

Application Data Sheet

No.30

GCMS

Gas Chromatograph Mass Spectrometer

Analysis of Chelate Compounds Through Direct Sample Introduction

Chelate compounds are generally refractory compounds composed of ligands and metal ions. Therefore, they are difficult to measure with gas chromatographs. To obtain an electron ionization mass spectrum of refractory compounds requires a pure sample that should be introduced by direct introduction (DI). In this Application Datasheet, a chelate with ring-shaped phthalocyanine ligands, which are cross linked to four phthalic imides by nitrogen atoms, is measured using the DI method.

Instrument

The direct introduction probe is shown in Figure 1 and a diagram of the principle is shown in Figure 2. The sample is dissolved in a solvent and inserted into a glass sample cup placed on the end of the probe. After drying the solvent, the probe is inserted into the mass spectrometer (MS) ion box. The sample is introduced to the ion source box by heating the sample cup with a heater, and then ionized by electrons emitted from a filament.



Fig. 1: Direct Introduction Probe (lower left) and Inserting the Probe into the MS Unit

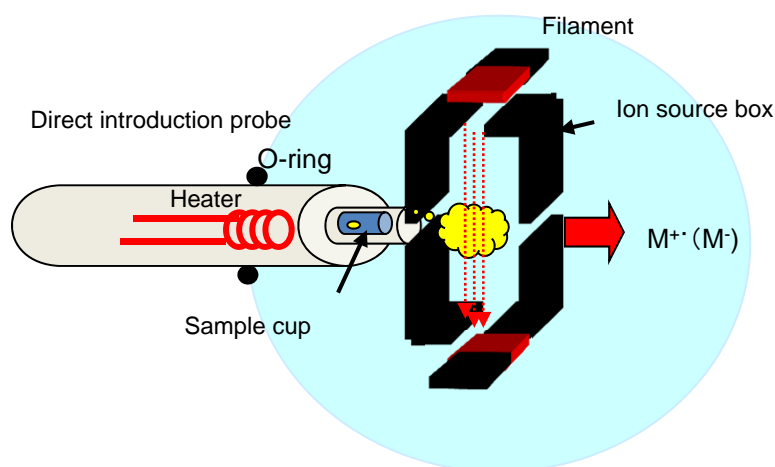


Fig. 2: Principle Behind Direct Sample Introduction

Analysis Conditions

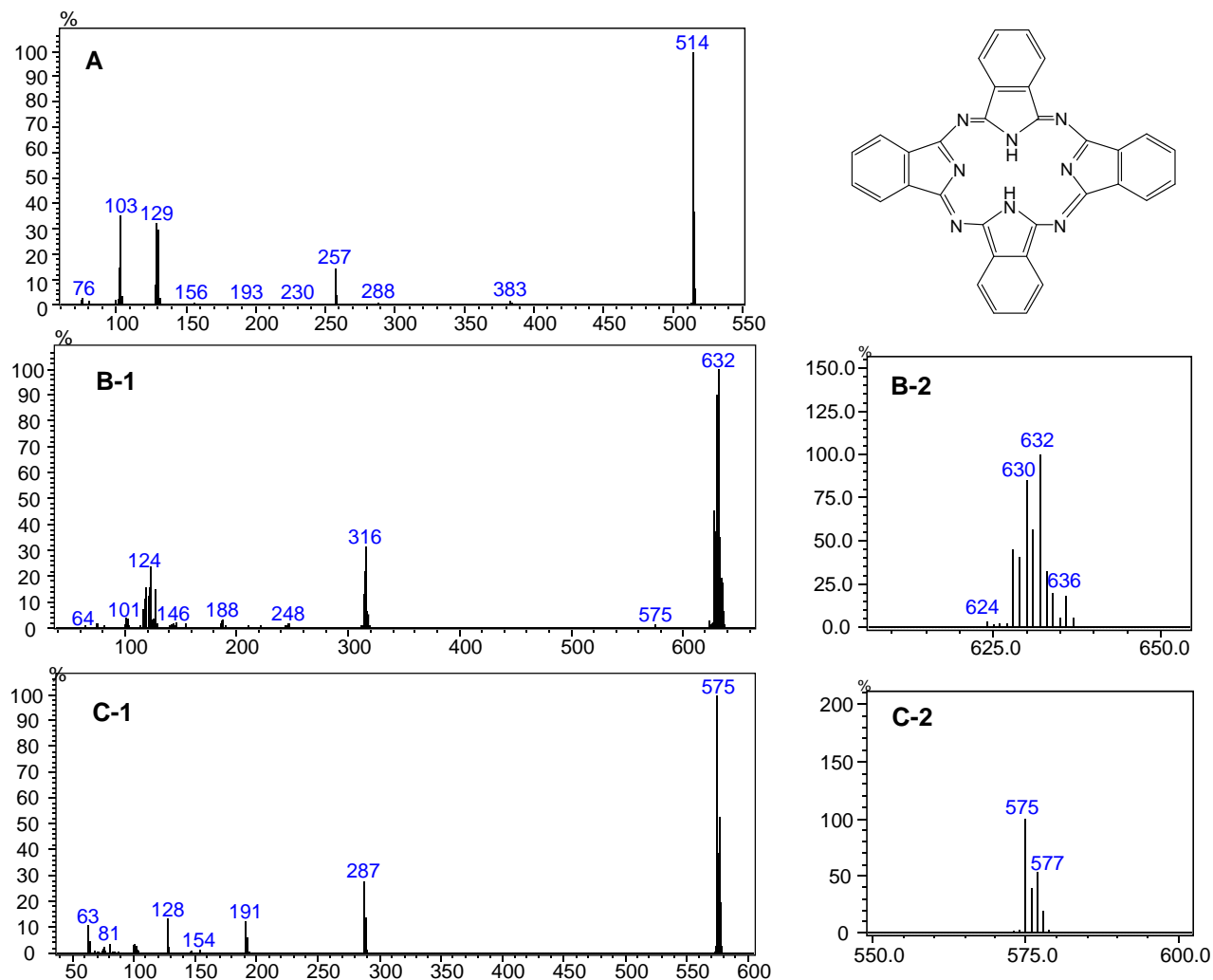
Table 1: Analysis Conditions

GC-MS	: GCMS-QP2010 Series	[MS]	
[DI]		Interface temperature	: 250°C
Sample quantity:	1,000 ng	Measurement mode	: Scan
Injection mode:	Direct Sample Introduction	Mass range	: m/z 50-600
DI temperature:	Room temperature \rightarrow (80 ° C/min) \rightarrow 500 ° C (10 min)	Event time	: 0.5 sec

Results

Figure 3 shows mass spectra obtained from measuring (A) phthalocyanine, (B) tin-chelated phthalocyanine, and (C) copper-chelated phthalocyanine by direct sample introduction. The structural formula for phthalocyanine is shown on the upper right.

In the mass spectrum for (A) phthalocyanine, the m/z molecular ion was detected as the base peak. For tin-chelated phthalocyanine, molecular ions corresponding to tin isotopes ^{116}Sn , ^{118}Sn , and ^{120}Sn were detected at 628, 630, and 632 (B-2), and an isotope pattern characteristic of tin was indicated. Similarly for copper, molecular ions corresponding to ^{63}Cu and ^{65}Cu isotopes were detected at m/z 575 and 577.



Summary

The direct sample introduction method (DI) enables the mass spectra of chelate and other refractory compounds to be obtained quickly and easily.

