

JMS-T100GC Application Data

Analysis of polydimethylsiloxanes by GC/TOFMS

Introduction

Three polydimethylsiloxane compounds; octamethylcyclotetrasiloxane (sample No. 1), decamethylcyclopentasiloxane (No. 2) and octa(dimethylsiloxy)silsesquioxane (No. 3), were analyzed using JMS-T100GC. The samples No. 1 and No. 2 were relatively small molecules with molecular weights of 296 and 370 respectively and could be introduced through the mass reference sample inlet (reservoir.) They can be used as internal mass references for accurate mass measurement. The sample No. 3 has a molecular weight of 1,016 and was difficult to be introduced through the reservoir. Sample No. 3 was analyzed by GC/MS.

Methods

Samples

1. Octamethylcyclotetrasiloxane, $C_8H_{24}O_4Si_4$, Neat
2. Decamethylcyclopentasiloxane, $C_{10}H_{30}O_5Si_5$, Neat
3. Octa(dimethylsiloxy)silsesquioxane, $C_{16}H_{56}O_{20}Si_{16}$, 2 mg/ml in acetone

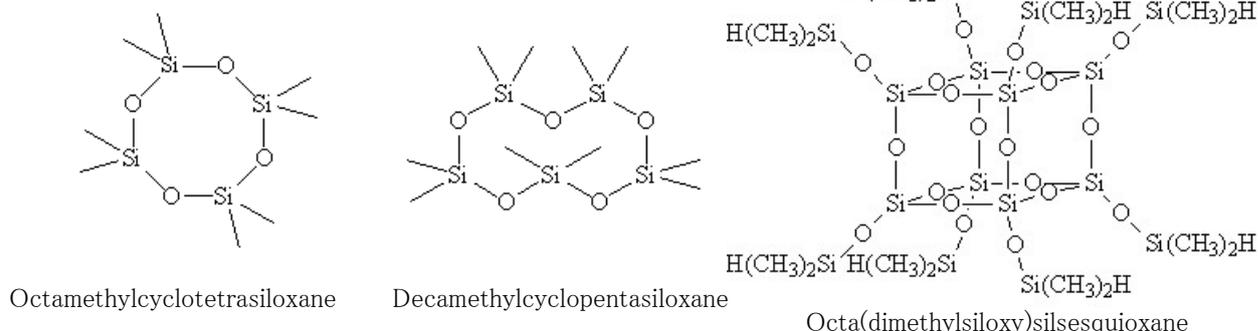


Fig.1 Structural formulae of the samples.

GC conditions

Column:	ZB-5ms, 30 m x 0.25 mm, 0.25 μ m
Injector:	320 $^{\circ}$ C, 1 mL/min (constant flow mode,) split mode (50:1)
Oven:	50 $^{\circ}$ C (1 min) \rightarrow 20 $^{\circ}$ C/min \rightarrow 320 $^{\circ}$ C (5.5 min)

MS conditions

Mass spectrometer:	JMS-T100GC "AccuTOF GC"
EI ionization:	Electron energy: 70 eV Ionization current: 300 μ A
FI ionization:	Cathode potential: -10 kV Emitter current: 7 mA for 20 msec between spectra
Temperatures:	Ion source: 280 $^{\circ}$ C GC-ITF: 280 $^{\circ}$ C Reservoir: 100 $^{\circ}$ C
Acquired mass range:	m/z 35 – 1,400
Spectral recording interval:	0.4 sec

Results and discussion

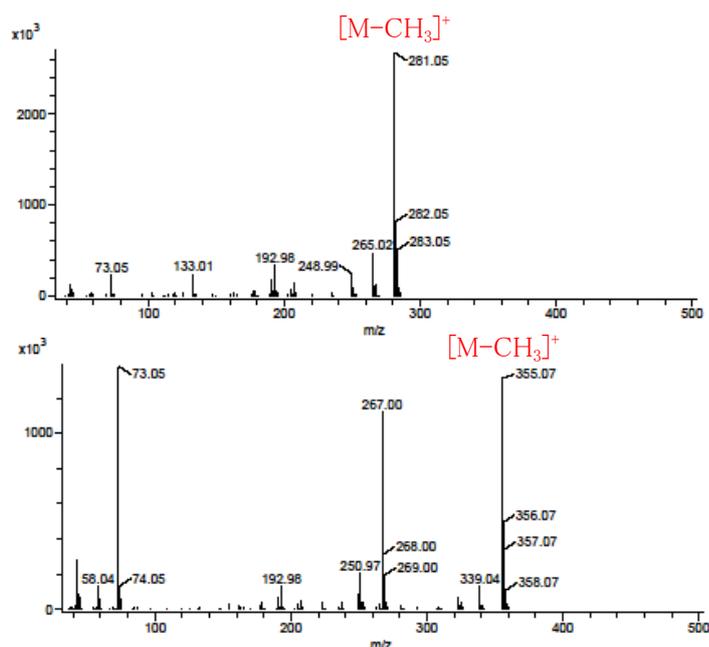


Fig.2 Mass spectra of Octamethylcyclotetrasiloxane (upper) and Decamethylcyclopentasiloxane (lower) by EI.

Table1 Information of calculated exact mass for each ions .

	Calculated exact mass	Fomula
Octamethylcyclo tetrasiloxane	73.04735	C ₃ H ₉ Si
	248.98909	C ₅ H ₁₃ O ₄ Si ₄
	265.02039	C ₆ H ₁₇ O ₄ Si ₄
	281.05169	C ₇ H ₂₁ O ₄ Si ₄
Decamethylcyclo pentasiloxane	73.04735	C ₃ H ₉ Si
	266.99965	C ₅ H ₁₅ O ₅ Si ₄
	355.07048	C ₉ H ₂₇ O ₅ Si ₅
Octa(dimethylsiloxy) silsesquioxane	1000.94384	C ₁₅ H ₅₃ O ₂₀ Si ₁₆
	1014.95949	C ₁₆ H ₅₅ O ₂₀ Si ₁₆

The acquired mass spectra for sample 1 and 2 are shown in Fig. 2. For both compounds, the base peaks were those of $[M-CH_3]^+$ ions, whereas the molecular ions were not detected. The major peaks, as documented in Table 1, can be used as internal mass references for accurate mass measurement. If these compounds are analyzed by field ionization (FI,) the base peaks would be those of $[M-CH_3]^+$ ions; virtually no molecular ions or fragment ions would be detected (cf. MS Tips No. 96.)

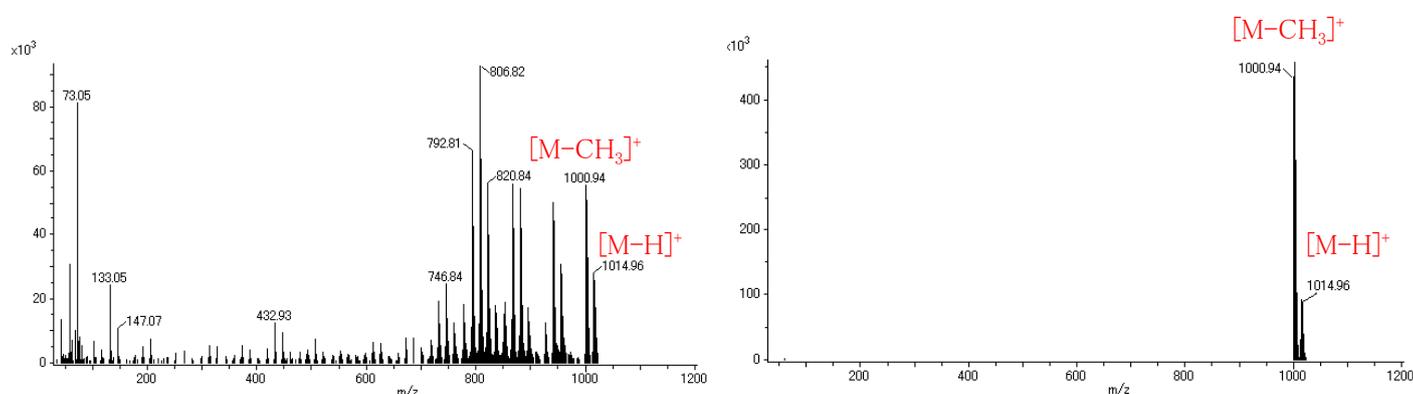


Fig.3 Mass spectrum of Octa(dimethylsiloxy)silsesquioxane by EI (left) and FI (right).

The mass spectrum of octa(dimethylsiloxy)silsesquioxane is shown in Fig. 3. Characteristic ions such as $[M-H]^+$ at m/z 1014.96 and $[M-CH_3]^+$ at m/z 1000.94 were observed in both electron ionization (EI) and FI. In EI, fragment ions that are common to sample 1 and 2, including that at m/z 73.05, were detected whereas no fragment ions other than those at m/z 1014.96 and m/z 1000.94 were detected in FI. Polydimethylsiloxanes are found to generate strong $[M-CH_3]^+$ ions in both EI and FI but detection of the molecular ions are found to be difficult even in FI.

Acknowledgement

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