

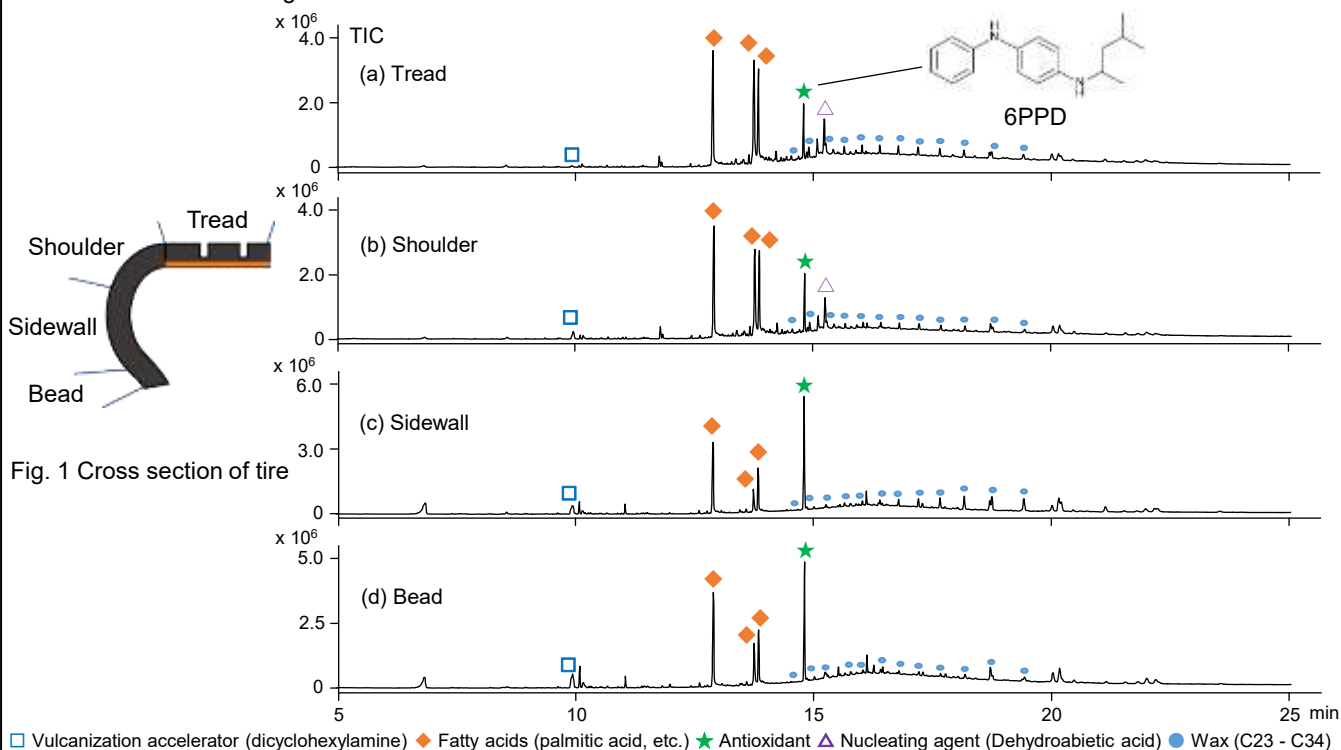
# Analysis of tire rubbers

## Part 1 Additives analysis by thermal desorption-GC/MS

**[Background]** Automobile tires are made up of many different parts. When manufacturing automobile tires, each part of the tire is specifically formulated to reflect the different demands for the tire functions. Different rubber formulations are used to ensure that the rubber is molded with additives and rubber materials appropriate for a specified part of the tire. Knowledge of how specific additives affect rubber performance is extremely important in product development. In this report, the additives contained in different parts of an automobile tire was analyzed using thermal desorption (TD)-GC/MS.

**[Experimental]** Rubber samples were collected from the (a) tread, (b) shoulder, (c) sidewall, and (d) bead parts of an automobile tire (Fig. 1) and were washed with water. Then, each sample was cut into approximately 1 mm squares. TD-GC/MS measurements were done using a GC/MS system in which a Multi-Shot Pyrolyzer (EGA/PY-3030D) was directly interfaced to the GC injector. For TD analysis, the pyrolyzer furnace was temperature-programmed from 100 to 320 °C (20 °C/min, 1 min hold), and the gases evolved from the sample were identified as various additives using GC/MS. Among identified additives, an antiaging agent N-phenyl-N'-(1,3-dimethylbutyl)-p-phenylenediamine (6PPD) was determined for each part of the tire rubber by the absolute calibration method.

**[Results]** The TD chromatograms of the four rubber samples are shown in Fig. 2. In Figs. 2 (a) and (b), vulcanization accelerators, fatty acids, antiaging agents, nucleating agents, waxes, etc. are identified. In Figs. 2 (c) and (d), the additives present in (a) and (b) are similarly detected, but no nucleating agent is detected. 6PPD contents in tread, shoulder, sidewall, and bead were 1452 ppm, 1744 ppm, 6122 ppm, and 4868 ppm, respectively (RSD < 5 %, n = 3). TD-GC/MS is a simple technique that can be used to quickly characterize the individual constituents found in complex matrices like those commonly encountered when working with various rubber formulations.



Thermal desorption temp.: 100 - 320 °C (20 °C/min, 1 min hold), Sample amount: ca.0.2 mg, MS scan range: *m/z* 29 - 600  
 Split ratio: 1/20, GC oven: 40 °C (2 min hold) - 320 °C (20 °C/min, 14 min hold), Column flow rate: 1 mL/min  
 Separation column: Ultra ALLOY-5 (5 % diphenyl-95 % dimethylpolysiloxane), L=30 m, i.d.=0.25 mm, df=0.25 μm

**Keywords :** Rubber, Additives, Thermal desorption-GC/MS

**Products used :** Multi-functional pyrolyzer, UA<sup>+</sup>-5, Vent-free GC/MS adapter

**Applications :** General polymer analysis, Rubber industry, Additive analysis, QA

**Related technical notes :** [PYA1-102E](#), [PYA1-095E](#), [PYA1-109E](#), [PYA-115E](#)

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