

Analysis of Phthalate by ChroZen GC/MS

- GC/MS Application



Abstract

Phthalates are used in a wide range of common products as plasticizers added to plastics to increase their flexibility, transparency, durability and longevity. However, studies of animals exposed to certain phthalates have shown the effect to change hormone levels and cause birth defects and they became concern and into force in restrictions on the use of certain substances in all products intended for children under specific ages in worldwide.

The Ministry of Food and Drug Safety prohibits the use of DEHP (Di-2-ethylhexyl phthalate) for food containers and packing materials that may be absorbed through food and regulates the tolerable daily intake (TDI).

There are many restrictions such as RoHS (Restriction of Hazardous Substances) to regulate phthalates for safety.

Instruments and Software

· ChroZen GC/MS System

Item	Description	Part No.
Oven	ChroZen GC Mainframe Assembly for Mass Spectrometer	6701012500
Inlet	Capillary Inlet Assembly for ChroZen GC	6701012550
Detector	ChroZen MS for ChroZen GC incl. built-in turbomolecular pump - Single Quadropole - EI source (UEIS:Ultimated Efficiency Ion Source) - Turbo pump(240 L/sec) - Include fore pump and spares kit - Higher Sensitivity (S/N for OFN 2500:1)	6901012110
CDS	YL-Clarity software for single instrument of YL GC	5301011020
	MS module of YL-Clarity	5301011180
	Library(NIST/EPA/NIH 2017 edition)-306,622 spectra	NIST17-MS-LIB
Column	YL-5MS (30m, 0.25 mm, 0.25 μ m)	1256120170
Kit	GC Start-up kit	1601011110
ACC	Big Universal Trap, 1/8" fttgs, Helium	RMSH-2

Reagents and Standards

Phthalate mix (2000 μ g/mL each component in dichloromethane)

Preparation of Standard Solution

20.0 mg/L of phthalate mix standard was prepared in dichloromethane (DCM) and diluted to the concentration of 0.050 mg/L, 0.075 mg/L, 0.100 mg/L, 0.250 mg/L, 0.500 mg/L and 1.000 mg/L for calibration curve.



Fig 1. ChroZen GC/MS

Preparation of Water Samples

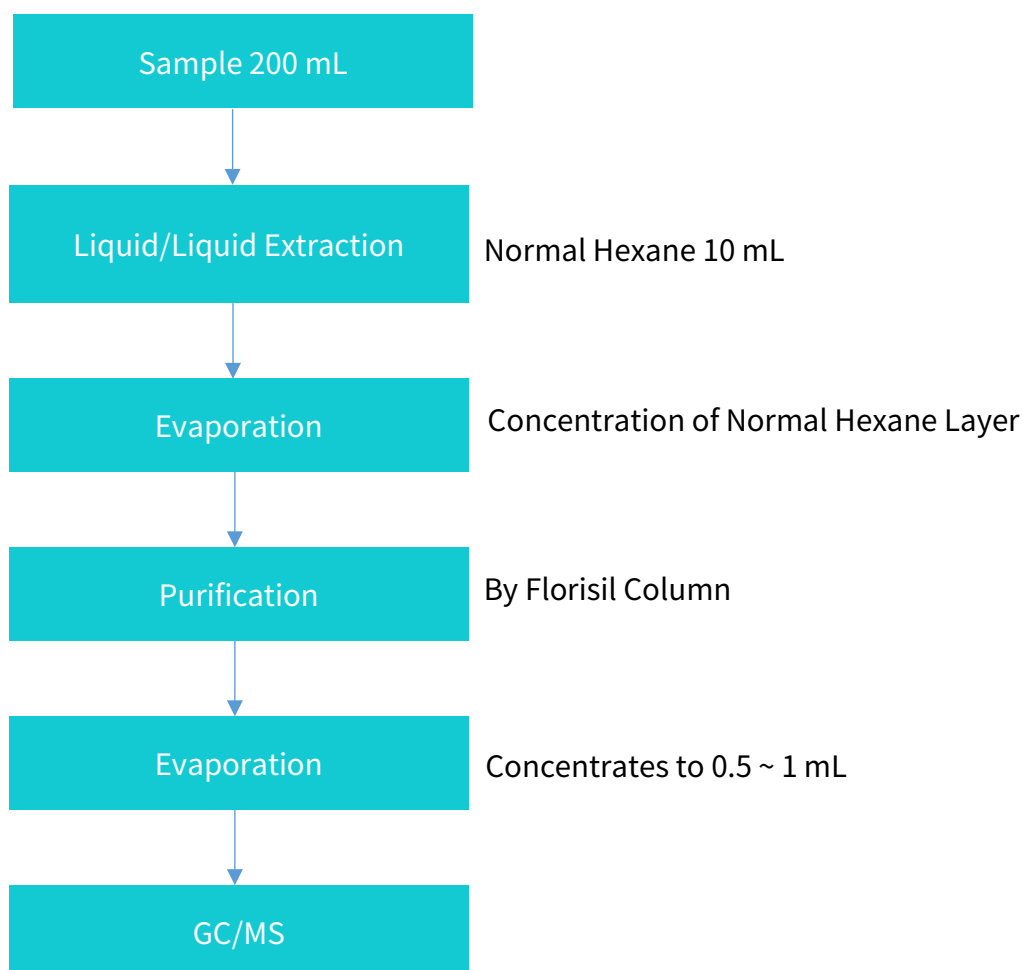


Table 1. Preparation of Liquid Sample

For the preparation of liquid sample, follow the steps described in Table 1. The sample amount can be changed depending on the sample status. Add n-Hexane to the sample to separate layers by Liquid/Liquid Extraction. Extract the n-Hexane layer after exposure of the sodium sulfate layer and concentrate to 0.5 ~ 1 ml. To eliminate the impurities in samples, you may need to purify it by Florisil column. Plastic container or lids may not be used for the storage of samples not to be contaminated. The concentrated sample is transferred to GC vials.

The instrument condition is described in Table 2 and each m/z for phthalate compounds were applied in SIM mode. [Table 3]

GC conditions	MS conditions
Column: YL 5ms (30m x 0.25 mm i.d. x 0.25µm)	Ion source 250°C, Transfer Line 290°C
Inlet: Splitless	
Oven temperature program: 80°C, 0.5min, 15°C/min to 260°C, 10min	
	Detection: SIM mode

Table 2. GC/MS condition

	Compound	Molecular Weight	Selected ions, m/z
1	Dimethyl phthalate (DMP)	194	163
2	Diethyl phthalate (DEP)	222	149
3	Dibutyl phthalate (DBP)	278	149
4	Benzyl butyl phthalate (BBP)	312	149
5	Bis(2-ethylhexyl) phthalate (DEHP)	390	149
6	Di-n-octyl phthalate (DNOP)	390	149

Table 3. GC/MS chromatographic conditions for Phthalate

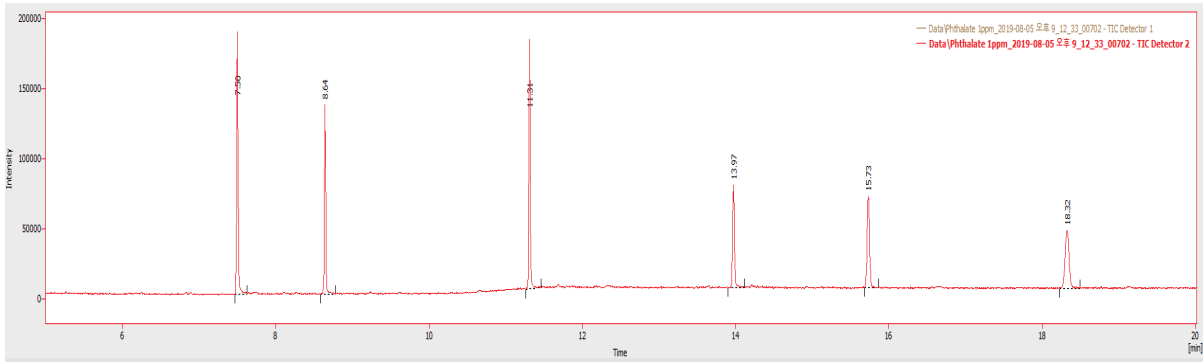
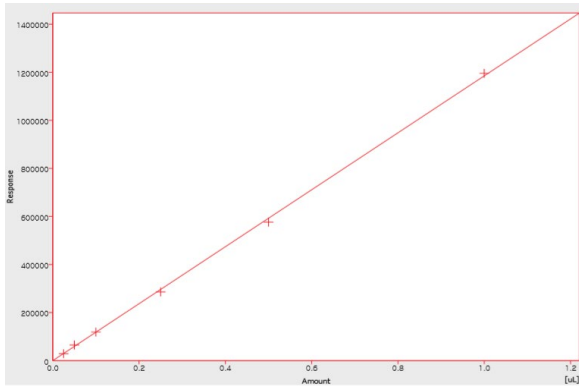


Fig 2. Phthalate 1ppm Chromatogram

[1. Dimethyl phthalate (DMP), 2. Diethyl phthalate (DEP), 3. Dibutyl phthalate (DBP),
4. Benzyl butyl phthalate (BBP), 5. Bis(2-ethylhexyl) phthalate (DEHP),
6. Di-n-octyl phthalate (DNOP)]

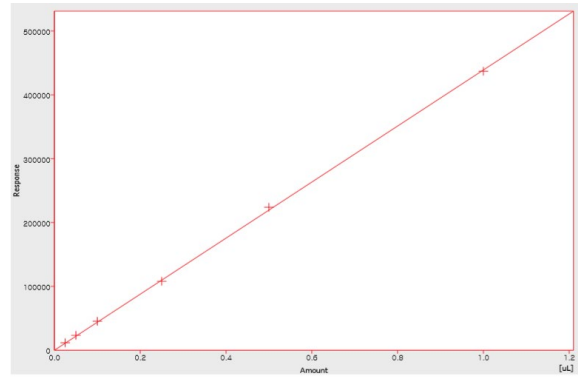
Analyte		R.T (min)	MDL (µg/L)	Accuracy (%)	Precision
1	Dimethyl phthalate (DMP)	7.5	0.0128	96.5	1.98
2	Diethyl phthalate (DEP)	8.6	0.0149	97.8	0.98
3	Dibutyl phthalate (DBP)	11.3	0.0134	101.5	1.71
4	Benzyl butyl phthalate (BBP)	13.9	0.0127	99.8	3.60
5	Bis(2-ethylhexyl) phthalate (DEHP)	15.7	0.0103	103.0	1.37
6	Di-n-octyl phthalate (DNOP)	18.3	0.0142	99.5	1.74

Table 4. Validity of test method



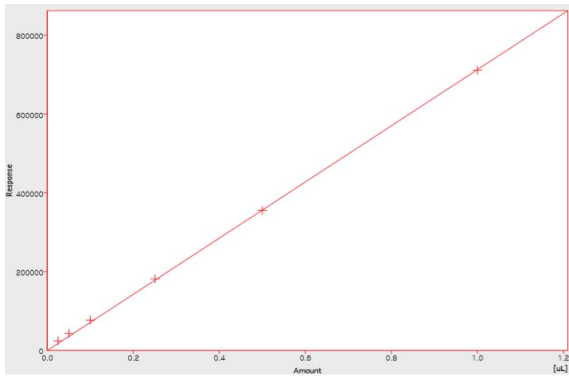
Dimethyl phthalate (DMP)

Correlation Factor : 0.9997838



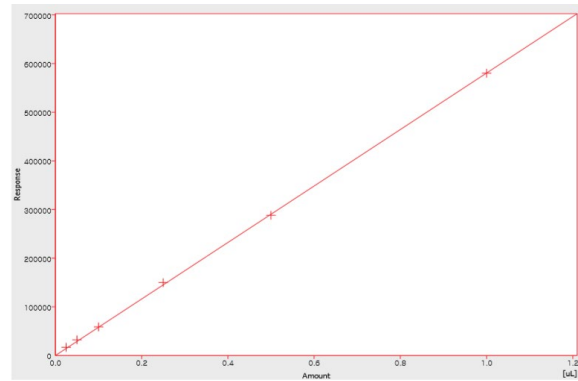
Benzyl butyl phthalate (BBP)

Correlation Factor : 0.9999113



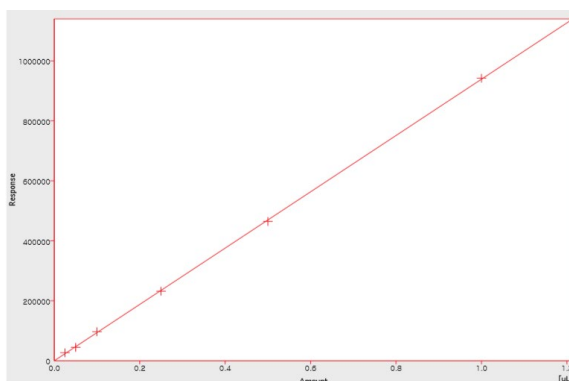
Diethyl Phthalate (DEP)

Correlation Factor : 0.9999450



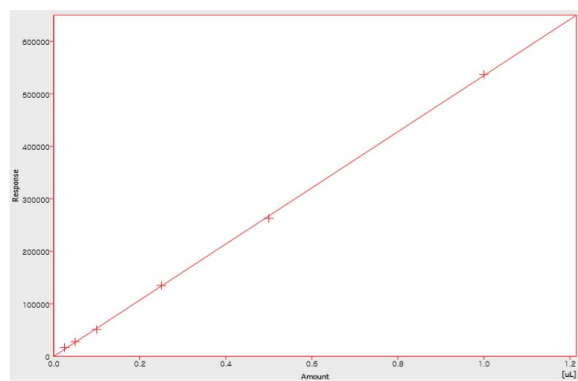
Bis(2-ethylhexyl) phthalate (DEHP)

Correlation Factor : 0.9999526



Dibutyl phthalate (DBP)

Correlation Factor : 0.9999527



Di-n-octyl phthalate (DNOP)

Correlation Factor : 0.9998937

Fig 3. Verification of Calibration Curve

Result

The linearity, accuracy, precision (RSD %) and method detection limit (MDL) were evaluated to verify the validity of analysis results [Table 4]. The correlation coefficients of calibration curve at each concentration was calculated to greater than 0.99 [Fig3]. The environmental research QA/QC handbook by National Institute of Environmental Research was referred for the results evaluation. The results are satisfied with ES 04051.1b (Water Quality Standard Test Method) for analysis of DEHP (Di-2-ethylhexyl phthalate) by gas chromatography/mass spectrometry and this study validates the achievement of the analysis of phthalates.

Conclusion

In this study, the phthalates in liquid sample was analyzed by ChroZen GC/MS. Referring to EPA method 8270E, the validity of analysis results was evaluated by 'Environment Research QA/QC Handbook_ National Institute of Environmental Research' and it verifies data reliability.

Reference

- EPA Method 8270E
Semivolatile Organic Compounds By Gas Chromatography/Mass Spectrometry
- Environment Research QA/QC Handbook_ National Institute of Environmental Research (2011)



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