

# 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 µl 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

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		MS	
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) → (60 °C/sec) → 420 °C (3 min) → 320 °C (hold)		Mass Range:	m/z 29 to 600
Carrier Gas:	Helium	Detector Voltage:	Relative to the Tuning Result 0 kV
Carrier Control Mode:	Flow control		
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		
GC			
Column Oven Temperature:			
40 °C (2 min) → (4 °C/min) → 230 °C → (10 °C/min) → 320 °C (1 min)			

## Results

The figures show the total ion current chromatogram (TICC) 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.

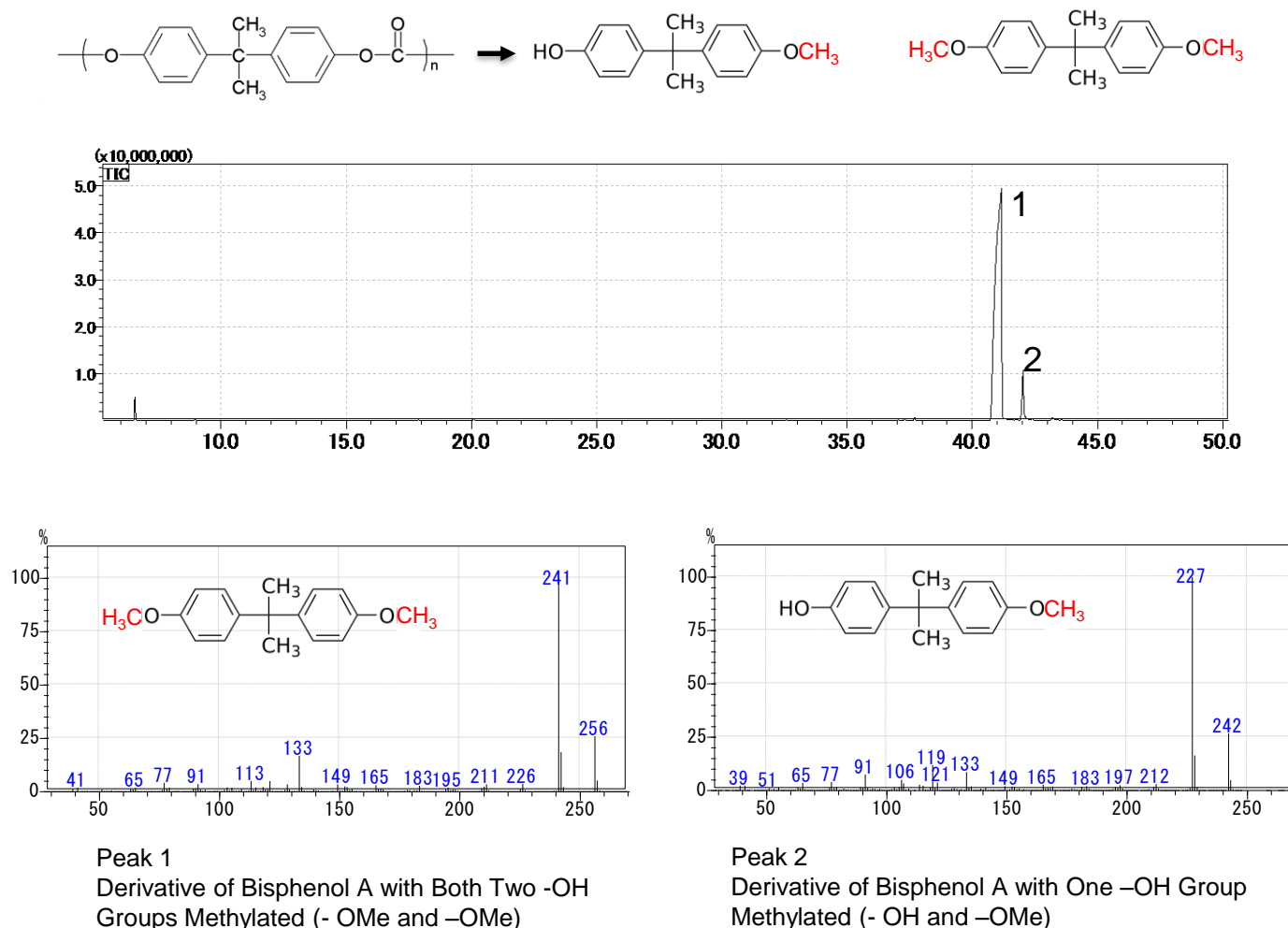


Fig. 1: Total Ion Current Chromatogram of Polycarbonate and Mass Spectra for Peaks Detected

## 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

First Edition: March, 2017



Shimadzu Corporation

[www.shimadzu.com/an/](http://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

# Related Products

Some products may be updated to newer models.



> OPTIC-4  
Multimode Inlet for Gas Chromatograph-Mass Spectro...



> GCMS-QP2020 NX  
Gas Chromatograph Mass Spectrometer



> AOC-6000 Plus  
AOC-6000 Plus Multifunctional Autosampler

# Related Solutions

Hydrocarbon  
> Processing Industry  
(Petrochemical, Ch

> Price Inquiry

> Product Inquiry

> Technical Service /  
Support Inquiry

> Other Inquiry