

AccuTOF-GCv Series

Type Analysis of Micro Crystalline Wax (Petroleum Wax) by Using Field Desorption (FD) Ionization

Introduction

Petroleum waxes are a class of hydrocarbons that are solid at room temperature and are classified by the Japan Industrial Standards (JIS K2235) into 3 types: paraffin wax, micro crystalline wax, and petrolatum. A typical micro crystalline wax contains hydrocarbons having a carbon number of 30 to 60 and molecular weights between 500 and 800. In addition to paraffins, these waxes also include large quantities of isoparaffins and cycloparaffins.

Field desorption (FD) is an ionization technique that utilizes the tunneling effect of electrons in the presence of a high electric field. The sample is applied directly onto an FD emitter filament, and then an electric current is applied to the filament to produce a high electric field across the emitter surface (including the whisker tips) to desorb and ionize the samples. As a soft ionization technique that minimizes fragmentation and produces molecular ions, FD has been previously used for analyzing refractory compounds and high molecular weight polymers.

In this work we ionized a micro crystalline wax by using a JMS-T100GC AccuTOF-GC with FD ionization to do a sample type analysis that was based on the mass and intensity of the resulting ions.

Method

Sample: Commercial micro crystalline wax product (10 mg/ml, chloroform solvent)

MS Conditions:

JMS-T100GC AccuTOF GC

Ionization mode: FD(+)

Cathode voltage: -10 kV

Emitter current: 0 mA → 6.4 mA/min → 45 mA

Mass range: m/z 20 to 1600

Recording interval: 3.2 s

Results and Discussion

Saturated hydrocarbon compounds were detected in the mass range under m/z 450, while unsaturated hydrocarbon compounds were detected in the mass range above m/z 450. A type analysis was conducted using the unsaturated hydrocarbon compounds. A total of 6 hydrocarbon compounds were used: C_nH_{2n+2} (S1), C_nH_{2n} (S2), C_nH_{2n-2} (S3), C_nH_{2n-4} (S4), C_nH_{2n-6} (S5), and C_nH_{2n-8} (S6). Table 1 shows the results for each series of hydrocarbons.

The content of C_nH_{2n} was the highest at 24.2% among the 6 hydrocarbons series shown in the table. This hydrocarbon series was followed by C_nH_{2n-6} at 22.5% and C_nH_{2n+2} at 17.3%. The micro crystalline wax contained large amounts of isoparaffins (equal to S1) and cycloparaffins (equal to S2), and the content of ion species in these series tended to be higher than observed for the other hydrocarbon types.

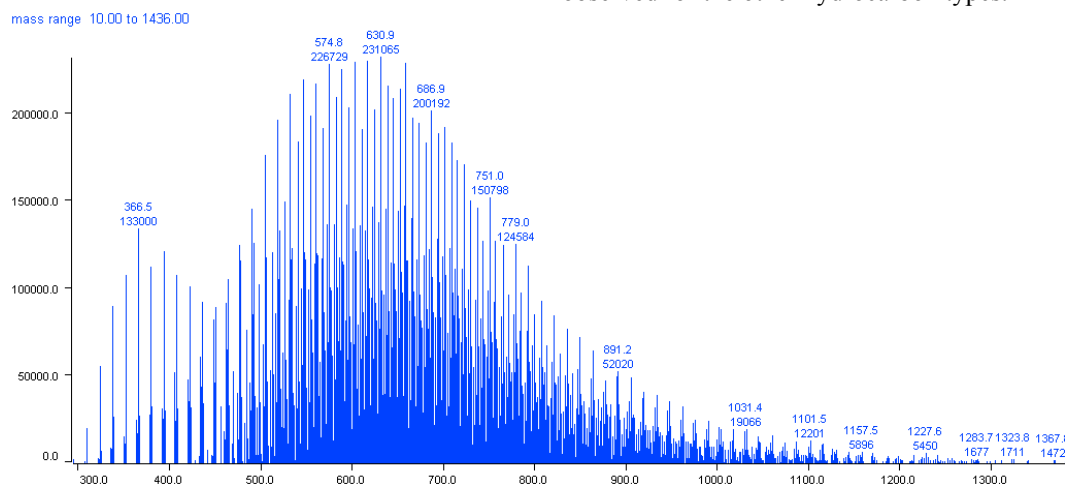
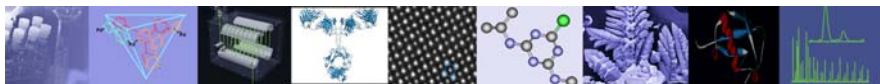


Figure 1. FD mass spectrum of micro crystalline wax



Series Label	M _n	M _w	M _z	PD	DP _n	DP _w	DP _z	Percent Series	Percent Spectrum
Total/Average	657.0	686.9	718.9	1.0	40.1	42.2	44.5	100.0	64.1
S1 C _n H _{2n+2}	571.1	617.3	665.3	1.1	33.6	36.9	40.3	17.3	11.1
S2 C _n H _{2n}	529.7	655.0	680.8	1.0	37.9	39.7	41.6	24.2	15.5
S3 C _n H _{2n-2}	670.5	692.3	715.6	1.0	41.0	42.5	44.2	13.1	8.4
S4 C _n H _{2n-4}	715.6	739.0	764.7	1.0	44.3	46.0	47.8	8.4	5.4
S5 C _n H _{2n-6}	688.3	717.9	751.1	1.0	42.5	44.7	47.0	22.5	14.5
S6 C _n H _{2n-8}	710.0	739.6	772.8	1.0	44.2	46.3	48.7	14.5	9.3

Mn: Number-average molecular weight DP_n: M_n/R
 Mw: Weight-average molecular weight DP_w: M_w/R
 Mz: Z-average molecular weight DP_z: M_z/R
 PD: Polydispersity (R: Mass of repeat unit)

Table 1. Type analysis of microcrystalline wax

Conclusions

These results demonstrate that by using the AccuTOF-GC with FD ionization, a solid wax can be analyzed at room temperature to produce high resolution mass spectra that provide detailed information about the sample, including the hydrocarbon types present in the samples, their average molecular weight information, and their compositional ratios relative to each other.

Acknowledgment

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