SHIMADZU

LC-MS Application Data Sheet No. 043

Analysis of Fullerenes using LC-MS

Fullerenes (C_{60}) have been attracting attention ever since their structure including five-membered and six-membered rings was proposed (Fig.1). Many applications are expected for them as semiconductive or superconductive materials, or as materials for new plastices, due to their high electrical conductivity and a high degree of superconductivity when internally doped with metal.

It is known that the MALDI-TOFMS is effective in the analysis of fullerenes. The mass spectrum of a fullerene mixture obtained using the Shimadzu/Kratos AXIMA-CFR is shown in Fig.2. Positive ions created through laser ionization have been detected for C_{60}, C_{70}, C_{78}, and C_{84}.

In LCMS measurements, the column performs a vital role since components are individually separated. Fig.3 shows a comparison of chromatograms obtained using the Develosil RPFULLERENE (made by Nomura Chemical), a column specially developed for fullerene, and a commercially available ODS column. The sample is a mixture of C_{60} and C_{70} (200ng each). A mixed solvent of A: 2-propanol and B: toluene/2-propanol = 90/10 was used for the mobile phase. (1) and (3) show a comparison with the same mobile phase. C30 (triaconthyl group) is chemically bonded in the RPFULLERENE, strengthening the retention of the components. (1) and (2) show a comparison at almost the same retention time. These comparisons prove that the use of RPFULLERENE improves the shape of the peaks (the symmetry coefficient of C_{60} is 1.16 with RPFULLERENE while it is 1.23 with ODS (2).

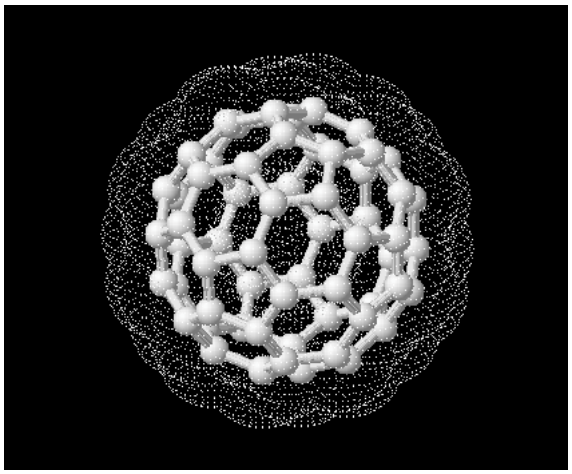

Fig. 1 Structure of C_{60}

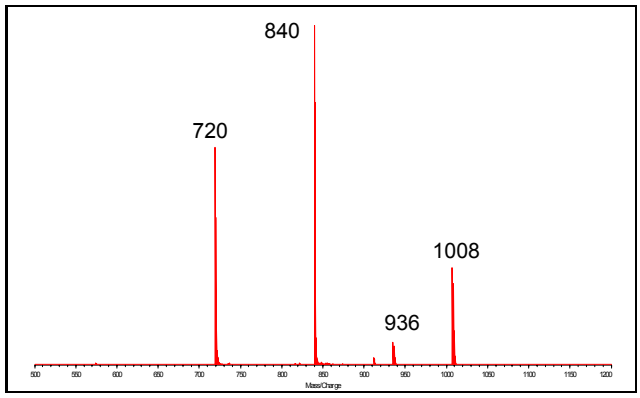

Fig. 2 Mass spectrum of fullerenes (MALDI-TOFMS, positive)

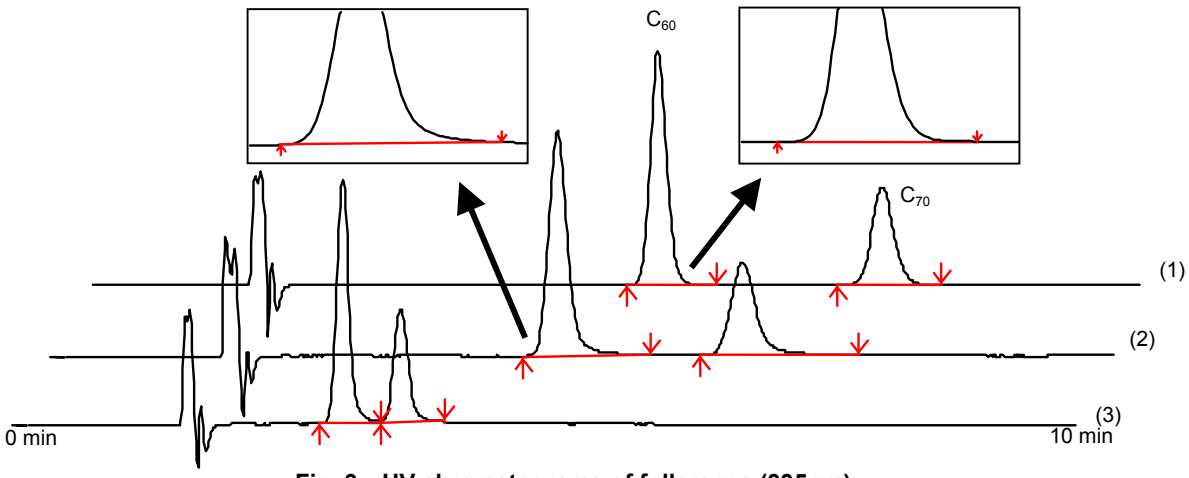

Fig. 3 UV chromatograms of fullerenes (335nm)
(1) RPFULLERENE, A/B = 50/50 (2) ODS, A/B = 65/35
(3) ODS, A/B = 50/50
A : 2-propanol, B : toluene/2-propanol = 90/10

The APCI ionization method was employed in the LC-MS measurement, and the fullerene was detected as a negative ion. C_{60} and C_{70} were detected as ions of m/z 720 and 840 respectively. Fig.4 shows the mass chromatogram and Fig.5 shows the mass spectra.

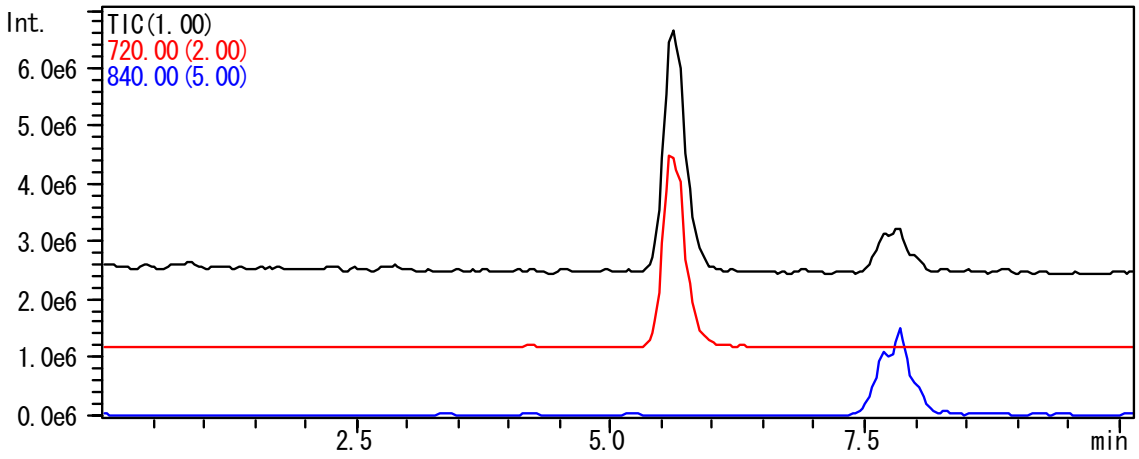

Fig.4 Mass chromatograms of fullerenes

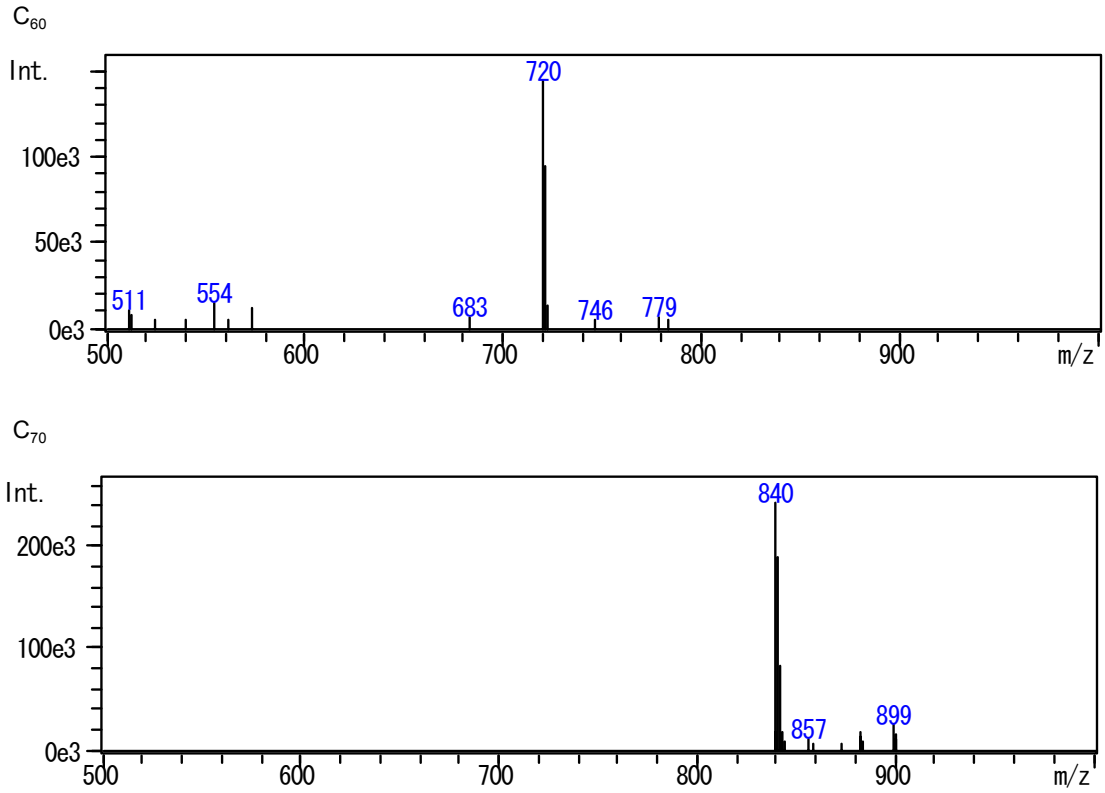

Fig.5 Mass spectra of C_{60} and C_{70}

Table 1 Analytical conditions for LC-MS

Column	: Develosil RPFULLERENE (2.0 mmI.D. x 150 mmL, 5μm, Nomura Chemical)		
Mobile phase	: A : 2-propanol, B : toluene/2-propanol = 90/10 A/B = 50/50		
Flow rate	: 0.2 mL/min		
Injection volume	: 2 μL	**Column temperature**	: 40 degree C
Probe voltage	: -3.0 kV (APCI-Negative mode)	**Probe temperature**	: 400 degree C
Nebulizing gas flow	: 2.5 L/min (Air)		

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