

# 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  $\mu\text{g}$  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

<b>Instrument</b>		<b>MS</b>	
Injection Port:	OPTIC-4	Interface Temperature:	250 °C
Liner:	L100011, DMI liner with taper	Ion Source Temperature:	200 °C
GC-MS:	GCMS-QP2020	Data Acquisition Time:	5 to 50.0 min
Autosampler:	AOC-6000 (LINEX-2 and CDC Station included)	Measurement Mode:	Scan
Column:	SH-Rxi-5SilMS (0.25 mm $\times$ 30 m, df = 0.25 $\mu\text{m}$ )	Event Time:	0.3 sec
<b>Injector</b>		Mass Range:	$m/z$ 29 to 600
Vent Time:	1 min	Detector Voltage:	Relative to the Tuning Result 0 kV
Method Type:	Split		
Equilibration Time:	5 sec		
End Time:	60 min		
Injector Temperature:			
40 °C (10 sec) $\rightarrow$ (60 °C/sec) $\rightarrow$ 600 °C (3 min) $\rightarrow$ 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		
<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

Fig. 1 shows the total ion current chromatogram (TIC) obtained, and the mass spectra for the major pyrolysates. In the obtained TIC, 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.

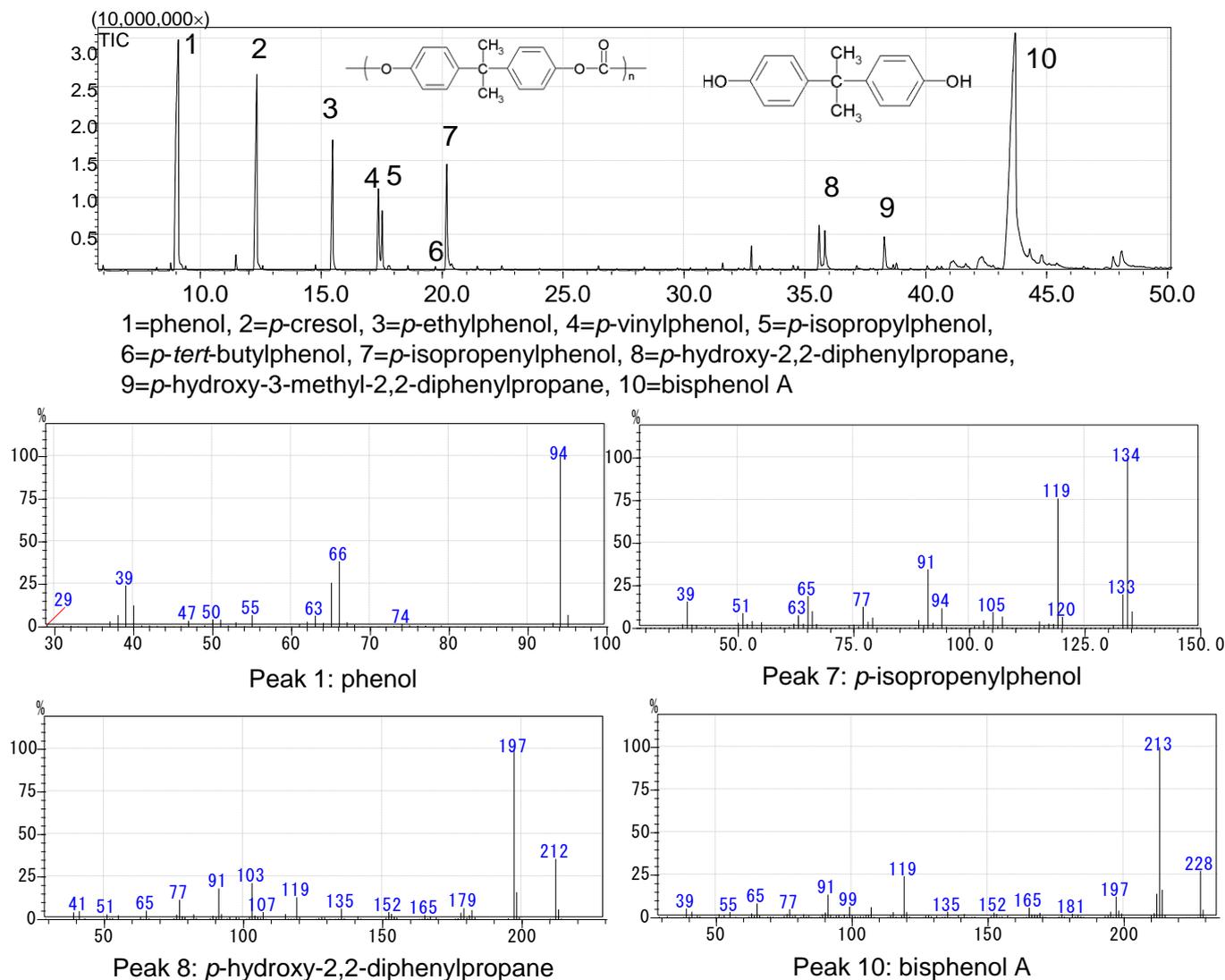


Fig. 1: Total Ion Current Chromatogram of Polycarbonate, and Mass Spectra for its Pyrolysates

## 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, Thermograms and MS of Pyrolyzers-, 1<sup>st</sup> Edition, Elsevier, 420 (2011)

First Edition: March, 2017



Shimadzu Corporation

www.shimadzu.com/an/

For Research Use Only. Not for use in diagnostic procedures.

This publication may contain references to products that are not available in your country. Please contact us to check the availability of these products in your country.

The content of this publication shall not be reproduced, altered or sold for any commercial purpose without the written approval of Shimadzu. Company names, products/service names and logos used in this publication are trademarks and trade names of Shimadzu Corporation, its subsidiaries or its affiliates, whether or not they are used with trademark symbol "TM" or "®".

Third-party trademarks and trade names may be used in this publication to refer to either the entities or their products/services, whether or not they are used with trademark symbol "TM" or "®".

Shimadzu disclaims any proprietary interest in trademarks and trade names other than its own.

The information contained herein is provided to you "as is" without warranty of any kind including without limitation warranties as to its accuracy or completeness. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.

© Shimadzu Corporation, 2017