

「Application Note」

Analysis of Octylphenol by ChroZen GC/MS According to ASTM D7065-06

GC/MS Application



Abstract

Octylphenol, as one of alkylphenols, is classified as endocrine disrupting compounds (EDCs), which are commonly known to us as environmental hormones. EDCs disrupt the physiological action of hormones in the endocrine organs and cause the increase of infertility.

Thus, alkylphenols are regulated by Hazardous Chemicals Control Act, the Public Health Control Act, and the Food Sanitation Act in Korea and they are also prohibited for the use of cleaning agents.

In this study, the samples were prepared in the process of liquid-liquid extraction, concentration and purification. The analysis of octylphenol in water was conducted by ChroZen GC/MS referring to ASTM D7065-06 and Water pollution test standard ES 04614.1 and this study approves that it complies with the regulated method.

Instruments and Software

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 Quadrupole - EI source (UEIS: Ultimate Efficiency Ion Source) - Turbo pump(240 L/sec) - Includes fore pump and spares kit - Higher Sensitivity (S/N for OFN 2500:1)	6901012110
Install. Option	Start-up kit	1601011110
YCM Liquid Autosampler	ChroZen PAL LSI system for liquid injection	6501011590
	Mounting Kit for ChroZen GC	PAL3-Kit-YI6700
CDS	YL-Clarity software for single instrument of YCM GC	5301011020
	MS module of YL-Clarity	5301011180
	Autosampler control of YL-Clarity	5301011040
	Library(NIST/EPA/NIH 2017 edition)-306,622 spectra	NIST17-MS-LIB
Column	YL-5ms (30m x 0.25mm i.d. x 0.25um)	1256120170
ACC	ChroZen PAL System Vial 2CV, Pk of 100 Pcs	Vial-1.5-ND9-CG-100
	ChroZen PAL System Screw Cap 2CV, Pk of 100 pcs	Cap-ND9-St-SP10-100



Fig.1. ChroZen GC/MS System

Reagents and Standards

- ① methanol, CH₃OH : Chromatographic Purity
- ② methylene chloride, CH₂Cl₂ : Chromatographic Purity
- ③ anhydrous sodium sulfate, Na₂SO₄
- ④ Sulfuric acid, H₂SO₄, 9 M
 - Prepare it by slowly adding pure H₂SO₄ and cooling it down
- ⑤ Octylphenol : Standards
 - Use the reagent grade octylphenol and dilute with methanol in chromatographic purity.
- ⑥ 4-n-Nonylphenol: Surrogates
 - Use the reagent grade 4-n-Nonylphenol and dilute with methanol in chromatographic purity.
- ⑦ phenanthrene-d10: Internal Standard
 - Use the reagent grade phenanthrene-d10 and dilute with methanol in

chromatographic purity.

Preparation of Standard Solution

- ① Octylphenol (Standards)

Use Octylphenol in analytical standard with the certificate.

Dilute with methanol in the concentration of 0.5~10.0mg/L for calibration curve.

- ② 4-n-Nonylphenol (Surrogate, SS)

Use 4-n-Nonylphenol in analytical standard with the certificate.

4-n-Nonylphenol as a surrogate is added in all blank samples, standards and samples and used to evaluate the efficiency of method for each sample.

It needs to be added prior to the preparation procedure to evaluate whole process from the preparation and extraction to the analysis report.

- ③ phenanthrene-d10 (Internal Standard, IS)

Use phenanthrene-d10 in analytical standard with the certificate.

Phenanthrene-d10 as an internal standard is added in all blank samples, standards and samples prior to the

analysis.

Phenanthrene-d10 has a similar chromatographic characteristic as it is the requirement of internal standard.

According to internal standard quantitation, the response of all analytes will be measured by comparing to internal standard response.

④ Octylphenol (Standards)

Use Octylphenol in analytical standard with the certificate.

Dilute with methanol in the concentration of 0.5~10.0mg/L for calibration curve.

⑤ 4-n-Nonylphenol (Surrogate, SS)

Use 4-n-Nonylphenol in analytical standard with the certificate.

4-n-Nonylphenol as a surrogate is added in all blank samples, standards and samples and used to evaluate the

efficiency of method for each sample.

It needs to be added prior to the preparation procedure to evaluate whole process from the preparation and extraction to the analysis report.

⑥ phenanthrene-d10 (Internal Standard, IS)

Use phenanthrene-d10 in analytical standard with the certificate.

Phenanthrene-d10 as an internal standard is added in all blank samples, standards and samples prior to the analysis.

Phenanthrene-d10 has a similar chromatographic characteristic as it is the requirement of internal standard.

According to internal standard quantitation, the response of all analytes will be measured by comparing to internal standard response.

Preparation of Water Samples

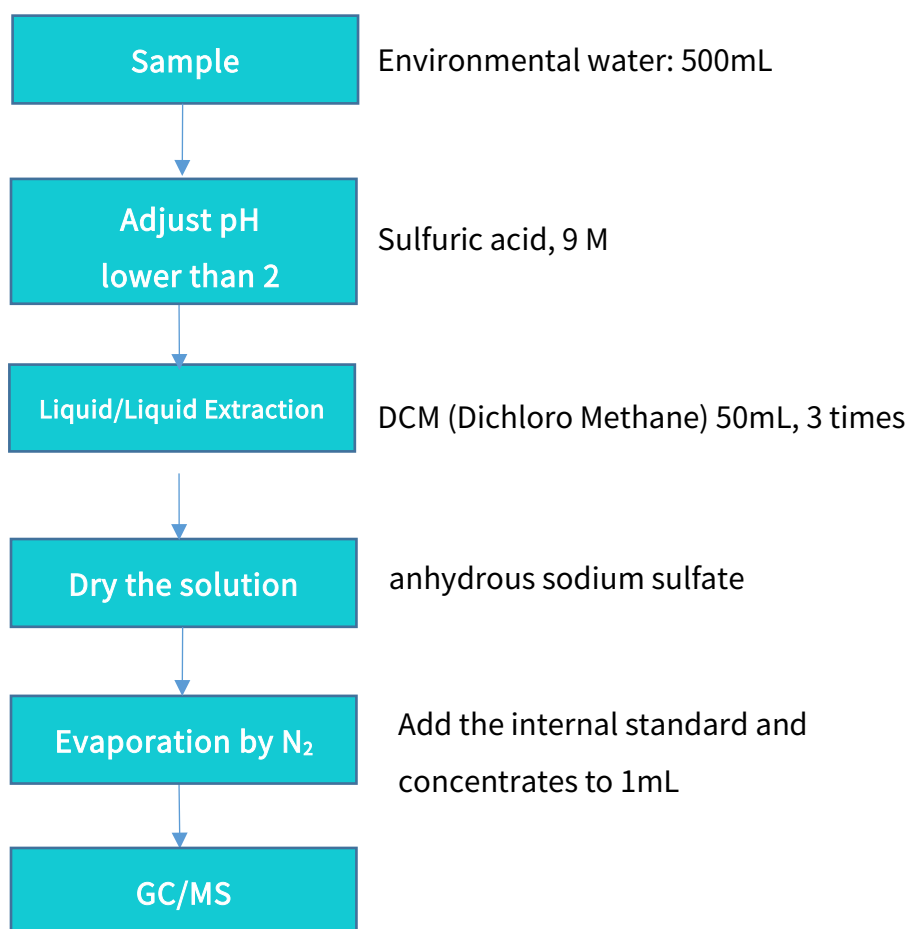


Table 1. Preparation of Water Sample

For the preparation of liquid sample, follow the steps described in Table 1. The sample amount can be varied depending on the sample status. Add 4-n-Nonylphenol as a surrogate prior to sample preparation and adjust pH level lower than 2. Add DCM for liquid/liquid extraction to separate the layers. Dry the solution (water) in the DCM layer by anhydrous sodium sulfate and collect it.

Repeat this procedure 3 times and evaporate it by Nitrogen. The purification by Silica Gel Column is necessary and the purified sample needs to be concentrated up to 1 ml. Put this sample to the GC vial. The GC/MS condition is indicated in Table 2 and the analysis of octylphenol was conducted in SIM (Selected Ion Monitoring) Mode.

GC conditions	MS conditions
Column: YL 5ms (30m x 0.25mm I.D. x 0.25µm)	Ion source: 200°C Transfer Line: 250°C
Inlet: splitless, 1µL Injection Inlet Temperature: 250°C, Split On Time : 1 min	Detection: SIM mode
Oven temperature program : 50°C, 2min, 10°C/min to 160°C, 5°C/min to 200°C, 2min, 30°C/min to 320°C, 3min	

Table 2. GC/MS Conditions

	Compound	Molecular Weight	Selected Ions, m/z
1	Octylphenol	206	135, 107
2	4-n-Nonylphenol (SS)	220	107, 220
3	Phenanthrene-d10 (IS)	188	188, 187

Table 3. GC/MS Chromatographic Conditions

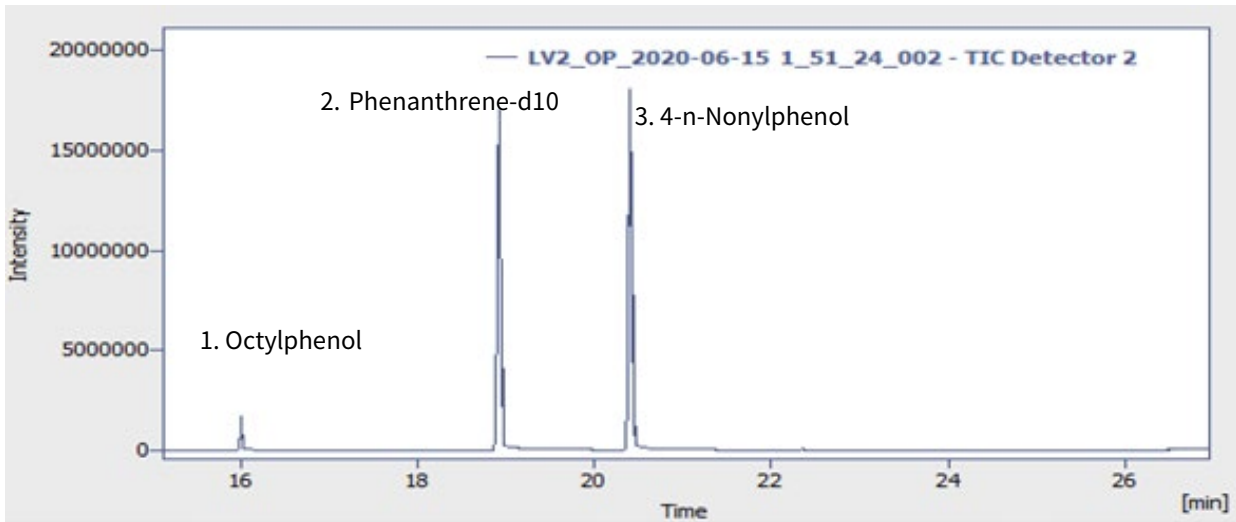


Fig 2. Chromatogram

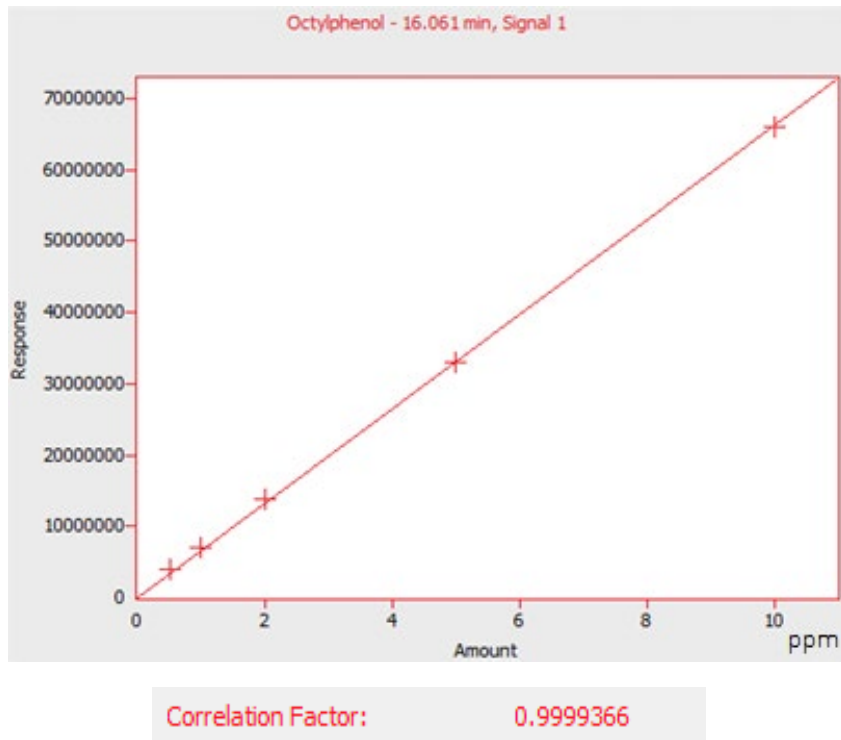


Fig 3. Verification of Calibration Curve

R.T (min)	Limit of Quantitation (µg /L)	Accuracy (%)	Precision
16.061	0.5062	98.68	0.05

Table 4. Validity of Test Method

Result

The linearity, accuracy, precision (RSD %) and method detection limit (MDL) were evaluated to verify the validity of analysis results [Fig 2] [Table 4]. As the result, the limit of quantitation (LOQ) of octylphenol was 0.5062 ug/L, the accuracy was 98.68%, and the precision was 0.05. And these results satisfy the target value in quality control by Water Quality Test Standard Method ES 04614.1.

The calibration curve was generated by internal standard quantitation with internal standard, Phenanthrene-d10, and the correlation coefficients of

calibration curve was calculated according to the environmental research QA/QC handbook by National Institute of Environmental Research [Fig3].

The surface water was used as a sample and the internal standard and the surrogate were added into it [Fig4].

Due to the matrix interference, the result in Scan Mode (in red) is hard to determine quantitation while the SIM Mode (in blue) provides the stable baseline for the accurate one. The recovery of the standard is in the range of 90~110%, which is acceptable.

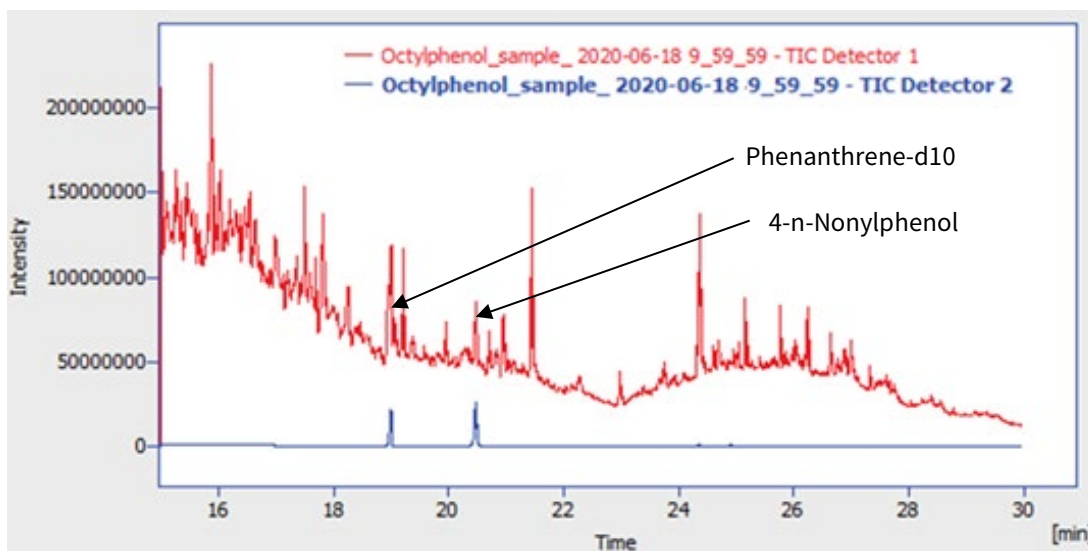


Fig 4. Sample Chromatogram

Conclusion

In this study, the analysis of octylphenol was conducted by ChroZen GC/MS by referring to ASTM D7065-06 and Water Quality Test Standard Method ES 04614.1. And the data was calculated according to the environmental research QA/QC handbook by National Institute of Environmental Research. The result shows that ChroZen GC/MS achieves the data verification.

Reference

- EPA Method 8270E_ Semi-volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
- ASTM D7065-06_ Standard Test Method for Determination of Nonylphenol,

Bisphenol A, p-tert-Octylphenol, Nonylphenol Monoethoxylate and Nonylphenol Diethoxylate in Environmental Waters by Gas Chromatography Mass Spectrometry

- Water Quality Test Standard Method ES 04614.1

- Environmental research QA/QC handbook by National Institute of Environmental Research (2011)



60, Anyangcheondong-ro, Dongan-gu, Anyang-si, Gyeonggi-do, 14042, Korea

TEL: +82-31-428-8700

FAX: +82-31-428-8787

E-mail: export@youngincm.com

Homepage: www.youngincm.com