

Determination of red phosphorus in phenol resin

[Background] The red phosphorus content in plastic electronic parts has attracted much concern because of the failure risk in electric circuits by exposing red phosphorus to moisture. The determination of red phosphorus can be accomplished either by pyrolysis-GC/MS¹⁾ or by evolved gas analysis (EGA)-MS²⁾, utilizing the tetramer formed during the thermal decomposition of red phosphorus. In this note, the determination of red phosphorus contained in a coil bobbin, which is made of phenol resin, using EGA-MS is described.

[Experimental] A sample was prepared by freeze-grinding the coil bobbin into a powder with a particle size-distribution of 50-100 µm. Commercially available red phosphorus (98% purity) was used to construct a calibration curve. An EGA-MS system consists of a Multi-Shot Pyrolyzer (EGA/PY-3030D) which was directly interfaced to a GC injector to which a quadrupole mass spectrometer was connected using a deactivated metal tube. The determination of red phosphorus was based on the calibration curve ($r^2 = 0.999$) prepared using the peak area of the extracted ion chromatogram (EIC) of the phosphorus tetramer ion (m/z 124) over the temperature zone 400-500°C as indicated by the EGA thermogram.

[Results] An EGA thermogram (TIC) of a coil bobbin sample (1.15 mg) is shown in Fig. 1. A peak at m/z 124 was observed in the average mass spectrum. From the thermogram, an EIC at m/z 124 was obtained. Using the peak area obtained over the 400-500°C zone and the calibration curve, the red phosphorus content was determined to be 0.26 wt% ($n = 3$) and the reproducibility (RSD) was 5.7%, assuming that the interference from the phenol resin was negligible. This value agreed well with 0.23 wt% (RSD = 5.6%, $n = 3$) obtained by pyrolysis-GC/MS¹⁾. As shown above, the determination of red phosphorus was successful using EGA-MS.

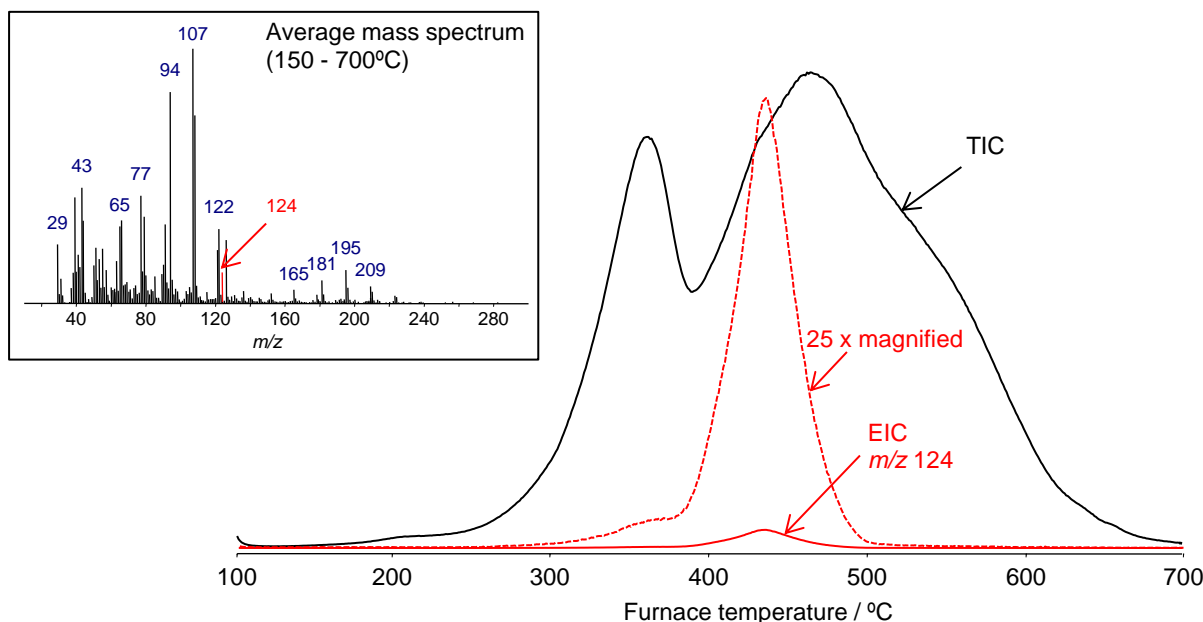


Fig. 1 EGA thermogram and average mass spectrum of a coil bobbin sample

Py furnace temp.: 100 – 700°C (20 °C/min), Split ratio: 1/100, GC oven temp.: 300°C,
 EGA tube: Deactivated metal tube, L = 2.5 m, i.d.= 0.15 mm, Column flow rate: 1 mL/min (He),
 Scan range: m/z 29 – 600
 Sample amount.: 1.15 mg.

1) M. Iida, K. Miyatake, A. Kimura, *Anal. Sci.* 24 (2008) 539-542, 2) Technical note [PYA3-018E](#)

Keywords : Red phosphorus, flame retardant, phenol resin, EGA-MS

Products used : Multi-functional Pyrolyzer, UADTM-2.5N, Vent-free GC/MS adapter

Applications : Quality assurance, receiving inspection

Related technical notes : [PYA3-018E](#)

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