

Analysis of additives in butadiene rubber using thermal desorption (TD)-GC/MS

[Background] One of the synthetic methods for producing butadiene rubber is the solution polymerization of a diene in a hydrocarbon solvent such as hexane or benzene. A lithium catalyst serves as an initiator and helps control the microstructure of the rubber. Functional butadiene rubbers can be produced by adding an organic additive containing sulfur which serves as a vulcanization accelerator and an antioxidant. Other inorganic reinforcing agents such as carbon black may also be present in the rubber. In this report, additives in a butadiene rubber sample were analyzed by thermal desorption (TD)-GC/MS.

[Experimental] A GC/MS system with a Multi-Shot Pyrolyzer (EGA/PY-3030D) interfaced directly to the GC injector was used for the TD analysis. Synthetic butadiene rubber sample (unvulcanized) was cut into small pieces and placed in a sample cup which was subsequently heated from 100 - 330 °C at 20 °C/min under a helium gas flow. The volatile compounds evolved from the sample were temporarily cryo-trapped at the head of a separation column using a Microjet Cryo-Trap (MJT-1035E). Then, the Cryo-Trap was turned off and the trapped compounds were analyzed by GC/MS, by which TD chromatogram of the volatile compounds was obtained. The polymeric fraction of the butadiene rubber remained in the sample cup as a residue.

[Results] A TD chromatogram of butadiene rubber is shown in Fig.1. Antioxidants octyl disulfide and Irganox1076 were detected. 1-Octane thiol, presumably a decomposition product, and 1-octadecanol were also detected. 2,2-di(2-tetrahydrofuryl)propane was detected as small peaks and is supposed to be used as an agent for modifying the microstructure. Using the standard addition method, the concentration of Irganox1076 was determined to be 2860 ppm.

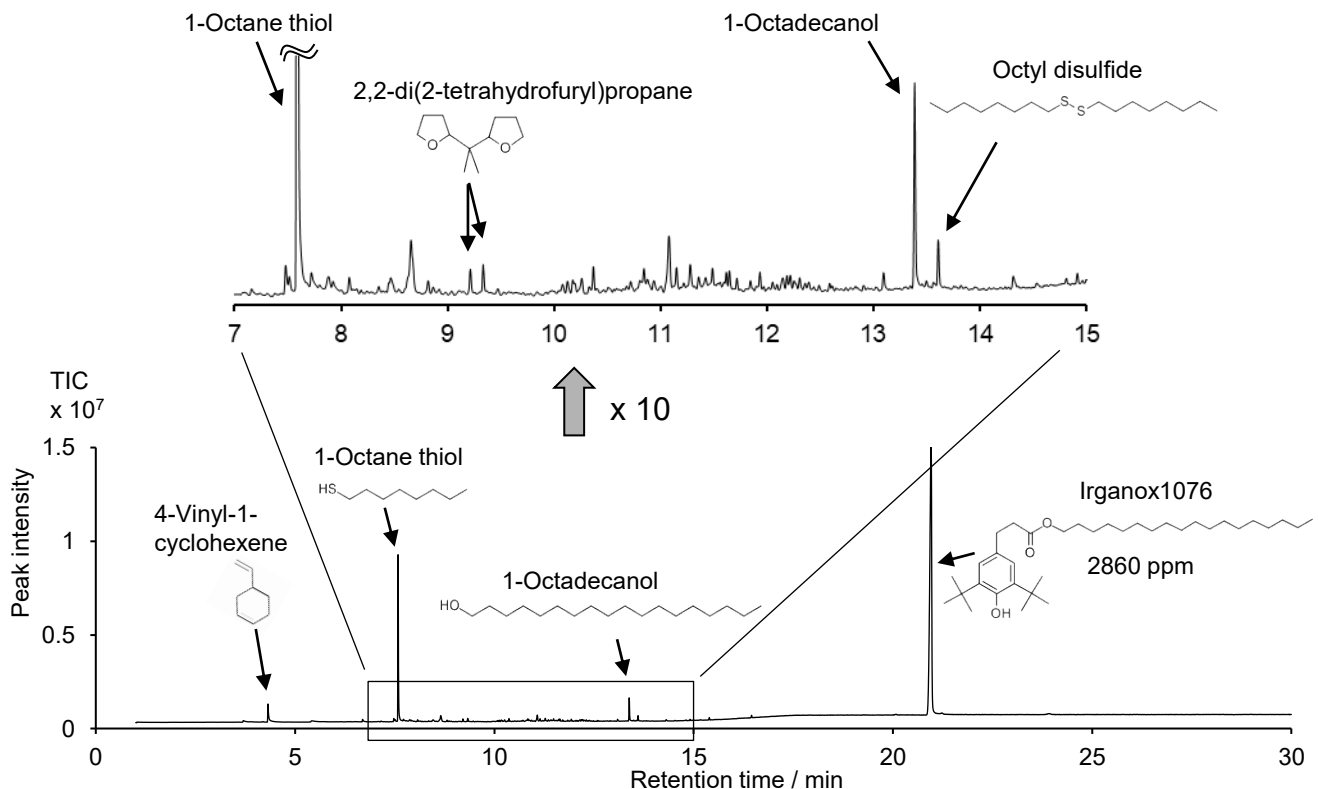


Fig.1 TD chromatogram of butadiene rubber sample

Furnace temp.: 100 - 330 °C (20 °C/min), Sample amount: 1.31 mg,
 Split ratio: 1/50, GC oven: 40 °C (2 min hold) - 320 °C (20 °C/min, 14 min hold),
 Separation column: UA⁻-5 (5 % diphenyl 95 % dimethylpolysiloxane, L=30 m, i.d.=0.25 mm, df=0.25 μm)
 Column flow rate: 1 mL/min (He), MS scan range: *m/z* 29 - 600

Keywords : Synthetic rubber, Butadiene rubber, Thermal desorption-GC/MS, Randomizer, Antioxidant, Stabilizer

Products used : Multi-Shot Pyrolyzer, UA⁻-5, Vent-free GC/MS adapter, F-Search, MicroJet Cryo-Trap

Applications : Rubber industry, Additive analysis, General polymer analysis

Related technical notes : PYA1-003, PYA1-015

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