JEOL MS Data Sheet

MS Tips

Mass Spectrometry Application Group
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Rapid analysis using inactivated fused silica tube (a.k.a. "guard column") as a sample inlet (1)

Introduction

Average molecular weight is an important reference for evaluating samples with molecular weight distributions, such as crude oils, which are complex mixtures, or synthetic polymers. Almost all the ions observed in field desorption (FD) and field ionization (FI) mass spectra are molecular ions since these are soft ionization methods. Average molecular weight of the sample can be calculated directly from the masses (or "*m/z*") and intensities of all ions observed by FD or FI. By applying group-type analysis, the components can be classified into types based on their functional groups and/or unsaturations. Average molecular weight, polydispersity index, or relative abundance of each type can be obtained.

A diesel fuel was analyzed on JMS-T100GC "AccuTOF GC" with 3 sample introduction methods and the results were compared:

- Capillary GC / FI
- Rapid FD in which the analysis time was shorten by ramping the emitter current much faster than that on conventional analyses on double-focusing mass spectrometers
- Blank tube inlet / FI in which a fused silica tube was used to connect the GC injector and the ion source

Methods

Blank tube inlet / FI

The schematics of the sample inlets are shown in Fig. 1. The specifications of the GC column and the fused silica tube used in the experiments are also shown in Fig. 1.
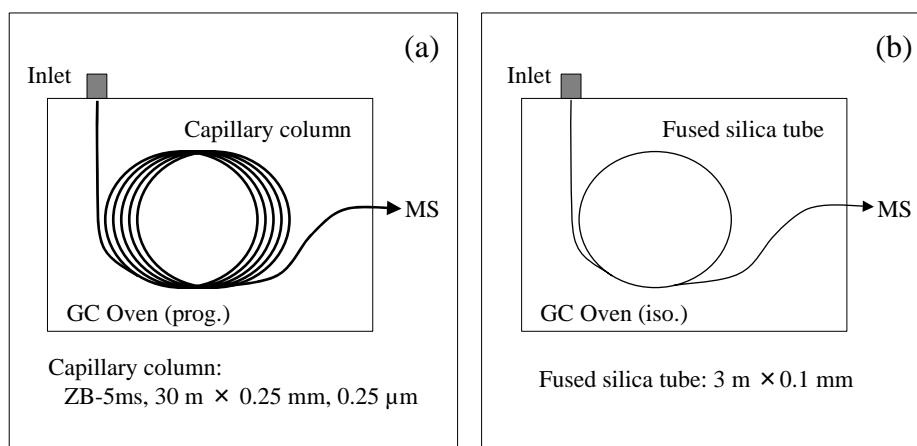

Fig.1 Schematic diagrams of sample introduction system by using GC,
(a) capillary GC/FI. (b) blank tube inlet/FI.

In ordinary GC/MS analysis, a mixture is separated by a GC column and then detected by MS. When

performing group-type analysis from GC/MS data, however, all acquired spectra are summed into a single spectrum. Component separation or individual spectra in the data are not really necessary.

In blank tube inlet / FI, a sample is introduced from the GC injection port through a short fused silica tube ("guard column"), held in the isothermal GC oven, to the ion source. The advantages are:

- Short analysis time
- Loss of low boiling point components are minimized compared with probe based methods, such as FD, DEI, and DCI, in which the sample is put on the probe at ambient pressure. However, low boiling point components are evaporated as the probe is introduced into the vacuum.
- Loss of high boiling point and trace components is minimized compared to the GC/MS method since there are no interaction with a GC stationary phase.
- GC conditions are not critical; no need to worry about separation.
- Lager amount of sample can be injected than in the GC/MS method; no GC column overloading.

Results and discussion

The results are shown on Fig. 2 and 3. The spectra on Fig. 3 were generated by summing the spectra in the time ranges shown by the arrows in Fig. 2.

With GC/FI, the analysis took 25 minutes although each component can be examined in detail due to the separation. FD and blank tube inlet / FI do not provide separation but the analysis times were very short; both within 1 minute.

By comparing the spectra from 3 methods, blank tube inlet / FI detected 1) low boiling point components (n-$C_{18}H_{38}$ (m/z 254) and smaller) that were missing in FD 2) high boiling point and trace components that were missing in GC/FI.
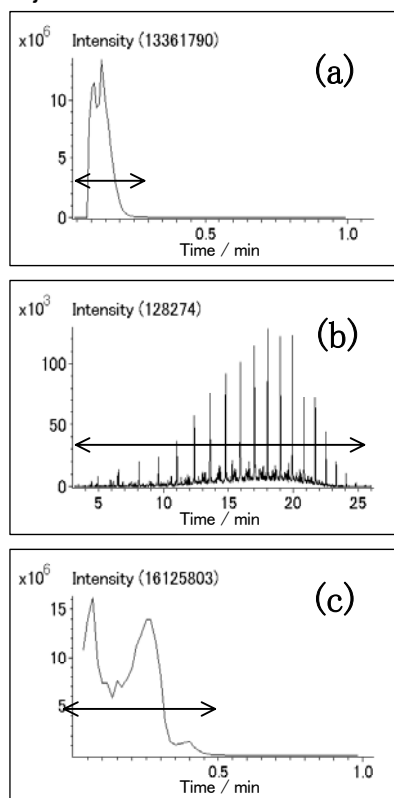

Fig.2 TICs of diesel oil
(a) blank tube inlet/FI,
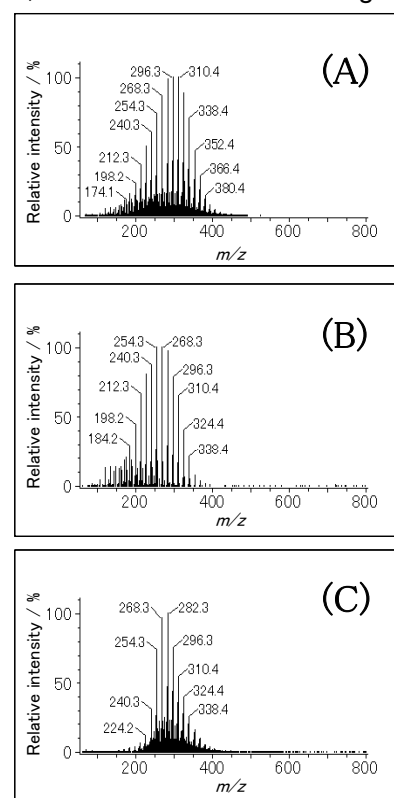(b) capillary GC/FI, (c) FD

Fig.3 Mass spectra of diesel oil
(A) blank tube inlet/FI,
(B) capillary GC/FI, (C) FD

For group-type analysis in which parameters such as relative abundance and average molecular weight for each type are obtained, m/z's and summed intensities of all the detected ions are used; no separation is required. The blank tube inlet / FI method is well suited for the group-type analysis of petrochemicals because of short analysis time, better detectability of low boiling point components than FD, better detectability of high boiling point and trace components than GC/FI. It can handle larger amount of sample injection than GC; well suited with FI whose ionization efficiency is lower than EI.

Reference

M. Ubukata et al., *J. Mass Spectrom. Soc. Jpn.,* **56**, 13-19 (2008).