Rapid analysis of phthalates for RoHs2 by Py-GC-MS

Related products: Mass spectrometer (MS)

Overview

Phthalates are used as plasticizers for resins, but their use is restricted by various regulations due to concerns about their effects on the human body as endocrine disruptors. The revised RoHS Directive (RoHS2), which came into effect on July 22, 2019, regulates four phthalats (diisobutyl phthalate: DIBP, dibutyl phthalate: DBP, butyl benzyl phthalate: BBP, di-2-ethylhexyl (DIBP, DBP, butyl benzyl phthalate: BBP, di-2-ethylhexyl phthalate: DEHP) were regulated. As measurement methods for these substances, the solvent extraction-GC/MS method for precise analysis and the Py-GC/MS method for screening are specified in IEC-62321-8. We have attempted a rapid analysis of Py-GC/MS method using JMS-Q1500GC by optimizing Py and GC oven conditions, and achieved a cycle time of 14.75 min per measurement. Here, we report the results.

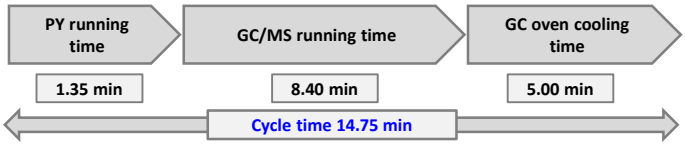

Figure1. Cycle time details

Experiment

PVC (NMIJ CRM 8152-a, about 0.1%), a certified reference material for the analysis of phthalates, was used as the measurement sample. A 0.50 mg sample was measured by Py-GC/MS to confirm the peak separation of the 4 phthalates and the repeatability of the area values at n=5. Table 1 shows the measurement conditions for Py-GC/MS.

Table 1. PY-GC/MS measurement conditions

PY	
Sample weight	0.50mg
Furnace Temp.	150℃→200℃/min→320℃(0.5min)
GC	
Column	ZB-1HT 15m length, 0.25mmi.d. 0.1μm film thickness (Phenomenex Inc.)
Gas flow	He 1.0mL/min, constant flow
Injection mode	Split 1/50
Inlet Temp.	300℃
Oven temp.	80℃→50℃/min→200℃ →25℃/min→350℃

MS : JMS-Q1500GC	
Ion source temp.	250℃
Interface temp.	300℃
Ionization	EI, 70eV
Measurement mode	Scan m/z 50 to 600 with SIM DIBP: *m/z* 149, 205, 223 DBP: *m/z* 149, 205, 223 BBP: *m/z* 91, 149, 206 DEHP: *m/z* 149, 167, 279 ※Under line : Quantitative ion

Results

The TIC chromatogram is shown in Figure 2. The 4 phthalates could be well separated.

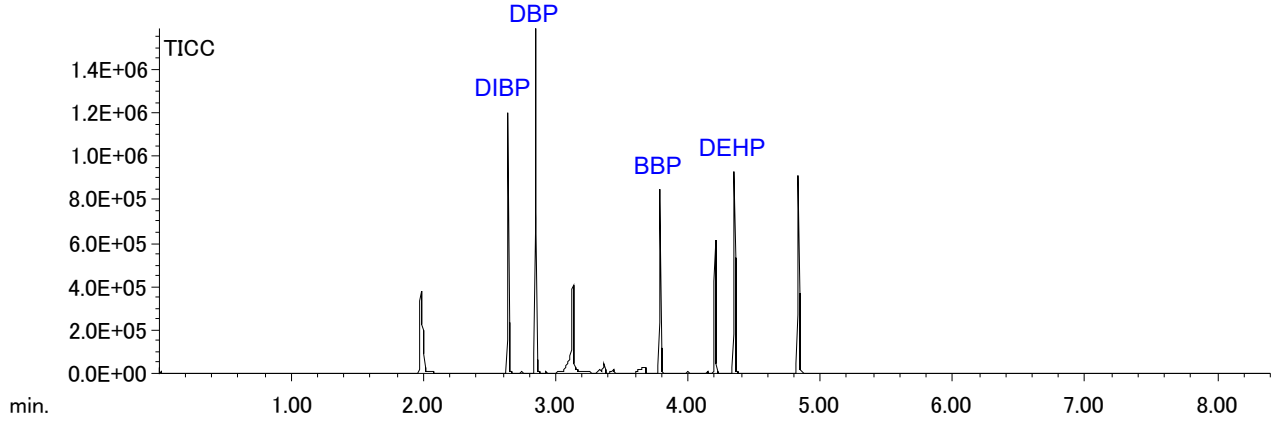

Figure2. TIC Chromatogram

Next, the extracted ion chromatogram (EIC) of the quantitation ion for each of the 4 phthalates is shown in Figure 3. All components showed good detection sensitivity.

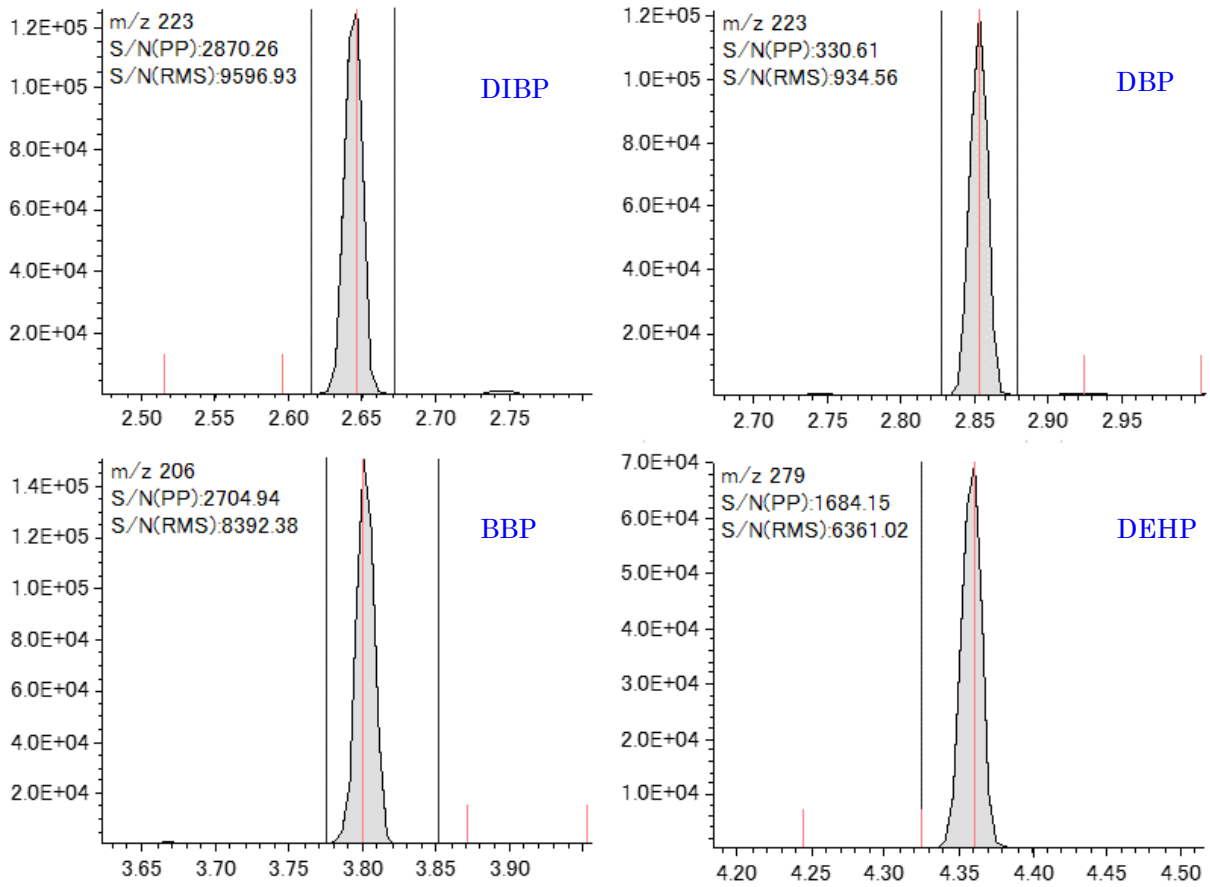

Figure3. Extracted Ion Chromatograms of phthalates in PVC

Finally, the area values of each measured sample at n=5 are shown in Table 2. For each phthalate, good results were obtained with a coefficient of variation of less than 10%.

Table 2. Repeatability of area value

	#1	#2	#3	#4	#5	Average	Standard deviation	CV%
DIBP	178051	168249	158209	157911	167761	166036	8359	5.0%
DBP	166170	150082	152769	142665	148792	152096	8695	5.7%
BBP	210573	212346	205639	205776	243147	215496	15735	7.3%
DEHP	110307	108873	97884	93277	102132	102495	7212	7.0%

Summary

A rapid analysis of four phthalates, which are regulated by RoHS2, by Py-GC/MS method was attempted and the data was obtained with a cycle time of 14.75 min based on good detection sensitivity and repeatability.

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