

Quantitative analysis of acrylic acid in acrylic pressure-sensitive adhesives by reactive pyrolysis GC/MS with two-step heating

Part 2: Quantitative analysis of acrylic acid in commercial adhesives

[Background] In the previous note (PYA2-035E), the two-step heating method for trimethylsilylation (TMS) of acrylic acid (AA) in acrylic pressure-sensitive adhesives (PSAs) using *N,O*-bis(trimethylsilyl)trifluoroacetamide as a TMS reagent was described. In this note, using the two-step heating method, a calibration curve for AA is prepared by reactive pyrolysis(Py-) GC/MS of PSA samples with known compositions, and AA in a commercial PSA sample is determined using the calibration curve.

[Experimental] Model acrylic PSAs were synthesized by solution polymerization using butyl acrylate (BA), 2-ethylhexyl acrylate (2-EHA), and AA monomers with the compositions shown in Table 1. After adding anthracene-*d*₁₀ (Anth-*d*₁₀) as an internal standard, the model acrylic PSA sample was introduced into the pyrolyzer furnace heated at 600 °C for flash pyrolysis. A Py-GC/MS system equipped with a pyrolyzer (EGA/PY-3030D) directly connected to the GC injector was used.

[Result] The pyrograms of the four PSA samples are shown in Fig. 1 (a), and the extracted ion chromatograms (EIC, *m/z* 129) of the TMS-derivatized AA (AA-TMS) are shown in Fig. 1 (b). For each of these PSAs, the peaks of pyrolyzates derived from each acrylic monomers and the peak of internal standard were clearly observed on the pyrograms. As shown in Fig. 1 (b), AA-TMS is undetectable in PSAs #1 and #2, which do not contain AA monomer as a raw material, confirming that only AA compounded in PSAs could be detected as AA-TMS. The peak areas of AA-TMS and Anth-*d*₁₀ were determined from the EIC peak characteristic to each compound. Excellent reproducibility of the peak area ratio of AA-TMS to Anth-*d*₁₀ was obtained, with the RSD of less than 5 % (*n*=5). Calibration curve for AA-TMS obtained using PSAs #1-#4 shows a good linearity with a determination coefficient (*R*²) of 0.9999, as shown in Fig. 2. Using the calibration curve, AA in a 50 µg commercial PSA sample was determined to be 2.71 µg. Alternatively, by conventional acid-base titration with KOH using phenolphthalein as an indicator, AA was determined to be 2.53 µg, which is almost consistent with the result obtained by reactive Py-GC/MS. From these results, AA in acrylic PSAs was able to be accurately quantified by reactive Py-GC/MS using the two-step heating TMS derivatization method.

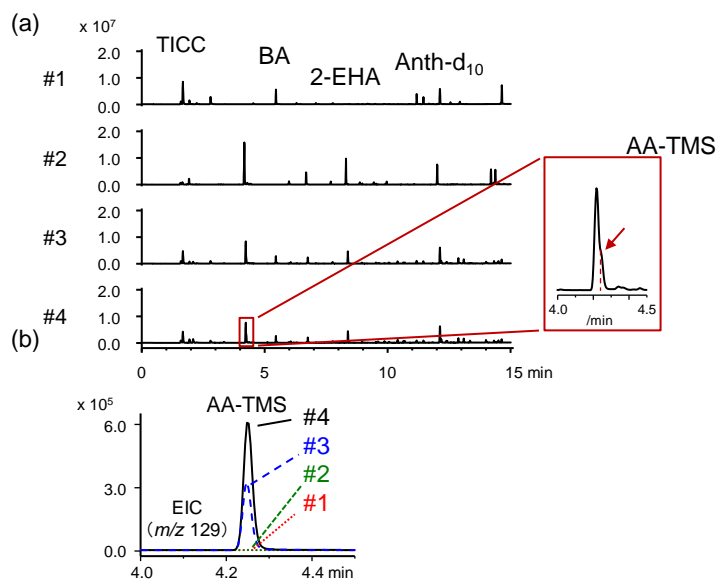


Fig. 1 (a) Pyrograms of PSA samples (TICC), (b) EICs of AA-TMS at *m/z* 129 for four samples.

Furnace temp.: 600 °C, GC Inj. temp.: 300 °C, Column flow rate: 1 mL/min, Split ratio: 1/100, GC oven temp.: 40 (2 min hold) - 320 °C (20 °C/min, 6 min hold), Separation column: UA⁺-5 (5 % diphenyl-95 % dimethylpolysiloxane; L=30 m, i.d.=0.25 mm, df=0.25 µm), Detector: Quadrupole MS, Sample amount: 50 µg.

1) M. Matsueda *et al.*, *J. Anal. Appl. Pyrolysis* 175 (2023) 106170.

Table 1 Composition of model acrylic PSAs.

Sample	Composition ratio (wt %)		
	BA	2-EHA	AA
#1	100.0	0.0	0.0
#2	0.0	100.0	0.0
#3	47.5	47.5	5.0
#4	45.0	45.0	10.0

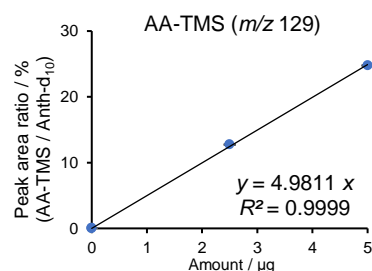


Fig. 2 Calibration curve for AA-TMS prepared by using the peak area ratio to Anth-*d*₁₀ (IS, *m/z* 188).

Keywords : Reactive pyrolysis, Pressure-sensitive adhesives, Pretreatment

Products used : Multi-Shot Pyrolyzer, Auto-Shot Sampler, UA⁺-5, Vent-free GC/MS adapter, F-Search

Applications : Quality assurance, Material analysis

Related technical notes : [PYA2-035E](#)

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Phone: (81)24-935-5100 Fax: (81)24-935-5102

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