

Application News

Thermal Analysis

No.T145

Evolved Gas Analysis by TG-FTIR

■ Introduction

In TG-FTIR, mass changes such as sample decomposition and vaporization due to changes in temperature are measured quantitatively by the TG, while at the same time, spectral analysis of the evolved gases by FTIR can be conducted for qualitative analysis. This analytical system can be applied to a wide range of analyses, such as

qualitative analysis of fragrances, trace analysis of additives, compositional analysis, unreacted substance analysis, and analysis of pyrolysis mechanisms, to name a few.

Here we introduce examples of calcium oxalate and polyethylene terephthalate (PET) analysis.

■ TG-FTIR System Configuration

Evolved gas from the heated sample is conducted from the TG to a gas cell in the FTIR via a temperature-controlled transfer line, and the spectrum is measured. The sample that passes through gas cell is monitored as an IR chromatogram at the FTIR based on changes in the infrared light absorption. After completion of the measurement, qualitative analysis can be performed by extracting spectra at specific times (temperatures).



Fig.1 TG-FTIR System

■ Analysis of Calcium Oxalate (CaC₂O₄ • H₂O)

Fig. 2 shows the TG-DTA curves when calcium oxalate monohydrate is heated at a rate of 20 °C/min. The 3-dimensional IR spectra are shown in Fig. 3. In addition, Fig. 4 shows the IR chromatograms of the change in absorbance plotted on the time axis at the absorption wavenumbers 2361 cm⁻¹ and 1508 cm⁻¹, corresponding to CO₂ and H₂O, respectively. Fig. 5 shows the spectra extracted at ① 175 °C, ② 460 °C, and ③ 720 °C from Fig. 4. From this, it can be seen in Fig. 2 that H₂O is volatilized at about ① 175 °C, the formation of CO and oxidization of CO₂ are observed in the vicinity of ② 460 °C, and the generation of CO₂ is observed at about ③ 720 °C.

Table 1 Analytical Conditions

Heating Rate	: 20 °C/min
Atmosphere	: Air
Resolution	: 8 cm ⁻¹
Interval	: 15 sec
Detector	: DLATGS
Cell Temperature	: 150 °C

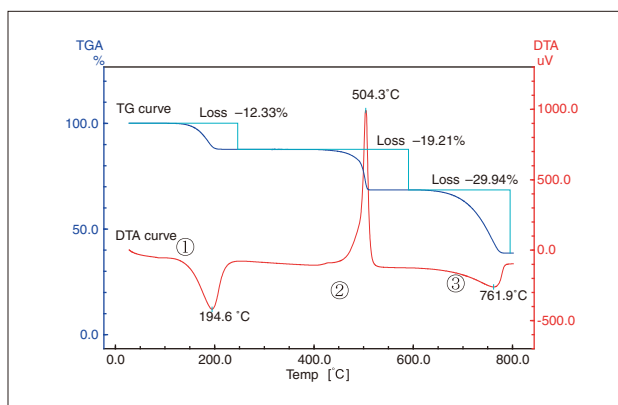


Fig.2 TG-DTA Curves of CaC₂O₄ in Air

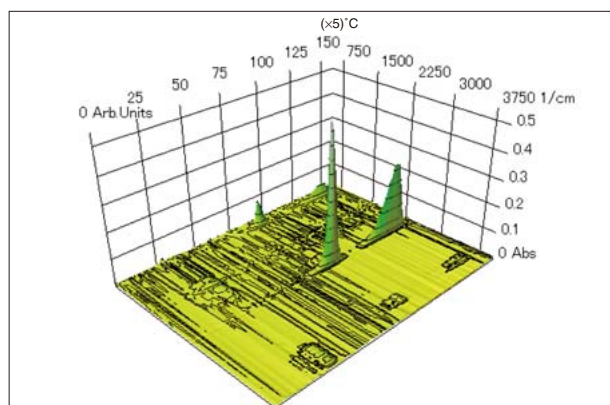


Fig.3 3D Display of IR Spectra

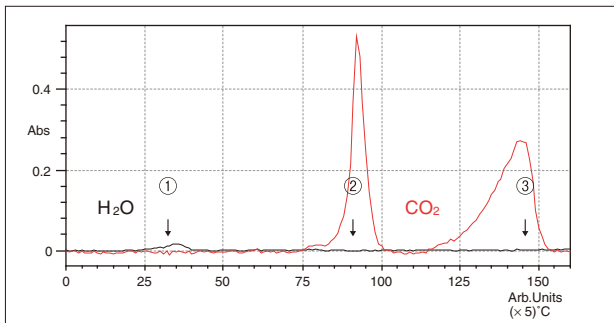


Fig.4 IR Chromatograms Extracted from Fig.3

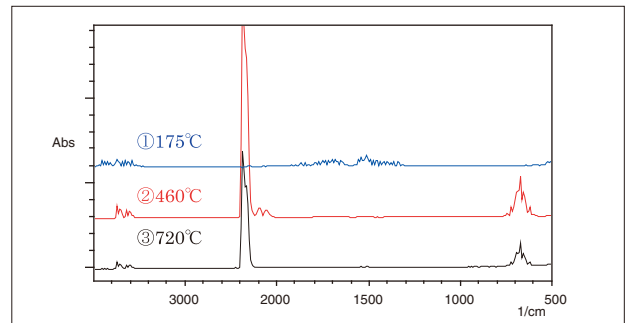


Fig.5 IR Spectra Extracted from Fig.4

■ Analysis of PET

Fig. 6 shows the TG and TG derivative curves when PET is heated in air at 20 °C/min. Two peaks appear in the D-TG curve, and the absorption peaks associated with these can be seen in the 3-dimensional IR spectra of Fig. 7. In addition, Fig. 8 shows IR chromatograms at the absorption wavenumbers 2361 cm⁻¹ and 1760 cm⁻¹, corresponding to CO₂ and the carbonyl group, respectively. The spectra extracted from the arrow-marked sites ① and ② of Fig. 8 are shown in Fig. 9. From analysis of these IR spectra, besides the generation of CO₂ 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.

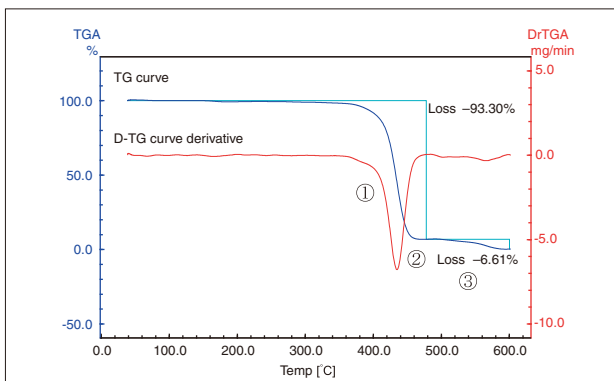


Fig.6 TG and D-TG Curves of PET in Air

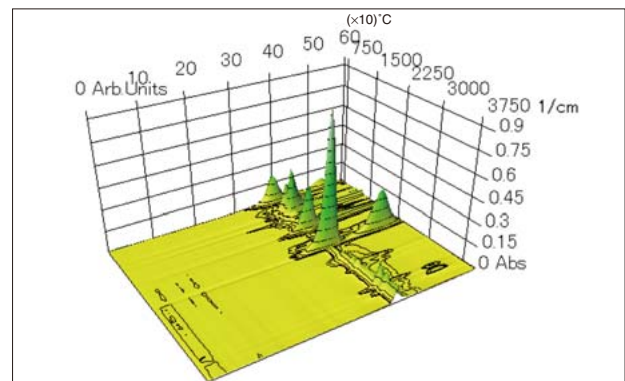


Fig.7 3D Display of IR Spectra

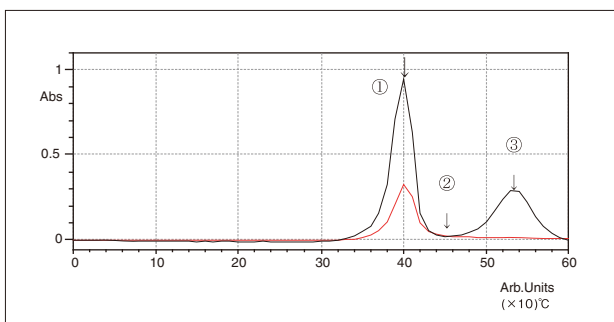


Fig.8 IR Chromatograms Extracted from Fig.7

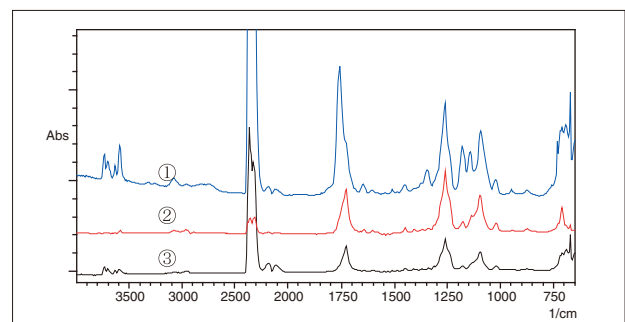


Fig.9 IR Spectra Extracted from Fig.8

Table 2 Analytical Conditions

Heating Rate	: 20 °C/min
Atmosphere	: Air
Resolution	: 4 cm ⁻¹
Interval	: 30 sec
Detector	: DLATGS
Cell Temperature	: 200 °C

* Fig. 6 - 9: Measured by Shimadzu (Asia Pacific) Pte. Ltd.