



Hybrid Pyrolyzer
JHI-08

Analysis using Hybrid Pyrolyzer 1 “Hybrid” Analysis of EPDM

Keyword:

Pyrolysis-GC/MS, Thermal Extraction-GC/MS, Additives Analysis, Polymer Composition Analysis

Introduction

JHI-08 is a pyrolyzer for GC/MS having two ways of heating system, high frequency induction heating and resistance heating. High frequency induction heating system can raise temperature instantly and therefore is good for pyrolysis, while resistance heating, where you can program heating rate and final temperature as you like, is good for thermal extraction analysis.

Further, JHI-08 has “Hybrid Mode” under which you can conduct thermal extraction analysis and pyrolysis analysis on one sample continuously without interruption.

The hybrid mode analysis is quite useful as more data can be obtained from limited amount of sample.

Here is an example of analysis using Hybrid Mode of JHI-08.

Experiment

Sample : Unvulcanized EPDM
Instruments : JHI-08 Hybrid Pyrolyzer, Column Inlet Cooler (Optional), GC/MS

Analysis Overview

Fig. 1 shows the entire analysis flow. Everything goes automatically under the hybrid mode.

The first analysis was thermal extraction-GC/MS analysis where residual solvents, unreacted monomers, additives and others were analyzed.

The second analysis, which started immediately after the first one, was pyrolysis-GC/MS for polymer composition analysis.

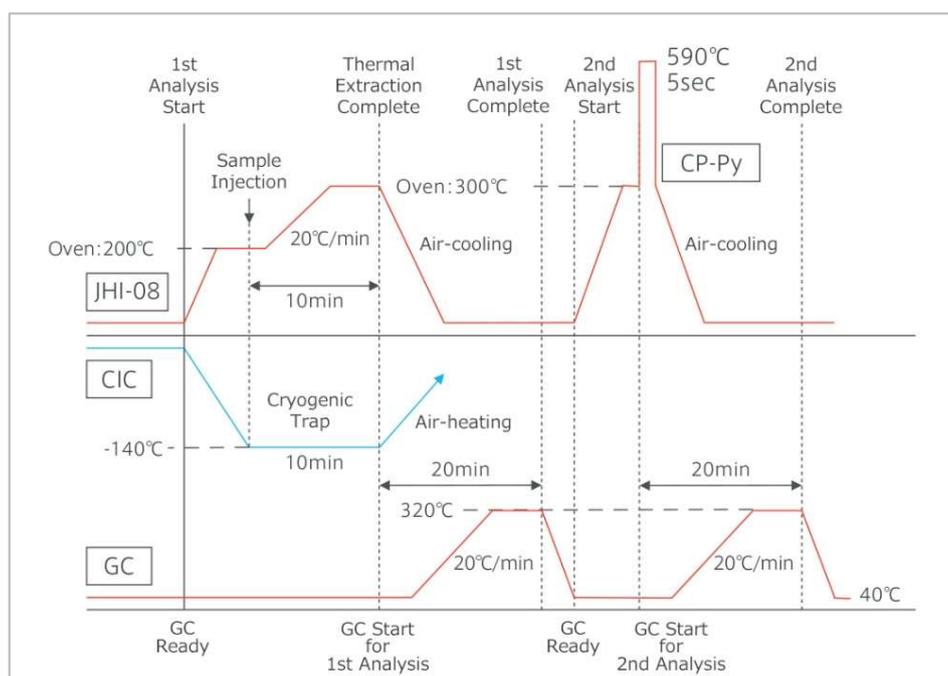


Fig. 1 Automatic Analysis Flow Overview

Results

The 1st analysis (Thermal Extraction-GC/MS)

Extraction Temperature
200°C (1 min) > 20°C/min > 300°C (4 min)

Thermally-extracted gas was cooled down to -140°C by CIC and was trapped at the column inlet. After extraction completed and the gas was totally trapped, GC column oven temperature was raised as programmed. Thermal extraction chromatogram was obtained as shown in Fig. 2.

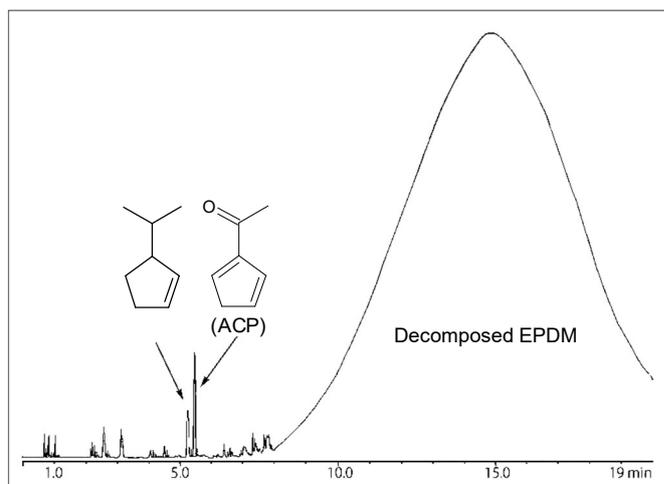


Fig. 2 Thermal extraction chromatogram of unvulcanized EPDM

The 2nd analysis (Pyrolysis-GC/MS)

Pyrolysis temperature : 590°C
(Pyrofoil® F590)

After pyrolysis-GC/MS analysis, the pyrogram was obtained as shown in Fig. 3.

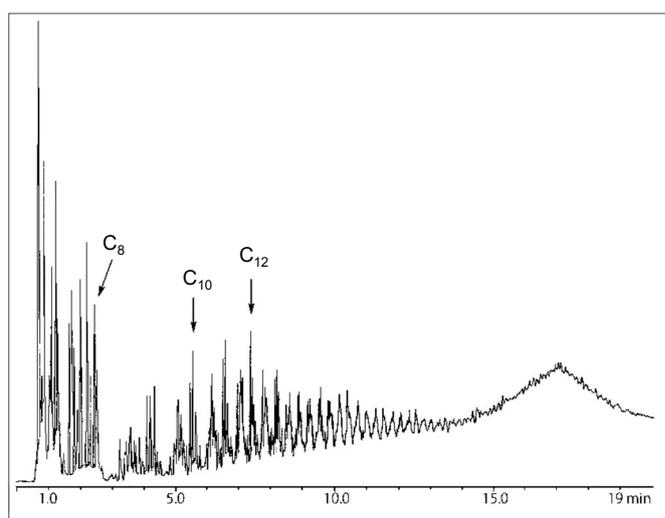


Fig. 3 590°C Pyrogram of unvulcanized EPDM

Conclusions and Discussion

Fig. 3 represents typical pyrogram of unvulcanized EPDM, however, it does not suggest existence of the third component, “diene monomer”.

In chromatogram (Fig. 2), on the other hand, compounds such as Isopropylcyclopentene and Acetylcyclopentadiene (ACP), which are assumed to have been generated during thermal extraction, were detected. So from the thermal extraction-GC/MS analysis, it is presumed that the third component is 5-Ethylidene-2-norbornene (ENB), from which these compounds were derived.