

GC-MS

APPLICATION NOTES

Phthalate detection scheme in electronic and electrical products by GC-MS



Experimental

Equipment and reagents

Instrument	Aludra gas chromatograph mass spectrometer
Laboratory Equipment	Grinder, liquid nitrogen, analytical balance, ultrasonic cleaner, 0.45 µm PTFE disposable filtration head
Standard Samples	Diisobutyl phthalate (DIBP), Dibutyl phthalate (DBP), Butyl benzyl phthalate (BBP), Di(2-ethylhexyl) phthalate (DEHP)
Solvents	Tetrahydrofuran, Acetonitrile (Chromatographic pure)

Sample preparation

Follow IEC-62321-8:2017 for sample processing:

- Cut the sample into small pieces using scissors and pliers.
- Freeze with liquid nitrogen, then crush with a grinder.
- Accurately weigh 0.3 g of the powdered sample using an analytical balance and transfer it into a 50 mL conical flask.
- Using a pipette, accurately transfer 10 mL of tetrahydrofuran into the flask. Seal the flask tightly and use sealing film to secure the cap (to prevent loosening during ultrasonic treatment).
- Place the flask in an ultrasonic cleaner and sonicate for 60 minutes.
- After ultrasonic treatment, cool the sample to room temperature.
- Use a pipette to transfer 20 mL of acetonitrile drop by drop into the flask to precipitate the polymer in the extract.
- Let it stand at room temperature for 30 minutes (the polymer will gradually settle at the bottom of the flask).
- Filter the sample using quantitative filter paper or a 0.45 µm PTFE disposable filter head into a 2 mL capped sample vial for analysis.

Analysis conditions

Gas chromatograph conditions

Column	Equity-5 (30 m × 0.25 mm × 0.25 µm) quartz capillary column
Flow Mode	Constant flow mode
Column Flow Rate	1 mL/min
Injection Mode	Non-split injection
Injection Volume	1 µL
Injector Temperature	260°C
Split Ratio	50:1
Purge Flow Rate	5 mL/min
Column Temperature Program	Start at 60°C and hold for one minute. Increase at 20°C/min to 300°C, then hold at 300°C for five minutes.

Mass spectrometer conditions

Ion Source	El Source
Ion Source Temperature	230°C
Electron Energy	70 eV
Interface Temperature	280°C
Solvent Peak Time	6 minutes
Scanning Mode	Selected Ion Monitoring (SIM) for quantification
Selected Ion Monitoring (SIM) Settings	6.25 minutes to 12 minutes: Ions 149, 205, 223 12 minutes to 16 minutes: Ions 91, 149, 167, 206, 279

Results

Sample chromatogram

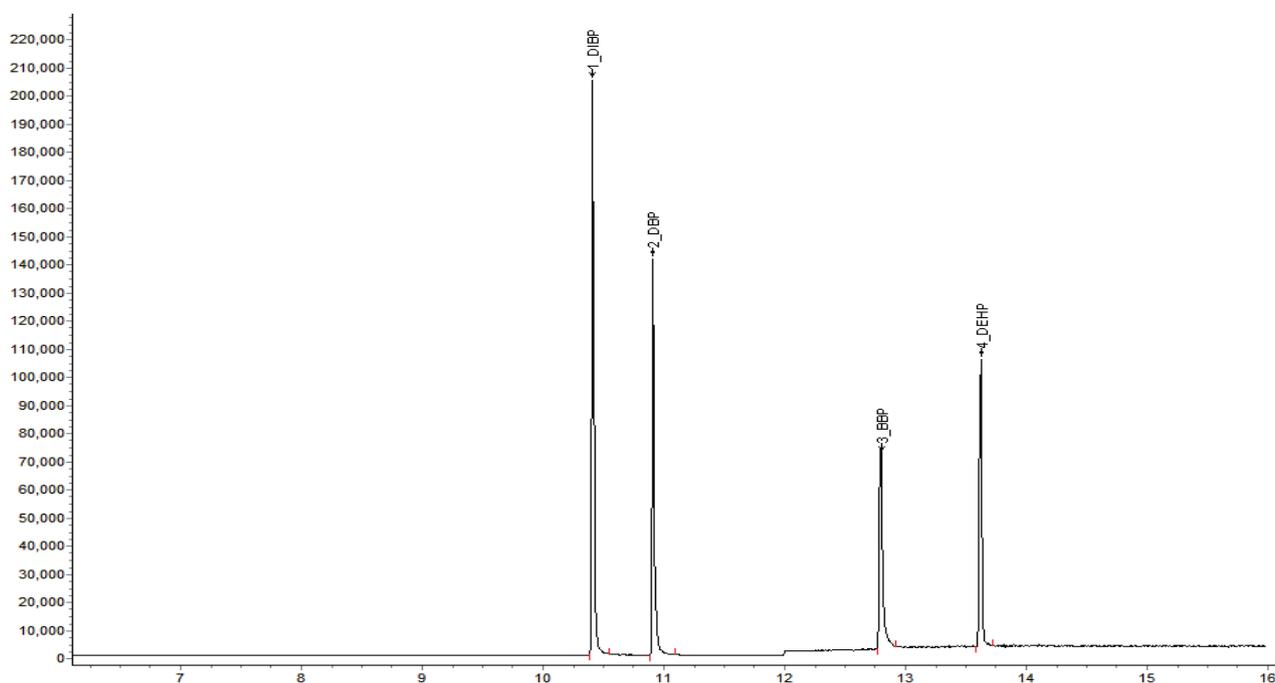


Figure 1: Chromatogram of four phthalate ester standard samples

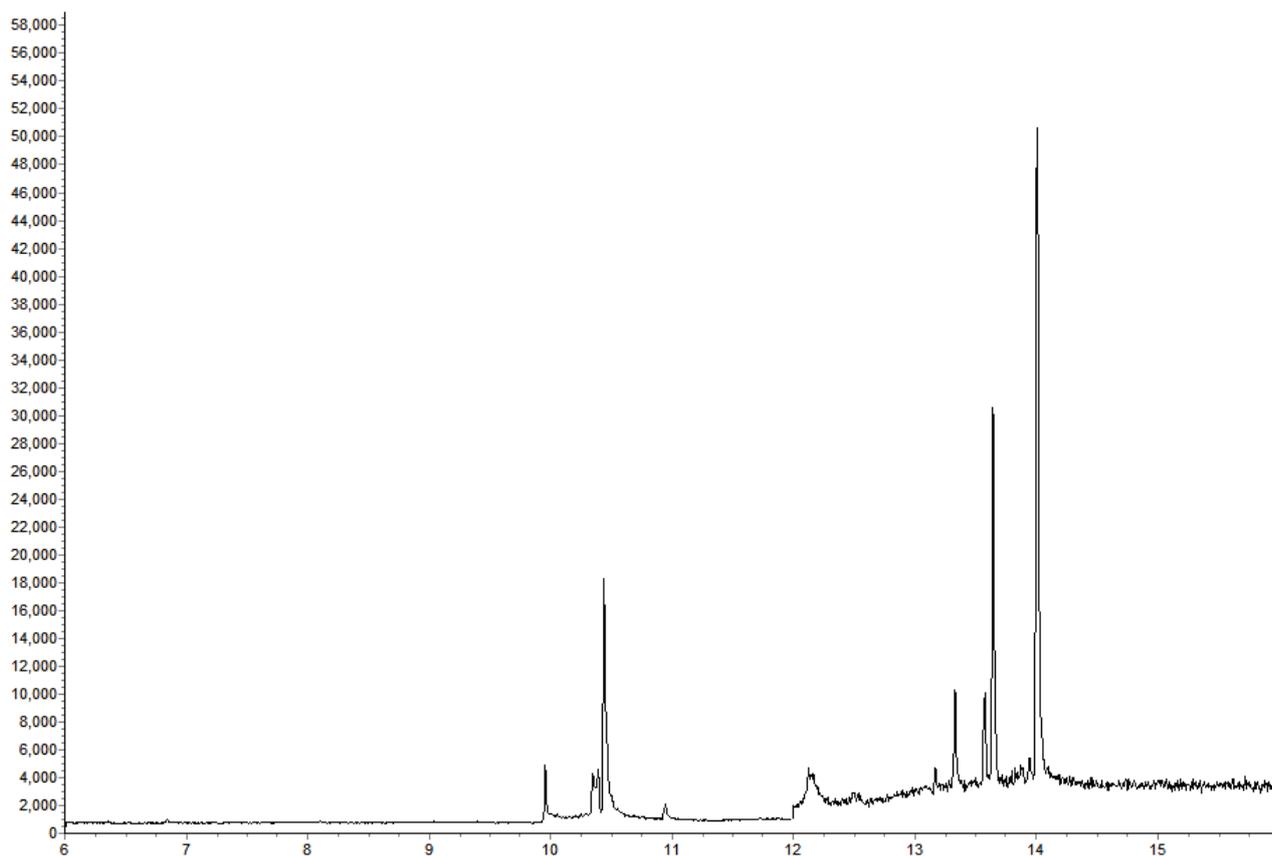


Figure 2: Chromatogram of plastic grid sample

Sample determination

No.	Description	Retention Time	Abbreviations	Ion Monitoring	CAS No.
1	Diisobutyl phthalate	10.41	DIBP	149, 205, 223	84-69-5
2	Dibutyl phthalate	10.91	DBP	149, 205, 223	84-74-2
3	Benzyl butyl phthalate	12.80	BBP	91, 149, 206	85-68-7
4	Di(2-ethylhexyl) phthalate	13.63	DEHP	149, 167, 279	117-81-7

Table 1: Quantification and qualification ions for phthalate esters

No.	Description	Retention Time	Linear Equation	Linear Correlation coefficient
1	Diisobutyl phthalate	10.41	$Y = 441220.6 X + 102125.2$	0.9993
2	Dibutyl phthalate	10.91	$Y = 429661.2 X + 23148.8$	0.9982
3	Benzyl butyl phthalate	12.80	$Y = 255021.1 X - 27240.2$	0.9956
4	Di(2-ethylhexyl) phthalate	13.63	$Y = 394753.6 X - 36722.4$	0.9968

Table 2: Linear equations and linear correlation coefficients for phthalate compounds

No.	Compound Name	Abbreviations	Retention Time	Sample Content mg/kg	ROHS Directive Limits mg/kg
1	Diisobutyl phthalate	DIBP	10.41	ND	1000
2	Dibutyl phthalate	DBP	10.91	ND	1000
3	Benzyl butyl phthalate	BBP	12.80	ND	1000
4	Di(2-ethylhexyl) phthalate	DEHP	13.63	30	1000

Table 3: Sample test results

NOTE:

The above results represent the measured results of the delivered samples according to the above pretreatment method. ND indicates not detected.

Conclusion

The detection method for phthalates in electrical and electronic products was established according to the IEC-62321-8:2017 standard. The ultrasonic extraction method was used to process the samples, which is relatively simple and allows for the simultaneous processing of multiple samples. Selective ion scanning was employed to reduce matrix interference and enhance the instrument's detection sensitivity. The linearity and precision are good, meeting the relevant testing requirements.