

Analysis of Polyethylene Terephthalate by TG-FTIR

TG-FTIR is a relatively simple FTIR-coupled analytical technique in which the FTIR is coupled with a thermogravimetric analyzer (TG). In TG-FTIR, while mass changes such as sample decomposition and vaporization that accompany changes in temperature are measured quantitatively by the TG, qualitative

analysis of the evolved gases can be conducted simultaneously by FTIR measurement of the obtained spectra. This Application News introduces an example of analysis of a polyethylene terephthalate sample in nitrogen and air atmospheres.

■ TG-FTIR System Configuration

The TG-FTIR system is shown in Fig.1. The gas exhaust from the TG is conducted to a gas cell installed in the FTIR sample compartment via a temperature-controlled transfer-line, and the spectrum is measured. The sample passing through the gas cell is monitored as an IR chromatogram at the FTIR based on changes in the infrared light absorption. After the measurement is completed, qualitative analysis can be performed by extracting the desired spectra.

As the gas cell used here had an optical path length of 10 cm, and the internal diameter was wide as well, the standard model detector was used.



Fig.1 TG-FTIR System

■ Analysis of PET

Fig.2 and Fig.3 show the TG and TG derivative curves (D-TG curve) of polyethylene terephthalate in nitrogen and air atmospheres, respectively, with heating conducted at 20 °C/min. In addition, the corresponding 3-dimensional IR spectra are shown in Fig.4 and Fig.5.

Table 1 Analytical Conditions

Resolution	: 4 cm ⁻¹
Interval	: 30 sec
Detector	: DLATGS
Cell Temperature	: 200 °C

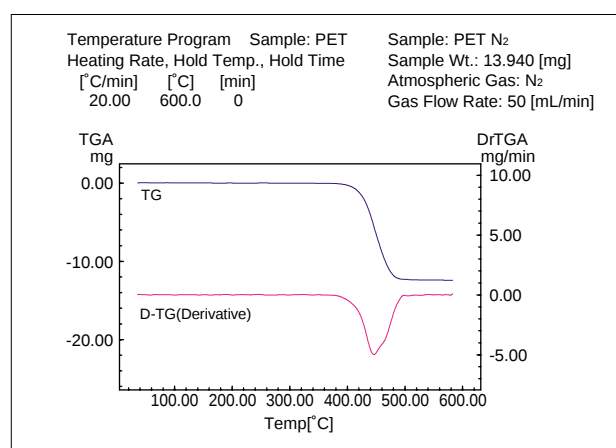


Fig.2 TG and D-TG Curves of PET (in nitrogen atmosphere)

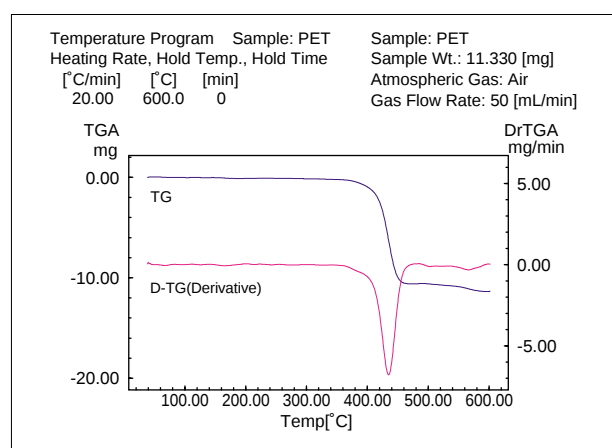


Fig.3 TG and D-TG Curves of PET (in air atmosphere)

In the measurements in air, two peaks appear at 440 °C and 570 °C, respectively, in the D-TG curve of Fig.3, and the corresponding absorption peaks are confirmed in the IR spectrum of Fig.5.

Fig.6 and Fig.7 are IR chromatograms showing the change in absorbance plotted on the time axis at the absorption wavenumbers 2361 cm^{-1} and 1760 cm^{-1} , corresponding to CO_2 and the carbonyl group. In this way, the state of the evolved gasses can be confirmed. Referring to this IR chromatogram, the 41st spectrum of Fig.6 was extracted and superimposed with the

search result, as shown in Fig.8. From this result, it can be predicted that in addition to CO_2 , benzoic acid is a principal ingredient in the nitrogen atmosphere measurement.

On the other hand, Fig.9 shows the spectra extracted at the arrow-marked sites in Fig.7, the result of the measurement in air. Besides the generation of CO_2 in two separate instances, the rapid generation of benzoic acid in the first half and the gradual generation of an ester compound are also observed.

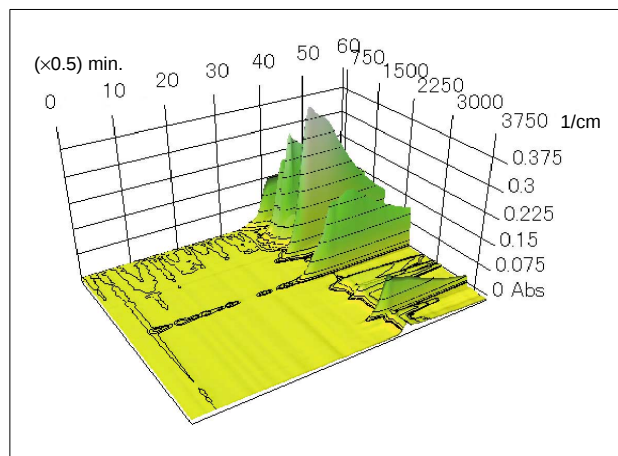


Fig.4 3D display of IR Spectra (in N_2 atmosphere)

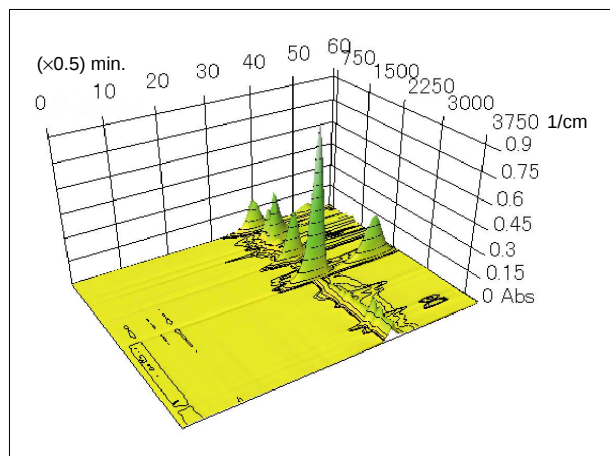


Fig.5 3D display of IR Spectra (in Air atmosphere)

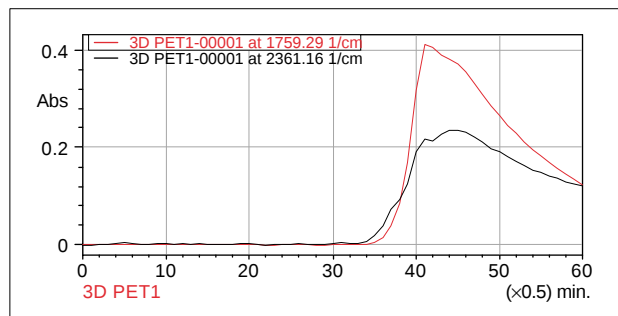


Fig.6 IR Chromatograms extracted from Fig.4

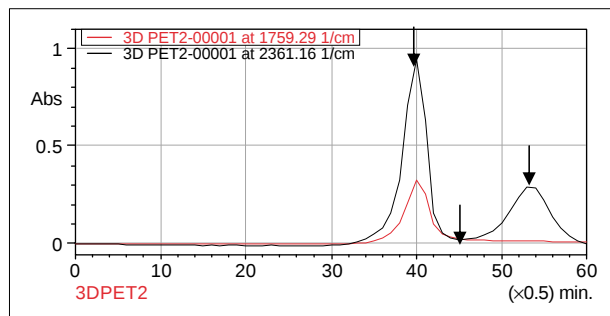


Fig.7 IR Chromatograms extracted from Fig.5

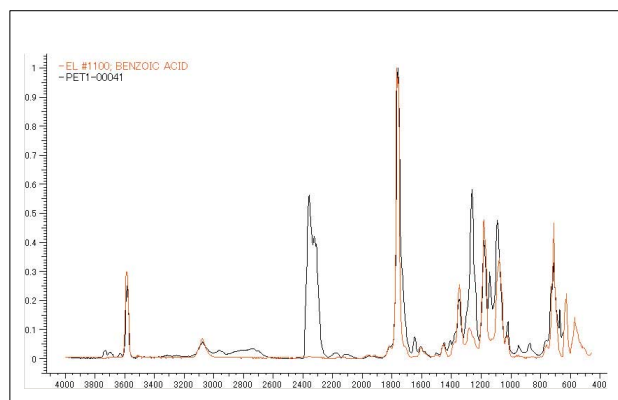


Fig.8 IR Overlay of Extracted Spectrum from Fig.6 with Search Result Spectrum

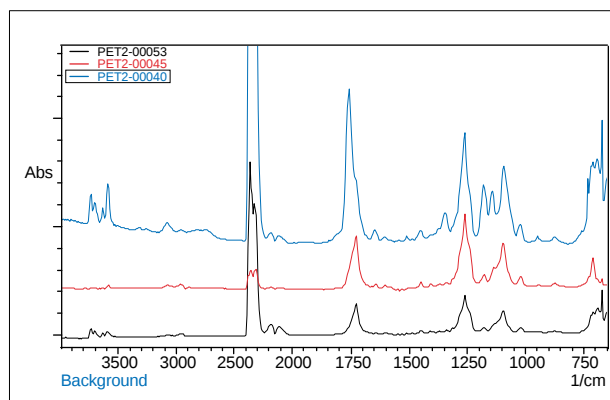


Fig.9 IR Spectra extracted from Fig.7

NOTES:

*This Application News has been produced and edited using information that was available when the data was acquired for each article. This Application News is subject to revision without prior notice.



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