SHIMADZU APPLICATION NEWS

LA146-E019

● GASCHROMATOGRAPHY MASS SPECTROMETRY

No.M196

Analysis of Ethylene Vinylacetate (EVA) Copolymer by Double-shot Pyrolyzer PY-2020D

Pyrolysis Gas Chromatography (Py-GC) is employed as a rapid and highly sensitive method for analyzing polymers. Using the furnaces with the temprature programing function thermal extraction analysis of trace amounts of residual monomers and oligomers in polymer compounds has become possible.

This Application News introduces an analysis system that combines the GC/MS with a double-shot pyrolyzer (Frontier Laboratories Ltd.), which is capable of programed heating.

The PY-2020D Double-Shot Pyrolyzer is capable of three analytical modes.
(1) Single-Shot Mode (Flash Pyrolysis)
(2) Double-Shot Mode (Multi-Step Pyrolysis)
(3) Evolved Gas Analysis Mode

(Portions of literatures from Frontier Laboratories Ltd. were used for this document.)

Fig.1 Double-Shot Pyrolyzer (PY-2020D)

The difference between the single-shot and double-shot methods is outlined below. The double-shot method first analyzes the volatile substances in the polymer by thermal desorption, and then analyzes the remaining polymer substrate by flash pyrolysis, providing two sets of information from one sample.

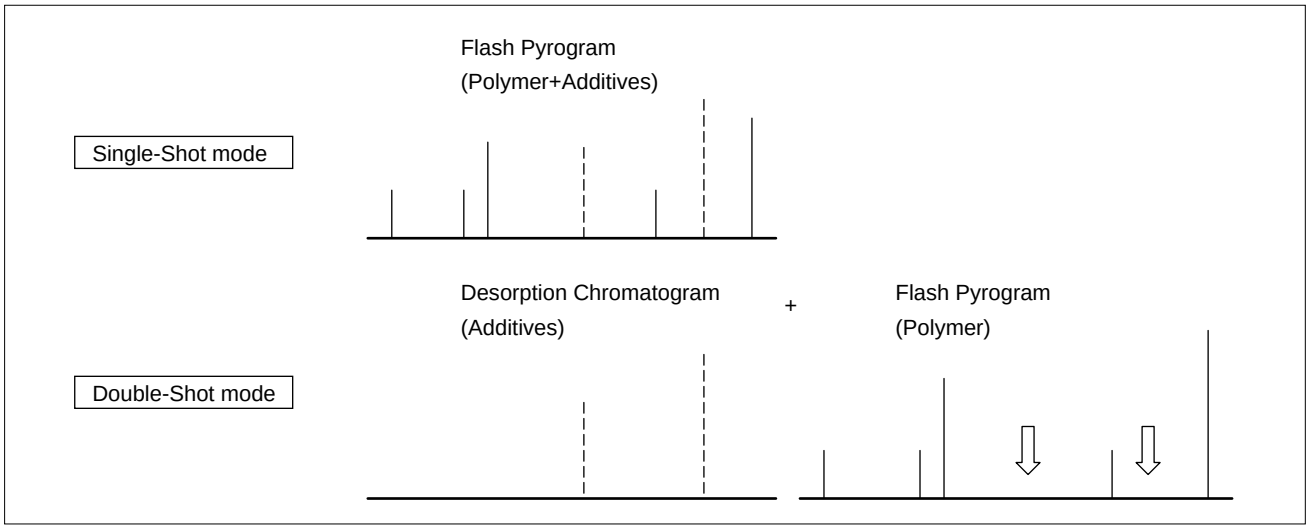

Fig.2 Comparison of Single-shot and Double-shot Methods (Provided by Frontier Lab Ltd.)

The following shows an example of analyzing an actual sample. Fig. 3 shows the results of evolved gas analysis (EGA-Direct MS) by heating EVA (ethylene vinyl acetate) from 50˚C to 650˚C at a heating rate of 20˚C per minute.

Small peaks are observed between 100˚C and 260˚C, and peaks due to the decomposition of the polyvinyl structure are observed between 260˚C and 550˚C. The peaks between 100˚C and 260˚C are considered to be generated by the vaporization of residual monomers and additives in the sample. Thus the mass chromatograms for m/z=60 (the molecular ion of acetic acid CH_3COOH), and for m/z=57 (a hydrocarbon fragment)are shown. It revealed that m/z=60 appears between 100˚C and 240˚C, and m/z=57 appears at 260˚C.

There is two peaks in ECA curve of EVA (Fig. 3). Peak 1 is observed between 260˚C and 400˚C. Peak 2 is observed between 400˚C and 550˚C. To ideneify the compounds in these peaks the double-shot method was used. The sample firstly is heated up to 400˚C and the generated gases are analyzed. And then the sample is heated up to 500˚C and analyzed. These results are shown at Fig. 4 and Fig. 5.

Table 1 Analytical conditions

-GC-	
Column	: Ultra ALLOY+5
Column Temp.	: (30m×0.25mm I.D. df=0.25μm) 50˚C (5min)-10˚C/min-320˚C (30min)
Carrier Gas	: 100kPa
Injection Temp.	: 300˚C
Injection Method	: Split 1:50
-MS-	
Interface Temp.	: 280˚C
Ionization Method	: EI
Scan Range	: m/z10-500
Scan Interval	: 0.5sec

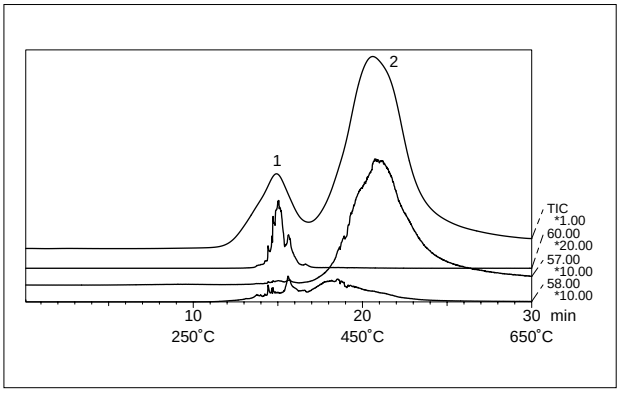

Fig.3 EGA Curve of EVA

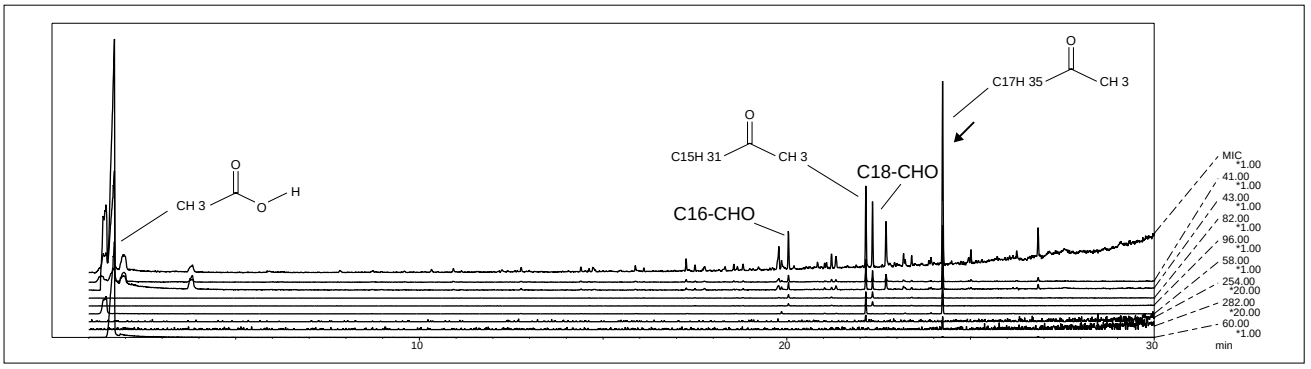

Fig.4 Mass Chromatograms of EVA (Pyrolysis Temp. 400˚C)

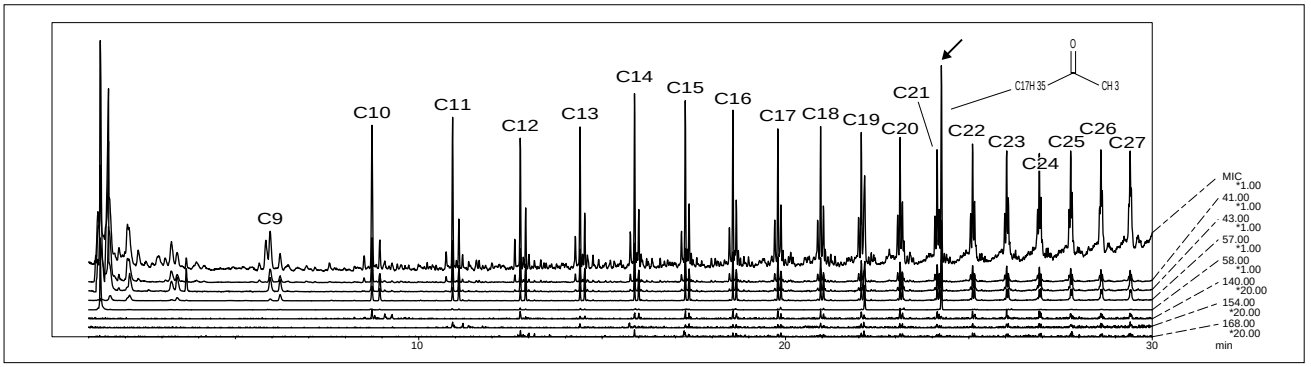

Fig.5 Mass Chromatograms of EVA (Pyrolysis Temp. 550˚C)

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