

Identification of an unknown vulcanization accelerator in vulcanized rubber using F-Search and the Additive MS library

[Background] Solvent extraction-GC/MS and thermal desorption (TD)-GC/MS are generally used for the analysis of vulcanization accelerators (VAs) present in vulcanized rubber. However, it is often difficult to identify VAs because the original chemical structure of VAs is altered due to their reaction with sulfur during the vulcanizing process. This reduces the identification accuracy when using most general-purpose MS libraries. In addition, the number of mass spectra of VAs in general-purpose MS libraries is limited. This note describes how F-Search and an Additive MS library (P/N: PY-1114E-161) can be used to identify an unknown VA in a vulcanized rubber.

[Experimental] A vulcanized rubber which contained an unknown VA was analyzed by TD-GC/MS. The EGA thermogram was used to determine the best TD temperature profile: 100 - 340°C. Using this profile, thermal decomposition of the base rubber was minimized.

[Results] A chromatogram of the vulcanized rubber is shown in Fig. 1(a). Peaks 1 to 3 in the chromatogram were identified as shown in Fig. 1(b) based on the similarities of their retention indices (RI) and mass spectra with those of compounds stored in the Additive MS library. A rubber vulcanization accelerator TBBS (*N-tert-Butyl-2-benzothiazolesulfenamide*, trade name: Accel BNS-R, Sanceler NS, etc.) was identified by comparing each of the three peaks shown in Figs. 1(a) and (b). Although the original VA was not detected as an intact molecule, it was possible to identify the original VA from the decomposition products using F-Search and the Additive MS library.

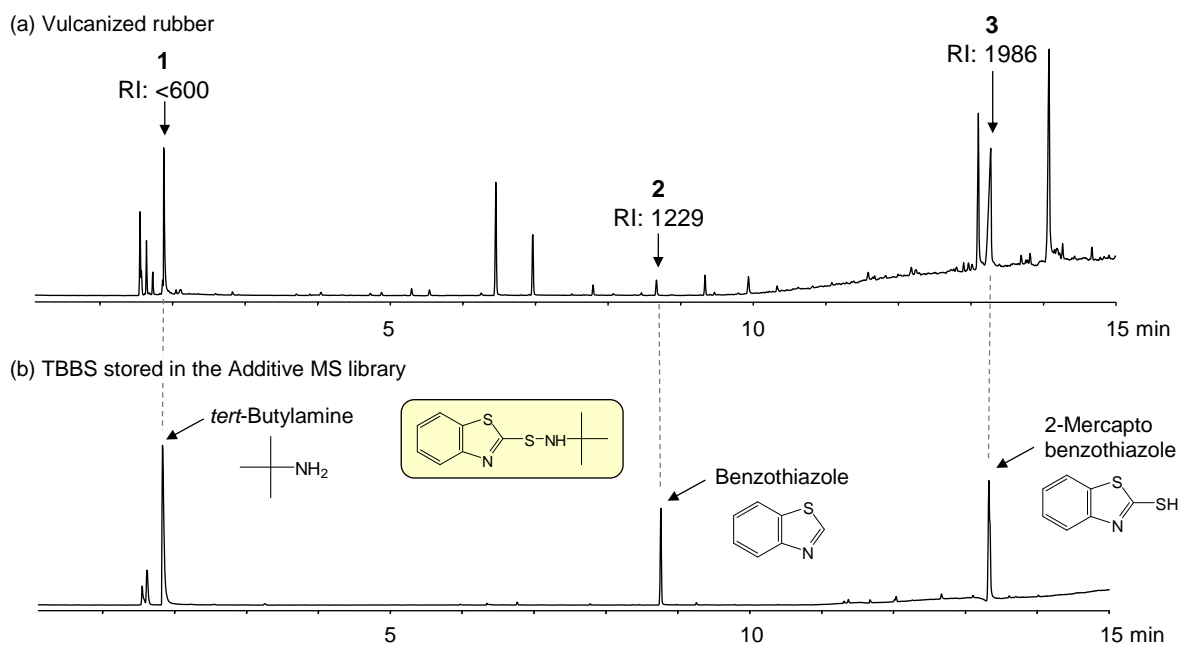


Fig. 1 Chromatograms obtained by TD-GC/MS

Furnace temp.: 100-340 °C (40 °C/min), GC oven temp.: 40 (2 min hold) – 320 °C (20 °C/min, 14 min hold)
Column: Ultra ALLOY-5(MS/HT), L=30 m, i.d.=0.25 mm, df=0.25 µm, split ratio: 1/10, sample amount: 1.0 mg

Ref. Shiono *et al.*, 20th Polym. Anal. & Characterization, 2015.

Keywords : Rubber, Vulcanization accelerator, MS library, Thermal desorption, Additives

Products used : Multi-functional pyrolyzer, Auto-Shot Sampler, MicroJet Cryo-Trap,
Vent-free GC/MS adapter, UA-5(MS/HT), F-Search, Additive MS library (P/N: PY-1114E-161)

Applications : General polymer analysis, Rubber and related industries, Additive analysis

Related technical notes : PYA1-054E, PYA1-057E, PYA1-066E

Please forward your inquiries via our web page or send us a fax message.

R&D and manufactured by :
Frontier Laboratories Ltd.

Phone: (81)24-935-5100 Fax: (81)24-935-5102
<http://www.frontier-lab.com/>