Dispersal rates of astatine-211 from aqueous solutions and chloroform

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The dispersal rates of 211At, which is a promising nuclide for targeted alpha therapy, from aqueous solutions and chloroform were studied to provide experimental evidence for the reasonable evaluation of its airborne concentration in a radiation-controlled area. Using a collection unit for dispersed 211At during ventilation, radioactivity of the trapped 211At was quantified. The dispersal rates of 211At in chloroform as well as acidic, neutral, and alkaline solutions, in addition to a neutral solution containing ascorbic acid, were determined. Thin-layer chromatography was also performed to identify the formed chemical species of 211At. The dispersal rate of 211At was very low in the neutral solution containing ascorbic acid and in chloroform. The chemical forms of 211At in aqueous solutions are also briefly discussed.

Key Words: astatine-211, dispersal rate, aqueous solution, thin-layer chromatography

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1. Introduction

Airborne concentrations of radioactive materials in their gaseous or aerosol forms are crucial for evaluation of human exposure and radiation protection protocols. In a facility where unsealed radioactive materials are used, regulations restrict their airborne concentrations in a working room and in exhaust air for the safety of the workers1). An important factor influencing airborne radioactivity concentration is the dispersal rate from the originally prepared radioactive solid or liquid sample. Dispersion occurs via mechanisms such as vaporization, evaporation, escape of radioactive materials, evaporation of impurities, decomposition of materials mixed with radioactive materials, and scattering of radioactive aerosols3). Dispersion also depends on the state (gas, liquid, or solid) and chemical forms of the radioactive materials. Therefore, to evaluate the airborne radiation concentration, it is indispensable to experimentally determine the dispersal rates of the radioactivity of interest under various conditions.

Recently, targeted alpha therapy using a short-lived radioisotope emitting α particles has been developed3). In 2016, the short-lived α-emitter 223Ra with a half-life (T1/2) of 11.43 d was approved for clinical use as 223RaCl2 (commercial name Xofigo®) for the treatment of patients with prostate cancer resistant to medical or surgical treatments that lower
testosterone\(^4\). In the near future, other short-lived \(\alpha\)-emitters are expected to be applied to targeted alpha therapy. For realistic and effective clinical use of short-lived \(\alpha\)-emitters, evaluation of the airborne concentration of each nuclide is necessary.

The nuclide \(^{211}\text{At}\) with a \(T_{1/2}\) of 7.2 h is a promising \(\alpha\)-emitter for targeted alpha therapy. This isotope disintegrates via 42\% \(\alpha\)-particle emission and 58\% electron capture (EC). The EC-decay daughter \(^{211}\text{Po}\) decays via 100\% often used as a solvent for \(^{211}\text{At}\). In addition, we used 100 mL a-émitters for targeted alpha therapy. This isotope disintegrates via 42\% of distilled water, which was then used as an \(^{211}\text{At}\) stock solution.

Lack of long-lived \(\text{At}\) isotopes, \(\text{At}\) dispersion has been rarely examined and medical tests\(^4,5\)). However, because of the somewhat similar to those of iodine\(^5,7\), the dispersal rates of exist in various oxidation states and chemical species, formed chemicals species of \(^{211}\text{At}\).

Formation of dry-distillation separation described in Section 2.1, and the radioactivity of \(^{211}\text{At}\) is expected to be applied to targeted alpha therapy. For realistic and effective clinical use of short-lived \(\alpha\)-emitters, evaluation of the airborne concentration of each nuclide is necessary.

2-2. Dispersal rate measurement

An experimental arrangement was prepared to measure the dispersal rate of \(^{211}\text{At}\) shown in Fig. 1. A plastic cylinder with a 75 mm inner diameter (i.d.) was connected to a holder (Sibata Scientific Technology Ltd., Radioactive iodine sampling holder, RI-55) of 74 mm of i.d. and 130 mm of length, in which a glass-fiber filter paper, charcoal-impregnated filter paper, and two charcoal cartridges were placed in this order from the bottom (upstream) side with a SUS mesh support, as illustrated in Fig. 2. The parts were all sealed with O-rings, and the active length of the cylinder was set to 300 mm. The inside of the cylinder was covered with a thin polyethylene terephthalate (PET) sheet to catch dispersed \(^{211}\text{At}\) on its surface. An air pump for ventilation was connected to the top of the filter holder using rubber tubing. This system was placed in a glove box except for the air pump.

To a 100 mL beaker, a 0.010 mL aliquot of the \(^{211}\text{At}\) stock solution was added to 20 mL of aqueous solution (details listed in Table 1) and chloroform. To a 1.5 mL microtube, 0.002 mL of the \(^{211}\text{At}\) solution was pipetted into 0.50 mL of the aqueous solutions listed in Table 1. Chloroform was used only in the experiment with the 100 mL beaker. In the experiment with chloroform, \(^{211}\text{At}\) was dissolved in 0.1 mL of chloroform during the dry-distillation separation described in Section 2.1, and the 0.1 mL aliquot was used as an \(^{211}\text{At}\) stock. The radioactivity of \(^{211}\text{At}\) used in a single run was 0.4–2 MBq at the start of the experiment. The vessel was first placed on a magnetic stirrer,
and the outside of the beaker and top surface of the magnetic stirrer were covered with Parafilm. The interior of the system was subsequently ventilated at an air flow rate of 30 L/min, which is equivalent to 110 mm/s in the upstream direction. The solution was stirred using a stirrer during ventilation, which was performed for 60 min.

The characteristic 79 keV X-ray of Po attributed to the EC decay of $^{211}$At in/on the sample solution, vessel, filter papers, cartridges, and PET-film cover, which was cut in pieces after the procedure, was measured using a Ge detector. The energy calibration of the Ge detector was performed using standard $^{133}$Ba and $^{152}$Eu sources. To determine the efficiency of the Ge detector at a fixed far position (1220 mm), the detector was calibrated using the standard sources to quantify the radioactivity of the initially added $^{211}$At in the 1.5 mL microtube. This measurement at the far position was performed to achieve negligible geometric differences between the point standard sources and microtube. The efficiency of each sample (with $^{211}$At, in a 100 mL beaker, filter papers, cartridges, etc.) was determined at both the far (1220 mm) and closer (50 and/or 235 mm) positions. This is because samples with small amounts of $^{211}$At must be measured at closer positions to obtain higher efficiencies and shorter measurement times. The $^{211}$At radioactivity was evaluated using the measured count, detection efficiency, and emission rate of the 79 keV X-ray with the radioactive decay correction.

The trapped rate can be defined as follows:

$$T_{\text{parts}} = \frac{100 \times A_{\text{parts}}}{A_{\text{ini}}},$$ (1)

where $A_{\text{parts}}$ is the trapped $^{211}$At radioactivity of each part (glass-fiber filter paper, charcoal impregnated filter paper, first and second cartridges, O-ring, and PET film) and $A_{\text{ini}}$ is the initially added radioactivity of $^{211}$At. The dispersal rate of $^{211}$At, $D_r$, can be defined as:

$$D_r = \sum T_{\text{parts}}.$$ (2)

The remaining rates in a vessel defined as:

$$R_v = \frac{100 \times (A_{\text{vessel}} + A_{\text{solv}})}{A_{\text{ini}}},$$ (3)

where $A_{\text{vessel}}$ and $A_{\text{solv}}$ are the $^{211}$At radioactivity on a vessel and in solution, respectively. Finally, the recovery rate ($R$) can be defined as follows:

$$R = D_r + R_v.$$ (4)

2-3. Thin layer chromatography of $^{211}$At

TLC analysis of $^{211}$At was performed to identify its species in the studied aqueous solutions. Before and after ventilation, as described in Section 2-2, a 2 µL aliquot of the aqueous $^{211}$At solution at pH 1, 7, 13, and pH 7 with the AA (Table 1) was spotted on a silica-gel-coated aluminum TLC plate (TLC Silica...
gel 60 F_{254}, Merck). The bottom of the plate was subsequently soaked in a mixed solvent of acetonitrile, water, and trifluoroacetic acid with a volume ratio of 2:1.0.005 for 15 min to develop the spotted $^{211}\text{At}$ along the silica surface of the TLC plate. After development, the TLC plate was subjected to X-ray measurements using a phosphor plate to visualize and quantify the $^{211}\text{At}$ spot retention.

### 3. Results and Discussion

Tables 2 and 3 list the trapped rates in each measured part ($T_{\text{parts}}$), dispersal rates ($D_r$), remaining rates in the vessel ($R_r$), and recovery rates ($R$) of $^{211}\text{At}$ under each solution condition and in the 100 mL beaker and 1.5 mL microtube, respectively. The error limits of the values include a 1σ error from counting statistics and deviation of the data. As listed in Tables 2 and 3, all recovery rates of $^{211}\text{At}$ were within the error, indicating that all dispersed $^{211}\text{At}$ was completely collected using the setup regardless of solution conditions.

For the 100 mL beaker data in Table 2, the dispersal rate of $^{211}\text{At}$ depended on the solution pH, where $^{211}\text{At}$ dispersed moderately in the acidic and basic solutions and largely in the neutral buffer. In contrast, for the microtube, the dispersal rates of $^{211}\text{At}$ were much smaller than those from the beaker and were largely unchanged among the studied conditions (Table 3). These results clearly show that $^{211}\text{At}$ dispersion was suppressed in the microtube because of the much smaller liquid surface area and was not strongly influenced by solution conditions. The vessel-size effect was also unambiguously observed in previous dispersal experiments using $^{223}\text{Ra}$.

Figure 3(a)–(d) shows the results of the TLC analysis. For all TLC plates, $^{211}\text{At}$ exhibited two dominant spots at the origin and front sides with rate of flow ($R_f$) values of 0.0 and 0.92–0.93, respectively, while the relative quantities of the $^{211}\text{At}$ spots differed among the studied solution conditions. At pH 1, 7, and 13, $^{211}\text{At}$ remained at the original position with rates of approximately 30%, 80%, and 46%, respectively. This suggested that polarized, non-mobile $^{211}\text{At}$ species, which exhibited strong interactions with SiO$_2$, were present in aqueous solution with a maximum at pH 7. In contrast, it was expected that neutral At compounds are formed in the aqueous solution.

### Table 1 Aqueous solutions used in this study.

| pH   | Solution                     |
|------|------------------------------|
| 1    | 1 M sulfuric acid solution at pH 1 |
| 7    | 0.025 M phosphoric buffer solution |
| 13   | 1 M sodium hydroxide solution |
| 7 + AA | 0.025 M phosphoric buffer solution with 1.2 wt% ascorbic acid |

### Table 2 Trapped rates in each part, dispersal rates, remaining rates in the vessels, and recovery rates of $^{211}\text{At}$ using the 100 mL beaker.

|                  | pH 1 | pH 7 | pH 13 | pH 7 + AA | Chloroform |
|------------------|------|------|-------|-----------|------------|
| Dispersal rate ($D_r$) |      |      |       |           |            |
| Glass-fiber filter paper | 13 ± 4 | 31 ± 3 | 6.8 ± 0.6 | 0.18 ± 0.01 | 0.06 ± 0.06 |
| Charcoal impregnated filter paper | 8 ± 3 | 15 ± 3 | 6.2 ± 0.6 | 0.36 ± 0.03 | 0.6 ± 0.7 |
| Cartridge (first) | 0.6 ± 0.4 | 2 ± 1 | 0.89 ± 0.06 | 0.27 ± 0.02 | 0.10 ± 0.08 |
| Cartridge (second) | 0.03 ± 0.03 | 0.8 ± 1.0 | 0.0078 ± 0.0008 | 0.0057 ± 0.0006 | 0.01 ± 0.01 |
| Trapped rate ($T_{\text{parts}}$) |      |      |       |           |            |
| O-ring | 0.3 ± 0.2 | 0.7 ± 0.2 | 1.0 ± 0.06 | 0.22 ± 0.02 | 0.003 ± 0.003 |
| PET film | 0.5 ± 0.4 | 0.8 ± 0.3 | 0.58 ± 0.06 | 0.034 ± 0.003 | 0.010 ± 0.002 |
| Stirrer cover | 0.13 ± 0.09 | 0.3 ± 0.3 | 0.032 ± 0.002 | 0.043 ± 0.003 | 0.01 ± 0.01 |
| Vessel cover | 0.4 ± 0.4 | 0.3 ± 0.3 | 0.105 ± 0.004 | 0.028 ± 0.002 | 0.02 ± 0.02 |
| Remaining rate in vessel ($R_r$) |     |     |      |          |            |
| 82 ± 12 | 69 ± 4 | 85 ± 4 | 101 ± 6 | 99 ± 1 |
| Recovery rate ($R$) |      |      |       |           |            |
| 95 ± 5 | 99 ± 2 | 101 ± 4 | 102 ± 6 | 100 ± 2 |

a) bottom side: b) top side.
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Table 3  Trapped rates in each part, dispersal rates, remaining rates in the vessels, and recovery rates of $^{211}$At using the 1.5 mL microtube.

|                  | pH 1   | pH 7   | pH 13  | pH 7 + AA |
|------------------|--------|--------|--------|-----------|
| Dispersal rate ($D_r$) | 3.56 ± 0.09 | 4.02 ± 0.07 | 2.15 ± 0.07 | 2.51 ± 0.05 |
| Glass-fiber filter paper | 0.39 ± 0.01 | 0.074 ± 0.002 | 0.094 ± 0.004 | 0.116 ± 0.004 |
| Charcoal impregnated filter paper | 2.04 ± 0.04 | 0.89 ± 0.02 | 0.65 ± 0.02 | 1.48 ± 0.04 |
| Cartridge (first) | 0.83 ± 0.02 | 2.73 ± 0.06 | 0.196 ± 0.005 | 0.191 ± 0.005 |
| Cartridge (second) | 0.016 ± 0.001 | 0.0043 ± 0.0003 | 0.008 ± 0.001 | 0.091 ± 0.004 |
| O-ring | 0.19 ± 0.07 | 0.29 ± 0.01 | 1.16 ± 0.07 | 0.34 ± 0.03 |
| PET film | 0.09 ± 0.01 | 0.041 ± 0.002 | 0.039 ± 0.003 | 0.30 ± 0.01 |
| Trapped rate ($T_{parts}$) | 0.39 ± 0.01 | 0.074 ± 0.002 | 0.094 ± 0.004 | 0.116 ± 0.004 |
| Charcoal impregnated filter paper | 2.04 ± 0.04 | 0.89 ± 0.02 | 0.65 ± 0.02 | 1.48 ± 0.04 |
| Cartridge (first) | 0.83 ± 0.02 | 2.73 ± 0.06 | 0.196 ± 0.005 | 0.191 ± 0.005 |
| Cartridge (second) | 0.016 ± 0.001 | 0.0043 ± 0.0003 | 0.008 ± 0.001 | 0.091 ± 0.004 |
| O-ring | 0.19 ± 0.07 | 0.29 ± 0.01 | 1.16 ± 0.07 | 0.34 ± 0.03 |
| PET film | 0.09 ± 0.01 | 0.041 ± 0.002 | 0.039 ± 0.003 | 0.30 ± 0.01 |
| Remaining rate in vessel ($R_r$) | 98 ± 2 | 96 ± 2 | 98 ± 2 | 98 ± 2 |
| Recovery rate ($R$) | 102 ± 2 | 100 ± 2 | 100 ± 2 | 100 ± 2 |

Fig. 3. Results of the TLC analysis of $^{211}$At in aqueous solutions at (a) pH 1, (b) pH 7, (c) pH 13, and (d) pH 7 with ascorbic acid (AA).

solutions because neutral species typically show much higher volatility than charged ions$^{22}$. Champion et al. reported that At can exist in three oxidation states (At$, At^{+}$, and AtO$^-$) in a redox window under strongly acidic conditions$^{13}$. Champion et al. also showed that the neutral compound AtO(OH) gradually formed at pH 1 to 4 by hydrolysis of AtO$^-$ under oxidative conditions$^{14}$. More recently, Sergentu et al. suggested that the anionic species AtO(OH)$^-$ is formed at pH > 7 via further hydrolytic reaction of AtO(OH)$^{15}$. From the proposed Pourbaix diagram of At$^{15}$, At$^+$ is expected to exist only at pH < 5. In addition, the authors insisted that AtO(OH)$^-$ is predominant over At$^+$ at pH 11$^{15}$. From these speciation studies$^{13–15}$, it is clear that the neutral species AtO(OH) is strongly volatile and could contribute to the dispersion of $^{211}$At. In addition, the non-mobile $^{211}$At species in TLC may be AtO(OH) because a large amount of sample remained at the origin spot at pH 7. Although Champion et al. reported that AtO(OH)$^-$, which is likely non-volatile and ionic, is formed in pH > 7 and could become predominant in a more basic solution. This is because, herein, the dispersal rate of At remained high in the H$_3$PO$_4$ buffer at pH 7. The volatilities of $^{211}$At after 60 min of incubation at 23°C and 36°C reported previously$^{6}$ were much higher than the dispersal rates measured at pH 7 herein. This is likely because the addition of potassium iodide to Thyrode’s salt solution$^{6}$ resulted in the formation of the highly volatile AtI$^{16}$. Upon addition of AA to the neutral buffer, the dispersal rate of $^{211}$At was remarkably suppressed. This is because of the reduction in the originally present At species, presumably AtO(OH), to the monovalent ionic At$^-$.$^{7,14,15}$ The TLC results in Fig. 3(b) and (d) clearly demonstrates that the At species differed at pH 7 without and with AA. In our previous clinical study with $^{211}$At–[NaAt]$^8$, AA was required to be admixed at 1.2 weight/volume%$^8$ to $^{211}$At-stocked distilled water as a stabilizer in vivo. Thus, for the actual use of $^{211}$At–[NaAt], the dispersal rate of $^{211}$At can be extremely low. In chloroform, a very low dispersal rate of $^{211}$At was also observed. This suggests that $^{211}$At exhibits low dispersion during labeling in chloroform.
In Fig. 4, the normalized trapped rates of $^{211}$At are provided for each solution condition. The normalized trapped rates were obtained by dividing the trapped rate of the two filter papers or the two cartridges by the dispersion rate observed for the 100 mL beaker. It is clear that the normalized trapped rates in the filter papers were higher than those in the charcoal cartridges, indicating that the dispersed At was easily trapped with a glass-fiber filter. This is likely because At disperses as the polar AtO(OH), which is easily adsorbed on polar glass-fiber (SiO$_2$) and on aerosols that can be filtered with glass-fiber, suspended in the air. In contrast, its homolog iodine is vaporized as non-polar I$_2$, which is poorly adsorbed on SiO$_2$ and aerosols. However, in the neutral solution with AA, the normalized trapped rate of $^{211}$At by the filters was lower than the others, indicating that with increasing cartridges, the dispersal rate of $^{211}$At decreased, as listed in Table 1. Therefore, these results suggest that At could be easily trapped using common HEPA filters in a ventilation system. It should be noted that the larger error limits of the chloroform data are due to the large observed deviation, because only a small amount of $^{211}$At was dispersed and trapped by the filters and cartridges.

4. Conclusion

The dispersal rates of $^{211}$At from various solutions were examined, and the rates were found to vary depending on the solution conditions, with the maximum dispersion observed at pH 7. In the neutral solution containing ascorbic acid, the dispersion rate of $^{211}$At was quite low. This suggests that in future clinical studies with $^{211}$At-[NaAt] in a reductive solution, the dispersion of $^{211}$At should be negligible. In addition, At could be easily trapped using a common HEPA filter in a ventilation system, which differs from iodine isotopes.

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