

# Application Data Sheet

## No. 128

### GC-MS

Gas Chromatograph Mass Spectrometer

## Analysis of Resin Using the OPTIC-4 Multimode Inlet in Thermal Assisted Hydrolysis and Methylation Mode

The OPTIC-4 multimode inlet can be used for the thermal assisted hydrolysis and methylation-GC/MS (THM-GC/MS) method. In the THM-GC/MS method, the sample is subjected to alkaline hydrolysis while being heated. The resulting products are subjected to methylation derivatization, and the derivatized compounds are then measured with a GC/MS. THM-GC/MS is an effective method for measuring resin samples that produce polar compounds due to pyrolysis. The OPTIC-4 allows derivatization reactions within inert glass micro vials.

### Experiment

An approximately 0.1 mg of polycarbonate resin sample clipped with a cutter knife was placed in a micro vial. Then, 4  $\mu$ l of tetramethylammonium hydroxide (25 % in methanol) was added to the sample in the 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. For thermal assisted hydrolysis, measurements are generally performed with the temperature set to between 300 °C and 400 °C<sup>\*1, \*2</sup>. This is lower than the temperature used for typical pyrolysis-GC measurements without using a reaction reagent (500 °C to 600 °C). Accordingly, the inlet temperature was raised to 420 °C prior to the analysis.

Table 1: Analytical Conditions

<b>Instrument</b>		
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 $\times$ 30 m, df = 0.25 $\mu$ m)	
<b>Injector</b>		<b>MS</b>
Vent Time:	1 min	Interface Temperature: 250 °C
Method Type:	Split	Ion Source Temperature: 200 °C
Equilibration Time:	5 sec	Data Acquisition Time: 5 to 50.0 min
End Time:	60 min	Measurement Mode: Scan
Injector Temperature:		Event Time: 0.3 sec
40 °C (10 sec) $\rightarrow$ (60 °C/sec) $\rightarrow$ 420 °C (3 min) $\rightarrow$ 320 °C (hold)		Mass Range: <i>m/z</i> 29 to 600
Carrier Gas:	Helium	Detector Voltage: Relative to the Tuning Result
Carrier Control Mode:	Flow control	0 kV
Start Column Flow:	1.5 mL/min	
End Column Flow:	1.5 mL/min	
Initial Split Flow:	150 mL/min	
Split Flow:	450 mL/min	
Septum Purge Flow:	10 mL/min	
<b>GC</b>		
Column Oven Temperature:		
40 °C (2 min) $\rightarrow$ (4 °C/min) $\rightarrow$ 230 °C $\rightarrow$ (10 °C/min) $\rightarrow$ 320 °C (1 min)		

## Results

The figures show the total ion current chromatogram (TIC) obtained, and the mass spectra for the compounds detected. When the ester bonds were hydrolyzed, bisphenol A was produced. As shown in Fig. 1, a derivative of bisphenol A with one hydroxyl group methylated and a derivative with two hydroxyl groups methylated were detected.

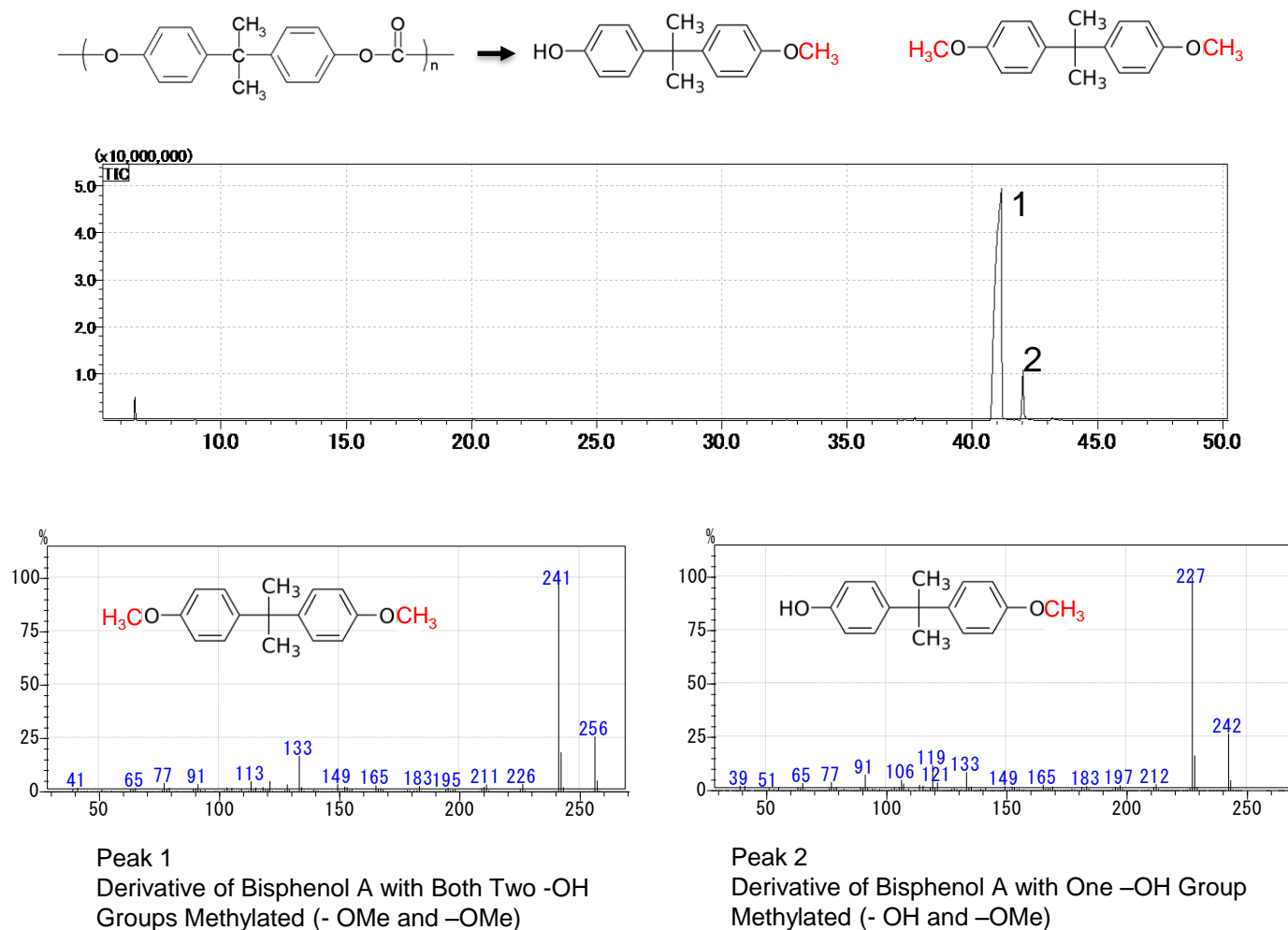


Fig. 1: Total Ion Current Chromatogram of Polycarbonate and Mass Spectra for Peaks Detected

## Conclusions

The OPTIC-4 is equipped with sample injection modes that are indispensable for the evaluation of polymer materials. In addition to THM-GC/MS, these include pyrolysis, difficult matrix introduction (DMI), and thermal desorption. As a result, it is effective for the multifaceted evaluation of materials. Furthermore, using it with the AOC-6000 enables consecutive analyses to be performed automatically.

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

\*2: H. Ohtani and T. Takarazaki edited: Synthetic Polymer Chromatography, Ohmsha, Ltd., 401, 2013

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