Analysis of Simazine and Thiobencarb 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, simazine and thiobencarb, pesticides subject to environmental standards for water pollution, were measured with hydrogen carrier gas. The results show good linearity of the calibration curve and reproducibility at the lower limit of quantification.

JMS-Q1600GC UltraQuad™ SQ-Zeta

2. Methods

Samples were prepared with simazine and thiobencarb at concentrations of 0.01, 0.05, 0.1, and 0.2 mg/L as test solutions. Measurements were made using a gas chromatograph quadrupole mass spectrometer "JMS-Q1600GC UltraQuad™ SQ-Zeta". Table 1 shows the measurement conditions. A calibration curve was created under these measurement conditions, and the coefficient of variation was calculated by continuously measuring a 0.01 mg/L sample with n=5 trials.

Table. 1 Measurement Condition

Parameter		Value
GC	Column	VF-5ms (Agilent Technologies, Inc.), length 30m, inner diameter 0.25mm, film thickness 0.25µm
	Oven temp.	50℃(1min)→20℃/min→300℃(1min), Total 14.5min
	Injection port temp.	250℃
	Injection mode, Volume	Pulsed Splitless, 1µL
	Pulsed press	200kPa
	Purge flow, Purge time	50mL/min, 1min
	Column flow (Hydrogen)	1.5mL/min, Constant flow
MS	Interface temp.	300℃
	Ion source temp.	280℃
	Ionization	EI(70eV, 50µA)
	Acquisition mode	SIM
	Monitor ion	Simazine(*m/z* 201, 186, 173), Thiobencarb(*m/z* 100, 72, 125)

3. Results

3.1. Confirmation of Mass Spectrum

The mass spectra (upper) and library spectra (lower) of simazine and thiobencarb acquired with hydrogen carrier gas are shown in Figure 1. The agreement of the library search in both cases was over 850 (maximum 999), and there was no significant difference in the mass spectra.

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

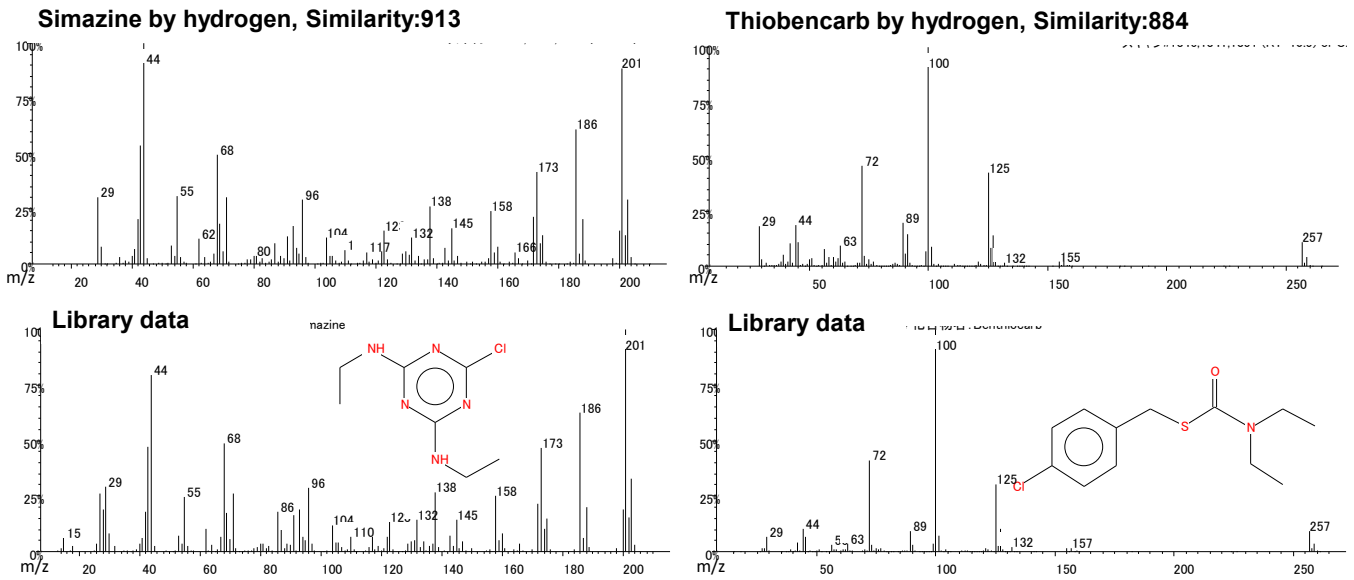

Figure 1. Mass spectra of Simazine and Thiobencarb acquired by hydrogen carrier gas(upper), and NIST Library data(lower)

3.2. Confirmation of calibration curve and reproducibility

Calibration curves for simazine and thiobencarb are shown in Figure 2. The correlation coefficients of the calibration curves were more than 0.999. SIM chromatograms and the coefficients of variation at quantitation values when a 0.01 mg/L sample was measured continuously at n=5 are respectively shown in Figure 3 and Table 2. Coefficients of variation of less than 10% were obtained for both simazine and thiobencarb. The environmental standard for simazine and thiobencarb is 0.003 mg/L for the lowest simazine, and assuming the concentration factor in the solid phase extraction-GC-MS method based on Notification No. 59 of the Environment Agency in 1971, Appendix Table 6, it was confirmed that less than 1/10 of the standard value can be measured.

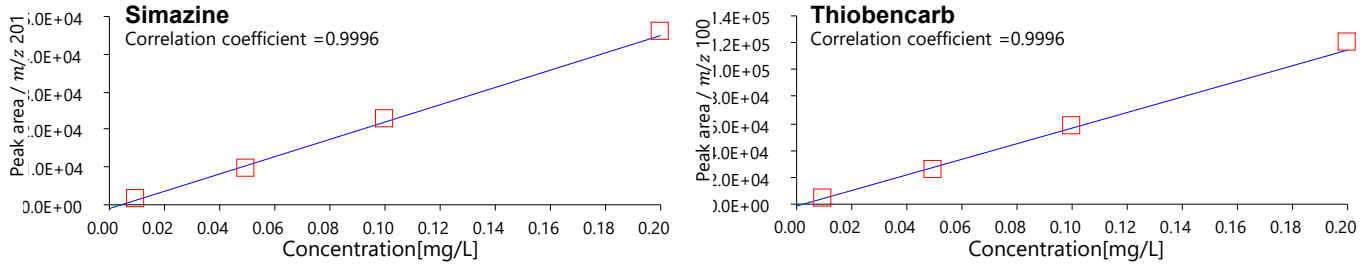

Figure 2. Calibration curve of Simazine and Thiobencarb

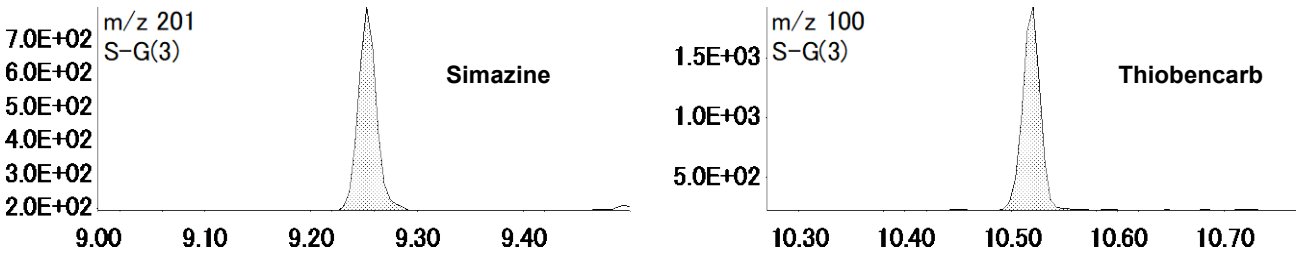

Figure 3. SIM chromatogram of Simazine and Thiobencarb at 0.01mg/L concentration by Hydrogen

Table 2. Coefficient variation (C.V.) of Simazine and Thiobencarb at 0.01mg/L

Compound name	Quantitation value(mg/L)					C.V.%
	#1	#2	#3	#4	#5	
Simazine	0.0101	0.0101	0.0092	0.0101	0.0101	4.1
Thiobencarb	0.0099	0.0098	0.0086	0.0099	0.0099	5.9

4. Summary

Using hydrogen carrier gas, measurements were made for simazine and thiobencarb, pesticides subject to environmental standards related to water pollution, and it was confirmed that less than 1/10 of the standard value could be measured.

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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