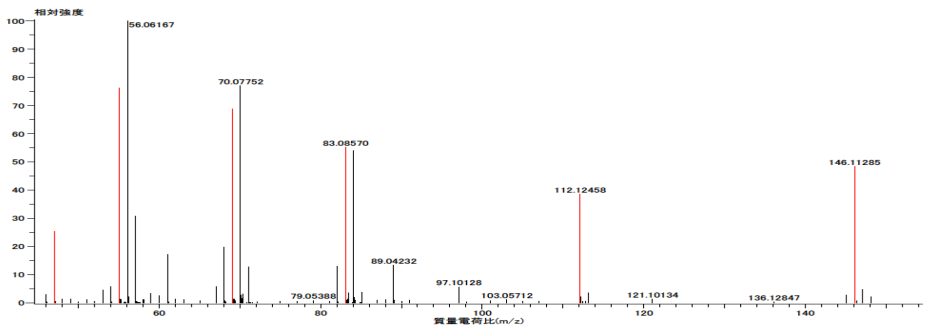


Fig. 3 Observed mass spectrum at 9.25 min; peaks in red were used for elemental composition calculations (Table 2)

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

Table 2. Determined elemental compositions

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