Quantitative analysis of fatty acids in vulcanized styrene-butadiene rubber by thermal desorption and thermally assisted hydrolysis and methylation-GC/MS

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Abstract:
Determination of stearic acid in rubber is very important from the viewpoint of quality control of products. In place of the conventional liquid-phase extraction procedures for the analysis of additives in rubber, stearic and palmitic acids in vulcanized styrene–butadiene rubber (SBR) samples were directly analyzed in the solid state by thermal desorption (TD)–gas chromatography (GC)/mass spectrometry (MS) and thermally assisted hydrolysis and methylation (THM)–GC/MS. It was found that the precision of analytical data was only fair in the TD-GC/MS analysis, with 7.8% relative standard deviation (RSD), because of the interaction between the polar fatty acids and basic sites in the GC chromatographic system. On the other hand, THM-GC/MS, in which the fatty acids are derivatized to the methyl esters using tetramethylammonium hydroxide, can overcome this problem. Under the optimized measurement conditions of this study for THM-GC/MS, the average determined value (0.62 wt%) of total fatty acids in the SBR samples was found to agree well with the compounded amount (0.64 wt%) used in the preparation stage of the SBR samples, and the RSD was 3.2%.

Frontier Labs products used:
Multi-Shot Pyrolyzer, Micro puncher, UAx-5, UADTM-2.5N, Eco-cup