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Application Data Sheet

No.129

GC-MS
Gas Chromatograph Mass Spectrometer

Analysis of Resin Using the OPTIC-4 Multimode Inlet in Pyrolysis Mode

The OPTIC-4 can be used for pyrolysis analysis of polymers. With the pyrolysis method, a small amount of a resin (a few dozen μg or less) is heated rapidly in a helium gas environment. The pyrolysates are then analyzed using a GC/MS. The structure of the resin can in turn be analyzed from the pyrolysates. The OPTIC-4 is capable of rapid heating up to 600 °C at 60 °C/sec, so data equivalent to that from a dedicated pyrolysis system can be obtained. This article describes the analysis of a polycarbonate resin using the OPTIC-4 in pyrolysis mode.

Experiment

An approximately 0.1 mg of polycarbonate resin sample clipped with a cutter knife was placed in a micro vial. The micro vial was placed in a liner, which was then passed through the O-ring for sealing the inlet. After both ends were capped, the liner was placed into the rack for the AOC-6000.
Table 1 shows the analytical conditions.

Table 1: Analytical Conditions

Instrument	
Injection Port:	OPTIC-4
Liner:	L100011, DMI liner with taper
GC-MS:	GCMS-QP2020
Autosampler:	AOC-6000 (LINEX-2 and CDC Station included)
Column:	SH-Rxi-5SilMS (0.25 mm × 30 m, df = 0.25 μm)

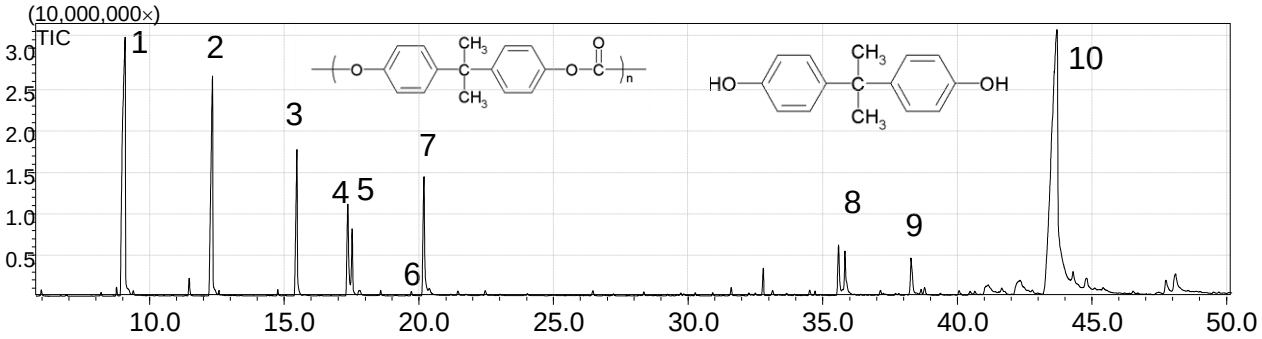
Injector	
Vent Time:	1 min
Method Type:	Split
Equilibration Time:	5 sec
End Time:	60 min
Injector Temperature:	
40 °C (10 sec) → (60 °C /sec) → 600 °C (3 min) → 320 °C (hold)	
Carrier Gas:	Helium
Carrier Control Mode:	Flow control
Transfer Column Flow:	0.7 mL/min
Start Column Flow:	1.5 mL/min
End Column Flow:	1.5 mL/min
Split Flow:	150 mL/min
Septum Purge Flow:	10 mL/min

MS	
Interface Temperature:	250 °C
Ion Source Temperature:	200 °C
Data Acquisition Time:	5 to 50.0 min
Measurement Mode:	Scan
Event Time:	0.3 sec
Mass Range:	*m/z* 29 to 600
Detector Voltage:	Relative to the Tuning Result 0 kV

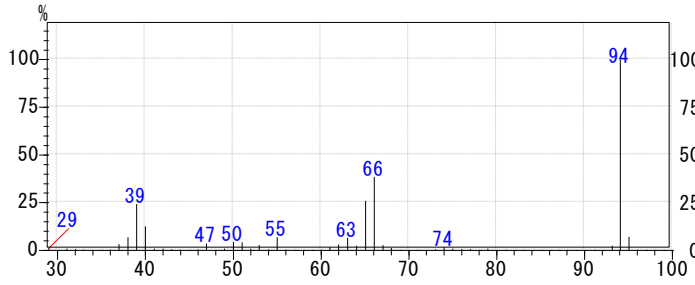
GC
Column Oven Temperature:
40 °C (2 min) → (4 °C/min) → 230 °C → (10 °C/min) → 320 °C (1 min)

Results

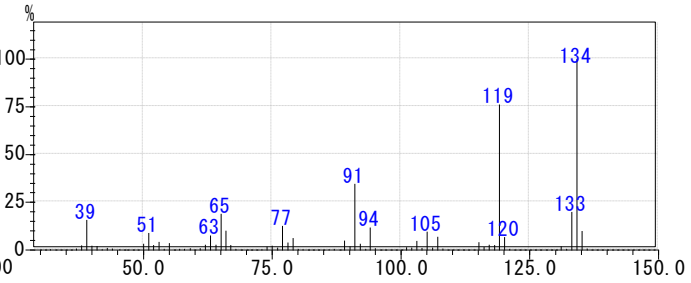
Fig. 1 shows the total ion current chromatogram (TICC) obtained, and the mass spectra for the major pyrolysates. In the obtained TICC, bisphenol A is detected as peak 10; a number of other phenol compounds are also detected. In other words, a typical pyrogram for a polycarbonate resin, which has already been reported*1, was obtained.



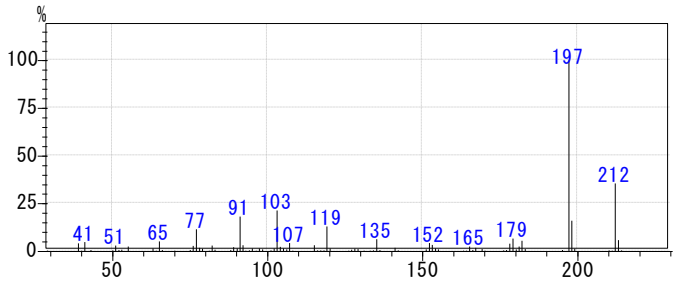
1=phenol, 2=*p*-cresol, 3=*p*-ethylphenol, 4=*p*-vinylphenol, 5=*p*-isopropylphenol, 6=*p*-*tert*-butylphenol, 7=*p*-isopropenylphenol, 8=*p*-hydroxy-2,2-diphenylpropane, 9=*p*-hydroxy-3-methyl-2,2-diphenylpropane, 10=bisphenol A



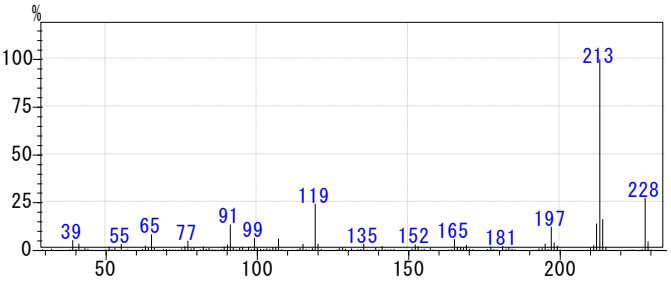
Peak 1: phenol



Peak 7: *p*-isopropenylphenol



Peak 8: *p*-hydroxy-2,2-diphenylpropane



Peak 10: bisphenol A

Fig. 1: Total Ion Current Chromatogram of Polycarbonate, and Mass Spectra for its Pyrolysates

Conclusions

In addition to the pyrolysis of resins, the OPTIC-4 is equipped with sample injection modes that are indispensable for the evaluation of high polymer materials, including difficult matrix introduction (DMI) and thermal desorption. As a result, it is an effective system for the multifaceted evaluation of materials. Furthermore, consecutive analyses can be performed automatically by combining it with the AOC-6000.

*1: S. Tsuge, H. Ohtani, C. Watanabe: Pyrolysis-GC/MS Data Book of Synthetic Polymers –Pyrograms, Thermorgams and MS of Pyrolyzers-, 1st Edition, Elsevier, 420 (2011)

First Edition: March, 2017

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