# Analysis of Simazine and Thiobencarb by GC-MS method using nitrogen carrier gas

Product used : Mass spectrometer (MS)

## 1. Overview

Helium (He), which is widely used as a carrier gas for GC, may have problems such as temporary price increase or unstable supply due to various reasons. As an alternative gas, hydrogen ($H_2$) has a wide linear velocity range for optimal separation and is suitable as a carrier gas for GC, but must be handled with care due to its influence on the mass spectrum pattern and the risk of being flammable and explosive. On the other hand, nitrogen ($N_2$) is relatively easy to introduce into GC-MS when safety is important, but there is concern about a sensitivity loss of about 1/10 due to decreased ionization efficiency. In this report, simazine and thiobencarb, which are pesticides subject to environmental standards related to water pollution, were measured using $N_2$ carrier gas, and the difference in sensitivity from He, linearity of the calibration curve, and reproducibility during continuous measurement were confirmed.

## 2. Methods

Samples were prepared with simazine and thiobencarb at concentrations of 10, 20, 50, and 100 µg/L as test solutions. Measurements were performed using a gas chromatograph quadrupole mass spectrometer "JMS-Q1600GC UltraQuad™ SQ-Zeta". The measurement conditions are shown in Table 1. A column with a narrow inner diameter of 0.18 mm was used, considering the optimal linear velocity range of the $N_2$ carrier gas. The mass spectrometer parameters were also set to an ionization energy of 20eV to suppress $N_2$ ionization.

**JMS-Q1600GC UltraQuad™ SQ-Zeta**

**Table. 1 Measurement Condition**

| Parameter | | Value |
|---|---|---|
| GC | Column | DB-5ms (Agilent Technologies, Inc.),<br>length 20m, inner diameter 0.18mm, film thickness 0.18µm |
| | Oven temp. | 70℃(2min)→20℃/min→150℃(0min)→10℃/min→300℃(5min) |
| | Injection port temp. | 250℃ |
| | Injection mode / Volume | Pulsed Splitless / 2µL |
| | Pulsed Press | 200kPa |
| | Carrier gas | $N_2$, 13.79kPa, Constant Pressure |
| MS | Interface temp. | 280℃ |
| | Ion source temp. | 250℃ |
| | Ionization | EI(20eV, 50µA) |
| | Acquisition mode | SIM |

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## 3.Results

### 3.1. Confirmation of sensitivity difference from He

SIM chromatograms of $N_2$ and He carrier gases at a 100 µg/L sample are shown in Figures 1 and 2. The sensitivity (S/N:PP) with $N_2$ carrier gas was about 2/3 for simazine and 1/4 for thiobencarb compared to He carrier gas.

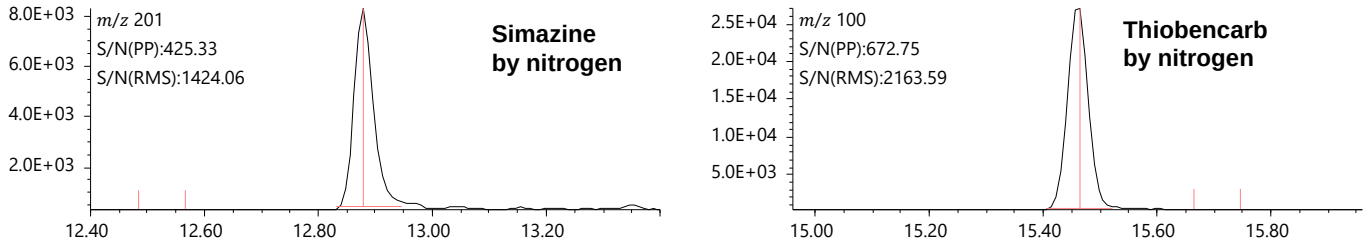


**Figure 1 SIM chromatogram of Simazine and Thiobencarb at 100µg/L concentration by $N_2$**

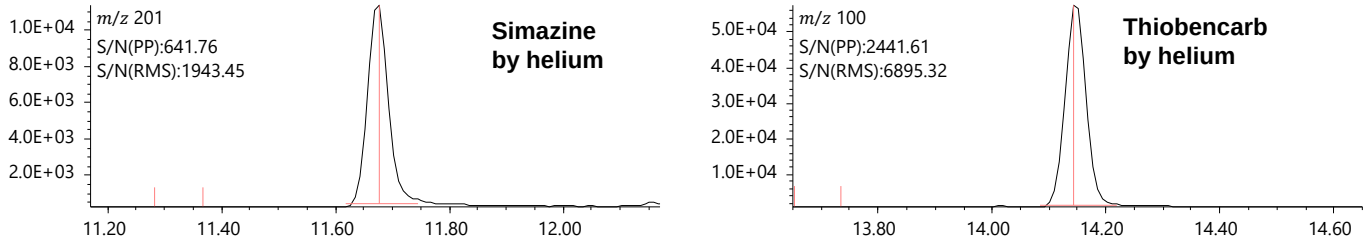


**Figure 2 SIM chromatogram of Simazine and Thiobencarb at 100µg/L concentration by He**

### 3.2. Confirmation of calibration curve and reproducibility

Calibration curves for simazine and thiobencarb are shown in Figure 3. The correlation coefficient of the calibration curves was more than 0.999. The first SIM chromatogram of a 10 µg/L sample measured continuously at n=5 is shown in Figure 4. The coefficients of variation of the quantification values during continuous measurement were 7.5% for simazine and 9.0% for thiobencarb, which were less than 10%. The environmental standard for simazine and thiobencarb is 3 µg/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 at least 1/10 or less of the standard value can be measured.

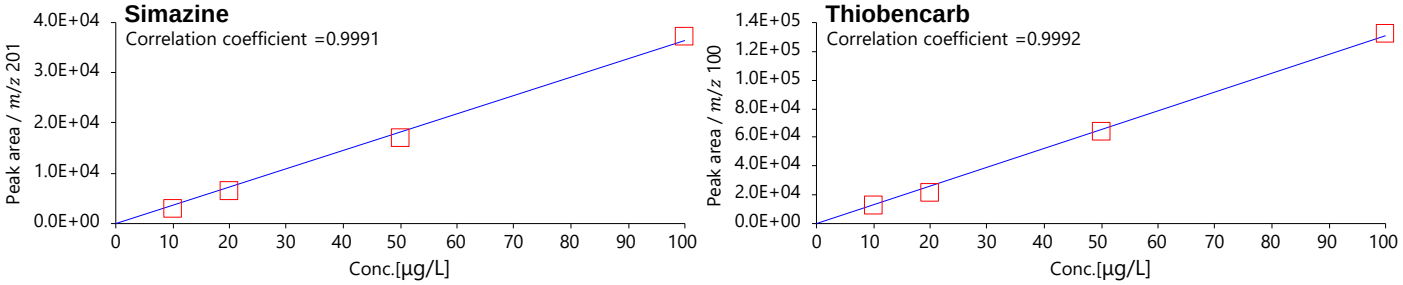


**Figure 3 Calibration curve of Simazine and Thiobencarb**

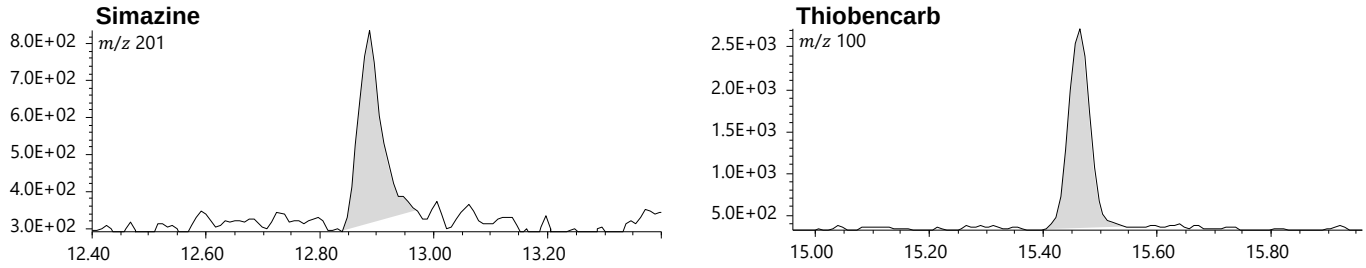


**Figure 4 SIM chromatogram of Simazine and Thiobencarb at 10µg/L concentration**

## 4. Summary

Simazine and thiobencarb, pesticides subject to the environmental standard on water pollution, were measured with $N_2$ carrier gas, which is said to have a sensitivity of about 1/10 of He carrier gas. As a result, even thiobencarb, which has the greatest sensitivity decrease, was measured with a sensitivity of about 1/4 of He. It was also confirmed that at least 1/10 or less of the standard value could be measured based on the coefficient of variation of the 10 µg/L sample.

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