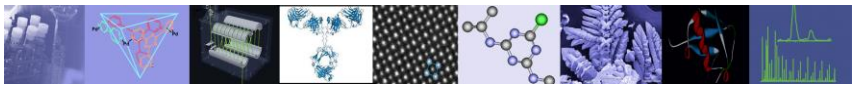

Semi-quantitative Analysis of Low-molecular-weight Cyclic Siloxane in Silicone Rubber via Pyrolysis GC/MS

Product: JMS-Q1500GC GC/MS System

Introduction

Silicone rubber is made from low-molecular-weight (LMW) cyclic siloxane. Most LMW cyclic siloxane is used up during the polymerization process, and the residual cyclic siloxane is removed by subsequent heating and depressurization steps. Generally, the residual level of cyclic siloxane is <3% in silicone rubber, but in the field of electronic equipment manufacturing, residual cyclic siloxane levels must be < 1% in order to avoid contact failure of relays, connectors, etc. due to gases evolved by LMW cyclic siloxane. In this application note, we show semi-quantitative analysis results of LMW cyclic siloxane in silicone rubber that was analyzed using the EGA/PY-3030D pyrolyzer (Py) (Frontier Laboratories, Ltd.) and the gas chromatography–quadrupole mass-spectrometer (GC/QMS) instrument JMS-Q1500GC (JEOL).

Experiment

A silicone rubber septum for a crimp vial was used as a measurement sample, which was weighed at 1 mg. Decamethylcyclopentasiloxane (Si D5) 1000 ng was used as a standard for quantification. Si D5 1000 ng was taken from 10 µL of Si D5 100ng/ µL methanol solution using a micro-syringe. Table 1 shows the measurement conditions.

Table 1. TG-MS measurement conditions.

Pyrolyser	
Furnace temp.	80˚C → 20˚C/min → 350˚C (1 min)
GC	
GC column	ZB-5MSI (Phenomenex, Inc.), 30m x 0.25 mm, 0.25µm
GC inlet temp.	300˚C
Oven temp.	40˚C → 10˚C/min → 300˚C (15 min)
Inlet mode	Split 10:1
Carrier gas	He, 1 mL/min

MS	
Ion source temp.	250˚C
Interface temp.	300˚C
Ionization mode	EI+: 70 eV, 50µA
Relative EM voltage	+200V
Measurement mode	SCAN
Scan range	*m/z* 50 – 1000

Results

Figure 1 shows the total ion current chromatograms (TICCs) for the silicone rubber septum sample (top) and Si D5 standard (bottom). For simple quantification, the sum chromatographic-area values of all LMW cyclic siloxane peaks in the sample were compared with the area value of Si D5 in the standard. As a result, the sample included 0.6 weight percentage (wt%) of Si D3–D10 and 2.0 wt% of Si D11–D20. Since the sample's quantitative value is larger than the residual value required by electrical and electronic equipment manufacturers, the sample is not of high-enough quality for use in electrical and electronic equipment.

These results show that the Py–GC/QMS system is a useful tool for quantitative analysis of impurities and/or additives in polymers.

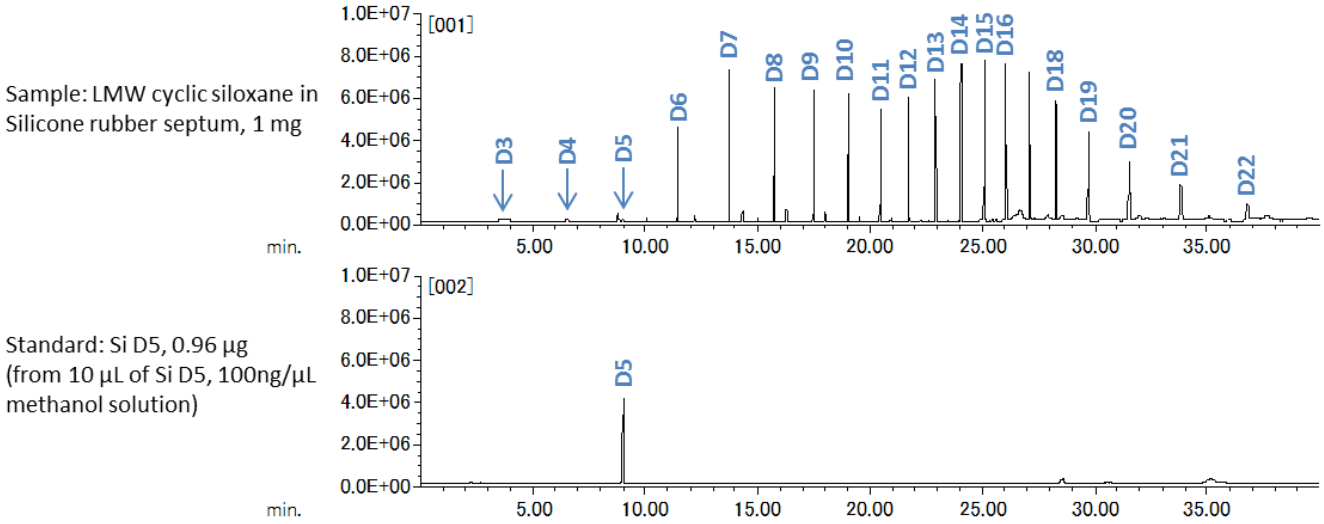

Figure 1. TICCs.

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