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Application Data Sheet

No.110

GC-MS
Gas Chromatograph Mass Spectrometer

Analysis of Phthalate Esters Using the Py-Screener (1)

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 using Py-GC/MS. It consists of polymer standard samples containing phthalate esters, a sample preparation sampling kit, and Py-GC/MS analysis files.

This Application Datasheet introduces measurements of phthalate ester standard samples using the Py-Screener.

Standard Samples Used in the Py-Screener

The Py-Screener uses the Phthalate Esters Polymer Standards for Py-GC/MS (P/N: S225-31003-91). These polymer standard samples can be prepared easily, without using organic solvents, by using a special sampling toolkit (P/N: PY1-K101 from Frontier Laboratories).

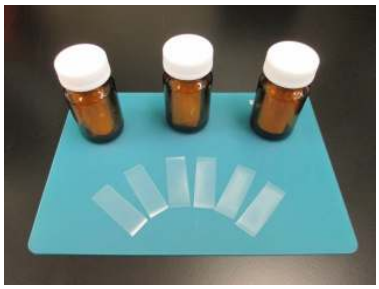

Fig. 1: Phthalate Esters Polymer Standards for Py-GC/MS (S225-31003-91), including seven phthalate esters

(Three types: Blank, 100 mg/kg, and 1000 mg/kg)

Preparing the Phthalate Ester Standard Samples

The phthalate ester resin standard samples are ribbon shaped and of uniform thickness. As a result, samples can be prepared just by placing two fragments of the sample (approximately 0.5 mg) in the Py Eco-cup using the 1.25 mm diameter micro puncher included in the special sampling toolkit. Fig. 1 shows the standard samples. Table 1 shows the repeatability for the weight of samples prepared seven times. The RSD is a favorable 2.24 %, and it is evident that the thickness is uniform.

Table 1: Sampling Weight Repeatability for the 100 mg/kg Phthalate Ester Standard

	1st	2nd	3rd	4th	5th	6th	7th	%RSD
Sample Weight (mg)	0.49	0.50	0.52	0.50	0.50	0.51	0.52	2.24

Analytical Conditions

The conditions registered in the Py-Screener were used as the GC-MS analysis conditions (Table 2).

Table 2: Analytical Conditions

Pyrolizer	:Multi-Shot Pyrolizer EGA/PY-3030D		
GC-MS	:GCMS-QP2010 Ultra		
Column	:Ultra ALLOY-PBDE (Length 15 m, 0.25 mm I.D., df = 0.05 µm)		
[Pyrolyzer]			
Furnace Temp.	:200 °C→(20 °C/min)→300 °C →(5 °C /min)→340 °C(1min)	[MS]	
Interface Temp.	:Manual (300 °C)	Interface Temp.	:320 °C
[GC]		Ion Source Temp.	:230 °C
Injection Temp.	:320 °C	Measurement Mode	:FASST (Scan/SIM mode)
Column Oven Temp.	:80 °C→(20 °C /min)→300 °C (5min)	Scan Mass Range	:*m/z* 50-1000
Injection Mode	:Split	Scan Event Time	:0.15 sec
Carrier Gas	:He	Scan Speed	:10,000 *u*/sec
Flow Control Mode	:Linear velocity (52.1cm/sec)	SIM Event Time	:0.3 sec
Purge Flow	:3.0 mL/min	SIM Micro Scan Width	:0.3 *u*
Split Ratio	:50		

Results

Fig. 2 shows the mass chromatograms for the phthalate esters obtained by measuring the 100 mg/kg phthalate ester standard sample. They were detected with sufficient sensitivity even at a concentration of 1/10th the prescribed concentration. Table 3 shows the repeatability and MDL calculated after measuring the 100 mg/kg phthalate ester standard sample seven times. The repeatability for the quantitative values obtained (% RSD) ranged from 4.1 % to 5.7 %, and the MDL was 12.3 mg/kg to 16.3 mg/kg, which are favorable results.

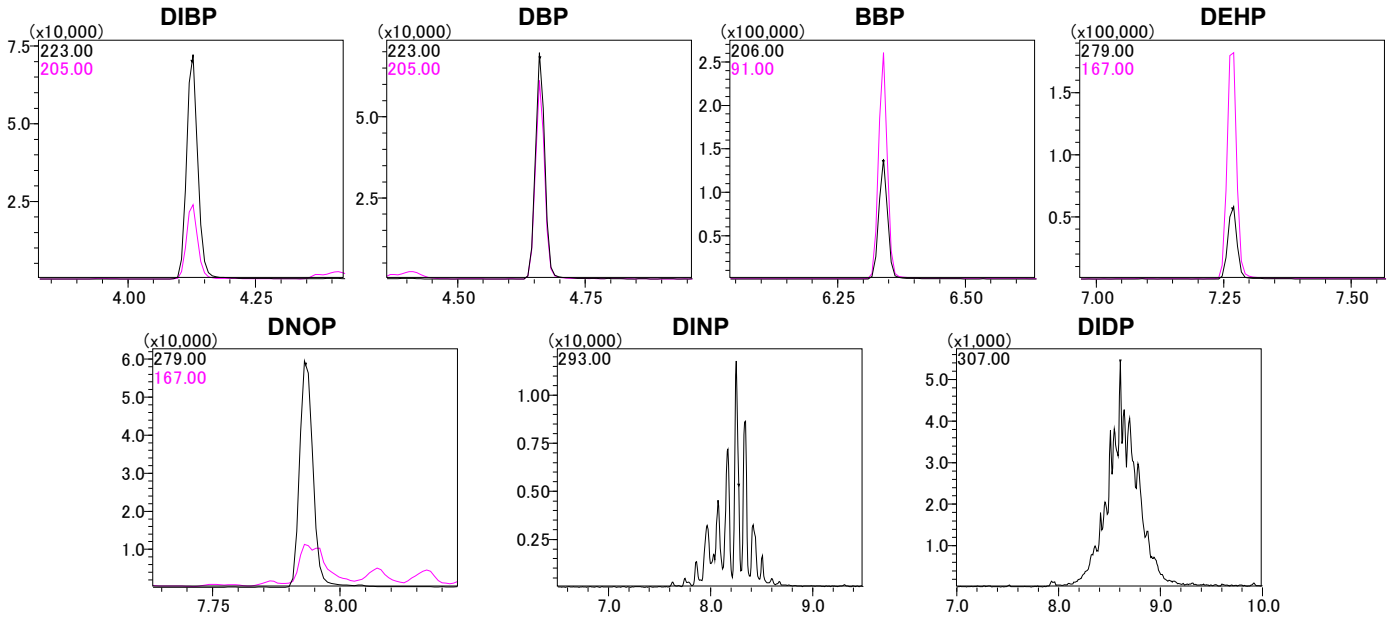

Fig. 2: Mass Chromatograms for the Phthalate Esters Measured in the 100 mg/kg Phthalate Ester Standard Sample

Table 3: Repeatability and MDL for the 100 mg/kg Phthalate Ester Standard Sample (n=7)

	Quantitation Value (mg/kg)							%RSD	MDL (mg/kg)
	1st	2nd	3rd	4th	5th	6th	7th		
DIBP	104.1	102.9	98.6	95.2	103.2	109.3	101.5	4.4	14.0
DBP	107.0	105.2	100.7	98.7	105.9	113.8	106.6	4.6	15.3
BBP	95.5	94.3	91.0	87.9	96.3	100.1	95.1	4.1	12.3
DEHP	110.7	108.5	101.1	101.5	111.2	115.3	108.4	4.8	16.3
DOP	101.2	101.9	93.9	90.3	99.3	103.9	99.1	4.8	15.0
DINP	94.3	95.7	87.4	84.8	92.8	96.8	92.5	4.8	13.7
DIDP	93.3	94.2	83.1	80.9	89.2	91.6	87.3	5.7	15.9

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