

Analysis of Phthalate Esters Using the Py-Screener (2)

In the RoHS directive (directive on the restriction of the use of certain hazardous substances in electrical and electronic equipment), four phthalate esters: diisobutyl phthalate (DIBP), dibutyl phthalate (DBP), benzyl butyl phthalate (BBP), and bis(2-ethylhexyl)phthalate (DEHP), will be added to the six conventionally limited substances starting in 2019.

Of these, the substances that can be measured with GC-MS are the brominated flame retardants, PBBs and PBDEs, and phthalate esters. The Soxhlet extraction-GC/MS method, while an accurate quantitation method, requires time-consuming pretreatment and uses organic solvents. In contrast, the pyrolysis-GC/MS (Py-GC/MS) method does not require complicated pretreatment and is therefore expected to be used as a screening method. The "Py-Screener" is a screening system for phthalate esters. It consists of resin standard samples containing phthalate esters, a sample preparation sampling kit, and Py-GC/MS analysis files.

This Application Datasheet introduces an example of the analysis of phthalate esters and brominated flame retardants using the Py-Screener.

Analytical Conditions

The conditions registered in the Py-Screener were used as the GC-MS analysis conditions. For the detailed analysis conditions, refer to GC-MS Application Datasheet No.110, "Analysis of Phthalate Esters Using the Py-Screener (1)."

Results

A calibration curve was created using the 1000 mg/kg phthalate ester resin standard sample. KRIS CRM113-03-006, which is sold as a phthalate ester certified standard sample, was then measured and quantified based on this calibration curve. Fig. 1 shows the mass chromatograms obtained. Table 1 shows the results of a comparison of the quantified values and the CRM certified values. The yield using the certified values as reference was in the range of 92.9 % to 109.0 %, so the quantitation results obtained were favorable with a view to screening.

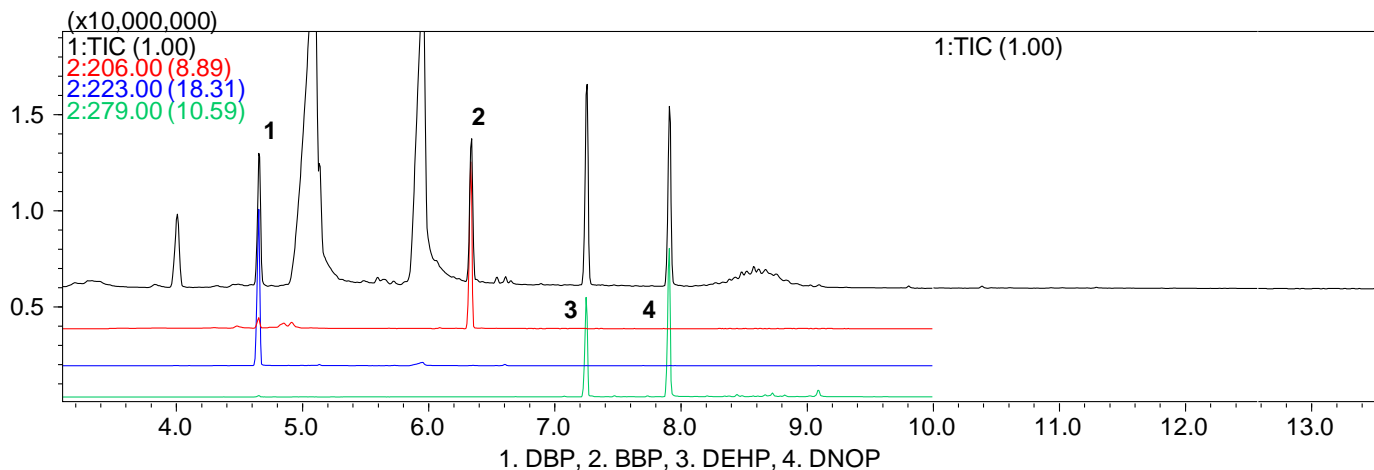


Fig. 1: Mass Chromatograms for the Phthalate Esters Measured in KRIS CRM113-03-006

Table 1: Comparative Results for the Certified Values and the Quantitation Results

	Quantitation Results (mg/kg)	Certified Values (mg/kg)	Yield (%) with the Certified Values as Reference
DBP	1059	972	109.0
BBP	894	962	92.9
DEHP	1015	989	102.6
DNOP	993	967	102.7

Figs. 2 and 3 show the mass chromatograms for a PVC cable and PBT resin measured as testing samples. DBP, DEHP, DINP, and DIDP were detected in the PVC cable, and DEHP and Deca-BDE were detected in the PBT resin. This system can accommodate screening for phthalate esters and brominated flame retardants with a single measurement cycle.

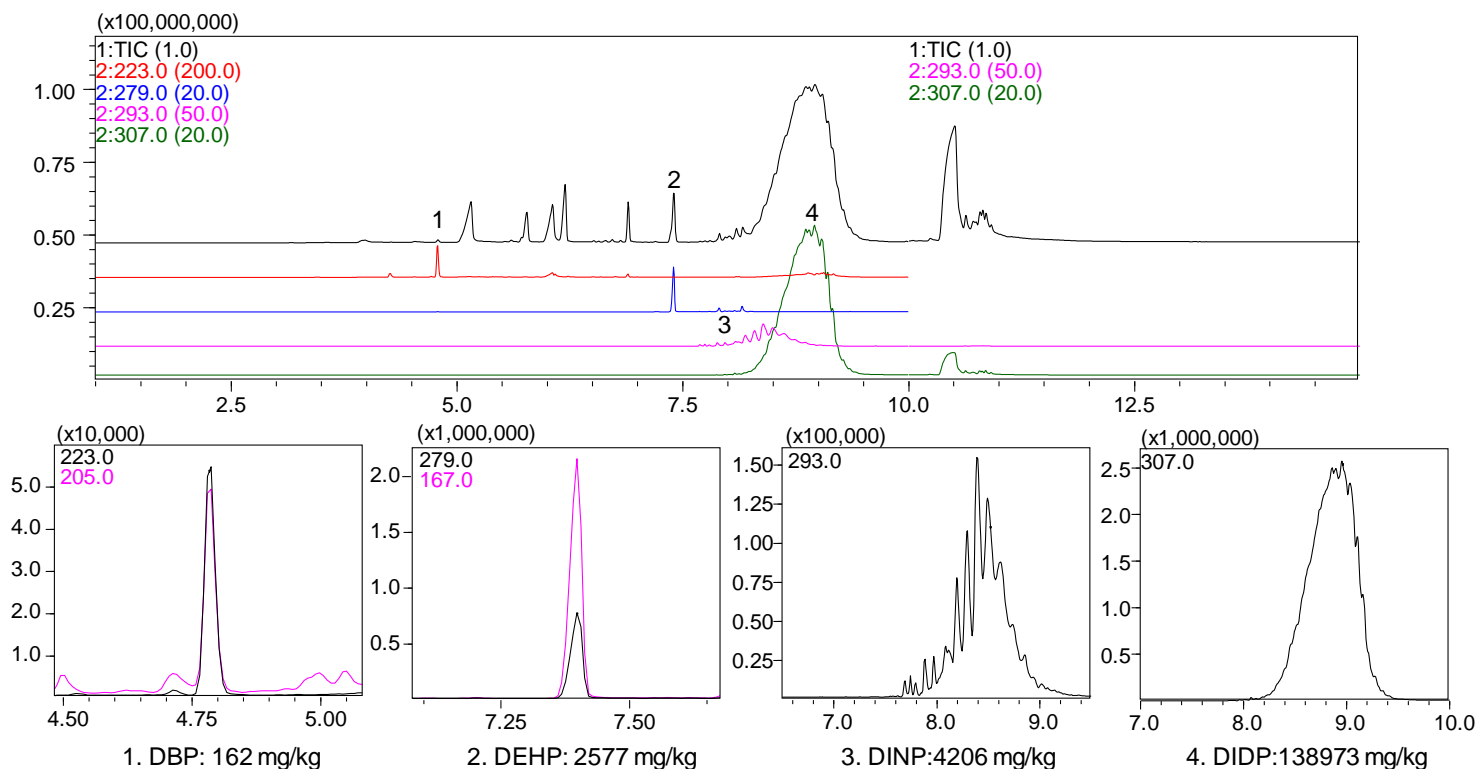


Fig. 2: Mass Chromatograms for Compounds Detected in a PVC Cable

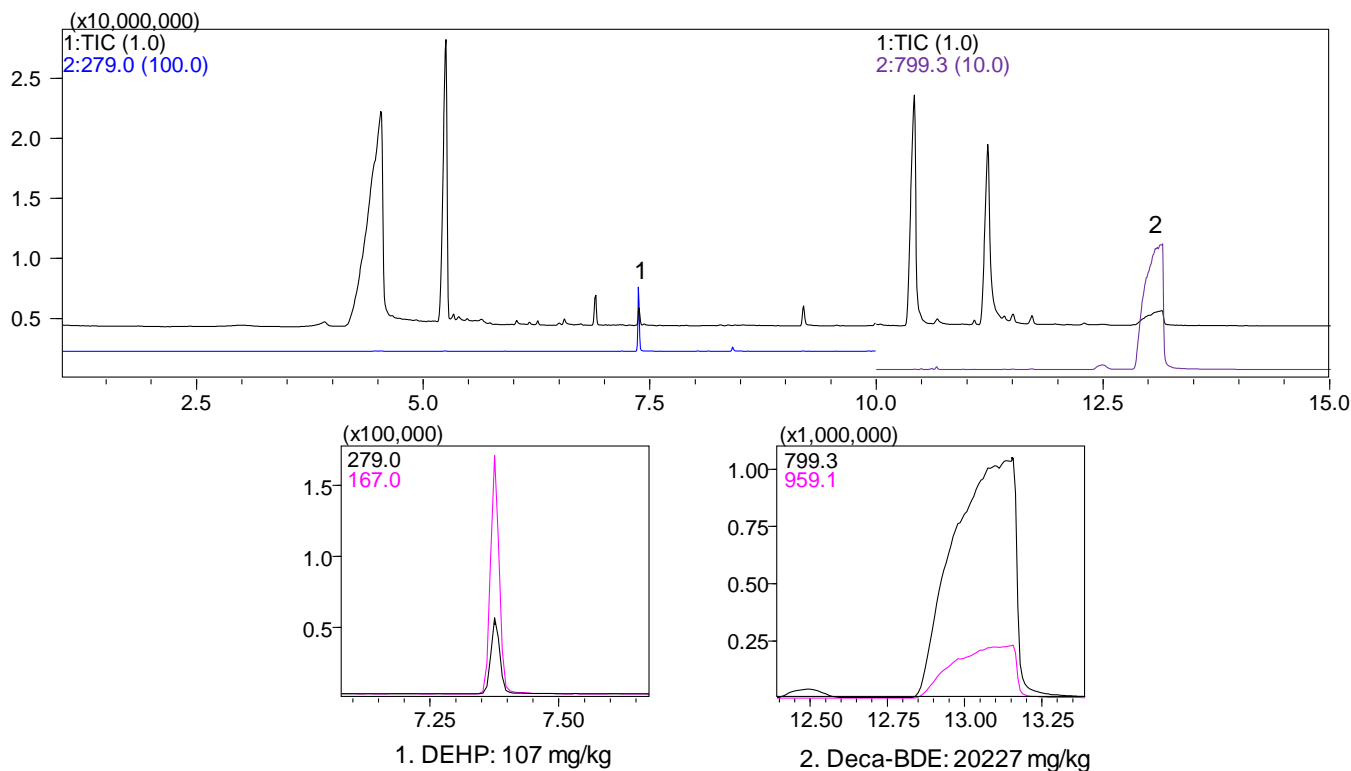


Fig. 3: Mass Chromatograms for Compounds Detected in a PBT Resin