Simple Quantitative Analysis of A Cationic Polymer with A Quaternary Ammonium Salt by Py-GC/MS Technique

[Background] Various cationic polymers with a quaternary ammonium salt are used as dye fixing agents and antimicrobials in textile, paper, and film industries. Because the amount of ammonium salt added to the products greatly influence the product performance, the quantitative analysis of the ammonium salt is very important. However, comsssplicated pretreatments such as solvent extractions of interfering species are usually required, because coexisting organic compounds may interfere the analysis. Therefore, the development of a simpler analytical methodology has long been desired. Here, a simple quantitative analysis of a quaternary ammonium salt by Py-GC/MS requiring no pretreatments is described.

[Experimental] A quaternary ammonium salt, polyquaternium-4 (PQ-4) with a chemical structure shown in Fig. 1, was used for this study. For the EGA-MS system used, a pyrolyzer (Frontier Lab. Ltd.) attached to the GC injection port, was connected to a quadrupole MS through a deactivated stainless-steel transfer capillary (Frontier Lab. Ltd.). In the thermal desorption-MS, a separation column was used in place of a transfer capillary. Also at the head of the separation column, MicroJet Cryo-Trap (Frontier Lab Ltd.) was attached to collect volatile components from the sample. They were later analyzed by GC/MS.

[Results] An EGA curve for PQ-4 is shown in Fig. 2. As shown in the blowup of 250~300ºC region, the formation of chloromethane was observed. Thus, the quantitative analysis of PQ-4 was attempted by the amount of chloromethane formed. Fig. 3 shows the chromatogram of the thermally desorbed components of PQ-4 between 100~340ºC obtained by GC/MS. At the retention time of about 1.5min, a peak due to chloromethane is markedly observed. This was repeated 8 times and the reproducibility of the peak area of chloromethane is shown in Table 1. The relative standard deviation was found to be 3.44% and this demonstrates that this is valid quantitative analytical method for PQ-4.

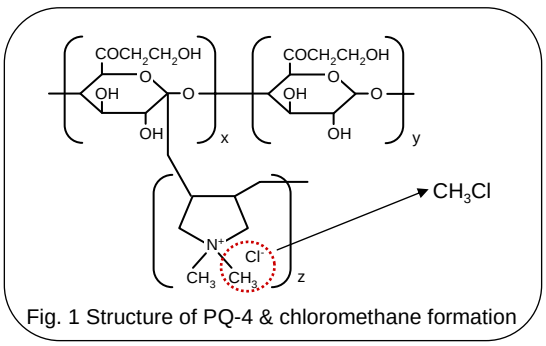

Fig. 1 Structure of PQ-4 & chloromethane formation

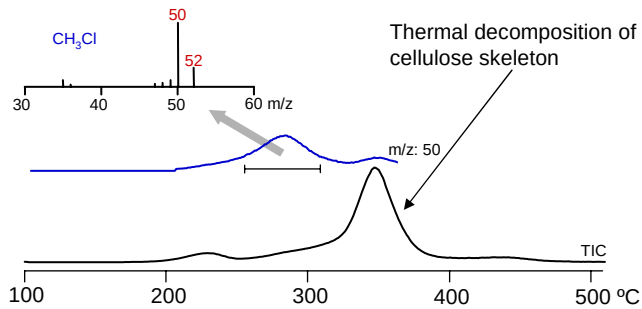

Fig. 2 EGA curve for PQ-4

Pyrolysus temp.:100-500ºC (20ºC/min), carrier gas : He 50kPa, split ratio : ca.1/50
EGA tube : id 0.15mm, length : 2.5m (UADTM-2.5N), GC oven temp.: 300ºC
Sample size : ca. 0.2mg, MS scan range : 29-600 (m/z), scan rate : 0.2 scan/sec

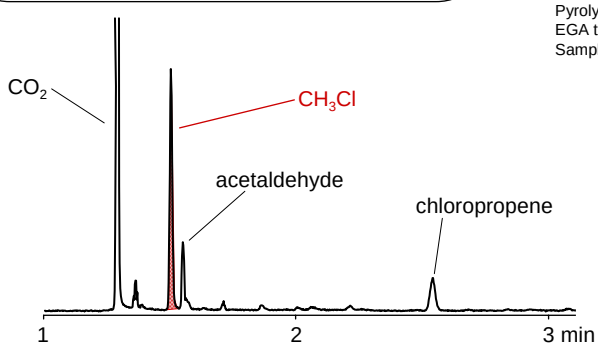

Fig. 3 Chromatogram of PQ-4 obtained by thermal desorption-GC/MS

Pyrolyzer furnace temp.: 100-340ºC (20ºC/min, 2min hold), flow rate : 1ml/min, split ratio : 1/50
Separation column : Ultra ALLOY5+ (5% diphenyl 95% dimethylpolysiloxane, length 30m
id 0.25mm, film thickness : 1.0µm), GC oven temp.: 40ºC (2min hold) - 300ºC (20ºC/min)
Sample size : 5.64µg, MS scan range : 29-600 (m/z), scan rate : 2 scan/sec

Table 1 Reproducibility of chloromethane peak area

n	Peak area (x10^5)
1	5.89
2	5.57
3	6.23
4	6.01
5	6.12
6	5.82
7	5.85
8	5.83
Average	5.92
RSD (%)	**3.44**

Keyword : Cationic polymer, quaternary ammonium salts, polyquaternium, EGA, Thermal desorption

Applications : Paper industry, Film manufacture, General polymer analysis

Related technical notes : PYA1-050E, PYT-004E, PYT-007E, PYT-014E

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