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Application News

GCMS-TQ™8050 NX Gas Chromatograph mass Spectrometer

Analysis of Dechlorane Plus Residues in Environmental Water Using GCMS-TQ8050 NX with Boosted Efficiency Ion Source(BEIS) and Long-Life Filament

Chen Shen[1], Zhenyu Shi[2]
1 Shimadzu (China) Cor. Ltd, 2 Jiangsu Environmental Monitoring Center, China

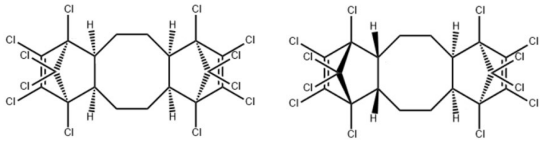
User Benefits

- The combination of the BEIS and long-life filament enables high sensitivity analysis while maintaining long-term stability.
- The ^{13}C-Dechlorane 602 is used as internal standard(IS) in the optimization method to avoid the interference of isotope IS with target components in MRM mode.

■ Introduction

Dechlorane Plus (DP) is a synthetic substance mainly used as an adhesive, sealant and flame retardant for polymers in the industrial field. Due to its strong persistence and bioaccumulation, there is a significant risk to human and environmental health. In 2018, the European Chemicals Agency (ECHA) identified it as a Substance of Very High Concern (SVHC), and in June 2021 proposed to add it to Appendix XVII of the REACH regulation to restrict it, proposing to ban the production and use of DPs and prohibit the production and launch on the market of goods with DP content equal to or greater than 0.1% (by weight). In May 2023, the Stockholm Convention on Persistent Organic Pollutants (POPs) listed DP in Appendix A. At the same time, DP is also included in China's List of new pollutants under Key Control (2023 version).

A method for the determination of residues of DPs in environmental water was established by using the Shimadzu GCMS-TQ8050 NX equipped with Boost Efficiency Ion Source and long-life filament. This application news provides an important analytical method for determine the residue levels of new pollutants in environment with high sensitivity and good stability.



syn-dechlorane plus (Syn-DP)　　anti-dechlorane plus (Anti-DP)

Fig. 1 The structural formula of DP

■ Sample Preparation

To prepare a 1L water sample, a liquid separation funnel should be used. Then, 80 mL of dichloromethane is added, and the mixture should be shaken for 5 minutes. The extraction should be repeated two times. The extract should be combined and washed with 6 mL of n-hexane under an anhydrous Na_2SO_4 drying column. After that, the extract and eluent solution should be combined, and the mixture should be concentrated to 1 mL. Next the solution should be transferred to a silica gel column and eluted with 120 mL of a dichloromethane-n-hexane (1:4) solution. Finally, the eluent should be collected, and it should be concentrated to 1 mL to obtain the test solution.

■ Analysis Conditions

The analytical condition for GCMS is shown in Table 1 and the MRM transitions are shown in Table 2.

Table 1 GC-MS/MS Analysis Conditions

GC	
Instrument:	GCMS-TQ8050 NX (with Boost Efficiency Ion Source and long-life filament)
Column:	SH-I-1HT, 15 m×0.25 mm×0.1 μm (P/N:227-36087-01)
Injection Mode:	Splitless
Sampling Time:	1 min
Injection Temp.:	280 ℃
Column Oven Temp.:	100 ℃(1 min)→20 ℃/min→310 ℃ (5 min)
Flow Control Mode:	Linear Velocity (64.4 cm/sec)
MS	
Ion Source Temp.:	230 ℃
Interface Temp.:	300 ℃
Detector Voltage:	+0.8 kV
Ion Source:	Boost Efficiency Ion Source (BEIS)
Filament:	long-life filament
Ionization Voltage:	70 eV
Emission Current:	185 μA
FWHM:	0.8

Table 2 Information on Compounds

Compounds	RT(min)	Quantitative ion (*m/z*) and CE*1	Ref. ions (*m/z*) and CE*1
^{13}C-Dechlorane 602 (IS)	7.825	281.0>245.8(18)	277.0>241.8(18) 279.0>243.8(18)
Syn-DP	10.166	272.0>236.8(18)	270.0>234.9(18) 274.0>238.8(18)
Anti-DP	10.358	272.0>236.8(18)	270.0>234.9(18) 274.0>238.8(18)

*1 CE (Collision Energies)

■ Quantitative Analysis

In the initial scheme, ^{13}C-DP was used as the IS. However, due to the same retention time of ^{13}C-DP and the target compound, the characteristic ions cause interference, resulting in a large calibration curve intercept and a high RF RSD (>80%). Therefore, ^{13}C-Dechlorane 602 is used as the IS instead. Its retention time differs from target DP, which helps avoid the interference.

■ Linearity

Standard solutions were prepared with concentrations of 0.2, 0.5, 1.0, 2.0, 5.0, 10.0, 20.0 and 50.0 ng/mL respectively (IS 50.0 ng/mL) with n-hexane as solvent. The standard curves of the IS method were drawn using the concentration ratio as the horizontal coordinate and the peak area ratio as the vertical coordinate. The details of linearity are shown in Table 3 and Fig. 2.

Table 3 Information on Calibration Curves Using different IS

IS	Correlation Coefficient (R)		RF RSD%	
	Syn-DP	Anti-DP	Syn-DP	Anti-DP
^{13}C-DP	0.9995	0.9996	82.77	89.09
^{13}C-Dechlorane 602	0.9997	0.9996	13.41	8.04

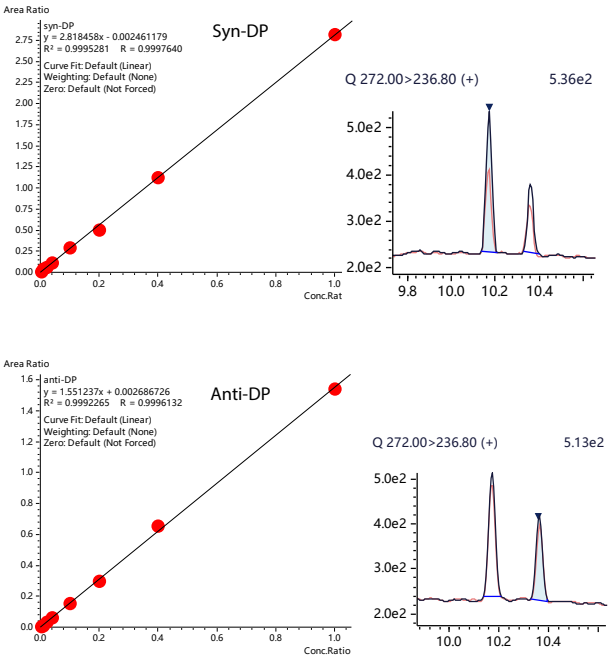


Fig. 2 Calibration Curves and MRM Chromatograms of DPs

■ The Limit of detection (LOD)

The standard solution of 0.2 ng /mL should be injected 10 times consecutively, and LOD should be calculated according to Appendix A of HJ168-2020 [1]. The details of LOD can be found in Table 4.

Table 4 Calculation of LOD

Name of Compounds	Syn-DP	Anti-DP
LOD (ng/mL)	0.063	0.103

■ Repeatability

The standard solution of 0.5 ng /mL should be injected 10 times consecutively to test the repeatability. The results are listed in Table 5.

Table 5 Repeatability Test Result

Name of Compounds		Syn-DP	Anti-DP
Area Ratio	1#	0.025	0.011
	2#	0.022	0.012
	3#	0.024	0.015
	4#	0.02	0.013
	5#	0.022	0.01
	6#	0.024	0.012
	7#	0.023	0.012
	8#	0.02	0.012
	9#	0.02	0.013
	10#	0.022	0.012
RSD (%)		8	11.5

■ Evaluation of Durability in Analysis of Standard Solution and Samples

The sample solution and 0.5 ng/mL standard solution were tested to evaluate the long-term durability of response. A total of 500 analyses of the sample and 80 analyses of standard solution were conducted. Fig.3 shows the durability test results of the standard solution. The horizontal axis shows the number of analyses, while the vertical axis shows the peak area ratio for each analysis number. The results indicate that the changes in peak area ratio and ion proportion were within ±20% of the average value (calculated from very beginning 20 injections).

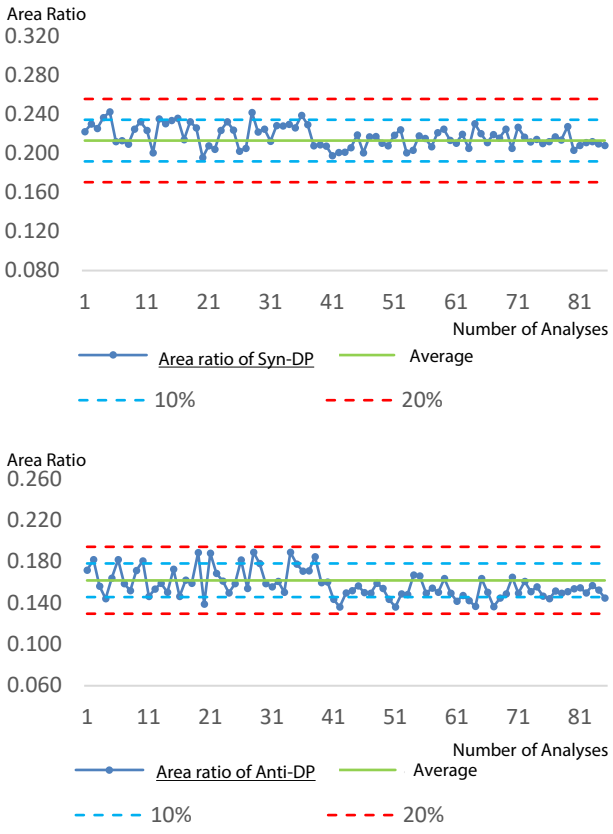


Fig. 3 Durability test results of DP in standard solution

■ Recovery Rate

The surface water samples should be tested after sample pre-treatment. No target compounds were detected. The blank sample should be used for the sample spike test at a concentration of 1 ng/mL, and the recovery rate results were listed in Table 6.

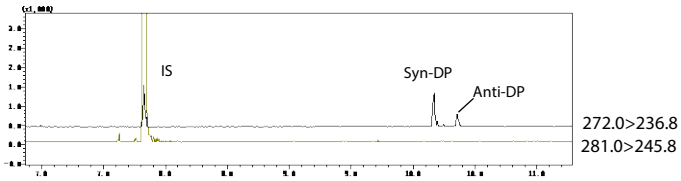


Fig. 4 Chromatograms of Surface Water Spike Sample

Table 6 Recovery Rate Result

Compounds	Concentration ng/mL	Spike Concentration (ng/mL)			
		1#	2#	3#	Average Recovery Rate (%)
Syn-DP	N.D.	1.01	1.09	0.93	101
Anti-DP	N.D.	1.01	1.18	0.86	102

■ Conclusion

A method for the determination of DP residues in environmental water was established by using the Shimadzu GCMS-TQ8050 NX combined with BEIS and long-life filament. The target compound exhibited good linearity in the concentration range of 0.2 to 50.0 ng/mL and the RF RSD of calibration curve was less than 15 %. The standard solution of 0.2 ng/mL was injected for 10 consecutive times to measure the LOD with results of 0.063 and 0.103 ng/mL. The durability of DP response was investigated by alternating test of sample solution and standard solution, and the peak area ratio in standard solution fluctuated within 20% in 500 injection of samples. The recovery rate of 1 ng/mL in blank samples was around 102 %. The method has high sensitivity and good repeatability and is suitable for the determination of DP residues in surface water, groundwater, industrial wastewater, and domestic sewage.

<References>

1) HJ168-2020: Technical guideline for the development of environmental monitoring analytical method standards

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