Analysis of di(2-ethylhexyl)phthalate, dichloroacetonitrile and chloral hydrate by GC-MS method using hydrogen carrier gas

Product used : Mass spectrometer (MS)

1. Overview

Helium (He), which is widely used as a carrier gas for GC, may face problems such as temporary price increases or unstable supply conditions due to various reasons, and when supply delays occur, it is necessary to consider using another type of carrier gas as an alternative. Hydrogen and nitrogen are the main alternative gases being considered. Hydrogen, in particular, has a wide linear velocity range for optimum separation and is suitable as a carrier gas for GC. In this report, di(2-ethylhexyl)phthalate, dichloroacetonitrile, and chloral hydrate, which are water quality control target items in water quality testing, were measured using hydrogen carrier gas by the same column. (For He, see MSTips No. 325.) As a result, good linearity of the calibration curve and reproducibility at the lower limit of quantification were obtained for all compounds, and are presented in this report.

JMS-Q1600GC UltraQuad™ SQ-Zeta

1.1. Measurement Condition

The measurements were performed using a gas chromatograph quadrupole mass spectrometer "JMS-Q1600GC UltraQuad™ SQ-Zeta". The measurement conditions are shown in Table 1. As mentioned above, the column used was the same, and each compound was measured by changing the GC conditions. The monitor ions for SIM described in the Japanese "Notification Law" were used.

Table. 1 Measurement Condition of Each Compound

Parameter		Value	
		Di(2-ethylhexyl)phthalate	Dichloroacetonitrile & Chloral hydrate
GC	Oven temp.	50℃(2min)→20℃/min→180℃(0min)→5℃/min→260℃(10.5min)→10℃/min→280℃(5min), Total 42min	35℃(3.5min)→15℃/min→100℃(0min)→20℃/min→250℃(3min), Total 18.3min
	Column flow (Hydrogen)	1.3mL/min	1.5mL/min
	Injection mode	Splitless, Purge time 1min	PulsedSplitless, Purge time 0.4min
	Injection volume	1uL	2uL
	Column	GL Sciences Inc. InertCap 1MS, 30m x 0.25mm id, 1μm film thickness	
	Inlet temp.	250℃	
MS	Interface temp.	250℃	
	Ion source temp.	250℃	
	Ionization	EI(70eV, 50μA)	
	Acquisition mode	SIM	

2. Di(2-ethylhexyl)phthalate

2.1. Measurement

The test solution was diluted with n-hexane to achieve concentrations of 5, 10, 15, and 20 µg/L of di(2-ethylhexyl)phthalate in the test water before pretreatment. Phenanthrene-D10 was added as an internal standard at a concentration of 250 µg/L.

2.2 Result

The calibration curve for di(2-ethylhexyl)phthalate is shown in Figure 1. The coefficient of correlation for the relative calibration curve was more than 0.999. SIM chromatogram and the coefficient of variation of the quantitation value for the 5 µg/L sample measured continuously at n=5 are shown in Figure 2 and Table 2, respectively. The coefficient of variation of the quantification value at 5 µg/L, which is less than 1/10 of the target value of 80 µg/L, was within 5%, a good result.

JEOL Ltd.
3-1-2 Musashino Akishima Tokyo 196-8558 Japan Sales Division Tel. +81-3-6262-3560 Fax. +81-3-6262-3577
www.jeol.com ISO 9001・ISO 14001 Certified

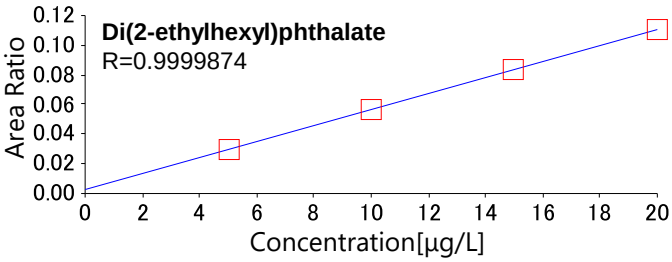

Figure 1. Calibration curve of Di(2-ethylhexyl)phthalate

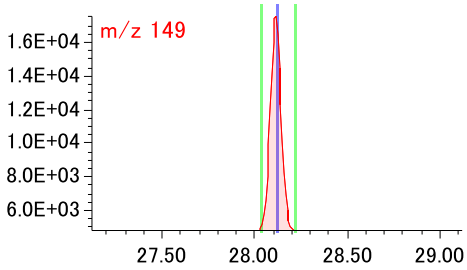

Figure 2. SIM chromatogram of Di(2-ethylhexyl)phthalate at 5µg/L

Table 2. Coefficient variation (C.V.) of Di(2–ethylhexyl)phthalate at 5µg/L

Quantitation value(µg/L)					C.V. %
#1	#2	#3	#4	#5	
5.24	5.04	5.33	5.24	5.10	2.2

3. Dichloroacetonitrile and Chloral hydrate

3.1. Measurement

The test solution was prepared by dilution with MTBE to achieve concentrations of 1, 3, 5, and 15 µg/L of dichloroacetonitrile and chloral hydrate in the test water before pretreatment. 1,2,3-trichloropropane was added as an internal standard at a concentration of 125 µg/L.

3.2. Result

The calibration curves for dichloroacetonitrile and chloral hydrate are shown in Figure 3. The correlation coefficients for the relative calibration curves were more than 0.999. The SIM chromatograms and the coefficient of variation values of the quantification values for the continuous measurement of 1 µg/L samples at n=5 are shown in Figure 4 and Table 3, respectively. The coefficient of variation of the quantification value at 1 µg/L, which is less than 1/10 of the target value (dichloroacetonitrile: 10 µg/L, chloral hydrate: 20 µg/L), was within 5%, a good result.

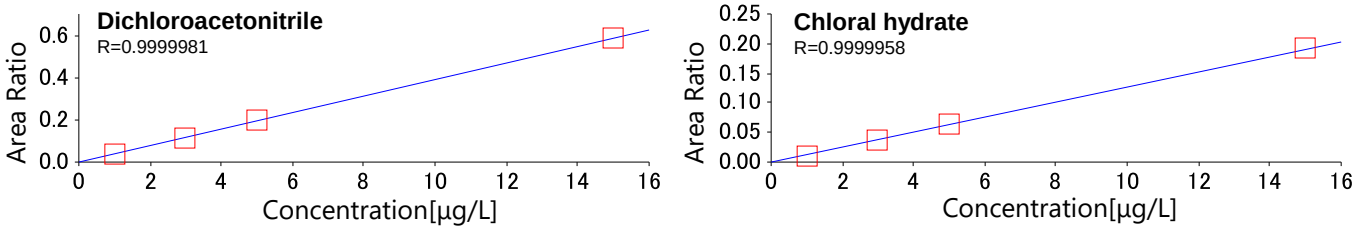

Figure 3. Calibration curve of dichloroacetonitrile & chloral hydrate

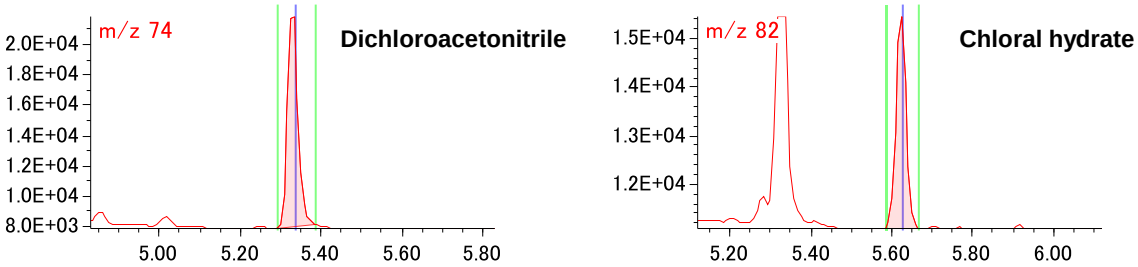

Figure 4. SIM chromatograms of dichloroacetonitrile & chloral hydrate at 1µg/L

Table 3. Coefficient variation of dichloroacetonitrile & chloral hydrate at 1µg/L

Compound name	Quantitation value (µg/L)					C.V.%
	#1	#2	#3	#4	#5	
Dichloroacetonitrile	1.01	1.00	1.03	1.01	1.01	0.7
Chloralhydrate	1.04	1.05	1.08	1.01	1.04	2.4

4. Summary

Di(2-ethylhexyl) phthalate, dichloroacetonitrile, and chloral hydrate were measured by the same column using a hydrogen carrier gas. The linearity of the calibration curve and the reproducibility at concentrations less than 1/10 of the target value were good, confirming that the measurement by the same column using a hydrogen carrier gas can be used adequately.

JEOL Ltd.
3-1-2 Musashino Akishima Tokyo 196-8558 Japan Sales Division Tel. +81-3-6262-3560 Fax. +81-3-6262-3577
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