JEOL MS Data Sheet

Mass Spectrometry Application Group Mass Spectrometry Business Unit JEOL Ltd.

No. 098

Degradation of rotary vacuum pump oil determined by field desorption (FD) - TOFMS and group-type analysis software

Introduction

Field desorption (FD) is an ionization method in which an electron of the analyte molecule is extracted by tunneling effect under very strong electric field at the surface or at the tips of the "whiskers" grown from the emitter. The analyte is applied as a thin film directly to the emitter and heated by passing a current through the emitter.

FD has been used for the analysis of nonvolatile compounds, synthetic polymers, etc., as a soft ionization method to produce molecular ions with little or no fragmentations.

New and used oil for rotary vane vacuum pump (RP hereafter) were analyzed and the change in their compositions was determined by performing group-type analysis on the FD mass spectra.

Methods

Samples

RP oil (new and used)

MS conditions

Mass spectrometer: JMS-T100GC "AccuTOF GC" Ionization mode: FD(+) Cathode potential: -10 kV Emitter current: $0 \text{ mA} \rightarrow 51.2 \text{ mA/min} \rightarrow 40 \text{ mA}$ Acquired mass range: m/z 35 - 1,600Spectral recording interval: 0.5 sec



Fig. 1 RP oil (left: used, right: new)

Results and discussion

The acquired FD mass spectra are shown below.



Fig. 2 Acquired FD mass spectra

As shown in Fig. 2, typical FD mass spectra for hydrocarbon mixture were obtained. Since the difference was rather subtle (more peaks were observed for the new oil at around m/z 500,) group-type analysis Copyright © 2007 JEOL Ltd.

was performed to determine the differences in their composition. The results are shown below.

Series								Percent
Label	Mn	Mw	Mz	PD	DPn	DPw	DPz	Series
Total/Average	453.82	483.97	531.27	1.07	25.57	27.73	31.10	100.00
C_nH_{2n+2}	447.44	479.98	533.06	1.07	24.78	27.10	30.89	15.47
C_nH_{2n}	435.82	462.47	504.05	1.06	24.10	26.00	28.96	23.19
C_nH_{2n-2}	451.28	482.02	530.39	1.07	25.34	27.54	30.99	16.70
C_nH_{2n-4}	464.22	498.14	551.19	1.07	26.41	28.83	32.61	12.69
C_nH_{2n-6}	459.36	486.90	529.76	1.06	26.21	28.17	31.23	18.90
C_nH_{2n-8}	478.50	511.38	561.52	1.07	27.72	30.06	33.64	13.06

Table 1 Type analysis result of new RP oil

Table 2 Type analysis result of used RP oil

Series								Percent
Label	Mn	Mw	Mz	PD	DPn	DPw	DPz	Series
Total/Average	446.82	483.77	546.99	1.08	25.05	27.68	32.20	100.00
C_nH_{2n+2}	434.74	473.54	544.08	1.09	23.88	26.64	31.68	16.76
C_nH_{2n}	425.93	455.81	507.39	1.07	23.39	25.52	29.20	26.43
C_nH_{2n-2}	444.90	481.25	543.39	1.08	24.89	27.48	31.91	17.57
C_nH_{2n-4}	461.91	506.41	581.41	1.10	26.24	29.42	34.77	12.03
C_nH_{2n-6}	458.94	494.97	555.72	1.08	26.18	28.75	33.08	16.65
C_nH_{2n-8}	485.15	530.63	603.67	1.09	28.19	31.44	36.65	10.58

Hydrocarbon types with different degrees of unsaturation were used. The hydrocarbon types used were C_nH_{2n+2} , C_nH_{2n} , C_nH_{2n-2} , C_nH_{2n-4} , C_nH_{2n-6} , and C_nH_{2n-8} . The difference in hydrocarbon compositions is shown below.

As shown in Table 3, fraction of highly unsaturated hydrocarbons, i.e., C_nH_{2n-4} , C_nH_{2n-6} , and C_nH_{2n-8} , decreased while fraction of moderately unsaturated and saturated hydrocarbons, i.e., C_nH_{2n-2} , C_nH_{2n} , and C_nH_{2n+2} increased in used RP oil. This suggests either opening of ring structures or hydrogenation of double bonds or triple bonds in C_nH_{2n-4} , C_nH_{2n-6} , and C_nH_{2n-8} .

	New		Used	
Series	Percent		Percent	
Label	Series		Series	
Total/Average	100.00		100.00	
C_nH2_{n+2}	15.47	– increase \rightarrow	16.76	
C_nH_{2n}	23.19	– increase \rightarrow	26.43	
C_nH_{2n-2}	16.70	– increase \rightarrow	17.57	
C_nH_{2n-4}	12.69	– decrease \rightarrow	12.03	
C_nH_{2n-6}	18.90	– decrease \rightarrow	16.65	
C _n H _{2n-8}	13.06	– decrease \rightarrow	10.58	

Table 3 Difference in hydrocarbon compositions

Another possibility is oxidative degradation

of unsaturated hydrocarbons. The exact masses of an oxidation product and its hydrocarbon isobar are very close and can not be mass-resolved with the analysis conditions used. However, the apparent increase of saturated and moderately unsaturated hydrocarbons and decrease of highly unsaturated hydrocarbons could be partly due to the oxidation products.

The difference in compositions of new and used RP oil was clearly revealed by group-type analysis.