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Gas Chromatograph-Mass Spectrometry / HS-20 NX, GCMS-QP2020 NX

Application News

Analysis of Epichlorohydrin in Water using HS-GC/MS

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User Benefits

- Water samples can be analyzed with a simple preparation.
- Using a headspace autosampler, the analysis of epichlorohydrin in water quality can be performed conveniently.

■ Introduction

Epichlorohydrin (ECH) is primarily used in the production of various resins (epoxy, ion exchange, etc.) and synthetic glycerin. Additionally, it is used as a stabilizer for carbon containing products in surfactants, plasticizers, dyes, and pharmaceuticals[1].

Some studies have demonstrated that damage to the nasal passages, respiratory tract and kidneys occurred in rodents exposed to epichlorohydrin. It has been observed that exposure to ECH induced a high nasal cancer response in rats, therefore, the United States Environmental Protection Agency (EPA) has classified epichlorohydrin as a "probable" human carcinogen (Group B2).

According Water Pollutants of Water Environment Conservation Act, the permissible discharge limits of ECH in water were 0.03 to 0.3 mg/L by the classified area. The test method for ECH analysis in water is "Epichlorohydrin-Solvent Extraction/Gas Chromatography-Mass Spectrometry (ES 04608.1)" as the official test method of water pollution.[2]

The ES 04608.1 was used to extraction method with a large volume of solvent, it caused high consumption of solvent and long time of preparation. To solve these disadvantages, analysis of ECH in water in this application was performed using HS-20 NX headspace sampler without sample pretreatment.

This application demonstrates the validation of a method for analysis of ECH in water samples using HS-GC/MS (Fig. 1) to meet the demanded levels of ES 04608.1 in Korea.

■ Materials and Method

The standard of ECH and fluorobenzene as internal standard (ISTD) were purchased from Sigma Aldrich. To create standard solution for a calibration curve, the levels of standard solution with ISTD solution were prepared in 20 mL HS vials to reach the final concentrations as shown table 1 below. A 10 mL water sample was transferred into the 20 mL HS vial and ISTD solution was added to the same concentration level (4 µg/L) as standard solution.

Table 1. Working solution for calibration curve

Compound	Concentration (µg/L)				
	1	2	3	4	5
ECH	0.5	1	2	5	10
ISTD	4	4	4	4	4

Fig 1. HS-20 NX + GCMS-QP2020 NX

The method detection limit (MDL) and limit of quantitation (LOQ) were assessed by analyzing the standard at a concentration of 2.5 µg/L in seven replicates. To assess for accuracy and precision of this method, the recovery test was evaluated by analyzing the standard at a concentration of 5 µg/L in three replicates. The analysis of instrumental conditions of HS-GC/MS were as shown in Table 2.

Table 2. Analytical Conditions

Headspace	HS-20 NX
Measurement mode	: Loop
Oven Temp.	: 75 ˚C
Sample Line Temp.	: 150 ˚C
Transfer Line Temp.	: 180 ˚C
Shaking level	: Level 3
Injection Time	: 1.0 min
Pressurizing Time	: 0.5 min
GC Chromatography	**Nexis GC-2030**
Carrier gas flow mode	: Column flow
Carrier gas	: He (1.0 mL/min)
Injection Mode	: Split (5:1)
Analytical Column	: SH-624 (60 m × 0.25 mm I.D., d.f.= 1.4 µm)
Column Temp.	: 40 ˚C (2 min) → 5 ˚C/min → 140 ˚C → 20 ˚C/min → 220 ˚C(4 min)
MS spectrometry	**QP2020 NX**
Ion Source Temp.	: 200 ˚C
Interface Temp.	: 230 ˚C
SIM m/z	: ECH (57, 27, 49, 62), ISTD (96, 70)

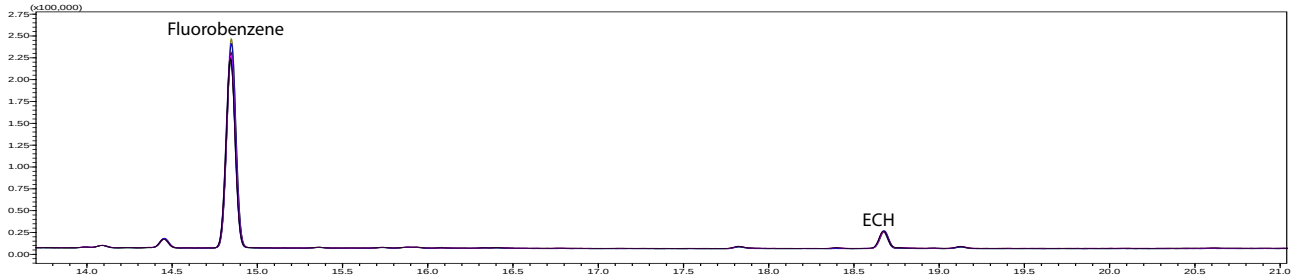

Fig 3. Chromatograms of ECH and ISTD (Fluorobenzene) corresponding to MDL and LOQ (n=7).

■ Results

Calibration curve

The concentration range of calibration was within 0.5 – 10 µg/L and the concentration of the internal standard solution was 4 µg/L. Calibration curve result was a good linearity with R^2 > 0.999. The calibration curve and chromatogram of five concentration levels for ECH is shown Fig 2.

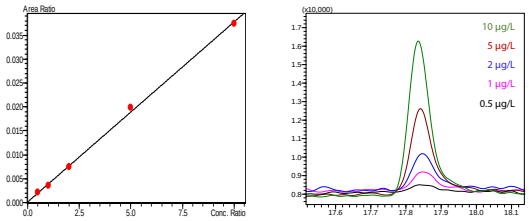

Fig. 2. Calibration curve and chromatograms of standard solutions.

MDL and LOQ

The detection limit of method (MDL) and the limit of quantitation (LOQ) were calculated respectively by multiplying 3.14 and 10 to the standard deviation obtained by seven repeated analysis of the 2.5 µg/L concentration. As a results, MDL and LOQ were 0.1 µg/L and 0.4 µg/L, respectively (Table 3, and Fig. 3).

Recovery test

Water samples for accuracy and precision were pretreated by adding the standard solution at a level of 5 µg/L in water. The results were calculated by repeated measurements three times. As shown in Table 4, a consistent recovery was determined 90 % with RSD 2.4 %.

Table 3. MDL and LOQ of ECH (2.5 µg/L, n=7)

ECH (µg/L)								RSD (%)	MDL (µg/L)	LOQ (µg/L)
1	2	3	4	5	6	7	Average			
2.3	2.4	2.3	2.4	2.4	2.3	2.3	2.3	1.6	0.1	0.4

Table 4. Accuracy and Precision of ECH (5 µg/L, n=3)

ECH (µg/L)				RSD (%)	Recovery(%)
1	2	3	Average		
4.7	4.4	4.4	4.5	2.4	90

■ Conclusions

This Application news demonstrated the use of a Shimadzu GCMS-QP2020 NX coupled HS-20 NX for measurement of ECH in water. The proposed analytical method was with good validation parameters, such as linearity, MDL, LOQ, accuracy and precision which was satisfied the criteria of standard method (ES 04608.1) in Korea.

■ Reference

1) Hazards Assessment and Workplace Management of Epichlorohydrin, J Korean Soc Occup Environ Hyg, 2012: 22(2): 164-173
2) Official test method of water pollution in Korea.

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