Mass Spectrometry Application Group Mass Spectrometry Business Unit JEOL Ltd.

No.091

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

~ analysis of organic borates, photoinitiators for polymerization ~

Introduction

Field desorption (FD) is an ionization method utilizing electron tunneling effect in a high electric field near the emitter surface or whisker tip. Sample is applied on the emitter and heated by applying an electric current through the emitter for desorption and ionization.

FD has been used to analyze nonvolatile compounds, polymers, etc. as soft ionization method, which yields intact molecular ions with very few fragment ions in most cases.

We have analyzed some quaternary borate ammonium salts that are designed as photoinitiators for the polymerization of functional polymers by FD.

Methods

Samples

Quaternary borate ammonium salts (Showa Denko K.K.)

$$\begin{bmatrix} n-C_4H_9 \\ \\ m-C_4H_9 \end{bmatrix} N-m-C_4H_9$$

$$n-C_4H_9$$

 $(1) \ \mathsf{P3B}([\mathsf{C}_{16}\mathsf{H}_{36}\mathsf{N}]^{\!+}\![\mathsf{C}_{22}\mathsf{H}_{24}\mathsf{B}]^{-})$

$$\begin{bmatrix} n-C_4H_9 & & \\ \oplus N & n-C_4H_9 \\ & n-C_4H_9 \end{bmatrix} \qquad n-C_4H_9 \qquad b$$

(2) BP3B($[C_{16}H_{36}N]^{+}[C_{34}H_{48}B]^{-}$)

MS conditions

Mass spectrometer: JMS-T100GC "AccuTOF GC"

Acquired mass range: m/z 30 - 900

Spectral recording interval: 0.3 sec

Ionization mode: FD+

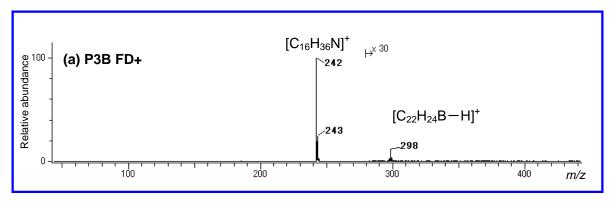
Cathode potential: -10 kV

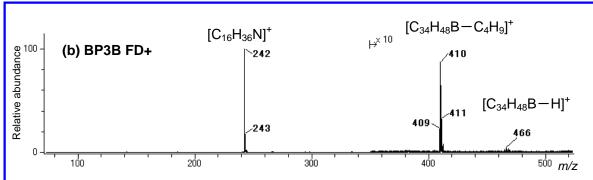
Emitter current program:

 $0 \text{ mA} \rightarrow 51.2 \text{ mA/min} \rightarrow 40 \text{ mA}$

(3) $N3B([C_{16}H_{36}N]^{+}[C_{34}H_{30}B]^{-})$

Results and discussion





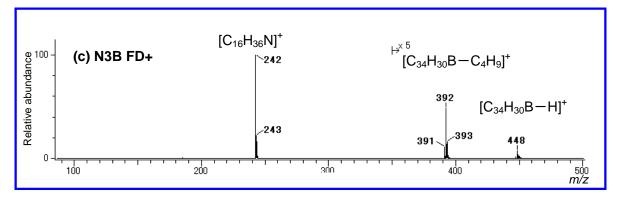


Fig. 1 FD+ mass spectra of organic borate ammonium salts

Molecular ions $[(Anion)(Cation)]^{+-}$ were not detected for all three quaternary borate ammonium salts. The quaternary ammonium cation at m/z 242 ($[C_{16}H_{36}N]^{+}$) was observed as the base peak for all samples. The ions that correspond to $[(Anion) - H]^{+}$ were observed for all samples, though they are relatively weak. For BP3B and N3B, ions that correspond to $[(Anion) - C_4H_9]^{+}$ at m/z 401 and at m/z 392 respectively, were also observed.

With FD, the cation part of salts can be clearly detected. For the quaternary borate salts analyzed here, we found that the anion part of the salts can be detected as de-protonated cations.

Acknowledgement

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