

[Quantitative analysis of acrylic acid in acrylic pressure-sensitive adhesives by reactive pyrolysis-GC/MS using N,O-bis\(trimethylsilyl\)trifluoroacetamide as a trimethylsilylation reagent with two-step heating](#)

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J. Anal. Appl. Pyrol. 175 (2023) 106170

Abstract:

Acrylic pressure-sensitive adhesives (APSAs) have been used in a variety of fields and the composition of their monomers is a very important factor in determining their physical properties. Monomers containing polar functional groups, such as acrylic acid (AA), are often used as raw materials for APSAs, and the number of these monomer units in the polymer chain has a significant effect on the adhesive properties. However, due to its high reactivity and polarity, it is not easy to accurately quantify AA in APSAs by conventional reactive pyrolysis gas chromatography mass spectrometry (Py-GC/MS). In this study, quantitative analysis of AA in APSAs was examined by reactive Py-GC/MS using N,O-bis(trimethylsilyl)trifluoroacetamide (BSTFA) as a trimethylsilylation (TMS) reagent (Py (BSTFA)-GC/MS). First, the APSA sample was mixed with BSTFA and heated at 60 °C for 30 min to accelerate TMS derivatization of any unpolymerized AA monomers (AA-TMS). If unreacted BSTFA remains after derivatization, it will react with AAs produced by the pyrolysis of acrylic ester units. These additional AA monomers will result in the over-estimation of the true amount of unpolymerized AA monomers. To prevent this undesirable effect by residual reagent, the TMS-derivatized sample was further heated at 100 °C for 10 min before pyrolysis in order to remove residual BSTFA and solvent. The derivatized sample was measured by Py-GC/MS at 600 °C and the relative standard deviation of the peak area ratio of AA-TMS derived from unpolymerized AA monomers to the internal standard (anthracene-d10) was less than 5% (n = 5). In the calibration curves made for four APSA samples with known compositions, good linearity was obtained for the raw materials of butyl acrylate and 2-ethylhexyl acrylate in addition to AA-TMS. Quantitative analysis of AA monomers in a commercial APSA sample showed fairly good agreement with the amount of AA determined by the official method of analysis using the acid-base titration (JIS K 5601-2-1). The results obtained in this study indicate that Py (BSTFA)-GC/MS is effective for the compositional analysis of APSAs.

* Excerpted from online journal website (Click the title)

Frontier Labs Products used:

Multi-Shot Pyrolyzer (EGA/PY-3030D), Auto-Shot Sampler (AS-1020E), Ultra ALLOY⁺-5, Vent-free GC/MS adapter, F-Search, Eco-Cup LF