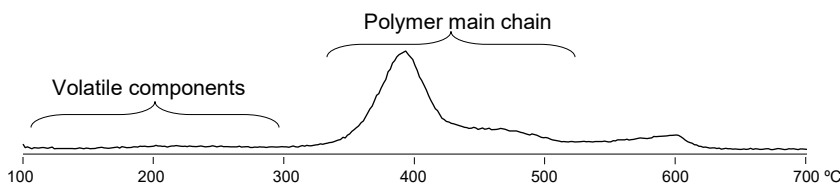


# Analysis of Compounded Rubber by Double-Shot Technique

Fig. 1 shows an evolved gas curve of a compounded rubber. Weak peaks are observed in 100~300 °C region due to the thermal desorption of various additives. In 300~500 °C region, a broad peak due to thermal decomposition of the rubber is observed. From this result on hand, thermal desorption, the first stage of double-shot analysis, was performed from 100 to 300 °C (20 °C/min), and flash pyrolysis, the second stage, was done at 550 °C.

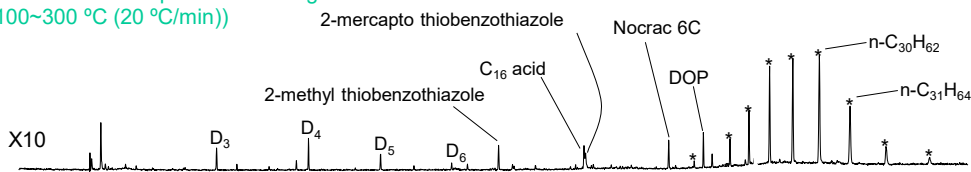
Fig. 2 shows the results of analysis. In the chromatogram of thermal desorption shown in Fig. 2a, cyclic siloxanes (D3~D6) originated from silane coupling agent, benzothiazole (vulcanization accelerator), higher aliphatic acid (vulcanizing agent), and waxes (antioxidants, marked with \*) were observed. Because isoprene and limonene were mainly observed in the pyrogram shown in Fig. 2b, the major component of this sample is natural rubber; further, it contains butadiene rubber since a small amount of butadiene was also observed.



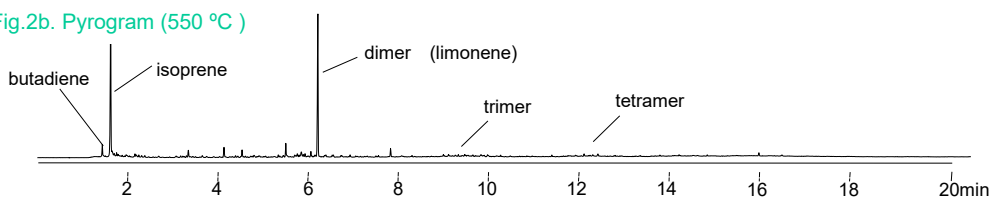
**Fig. 1 Evolved Gas Curve of a Compounded Rubber**

Pyrolysis temp.: 100~700 °C (20 °C/min), Carrier gas : He 50 kPa, Split ratio : ca. 1/20  
EGA capillary tube : 0.15 mm id, 2.5 m (UADTM-2.5N), GC oven temp.: 300 °C  
Injection temp.: 320 °C, Sample : ca. 500 µg, Detector : MS ( $m/z=29-400$ )

**Fig.2a. Thermal Desorption Chromatogram (100~300 °C (20 °C/min))**



**Fig.2b. Pyrogram (550 °C)**



**Fig. 2 GC/MS Analysis of Compounded Rubber by Double-Shot Technique**

Column flow rate : 1 mL/min (fixed flow rate), Split ratio : 1/20  
Separation column : Ultra ALLOY™-5 (5 % diphenyl polysiloxane), 30 m, 0.25 mm id, Film thickness : 0.25 µm  
GC oven temp.: 40~300 °C (20 °C/min), Sample : 500 µg, Detector : MS ( $m/z=29-400$ , 2 scan/sec)

**Keywords :** Double-Shot Technique, Rubber, Coupling Agent, Vulcanizing Agent, Antioxidant

**Products used :** Multi-functional pyrolyzer, UA-5, UADTM-2.5N

**Applications :** General Polymer Analysis

**Related technical notes :**

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