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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[*1, *2]. 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) |

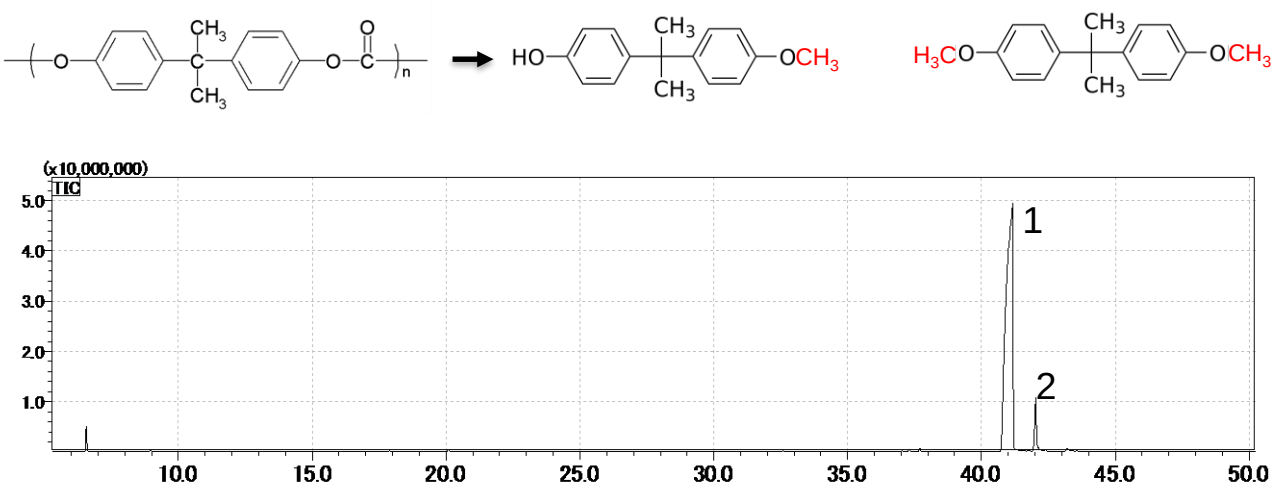
| | |
|---|---|
| Injector | |
| Vent Time: | 1 min |
| Method Type: | Split |
| Equilibration Time: | 5 sec |
| End Time: | 60 min |
| Injector Temperature: | |
| 40 °C (10 sec) → (60 °C/sec) → 420 °C (3 min) → 320 °C (hold) | |
| Carrier Gas: | Helium |
| Carrier Control Mode: | Flow control |
| Start 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 |

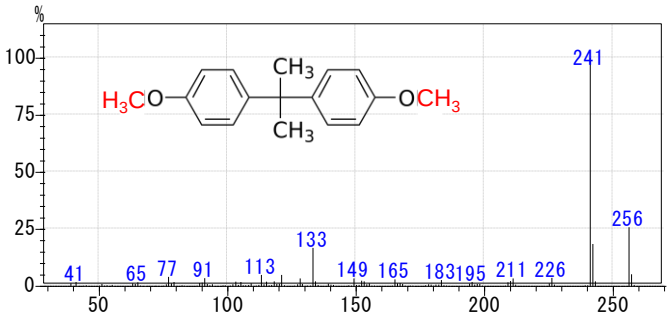
| | |
|---|---|
| MS | |
| Interface Temperature: | 250 °C |
| Ion Source Temperature: | 200 °C |
| Data Acquisition Time: | 5 to 50.0 min |
| Measurement Mode: | Scan |
| Event Time: | 0.3 sec |
| Mass Range: | *m/z* 29 to 600 |
| Detector Voltage: | Relative to the Tuning Result<br>0 kV |

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

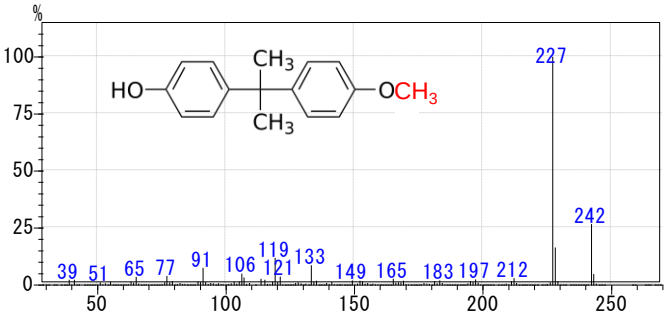
## Results

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.




Peak 1
Derivative of Bisphenol A with Both Two -OH Groups Methylated (- OMe and –OMe)




Peak 2
Derivative of Bisphenol A with One –OH Group Methylated (- OH and –OMe)

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, Thermorgams and MS of Pyrolyzers-, 1st Edition, Elsevier, 420 (2011)
*2: H. Ohtani and T. Takarazaki edited: Synthetic Polymer Chromatography, Ohmsha, Ltd., 401, 2013

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