Mass Spectrometry Application Group Mass Spectrometry Business Unit JEOL Ltd.

No.089

Analysis of advanced materials by FD/FI using "AccuTOF GC" Part I

~ analysis of polymer building blocks with isocyanate and pilazole groups ~

Introduction

Field Ionization (FI) is a soft ionization method which ionizes analytes by electron tunneling from analyte molecules to a solid surface (emitter) in a high electric field. Vaporized analyte molecules are introduced to the proximity of the emitter.

We have analyzed functional monomers (building blocks for functional polymers) by GC/EI and GC/FI methods and compared the obtained mass spectra.

Methods

Samples: functional monomers with isocyanate and pilazole groups (Showa Denko K.K.)

(1) Karenz AOI® $(C_6H_7NO_3)$

$$\begin{array}{c} CH_3 \\ H_2C \stackrel{|}{=\!\!\!=\!\!\!=} C - C - OCH_2CH_2 - NCO \\ \parallel \\ O \end{array}$$

(2) Karenz MOI® (C₇H₉NO₃)

$$\begin{array}{c} O \\ \parallel \\ H_2C=CH-C-O-CH_2 \\ C \\ H_2C=CH-C-O-CH_2 \\ \parallel \\ O \end{array} NCO$$

(3) Karenz BEI® $(C_{11}H_{13}NO_5)$

$$H_2C = \begin{matrix} CH_3 \\ -C - C - CCH_2CH_2 - N - C - N \end{matrix}$$

(4) Karenz MOI-BP® (C₁₂H₁₇N₃O₃)

GC conditions

Column: ZB-5ms, 30 m x 0.25 mm, film thickness: 0.25 µm Carrier gas: Helium, 1.0 mL/min (constant flow rate mode) 40 °C (2 min) \rightarrow 30 °C/min \rightarrow 280 °C (12 min) Oven:

MS conditions

Mass spectrometer: JMS-T100GC "AccuTOF GC"

Ionization mode: EI: Electron energy: 70 eV, Ionization current: 300 µA

> FI: Cathode potential: -10 kV, Emitter current: 0 mA

Acquired mass range: m/z 35 - 550

Spectral recording interval: 0.4 sec

Results and Discussion

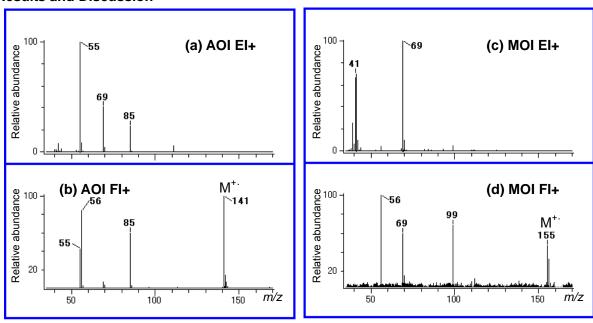


Fig. 1 Mass spectra of Karenz AOI®; (a) EI+, (b) FI+ Mass spectra of Karenz MOI®; (c) EI+, (d) FI+

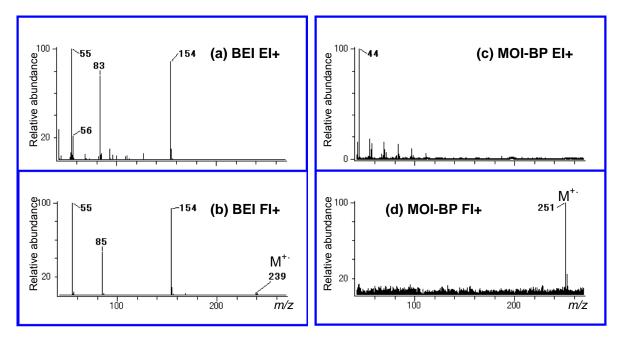


Fig. 2 Mass spectra of Karenz BEI®; (a) EI+, (b) FI+

Mass spectra of Karenz MOI-BP®; (c) EI+,(d) FI+

Karenz AOI®, MOI®, BEI® have isocyanate groups, and MOI-BP® has a pyrazole group. Observation of molecular ions is difficult for these types of compounds. (Refer (a) and (c) in Figs. 1 and 2 respectively). It was possible to observe molecular ions from all samples by using FI method. FI method was found suitable for the analysis of highly reactive compounds such as the ones above.

Acknowledgement

We acknowledge the generous offer of the samples by Fine Chemicals Group, Specialty Chemicals Department, Chemicals Division, SHOWA DENKO K.K.