Mass-spectrometric determination of iodine-129 using O₂–CO₂ mixed-gas reaction in inductively coupled plasma tandem quadrupole mass spectrometry

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Abstract
This paper presents a mass-spectrometric method for determining the radionuclide iodine-129 (¹²⁹I) from the significant amount of interference in inductively coupled plasma tandem quadrupole mass spectrometry (ICP–MS/MS) using a dynamic reaction cell passing a mixture gas of O₂ and CO₂. Thus far, mass spectrometry analysis of trace amounts of ¹²⁹I has been hampered by the presence of xenon-129 (¹²⁹Xe) and the formation of polyatomic ions from excess amounts of stable isotope ¹²⁷I. In this study, flowing a mixture gas of O₂ and CO₂ into the dynamic reaction cell (Q2) successfully removed both ¹²⁹Xe interference and polyatomic interference (¹²⁷IH₂) in the analysis of ¹²⁹I in ICP–MS/MS. The resulting ratio of (background noise of m/z 129)/¹²⁷I was 4.6 × 10⁻¹⁰ ± 3.3 × 10⁻¹⁰, which enables the analysis of 10 mBq/L of ¹²⁹I in the presence of 100 mg/L of stable ¹²⁷I without chemical separation. The detection limit of this method was 0.73 mBq/L (=0.11 ng/L) with an APEX-Q sample inlet desolvation device. For demonstration purposes, spike and recovery analysis of rainwater was performed, and good agreement between the spiked and recovered amounts was achieved.

Keywords Iodine-129 ⋅ Dynamic reaction cell ⋅ Mixture gas ⋅ Inductively coupled plasma tandem quadrupole mass spectrometry

Introduction
Radioactive iodine-129 (¹²⁹I), which has a long half-life of 1.57 × 10⁷ years, is a remarkable indicator for the investigation of influence to the environment from nuclear power activities. Over the past 50 years, the inventory of ¹²⁹I on Earth has increased from the pre-nuclear-age level of 10⁻¹²–10⁻¹⁵ to 10⁻¹¹–10⁻⁹ due to accumulation through artificial activities, such as discharge from nuclear fuel reprocessing plants, atmospheric nuclear weapons testing, and nuclear accidents (as a fission product of ²³⁵U and ²³⁹Pu) [1]. Monitoring anthropogenic ¹²⁹I as an indicator or tracer can help understand the transport and dynamics of ¹³¹I, which has a short half-life of 8 days, in radioactive wastes discharged from nuclear facilities or in materials deposited by fallout from nuclear accidents [2–5]. The simultaneous determination of radioactive ¹²⁹I and natural ¹²⁷I (i.e., the ratio of ¹²⁹I/¹²⁷I) has contributed to determine the origin and age of seawater, porewater, groundwater, methane hydrate, and meteorites [6–10]. Thus, the determination of ¹²⁹I contributes to many fields, such as environmental science, nuclear forensics, and geological science; however, the determination of ¹²⁹I is challenging using mass spectrometry (MS).

Accelerator mass spectrometry (AMS) is often used to determine the ratio of ¹²⁹I/¹²⁷I due to the high mass resolution and low background (¹²⁹I/¹²⁷I = 10⁻¹⁵–10⁻¹⁰) [11–14]; however, its cost is high, and the control of contaminants is difficult. Inductively coupled plasma mass spectrometry (ICP–MS) is a popular means to determine ultratrace elements and is employed by many institutions. The advantage...
of ICP–MS over AMS is that it performs analysis with higher sample throughput; however, it must remove mass-spectrometric interferences (xenon-129 \[^{129}\text{Xe}\] and \[^{130}\text{Xe}\]^+)\(^+\), which tend to increase the background noise (BGN) intensity of \(m/z\ 129\) and the detection limit.

\[^{129}\text{Xe}\] is the primary source of interference in MS analysis of \(^{129}\text{I}\) and is contained in argon (Ar) plasma gas as an impurity. It can be almost completely removed by the charge transfer reaction using \(\text{O}_2\) via the reaction cell in ICP–MS \(\text{Xe}^+ + \text{O}_2 \rightarrow \text{Xe} + \text{O}_2^+\) [15]. Ohno et al. reported suppression of the high BGN level caused by \(^{129}\text{Xe}\) via the \(\text{O}_2\) reaction using the Agilent triple quadrupole (Q) ICP–MS system. It should be noted that the MS configuration was \(m/z\ 129\) (Q1) \(\rightarrow\) \(\text{O}_2\) reaction (Q2) \(\rightarrow\) \(m/z\ 129\) (Q3). Previous studies achieved a ratio of \(^{129}\text{I}/^{127}\text{I} = 5 \times 10^{-9}\) by the \(\text{O}_2\) (mono-gas) reaction [15, 16]; however, a ratio of \(^{129}\text{I}/^{127}\text{I}\) with an order of magnitude less than \(10^{-9}\) is required for the analysis of actual rainwater [18–20]. Thus, the ICP–tandem quadrupole MS (ICP–MS/MS) technique faces challenges achieving values less than \(10^{-9}\).

The primary factor limiting the improvement of the ratio is the formation of hydrogen additives (i.e., \(^{127}\text{I}^2\text{H}_2\)) as polyatomic ions, which are created in Ar plasma and increase the ratio of \(^{129}\text{I}/^{127}\text{I}\). In current reaction techniques, such as dynamic reaction cell (DRC, i.e., Q2) or collision–reaction cell, the simultaneous removal of \(\text{IH}_2\) and \(\text{Xe}\) has been challenging.

This paper presents a method for removing interferences (\(\text{Xe}^+\) and \(\text{IH}_2^+\)) using a mixture of \(\text{O}_2–\text{CO}_2\) gas in Q2 (DRC) in ICP–MS/MS. Analysis of actual rainwater was performed to evaluate the ability of the proposed method to remove \(\text{IH}_2\) in Q2.

### Experimental

#### Reagents and preparation

A radioactive \(^{129}\text{I}^-\) stock solution (0.3 Bq/g [= 45.9 ng/g]), radioactive purity > 99%, used as potassium iodide) obtained from Kaken Corporation Ltd. (Ibaraki, Japan) was diluted to the desired concentration using Milli-Q water (18.2 MΩ-cm; Merck KGaA, Darmstadt, Germany). A stable iodine \(^{127}\text{I}^-\) solution was prepared from ammonium iodide (purity 99.5%) obtained from FUJIFILM Wako Pure Chemical Co. (Osaka, Japan). Tetramethylammonium hydroxide (TMASH; Tama Chemicals Co. Ltd., Kawasaki, Japan) was spiked into the \(^{127}\text{I}^-\) solution as a stabilizer. Cesium (Cs) was used as an internal standard solution (1000 mg/L; atomic absorption spectrometry grade; SPEX Certiprep Inc., Metuchen, NJ, USA) and was spiked into each test solution. All other analytical-grade reagents were purchased from FUJIFILM and were used without further purification unless otherwise noted.

### Instrumentation

A triple quadrupole ICP–MS/MS instrument (NexION 5000, PerkinElmer, Sheraton, CT, USA) equipped with a DRC (Q2) between two quadrupole mass-filters (Q1/Q3) and a four-channel reaction gas manifold was used for all experiments. Ultrapure-grade gases (\(\text{O}_2 > 99.99995\%), \text{CO}_2 > 99.995\%, \text{NO} > 99\%, \text{N}_2\text{O} > 99.999\%) were mixed in the gas manifold and directly introduced into the DRC. The sample solutions were injected by a PFA concentric nebulizer and cyclonic spray chamber (300–350 μL/min) or by a high-efficiency sample-injection device (APEX-Q, Elemental Scientific, Omaha, NE, USA) into the ICP–MS instrument. Optimization of the parameters or conditions in ICP-MS/MS was obtained using 200 ng/L of mixed element standard solution (Li, Mg, In, Ce, and U) and the solution was flowed at above mentioned flow rate in the STD mode (no gas mode), while the sensitivity (intensity) of \(^{115}\text{In}^+\) was maximized during the maintaining less than 3% of Ce\(^+\)/Ce\(^+\) ratio. The resultant obtained parameters for ICP–MS/MS analysis are listed in Table S1 in the Supplementary information (SI).

### Calculations

The normalized intensity \((N_I)\) values were calculated using the following equation:

\[
N_I = \frac{\text{Measured intensity (cps)}_f}{\text{Initial intensity (cps)}_0},
\]

where the initial intensity (cps) is the original intensity when no gases flow into the DRC (i.e., flow rate = 0 mL/min), and the measured intensity (cps) is the actual intensity in the presence of gases in the DRC at an arbitrary flow rate (mL/min). The subscript “\(f\)” represents the flow rates of gas in the DRC.

### Sample collection and pretreatment

Rainwater was collected in Fukushima Prefecture, Japan. The samples were filtered using a hydrophilic polytetrafluoroethylene (PTFE) polymer membrane filter (0.45 μm × 47 mm). The rainwater was prepared to 3% TMAH aqueous solution before ICP–MS/MS analysis. Although an acidic media solution (e.g., \(\text{HNO}_3\)) causes a memory effect in the spray chamber of ICP–MS by the production of volatile species such as HI and \(\text{I}_2\), the use of an alkaline media (e.g., TMAH) can circumvent the interferences with short washout time [21–24]. The washout was carefully confirmed using Milli-Q water.
Results and discussion

Behavior of the BNG of m/z 129 in the presence or absence of mono-gas in the DRC (Q2)

In the absence of any gases in the DRC (Q2), the average BGN of m/z 129 was \(1.4 \times 10^3 \pm 9.5 \times 10^1\) cps (gas flow rate = 0 mL/min; Q1 → m/z 129 / Q3 → m/z 129). The values depended on the employed Ar gas cylinders; the intensity ranged from \(1.4 \times 10^3\) to \(5.0 \times 10^3\) cps for each cylinder. When the reaction gases flowed into the DRC (Q2), the BGN of m/z 129 was effectively removed using O₂, as illustrated in Fig. 1a. The actual BGN was finally decreased to 0–1 cps. In addition, Fig. 1b demonstrates that the intensity of I⁺ as the analytical target was almost maintained by using O₂ gas. For example, the signal intensity of \(1.2 \times 10^5\) cps was maintained with the injection of 1.0 µg/L for 127I, as illustrated in Fig. S1 in the SI. These results reveal that the O₂ reaction in the DRC (Q2) is effective in determining 129Xe⁺ and 129I⁺, and the results are similar to those of previous studies [15, 16, 20].

Effect of the mixture of reaction gases on BGN suppression

Figure 2 displays the contribution of O₂ as a mono-gas reaction in the DRC (Q2) for the removal of the external interference. Whereas the BGN of m/z 129 remained at a low level in a sample solution not containing 127I, it rose in the presence of a relatively high concentration of 127I (1000 mg/L) in the sample solution. This may have been caused by the tailing of 127I in MS or the formation of 127IH₂⁺ in Ar plasma; however, the primary cause of the external interference could not be determined. It was difficult to significantly reduce the BGN in the presence of an excess amount of 127I to the lowest BGN (i.e., in the absence of 127I) for any flow rate of O₂ in the DRC (Q2).

An oxygen-based gas mixture (two variants: O₂–N₂O and O₂–CO₂) was used to remove the interference. Under a constant flow of O₂ (1.0 mL/min), N₂O or CO₂ was added to the O₂ gas at a rate ranging from 0.0 to 2.0 mL/min. Figure 3 presents the effect of additional gas on the BGN. The mixture of O₂–N₂O improved N₁ by reducing it to \(5.5 \times 10^{-3}\) (black and white triangle symbols in Fig. 3) as compared with the no-gas condition. Notably, the O₂–CO₂ mixture
improved N\textsubscript{2}\textsubscript{f} further, reducing it to $2.8 \times 10^{-4}$. The contributions of the gases to the N\textsubscript{1} value were $5.4 \times 10^{-2}$ and $5.2 \times 10^{-3}$ for O\textsubscript{2} and CO\textsubscript{2}, respectively. Whereas the use of mono-gas in the DRC (Q2) had difficulty simultaneously removing the two interference states (\textsuperscript{129}Xe and \textsuperscript{127}IH\textsubscript{2}) and discriminating \textsuperscript{129}I in the presence of a high concentration of \textsuperscript{127}I, the use of a mixture gas resulted in the simultaneous elimination of the interference states. This method can be combined to the preconcentration of the sample water to enhance the analytical sensitivity of MS due to the allowance of the higher ratio of BGN (\textsuperscript{129}I/\textsuperscript{127}I) since the actual concentration of radioactive \textsuperscript{129}I was at an ultra-low level.

The mechanism of the suppression of IH\textsubscript{2}\textsuperscript{+} by CO\textsubscript{2} is discussed based on the experimental data as follows. Based on the experimental results that CO\textsubscript{2} oxidation was slower than that of O\textsubscript{2} and N\textsubscript{2}O, the oxidative reaction was unlikely to occur. Furthermore, because the ionization potential of CO\textsubscript{2} (13.79 eV) is much higher than that of I (10.45 eV) and IH (10.39 eV) (cf. no data found for IH\textsubscript{2}), the reaction by the charge transfer effect was unlikely to occur. Although H\textsubscript{2} gas was intentionally introduced into the DRC, little or no IH\textsubscript{2}\textsuperscript{+} was formed. In other words, even if a small quantity of I in O\textsubscript{2} gas existed as an impurity, it would not form IH\textsubscript{2}. In addition, based on the reaction degree illustrated in Fig. 3, the reaction suppressing the formation of IH\textsubscript{2}\textsuperscript{+} was preferentially caused only for hydrogen additive (IH\textsubscript{2}\textsuperscript{+}) passing through Q1 (m/z \textsuperscript{129}), not for the I\textsuperscript{+} ions. This suggests that the kinetic energy of IH\textsubscript{2}\textsuperscript{+} was preferentially reduced by the collision reaction of CO\textsubscript{2} with polyatomic ions (IH\textsubscript{2}\textsuperscript{+}) due to the larger cross section of CO\textsubscript{2} than that of I\textsuperscript{+}. Similar behavior has been reported, namely, that the kinetic energies of medium or heavy elements are more effectively reduced by inert gases with larger atomic sizes, such as neon (Ne) and krypton (Kr), in the collision reaction, than by gases with smaller atomic sizes, such as He [28–30].

**Determination of \textsuperscript{129}I/\textsuperscript{127}I ratio and analytical performance**

A blank solution (0 Bq/L of \textsuperscript{129}I) containing 0.5 w/w\% of \textsuperscript{127}I (as \textsuperscript{127}I in NH\textsubscript{4}I) with 1.0 μg/L of \textsuperscript{133}Cs as an internal standard was measured by the proposed method (O\textsubscript{2}: 1.0 mL/min and CO\textsubscript{2}: 2.0 mL/min). The axial field technology voltage was adjusted to a negative value of $-25$ to suppress the BGN of m/z \textsuperscript{129} (Fig. S2 in SI). An intensity ratio of (m/z \textsuperscript{129})/\textsuperscript{127}I of $4.6 \times 10^{-10}$ ± $3.3 \times 10^{-10}$ was achieved. Table 1 presents a comparison between the proposed method and previous methods for obtaining the ratio of \textsuperscript{129}I/\textsuperscript{127}I. The value of \textsuperscript{129}I/\textsuperscript{127}I in the proposed method was relatively lower than other previous methods with high precision. The previous methods had difficulty measuring rainwater due to the relatively high \textsuperscript{129}I/\textsuperscript{127}I ratio. The calibration curve of \textsuperscript{129}I was shown in Fig. 4. The relative standard deviation (RSD) of 1.0 Bq/L of \textsuperscript{129}I was 2.5% (45.8 ± 1.2 cps) and the detection limit (3σ) was 6.7 mBq/L (equal to 1.0 ng/L) (sample inlet: a PFA concentric nebulizer and a cyclonic spray chamber). In addition, the signal stabilities were confirmed under the flowing O\textsubscript{2}–CO\textsubscript{2} mixed gas for an hour in the presence of 1.0 Bq/L of \textsuperscript{129}I or blank solution (3% TMAH only), as shown in Fig. 5. The intensity of \textsuperscript{129}I was maintained within

**Table 1** Comparison of the \textsuperscript{129}I/\textsuperscript{127}I ratio of background (e.g., abundance sensitivity) with that of previous ICP–MS and ICP–MS/MS studies

| Method       | Reaction gas | \textsuperscript{129}I/\textsuperscript{127}I | References |
|--------------|--------------|-------------------------------------------|------------|
| ICP–MS       | O\textsubscript{2} | $5.0 \times 10^{-9}$ | [16]        |
| ICP–MS       | O\textsubscript{2} + He | $7.0 \times 10^{-9}$ | [31]        |
| ICP–MS/MS    | O\textsubscript{2} | $5.0 \times 10^{-9}$ | [17]        |
| ICP–MS/MS    | O\textsubscript{2} | $7.0 \times 10^{-9}$ | [27]        |
| ICP–MS/MS    | O\textsubscript{2} + CO\textsubscript{2} | $4.6 \times 10^{-10}$ | This study  |

![Fig. 4](https://example.com/fig4.png) The calibration curve of \textsuperscript{129}I via flowing mixed reaction gas of O\textsubscript{2} (1.0 mL/min) and CO\textsubscript{2} (2.0 mL/min). Experimental condition: the sample \textsuperscript{129}I solution in 3% TMAH. MS configuration: (Q1) m/z 129/(Q3) 129

![Fig. 5](https://example.com/fig5.png) The signal trends of \textsuperscript{129}I and BGN via flowing mixed reaction gas of O\textsubscript{2} (1.0 mL/min) and CO\textsubscript{2} (2.0 mL/min). Experimental conditions: for measurement of BGN: sample 3% TMAH solution. MS configuration: (Q1) m/z 129/(Q3) 129. For the measurement of \textsuperscript{129}I: sample 1.0 Bq/L of \textsuperscript{129}I in 3% TMAH. MS configuration: (Q1) 129/(Q3) 129
46.4 ± 4.4 cps (RSD: 9.5%), and the intensity of BGN (m/z 129) was lower than 4.0 cps (average: 0.4 cps).

**Application to actual rainwater**

Rainwater samples were prepared according to a previous study on rainwater analysis by AMS measurement [19]. Two samples were prepared: a sample solution spiked with 10 mBq/L of $^{129}$I and 100 mg/L of $^{127}$I, and a solution not spiked with $^{129}$I despite the presence of 100 mg/L of $^{127}$I. In the analysis of actual rainwater, an APEX-Q sample inlet desolvation device was employed. In the results of the quantification analysis, the spiked sample was found to contain 9.8 ± 0.9 mBq/L of $^{129}$I ($^{129}$I/$^{127}$I ratio of $1.63 \times 10^{-8}$) despite the unspiked sample showed less than DL (0.73 mBq/L, 3σ). Both quantification values of $^{127}$I corresponded well with the spiked amount (100 mg/L).

**Conclusions**

$\text{O}_2$ mono-gas is commonly used in the removal of isobaric interference by $^{129}$Xe present in Ar gas cylinders; however, the elimination of polyatomic interference ($^{127}$IH$_2$) caused by the presence of high concentrations of the stable isotope $^{127}$I remains a challenge. The concentration of solutions containing $^{129}$I must be highly enhanced using preconcentration methods due to the ultra-low levels of $^{129}$I concentration in the environment. Thus, a method allowing a high ratio of (BGN of $m/z$ 129)/$^{127}$I is required. In this study, flowing a mixture gas of $\text{O}_2$ and $\text{CO}_2$ into the DRC (Q2) succeeded in removing the interference of $^{129}$Xe as well as polyatomic interference ($^{127}$IH$_2$) in the analysis of radioactive $^{129}$I in ICP–MS/MS. The resulting ratio (BGN of $m/z$ 129)/$^{127}$I was $4.6 \times 10^{-10} \pm 3.3 \times 10^{-10}$, which is equal to the ability to analyze 10 mBq/L of $^{129}$I in the presence of 100 mg/L of stable $^{127}$I without any chemical separation. As a demonstration, spike and recovery analysis for rainwater was performed, and good agreement between the spiked and recovered amounts was achieved.

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**Declarations**

**Conflict of interest** The authors declare no competing financial interest.

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