

Interference of Toluene in Two-Phase Capture Solution on Determination of Iodine Concentration

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1. Introduction

Radioactive iodines have attracted a lot of attention because they are considered as important radionuclides in the fields of nuclear accident and radioactive waste disposal [1-7]. In particular, the concentration determination of volatile iodines has been mainly performed in order to predict the behavior of radioactive iodines [6-7].

In order to separate the molecular I_2 from the aerosol iodines and measure their concentrations in the mixture samples of aerosol and I_2 , a two-phase capture solution composed of an organic solvent and an aqueous solution is generally used. However, the organic solvent may interfere with iodine concentration measurement by dissolving in aqueous solution, and the organic solvent easily evaporates at room temperature. For these reasons, it may be difficult to estimate the captured iodine concentration. In this study, the interference of toluene used as an organic solvent in the capture solution was confirmed and a method to minimize the effect of the interference was proposed. In addition, I_2 concentration was measured by reflecting the evaporation of toluene from the iodine capture solution.

2. Methods and Results

2.1 Iodine Capture by Two-Phase Capture Solution

When $NaIO_3$ powder is pyrolyzed in a high-temperature furnace, various volatile iodine species such as NaI , I_2 , and IO_x are formed at about $450^\circ C$ or more. To measure the concentrations of molecular I_2 and aerosol iodine components present in these decomposition products, a two-phase capture solution consisting of an organic phase and an aqueous phase was used. In order to completely capture the iodine component, three capture solutions were connected in series as shown in Fig. 1. In addition, the temperature of the capture solution was maintained at $4^\circ C$ to suppress the evaporation of toluene. Molecular I_2 was captured in toluene phase and NaI , IO_x , etc. in $d-HNO_3$ (dilute nitric acid) phase.

2.2 Determination of Iodine Concentration

The concentration of molecular I_2 captured on toluene was determined by absorbance at 498 nm. Because the UV-VIS absorption of I^- and IO_3^- overlapped

considerably, the concentration of the mixture of these two species could not be determined by UV-VIS spectrophotometry.

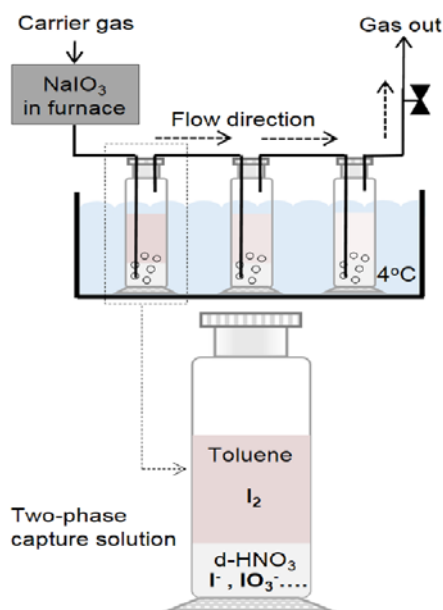


Fig. 1. Schematic diagrams of a two-phase capture system

Because the two-phase capture solution was contact with each other phase, a trace amount of toluene may have dissolved in the $d-HNO_3$ solution. Figure 2 shows the UV-VIS absorption spectrum of $d-HNO_3$ solution after the sample capture process. The spectrum indicates toluene absorption at 261 nm partially overlapping with I^- absorption at 228 nm. The I^- concentrations were determined after corrected using UV-VIS spectra of pure toluene diluted in water. A corrected spectrum was obtained by subtracting the absorption spectrum of toluene from that of the I^- capture solution.

2.3 Evaporation of Toluene from Capture Solution

Although the temperature of the capture solution was maintained below $4^\circ C$, the evaporation of toluene was unavoidable. Therefore, the effect of toluene evaporation on the I_2 evaporation was estimated to ensure analytical accuracy. Three identical two-phase capture solutions were connected in series to ensure the iodine capture. Table I shows the volume loss of the toluene capture solutions after three independent capture experiments. In all first capture solutions, it was

observed that 10–13% of toluene compared to the initial volume was evaporated during the capture process.

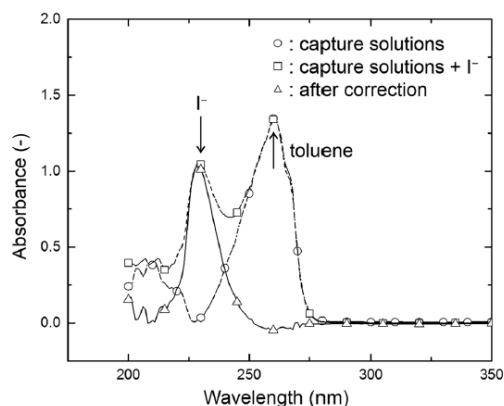


Fig. 2. UV-VIS absorption spectra of the I⁻ capture solution contaminated with toluene (□) and the pure toluene diluted in water (○). The corrected spectrum (△) was obtained by subtracting the absorption spectrum of toluene from that of the I⁻ capture solution (□-○) [7].

The vapor pressures of toluene and molecular I₂ at 4 °C were calculated as 1,153 and 27 Pa, respectively [8]. In addition, in our capturing system, the I₂ concentration captured in toluene was less than 9 mM, which is less than about 0.3% of the toluene mass. Applying Raoult’s law and Dalton’s law under these conditions, the vapor pressure ratio of I₂ to toluene within mixed system was calculated using Eq. (1). Where P^oI₂ is the vapor pressure of iodine, and P^otoluene is the vapor pressure of toluene. P_{I₂} is the vapor pressure of iodine, and P_{toluene} is the vapor pressure of toluene in the mixed vapor conditions.

$$P_{I_2}/P_{toluene} = (P_{I_2}^o X_{I_2}) / (P_{toluene}^o X_{toluene}) \quad (1)$$

Under our capture conditions, P_{I₂}/P_{toluene} was calculated to be 2.3 × 10⁻⁵. This value can be applied to estimate the evaporated I₂ from the first two-phase capture solution during the capture process. In our capture system, the evaporated I₂ was captured again in the second toluene capture solution connected in the series, and this value was added to the total amount of I₂.

Table I: The evaporated volumes of toluene from the two-phase capture solutions after 4.0 h capturing. The initial volume of each toluene capture solution: 150 mL [7]

Experiment	Evaporated volume of toluene capture solution (mL)		
	1st	2nd	3rd
#1	15	3	1
#2	17	4	1
#3	16	2	1

3. Conclusions

Molecular I₂ and aerosol iodine (NaI, IO_x, etc.) were separately captured with a two-phase capture solution composed of toluene and d-HNO₃. The concentration of I₂ captured on toluene phase was determined by absorbance at 498 nm, and the concentration of aerosol iodine captured in d-HNO₃ was determined by ICP-MS. On the other hand, the evaporation of toluene from the capture solution affected the estimation of I₂ concentration, but in our experimental condition where three capture solutions were connected in series, the effect on this was insignificant and could be calculated.

Acknowledgments

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