Identification of unknown vulcanization accelerator in vulcanized rubber samples using additive MS library (Part 2)

[**Background**] Solvent extraction-GC/MS and thermal desorption (TD)-GC/MS are commonly used to analyze accelerators in vulcanized rubber samples. However, it is sometimes difficult to identify accelerators because accelerators react with sulfur during the vulcanization process and general-purpose MS libraries do not contain a sufficient number of accelerators and their degradation products. In this report, the identification of an unknown vulcanization accelerator in a vulcanized rubber sample is attempted using the additive MS library ADD-MS22B (Frontier Labs).

[**Experimental**] A vulcanized rubber sample containing about 1 % of unknown vulcanization accelerator was used and measured by TD-GC/MS. The thermal desorption temperature was programmed from 100 to 340 ºC, at which temperature the base rubber hardly thermally decomposes. Search software F-Search (Frontier Labs) and the additive MS library ADD-MS22B (Frontier Labs) and were used for data analysis.

[**Results**] The TD chromatogram of the vulcanized rubber sample is shown in Fig. 1(a). The major peaks 1 through 4 of the chromatograms obtained were identified as the compounds shown in Fig. 1(b) based on the match quality of mass spectra and retention indices (RI). Furthermore, comparing the peak intensity ratio with the one in the chromatogram stored in this MS library shown in Fig. 1(b) revealed the vulcanization accelerator should be Accel D or Sanceler D (compound name: 1,3-Diphenylguanidine). Peak 4 is a compound of the original vulcanization accelerator itself, and peaks 1 through 3 of decomposition products are observed on the chromatogram, so the vulcanization accelerator in question is certainly contained in the rubber sample. As described the above, this MS library was found to be useful for qualitative identification of unknown vulcanization accelerators.

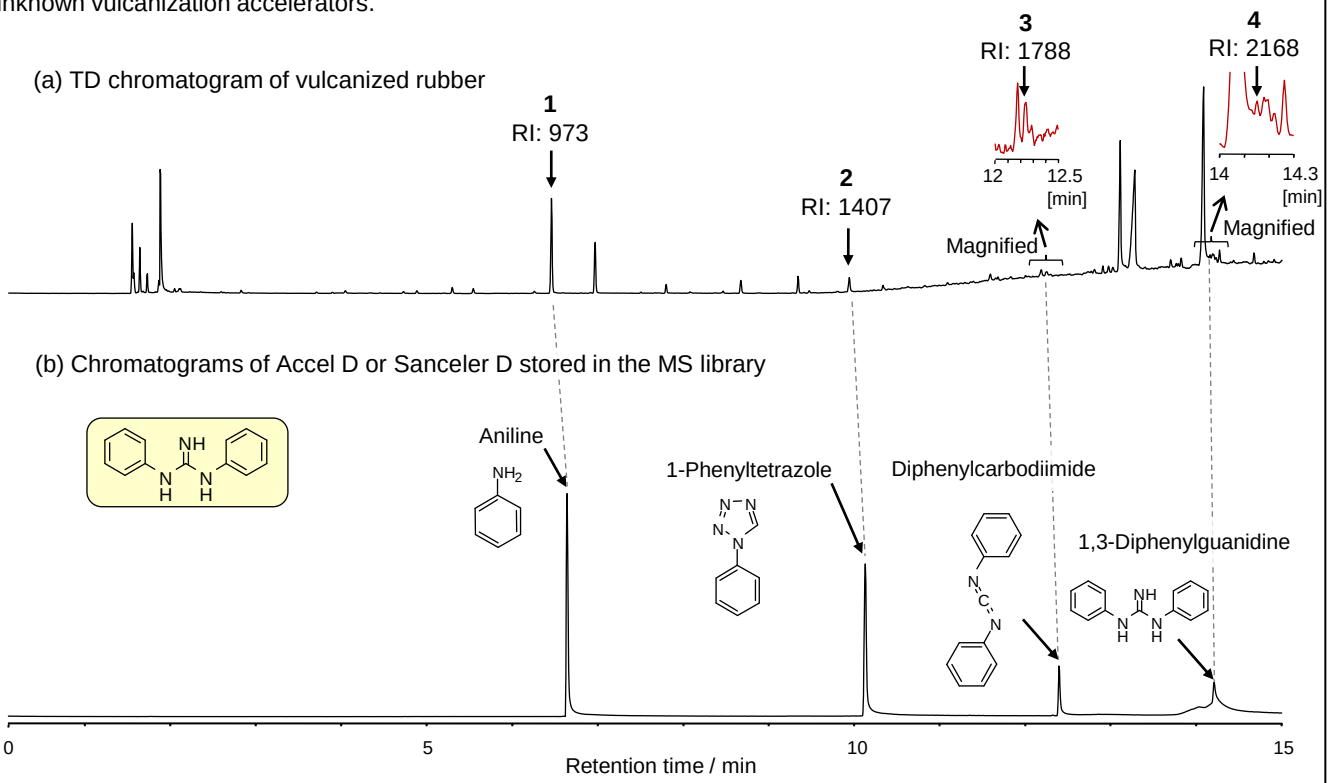

Fig. 1 Chromatograms obtained by TD-GC/MS

TD temp.: 100-340 ºC (20 ºC/min), GC Oven Temp.: 40 (2 min hold) - 320 ºC (20 ºC/min, 14 min hold),
Separation Column: Ultra ALLOY-5 (MS/HT), L=30 m, i.d.=0.25 mm, df=0.25 µm,
Column flow rate: 1 mL/min, Split ratio: 1/10, Sample weight: 1.0 mg.

Keywords : Rubber, Vulcanization accelerator, MS Library, Thermal Desorption

Products used : Multi-functional Pyrolyzer, Auto-Shot Sampler, MicroJet Cryo-Trap, Vent-free GC/MS Adapter, UA⁺-5, F-Search

Applications : General Polymer Analysis, Rubber Industry, Additive Analysis

Related technical notes : PYA1-054E, PYA1-057E, PYA1-066E, PYA1-087E (Part 1)

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