The Series of Environmental Radioactivity Measuring Methods

AN-Sr
(No.2)

Radiostrontium Analysis

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Radiation Monitoring Division
Radiation Protection Department
The Secretariat of
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NRA JAPAN
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This document was written in Japanese and translated into English.
The Japanese version is the original and the English version was written as a reference.

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Introduction

Strontium (Sr) belongs to the Group 2 elements (alkaline earth metals) of the periodic table. It exhibits similar chemical behavior to its homologous elements, such as calcium (Ca) and barium (Ba). Although there are several radioactive isotopes of Sr, environmental radiation monitoring targets ^{89}Sr and ^{90}Sr because of their half lives and radiation emission properties[*1,*2,*3]. Both nuclides are pure β-emitting nuclides that emit β-rays through beta decay, but not γ-rays. The half lives are 50.563 days for ^{89}Sr and 28.91 years for ^{90}Sr (refer to Appendix A).

Radiostrontium is primarily produced by the fission of uranium 235 (^{235}U) and other nuclides, and is known for its high fission yield. When introduced into the body, it is deposited in bone tissues along with Ca, which may cause exposure of the bone marrow and other effects[*4]. Therefore, alongside radioiodine (I) and radiocesium (Cs), radiostrontium is an important nuclide for exposure dose evaluations and must be monitored in the event it is released into the environment as a result of atmospheric nuclear tests or nuclear power plant accidents.

The first edition of this document was issued in 1960 out of a necessity to assess the environmental impact of radioactive fallout from atmospheric nuclear tests. Over the course of several subsequent revisions, the measuring methods introduced in this document have been used not only for environmental radiation monitoring in general environments but also for measuring radiostrontium in environmental radiation monitoring around facilities such as nuclear power plants. This document defines general methods of environmental radioactivity measurement for the determination of ^{89}Sr and ^{90}Sr, which are key radiostrontium isotopes in environmental radiation monitoring.

The most recent revision was made in 2003 to reflect societal developments and the improved performance of analytical and other instruments. The key revision was designating the ion exchange method, which is highly safe for analytical operations, as the primary analytical method. This revision was made in light of the fact that the primary analysis method, the fuming nitric acid method, uses a reagent that requires careful handling, and the Water Pollution Prevention Act[*5] specified effluent standards for nitric acid compounds and other effluents. Furthermore, the measuring method involving the use of an inductively coupled plasma atomic emission spectrometer (hereinafter referred to as "ICP-AES") was added as a method of determining stable Sr and other nuclides. The scope of application of the ion exchange method has been expanded to include analysis samples that contain up to 5 g of Ca, as well as seawater samples of up to 40 L, due to the addition of pretreatment procedures and an increased amount of ion exchange resin.

[*1] The Nuclear Regulation Authority: NRA Guideline for Emergency Preparedness and Response (fully amended on September 11, 2024)

[*2] Radiation Monitoring Division, the Secretariat of the Nuclear Regulation Authority: Ordinary Monitoring (Supplementary Reference Material for The Guideline for Emergency Preparedness and Response) (Revised on December 21, 2021)

[*3] Radiation Monitoring Division, the Secretariat of the Nuclear Regulation Authority: Emergency Monitoring (Supplementary Reference Material for The Guideline for Emergency Preparedness and Response) (Revised on March 21, 2024)

[*4] N.Vajda, C.Kim:Determination of radiostrontium isotopes:A review of analytical methodology, Appl.Radiat.Isot., 68(12), 2306-2326(2010).

[*5] The purpose of this law is to prevent the pollution of water in public water areas and in ground water by regulating effluent discharged by factories and workplaces into public water areas and the permeation of effluent underground, thereby protecting public health and preserving living conditions.

This revision does not include any significant changes to the primary analytical method. However, the solvent extraction method[*6], which was applied only to milk ash samples, has been deleted because no analytical laboratories use this method, as ^{90}Sr is the only nuclide to be measured and the target samples are limited. The Appendix titled "Strontium 90 Concentrations in Environmental Samples" has been deleted because the database[*7] has been in common use in recent years. When using the method deleted in this revision, refer to the archive of the fourth revision of this document.

On the other hand, descriptions of internal and external quality control have been added to maintain and improve analytical and measuring technology. Furthermore, regarding the uncertainty evaluation method, which has been widely used in recent years, and the calculation method of the lower detection limit, which was not previously documented, a calculation method based on ISO 11929 has been introduced as a means of obtaining the lower detection limit, in addition to the Kaiser method.

In addition, among the methods currently under consideration for application to radiostrontium analysis and measurement, the chemical separation method using solid-phase extraction (hereinafter referred to as the "solid-phase extraction method") and the determination method of radiostrontium using mass spectrometer (hereinafter referred to as "mass spectrometry") are partially described for reference. These methods were developed in response to the need to obtain faster measurement results for emergency monitoring after the accident at the Fukushima Daiichi Nuclear Power Plant of Tokyo Electric Power Company (TEPCO) (hereinafter referred to as the "Fukushima Daiichi Nuclear Power Plant Accident") caused by the Great East Japan Earthquake on March 11, 2011, as the previous methods required approximately one month to obtain measurement results. These methods are not generally used in environmental radiation monitoring due to issues related to detectable levels and device costs. However, they have been documented for reference because they require a smaller quantity of analytical samples than the existing methods, allowing measurement results to be obtained promptly. Although the methods described in this document are used as the standard, the methods introduced in the Information section or the newly developed methods may also be used, depending on the purpose. However, it should be noted that the quality assurance requirements in Chapter 8 must be satisfied even when these methods are used.

In addition to the above, photos and other references have been added to the section on the frequently used ion exchange method, where the operations and sample conditions are difficult to conceptualize based on the text alone. This is because the radiostrontium analysis method requires longer analysis processes than the radiochemical analysis method for other nuclides, making it difficult for analytical laboratories to pass on the analytical techniques. For reference, the Appendix provides the analytical flow of the primary analytical methods (ion exchange, fuming nitric acid, and oxalate analysis) to illustrate the operational flow and provide a general picture.

The following outlines the primary analytical methods used in this document: ion exchange, fuming nitric acid, and oxalate analysis.

Ion exchange method:

This method uses a column filled with strongly acidic cation-exchange resin to separate

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[*6] 三橋俊彦, 阪上正信：牛乳中 ^{90}Sr の ^{90}Y 溶媒抽出による定量, Radioisotopes, 26(10), 673-678(1977).

[*7] For example, the "Environmental Radiation Database" on the "Environmental Radioactivity and Radiation in Japan" website contains the results of the environmental radioactivity survey conducted by the Secretariat of the Nuclear Regulation Authority with the cooperation of relevant ministries and prefectural governments. These results are publicly available. (https://www.envraddb.go.jp/en/)

Sr from other alkaline earth metal elements, such as Ca, by utilizing the difference in the distribution coefficient between the cation metal and the cation exchange resin. Multiple resin columns will be used if a sample contains a large amount of Ca that exceeds the exchange capacity of the resin column.

Fuming nitric acid method:

This method separates Sr from Ca by utilizing the difference in solubility between strontium nitrate and calcium nitrate in nitric acid at a specific gravity of 1.45 (a mass fraction of approx. 77%). Since Ca cannot be completely separated and removed in a single operation, it is necessary to repeat the production of nitrate precipitation. Careful attention is required when handling fuming nitric acid. In addition, a separate operation is required to separate and remove homologous elements other than Ca.

Oxalate method:

This method separates Sr from many cationic metals by using the difference in the solubility of oxalates. The method is simple as it does not involve separating and removing Ca. However, there are disadvantages to the method, such as the need for large capacity instruments and complex analytical operations relative to the ion exchange method. When strontium carbonate precipitate is used as a measurement sample, it will contain calcium carbonate precipitate, as Ca is not separated and removed. Consequently, ^{89}Sr cannot be measured, making ^{90}Sr the only nuclide that can be analyzed using this method.

In addition to the radiostrontium measuring methods in this document, "(AI-LSC) Radionuclide Analysis using Liquid Scintillation Counter" in the Series of Environmental Radioactivity Measuring Methods No. 23 provides different measuring methods for seawater, soil, sea bottom sediment, and ash samples.

This document provides methods generally used in environmental radiation monitoring. However, it does not preclude the use of new technology, including ever-improving analytical technology. As such, the method should be selected according to the purpose and accuracy of the analysis. To aid in method selection, Summary Tables 1.1 and 1.2 show the detectable level of ^{90}Sr[*8] for each sample type using two measuring methods: a method using a low background 2π gas flow counter (hereinafter referred to as "LBC") and a method using a liquid scintillation counter (hereinafter referred to as "LSC") method. The following are guidelines for selecting each measuring method.

Measuring method using LBC:

The target samples are fallout, airborne dust, land water, seawater, soil and sea bottom sediment, and ash. The oxalate method can only measure ^{90}Sr. If the nuclides to be analyzed contain ^{89}Sr, either the ion exchange or fuming nitric acid methods should be selected, as they can be used to measure ^{89}Sr and ^{90}Sr.

Measuring method using LSC:

The target samples are seawater, soil, sea bottom sediment, and ash. Compared to the measuring method using LBC, the preparation of the measurement sample is simpler. Measurements with LSC use liquid samples, which do not require precipitation production. Therefore, ^{89}Sr can also be measured using the oxalate method. In other words, both analytical methods can be used to measure ^{89}Sr and ^{90}Sr.

[*8] The detectable levels for the measuring method using LSC are based on the values indicated in "(AI-LSC) Radionuclide Analysis using Liquid Scintillation Counter" in the Series of Environmental Radioactivity Measuring Methods No. 23.

Summary Table 1.1: Analysis sample quantity and detectable ^{90}Sr level by sample type for the measuring method using LBC

Sample name	Analysis method	Sample quantity analyzed	Detectable level	Unit
Fallout	Ion exchange method Fuming nitric acid method Oxalate method	0.5 m^2	0.07	MBq/km^2
Airborne dust	Ion exchange method Fuming nitric acid method Oxalate method	10000 m^3	0.004	mBq/m^3
Inland water	Ion exchange method Fuming nitric acid method Oxalate method	100 L	0.4	mBq/L
Seawater	Ion exchange method Fuming nitric acid method	40 L	0.9	mBq/L
	Oxalate method	20 L	2	mBq/L
Soil and sea bottom sediment	Ion exchange method Fuming nitric acid method Oxalate method	100 g (dry soil)	0.4	Bq/kg (dry soil)
Ash sample	Ion exchange method Fuming nitric acid method Oxalate method	Equivalent to 1 kg of raw sample	0.04	Bq/kg (raw)

* The following conditions apply: the LBC counting efficiency for ^{90}Y is 25%; the measurement time is 60 minutes; the background counting rate is 0.4 cpm; and the Sr recovery rate is 80%.

Summary table 1.2: Analysis sample quantity and detectable ^{90}Sr level by sample type for the measuring method using LSC

Sample name	Analysis method	Sample quantity analyzed	Detectable level	Unit
Seawater	Ion exchange method	2 L	30	mBq/L
	Oxalate method	10 L	5	mBq/L
Soil and sea bottom	Ion exchange method	100 g (dry soil)	0.5	Bq/kg (dry soil)

sediment	Oxalate method			
Ash sample	Ion exchange method Oxalate method	10 g	0.005	Bq/g (ash)

* The following conditions apply: the LSC counting efficiency for ^{90}Y is 45%; the measurement time is 100 minutes; the background counting rate is 5 cpm; and the Sr recovery rate is 80%; and there is no ^{89}Sr content. To ensure safety in laboratories, the fuming nitric acid method has not been included in "(AI-LSC) Radionuclide Analysis using Liquid Scintillation Counter" in the Series of Environmental Radioactivity Measuring Methods No. 23.

Chapter 1 Fallout

Radiostrontium in fallout is determined using the ion exchange, oxalate, or fuming nitric acid method after samples are concentrated by evaporation or other methods.

1.1 Ion exchange method

1.1.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 2: 18.5 cm diameter
   - No. 5A: 11 cm diameter
   - No. 5B: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter, 18.5 cm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 1G4
10) Chromatography column: With glass joints, ID: 30 mm, column length: 30 cm
11) Separatory funnel: 500 mL, globular shape, 24/40 top and 24/40 bottom joints
12) Separation filter: Glass. Refer to Figure 7.1 below
13) Sample dish: 1 inch diameter x 6.2 mm stainless steel
14) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
15) Filter paper holder: See Figure 7.3 below.
16) Volumetric flask: 50 mL, 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass

and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*9].

1) Pure water: Water purified using ion exchange and other methods[*10]
2) Hydrochloric acid (HCl)[*11,*12,*13]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
   - Hydrochloric acid (1+23): Add one volume of hydrochloric acid to 23 volumes of pure water and mix.
3) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*12,*13]: Special grade reagent
4) Nitric acid (HNO_3)[*11,*12,*13]: Special grade reagent, mass fraction of 60%–61%
   - Aqua regia: Add one volume of nitric acid to three volumes of hydrochloric acid and mix. Prepare it immediately before use.
5) Aqueous ammonia (NH_3)[*11,*12,*13]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.
6) Sodium hydroxide (NaOH)[*12,*13]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water

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[*9] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

[*10] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*11] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*12] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*13] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and measures must be taken to prevent theft, loss, leakage, etc.

to make 1 L.

7) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*11,*12]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.
8) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
9) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*12]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
10) Strontium nitrate ($Sr(NO_3)_2$)[*14]: Special grade reagent
   - Sr carrier solution (Sr^{2+}: 10 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 24.15 g of the solution to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
11) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*15] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
12) Ammonium chloride (NH_4Cl)[*12]: Special grade reagent
13) Ammonium acetate (CH_3COONH_4): Special grade reagent
   - Ammonium acetate solution (15.4 w/v%): Dissolve 154 g of ammonium acetate in pure water to make 1 L.
14) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*13]: Special grade reagent
   - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
   - Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in water to make 1 L.
15) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
   - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.
16) Sodium carbonate (Na_2CO_3): Special grade reagent
   - Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused

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[*14] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*15] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

17) Ethanol (C_2H_5OH)[*12,*16]: Special grade reagent
18) Methanol (CH_3OH)[*12,*13,*16,*17]: Special grade reagent
19) pH test paper: Measurement range pH 1.0–11.0
20) Phenolphthalein ($C_{20}H_{14}O_4$): Special grade reagent, transition range: pH 7.8–pH 10.0
    - Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.
21) Bromocresol green (BCG) solution (0.04 w/v%): For pH test
22) Strongly acidic cation-exchange resin (100 mesh–200 mesh): Crosslinking degree of 8%, 100 mesh–200 mesh
23) Strongly acidic cation-exchange resin (50 mesh–100 mesh): Crosslinking degree of 8%, 50 mesh–100 mesh

1.1.2 Conditioning of the ion exchange resin and column preparation

(1) Strongly acidic cation-exchange resin (50 mesh–100 mesh)

1) Place the strongly acidic cation-exchange resin (50 mesh–100 mesh) in a beaker.
2) Add pure water to allow it to swell, stir well, and let it stand.
3) Remove fine resin, soluble impurities, and other impurities by decantation.
4) Repeat steps 2 and 3 until the supernatant becomes transparent.
5) Add an amount of hydrochloric acid (1+1) approximately equal to the resin capacity, stir well, and allow it to stand.
6) Remove the supernatant by decantation.
7) Use pure water instead of hydrochloric acid (1+1), then repeat steps 5 and 6 until the pH of the supernatant reaches 7.
8) Use sodium hydroxide solution (24 w/v%) instead of hydrochloric acid (1+1), then repeat steps 5 and 6.
9) Use pure water instead of hydrochloric acid (1+1), then repeat steps 5 and 6 until the pH of the supernatant reaches 7.
10) Use sodium hydroxide solution (24 w/v%) instead of hydrochloric acid (1+1), then repeat steps 5 and 6.
11) Use pure water instead of hydrochloric acid (1+1), then repeat steps 5 and 6 until the pH of the supernatant reaches 7.
12) Inject approx. 500 mL of conditioned sodium ion ($^{Na+}$) resin into a chromatography tube [*18]

[*16] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

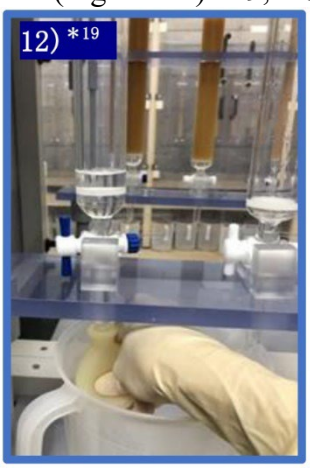
[*17] Class 2 Organic Solvents in the Ordinance on Prevention of Organic Solvent Poisoning: Measures must be taken to reduce the exposure concentrations, such as installing equipment to seal off the source of organic solvent vapor and a local exhaust ventilation system in the workplace.

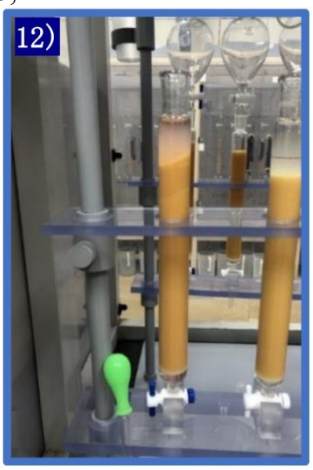
[*18] Newly purchased chromatography tubes contain impurities. For this reason, use tubes that have been soaked in hydrochloric acid (1+10) for at least one night, then wash and rinse with pure water.

with an inner diameter of 5–6 cm to form a resin column[*19,*20,*21].

(2) Strongly acidic cation-exchange resin (100 mesh–200 mesh)

1) Place the strongly acidic cation-exchange resin (100 mesh–200 mesh) in a beaker.
2) Add pure water to allow it to swell, stir well, and let it stand.
3) Remove fine resin, soluble impurities, and other impurities by decantation.
4) Repeat steps 2 and 3 until the supernatant becomes transparent.
5) Add an amount of hydrochloric acid (1+1) approximately equal to the resin capacity, stir well, and allow it to stand.
6) Remove the supernatant by decantation.
7) Use pure water instead of hydrochloric acid (1+1), then repeat steps 5 and 6 until the pH of the supernatant reaches 7.
8) Use sodium hydroxide solution (24 w/v%) instead of hydrochloric acid (1+1), then repeat steps 5 and 6.
9) Use pure water instead of hydrochloric acid (1+1), then repeat steps 5 and 6 until the pH of the supernatant reaches 7.
10) Use hydrochloric acid (1+1) and perform steps 5 and 6.
11) Use pure water instead of hydrochloric acid (1+1), then repeat steps 5 and 6 until the pH of the supernatant reaches 7.
12) Inject conditioned hydrogen ion (H+) resin into a chromatography tube[*18] (Figure 1.1) with an inner diameter of 3 cm so that the length reaches 26 cm ± 0.2 cm, forming a resin column (Figure 1.2) *19,*20,[*22].



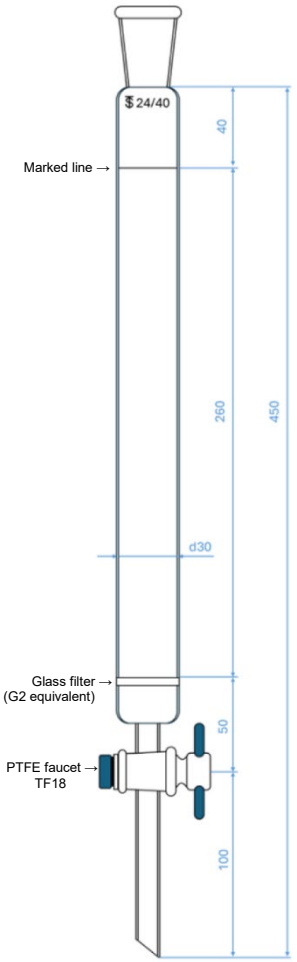


[*19] Fill a dropper with pure water and inject it from the bottom of the column. Fill it up to the glass filter to prevent air bubbles from entering, and then add resin using a Komagome pipette or another instrument. Be careful not to allow air bubbles to enter even when filling the resin.

[*20] When the column approximately half filled with resin, open the faucet and continue filling by pouring the resin.

[*21] After the separation operation, pass 100 mL of pure water, sodium hydroxide solution (24 w/v%), and 1000 mL of pure water through the resin column in that order. This regenerates the resin, enabling repeated use.

[*22] After the separation operation, pass 100 mL of pure water, 1500 mL of hydrochloric acid (1+1), and 1000 mL of pure water through the resin column in that order. This regenerates the resin, enabling repeated use.



Unit: mm

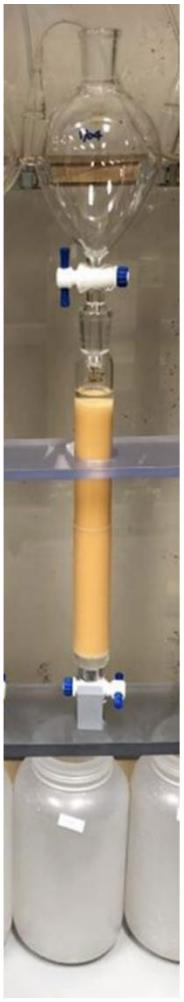


Figure 1.1 Example of a chromatography tube

Figure 1.2 Example of a resin column

1.1.3 Analytical operation

The sample is pretreated using either the evaporation method (a-1)[*23] or the ion exchange method[*24] (a-2) with strongly acidic cation-exchange resin (50–100 mesh). Then, chemical separation is performed using the ion exchange method with a strongly acidic cation-exchange resin (b) (100–200 mesh).

(a-1) Evaporation method

1) Add exactly 5 mL of the Sr carrier solution [*25](Sr^{2+}: 10 mg/mL) to the total volume of the fallout and stir well.
2) Evaporate the mixture to dryness using a 5 L beaker or an evaporating dish (porcelain, approx. 30 cm in diameter) on a hot plate.
3) Dissolve the dry solid matter using hydrochloric acid (1+3) and completely transfer to a 1 L beaker.
4) Add 100 mL of aqua regia, cover the sample with a watch glass, and decompose it by heating [*26] it on a hot plate.

[*23] This method is also used for γ-ray spectrometry and other applications.

[*24] This method is only used when performing radiostrontium analysis.

[*25] When simultaneously analyzing radiocesium by radiochemical analysis, add a Cs carrier as well.

[*26] Set the temperature to approx. 200 °C and evaporate to dryness: Adjust the temperature

5) If organic matter remains in the sample, add nitric acid and decompose it by heating[*27].
6) Evaporate the sample to dryness, add 200 mL of hydrochloric acid (1+1), heat for two to three hours, and cool the mixture.
7) Perform vacuum filtration using a Büchner funnel and filter paper (No. 5C), then wash with pure water.
8) Transfer the filtrate and the washings to a 1 L beaker and add 5 mL of the Ca carrier solution (Ca^{2+}: 50 mg/mL). Then, add pure water to make approx. 700 mL of analytical sample.

(a-2) Ion exchange method using strongly acidic cation-exchange resin (50 mesh–100 mesh)[*28]

1) Add exactly 5 mL of the Sr carrier solution [*25](Sr^{2+}: 10 mg/mL) to the total volume of the fallout.
2) Mix well and perform vacuum filtration using a Büchner funnel and large filter paper (No. 2).
3) Pass the filtrate through a column of Na^{+} type, strongly acidic cation-exchange resin (50–100 mesh) at a flow rate of approx. 80 mL/min to adsorb the cations. Discard the effluent.
4) Pass 2 L of hydrochloric acid (1+3) at a flow rate of approx. 20 mL/min to elute Sr and other elements. Collect the eluate in a 3 L beaker.
5) Incinerate the filter paper prepared in step 2 by heating it in an electric furnace set at 500°C for approx. five hours.
6) Transfer the ash to a 200 mL beaker and add 20 mL of aqua regia. Then, cover it with a watch glass and evaporate on a hot plate to dryness.
7) Add 40 mL of hydrochloric acid (1+1) to the dry solid matter and heat on a hot plate for approx. 30 minutes.
8) Filter through filter paper (No. 5B) and wash the residue thoroughly with hydrochloric acid (1+11) and warm water.
9) Combine the filtrate and the washings with the eluate obtained in step 4, add 5 mL of the Ca carrier solution (Ca^{2+}: 50 mg/mL), and use this solution as the analysis sample.

(b) Chemical separation through the ion exchange method using a strongly acidic cation-exchange resin (100–200 mesh)[*28]

1) Gradually add sodium hydroxide while stirring the analysis sample to adjust the pH to 10 or higher[*29].
2) Add 20 g of sodium carbonate and stir until it is dissolved.
3) Cover the sample with a watch glass and heat to a boil to mature the precipitate.
4) Allow it to cool and stand until the suspension has settled[*30].

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appropriately, while taking care to avoid dispersion of the sample due to the generation of air bubbles. Precipitate formation: Continue boiling to prevent precipitate from settling.

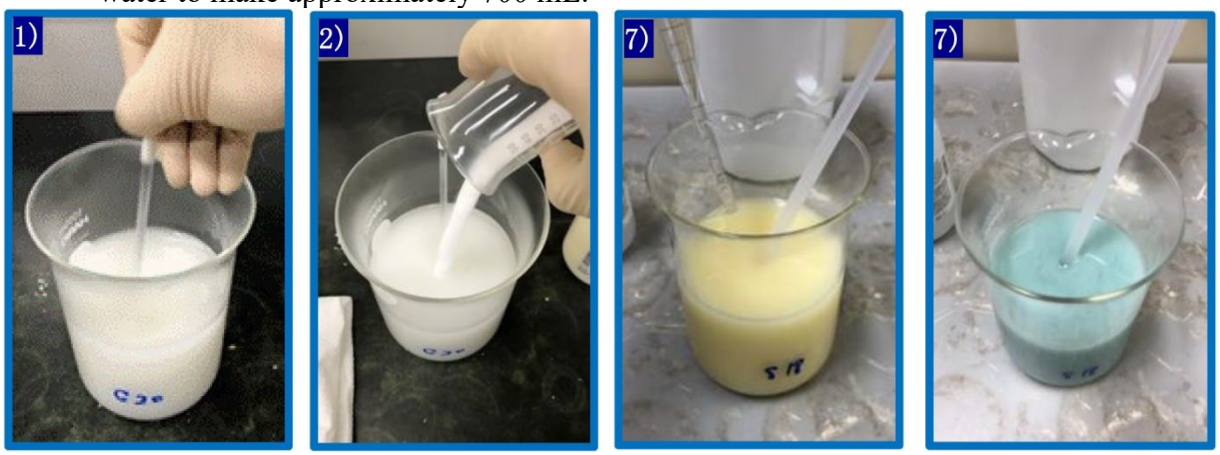
[*27] In some cases, it is possible to determine whether there is any remaining organic matter by checking the color of the dry solid matter. In other cases, however, the color does not change significantly. Therefore, the number of tests may be predetermined based on experience.

[*28] The elution conditions shown in this document are examples. The elution conditions may vary depending on the resin and lot used (refer to Explanation A). Therefore, it is necessary to develop separate elution curves for Sr and Ca for each resin used.

[*29] Exercise caution, as this operation generates heat. The pH test paper should be used to measure pH.

[*30] Add the sodium carbonate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in carbonate precipitate. If turbidity forms, it indicates

5) Remove most of the supernatant by decantation and centrifuge the remainder[*31].
6) Dissolve the precipitate in hydrochloric acid (1+1), transfer to a 1 L beaker, and add pure water to make approximately 700 mL.



7) After heating to a boil, add 10 g of oxalic acid and 5 mL of the BCG solution and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding aqueous ammonia[*32].
8) Reheat to a boil to mature the precipitate. Allow it to cool and stand until the suspension has settled[*33].
9) Remove most of the supernatant by decantation.
10) Perform vacuum filtration using a Büchner funnel and filter paper (No. 5C).
11) Wash the precipitate on the filter paper with 50 mL of ammonium oxalate solution (0.2 w/v%). Add ammonium oxalate solution (0.2 w/v%) in three portions.
12) Transfer the precipitate along with the filter paper to an evaporating dish (porcelain, approx. 10 cm in diameter), dry it in an electric furnace, and heat at 600°C for three hours or more[*34].
13) Transfer the sample to a 1 L beaker and dissolve it in hydrochloric acid (1+3).
14) Evaporate the sample on a hot plate to dryness.

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insufficient sodium carbonate. Therefore, add more sodium carbonate and repeat the operation to generate precipitate. Typically, the sample becomes a white suspension. However, a sample containing a large amount of coexisting iron may become a reddish-brown suspension.

[*31] Precipitate Sr, Ca, etc., as carbonates, and remove Cs, potassium (K), and other elements in the supernatant. The supernatant can be used to analyze radiocesium.

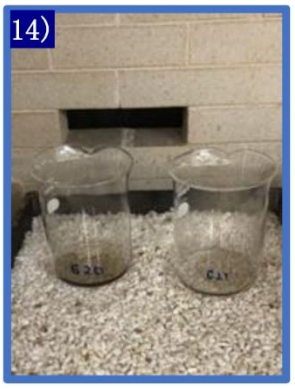
[*32] Note that if the pH is high, Fe, which should be separated, will precipitate as hydroxide. If the pH is high, the sample solution turns blue. Depending on the sample, a red-brown ion precipitate may be generated. In this case, the pH should be adjusted by adding a small amount of hydrochloric acid. Furthermore, note that the amount of precipitate varies depending on the amount of Sr and Ca contained in the sample.

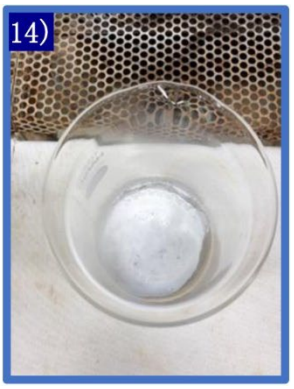
[*33] Add the ammonium oxalate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in the oxalate precipitate. If turbidity forms, it indicates insufficient oxalic acid. Therefore, add more oxalic acid and repeat the operation to generate precipitate.

[*34] In order to avoid damaging the evaporating dish due to a sudden temperature change, allowing a temperature rise time of approx. three hours is recommended.







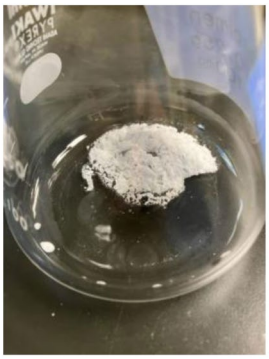


15) Add 200 mL of hydrochloric acid (1+23) to dissolve the dry solid matter.
16) Perform vacuum filtration using a Büchner funnel and filter paper (No. 5C) and wash with hydrochloric acid (1+23).
17) Combine the filtrate and the washings and add hydrochloric acid (1+23) to make approx. 500 mL.
18) Pass the sample solution through an ion-exchange resin column at a flow rate of 4–6 mL/min, and then pass 30 mL of pure water. Discard the effluent[*35].
19) Pass 1100 mL of eluate A [ammonium acetate solution (15.4 w/v%)-methanol (volume ratio 1:1)] at a flow rate of 4–6 mL/min, and discard the effluent[*36].
20) Pass 600 mL of eluate B [ammonium acetate solution (15.4 w/v%)] at a flow rate of 4–6 mL/min to elute Sr[*37].
21) Heat the eluate and evaporate it to dryness.
22) Add 10 mL of pure water and 10 mL of nitric acid to dissolve the dry solid matter, then evaporate again to dryness[*38].
23) Dissolve the dry solid matter in 20 mL of pure water and transfer to a 100 mL beaker.
24) Add 1 mL of the iron(III) chloride solution (Fe^{3+}: 5 mg/mL), 1 g of ammonium chloride, and 0.5 mL of phenolphthalein solution, and heat the mixture until it boils to drive off the

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[*35] With the hydrochloric acid (1+23) solution, Sr, iron (Fe), radium (Ra), Ba, Ca, bismuth (Bi), lead (Pb), and other elements are adsorbed onto the resin and retained.
[*36] When using eluate A, elements such as Ca, Bi, and Pb are eluted.
[*37] When eluate B is used, Sr is eluted. Fe, Ra, Ba, and other elements remain on the resin, but because they gradually elute, it is necessary to establish an elution curve and adjust the volume of eluent accordingly.
[*38] Smooth strontium nitrate (white crystals) can be observed. If a deliquescent, sticky white precipitate is observed, it indicates insufficient separation of Ca. Therefore, perform the separation operation again using an ion exchange column. The difference in appearance of strontium nitrate mixed with calcium nitrate is shown in the following photos.


Sr:100 mg Ca:0 g


Sr:100 mg Ca:0.25 g

carbon dioxide[*39].

25) Gradually add aqueous ammonia[*40] to the solution to precipitate iron(III) hydroxide until the solution turns red.
26) Add 1 mL of aqueous ammonia and heat the precipitate to mature it.
27) Quickly filter with filter paper (No. 5A) (record the date and time as the "scavenging date and time") and wash the precipitate with warm aqueous ammonia (1+500). Discard the precipitate.
28) When determining Sr using the gravimetric method in 1.1.4 (a), collect the filtrate and the washings in a 100 mL beaker and stir the mixture (hereinafter referred to as "the post-scavenging[*41] solution") well. When determining Sr using ICP-AES or atomic absorption spectrometry as described in 2.1.4 (b) and (c), respectively, collect the filtrate and the washings in a 100 mL volumetric flask up to the mark, and stir the mixture (the post-scavenging solution) well.

1.1.4 Determination of recovered Sr

(a) Gravimetric method

a) When determining only the radioactive concentration of ^{90}Sr:

1) Add 5 mL of ammonium carbonate solution (saturated) to the post-scavenging solution and heat the carbonate precipitate on a hot plate to mature it.
2) Filter the precipitate through a glass filter (1G4) of a known mass.
3) Wash with aqueous ammonia (1+100) and ethanol.
4) Dry the glass filter at 110 °C for one hour.
5) Allow it to cool in a desiccator.
6) Measure the mass of the precipitate and calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg) [*42]

7) Allow the precipitate to stand for more than two weeks.

b) When determining the radioactive concentrations of ^{90}Sr and ^{89}Sr simultaneously:

1) Add 5 mL of ammonium carbonate solution (saturated) to the post-scavenging solution and heat the carbonate precipitate on a hot plate to mature it.
2) Perform vacuum filtration on the precipitate using a separation filter (refer to Figure 7.1) and filter paper of a known mass (No. 5C).
3) Wash with aqueous ammonia (1+100) and ethanol.
4) Dry the precipitate on the filter paper at 110°C for one hour.

[*39] Air bubbles that begin to appear when the sample is heated indicate sufficient heating.

[*40] If the aqueous ammonia contains carbon dioxide, Sr precipitates as a carbonate and is filtered together with the iron hydroxide precipitation. Therefore, use unopened aqueous ammonia containing little or no carbon dioxide.

[*41] Remove ^{90}Y formed from ^{90}Sr in the sample by performing steps 24 to 28. This operation is called scavenging.

[*42] Atomic weight of Sr/formula weight of strontium carbonate = 0.594. Therefore, W_2 = mass of the recovered strontium carbonate precipitate × 0.594.

5) Allow it to cool in a desiccator.
6) Measure the mass of the precipitate and calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg)[*42]

7) Secure the precipitate together with the filter paper using the filter paper holder ring (refer to Figure 7.3) and allow it to stand for more than two weeks.

(b) ICP-AES

a) Internal standard method

1) Measure out exactly 1 mL of the post-scavenging solution, transfer it to a 50 mL volumetric flask, add pure water up to the marked line, and stir well.
2) Measure out exactly 1 mL of the solution prepared in step 1 and place it in a 50 mL volumetric flask. Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L), add pure water up to the marked line, stir well, and use this solution as the sample solution for measurement.
3) Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L) to each of the five 50 mL volumetric flasks. Then, add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to each flask, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Using ICP-AES, measure the ratio (Sr emission intensity/Y emission intensity) of Sr emission intensity (measurement wavelength: 407.771 nm) to Y emission intensity (measurement wavelength: 371.029 nm) for the calibration curve solutions and the measurement sample solutions.
5) Determine the Sr concentration, C_{Sr} (mg/L), of the measurement sample solution from the calibration curve, then determine the recovered Sr content, W_2 (mg), from the dilution factor.
6) Calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg)[*43]

b) Calibration curve method

1) Measure out exactly 1 mL of the post-scavenging solution, transfer it to a 50 mL volumetric flask, add pure water up to the marked line, and stir well.
2) Measure out exactly 1 mL of the solution prepared in step 1 and place it in a 50 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
3) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50

[*43] Sr concentration × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (volume of post-scavenging solution/aliquot volume of sample) × (converted to liters). For example, at this dilution factor, the calculation is $W_2 = C_{Sr} \times 50 \times (50/1) \times (100/1) \times (1/10^3)$ (mg).

mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).

4) Measure the emission intensity (wavelength: 407.771 nm) of the calibration curve solution and the sample solution using ICP-AES.
5) Determine the Sr concentration, C_{Sr} (mg/L), of the measurement sample solution from the calibration curve, then determine the recovered Sr content, W_2 (mg), from the dilution factor.
6) Calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg)[*43]

(c) Atomic absorption spectrometry

1) Measure out exactly 1 mL of the post-scavenging solution, transfer it to a 50 mL volumetric flask, add pure water up to the marked line, and stir well.
2) Measure out exactly 1 mL of the solution prepared in step 1 and place it in a 50 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
3) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50 mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Using an atomic absorption spectrometer, measure the emission intensity (measurement wavelength: 460.7 nm) for the calibration solutions and the measurement sample solutions.
5) Determine the Sr concentration, C_{Sr} (mg/L), of the measurement sample solution from the calibration curve, then determine the recovered Sr content, W_2 (mg), from the dilution factor.
6) Calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg)[*43]

1.2 Fuming nitric acid method

1.2.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Graduated cylinder (or dispenser)
3) Evaporating dish
4) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
5) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
6) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
7) Funnel: 65 mm diameter x 8 x 150 mm
8) Büchner funnel: 90 mm diameter, 110 mm diameter
9) Suction flask: 1 L, borosilicate glass
10) Filtration bell jar
11) Glass filter: 1G4
12) Erlenmeyer flask with a stopper
13) Separation filter: Glass. Refer to Figure 7.1 below
14) Sample dish: 1 inch diameter x 6.2 mm stainless steel
15) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
16) Filter paper holder: See Figure 7.3 below.
17) Volumetric flask: 50 mL, 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*44].

[*44] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be

1) Pure water: Water purified using ion exchange and other methods[*45]
2) Hydrochloric acid (HCl)[*46,*47,*48]: Special grade reagent
- Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
- Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
- Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.

3) Acetic acid (CH_3COOH)[*47,*49]: Special grade reagent
- Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.

4) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*47,*48]: Special grade reagent
5) Nitric acid (HNO_3)[*46,*47,*48]: Special grade reagent, mass fraction of 60%–61%
- Aqua regia: Add one volume of nitric acid to three volumes of hydrochloric acid and mix. Prepare it immediately before use.

6) Fuming nitric acid (1.52)[*46,*47,*48,*50]: Special grade reagent, density: 1.52 g/mL
7) Fuming nitric acid (1.45)[*46,*47,*48]: Special grade reagent, density: 1.45 g/mL
- For preparation: Gradually add three volumes of fuming nitric acid (1.52) to one volume of

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expressed by the mixing ratio with water."

[*45] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*46] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*47] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*48] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

[*49] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*50] Classified as a Category 6 hazardous substance (oxidizing liquids) under the Fire Service Act. Appropriate precautions are required, such as ensuring that the substance does not come into contact with the human body, keeping it away from combustibles and metals, storing it in acid-resistant containers, and preventing exposure to moisture and humidity.

pure water while cooling it in running water[*51]. After cooling it sufficiently in running water, gradually add pure water until the specific gravity reaches 1.45 as measured with a hydrometer.

8) Aqueous ammonia (NH_3)[*46,*47,*48]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.
9) Sodium hydroxide (NaOH)[*47,*48]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
10) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*46,*47]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.
11) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
12) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*47]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
13) Strontium nitrate ($Sr(NO_3)_2$)[*52]: Special grade reagent
   - Sr carrier solution (Sr^{2+}: 10 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 24.15 g of the solution to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
14) Barium hydroxide octahydrate (Ba (OH) 2 · 8H2O)[*47,*48,*53]: Special grade reagent
   - Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.
15) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*54] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
16) Ammonium chloride (NH_4Cl)[*47]: Special grade reagent
17) Potassium chromate (K2CrO4)[*47,*48,*55]: Special grade reagent

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[*51] This process generates significant heat. Gradually add the substance while cooling, exercising caution to avoid overheating and bumping.

[*52] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*53] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*54] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

[*55] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical

- Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.

18) Ammonium acetate (CH_3COONH_4): Special grade reagent
- Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water to make 100 mL.
- Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.

19) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*48]: Special grade reagent
- Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
- Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.

20) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
- Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.

21) Sodium carbonate (Na_2CO_3): Special grade reagent
- Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

22) Ethanol (C_2H_5OH)[*47,*49]: Special grade reagent
23) pH test paper: Measurement range pH 1.0–11.0
24) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
- Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.

25) Bromocresol green (BCG) solution (0.04 w/v%): For pH test
26) Strongly acidic cation-exchange resin (50 mesh–100 mesh): Crosslinking degree of 8%, 50 mesh–100 mesh

1.2.2 Conditioning of the ion exchange resin and column preparation

Same as 1.1.2 (1).

1.2.3 Analytical operation

The sample is pretreated using either the evaporation method (a-1)[*56] or the ion exchange

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Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a system that seals off the source of vapor or dust or installing a local exhaust ventilation system, must be taken to reduce diffusion into the atmosphere.

[*56] This method is also used for γ-ray spectrometry and other applications.

method[*57] (a-2) using a strongly acidic cation-exchange resin (50–100 mesh). Then, chemical separation is performed using the fuming nitric acid method (b).

(a-1) Evaporation method
Same as 1.1.3 (a-1).

(a-2) Ion exchange method using strongly acidic cation-exchange resin (50–100 mesh)
Same as 1.1.3 (a-2).

(b) Fuming nitric acid method

1) Gradually add sodium hydroxide while stirring the analysis sample to adjust the pH to 10 or higher[*58].
2) Add 20 g of sodium carbonate and stir until it is dissolved.
3) Cover the sample with a watch glass and heat to a boil to mature the precipitate.
4) Allow it to cool and stand until the suspension has settled[*59].
5) Remove most of the supernatant by decantation and centrifuge the remainder[*60].
6) Dissolve the precipitate in hydrochloric acid (1+1), then transfer the sample to a 1 L beaker, and add pure water to make approx. 700 mL.
7) After heating to a boil, add 10 g of oxalic acid and 5 mL of the BCG solution and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding aqueous ammonia[*61].
8) Reheat to a boil to mature the precipitate. Allow it to cool and stand until the suspension has settled[*62].
9) Remove as much of the supernatant liquid as possible by decantation[*63]. Discard the supernatant.
10) Gradually add approx. three times the volume of fuming nitric acid (1.52) to the solution containing the precipitate a while cooling it in running water. Then, stir for an additional two to three minutes.
11) Cover it with a watch glass and allow it to stand in a water bath overnight to settle the suspension.
12) Remove the supernatant by decantation and filter the remainder through a glass filter (1G4) under vacuum. Discard the supernatant and the filtrate.

[*57] This method is only used when performing radiostrontium analysis.

[*58] Exercise caution, as this operation generates heat. The pH test paper should be used to measure pH.

[*59] Add the sodium carbonate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in carbonate precipitate. If turbidity forms, it indicates insufficient sodium carbonate. Therefore, add more sodium carbonate and repeat the operation to generate precipitate.

[*60] The supernatant can be used to analyze radiocesium.

[*61] Exercise caution, as Fe precipitates as a hydroxide at a high pH.

[*62] Add the ammonium oxalate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in the oxalate precipitate. If turbidity forms, it indicates insufficient oxalic acid. Therefore, add more oxalic acid and repeat the operation to generate precipitate.

[*63] If a large amount of supernatant remains, the amount of fuming nitric acid that needs be added will increase, so remove as much as possible.

13) Wash the strontium nitrate precipitate with a small amount of fuming nitric acid (1.45) and discard the washings.
14) Transfer the glass filter to a filtration bell jar containing a 100 mL volumetric flask with a stopper.
15) Wash the inner wall of the beaker with 20 mL of pure water, transfer the washings to the glass filter, and then perform vacuum filtration.
16) Dissolve the precipitate in 20 mL of pure water and perform vacuum filtration.
17) Wash the glass filter with 20 mL of pure water and perform vacuum filtration.
18) Evaporate the filtrate and the washings gently on a hot plate to dryness and allow them to cool.
19) Add 10 mL of pure water to dissolve the dry solid matter.
20) Gradually add 26 mL of fuming nitric acid (1.52) while cooling it with ice, then cool it in ice water for four to five hours.
21) Filter the precipitate through a glass filter (1G4) under vacuum.
22) Perform steps 13 to 18.
23) If Ca remains, repeat steps 19 to 22 to remove Ca completely[*64].
24) Dissolve the dry solid matter in the conical flask in 10 mL of pure water and transfer to a 100 mL beaker.
25) Wash the conical flask with 30 mL of pure water and combine the washings with the solution.
26) Add 1 mL of acetic acid (7+13), 2 mL of ammonium acetate solution (46 w/v%), and 1 mL of the Ba carrier solution (Ba^{+2}:10 mg/mL)[*65] and heat the mixture[*66].
27) Add 1 mL of the potassium chromate solution (30 w/v%), continue heating to mature the precipitate, and allow it to stand at room temperature until the suspension has settled.
28) Filter the yellow barium chromate precipitate using filter paper (No. 5C) and wash the precipitate with warm ammonium acetate solution (0.6 w/v%). Collect the filtrate and the washings in a 100 mL beaker and discard the precipitate.
29) Add 1 mL of aqueous ammonia to adjust the pH to approx. 9[*67].
30) Add 5 mL of ammonium carbonate solution (saturated) and heat it on a hot plate to mature the precipitate.
31) Filter the precipitate under vacuum using a glass filter (1G4) and wash it with a small amount of aqueous ammonia (1+100).
32) Dissolve the precipitate in the glass filter in a small amount of hydrochloric acid (1+1) and wash the glass filter with water. Combine the filtrate and the washings to make approx. 40 mL.
33) Add 1 mL of the iron(III) chloride solution (Fe^{3+}: 5 mg/mL), 1 g of ammonium chloride, and 0.5 mL of phenolphthalein solution, and heat the mixture to drive off the carbon dioxide.
34) Gradually add aqueous ammonia[*68] to the solution to precipitate iron(III) hydroxide until the solution turns red.
35) Add 1 mL of aqueous ammonia and heat to mature the precipitate.

[*64] Once Ca has been completely removed, the dry solid matter will consist only of square crystals. If the dry solid matter is massive and deliquescent, this indicates Ca contamination. Three or four operations are usually required to remove Ca completely.

[*65] The pH of the solution is 5.0–5.5.

[*66] Heat to a gentle boil.

[*67] A change in the solution color from orange to yellow indicates completion.

[*68] If the aqueous ammonia contains carbon dioxide, Sr precipitates as a carbonate and is filtered together with the iron hydroxide precipitation. Therefore, use unopened aqueous ammonia containing little or no carbon dioxide.

36) Quickly filter with filter paper (No. 5A) (record the date and time as the “scavenging date and time”) and wash the precipitate with warm aqueous ammonia (1+500). Discard the precipitate.
37) Same as 1.1.3 (b), step 28 and onward.

1.2.4 Determination of recovered Sr

Same as 1.1.4.

1.3 Oxalate method

1.3.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 2: 18.5 cm diameter
   - No. 5A: 11 cm diameter
   - No. 5B: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter, 180 mm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 25G4
10) Separation filter: Glass. Refer to Figure 7.1 below
11) Sample dish: 1 inch diameter x 6.2 mm stainless steel
12) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
13) Filter paper holder: See Figure 7.3 below.
14) Volumetric flask: 50 mL, 100 mL, 250 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*69].

[*69] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

1) Pure water: Water purified using ion exchange and other methods[*70]
2) Hydrochloric acid (HCl)[*71,*72,*73]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
3) Hydrogen peroxide (H_2O_2)[*72,*73]: Special grade reagent
4) Acetic acid (CH_3COOH)[*72,*74]: Special grade reagent
   - Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.
5) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*72,*73]: Special grade reagent
6) Nitric acid (HNO_3)[*71,*72,*73]: Special grade reagent, mass fraction of 60%–61%
   - Nitric acid (1+1): Add one volume of nitric acid to one volume of pure water and mix.
   - Aqua regia: Add one volume of nitric acid to three volumes of hydrochloric acid and mix. Prepare it immediately before use.
7) Aqueous ammonia (NH_3)[*71,*72,*73]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+1): Add one volume of aqueous ammonia to one volume of pure water and mix.
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
8) Sodium hydroxide (NaOH)[*72,*73]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water

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[*70] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*71] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*72] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*73] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and measures must be taken to prevent theft, loss, leakage, etc.

[*74] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

to make 1 L.

9) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*71,*72]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.
10) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
11) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*72]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
12) Yttrium oxide (Y_2O_3)[*72]: Mass fraction of 99.99% or higher
   - Y carrier solution (Y^{3+}: 10 mg/mL): Dissolve 1.270 g of yttrium oxide in 50 mL of nitric acid (1+1) while heating. Allow it to cool, then transfer it to a 100 mL volumetric flask, add pure water up to the marked line, and stir well.
13) Lanthanum oxide (La_2O_3): Mass fraction of 99.99% or higher
   - La carrier solution (La^{3+}: 10 mg/mL): Dissolve 11.728 g of lanthanum oxide in 10 mL of hydrochloric acid (1+1). Then, transfer it to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
14) Strontium nitrate ($Sr(NO_3)_2$))[*75]: Special grade reagent
   - Sr carrier solution (Sr^{2+}: 100 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 241.5 g to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
15) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*72,*73,*76]: Special grade reagent
   - Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.
16) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*77] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
17) Potassium chromate (K_2CrO_4)[*72,*73,*78]: Special grade reagent
   - Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.

[*75] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*76] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*77] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

[*78] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a local exhaust ventilation system, must be taken to reduce exposure concentrations.

18) Ammonium acetate (CH_3COONH_4): Special grade reagent
- Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water to make 100 mL.
- Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.

19) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*73]: Special grade reagent
- Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
- Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.

20) Lanthanum nitrate hexahydrate ($La(NO_3)_3 \cdot 6H_2O$)[*75]: Mass fraction of 95.0% or higher
- Lanthanum nitrate solution (0.2 w/v%): Dissolve 2.66 g of lanthanum nitrate hexahydrate in pure water to make 1 L.

21) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
- Ammonium carbonate solution (1 w/v%): Dissolve 1 g of ammonium carbonate in pure water to make 100 mL.

22) Sodium carbonate (Na_2CO_3): Special grade reagent
- Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

23) Strongly acidic cation-exchange resin (50 mesh–100 mesh): Crosslinking degree of 8%, 50 mesh–100 mesh

24) pH test paper: Measurement range pH 1.0–11.0

25) Bromocresol green (BCG) solution (0.04 w/v%): For pH test

1.3.2 Conditioning of the ion exchange resin and column preparation

Same as 1.1.2 (1).

1.3.3 Analytical operation

The sample is pretreated using either the evaporation method (a-1)[*79] or the ion exchange method[*80] (a-2) with a strongly acidic cation-exchange resin (50–100 mesh). Then, chemical separation is performed using the oxalate method (b). Make sure to add exactly 10 mL of the Sr carrier solution (Sr^{2+}+: 100 mg/mL) to each sample.

(a-1) Evaporation method

Same as 1.1.3 (a-1). Make sure to add exactly 10 mL of the Sr carrier solution (Sr^{2+}+: 100 mg/mL).

(a-2) Ion exchange method using strongly acidic cation-exchange resin (50–100 mesh)

[*79] This method is also used for γ-ray spectrometry and other applications.

[*80] This method is only used when performing radiostrontium analysis.

Same as 1.1.3 (a-2). Make sure to add exactly 10 mL of the Sr carrier solution (Sr^{2+}+: 100 mg/mL).

(b) Oxalate method

1) Add sodium hydroxide a little at a time while stirring the analysis sample to adjust the pH to 10 or higher[*81].
2) Add 20 g of sodium carbonate and stir until it is dissolved.
3) Cover the sample with a watch glass and heat to a boil to mature the precipitate.
4) Allow it to cool and stand until the suspension has settled[*82].
5) Remove most of the supernatant by decantation and centrifuge the remainder[*83].
6) Dissolve the precipitate in hydrochloric acid (1+1), transfer to a 1 L beaker, and add pure water to make approximately 700 mL.
7) After heating to a boil, add 10 g of oxalic acid and 5 mL of the BCG solution and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding aqueous ammonia[*84].
8) Reheat to a boil to mature the precipitate. Allow it to cool and stand until the suspension has settled[*85].
9) Age the precipitate by heating for several hours and allow it to cool and stand until the suspension has settled completely.
10) Remove most of the supernatant by decantation.
11) Perform vacuum filtration using a Büchner funnel and filter paper (No. 5C).
12) Wash the precipitate on the filter paper with 50 mL of ammonium oxalate solution (0.2 w/v%). Add ammonium oxalate solution (0.2 w/v%) in three portions.
13) Transfer the precipitate with the filter paper to an evaporating dish (porcelain, approx. 10 cm in diameter), dry it, and heat it at 600°C for three hours.
14) Transfer the sample to a 500 mL beaker, dissolve it in nitric acid, and add pure water to make approx. 200 mL.
15) Add 2 mL of the iron(III) chloride solution (Fe^{3+}: 5 mg/mL) and heat to drive off carbon dioxide.
16) Gradually add aqueous ammonia to adjust the pH to approx. 8 and precipitate iron(III) hydroxide.
17) Continue heating for an additional five minutes to age the precipitate.
18) Quickly filter the precipitate through filter paper (No. 5A) and wash it with aqueous ammonia (1+100). Collect the filtrate and the washings in a 300 mL beaker and discard the precipitate.

[*81] Exercise caution, as this operation generates heat. The pH test paper should be used to measure pH.

[*82] Add the sodium carbonate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in carbonate precipitate. If turbidity forms, it indicates insufficient sodium carbonate. Therefore, add more sodium carbonate and repeat the operation to generate precipitate.

[*83] The supernatant can be used to analyze radiocesium.

[*84] Exercise caution, as Fe precipitates as a hydroxide at a high pH.

[*85] Add the ammonium oxalate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in the oxalate precipitate. If turbidity forms, it indicates insufficient oxalic acid. Therefore, add more oxalic acid and repeat the operation to generate precipitate.

19) Heat the solution to concentrate it to approx. 100 mL.
20) Add 2 mL of the Ba carrier solution (Ba^{2+}: 10 mg/mL) and 2 mL of the ammonium acetate solution (46 w/v%). Adjust pH to between 5.0 and 5.5 by adding aqueous ammonia (1+1) and acetic acid (7+13).
21) Heat to approx. 60°C and add 1 mL of potassium chromate solution (30 w/v%).
22) Continue heating to age the precipitate.
23) Allow it to stand at room temperature for approx. 1 hour.
24) Filter the yellow barium chromate precipitate using filter paper (No. 5C) and wash the precipitate with ammonium acetate solution (0.6 w/v%). Collect the filtrate and the washings in a 1 L beaker and discard the precipitate.
25) Add 4 g of sodium hydroxide to adjust the pH to approx. 10.
26) Add 40 g of ammonium carbonate and heat for 30 minutes to precipitate the white carbonate.
27) Filter the precipitate under vacuum using a glass filter (25G4) and wash with ammonium carbonate (1 w/v%).
28) Dissolve the precipitate in nitric acid (1+1) and wash the glass filter with pure water three times. Combine the filtrate and the washings, then heat to concentrate to approx. 100 mL.
29) Add 2 mL of an iron(III) chloride solution (Fe^{3+}: 5 mg/mL), 0.5 mL of the La carrier solution (La^{2+}: 10 mg/mL), and 0.5 mL of the Y carrier solution (Y^{3+}+: 10 mg/mL) and heat to drive off carbon dioxide.
30) Gradually add aqueous ammonia to adjust the pH to approx. 8 and precipitate iron(III) hydroxide.
31) Continue heating for an additional ten minutes to age the precipitate.
32) Quickly filter the precipitate with filter paper (No. 5A) (record the date and time as the "scavenging date and time") and wash it with aqueous ammonia (1+100). Collect the filtrate and the washings in a 500 mL beaker and discard the precipitate.
33) Heat it to drive off ammonia.
34) Add 7 mL of nitric acid, transfer to a 250 mL volumetric flask, and add pure water to the marked line.

1.3.4 Determination of recovered Sr

Same as 1.1.4. However, note that as the oxalate method does not remove Ca, quantification by gravimetric analysis is not possible. In addition, because this method uses a larger amount of Sr carrier than others, care must also be taken with the aliquot volume, dilution factor, and concentration of the calibration solution. Furthermore, when performing atomic absorption spectrometry (c), lanthanum nitrate solution (0.2 w/v%) must be added.

Chapter 2 Airborne dust

Radiostrontium in airborne dust is determined using the ion exchange, oxalate, or fuming nitric acid method after extraction with hydrochloric acid.

2.1 Ion exchange method

2.1.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 1G4
10) Chromatography column: With glass joints, ID: 30 mm, column length: 30 cm
11) Separatory funnel: 500 mL, globular shape, 24/40 top and 24/40 bottom joints
12) Separation filter: Glass. Refer to Figure 7.1 below
13) Sample dish: 1 inch diameter x 6.2 mm stainless steel
14) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
15) Filter paper holder: See Figure 7.3 below.
16) Volumetric flask: 50 mL, 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is

indicated as the reagent name (a+b)[*86].

1) Pure water: Water purified using ion exchange and other methods[*87]
2) Hydrochloric acid (HCl)[*88,*89,*90]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
   - Hydrochloric acid (1+23): Add one volume of hydrochloric acid to 23 volumes of pure water and mix.
3) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*89,*90]: Special grade reagent
4) Nitric acid (HNO_3)[*88,*89,*90]: Special grade reagent, mass fraction of 60%–61%
5) Aqueous ammonia (NH_3)[*88,*89,*90]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.
6) Sodium hydroxide (NaOH)[*89,*90]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
7) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*88,*89]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the

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[*86] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

[*87] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*88] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*89] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*90] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

exact required concentration.

8) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
9) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*89]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
10) Strontium nitrate ($Sr(NO_3)_2$)[*91]: Special grade reagent
   - Sr carrier solution (Sr^{2+}: 10 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 24.15 g of the solution to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
11) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*92] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved and add pure water to make 1 L.
12) Ammonium chloride (NH_4Cl)[*89]: Special grade reagent
13) Ammonium acetate (CH_3COONH_4): Special grade reagent
   - Ammonium acetate solution (15.4 w/v%): Dissolve 154 g of ammonium acetate in pure water to make 1 L.
14) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*90]: Special grade reagent
   - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
   - Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.
15) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
   - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.
16) Sodium carbonate (Na_2CO_3): Special grade reagent
   - Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.
17) Ethanol (C_2H_5OH)[*89,*93]: Special grade reagent

[*91] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*92] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

[*93] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act.

18) Methanol (CH_3OH)[*89,*90,*93,*94]: Special grade reagent
19) pH test paper: Measurement range pH 1.0–11.0
20) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
    - Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.
21) Bromocresol green (BCG) solution (0.04 w/v%): For pH test
22) Strongly acidic cation-exchange resin (100 mesh–200 mesh): Crosslinking degree of 8%, 100 mesh–200 mesh

2.1.2 Conditioning of the ion exchange resin and column preparation

Same as 1.1.2 (2).

2.1.3 Analytical operation

1) Incinerate the airborne dust samples at 500°C for approx. one day[*95].
2) Transfer the ash to a beaker and add exactly 5 mL of Sr carrier solution [*96](Sr^{2+}: 10 mg/mL).
3) Add enough hydrochloric acid (1+11) to cover the ash and heat on a hot plate for approx. two hours.
4) Perform vacuum filtration using a Büchner funnel and filter paper (No. 5C), wash the residue thoroughly with warm water, and combine the filtrate with the washings.
5) Return the residue to the original beaker and add enough hydrochloric acid (1+11) to cover the ash. Then, repeat steps and 4[*97].
6) Combine the two extracts prepared in steps 4 and 5 and heat to concentrate them to approx. 1.5 L.

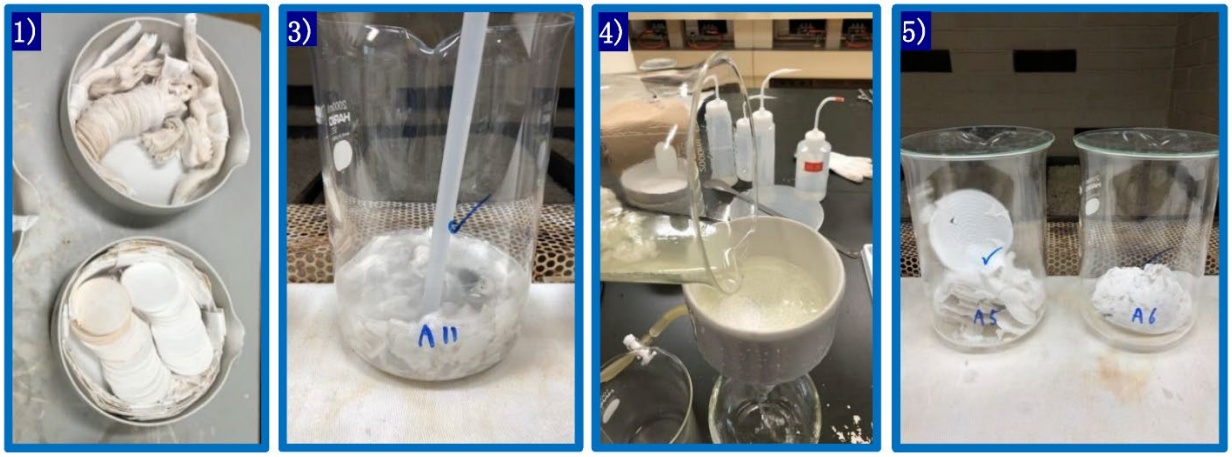
Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*94] Class 2 Organic Solvents in the Ordinance on Prevention of Organic Solvent Poisoning: Measures must be taken to reduce the exposure concentrations, such as installing equipment to seal off the source of organic solvent vapor and a local exhaust ventilation system in the workplace.

[*95] When simultaneously analyzing radiocesium by radiochemical analysis, incinerate the dust at 450°C for approx. one day.

[*96] When simultaneously analyzing radiocesium by radiochemical analysis, add a Cs carrier as well.

[*97] Caution is required for the second extraction, as the glass fibers are loosen and disperse, which increases the viscosity of the sample solution and makes bumping more likely.



7) Add 5 mL of Ca carrier solution (Ca^{2+}: 50 mg/mL).
8) Same as 1.1.3 (b).

2.1.4 Determination of recovered Sr

Same as 1.1.4.

2.2 Fuming nitric acid method

2.2.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Graduated cylinder (or dispenser)
3) Evaporating dish
4) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
5) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
6) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
7) Funnel: 65 mm diameter x 8 x 150 mm
8) Büchner funnel: 90 mm diameter, 110 mm diameter
9) Suction flask: 1 L, borosilicate glass
10) Filtration bell jar
11) Glass filter: 1G4
12) Erlenmeyer flask with a stopper
13) Separation filter: Glass. Refer to Figure 7.1 below
14) Sample dish: 1 inch diameter x 6.2 mm stainless steel
15) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
16) Filter paper holder: See Figure 7.3 below.
17) Volumetric flask: 50 mL, 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is

indicated as the reagent name (a+b)[*98].

1) Pure water: Water purified using ion exchange and other methods[*99]
2) Hydrochloric acid (HCl)[*100,*101,*102]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
3) Acetic acid (CH_3COOH)[*101,*103]: Special grade reagent
   - Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.
4) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*101,*102]: Special grade reagent
5) Nitric acid (HNO_3)[*100,*101,*102]: Special grade reagent, mass fraction of 60%–61%
6) Fuming nitric acid (1.52)[*100,*101,*102,*104]: Special grade reagent, density: 1.52 g/mL

[*98] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

[*99] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*100] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*101] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*102] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

[103] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*104] Classified as a Category 6 hazardous substance (oxidizing liquids) under the Fire Service Act. Appropriate precautions are required, such as ensuring that the substance does not come into contact with the human body, keeping it away from combustibles and metals, storing it in acid-resistant containers, and preventing exposure to moisture and humidity.

7) Fuming nitric acid (1.45)[*100,*101,*102]: Special grade reagent, density: 1.45 g/mL
   - For preparation: Gradually add three volumes of fuming nitric acid (1.52) to one volume of pure water while cooling it in running water[*105]. After cooling it sufficiently in running water, gradually add pure water until the specific gravity reaches 1.45 as measured with a hydrometer.
8) Aqueous ammonia (NH_3)[*100,*101,*102]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.
9) Sodium hydroxide (NaOH)[*101,*102]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
10) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*100,*101]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.
11) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
12) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*101]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate into a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
13) Strontium nitrate ($Sr(NO_3)_2$)[*106]: Special grade reagent
   - Sr carrier solution (Sr^{2+}: 10 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 24.15 g of the solution to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
14) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*101,*102,*107]: Special grade reagent
   - Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.
15) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*108] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
16) Ammonium chloride (NH_4Cl)[*101]: Special grade reagent

[*105] This process generates significant heat. Gradually add the substance while cooling, exercising caution to avoid overheating and bumping.

[*106] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*107] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*108] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

17) Potassium chromate (K_2CrO_4)[*101,*102,*109]: Special grade reagent
   - Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.
18) Ammonium acetate (CH_3COONH_4): Special grade reagent
   - Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water to make 100 mL.
   - Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.
19) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*102]: Special grade reagent
   - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
   - Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.
20) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
   - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.
21) Sodium carbonate (Na_2CO_3): Special grade reagent
   - Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.
22) Ethanol (C_2H_5OH)[*101,*103]: Special grade reagent
23) pH test paper: Measurement range pH 1.0–11.0
24) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
   - Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.
25) Bromocresol green (BCG) solution (0.04 w/v%): For pH test

2.2.2 Analytical operation

Perform the analytical operation steps from 1.2.3 (b) and onward on the sample solution obtained in steps 1 through 7 in 2.1.3.

2.2.3 Determination of recovered Sr

Same as 1.1.4.

[*109] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a system that seals off the source of vapor or dust or installing a local exhaust ventilation system, must be taken to reduce diffusion into the atmosphere.

2.3 Oxalate method

2.3.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 25G4
10) Separation filter: Glass. Refer to Figure 7.1 below
11) Sample dish: 1 inch diameter x 6.2 mm stainless steel
12) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
13) Filter paper holder: See Figure 7.3 below.
14) Volumetric flask: 50 mL, 100 mL, 250 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*110].

[*110] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

1) Pure water: Water purified using ion exchange and other methods[*111]
2) Hydrochloric acid (HCl)[*112,*113 ,*114]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
3) Hydrogen peroxide (H_2O_2)[*113,*114]: Special grade reagent
4) Acetic acid (CH_3COOH)[*113,*115]: Special grade reagent
   - Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.
5) Nitric acid (HNO_3)[*112,*113,*114]: Special grade reagent, mass fraction of 60%–61%
   - Nitric acid (1+1): Add one volume of nitric acid to one volume of pure water and mix.
6) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*113,*114]: Special grade reagent
7) Aqueous ammonia (NH_3)[*112,*113,*114]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+1): Add one volume of aqueous ammonia to one volume of pure water and mix.
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
8) Sodium hydroxide (NaOH)[*113,*114]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
9) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*112,*113]: For ICP analysis (certified reference

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[*111] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*112] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*113] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*114] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and measures must be taken to prevent theft, loss, leakage, etc.

[*115] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

material)

- For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.

10) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
    - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
11) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*113]: Special grade reagent
    - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
12) Yttrium oxide (Y_2O_3)[*113]: Mass fraction of 99.99% or higher
    - Y carrier solution (Y^{3+}: 10 mg/mL): Dissolve 1.270 g of yttrium oxide in 50 mL of nitric acid (1+1) while heating. Allow it to cool, then transfer it to a 100 mL volumetric flask, add pure water up to the marked line, and stir well.
13) Lanthanum oxide (La_2O_3): Mass fraction of 99.99% or higher
    - La carrier solution (La^{3+}: 10 mg/mL): Dissolve 11.728 g of lanthanum oxide in 10 mL of hydrochloric acid. Then, transfer to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
14) Strontium nitrate ($Sr(NO_3)_2$))[*116]: Special grade reagent
    - Sr carrier solution (Sr^{2+}: 100 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 241.5 g to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
15) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*113,*114,*117]: Special grade reagent
    - Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.
16) Calcium carbonate ($CaCO_3$): Special grade reagent
    - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*118] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
17) Potassium chromate (K_2CrO_4)[*113,*114,*119]: Special grade reagent
    - Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.
18) Ammonium acetate (CH_3COONH_4): Special grade reagent
    - Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water

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[*116] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*117] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*118] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

[*119] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a local exhaust ventilation system, must be taken to reduce exposure concentrations.

to make 100 mL.
- Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.

19) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \bullet H_2O$)[*114]: Special grade reagent
- Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
- Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.

20) Lanthanum nitrate hexahydrate ($La(NO_3)_3 \bullet 6H_2O$)[*116]: Mass fraction of 95.0% or higher
- Lanthanum nitrate solution (0.2 w/v%): Dissolve 2.66 g of lanthanum nitrate hexahydrate in pure water to make 1 L.

21) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
- Ammonium carbonate solution (1 w/v%): Dissolve 1 g of ammonium carbonate in pure water to make 100 mL.

22) Sodium carbonate (Na_2CO_3): Special grade reagent
- Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

23) pH test paper: Measurement range pH 1.0–11.0

24) Bromocresol green (BCG) solution (0.04 w/v%): For pH test

2.3.2 Analytical operation

Perform the analytical operation steps from 1.3.3 (b) and onward on the sample solution obtained in steps 1 through 7 in 2.1.3. Make sure to add exactly 10 mL of the Sr carrier solution (Sr^{2+}+: 100 mg/mL).

2.3.3 Determination of recovered Sr

Same as 1.1.4. However, note that as the oxalate method does not remove Ca, quantification by gravimetric analysis is not possible. In addition, because this method uses a larger amount of Sr carrier than others, care must also be taken with the aliquot volume, dilution factor, and concentration of the calibration solution. Furthermore, when performing atomic absorption spectrometry (c), lanthanum nitrate solution (0.2 w/v%) must be added.

Chapter 3 Inland water

Radiostrontium in inland water is determined using the ion exchange, oxalate, or fuming nitric acid methods after evaporating the sample water to dryness. This methodology can also be applied to drinking water, river water, lake water, and other land waters.

3.1 Ion exchange method

3.1.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 1G4
10) Chromatography column: With glass joints, ID: 30 mm, column length: 30 cm
11) Separatory funnel: 500 mL, globular shape, 24/40 top and 24/40 bottom joints
12) Separation filter: Glass. Refer to Figure 7.1 below
13) Sample dish: 1 inch diameter x 6.2 mm stainless steel
14) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
15) Filter paper holder: See Figure 7.3 below.
16) Volumetric flask: 10 mL, 50 mL, 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may

be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*120].

1) Pure water: Water purified using ion exchange and other methods[*121]
2) Hydrochloric acid (HCl)[*122,*123 ,*124]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+23): Add one volume of hydrochloric acid to 23 volumes of pure water and mix.
3) Oxalic acid dihydrate ($H_2C_2O_4$•$2H_2O$)[*123,*124]: Special grade reagent
4) Nitric acid (HNO_3)[*122,*123,*124]: Special grade reagent, mass fraction of 60%–61%
   - Aqua regia: Add one volume of nitric acid to three volumes of hydrochloric acid and mix. Prepare it immediately before use.
5) Aqueous ammonia (NH_3)[*122,*123,*124]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add 500 volumes of aqueous ammonia to one volume of pure water and mix.
6) Sodium hydroxide (NaOH)[*123,*124]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
7) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*122,*123]: For ICP analysis (certified reference material)

[*120] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

[*121] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*122] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*123] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*124] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

- For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.

8) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
9) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*123]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate into a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
10) Strontium nitrate ($Sr(NO_3)_2$)[*125]: Special grade reagent
   - Sr carrier solution (Sr^{2+}: 10 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 24.15 g of the solution to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
11) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*126] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
12) Ammonium chloride (NH_4Cl)[*123]: Special grade reagent
13) Ammonium acetate (CH_3COONH_4): Special grade reagent
   - Ammonium acetate solution (15.4 w/v%): Dissolve 154 g of ammonium acetate in pure water to make 1 L.
14) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*124]: Special grade reagent
   - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
   - Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.
15) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
   - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.
16) Sodium carbonate (Na_2CO_3): Special grade reagent
   - Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

[*125] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*126] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

17) Ethanol (C_2H_5OH)[*123,*127]: Special grade reagent
18) Methanol (CH_3OH)[*123,*124,*127,*128]: Special grade reagent
19) pH test paper: Measurement range pH 1.0–11.0
20) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
    - Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.
21) Bromocresol green (BCG) solution (0.04 w/v%): For pH test
22) Strongly acidic cation-exchange resin (100 mesh–200 mesh): Crosslinking degree of 8%, 100 mesh–200 mesh

3.1.2 Conditioning of the ion exchange resin and column preparation

Same as 1.1.2 (2).

3.1.3 Analytical operation

1) Add exactly 5 mL of the Sr carrier solution (Sr^{2+}: 10 mg/mL)[*130] to 100 mL of the inland water sample[*129] and stir well.
2) Evaporate the mixture to dryness using a 5 L beaker or an evaporating dish (porcelain, approx. 30 cm in diameter).
3) Use hydrochloric acid (1+3) to dissolve the dry solid matter and completely transfer to a 2 L beaker. Then, add 100 mL of aqua regia and decompose the sample carefully by heating it on a hot plate.
4) If organic matter remains in the sample, add more nitric acid and decompose it by heating[*131].
5) Add 200 mL of hydrochloric acid (1+1) to the dry solid matter, heat the mixture for two to three hours, and allow it to cool.
6) Perform vacuum filtration using a Büchner funnel and filter paper (No. 5C), then wash with pure water.

[*127] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*128] Class 2 Organic Solvents in the Ordinance on Prevention of Organic Solvent Poisoning: Measures must be taken to reduce the exposure concentrations, such as installing equipment to seal off the source of organic solvent vapor and a local exhaust ventilation system in the workplace.

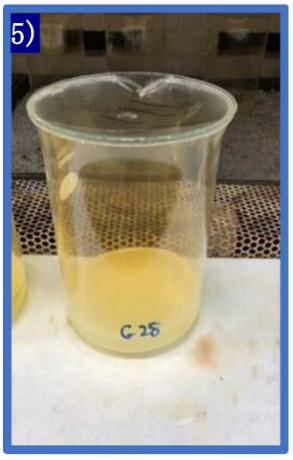
[*130] When simultaneously analyzing radiocesium by radiochemical analysis, add a Cs carrier as well.

[*129] If the sample is expected to contain insoluble matter, filtration may be performed in light of the purpose of the investigation.

[*131] In some cases, it is possible to determine whether there is any remaining organic matter by checking the color of the dry solid matter. In other cases, however, the color does not change significantly. Therefore, the number of tests may be predetermined based on experience. Inland water samples often splatter. Cover the beaker with a watch glass, ensuring there are no gaps.









7) Transfer the filtrate and the washings to a 1 L beaker and add pure water to make approx. 700 mL.
8) Add 5 mL of Ca carrier solution (Ca^{2+}: 50 mg/mL).
9) Same as 1.1.3 (b).

3.1.4 Quantification of stable Sr[*132]

(a) ICP-AES

a) Internal standard method

1) Filter suspended solids in inland water samples using a membrane filter (0.45 μm).
2) Measure out exactly 40 mL of the filtrate and transfer it to a 50 mL volumetric flask. Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L), then add pure water up to the marked line, stir well, and use this solution as the sample solution for measurement.
3) Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L) to each of the five 50 mL volumetric flasks, and then add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to each flask, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Using ICP-AES, measure the ratio (Sr emission intensity/Y emission intensity) of Sr emission intensity (measurement wavelength: 407.771 nm) to Y emission intensity (measurement wavelength: 371.029 nm) for the calibration curve solutions and the measurement sample solutions.
5) Determine the Sr concentration, C_P (mg/L), of the measurement sample solution from the calibration curve, then determine the Sr content, C_{st} (mg/L), in the inland water sample from the dilution factor[*133].

b) Calibration curve method

1) Filter suspended solids in inland water samples using a membrane filter (0.45 μm).
2) Measure out exactly 40 mL of the filtrate and transfer it to a 50 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
3) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50 mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Measure the emission intensity (wavelength: 407.771 nm) of the calibration curve solution and the sample solution using ICP-AES.
5) Determine the Sr concentration, C_P (mg/L), of the measurement sample solution from the

[*132] Typically, 100 L of inland water contains several mg of stable Sr.
[*133] For example, at this dilution factor, C_{st} (mg/L) = C_p (mg/L) × (50/40)

calibration curve, then determine the Sr content, C_{st} (mg/L), in the inland water sample from the dilution factor[*133].

(b) Atomic absorption spectrometry (standard addition method)

1) Add 1 mL of the Ca carrier solution (Ca^{2+}: 50 mg/mL)[*134] to 2 L of the inland water sample.
2) Add 10 g of sodium carbonate, heat to a boil, and age the precipitate.
3) Allow it to cool and stand until the suspension has settled completely.
4) Filter the precipitate under vacuum using a glass filter (1G4) and wash with a small amount of pure water.
5) Transfer the glass filter to a filtration bell jar containing a 50mL volumetric flask. Dissolve the precipitate in a small amount of hydrochloric acid (1+1) and perform vacuum filtration.
6) Wash the glass filter with pure water, add pure water up to the marked line, and stir well.
7) Measure out exactly 5 mL of the solution in step 6 and transfer it to each of the four 10 mL volumetric flasks[*135]. Then, add exactly 0, 1, 2, and 3 mL of the Sr standard solution (Sr^{2+}: 10 mg/L) to each flask, respectively, add pure water up to the marked line, and stir well.
8) Determine the Sr concentration, C_p (mg/L), by measuring the absorbance using an atomic absorption spectrometer (refer to Figure 3.1), then determine the Sr content, C_{st} (mg/L), in the inland water sample from the dilution factor[*136].

[*134] The reagent may contain Sr as an impurity. Measuring and subtracting the reagent blank or purifying the reagent is recommended.

[*135] Dilution to a Sr concentration of approx. 1.5 mg/l is recommended. The aliquot volume shown here is an example.

[*136] Sr concentration × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (1/sample volume) × (converted to liters).

For example, at this dilution factor, CC_{st}(mg/L) = C_p(mg/L) × 10 × (50/5) × (1/2) × ($1/10^3$).

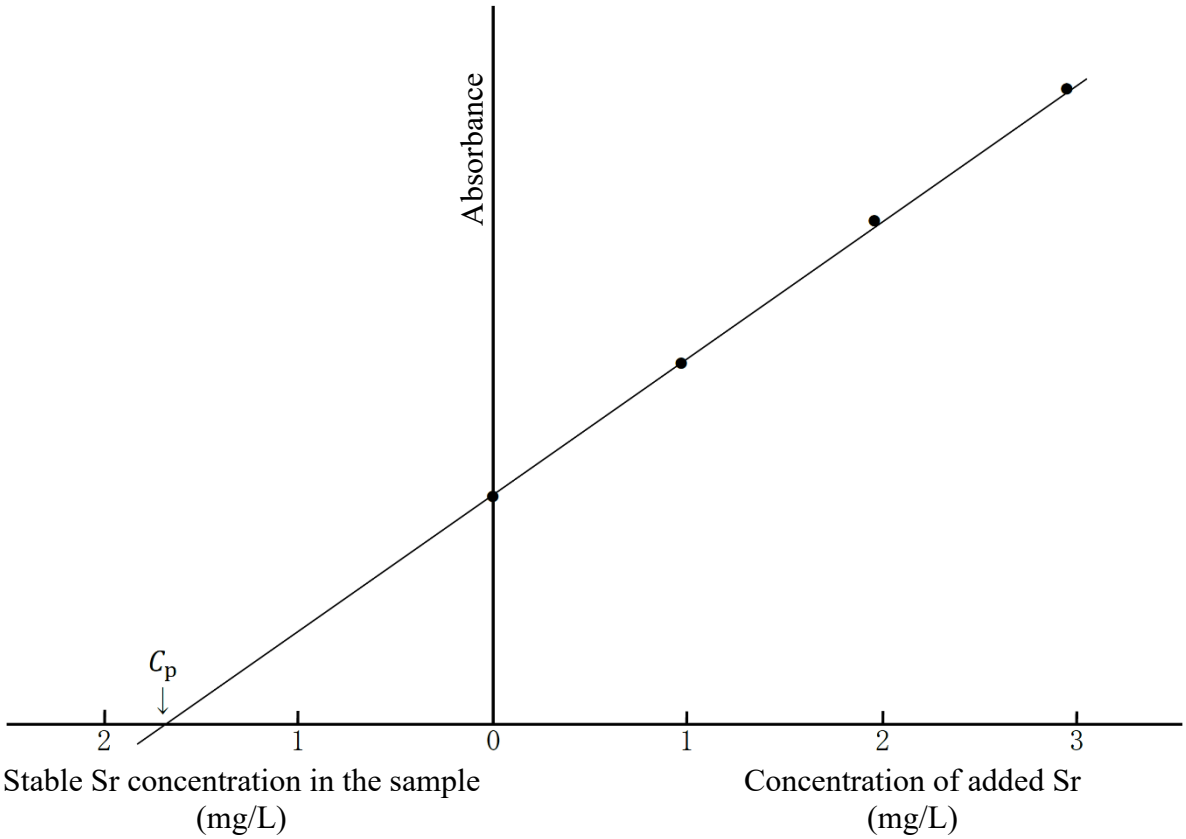


Figure 3.1 Determination of Sr concentration using the standard addition method

3.1.5 Determination of recovered Sr

(a) Gravimetric method

a) When determining only the radioactive concentration of ^{90}Sr:

1) Add 5 mL of ammonium carbonate solution (saturated) to the post-scavenging solution and heat the carbonate precipitate on a hot plate to mature it.
2) Filter the precipitate through a glass filter (1G4) of a known mass.
3) Wash with aqueous ammonia (1+100) and ethanol.
4) Dry the glass filter at 110 °C for one hour.
5) Allow it to cool in a desiccator.
6) Measure the mass of the precipitate and calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1 + C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg)*137
C_{st} : Sr concentration in the inland water sample (mg/L)*138
V : Sample quantity (liters)

7) Allow the precipitate to stand for more than two weeks.

b) When determining the radioactive concentrations of ^{90}Sr and ^{89}Sr simultaneously:

1) Add 5 mL of ammonium carbonate solution (saturated) to the post-scavenging solution and heat the carbonate precipitate on a hot plate to mature it.
2) Perform vacuum filtration on the precipitate using a separation filter (refer to Figure 7.1) and filter paper of a known mass (No. 5C).
3) Wash with aqueous ammonia (1+100) and ethanol.
4) Dry the precipitate on the filter paper at 110°C for one hour.
5) Allow it to cool in a desiccator.
6) Measure the mass of the precipitate and calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1 + C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg)*137
C_{st} : Sr concentration in the inland water sample (mg/L)*138
V : Sample quantity (liters)

7) Secure the precipitate together with the filter paper using the filter paper holder ring (refer

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*137 Atomic weight of Sr/formula weight of strontium carbonate = 0.594. Therefore, W_2 = mass of the recovered strontium carbonate precipitate × 0.594.

*138 When stable Sr has been determined by ICP-AES, the calculation is Sr concentration C_p × (volume of the volumetric flask/aliquot volume of sample). For example, at this dilution factor, C_{st}(mg/L) = C_p(mg/L) × (50/40).
When stable Sr has been determined by atomic absorption spectrometry, the calculation is Sr concentration P × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (1/sample volume) × (converted to liters).
For example, at this dilution factor, C_{st}(mg/L) = C_p (mg/L) × 10 × (50/5) × (1/2) × (1/10^3).

to Figure 7.3) and allow it to stand for more than two weeks.

(b) ICP-AES

a) Internal standard method

1) Measure out exactly 1 mL of the post-scavenging solution, transfer it to a 50 mL volumetric flask, add pure water up to the marked line, and stir well.
2) Measure out exactly 1 mL of the solution prepared in step 1 and place it in a 50 mL volumetric flask. Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L), add pure water up to the marked line, stir well, and use this solution as the sample solution for measurement.
3) Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L) to each of the five 50 mL volumetric flasks, and then add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to each flask, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Using ICP-AES, measure the ratio (Sr emission intensity/Y emission intensity) of Sr emission intensity (measurement wavelength: 407.771 nm) to Y emission intensity (measurement wavelength: 371.029 nm) for the calibration curve solutions and the measurement sample solutions.
5) Determine the Sr concentration, C_p (mg/L), of the measurement sample solution from the calibration curve, then determine the recovered Sr content, W_2 (mg), from the dilution factor.
6) Calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1 + C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg)[*139]
C_{st} : Sr concentration in the inland water sample (mg/L)[*140]
V : Sample quantity (liters)

b) Calibration curve method

1) Measure out exactly 1 mL of the post-scavenging solution, transfer it to a 50 mL volumetric flask, add pure water up to the marked line, and stir well.
2) Measure out exactly 1 mL of the solution prepared in step 1 and place it in a 50 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
3) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50

[*139] Sr concentration × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (volume of post-scavenging solution /aliquot volume of sample) × (converted to liters). For example, at this dilution factor, W_2 (mg) = C_p (mg/L) × 50 × (50/1) × (100/1) × ($1/10^3$)

[*140] When stable Sr has been determined through ICP-AES, the calculation is Sr concentration × (volume of the volumetric flask/aliquot volume of sample).
For example, at this dilution factor, C_{st} (mg/L) = C_p(mg/L) × (50/40).
When stable Sr has been determined by atomic absorption spectrometry, the calculation is Sr concentration × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (1/sample volume) × (converted to liters).
For example, at this dilution factor, C_{st} (mg/L) = C_p (mg/L) × 10 × (50/5) × (1/2) × ($1/10^3$).

mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).

4) Measure the emission intensity (wavelength: 407.771 nm) of the calibration curve solution and the sample solution using ICP-AES.
5) Determine the Sr concentration, C_p (mg/L), of the measurement sample solution from the calibration curve, then determine the recovered Sr content, W_2 (mg), from the dilution factor.
6) Calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1 + C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg)*141
C_{st} : Sr concentration in the inland water sample (mg/L)*142
V : Sample quantity (liters)

(c) Atomic absorption spectrometry

1) Measure out exactly 1 mL of the post-scavenging solution, transfer it to a 50 mL volumetric flask, add pure water up to the marked line, and stir well.
2) Measure out exactly 1 mL of the solution prepared in step 1 and transfer it to a 50 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
3) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50 mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Using an atomic absorption spectrometer, measure the absorbance (measurement wavelength: 460.7 nm) for the calibration solutions and the measurement sample solutions.
5) Determine the Sr concentration, C_p (mg/L), of the measurement sample solution from the calibration curve, then determine the recovered Sr content, W_2 (mg), from the dilution factor.

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*141 Sr concentration × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (volume of post-scavenging solution /aliquot volume of sample) × (converted to liters). For example, at this dilution factor, W_2 (mg) = C_p (mg/L) × 50 × (50/1) × (100/1) × ($1/10^3$)

*142 When stable Sr has been determined through ICP-AES, the calculation is Sr concentration × (volume of the volumetric flask/aliquot volume of sample).
For example, at this dilution factor, C_{st}(mg/L) = C_p (mg/L) × (50/40).
When stable Sr has been determined by atomic absorption spectrometry, the calculation is Sr concentration × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (1/sample volume) × (converted to liters).
For example, at this dilution factor, C_{st} (mg/L) = C_p (mg/L) × 10 × (50/5) × (1/2) × ($1/10^3$).

6) Calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{W_1 + C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_1 : Amount of Sr carrier added (mg)
W_2 : Recovered Sr content (mg)*143
C_{st} : Amount of Sr in the inland water sample (mg/L)*142
V : Sample quantity (liters)

*143 Sr concentration × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (volume of post-scavenging solution/aliquot volume of sample) × (converted to liters).
For example, at this dilution factor, W_2 (mg) = C_p (mg/L) × 50 × (50/1) × (100/1) × ($1/10^3$)

3.2 Fuming nitric acid method

3.2.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Graduated cylinder (or dispenser)
3) Evaporating dish
4) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
5) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
6) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
7) Funnel: 65 mm diameter x 8 x 150 mm
8) Büchner funnel: 90 mm diameter, 110 mm diameter
9) Suction flask: 1 L, borosilicate glass
10) Filtration bell jar
11) Glass filter: 1G4
12) Erlenmeyer flask with a stopper
13) Separation filter: Glass. Refer to Figure 7.1 below
14) Sample dish: 1 inch diameter x 6.2 mm stainless steel
15) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
16) Filter paper holder: See Figure 7.3 below.
17) Volumetric flask: 10 mL, 50 mL, 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*144].

[*144] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

1) Pure water: Water purified using ion exchange and other methods[*145]
2) Hydrochloric acid (HCl)[*146,*147,*148]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
3) Acetic acid (CH_3COOH)[*147,*149]: Special grade reagent
   - Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.
4) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*147,*148]: Special grade reagent
5) Nitric acid (HNO_3)[*146,*147,*148]: Special grade reagent, mass fraction of 60%–61%
   - Aqua regia: Add one volume of nitric acid to three volumes of hydrochloric acid and mix. Prepare it immediately before use.
6) Fuming nitric acid (1.52)[*146,*147,*148,*150]: Special grade reagent, density: 1.52 g/mL
7) Fuming nitric acid (1.45)[*146,*147,*148]: Special grade reagent, density: 1.45 g/mL
   - For preparation: Gradually add three volumes of fuming nitric acid (1.52) to one volume of pure water while cooling it in running water[*151]. After cooling it sufficiently in running

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[*145] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*146] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*147] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*148] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

[*149] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*150] Classified as a Category 6 hazardous substance (oxidizing liquids) under the Fire Service Act. Appropriate precautions are required, such as ensuring that the substance does not come into contact with the human body, keeping it away from combustibles and metals, storing it in acid-resistant containers, and preventing exposure to moisture and humidity.

[*151] This process generates significant heat. Gradually add the substance while cooling, exercising

water, gradually add pure water until the specific gravity reaches 1.45 as measured with a hydrometer.

8) Aqueous ammonia (NH_3)[*146,*147,*148]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.
9) Sodium hydroxide (NaOH)[*147,*148]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
10) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*146,*147]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.
11) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
12) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*147]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
13) Strontium nitrate ($Sr(NO_3)_2$)[*152]: Special grade reagent
   - Sr carrier solution (Sr^{2+}: 10 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 24.15 g of the solution to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
14) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*147,*148,*153]: Special grade reagent
   - Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.
15) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*154] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved and add pure water to make 1 L.
16) Ammonium chloride (NH_4Cl)[*147]: Special grade reagent

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caution to avoid overheating and bumping.

[*152] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*153] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*154] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

17) Potassium chromate (K2CrO4)[*147,*148,*155]: Special grade reagent
   - Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.
18) Ammonium acetate (CH_3COONH_4): Special grade reagent
   - Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water to make 100 mL.
   - Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.
19) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \bullet H_2O$)[*148]: Special grade reagent
   - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
   - Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.
20) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
   - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.
21) Sodium carbonate (Na_2CO_3): Special grade reagent
   - Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.
22) Ethanol (C_2H_5OH)[*147,*149]: Special grade reagent
23) pH test paper: Measurement range pH 1.0–11.0
24) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
   - Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.
25) Bromocresol green (BCG) solution (0.04 w/v%): For pH test

3.2.2 Analytical operation

Perform the analytical operation steps from 1.2.3 (b) and onward on the sample solution obtained in steps 1 through 8 in 3.1.3.

3.2.3 Determination of stable Sr

Same as 3.1.4.

[*155] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a system that seals off the source of vapor or dust or installing a local exhaust ventilation system, must be taken to reduce diffusion into the atmosphere.

3.2.4 Determination of recovered Sr

Same as 3.1.5.

3.3 Oxalate method

3.3.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 25G4
10) Separation filter: Glass. Refer to Figure 7.1 below
11) Sample dish: 1 inch diameter x 6.2 mm stainless steel
12) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
13) Filter paper holder: See Figure 7.3 below.
14) Volumetric flask: 10 mL, 50 mL, 100 mL, 250 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*156].

1) Pure water: Water purified using ion exchange and other methods[*157]

[*156] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

[*157] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General

2) Hydrochloric acid (HCl)[*158,*159,*160]: Special grade reagent
- Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
- Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
- Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.

3) Hydrogen peroxide (H_2O_2)[*159,*160]: Special grade reagent
4) Acetic acid (CH_3COOH)[*159,*161]: Special grade reagent
- Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.

5) Nitric acid (HNO_3)[*159,*160]: Special grade reagent, mass fraction of 60%–61%
- Nitric acid (1+1): Add one volume of nitric acid to one volume of pure water and mix.
- Aqua regia: Add one volume of nitric acid to three volumes of hydrochloric acid and mix. Prepare it immediately before use.

6) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*159,*160]: Special grade reagent
7) Aqueous ammonia (NH_3)[*158,*159,*160]: Special grade reagent, mass fraction of 28.0%–30.0%
- Aqueous ammonia (1+1): Add one volume of aqueous ammonia to one volume of pure water and mix.
- Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.

8) Sodium hydroxide (NaOH)[*159,*160]: Special grade reagent
- Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.

9) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*158,*159]: For ICP analysis (certified reference material)

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rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

*158 Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

*159 Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

*160 Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and measures must be taken to prevent theft, loss, leakage, etc.

*161 Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

- For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.

10) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
    - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
11) Yttrium oxide (Y_2O_3)[*159]: Mass fraction of 99.99% or higher
    - Y carrier solution (Y^{3+}: 10 mg/mL): Dissolve 1.270 g of yttrium oxide in 50 mL of nitric acid (1+1) while heating. Allow it to cool, then transfer it to a 100 mL volumetric flask, add pure water up to the marked line, and stir well.
12) Lanthanum oxide (La_2O_3): Mass fraction of 99.99% or higher
    - La carrier solution (La^{3+}: 10 mg/mL): Dissolve 11.728 g of lanthanum oxide in 10 mL of hydrochloric acid. Then, transfer to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
13) Strontium nitrate ($Sr(NO_3)_2$))[*162]: Special grade reagent
    - Sr carrier solution (Sr^{2+}: 100 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 241.5 g to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
14) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*159,*160,*163]: Special grade reagent
    - Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.
15) Calcium carbonate ($CaCO_3$): Special grade reagent
    - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*164] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
16) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*159]: Special grade reagent
    - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
17) Potassium chromate (K_2CrO_4)[*159,*160,*165]: Special grade reagent
    - Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.
18) Ammonium acetate (CH_3COONH_4): Special grade reagent
    - Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water to make 100 mL.

[*162] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*163] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*164] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

[*165] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a local exhaust ventilation system, must be taken to reduce exposure concentrations.

- Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.

19) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \bullet H_2O$)[*160]: Special grade reagent
- Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
- Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.

20) Lanthanum nitrate hexahydrate ($La(NO_3)_3 \bullet 6H_2O$)[*162]: Mass fraction of 95.0% or higher
- Lanthanum nitrate solution (0.2 w/v%): Dissolve 2.66 g of lanthanum nitrate hexahydrate in pure water to make 1 L.

21) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
- Ammonium carbonate solution (1 w/v%): Dissolve 1 g of ammonium carbonate in pure water to make 100 mL.

22) Sodium carbonate (Na_2CO_3): Special grade reagent
- Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

23) pH test paper: Measurement range pH 1.0–11.0

24) Bromocresol green (BCG) solution (0.04 w/v%): For pH test

3.3.2 Analytical operation

Perform the analytical operation steps from 1.3.3 (b) and onward on the sample solution obtained in steps 1 through 8 in 3.1.3. Make sure to add exactly 10 mL of the Sr carrier solution (Sr^{2+}+: 100 mg/mL).

3.3.3 Determination of stable Sr

Same as 3.1.4.

3.3.4 Determination of recovered Sr

Same as 3.1.5. However, note that as the oxalate method does not remove Ca, quantification by gravimetric analysis is not possible. In addition, because this method uses a larger amount of Sr carrier than others, care must also be taken with the aliquot volume, dilution factor, and concentration of the calibration solution. Furthermore, when performing atomic absorption spectrometry (c), lanthanum nitrate solution (0.2 w/v%) must be added.

Chapter 4 Seawater

Radiostrontium in seawater is determined using the ion exchange, oxalate, or fuming nitric acid method.

4.1 Ion exchange method

4.1.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 5 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 1G4
10) Chromatography column: With glass joints, ID: 30 mm, column length: 30 cm
11) Separatory funnel: 500 mL, globular shape, 24/40 top and 24/40 bottom joints
12) Separation filter: Glass. Refer to Figure 7.1 below
13) Sample dish: 1 inch diameter x 6.2 mm stainless steel
14) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
15) Filter paper holder: See Figure 7.3 below.
16) Volumetric flask: 50 mL, 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is

indicated as the reagent name (a+b)[*166].

1) Pure water: Water purified using ion exchange and other methods[*167]
2) Hydrochloric acid (HCl)[*168,*169,*170]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
   - Hydrochloric acid (1+23): Add one volume of hydrochloric acid to 23 volumes of pure water and mix.
3) Nitric acid (HNO_3)[*168,*169,*170]: Special grade reagent, mass fraction of 60–61%
4) Aqueous ammonia (NH_3)[*168,*169,*170]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.
5) Sodium hydroxide (NaOH)[*169,*170]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
6) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*168,*169]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.

[*166] JIS K 0050: 019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

[*167] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*168] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*169] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*170] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

7) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
8) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*169]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
9) Ammonium chloride (NH_4Cl)[*169]: Special grade reagent
10) Ammonium acetate (CH_3COONH_4): Special grade reagent
   - Ammonium acetate solution (15.4 w/v%): Dissolve 154 g of ammonium acetate in pure water to make 1 L.
11) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*170]: Special grade reagent
   - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
   - Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.
12) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
   - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.
13) Sodium carbonate (Na_2CO_3): Special grade reagent
   - Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.
14) Ethanol (C_2H_5OH)[*169,*171]: Special grade reagent
15) Methanol (CH_3OH)[*169,*170,*171,*172]: Special grade reagent
16) pH test paper: Measurement range pH 1.0–11.0
17) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
   - Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.
18) Bromocresol green (BCG) solution (0.04 w/v%): For pH test

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[*171] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*172] Class 2 Organic Solvents in the Ordinance on Prevention of Organic Solvent Poisoning: Measures must be taken to reduce the exposure concentrations, such as installing equipment to seal off the source of organic solvent vapor and a local exhaust ventilation system in the workplace.

19) Strongly acidic cation-exchange resin (100 mesh–200 mesh): Crosslinking degree of 8%, 100 mesh–200 mesh

4.1.2 Conditioning of the ion exchange resin and column preparation

(1) Preparation of a column for preconcentration[*173]

1) Perform conditioning in the same manner as described in 1 through 7 in 1.1.2.
2) Inject conditioned H^+ resin into a chromatography tube with an inner diameter of 9 cm while taking care not to allow air bubbles to enter, so that the length reaches 26 cm ± 0.2 cm and forms a resin column (hereinafter referred to as the "large column")[*174].

(2) Preparation of analytical columns

Same as 1.1.2 (2).

4.1.3 Analytical operation

1) Measure out 40 L[*176] of seawater sample[*175] and transfer it to a container.
2) Adjust the pH to 1.9 using hydrochloric acid (1+1) and sodium hydroxide solution (24 w/v%).
3) Filter through filter paper (No. 5C).
4) Pass the sample solution through the large column at a flow rate of approx. 30 mL/min, then discard the effluent.
5) Pass 2.5 L of eluate A [ammonium acetate solution (15.4 w/v%)-methanol (volume ratio 1:1)] at a flow rate of approx. 30 mL/min, then discard the effluent.
6) Pass 6 L of hydrochloric acid (1+2) at a flow rate of approx. 30 mL/min to elute Sr and other elements.
7) Equally divide the eluate into two 5 L beakers. Then, perform the following steps on each beaker.
8) Gradually add sodium hydroxide while stirring to adjust the pH to 8 or higher[*177].
9) Add 50 g of sodium carbonate and stir to dissolve it. Then, cover it with a watch glass, heat, and boil to age the precipitate.

[*173] Refer to Explanation B.

[*174] Pass 40 L of seawater sample through the large column for preconcentration. After the preconcentration, pass 10 L of hydrogen chloride (1+1) through the large column, followed by pure water until the sample is neutralized. This regenerates the resin, enabling repeated use.

[*176] When increasing or decreasing the sample volume, the volume of the ion exchange resin and the eluate used in the large column should be adjusted proportionally. For example, for a 20 L seawater sample, the size of the large column is first determined to be 6 cm by calculating the diameter [φ9 cm × (20 L/40 L) 1/2 = φ6.36 cm]. Then, the length is determined to be 29.3 cm ± 0.2 by calculating [$(\varphi 9\ \text{cm})^2 \times 26\ \text{cm} \times (20\ \text{L}/40\ \text{L}) \div (\varphi 6\ \text{cm})^2 = 29.25\ \text{cm}$]. The volume of the eluate can be half (= 20 L out of 40 L).

[*175] The acid concentration of the seawater (1 mL of hydrochloric acid per 1 L of seawater) sampled according to the "(AS) Method for Sampling of Environmental Materials" in the Series of Environmental Radioactivity Measuring Methods, No. 16, is hydrochloric acid (1+1000) acidic. When simultaneously analyzing radiocesium using radiochemical analysis, a Cs carrier is added to the seawater sample. Then, radiocesium is adsorbed and collected using ammonium phosphomolybdate. Ammonium phosphomolybdate is used for radiocesium analysis, and the supernatant is used for radiostrontium analysis. Perform vacuum filtration on the supernatant using a Büchner funnel and filter paper (No. 5C) and pass it through the large column.

[*177] Exercise caution, as this operation generates heat. The pH test paper should be used to measure pH.

10) Allow it to cool and stand until the suspension has settled[*178].
11) Remove most of the supernatant by decantation and centrifuge the remainder.
12) Dissolve the precipitate in 50 mL of combined hydrochloric acid, transfer to a 1 L beaker, and evaporate to dryness.
13) Dissolve the dry solid matter in 200 mL of hydrochloric acid (1+23).
14) Same as 1.1.3 (b), steps 16 to 28.

4.1.4 Determination of stable Sr

(a) ICP-AES

a) Internal standard method

1) Filter suspended solids in seawater samples[*179] using a membrane filter (0.45 μm).
2) Measure out exactly 1 mL of the filtrate and transfer it to a 100 mL volumetric flask. Add exactly 1 mL of the Y standard solution (Y^{3+}: 100 mg/L), add pure water up to the marked line, stir well, and use this solution as the sample solution for measurement.
3) Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L) to each of the five 50 mL volumetric flasks, and then add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to each flask, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Using ICP-AES, measure the ratio (Sr emission intensity/Y emission intensity) of Sr emission intensity (measurement wavelength: 407.771 nm) to Y emission intensity (measurement wavelength: 371.029 nm) for the calibration curve solutions and the measurement sample solutions.
5) Determine the Sr concentration, C_P (mg/L), of the measurement sample solution from the calibration curve, then determine the Sr content, C_{st} (mg/L), of the seawater sample from the dilution factor[*180].

b) Calibration curve method

1) Filter suspended solids in seawater samples[*181] using a membrane filter (0.45 μm).
2) Measure out exactly 1 mL of the filtrate and transfer it to a 100 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
3) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50 mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Measure the emission intensity (wavelength: 407.771 nm) of the calibration curve solution and the sample solution using ICP-AES.
5) Determine the Sr concentration, C_P (mg/L), of the measurement sample solution from the calibration curve, then determine the Sr content, C_{st} (mg/L), of the seawater sample from the dilution factor[*180].

[*178] Add the sodium carbonate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in carbonate precipitate. If turbidity forms, it indicates insufficient sodium carbonate. Therefore, add more sodium carbonate and repeat the operation to generate precipitate.

[*179] Acid is usually added to seawater samples. Here, however, seawater samples are collected and used without adding acid.

[*180] For example, at this dilution factor, C_{st} (mg/L) = C_p (mg/L) × (100/1).

[*181] Acid is usually added to seawater samples. Here, however, seawater samples are collected and used without adding acid.

(b) Atomic absorption spectrometry (standard addition method)

1) Measure out exactly 10 mL of the seawater sample and transfer to each of the four 50 mL volumetric flasks. Then, add exactly 0, 1, 2, and 3 mL of the Sr standard solution (Sr^{2+}: 50 mg/L) to each flask, respectively, add pure water up to the marked line, and stir well.
2) Determine the Sr concentration, C_p (mg/L), by measuring the absorbance using an atomic absorption spectrometer (refer to Figure 3.1), then determine the Sr content, C_{st} (mg/L), in the seawater sample from the dilution factor[*182].

4.1.5 Determination of recovered Sr

(a) Gravimetric method

a) When determining only the radioactive concentration of ^{90}Sr:

1) Add 5 mL of ammonium carbonate solution (saturated) to the post-scavenging solution and heat the carbonate precipitate on a hot plate to mature it.
2) Filter the precipitate through a glass filter (1G4) of a known mass.
3) Wash with aqueous ammonia (1+100) and ethanol.
4) Dry the glass filter at 110 °C for one hour.
5) Allow it to cool in a desiccator.
6) Measure the mass of the precipitate and calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_2 : Recovered Sr content (mg)[*183]
C_{st} : Sr concentration in seawater (mg/L)[*184]
V : Sample quantity (liters)

7) Allow the precipitate to stand for more than two weeks.

b) When determining the radioactive concentrations of ^{90}Sr and ^{89}Sr simultaneously:

1) Add 5 mL of ammonium carbonate solution (saturated) to the post-scavenging solution and heat the carbonate precipitate on a hot plate to mature it.
2) Perform vacuum filtration on the precipitate using a separation filter (refer to Figure 7.1) and filter paper of a known mass (No. 5C).
3) Wash with aqueous ammonia (1+100) and ethanol.
4) Dry the precipitate on the filter paper at 110°C for one hour.
5) Allow it to cool in a desiccator.
6) Measure the mass of the precipitate and calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

[*182] For example, at this dilution factor, C_{st} (mg/L) = C_p (mg/L) × (50/10).

[*183] Atomic weight of Sr/formula weight of strontium carbonate = 0.594. Therefore, W_2 = mass of the recovered strontium carbonate precipitate × 0.594.

[*184] When stable Sr has been determined by ICP-AES, the calculation is Sr concentration C_p × (volume of the volumetric flask/aliquot volume of sample).
For example, at this dilution factor, C_{st}(mg/L) = C_p(mg/L) × (100/1).
When stable Sr is determined by atomic absorption spectrometry, the Sr concentration is C_p × (volume of the volumetric flask/aliquot volume of sample).
For example, at this dilution factor, C_{st} (mg/L) = C_p (mg/L) × (50/10).

$$Y_{Sr} = \frac{W_2}{C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_2 : Recovered Sr content (mg)*183
C_{st} : Amount of Sr in seawater (mg/L)*184
V : Sample quantity (liters)

7) Secure the precipitate together with the filter paper using the filter paper holder ring (refer to Figure 7.3) and allow it to stand for more than two weeks.

(b) ICP-AES

a) Internal standard method

1) Measure out exactly 1 mL of the post-scavenging solution, transfer it to a 100 mL volumetric flask, add pure water up to the marked line, and stir well.
2) Measure out exactly 1 mL of the solution prepared in step 1 and transfer it to a 100 mL volumetric flask. Add exactly 1 mL of the Y standard solution (Y^{3+}: 100 mg/L), add pure water up to the marked line, stir well, and use this solution as the sample solution for measurement.
3) Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L) to each of the five 50 mL volumetric flasks. Then, add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to each flask, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Using ICP-AES, measure the ratio (Sr emission intensity/Y emission intensity) of Sr emission intensity (measurement wavelength: 407.771 nm) to Y emission intensity (measurement wavelength: 371.029 nm) for the calibration curve solutions and the measurement sample solutions.
5) Determine the Sr concentration, C_p (mg/L), of the measurement sample solution from the calibration curve, then determine the recovered Sr content, W_2 (mg), from the dilution factor.
6) Calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_2 : Recovered Sr content (mg)*185
C_{st} : Sr concentration in the sample water (mg/L)*184
V : Sample quantity (liters)

b) Calibration curve method

1) Measure out exactly 1 mL of the post-scavenging solution, transfer it to a 100 mL volumetric flask, add pure water up to the marked line, and stir well.
2) Measure out exactly 1 mL of the solution prepared in step 1 and transfer it to a 100 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
3) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50 mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to

*185 Sr concentration × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (volume of post-scavenging solution/aliquot volume of sample) × (converted to liters). For example, at this dilution factor, W_2 (mg) = C_p (mg/L) × 100 × (100/1) × (100/1) × ($1/10^3$).

prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).

4) Measure the emission intensity (wavelength: 407.771 nm) of the calibration curve solution and the sample solution using ICP-AES.
5) Determine the Sr concentration, C_p (mg/L), of the measurement sample solution from the calibration curve, then determine the recovered Sr content, W_2 (mg), from the dilution factor.
6) Calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_2 : Recovered Sr content (mg)*186
C_{st} : Sr concentration in the sample water (mg/L)*184
V : Sample quantity (liters)

(c) Atomic absorption spectrometry

1) Measure out exactly 1 mL of the post-scavenging solution, transfer it to a 100 mL volumetric flask, add pure water up to the marked line, and stir well.
2) Measure out exactly 1 mL of the solution prepared in step 1 and transfer it to a 100 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
3) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50 mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
4) Using an atomic absorption spectrometer, measure the absorbance (measurement wavelength: 460.7 nm) for the calibration solutions and the measurement sample solutions.
5) Determine the Sr concentration, C_p (mg/L), of the measurement sample solution from the calibration curve, then determine the recovered Sr content, W_2 (mg), from the dilution factor.
6) Calculate the recovery rate of Sr (Y_{Sr}) using the following equation:

$$Y_{Sr} = \frac{W_2}{C_{st} \times V} \times 100$$

Y_{Sr} : Recovery rate of Sr (%)
W_2 : Recovered Sr content (mg)*187
C_{st} : Sr concentration in the sample water (mg/L)*184
V : Sample quantity (liters)

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*186 Sr concentration × volume of measurement solution in volumetric flask × (volume of the dilution volumetric flask/aliquot volume) × (volume of post-scavenging solution/aliquot volume of sample) × (converted to liters). For example, at this dilution factor, W_2 (mg) = C_p (mg/L) × 100 × (100/1) × (100/1) × ($1/10^3$).

*187 Sr concentration × volume of measurement solution in volumetric flask × (volume of post-scavenging solution/aliquot volume of sample) × (converted to liters).
For example, at this dilution factor, W_2 (mg) = C_p (mg/L) × 100 × (100/1) × (100/1) × ($1/10^3$).

4.2 Fuming nitric acid method

4.2.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Agitator
3) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
4) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
5) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
6) Electric dryer: Temperature can be set from room temperature to approximately 120°C
7) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
8) ICP-AES
9) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Graduated cylinder (or dispenser)
3) Evaporating dish
4) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
5) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
6) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
7) Funnel: 65 mm diameter x 8 x 150 mm
8) Büchner funnel: 90 mm diameter, 110 mm diameter
9) Suction flask: 1 L, borosilicate glass
10) Filtration bell jar
11) Glass filter: 1G4
12) Erlenmeyer flask with a stopper
13) Separation filter: Glass. Refer to Figure 7.1 below
14) Sample dish: 1 inch diameter x 6.2 mm stainless steel
15) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
16) Filter paper holder: See Figure 7.3 below.
17) Volumetric flask: 50 mL, 100 mL, 1 L
18) Teflon-coated stainless steel tank

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*188].

[*188] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to

1) Pure water: Water purified using ion exchange and other methods[*189]
2) Hydrochloric acid (HCl)[*190,*191,*192]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
3) Acetic acid (CH_3COOH)[*191,*193]: Special grade reagent
   - Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.
4) Nitric acid (HNO_3)[*190,*191,*192]: Special grade reagent, mass fraction of 60%–61%
5) Fuming nitric acid (1.52)[*190,*191,*192,*194]: Special grade reagent, density: 1.52 g/mL
6) Fuming nitric acid (1.45)[*190,*191,*192]: Special grade reagent, density 1.45 g/mL
   - For preparation: Gradually add three volumes of fuming nitric acid (1.52) to one volume of pure water while cooling it in running water[*195]. After cooling it sufficiently in running

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reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

[*189] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*190] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*191] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*192] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

[*193] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*194] Classified as a Category 6 hazardous substance (oxidizing liquids) under the Fire Service Act. Appropriate precautions are required, such as ensuring that the substance does not come into contact with the human body, keeping it away from combustibles and metals, storing it in acid-resistant containers, and preventing exposure to moisture and humidity.

[*195] This process generates significant heat. Gradually add the substance while cooling, exercising caution to avoid overheating and bumping.

water, gradually add pure water until the specific gravity reaches 1.45 as measured with a hydrometer.

7) Aqueous ammonia (NH_3)[*190,*191,*192]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.
8) Sodium hydroxide (NaOH)[*191,*192]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
9) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*190,*191]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.
10) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
11) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*191]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate into a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
12) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*191,*192,*196]: Special grade reagent
   - Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.
13) Ammonium chloride (NH_4Cl)[*191]: Special grade reagent
14) Potassium chromate (K_2CrO_4)[*191,*192,*197]: Special grade reagent
   - Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.
15) Ammonium acetate (CH_3COONH_4): Special grade reagent
   - Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water to make 100 mL.
   - Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.
16) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*192]: Special grade reagent
   - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.

[*196] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*197] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a system that seals off the source of vapor or dust or installing a local exhaust ventilation system, must be taken to reduce diffusion into the atmosphere.

- Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.

17) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
- Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.

18) Sodium carbonate (Na_2CO_3): Special grade reagent
- Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

19) Ethanol (C_2H_5OH)[*191,*193]: Special grade reagent
20) pH test paper: Measurement range pH 1.0–11.0
21) Bromocresol green (BCG) solution (0.04 w/v%): For pH test
22) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
- Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.

4.2.2 Analytical operation

1) Measure out 40 L of the seawater sample and transfer to a Teflon-coated stainless steel tank. Add 400 g of sodium carbonate while slowly stirring the sample with an agitator.
2) After the sodium carbonate has been dissolved, gradually add hydrochloric acid (1+1) to adjust the pH to 9.7 ± pH 0.1[*198].
3) Heat on a gas burner while stirring and boil for five hours to age the precipitate.
4) Allow it to cool and stand until the suspension has settled[*199].
5) Remove most of the supernatant by decantation and centrifuge the remainder[*200].
6) Dissolve the precipitate in nitric acid and transfer to a 5 L beaker. Heat the sample solution to concentrate it to approx. 1L.
7) After allowing it to cool, perform vacuum filtration using a Büchner funnel and filter paper (No. 5C), then wash with pure water. Return the filtrate and the washings to the original 5 L beaker.
8) Gradually add approx. 3.3 L of fuming nitric acid (with a specific gravity of 1.52) while cooling it in running water.
9) Leave it in running water for an hour, covered with a watch glass. Then, stir with a glass rod to form a nitrate precipitate and allow it to stand in a water bath overnight.
10) After using a hydrometer to confirm that the specific gravity of the supernatant is 1.45 or higher, remove the supernatant by decantation.

[*198] If the pH is 10 or higher, a magnesium precipitate forms, and the volume increases. Use a pH meter to adjust the pH accurately.

[*199] Add the sodium carbonate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in carbonate precipitate. If turbidity forms, it indicates insufficient sodium carbonate. Therefore, add more sodium carbonate and repeat the operation to generate precipitate.

[*200] The supernatant can be used to analyze radiocesium.

11) Dissolve the precipitate with 370 mL of pure water and transfer to a 2 L beaker.
12) Gradually add approx. 990 mL of fuming nitric acid (with a specific gravity of 1.52) while cooling in running water.
13) Cover it with a watch glass and leave it in running water for an hour.
14) Stir with a glass rod to form a nitrate precipitate, then allow it to stand in a water bath overnight.
15) After using a hydrometer to confirm that the specific gravity of the supernatant is 1.45 or higher, remove the supernatant by decantation.
16) Dissolve the precipitate with 240 mL of pure water and transfer to a 1 L beaker.
17) Gradually add approx. 660 mL of fuming nitric acid (with a specific gravity of 1.52) while cooling in running water.
18) Cover it with a watch glass and leave it in running water for an hour.
19) Stir with a glass rod to form a nitrate precipitate, then allow it to stand in a water bath overnight.
20) Same as 1.2.3 (b), step 12 and onward.

4.2.3 Determination of stable Sr

Same as 4.1.4.

4.2.4 Determination of recovered Sr

Same as 4.1.5.

4.3 Oxalate method

4.3.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 25G4
10) Separation filter: Glass. Refer to Figure 7.1 below
11) Sample dish: 1 inch diameter x 6.2 mm stainless steel
12) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
13) Filter paper holder: See Figure 7.3 below.
14) Volumetric flask: 50 mL, 100 mL, 250 mL, 500 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*201].

[*201] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

1) Pure water: Water purified using ion exchange and other methods[*202].
2) Hydrochloric acid (HCl)[*203,*204,*205]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
3) Hydrogen peroxide (H_2O_2)[*204,*205]: Special grade reagent
4) Acetic acid (CH_3COOH)[*204,*206]: Special grade reagent
   - Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.
5) Nitric acid (HNO_3)[*203,*204,*205]: Special grade reagent, mass fraction of 60%–61%
   - Nitric acid (1+1): Add one volume of nitric acid to one volume of pure water and mix.
6) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*204,*205]: Special grade reagent
7) Aqueous ammonia (NH_3)[*203,*204,*205]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+1): Add one volume of aqueous ammonia to one volume of pure water and mix.
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
8) Sodium hydroxide (NaOH)[*204,*205]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
9) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*203,*204]: For ICP analysis (certified reference

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[*202] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*203] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*204] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*205] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and measures must be taken to prevent theft, loss, leakage, etc.

[*206] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

material)
- For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.

10) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
- For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.

11) Yttrium oxide (Y_2O_3)[*204]: Mass fraction of 99.99% or higher
- Y carrier solution (Y^{3+}: 10 mg/mL): Dissolve 1.270 g of yttrium oxide in 50 mL of nitric acid (1+1) while heating. Allow it to cool, then transfer it to a 100 mL volumetric flask, add pure water up to the marked line, and stir well.

12) Lanthanum oxide (La_2O_3): Mass fraction of 99.99% or higher
- La carrier solution (La^{3+}: 10 mg/mL): Dissolve 11.728 g of lanthanum oxide in 10 mL of hydrochloric acid. Then, transfer to a 1 L volumetric flask, add pure water up to the marked line, and stir well.

13) Strontium nitrate ($Sr(NO_3)_2$)[*207]: Special grade reagent
- Sr carrier solution (Sr^{2+}: 100 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 241.5 g to a 1 L volumetric flask, add pure water up to the marked line, and stir well.

14) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*204,*205,*208]: Special grade reagent
- Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.

15) Calcium carbonate ($CaCO_3$): Special grade reagent
- Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*209] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.

16) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*204]: Special grade reagent
- Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.

17) Potassium chromate (K_2CrO_4)[*204,*205,*210]: Special grade reagent
- Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.

18) Ammonium acetate (CH_3COONH_4): Special grade reagent
- Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water

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[*207] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*208] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*209] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

[*210] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a local exhaust ventilation system, must be taken to reduce exposure concentrations.

to make 100 mL.
- Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.

19) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \bullet H_2O$)[*205]: Special grade reagent
- Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
- Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in water to make 1 L.

20) Lanthanum nitrate hexahydrate ($La(NO_3)_3 \bullet 6H_2O$)[*207]: Mass fraction of 95.0% or higher
- Lanthanum nitrate solution (0.2 w/v%): Dissolve 2.66 g of lanthanum nitrate hexahydrate in pure water to make 1 L.

21) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
- Ammonium carbonate solution (1 w/v%): Dissolve 1 g of ammonium carbonate in pure water to make 100 mL.

22) Sodium carbonate (Na_2CO_3): Special grade reagent
- Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

23) pH test paper: Measurement range pH 1.0–11.0

24) Bromocresol green (BCG) solution (0.04 w/v%): For pH test

4.3.2 Analytical operation

1) Measure out 20 L of the seawater sample and divide it into two 10 L beakers. Then, add exactly 5 mL of the Sr carrier solution (Sr^{2+}+: 100 mg/mL) to each beaker.
2) Gradually add sodium hydroxide while stirring the analysis sample to adjust the pH to 10 or higher[*211].
3) Add 100 g of sodium carbonate at a time and heat at approx. 80 °C for one hour while stirring.
4) Allow it to cool and stand until the suspension has settled[*212].
5) Remove most of the supernatant by decantation and centrifuge the remainder[*213].
6) Dissolve and combine the precipitates in hydrochloric acid, transfer to a 5 L beaker, and add water to make approx. 4.5 L.
7) After heating to approx. 80°C, add 100 g of oxalic acid and 5 mL of the BCG solution and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding

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[*211] Exercise caution, as this operation generates heat. The pH test paper should be used to measure pH.

[*212] Add the sodium carbonate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in carbonate precipitate. If turbidity forms, it indicates insufficient sodium carbonate. Therefore, add more sodium carbonate and repeat the operation to generate precipitate.

[*213] The supernatant can be used to analyze radiocesium.

aqueous ammonia[*214].

8) Age the precipitate by heating it at 100°C for several hours. Then, allow it to cool and stand until the suspension has settled completely[*215].
9) Perform vacuum filtration using a Büchner funnel and filter paper (No. 5C).
10) Wash with 50 mL of ammonium oxalate solution (0.2 w/v%) in three portions.
11) Transfer the precipitate with the filter paper to an evaporating dish (porcelain, approx. 10 cm in diameter), dry it, and heat it at 600°C for three hours.
12) Dissolve the sample in nitric acid, transfer to a 1 L beaker, and add water to make approx. 500 mL.
13) Add 8 mL of an iron(III) chloride solution (Fe^{3+}: 5 mg/mL) and heat to drive off carbon dioxide.
14) Gradually add aqueous ammonia to adjust the pH to approx. 8 and precipitate iron(III) hydroxide.
15) Continue heating for an additional ten minutes to age the precipitate.
16) Quickly filter the precipitate through filter paper (No. 5A) and wash it with aqueous ammonia (1+100). Collect the filtrate and the washings in a 1 L beaker and discard the precipitate.
17) Heat the solution to evaporate and concentrate it to approx. 300 mL. Then, add 500 mL of water.
18) Add 4 mL of the Ba carrier solution (Ba^{2+}: 10 mg/mL) and 4 mL of the ammonium acetate solution (46 w/v%). Then, adjust the pH to 5.0–5.5 by adding aqueous ammonia (1+1) and acetic acid (7+13).
19) Heat to approx. 60 °C, add 2 mL of potassium chromate solution (30 w/v%)[*216], and rub the inner wall of the beaker with a policeman (glass rod) to form a precipitate.
20) Continue heating to age the precipitate.
21) Allow it to stand at room temperature for approx. 1 hour.
22) Filter the yellow barium chromate (containing radium) precipitate using filter paper (No. 5C) and wash the precipitate with ammonium acetate solution (0.6 w/v%). Collect the filtrate and the washings in a 3 L beaker and discard the precipitate. Add water to make approx. 2.5 L.
23) Add 8 g of sodium hydroxide to adjust the pH to approx. 10[*217].
24) Add 100 g of ammonium carbonate and heat for one hour to precipitate the white carbonate.
25) Filter the precipitate under vacuum using a glass filter (25G4) and wash the precipitate on the filter paper with 200 mL of ammonium carbonate (1 w/v%). Add ammonium carbonate solution (1 w/v%) in three portions.
26) Dissolve the precipitate in nitric acid (1+1) and wash the glass filter with pure water three times. Combine the filtrate and the washings in a 1 L beaker and concentrate by heating to make approx. 300 mL.
27) Add two drops of hydrogen peroxide solution to reduce chromic acid.
28) Add pure water to make 500 mL.
29) Add 8 mL of the iron(III)chloride solution (Fe^{3+}: 5 mg/mL), 1 mL of the La carrier solution

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[*214] Exercise caution, as Fe precipitates as a hydroxide at a high pH.

[*215] Add the ammonium oxalate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in the oxalate precipitate. If turbidity forms, it indicates insufficient oxalic acid. Therefore, add more oxalic acid and repeat the operation to generate precipitate.

[*216] If no precipitate forms, add potassium chromate solution (30 w/v%).

[*217] A change in the solution color from orange to yellow indicates completion.

(La: 10 mg/mL), and 1 mL of the Y carrier solution (Y^{3+}+: 10 mg/mL), and heat to drive off carbon dioxide.

30) Gradually add aqueous ammonia[*218] to adjust the pH to approx. 8 and precipitate iron(III) hydroxide.
31) Continue heating for an additional ten minutes to age the precipitate.
32) Quickly filter the precipitate with filter paper (No. 5A) (record the date and time as the "scavenging date and time") and wash it with aqueous ammonia (1+100). Collect the filtrate and the washings in a 1 L beaker and discard the precipitate.
33) Heat it to drive off ammonia.
34) Add 14 mL of nitric acid, transfer to a 500 mL volumetric flask, and add pure water to the marked line.

4.3.3 Determination of stable Sr

Same as 4.1.4.

4.3.4 Determination of recovered Sr

Same as 4.1.5. However, note that as the oxalate method does not remove Ca, quantification by gravimetric analysis is not possible. In addition, because this method uses a larger amount of Sr carrier than others, care must also be taken with the aliquot volume, dilution factor, and concentration of the calibration solution. Furthermore, when performing atomic absorption spectrometry (c), lanthanum nitrate solution (0.2 w/v%) must be added.

[*218] If the aqueous ammonia contains carbon dioxide, Sr precipitates as a carbonate and is filtered together with the iron hydroxide precipitation. Therefore, use unopened aqueous ammonia containing little or no carbon dioxide.

Chapter 5 Soil and sea bottom sediment

Radiostrontium in soil and sea bottom sediment is determined using the ion exchange, oxalate, or fuming nitric acid methods after heating the sample at 500°C and performing hydrochloric acid or nitric acid extraction. This methodology can also be applied to riverbed soil and lakebed soil.

5.1 Ion exchange method

5.1.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
   - Glass fiber filter: 185 mm diameter6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter, 185 mm diameter
8) Suction flask: Borosilicate glass, 1 L, 5 L
9) Filtration bell jar
10) Glass filter: 1G4
11) Chromatography column: With glass joints, ID: 30 mm, column length: 30 cm
12) Separatory funnel: 500 mL, globular shape, 24/40 top and 24/40 bottom joints
13) Separation filter: Glass. Refer to Figure 7.1 below
14) Sample dish: 1 inch diameter x 6.2 mm stainless steel
15) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
16) Filter paper holder: See Figure 7.3 below.
17) Volumetric flask: 25 mL, 50 mL, 100 mL, 500 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may

be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*219].

1) Pure water: Water purified using ion exchange and other methods[*220]
2) Hydrochloric acid (HCl)[*221,*222,*223]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
   - Hydrochloric acid (1+23): Add one volume of hydrochloric acid to 23 volumes of pure water and mix.
3) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*222,*223]: Special grade reagent
4) Nitric acid (HNO_3)[*221,*222,*223]: Special grade reagent, mass fraction of 60%–61%
5) Aqueous ammonia (NH_3)[*221,*222,*223]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (aqueous ammonia 1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.
6) Sodium hydroxide (NaOH)[*222,*223]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
7) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*221,*222]: For ICP analysis (certified reference material)

[*219] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

[*220] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*221] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*222] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*223] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and measures must be taken to prevent theft, loss, leakage, etc.

- For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.

8) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
9) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*222]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
10) Strontium nitrate ($Sr(NO_3)_2$)[*224]: Special grade reagent
   - Sr carrier solution (Sr: 10 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 24.15 g of the solution to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
11) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*225] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
12) Ammonium chloride (NH_4Cl)[*222]: Special grade reagent
13) Ammonium acetate (CH_3COONH_4): Special grade reagent
   - Ammonium acetate solution (15.4 w/v%): Dissolve 154 g of ammonium acetate in pure water to make 1 L.
14) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*223]: Special grade reagent
   - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
   - Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.
15) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
   - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.
16) Sodium carbonate (Na_2CO_3): Special grade reagent
   - Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

[*224] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*225] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

17) Ethanol (C_2H_5OH)[*222,*226]: Special grade reagent
18) Methanol (CH_3OH)[*222,*223,*226,*227]: Special grade reagent
19) pH test paper: Measurement range pH 1.0–11.0
20) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
    - Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.
21) Bromocresol green (BCG) solution (0.04 w/v%): For pH test
22) Strongly acidic cation-exchange resin (100 mesh–200 mesh): Crosslinking degree of 8%, 100 mesh–200 mesh

5.1.2 Conditioning of the ion exchange resin and column preparation

Same as 1.1.2 (2).

5.1.3 Analytical operation

1) Measure out 100 g[*228] of air-dried fine soil, place it on an evaporating dish (porcelain, approx. diameter of 15 cm), and heat it at 500°C[*229] for five hours.
2) After allowing it to cool, transfer to a 2 L beaker, add exactly 5 mL of the Sr carrier solution (Sr: 10 mg/mL) [*230], and stir well.
3) Gradually add 1 L of hydrochloric acid or nitric acid, taking care to prevent foaming[*231].
4) Cover it with a watch glass and heat it on a hot plate for three hours until it boils gently.
5) Filter under vacuum by placing a glass fiber filter paper over a Büchner funnel and filter paper (No. 5C)[*232]. Wash the residue with warm pure water.

[*226] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*227] Class 2 Organic Solvents in the Ordinance on Prevention of Organic Solvent Poisoning: Measures must be taken to reduce the exposure concentrations, such as installing equipment to seal off the source of organic solvent vapor and a local exhaust ventilation system in the workplace.

[*228] For samples containing large amounts of sulfur compounds, such as sulfate and sulfide, strontium sulfate may be produced during the analysis process, resulting in a greatly reduced recovery rate. In such cases, add 200 mL of sodium carbonate solution (30 w/v%), stir for 4 hours, then filter and perform acid leaching to prevent loss of recovery rate.

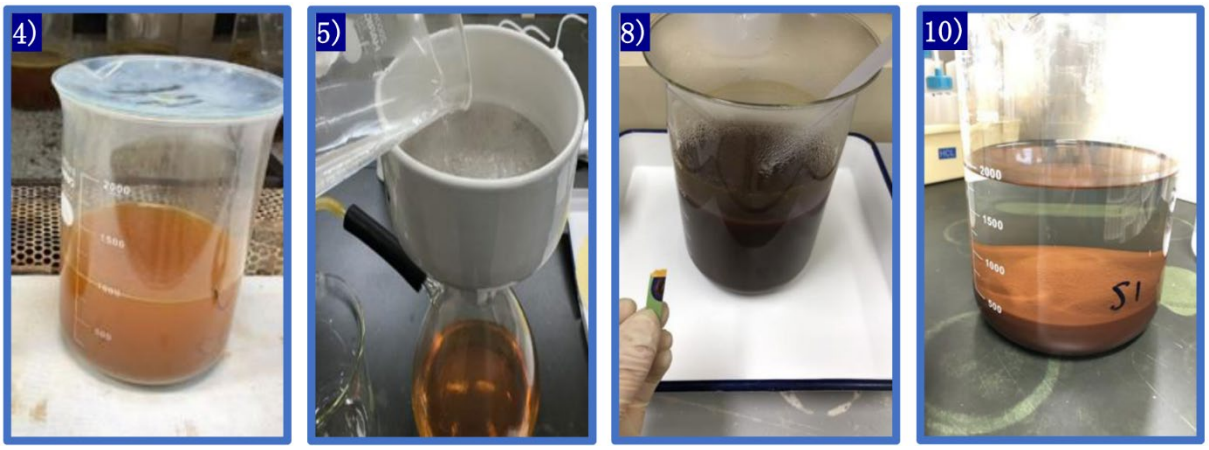
[*229] When simultaneously analyzing radiocesium by radiochemical analysis, incinerate the sample at 450°C for three hours.

[*230] When simultaneously analyzing radiocesium by radiochemical analysis, add a Cs carrier as well.

[*231] In samples rich in calcium carbonate (e.g., shell fragments), vigorous foaming will occur. Take care to prevent splashing and dispersion.

[*232] Since the heated sample solution generates hydrochloric acid gas, the filtration operation must be performed under local exhaust ventilation equipment, such as a fume hood, after the solution has cooled sufficiently. Furthermore, even after cooling, if the hydrochloric acid concentration of the sample solution is 25% or higher, hydrochloric acid gas will still be generated. For this reason, the filtration must be performed under local exhaust ventilation

6) Combine the filtrate and the washings and add pure water to make approx. 2.5 L.
7) Add 5 mL of Ca carrier solution (Ca^{2+}: 50 mg/mL).
8) Gradually add sodium hydroxide while stirring to adjust the pH to 10 or higher[*233].
9) Add 50 g of sodium carbonate and stir to dissolve it. Then, cover it with a watch glass, heat, and boil to age the precipitate.
10) Allow it to cool and stand until the suspension has settled[*234].



11) Remove most of the supernatant by decantation and centrifuge the remainder[*235].
12) Dissolve the precipitate in a slight excess of hydrochloric acid, transfer to a 2 L beaker, and add pure water to make approx. 1.5 L.
13) After heating to a boil, add 50 g of oxalic acid and 5 mL of the BCG solution[*236] and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding aqueous ammonia[*237].

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equipment, such as a fume hood.

[*233] In this operation, a strongly basic reagent is added to a strongly acidic sample solution. As this generates a large amount of neutralization heat, add the reagent carefully in small portions while stirring well. Alternatively, the beaker may be placed in an ice bath. Near neutral pH, the neutralization reaction proceeds rapidly, increasing the likelihood of bumping. Therefore, it is recommended that the operation be performed while checking the pH of the sample solution. The pH test paper should be used to measure pH. When doing so, the iron contained in the sample precipitates as iron hydroxide, and the sample solution turns from yellow and transparent to red-brown and turbid.

[*234] Add the sodium carbonate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in carbonate precipitate. If turbidity forms, it indicates insufficient sodium carbonate. Therefore, add more sodium carbonate and repeat the operation to generate precipitate.

[*235] The supernatant can be used to analyze radiocesium.

[*236] If a sample contains a large amount of iron, adding a large amount of the BCG solution will make it difficult to observe the change in color. Therefore, adjust the amount added appropriately.

[*237] The sample solution turns emerald green due to the iron oxalate complex ion that forms. As a result, the color resembles the blue-green color of the BCG solution with a pH of 4.0–4.2, making it difficult to determine when to stop adjusting the pH. If it is difficult to judge, adjust the color to a blue-green leaning toward blue and keep the pH on the higher side, as this will improve the recovery of strontium. At this point, the mixed iron hydroxide precipitate can be removed by reprecipitation. With insufficient oxalic acid, even if the pH is adjusted to 4.0–4.2, iron not complexed with oxalic acid will precipitate as iron hydroxide. Consequently, the

14) Reheat to age the precipitate. Allow it to cool and stand until the suspension has settled[*238].
15) Transfer as much of the supernatant as possible to a 3 L beaker by decantation[*239]. Transfer the remainder, which contains the precipitate, to a 1 L beaker by dissolving it with hydrochloric acid (1+3).



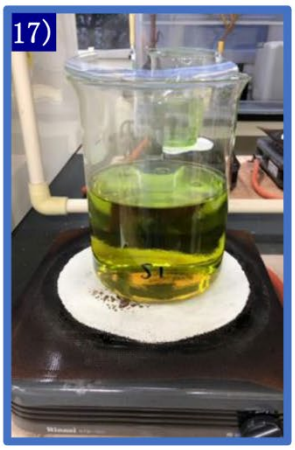






16) Add 5 mL of the Ca carrier solution (Ca^{2+}: 50 mg/mL) to the supernatant.
17) Heat and gradually add hydrochloric acid while stirring to dissolve the white calcium oxalate precipitate produced.
18) After heating to a boil, add 10 g of oxalic acid and 5 mL of the BCG solution[*236] and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding aqueous ammonia[*237].









19) Reheat to age the precipitate.
20) Allow it to cool and stand until the suspension has settled.
21) Discard as much of the supernatant as possible by decantation. Then, add the remainder, which contains the precipitate, to the 1 L beaker prepared in step 15 by dissolving it in hydrochloric acid (1+3).

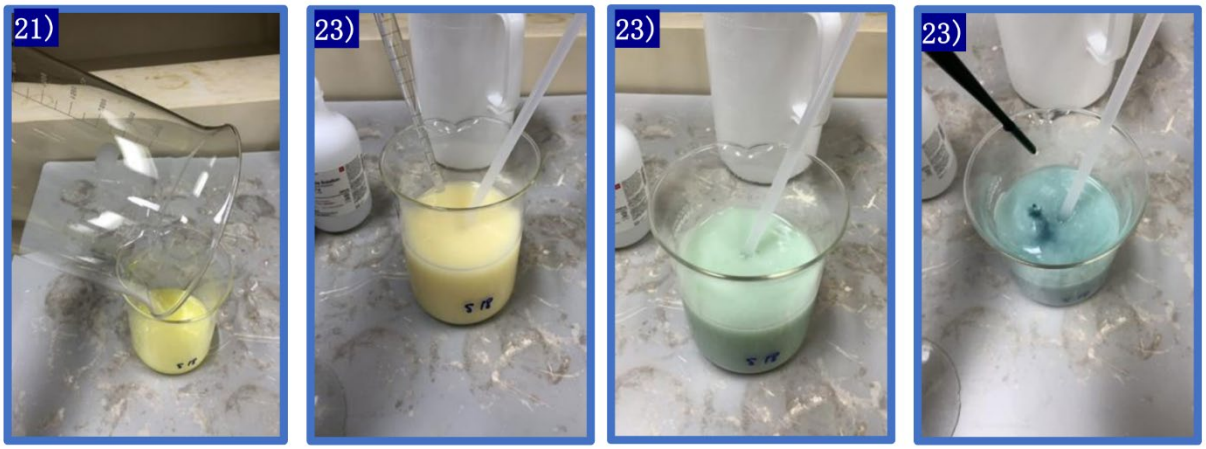
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sample solution turns reddish brown and turbid. In such cases, add approx. 10 g of oxalic acid at a time until the sample solution turns emerald green. Then, since adding oxalic acid makes the solution more acidic, add aqueous ammonia again to adjust the pH to 4.0–4.2.

*238 Add the ammonium oxalate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in the oxalate precipitate. If turbidity forms, it indicates insufficient oxalic acid. Therefore, add more oxalic acid and repeat the operation to generate precipitate.

*239 Since some Sr may remain in the supernatant, perform oxalate precipitation again using the supernatant to improve the recovery rate.

22) Gradually add hydrochloric acid while heating to dissolve the precipitate completely. Then, add pure water to make approx. 600 mL.
23) After heating to a boil, add 5 g of oxalic acid and 5 mL of the BCG solution[*236] and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding aqueous ammonia[*237].



24) Reheat to age the precipitate. Allow it to cool and stand until the suspension has settled.
25) Same as 1.1.3 (b), step 9 and onward.

5.1.4 Determination of stable Sr

(a) ICP-AES

1) Measure out 10 g of air-dried fine soil, place it on an evaporating dish (porcelain, approx. diameter of 5 cm), and heat it at 500°C for five hours.
2) After allowing it to cool, transfer the sample to a 500-mL beaker and gradually add approx. 100 mL of hydrochloric acid or nitric acid.
3) Cover with a watch glass and heat on a hot plate to a gentle boil for 3 hours[*240].
4) Filter under vacuum by placing a glass fiber filter paper over a Büchner funnel and filter paper (No. 5C). Wash the residuewith warm pure water.
5) Combine the filtrate and the washings and transfer to a 500 mL volumetric flask. Then, add pure water to the marked line and stir well to use it as the sample solution.

a) Internal standard method

1) Measure out exactly 5 mL of the sample solution and transfer it to a 50 mL volumetric flask. Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L), add pure water up to the marked line, stir well, and use this solution as the sample solution for measurement.
2) Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L) to each of the five 50 mL volumetric flasks, and then add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to each flask, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
3) Using ICP-AES, measure the ratio (Sr emission intensity/Y emission intensity) of Sr emission intensity (measurement wavelength: 407.771 nm) to Y emission intensity (measurement wavelength: 371.029 nm) for the calibration curve solutions and the

[*240] Since stable Sr is also extracted from soil minerals, different extraction conditions may alter the extraction ratio of stable Sr, affecting the recovery ratio. Therefore, the temperature and time should be appropriately controlled so that extraction can be performed under the same conditions.

measurement sample solutions.

4) Determine the Sr concentration, C_P (mg/L), of the measurement sample solution from the calibration curve, then determine the Sr content, C_{st} (mg/L), of the dried fine soil from the dilution factor.

b) Calibration curve method

1) Measure out exactly 5 mL of the sample solution and transfer it to a 50 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
2) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50 mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
3) Measure the emission intensity (wavelength: 407.771 nm) of the calibration curve solution and the sample solution using ICP-AES.
4) Determine the Sr concentration, C_P (mg/L), of the measurement sample solution from the calibration curve, then determine the Sr content, C_{st} (mg/L), of the dried fine soil from the dilution factor.

(b) Atomic absorption spectrometry (standard addition method)

1) Measure out 10 g of air-dried fine soil, place it on an evaporating dish (porcelain, approx. diameter of 5 cm), and heat it at 500°C for five hours.
2) After allowing it to cool, transfer the sample to a 500-mL beaker and gradually add approx. 100 mL of hydrochloric acid or nitric acid.
3) Cover it with a watch glass and heat it on a hot plate for three hours until it boils gently.
4) Filter under vacuum by placing a glass fiber filter paper over a Büchner funnel and filter paper (No. 5C). Wash the residuewith warm pure water.
5) Combine the filtrate and the washings and add pure water to make approx. 600 mL.
6) Add 1–2mL of the Ca carrier solution [*241](Ca^2: 50 mg/mL) and heat to a boil. Then, add 10 g of oxalic acid and 5 mL of the BCG solution[*242] and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding aqueous ammonia.
7) Reheat to age the precipitate and allow it to cool.
8) After cooling, filter the precipitate under vacuum using a glass filter (1G4) and wash with a small amount of ammonium oxalate solution (0.2 w/v%).
9) Transfer the glass filter to a filtration bell jar containing a 50mL volumetric flask. Dissolve the precipitate in hydrochloric acid (1+1) and perform vacuum filtration.
10) Wash the glass filter with pure water, add pure water up to the marked line, and stir well.
11) Measure out exactly 5 mL of this solution and transfer it to each of the four 25 mL volumetric flasks[*243]. Then, add exactly 0, 1, 2, and 3 mL of the Sr standard solution (Sr^{2+}: 25 mg/mL) to each flask, respectively, add pure water up to the marked line, and stir well.
12) As in 3.1.4 (b), determine the Sr concentration, C_p (mg/L), by measuring the absorbance

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[*241] The reagent may contain Sr as an impurity. Measuring and subtracting the reagent blank or purifying the reagent is recommended. It is not necessary to add it to samples with a high Ca content.

[*242] If a sample contains a large amount of iron, adding a large amount of the BCG solution will make it difficult to observe the change in color. Therefore, adjust the amount added appropriately.

[*243] Dilution to a Sr concentration of approx. 1.5 mg/l is recommended. The aliquot volume shown here is an example.

using an atomic absorption spectrometer, and determine the Sr content, $C_{st,}$ in the air-dried fine soil from the dilution factor[*244].

5.1.5 Determination of recovered Sr

Same as 3.1.5[*245].

[*244] For example, at this dilution factor, C_{st}(mg/g) = C_p (mg/L) × 25 × (50/5) × (1/10) × ($1/10^3$).

[*245] For soil and seafloor soil samples, the Sr concentration (C_{st}) in the sample is expressed in mg/g and the sample quantity (V) is expressed as W_s in g.

5.2 Fuming nitric acid method

5.2.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Graduated cylinder (or dispenser)
3) Evaporating dish
4) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
5) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
6) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
   - Glass fiber filter: 185 mm diameter
7) Funnel: 65 mm diameter x 8 x 150 mm
8) Büchner funnel: 90 mm diameter, 110 mm diameter, 185 mm diameter
9) Suction flask: Borosilicate glass, 1 L, 5 L
10) Filtration bell jar
11) Glass filter: 1G4
12) Erlenmeyer flask with a stopper
13) Separation filter: Glass. Refer to Figure 7.1 below
14) Sample dish: 1 inch diameter x 6.2 mm stainless steel
15) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
16) Filter paper holder: See Figure 7.3 below.
17) Volumetric flask: 25 mL, 50 mL, 100 mL, 500 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*246].

[*246] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be

1) Pure water: Water purified using ion exchange and other methods[*247]
2) Hydrochloric acid (HCl)[*248,*249,*250]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
3) Acetic acid (CH_3COOH)[*249,*251]: Special grade reagent
   - Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.
4) Oxalic acid dihydrate ($H_2C_2O_4•2H_2O$)[*249,*250]: Special grade reagent
5) Nitric acid (HNO_3)[*248,*249,*250]: Special grade reagent, mass fraction of 60%–61%
6) Fuming nitric acid (1.52)[*248,*249,*250,*252]: Special grade reagent, density: 1.52 g/mL
7) Fuming nitric acid (1.45)[*248,*249,*250]: Special grade reagent, density 1.45 g/mL
   - For preparation: Gradually add three volumes of fuming nitric acid (1.52) to one volume of pure water while cooling it in running water[*253]. After cooling it sufficiently in running

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expressed by the mixing ratio with water."

[*247] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*248] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*249] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*250] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

[*251] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*252] Classified as a Category 6 hazardous substance (oxidizing liquids) under the Fire Service Act. Appropriate precautions are required, such as ensuring that the substance does not come into contact with the human body, keeping it away from combustibles and metals, storing it in acid-resistant containers, and preventing exposure to moisture and humidity.

[*253] This process generates significant heat. Gradually add the substance while cooling, exercising caution to avoid overheating and bumping.

water, gradually add pure water until the specific gravity reaches 1.45 as measured with a hydrometer.

8) Aqueous ammonia (NH_3)[*248,*249,*250]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
   - Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.
9) Sodium hydroxide (NaOH)[*249,*250]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
10) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*248,*249]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.
11) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
   - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
12) Strontium nitrate ($Sr(NO_3)_2$)[*254]: Special grade reagent
   - Sr carrier solution (Sr: 10 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 24.15 g of the solution to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
13) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*249,*250,*255]: Special grade reagent
   - Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.
14) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*249]: Special grade reagent
   - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
15) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*256] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
16) Ammonium chloride (NH_4Cl)[*249]: Special grade reagent
17) Potassium chromate (K_2CrO_4)[*249,*250,*257]: Special grade reagent

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[*254] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*255] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*256] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

[*257] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a system that seals off the source of vapor or dust or installing a local exhaust ventilation system, must be taken to reduce diffusion into the

- Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.

18) Ammonium acetate (CH_3COONH_4): Special grade reagent
- Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water to make 100 mL.
- Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.

19) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \bullet H_2O$)[*250]: Special grade reagent
- Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
- Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.

20) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
- Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.

21) Sodium carbonate (Na_2CO_3): Special grade reagent
- Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

22) Ethanol (C_2H_5OH)[*249,*251]: Special grade reagent
23) pH test paper: Measurement range pH 1.0–11.0
24) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
- Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.

25) Bromocresol green (BCG) solution (0.04 w/v%): For pH test

5.2.2 Analytical operation

1) Same as 5.1.3, steps 1 through 24.
2) Same as 1.2.3 (b), step 9 and onward[*258].

5.2.3 Determination of stable Sr

Same as 5.1.4.

5.2.4 Determination of recovered Sr

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atmosphere.

[*258] For soil samples containing large amounts of uranium and thorium (Th), the measured decay of 90Y may deviate from its theoretical half-life of 64.05 hours. In such cases, an effective solution is to repeat the removal of Ba (1.2.3 (b), steps 26 through 32).

Same as 3.1.5[*259].

[*259] For soil and seafloor soil samples, the Sr concentration (C_{st}) in the sample is expressed in mg/g and the sample quantity (V) is expressed as W_s in g.

5.3 Oxalate method

5.3.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
   - Glass fiber filter: 185 mm diameter
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter, 185 mm diameter
8) Suction flask: Borosilicate glass, 1 L, 5 L
9) Filtration bell jar
10) Glass filter: 25G4
11) Separation filter: Glass. Refer to Figure 7.1 below
12) Sample dish: 1 inch diameter x 6.2 mm stainless steel
13) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
14) Filter paper holder: See Figure 7.3 below.
15) Volumetric flask: 25 mL, 50 mL, 100 mL, 250 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*260].

[*260] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

1) Pure water: Water purified using ion exchange and other methods[*261]
2) Hydrochloric acid (HCl)[*262,*263,*264]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
3) Hydrogen peroxide (H_2O_2)[*263,*264]: Special grade reagent
4) Acetic acid (CH_3COOH)[*263,*265]: Special grade reagent
   - Acetic acid (7+13): Add seven volumes of acetic acid to 13 volumes of pure water and mix.
5) Nitric acid (HNO_3)[*262,*263,*264]: Special grade reagent, mass fraction of 60%–61%
   - Nitric acid (1+1): Add one volume of nitric acid to one volume of pure water and mix.
6) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*263,*264]: Special grade reagent
7) Aqueous ammonia (NH_3)[*262,*263,*264]: Special grade reagent, mass fraction of 28.0%–30.0%
   - Aqueous ammonia (1+1): Add one volume of aqueous ammonia to one volume of pure water and mix.
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.
8) Sodium hydroxide (NaOH)[*263,*264]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
9) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*262,*263]: For ICP analysis (certified reference

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[*261] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*262] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*263] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*264] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and measures must be taken to prevent theft, loss, leakage, etc.

[*265] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

material)

- For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.

10) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
    - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
11) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*263]: Special grade reagent
    - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
12) Yttrium oxide (Y_2O_3)[*263]: Mass fraction of 99.99% or higher
    - Y carrier solution (Y^{3+}: 10 mg/mL): Dissolve 1.270 g of yttrium oxide in 50 mL of nitric acid (1+1) while heating. Allow it to cool, then transfer it to a 100 mL volumetric flask, add pure water up to the marked line, and stir well.
13) Lanthanum oxide (La_2O_3): Mass fraction of 99.99% or higher
    - La carrier solution (La^{3+}: 10 mg/mL): Dissolve 11.728 g of lanthanum oxide in 10 mL of hydrochloric acid. Then, transfer to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
14) Strontium nitrate ($Sr(NO_3)_2$))[*266]: Special grade reagent
    - Sr carrier solution (Sr^{2+}: 100 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 241.5 g to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
15) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*263,*264,*267]: Special grade reagent
    - Ba carrier solution ($^{Ba2+}$: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, gradually add hydrochloric acid (1+1) until the pH reaches approx. 4 and the substance is completely dissolved, then add pure water to make 250 mL.
16) Calcium carbonate ($CaCO_3$): Special grade reagent
    - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*268] and add 500 mL of pure water. Then, gradually add hydrochloric acid until it is completely dissolved, and add pure water to make 1 L.
17) Potassium chromate (K_2CrO_4)[*263,*264,*269]: Special grade reagent
    - Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.
18) Ammonium acetate (CH_3COONH_4): Special grade reagent
    - Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water

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[*266] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

[*267] Radioactive materials may be present. A reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*268] Vigorous foaming will occur when dissolved in hydrochloric acid. It is preferable to prepare the solution in a beaker, as there is a risk that the stopper of the volumetric flask may be forced off.

[*269] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a local exhaust ventilation system, must be taken to reduce exposure concentrations.

to make 100 mL.
- Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.

19) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \bullet H_2O$)[*264]: Special grade reagent
- Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
- Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in water to make 1 L.

20) Lanthanum nitrate hexahydrate ($La(NO_3)_3 \bullet 6H_2O$)[*266]: Mass fraction of 95.0% or higher
- Lanthanum nitrate solution (0.2 w/v%): Dissolve 2.66 g of lanthanum nitrate hexahydrate in pure water to make 1 L.

21) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
- Ammonium carbonate solution (1 w/v%): Dissolve 1 g of ammonium carbonate in pure water to make 100 mL.

22) Sodium carbonate (Na_2CO_3): Special grade reagent
- Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.

23) pH test paper: Measurement range pH 1.0–11.0

24) Bromocresol green (BCG) solution (0.04 w/v%): For pH test

5.3.2 Analytical operation

1) Measure out 100 g[*270] of air-dried fine soil, place it on an evaporating dish (porcelain, approx. diameter of 15 cm), and heat it at 500°C for five hours.
2) After cooling, transfer to a 2 L beaker, add exactly 10 mL of Sr carrier solution (Sr: 100 mg/mL), and mix well.
3) Gradually add 1 L of hydrochloric acid or nitric acid, taking care to prevent foaming[*271].
4) Cover it with a watch glass and heat it on a hot plate for three hours until it boils gently.
5) Filter under vacuum[*272] by placing a glass fiber filter paper over a Büchner funnel and filter paper (No. 5C). Wash the residue three times with warm pure water.

[*270] For samples containing large amounts of sulfur compounds, such as sulfate and sulfide, strontium sulfate may be produced during the analysis process, resulting in a greatly reduced recovery rate. In such cases, add 200 mL of sodium carbonate solution (30 w/v%), stir for 4 hours, then filter and perform acid leaching to prevent loss of recovery rate.

[*271] In samples rich in calcium carbonate (e.g., shell fragments), vigorous foaming will occur. Take care to prevent splashing and dispersion.

[*272] Since the heated sample solution generates hydrochloric acid gas, the filtration operation must be performed under local exhaust ventilation equipment, such as a fume hood, after the solution has cooled sufficiently. Furthermore, even after cooling, if the hydrochloric acid concentration of the sample solution is 25% or higher, hydrochloric acid gas will still be generated. For this reason, the filtration must be performed under local exhaust ventilation equipment, such as a fume hood.

6) Add 1 L of pure water, heat it on a hot plate, and bring to a boil.
7) Gradually add aqueous ammonia to adjust the pH to approx. 8 and precipitate iron(III) hydroxide.
8) Add boiled pure water to make a total volume of 2 L, and allow to stand overnight to settle the suspension.
9) Remove most of the supernatant by decantation and centrifuge the remainder. Transfer the supernatant to a 5 L beaker.
10) Dissolve the precipitate by warming it in 30 mL of hydrochloric acid, then place it in the original 3 L beaker.
11) Repeat steps 6 to 10 twice.
12) Combine the supernatants, heat to approx. 80°C, add 40 g of oxalic acid, and adjust the pH to 4.0–4.2 using aqueous ammonia while measuring with a pH meter. For samples rich in calcium, add more oxalic acid.
13) Age the precipitate by heating it for several hours. Then, allow it to cool and stand until the suspension has settled completely[*273].
14) Remove most of the supernatant liquid by decantation, then perform vacuum filtration using a Büchner funnel and filter paper (No. 5C).
15) Wash the precipitate on the filter paper with 50 mL of ammonium oxalate solution (0.2 w/v%). Add ammonium oxalate solution (0.2 w/v%) in three portions.
16) Transfer the precipitate with the filter paper to an evaporating dish (porcelain, approx. 10 cm in diameter), dry it, and heat it at 600°C for three hours.
17) Transfer the sample to a 500 mL beaker, dissolve it in nitric acid, and add pure water to make approx. 200 mL.
18) Add 4 mL of a solution of iron(III) chloride (Fe^{3+}: 5 mg/mL), and heat to drive out carbon dioxide.
19) Gradually add aqueous ammonia to adjust the pH to approx. 8 and precipitate iron(III) hydroxide.
20) Heat for approx. 10 minutes to age the precipitate.
21) Quickly filter through filter paper (No. 5A) and wash the precipitate three times with aqueous ammonia (1+100). Collect the filtrate and the washings in a 500 mL beaker and discard the precipitate.
22) Heat the solution and evaporate to approx. 200 mL.
23) Add 2 mL of the Ba carrier solution (Ba^{2+}: 10 mg/mL) and 2 mL of the ammonium acetate solution (46 w/v%). Adjust pH to between 5.0 and 5.5 by adding aqueous ammonia (1+1) and acetic acid (7+13).
24) Heat to approx. 60°C, add 1 mL of potassium chromate solution (30 w/v%)[*274], and rub the inner wall of the beaker with a policeman (glass rod) to form a precipitate.
25) Continue heating to age the precipitate.
26) Allow it to stand at room temperature for approx. 1 hour.
27) Filter the yellow barium chromate (including radium) precipitate using filter paper (No.5C) and wash the precipitate three times with ammonium acetate solution (0.6 w/v%). Collect the filtrate and the washings in a 1 L beaker and discard the precipitate.

[*273] Add the ammonium oxalate solution (saturated) dropwise to the supernatant and confirm that no white turbidity has formed in the oxalate precipitate. If turbidity forms, it indicates insufficient oxalic acid. Therefore, add more oxalic acid and repeat the operation to generate precipitate.

[*274] If no precipitate forms, add potassium chromate solution (30 w/v%).

28) Add 4 g of sodium hydroxide to adjust the pH to approx. 10[*275].
29) Add 40 g of ammonium carbonate, and when a precipitate begins to appear, heat for 30 minutes, and allow to stand so that the white carbonate can precipitate.
30) Filter the precipitate under vacuum using a glass filter (25G4) and wash three times with ammonium carbonate (1 w/v%).
31) Dissolve the precipitate in nitric acid[*276], and wash the glass filter three times with pure water. Combine the filtrate and the washings in a 500 mL beaker, then heat to concentrate to approx. 200 mL.
32) Add two drops of hydrogen peroxide solution to reduce chromic acid.
33) Add 4 mL of iron(III) chloride solution (Fe^{3+}: 5 mg/mL), 0.5 mL of a La carrier solution (La: 10 mg/mL), and 0.5 mL of a Y carrier solution (Y^{3+}: 10 mg/mL), and heat to drive out carbon dioxide.
34) Gradually add aqueous ammonia[*277] to adjust the pH to approx. 8 and precipitate iron(III) hydroxide.
35) Heat for approx. 10 minutes to age the precipitate.
36) Quickly filter the precipitate with filter paper (No. 5A) (record the date and time as the "scavenging date and time") and wash it three times with aqueous ammonia (1+100). Collect the filtrate and the washings in a 500 mL beaker and discard the precipitate.
37) Heat it to drive off ammonia.
38) Add 7 mL of nitric acid, transfer to a 250 mL volumetric flask, and add pure water to the marked line.

5.3.3 Determination of stable Sr

Same as 5.1.4.

5.3.4 Determination of recovered Sr

Same as 3.1.5[*278]. However, note that as the oxalate method does not remove Ca, quantification by gravimetric analysis is not possible. In addition, because this method uses a larger amount of Sr carrier than others, care must also be taken with the aliquot volume, dilution factor, and concentration of the calibration solution. Furthermore, when performing atomic absorption spectrometry (c), lanthanum nitrate solution (0.2 w/v%) must be added.

[*275] A change in the solution color from orange to yellow indicates completion.

[*276] Adjust the amount of nitric acid according to the amount of precipitate. Cover with a watch glass and add nitric acid in small portions.

[*277] If the aqueous ammonia contains carbon dioxide, Sr precipitates as a carbonate and is filtered together with the iron hydroxide precipitation. Therefore, use unopened aqueous ammonia containing little or no carbon dioxide.

[*278] For soil and seafloor soil samples, the Sr concentration (C_{st}) in the sample is expressed in mg/g and the sample quantity (V) is expressed as W_s in g.

Chapter 6 Ash sample

Radioactive Sr in cereals, vegetables, tea leaves, pine needles, milk, seafood, and other common food products is determined by dissolving ash samples[*279] in acid and using the ion exchange, fuming nitric acid, or oxalate methods.

6.1 Ion exchange method

6.1.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Capable of operating for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Equipment

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 1G4
10) Chromatography column: With glass joints, ID: 30 mm, column length: 30 cm
11) Separatory funnel: 500 mL, globular shape, 24/40 top and 24/40 bottom joints
12) Separation filter: Glass. Refer to Figure 7.1 below
13) Sample dish: 1 inch diameter x 6.2 mm stainless steel
14) Desiccator: A vacuum type is preferred. A desiccant type may also be used.
15) Filter paper holder: See Figure 7.3 below.
16) Volumetric flask: 25 mL, 50 mL, 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used. For those not

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[*279] For ashing procedures, see "(AS) Method for Sampling of Environmental Materials" in the Series of Environmental Radioactivity Measuring Methods, No. 16.

specified in the standards, the reagent with the highest possible purity should be selected. The mass and volume figures in this book are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is indicated as the reagent name (a+b)[*280].

1) Pure water: Water purified using ion exchange and other methods[*281]
2) Hydrochloric acid (HCl)[*282,*283,*284]: Special grade reagent
   - Hydrochloric acid (1+1): Add one volume of hydrochloric acid to one volume of pure water and mix.
   - Hydrochloric acid (1+3): Add one volume of hydrochloric acid to three volumes of pure water and mix.
   - Hydrochloric acid (1+11): Add one volume of hydrochloric acid to 11 volumes of pure water and mix.
   - Hydrochloric acid (1+23): Add one volume of hydrochloric acid to 23 volumes of pure water and mix.
3) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*283,*284]: Special grade reagent
4) Nitric acid (HNO_3)[*282,*283,*284]: Special grade reagent, mass fraction 60%–61%
   - Aqua regia: Add one volume of nitric acid to three volumes of hydrochloric acid and mix. Prepare it immediately before use.
5) Sulfuric acid (H_2SO_4)[*282,*283,*284]: Special grade reagent, mass fraction of 95% or higher
   - Sulfuric acid (1+3): Add one volume of Sulfuric acid to three volumes of pure water and mix.
6) Aqueous ammonia (NH_3)[*282,*283,*284]: Special grade reagent, mass fraction 28.0%–30.0%
   - Aqueous ammonia (1+100): Add one volume of aqueous ammonia to 100 volumes of pure water and mix.

[*280] JIS K 0050:2019 "General rules for chemical analysis" states that it is applicable only to reagents with the purity, molar concentration, and density indicated in "reagents that can be expressed by the mixing ratio with water."

[*281] All water used for analysis shall be pure water. For the quantitative analysis of elements using an ICP-AES or an atomic absorption spectrometer, as described in JIS K 0116: 2014, "General rules for atomic emission spectrometry" and JIS K 0121:2006, "General rules for atomic absorption spectrometry," pure water of grade A3 or A4 (e.g., with a specific electrical resistance of 1 MΩ•cm or higher), as specified in JIS K 0557, should be used. Furthermore, it is more preferable to use water of a higher purity (ultrapure water with a specific electrical resistance of 18.2 MΩ•cm or higher).

[*282] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): Measures must be taken to reduce the exposure concentrations, such as installing a local exhaust ventilation system.

[*283] Harmful substances subject to labeling under the Industrial Safety and Health Act (those that may cause risk or health impairment to workers): Labeling, issuance of safety data sheets, and risk assessments (e.g., risk reduction measures) are mandatory requirements, and workers must be informed and educated about these matters.

[*284] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

- Aqueous ammonia (1+500): Add one volume of aqueous ammonia to 500 volumes of pure water and mix.

7) Sodium hydroxide (NaOH)[*283,*284]: Special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
8) Yttrium standard solution (Y^{3+}, 1000 mg/L)[*282,*283]: For ICP analysis (certified reference material)
   - For use, dilute the above commercially available Y standard solution with pure water to the exact required concentration.
9) Potassium permanganate standard solution (3.16 g/L)[*283]: For volumetric analysis
10) Calcium standard solution (Ca^{2+}: 1000 mg/L): JCSS grade
    - For use, dilute the above commercially available Ca standard solution with pure water to the exact required concentration.
11) Sodium oxalate ($(COONa)_2$)[*284]: Certified reference material
    - Dry sodium oxalate at 100°C for 45 to 60 minutes. Use it after allowing it to cool in a desiccator.
12) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS Grade
    - For use, dilute the above commercially available Sr standard solution with pure water to the exact required concentration.
13) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*283]: Special grade reagent
    - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
14) Strontium nitrate ($Sr(NO_3)_2$)[*285]: Special grade reagent
    - Sr carrier solution (Sr: 10 mg/mL): Dry strontium nitrate at 300°C for two to three hours and allow it to cool in a desiccator. Then, transfer 24.15 g of the solution to a 1 L volumetric flask, add pure water up to the marked line, and stir well.
15) Ammonium chloride (NH_4Cl)[*283]: Special grade reagent
16) Ammonium acetate (CH_3COONH_4): Special grade reagent
    - Ammonium acetate solution (15.4 w/v%): Dissolve 154 g of ammonium acetate in pure water to make 1 L.
17) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*284]: Special grade reagent
    - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium oxalate remains at the bottom of the container.
    - Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.
18) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
    - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water into a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid ammonium carbonate remains at the bottom of the container.

[*285] Classified as a Category 1 hazardous substance (oxidizing solids) under the Fire Service Act. Exercise caution to prevent the substance from coming into contact with strong acids. Avoid storage with flammable substances and exposure to heat, impact, and friction.

19) Sodium carbonate (Na_2CO_3): Special grade reagent
   - Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow the mixture to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be reused as a saturated solution by adding pure water and shaking, as long as solid sodium carbonate remains at the bottom of the container.
20) Ethanol (C_2H_5OH)[*283,*286]: Special grade reagent
21) Methanol (CH_3OH)[*283,*284,*286,*287]: Special grade reagent
22) pH test paper: Measurement range pH 1.0–11.0
23) Phenolphthalein: Special grade reagent, transition range: pH 7.8–pH 10.0
   - Phenolphthalein solution: Place 0.5 g of phenolphthalein into a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.
24) Bromocresol green (BCG) solution (0.04 w/v%): For pH test
25) Strongly acidic cation-exchange resin (100 mesh–200 mesh): Crosslinking degree of 8%, 100 mesh–200 mesh

6.1.2 Conditioning of the ion exchange resin and column preparation

Same as 1.1.2 (2).

6.1.3 Analytical operation

1) Place an ash sample equivalent to 1 kg of fresh mass in a beaker[*288], moisten it with pure water, and add exactly 5 mL of Sr carrier solution (Sr: 10 mg/mL)[*289].
2) Add aqua regia and evaporate on a hot plate to dryness.
3) Add nitric acid, and evaporate to dryness approx. three times[*290].
4) Add 100 mL to 300 mL of hydrochloric acid (1+1), and heat on a hot plate for one hour[*291].
5) Perform vacuum filtration using a Büchner funnel and filter paper (No. 5C), then wash the residue with warm pure water.

[*286] Classified as a Category 4 hazardous substance (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping Category 1 and Category 5 hazardous substances away from it, preventing the generation of static electricity, and avoiding filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

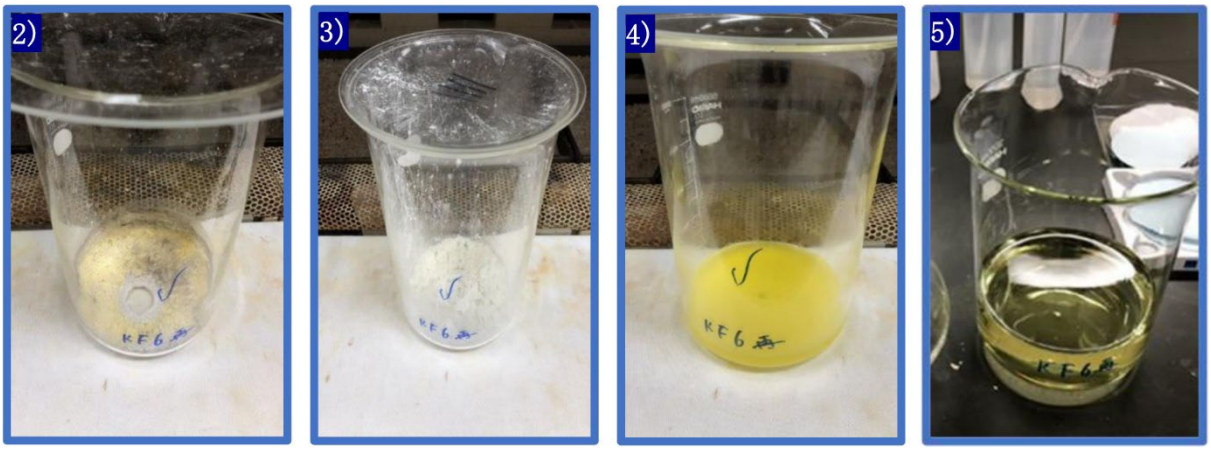
[*287] Class 2 Organic Solvents in the Ordinance on Prevention of Organic Solvent Poisoning: Measures must be taken to reduce the exposure concentrations, such as installing equipment to seal off the source of organic solvent vapor and a local exhaust ventilation system in the workplace.

[*288] The standard beaker sizes are 1 L for 10 to 20 g of ash, 2 L for 30 g of ash, and 3 L for 40 to 50 g of ash.

[*289] When simultaneously analyzing radiocesium by radiochemical analysis, add a Cs carrier as well.

[*290] If organic matter (black or dark brown) remains in the sample, add nitric acid and decompose it by heating.

[*291] Insoluble residues often remain in plant samples such as cedar leaves, pasture grass, Sargassum, and seaweed samples. Samples with remaining insoluble residue are difficult to distinguish from undissolved samples. In such cases, add 100 mL of hydrochloric acid (1+1), heat again, and check whether the residue has decreased to determine whether the residue is insoluble.



6) Combine the filtrate and washings, transfer to a beaker[*292], and fill 70% of the beaker volume with pure water[*293].
7) Same as 1.1.3 (b)[*294].

6.1.4 Determination of stable Sr[*295]

(a) ICP-AES

1) Weigh 1 g of ash sample, place it in a 200 mL beaker, moisten with pure water, add 15 mL of nitric acid, and evaporate on a hot plate to dryness.
2) Add 10 mL of nitric acid and evaporate to dryness again.
3) Add 20 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
4) Add 30 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
5) Allow to cool, filter through filter paper (No.5C), and transfer the filtrate to a 100 mL volumetric flask.
6) Add pure water up to the marked line, stir well, and use this solution as the sample solution.

a) Internal standard method

1) Measure out exactly 5 mL of the sample solution and transfer it to a 50 mL volumetric flask. Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L), add pure water up to the marked line, stir well, and use this solution as the sample solution for measurement.
2) Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L) to each of the five 50 mL volumetric flasks, and then add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to each flask, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
3) Using ICP-AES, measure the ratio (Sr emission intensity/Y emission intensity) of Sr emission intensity (measurement wavelength: 407.771 nm) to Y emission intensity (measurement wavelength: 371.029 nm) for the calibration curve solutions and the

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[*292] The standard beaker sizes are 1 L for 10 g of ash, 2 L for 20 g of ash, and 3 L for 30 g of ash or more.

[*293] For samples with low Ca content, such as milled rice and tea leaves, adding 5 mL of a Ca carrier solution (Ca^{2+}: 50 mg/mL) is recommended.

[*294] The standard amount of sodium carbonate to be added is 10 g per 10 g of ash, up to a maximum of 30 g. The standard beaker sizes used for producing oxalate precipitation are 1 L for 10 to 20 g of ash, 2 L for 30 to 40 g of ash, and 3 L for 50 g or more of ash. The standard amounts of oxalic acid to be added are 30 g for fish and skim milk, 20 g for seaweed and dry milk, and 10 g for others.

[*295] Milled rice, milk, etc. have very low stable Sr content and do not need to be quantified.

measurement sample solutions.

4) Determine the Sr concentration, C_P (mg/L), of the sample solution from the calibration curve, then determine the Sr content, C_{st} (% in ash), of the ash sample from the dilution factor[*296].

b) Calibration curve method

1) Measure out exactly 5 mL of the sample solution and transfer it to a 50 mL volumetric flask. Add pure water up to the mark line, stir well, and use this solution as the sample solution.
2) Add exactly 0, 0.1, 0.5, 1, and 5 mL of the Sr standard solution (Sr^{2+}: 5 mg/L) to the five 50 mL volumetric flasks, respectively. Add pure water up to the marked line and stir well to prepare the solution for the calibration curve (Sr^{2+}: 0, 0.01, 0.05, 0.1, 0.5 mg/L).
3) Measure the emission intensity (wavelength: 407.771 nm) of the calibration curve solution and the sample solution using ICP-AES.
4) Determine the Sr concentration, C_P (mg/L), of the sample solution from the calibration curve, then determine the Sr content, C_{st} (% in ash), of the ash sample from the dilution factor[*296].

(b) Atomic absorption spectrometry (standard addition method)

1) Weigh 1 g of ash sample, place it in a 200 mL beaker, moisten with pure water, add 15 mL of nitric acid, and evaporate on a hot plate to dryness.
2) Add 10 mL of nitric acid and evaporate to dryness again.
3) Add 20 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
4) Add 30 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
5) Allow to cool, filter through filter paper (No.5C), and transfer the filtrate to a 100 mL volumetric flask.
6) Add pure water up to the marked line, stir well, and use this solution as the sample solution.
7) Measure out exactly 50 mL of the sample solution, transfer to a 200 mL beaker, then add pure water to make approx. 100 mL.
8) After heating to approx. 80°C, add 5 g of oxalic acid and 5 mL of the BCG solution and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding aqueous ammonia.
9) Reheat to age the precipitate.
10) After cooling, filter the precipitate under vacuum using a glass filter (1G4) and wash with a small amount of ammonium oxalate solution (0.2 w/v%).
11) Transfer the glass filter to a filtration bell jar containing a 50mL volumetric flask. Dissolve the precipitate in the smallest possible amount of hydrochloric acid (1+1) and perform vacuum filtration.
12) Wash the glass filter with pure water, add pure water up to the marked line, and stir well.
13) Dispense exactly 5 mL of the solution into each of four 25 mL volumetric flasks [*297].
14) Add exactly 0, 1, 2, and 3 mL of Sr standard solution (Sr^{2+}: 25 mg/L) to the respective flasks. Then, add pure water up to the marked line, stir well, and use this solution as the solution for the test sample (Sr^{2+}: 0, 1, 2 and 3 mg/l).
15) As in 4.1.5 (b), determine the Sr concentration C_p (mg/L) by measuring the absorbance using an atomic absorption spectrometer, and determine the Sr content C_{st} (% in ash) in the ash

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[*296] For example, at this dilution factor, C_{st} (% in ash) = C_p (mg/mL) × 50 × (100/5) × ($1/10^3$) × (1/1) × (1/10).

[*297] Dilution to a Sr concentration of approx. 1.5 mg/l is recommended. The aliquot volume shown here is an example.

sample from the dilution factor[*298].

6.1.5 Determination of Ca

(a) ICP-AES

1) Weigh 1 g of ash sample, place it in a 200 mL beaker, moisten with pure water, add 15 mL of nitric acid, and evaporate on a hot plate to dryness.
2) Add 10 mL of nitric acid and evaporate to dryness again.
3) Add 20 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
4) Add 30 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
5) Allow to cool, filter through filter paper (No.5C), and transfer the filtrate to a 100 mL volumetric flask.
6) Add pure water up to the marked line, stir well, and use this solution as the sample solution.

a) Internal standard method

1) Dilute repeatedly with water so that the amount of Ca in the 50 mL volumetric flask is approx. 50 μg. Then, add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L), add pure water up to the marked line, stir well, and use this solution as the sample solution.
2) Add exactly 1 mL of the Y standard solution (Y^{3+}: 50 mg/L) to each of the six 50 mL volumetric flasks, and then add exactly 0, 0.5, 1, 5, 10, and 30 mL of the Ca standard solution (Ca^{2+}: 5 mg/L) to each flask, respectively. Add pure water up to the marked line, stir well, and use the solution for the calibration curve (Ca^{2+}: 0, 0.05, 0.1, 0.5, 1, 3 mg/l).
3) Using ICP-AES, measure the ratio (Ca emission intensity/Y emission intensity) of Ca emission intensity (measurement wavelength: 393.366 nm) to Y emission intensity (measurement wavelength: 371.029 nm) for the calibration curve solutions and the measurement sample solutions.
4) Determine the Ca concentration, C_P (mg/L), of the sample solution from the calibration curve, and determine the Ca content, W_2 (mg), of the ash sample from the dilution ratio[*299].
5) The amount of Ca in the ash, C_{ca}, is calculated using the following equation.

$$C_{Ca} = \frac{W_2}{W_1} \times \frac{1}{10^3} \times 100$$

C_{ca} : Amount of Ca in ash (% in ash)
W_1 : Mass of ash sample weighed (g)
W_2 : Mass of Ca in the weighed ash sample (mg)[*299]

b) Calibration curve method

1) Dilute repeatedly with pure water so that the amount of Ca in the 50 mL volumetric flask is approx. 50 μg. Add pure water up to the marked line, stir well, and use this solution as the sample solution.
2) Add exactly 0, 0.5, 1, 5, 10, and 30 mL of the Ca standard solution (Ca^{2+}: 5 mg/L) to the six 50 mL volumetric flasks, respectively. Add pure water up to the marked line, stir well, and use the solution for the calibration curve (Ca^{2+}: 0, 0.05, 0.1, 0.5, 1, 3 mg/l).
3) Measure the emission intensity (wavelength: 393.366 nm) of the calibration curve solution and the sample solution using ICP-AES.

[*298] For example, at this dilution factor, C_{st} (% in ash) = C_p (mg/mL) × 25 × (50/5) × (100/50) × (1/103) × (1/1) × (1/10).

[*299] W_2 (mg) = C_P (mg/L) × volumetric flask volume of the measuring solution × (volume of diluted volumetric flask/aliquot volume)

4) Determine the Ca concentration, C_P (mg/L), of the sample solution from the calibration curve, then determine the Ca content, W_2 (mg), of the ash sample from the dilution ratio[*299].
5) The amount of Ca in the ash, C_{ca}, is calculated using the following equation.

$$C_{Ca} = \frac{W_2}{W_1} \times \frac{1}{10^3} \times 100$$

C_{ca} : Amount of Ca in ash (% in ash)
W_1 : Mass of ash sample weighed (g)
W_2 : Mass of Ca in the weighed ash sample (mg)[*299]

(b) Atomic absorption spectrometry

1) Weigh 1 g of ash sample, place it in a 200 mL beaker, moisten with pure water, add 15 mL of nitric acid, and evaporate on a hot plate to dryness.
2) Add 10 mL of nitric acid and evaporate to dryness again.
3) Add 20 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
4) Add 30 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
5) Allow to cool, filter through filter paper (No.5C), and transfer the filtrate to a 100 mL volumetric flask.
6) Add pure water up to the marked line, stir well, and use this solution as the sample solution.
7) Dilute repeatedly with pure water so that the Ca content in the 50 mL volumetric flask is approx. 0.05 mg to 0.1 mg, then add exactly 5 mL of Sr carrier solution (Sr^{2+}: 10 mg/mL). Add pure water up to the marked line, stir well, and use the solution for the test sample (Ca^{2+}: 1 mg/mL to 2 mg/mL).
8) Dispense exactly 5 mL of Sr carrier solution (Sr^{2+}: 10 mg/mL), in each of four 50 mL volumetric flasks, then add exactly 0, 1, 2, and 3 mL of Ca standard solution (Ca^{2+}: 50 mg/L), respectively. Add pure water up to the marked line, stir well, and use the solution for the calibration curve (Ca^{2+}: 0, 1, 2, 3 mg/l).
9) Measure the emission intensity (wavelength: 422.7 nm) of the calibration curve solution and the sample solution using the atomic absorption spectrometer.
10) Determine the Ca concentration, C_P (mg/L), of the sample solution from the calibration curve, then determine the Ca content, W_2 (mg), of the ash sample from the dilution ratio[*299].
11) The amount of Ca in the ash, C_{ca}, is calculated using the following equation.

$$C_{Ca} = \frac{W_2}{W_1} \times \frac{1}{10^3} \times 100$$

C_{ca} : Amount of Ca in ash (% in ash)
W_1 : Mass of ash sample weighed (g)
W_2 : Mass of Ca in the weighed ash sample (mg)[*299]

(c) Potassium permanganate titration

1) Weigh 1 g of ash sample, place it in a 200 mL beaker, moisten with pure water, add 15 mL of nitric acid, and evaporate on a hot plate to dryness.
2) Add 10 mL of nitric acid and evaporate to dryness again.
3) Add 20 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
4) Add 30 mL of hydrochloric acid (1+1) and evaporate on a hot plate to dryness.
5) Allow to cool, filter through filter paper (No.5C), and transfer the filtrate to a 100 mL volumetric flask.
6) Add pure water up to the marked line, stir well, and use this solution as the sample solution.
7) Transfer an amount of the sample solution equivalent to 20 mg to 40 mg of Ca into a 300 mL beaker, and add pure water to make approx. 200 mL.

8) After heating to approx. 80°C, add 5 g of oxalic acid and 5 mL of the BCG solution and adjust the pH to 4.0–4.2 (the BCG solution turns from yellow to blue-green) by adding aqueous ammonia.
9) Reheat to age the precipitate.
10) After cooling, filter the precipitate using filter paper (No.5C). Wash the filter paper and the precipitate thoroughly (approx. 10 times) with warm pure water.
11) Transfer the precipitate with the filter paper to a 300 mL beaker, then dissolve the precipitate by adding 200 mL of warm pure water and 30 mL of sulfuric acid (1+3).
12) Titrate with potassium permanganate standard solution (3.16 g/L), keeping the temperature of the solution at approx. 70 °C. The end point is the point at which a pale red color remains for approx. 15 seconds after stirring.
13) Using the following equation, calculate the amount of Ca in the ash, C_{ca} (% in ash), from the amount of potassium permanganate standard solution, V_1 (mL), required to reach the end point.

$$C_{\mathrm{Ca}} = \frac{(V_1 - V_2 - V_3) \times F \times 0.002004}{W_1} \times \frac{100}{V_4} \times 100$$

C_{ca} : Amount of Ca in ash (% in ash)
W_1 : Mass of ash sample weighed (g)
V_1 : Amount (mL) of potassium permanganate standard solution (3.16 g/L) required for the aliquot
V_2 : Amount (mL) of potassium permanganate standard solution (3.16 g/L) required for the blank
V_3 : Amount (mL) of potassium permanganate standard solution (3.16 g/L) consumed by precipitation of oxalate formed from Sr coexisting in the ash sample[*300]
F : factor of potassium permanganate standard solution (3.16 g/L)[*301]
V_4 : aliquot volume (mL) of 7)

6.1.6 Determination of recovered Sr

Same as 3.1.5[*302].

[*300] Using the result in 6.1.4 (Sr content in ash sample, C_{st} (% in ash)), perform the calculation according to the following formula:
$V_3=(W_1 \times C_{st}/0.4381 \times F) \times (V_4/100)$

[*301] Precisely measure out 100 mg to 200 mg of sodium oxalate (W_3 (mg)), titrate with potassium permanganate standard solution (3.16 g/L), subtract the amount (mL) required for the blank from the amount (mL) required for the titration to obtain V_5 (mL), then perform the calculation according to the following formula. $F = W_3/(6.701 \times V_5)$

[*302] In the case of ash samples, the Sr concentration (C_{sr}) in the sample is expressed in mg/g of ash, and the test quantity (V) is expressed as W_s in grams of ash.

6.2 Fuming nitric acid method

6.2.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: can be set to approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Can operate for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Devices

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Graduated cylinder (or dispenser)
3) Evaporating dish
4) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
5) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
6) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
7) Funnel: 65 mm diameter x 8 x 150 mm
8) Büchner funnel: 90 mm diameter, 110 mm diameter
9) Suction flask: 1 L, borosilicate glass
10) Filtration bell jar
11) Glass filter: 1G4
12) Erlenmeyer flask with stopper
13) Separable filter assembly: Glass, see Figure 7.1 below.
14) Sample dish: 1 inch diameter x 6.2 mm stainless steel
15) Desiccator: Vacuum type is preferred. A desiccant type may be used.
16) Filter paper holder: See Figure 7.3 below.
17) Volumetric flask: 25 mL, 50 mL, 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used, and for those not specified in the standards, the highest possible purity should be selected. The weight and volume figures in the text are merely illustrative of the percentage of preparation and may be adjusted as necessary. A reagent prepared by mixing reagent volume a and water volume b is described as reagent name (a+b)[*303].

[*303] JIS K 0050:2019 "General rules for chemical analysis," states that it is applicable only to reagents with a purity, molar concentration and density described in "reagents that can be expressed by mixing ratio with water."

1) Pure water: Water purified by ion exchange, etc.[*304]
2) Hydrochloric acid (HCl)[*305,*306,*307]: Special grade reagent
   - Hydrochloric acid (1+1): Mix one part of hydrochloric acid with one part of pure water by volume.
   - Hydrochloric acid (1+3): Mix one part of hydrochloric acid with three parts of pure water by volume.
   - Hydrochloric acid (1+11): Mix one part of hydrochloric acid with eleven parts of pure water by volume.
3) Acetic acid (CH_3COOH)[*306,*308]: Special grade reagent
   - Acetic acid (7+13): Mix seven parts of acetic acid with thirteen parts of pure water by volume.
4) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$): Special grade reagent
5) Nitric acid (HNO_3)[*305,*306,*307]: special grade reagent, mass fraction 60% to 61%
   - Aqua regia: Mix three parts of hydrochloric acid with one part of nitric acid by volume. Prepare immediately before use.
6) Fuming nitric acid (1.52)[*305,*306,*307,*309]: Special grade reagent, density 1.52 g/mL
7) Fuming nitric acid (1.45)[*305,*306,*307]: Special grade reagent, density 1.45 g/mL
   - For preparation: Add three parts of fuming nitric acid (1.52) to one part of pure water by volume, a little at a time, while cooling it in running water[*310]. After cooling it sufficiently

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[*304] All water used for analysis shall be pure water. For the quantitative analysis of elements using ICP-AES or atomic absorption spectrometry, as described in JIS K 0116: 2014 "General rules for atomic emission spectrometry" and JIS K 0121:2006 "General rules for atomic absorption spectrometry," use A3 or A4 pure water (resistivity 1 MΩ•cm or more, etc.) as specified in JIS K 0557. Further, it is preferable to use water of higher purity (ultrapure water: specific resistivity of 18.2 MΩ•cm or more).

[*305] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): It is necessary to take measures to reduce the exposure concentration, such as installing a local exhaust ventilation system.

[*306] Substances subject to hazardous substances labeling under the Industrial Safety and Health Law (those that may cause danger or health impairment to workers): Labeling, issuance of Safety Data Sheets, and risk assessment (risk reduction measures, etc.) are obligatory, and workers must be informed and educated regarding these matters.

[*307] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

[*308] Classified as a Class 4 hazardous material (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping hazardous materials of Class 1 and Class 5 away from it, preventing the generation of static electricity, and not filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*309] Classified as a Class 6 dangerous material (oxidizing liquid), by the Fire Service Act. It is necessary to prevent it from coming into contact with the human body, to keep combustibles and metals away, and to prevent it from coming into contact with moisture and humidity by making the storage container acid-resistant.

[*310] Since the heat generation is very intense, add a little at a time while performing cooling, and

in running water, add pure water little by little until the specific gravity reaches 1.45 as measured with a hydrometer.

8) Aqueous ammonia (NH_3)[*305,*306,*307]: Special grade reagent, mass fraction 28.0% to 30.0%
   - Aqueous ammonia (1+100): Mix one part of aqueous ammonia with one hundred parts of pure water by volume.
   - Aqueous ammonia (1+500):Mix one part of aqueous ammonia with five hundred parts of pure water by volume.
9) Sodium hydroxide (NaOH)[*306,*307]: special grade reagent
   - Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.
10) Yttrium standard solution (Y^{3+}, 1000 mg/L)[*305,*306]: for ICP analysis (certified reference material)
   - For use, dilute the above commercial Y standard solution accurately with pure water to the required concentration.
11) Potassium permanganate standard solution (3.16 g/L)[*306]: for volumetric analysis
12) Calcium standard solution Ca^{2+}: 1000 mg/L): JCSS grade
   - For use, dilute the above commercial Ca standard solution accurately with pure water to the required concentration.
13) Sodium oxalate ($(COONa)_2$)[*307]: Certified reference material
   - Dry sodium oxalate at 100°C for 45 to 60 minutes, allow to cool in a desiccator, and use.
14) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS grade
   - For use, dilute the above commercial Sr standard solution accurately with pure water to the required concentration.
15) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*306]: Special grade reagent
   - Iron(III) chloride solution (5 mg Fe^{3+}/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker, and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
16) Strontium nitrate ($Sr(NO_3)_2$)[*311]: Special grade reagent
   - Sr carrier solution (Sr: 10 mg/mL): Dry strontium nitrate at 300 °C for 2 to 3 hours, allow to cool in a desiccator, transfer 24.15 g of the dried strontium nitrate to a 1 L volumetric flask, fill to the mark with pure water, and mix well.
17) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*306,*307,*312]: special grade reagent
   - Ba carrier solution (Ba^{2+}: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, add 200 mL of pure water. Then, add hydrochloric acid (1+1) a little at a time until the barium is completely dissolved at approximately pH 4, and add pure water to make 250 mL.
18) Calcium carbonate ($CaCO_3$): Special grade reagent
   - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*313], add 500 mL of pure water. Then, add hydrochloric acid a little at a time until the calcium carbonate is completely dissolved, and add pure water to make 1 L.

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pay particular attention to avoiding overheating and bumping.

[*311] Classified as a Class 1 hazardous material (oxidizing solid) by the Fire Service Act. Caution is required, such as preventing it from coming into contact with strong acids, avoiding storage with flammable materials, and avoiding heat, impact, and friction.

[*312] As radioactive materials may be contained, a reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*313] It foams intensely when dissolved in hydrochloric acid. It is preferable to prepare it in a beaker because there is a risk that the stopper of the volumetric flask will blow off.

19) Ammonium chloride (NH_4Cl)[*306]: Special grade reagent
20) Potassium chromate (K_2CrO_4)[*306,*307,*314]: Special grade reagent
    - Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.
21) Ammonium acetate (CH_3COONH_4)): Special grade reagent
    - Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water to make 100 mL.
    - Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.
22) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*307]: Special grade reagent
    - Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be used as a saturated solution by adding pure water and shaking as long as the solid ammonium oxalate remains at the bottom of the container.
    - Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in pure water to make 1 L.
23) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
    - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be used as a saturated solution by adding pure water and shaking as long as the solid ammonium carbonate remains at the bottom of the container.
24) Sodium carbonate (Na_2CO_3): Special grade reagent
    - Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be used as a saturated solution by adding pure water and shaking as long as the solid sodium carbonate remains at the bottom of the container.
25) Ethanol (C_2H_5OH)[*306,*308]: Special grade reagent
26) pH test paper: Measurement range pH 1.0 to 11.0
27) Phenolphthalein: Special grade reagent, transition range: pH 7.8 to pH 10.0
    - Phenolphthalein solution: Place 0.5 g of phenolphthalein in a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.
28) Bromocresol green (BCG) solution (0.04 w/v%) for pH test

6.2.2 Analytical procedure

1) Same as 6.1.3 1) through 6).
2) Same as 1.2.3(b)[*315].

[*314] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a system that seals off the source of vapor or dust or installing a local exhaust ventilation system, must be taken to reduce diffusion into the atmosphere.

[*315] The standard amount of sodium carbonate to be added is 10 g per 10 g of ash, up to a maximum of 30 g. The standard beaker size used for producing oxalate precipitation is a 1 L beaker for 10 g to 20 g of ash, a 2 L beaker for 30 g to 40 g of ash, and a 3 L beaker for 50 g or more of

6.2.3 Quantification of stable Sr[*316]

Same as 6.1.4.

6.2.4 Quantification of Ca

Same as 6.1.5.

6.2.5 Quantification of recovered Sr

Same as 3.1.5[*317].

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ash. The standard amount of oxalic acid to be added is 30 g for fish and skim milk, 20 g for seaweed and dry milk, and 10 g for others.

[*316] Milled rice, milk, etc. have a very low stable Sr content and the Sr content does not need to be quantified.

[*317] In the case of ash samples, the Sr concentration (C_{sr}) in the sample is expressed in mg/g ash, and the test quantity (V) is expressed in W_s and in g ash.

6.3 Oxalate method

6.3.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: Temperature adjustable from approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Centrifuge: Can operate for 10 minutes at a relative centrifugal acceleration of 2000 x *g* or higher
4) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
5) Electric dryer: Temperature can be set from room temperature to approximately 120°C
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) ICP-AES
8) Atomic absorption spectrometer

(2) Devices

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Evaporating dish
3) Beaker: 100 mL, 200 mL, 500 mL, 1 L, 2 L, 3 L, 5 L
4) Watch glass: 75 mm diameter, 105 mm diameter, 120 mm diameter
5) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 11 cm diameter, 9 cm diameter, 18.5 cm diameter, 24 mm diameter
   - Membrane filter: 11 cm diam. (0.45 μm pore size); 25 mm diam. (0.45 μm)
6) Funnel: 65 mm diameter x 8 x 150 mm
7) Büchner funnel: 90 mm diameter, 110 mm diameter
8) Suction flask: 1 L, borosilicate glass
9) Glass filter: 25G4
10) Separable filter assembly: Glass, see Figure 7.1 below.
11) Sample dish: 1 inch diameter x 6.2 mm stainless steel
12) Desiccator: Vacuum type is preferred. A desiccant type may be used.
13) Filter paper holder: See Figure 7.3 below.
14) Volumetric flask: 25 mL, 50 mL, 100 mL, 250 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used, and for those not specified in the standards, the highest possible purity should be selected. The weight and volume figures in the text are merely illustrative of the percentage of preparation and may be adjusted as necessary. Reagent prepared by mixing reagent volume a and water volume b is described as reagent name (a+b)[*318].

1) Pure water: Water purified by ion exchange, etc.[*319]

[*318] JIS K 0050:2019 "General rules for chemical analysis," states that it is applicable only to reagents with a purity, molar concentration and density described in "reagents that can be expressed by mixing ratio with water."

[*319] All water used for analysis shall be pure water. For the quantitative analysis of elements using ICP-AES or atomic absorption spectrometry, as described in JIS K 0116: 2014 "General rules

2) Hydrochloric acid (HCl)[*320,*321,*322]: Special grade reagent
- Hydrochloric acid (1+1): Mix one part of hydrochloric acid with one part of pure water by volume.
- Hydrochloric acid (1+3): Mix one part of hydrochloric acid with three parts of pure water by volume.
- Hydrochloric acid (1+11): Mix one part of hydrochloric acid with eleven parts of pure water by volume.

3) Hydrogen peroxide (H_2O_2)[*321,*322]: Special grade reagent
4) Acetic acid (CH_3COOH)[*321,*323]: Special grade reagent
- Acetic acid (7+13): Mix seven parts of acetic acid with thirteen parts of pure water by volume.

5) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*321,*322]: Special grade reagent
6) Nitric acid (HNO_3)[*320,*321,*322]: Special grade reagent, mass fraction 60% to 61%
- Nitric acid (1+1): Mix one part of nitric acid to one part of pure water by volume.
- Aqua regia: Mix three parts of hydrochloric acid with one part of nitric acid by volume. Prepare immediately before use.

7) Aqueous ammonia (NH_3)[*320,*321,*322]: Special grade reagent, mass fraction 28.0% to 30.0%
- Aqueous ammonia (1+1): Mix one part of aqueous ammonia with one part of pure water by volume.
- Aqueous ammonia (1+100): Mix one part of aqueous ammonia with one hundred parts of pure water by volume.

8) Sodium hydroxide (NaOH)[*321,*322]: Special grade reagent
- Sodium hydroxide solution (24 w/v%): Dissolve 240 g of sodium hydroxide in pure water to make 1 L.

9) Yttrium standard solution (Y^{3+}: 1000 mg/L)[*320,*321]: JCSS Grade

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for atomic emission spectrometry" and JIS K 0121:2006 "General rules for atomic absorption spectrometry," use A3 or A4 pure water (resistivity 1 MΩ•cm or more, etc.) as specified in JIS K 0557. Further, it is preferable to use water of higher purity (ultrapure water: specific resistivity of 18.2 MΩ•cm or more).

[*320] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): It is necessary to take measures to reduce the exposure concentration, such as installing a local exhaust ventilation system.

[*321] Substances subject to hazardous substances labeling under the Industrial Safety and Health Law (those that may cause danger or health impairment to workers): Labeling, issuance of Safety Data Sheets, and risk assessment (risk reduction measures, etc.) are obligatory, and workers must be informed and educated regarding these matters.

[*322] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled "poisonous substance not for medical use" and "deleterious substance not for medical use," and necessary measures must be taken to prevent theft, loss, leakage, and other incidents.

[*323] Classified as a Class 4 hazardous material (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping hazardous materials of Class 1 and Class 5 away from it, preventing the generation of static electricity, and not filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

- For use, dilute the above commercial Y standard solution accurately with pure water to the required concentration.

10) Potassium permanganate standard solution (3.16 g/L)[*321]: for volumetric analysis
11) Calcium standard solution (Ca^{2+}: 1000 mg/L): JCSS grade
    - For use, dilute the above commercial Ca standard solution accurately with pure water to the required concentration.
12) Sodium oxalate ((COONa) 2) [*322]: Certified reference material
    - Dry sodium oxalate at 100°C for 45 to 60 minutes, allow to cool in a desiccator, and use.
13) Strontium standard solution (Sr^{2+}: 1000 mg/L): JCSS grade
    - For use, dilute the above commercial Sr standard solution accurately with pure water to the required concentration.
14) Yttrium oxide (Y_2O_3)[*321]: Mass fraction of 99.99% or higher
    - Y carrier solution (Y^{3+}: 10 mg/mL): Dissolve 1.270 g of yttrium oxide in 50 mL of nitric acid (1+1) while heating. Allow it to cool, then transfer it to a 100 mL volumetric flask, fill to the mark with pure water, and mix well.
15) Lanthanum oxide (La_2O_3): Mass fraction of 99.99% or higher
    - La carrier solution (La^{3+}: 10 mg/mL): Dissolve 11.728 g of lanthanum oxide in 10 mL of hydrochloric acid. Then, transfer it to a 1 L volumetric flask, fill to the mark with pure water, and mix well.
16) Strontium nitrate ($Sr(NO_3)_2$))[*324]: Special grade reagent
    - Sr carrier solution (Sr^{2+}: 100 mg/mL): Dry strontium nitrate at 300°C for two to three hours, allow it to cool in a desiccator, transfer 241.5 g to a 1 L volumetric flask, fill to the mark with pure water, and mix well.
17) Barium hydroxide octahydrate ($Ba(OH)_2 \cdot 8H_2O$)[*321,*322,*325]: Special grade reagent
    - Ba carrier solution (Ba^{2+}: 10 mg/mL): Place 5.7 g of barium hydroxide octahydrate in a 300 mL beaker, and add 200 mL of pure water. Then, add hydrochloric acid (1+1) a little at a time until the barium hydroxide octahydrate is completely dissolved at approximately pH 4, and add pure water to make 250 mL.
18) Calcium carbonate ($CaCO_3$): Special grade reagent
    - Ca carrier solution (Ca^{2+}: 50 mg/mL): Place 125 g of calcium carbonate in a 1 L beaker[*326], add 500 mL of pure water. Then, add hydrochloric acid a little at a time until the calcium carbonate is completely dissolved, and add pure water to make 1 L.
19) Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$)[*321]: Special grade reagent
    - Iron(III) chloride solution (Fe^{3+}: 5 mg/mL): Place 6.0 g of iron(III) chloride hexahydrate in a 500 mL beaker, and dissolve in 5 mL of hydrochloric acid (1+1) and pure water to make 250 mL.
20) Potassium chromate (K_2CrO_4)[*321,*322,*327]: Special grade reagent

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[*324] Classified as a Class 1 hazardous material (oxidizing solid) by the Fire Service Act. Caution is required, such as preventing it from coming into contact with strong acids, avoiding storage with flammable materials, and avoiding heat, impact, and friction.

[*325] As radioactive materials may be contained, a reagent blank test must be conducted before use to verify that their concentration will not interfere with analysis.

[*326] It foams intensely when dissolved in hydrochloric acid. It is preferable to prepare it in a beaker because there is a risk that the stopper of the volumetric flask will blow off.

[*327] Class 2 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that cause chronic disorders, such as cancer, and do not fall under Class 1 substances): Measures, such as installing a local exhaust ventilation system, must be taken to reduce exposure concentrations.

- Potassium chromate solution (30 w/v%): Dissolve 30 g of potassium chromate in pure water to make 100 mL.

21) Ammonium acetate (CH_3COONH_4)): Special grade reagent
- Ammonium acetate solution (46 w/v%): Dissolve 46 g of ammonium acetate in pure water to make 100 mL.
- Ammonium acetate solution (0.6 w/v%): Dissolve 6 g of ammonium acetate in pure water to make 1 L.

22) Ammonium oxalate monohydrate ($(NH_4)_2C_2O_4 \cdot H_2O$)[*322]: Special grade reagent
- Ammonium oxalate solution (saturated): Place 30 g of ammonium oxalate monohydrate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow to stand. Use the supernatant after the undissolved ammonium oxalate has settled. After use, it can be used as a saturated solution by adding pure water and shaking as long as the solid ammonium oxalate remains at the bottom of the container.
- Ammonium oxalate solution (0.2 w/v%): Dissolve 2 g of ammonium oxalate monohydrate in water to make 1 L.

23) Lanthanum nitrate hexahydrate ($La(NO_3)_3 \cdot 6H_2O$)[*324]: Mass fraction of 95.0% or higher
- Lanthanum nitrate solution (0.2 w/v%): Dissolve 2.66 g of lanthanum nitrate hexahydrate in pure water to make 1 L.

24) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
- Ammonium carbonate solution (1 w/v%): Dissolve 1 g of ammonium carbonate in pure water to make 100 mL.

25) Sodium carbonate (Na_2CO_3): Special grade reagent
- Sodium carbonate solution (saturated): Place 110 g of sodium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow to stand. Use the supernatant after the undissolved sodium carbonate has settled. After use, it can be used as a saturated solution by adding pure water and shaking as long as the solid sodium carbonate remains at the bottom of the container.

26) pH test paper: Measurement range pH 1.0 to 11.0

27) Bromocresol green (BCG) solution (0.04 w/v%) for pH test

6.3.2 Analytical procedure

1) Place an ash sample equivalent to 1 kg of fresh mass in a beaker[*328], moisten it with pure water, and add exactly 10 mL of Sr carrier solution (Sr: 100 mg/mL)[*329].
2) Add aqua regia and evaporate on a hot plate to dryness.
3) Add nitric acid, and evaporate to dryness again[*330].
4) Add 100 mL to 300 mL of hydrochloric acid (1+1), and heat on a hot plate for 1 hour.
5) Filter by suction using a Büchner funnel and filter paper (No. 5C), and wash the residue with warm pure water.
6) Combine the filtrate and washings, transfer to a beaker[*331], and add pure water to 70% of

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[*328] The standard beaker size is 1 L beaker for 10 g to 20 g of ash, 2 L beaker for 30 g of ash, and 3 L beaker for 40 g to 50 g of ash.

[*329] When radioactive Cs is analyzed simultaneously by radiochemical analysis, a Cs carrier is also added.

[*330] If organic matter (black or dark brown) remains in the sample, add nitric acid and decompose by heating.

[*331] Standard beaker sizes to be used are 1 L beaker for 10 g of ash, 2 L beaker for 20 g of ash, and 3 L beaker for 30 g or more of ash.

the beaker volume[*332].

7) Perform the analytical procedure described in 1.3.3 and the subsequent steps.[*333]

6.3.3 Quantification of stable Sr

Same as 6.1.4.

6.3.4 Quantification of Ca

Same as 6.1.5.

6.3.5 Quantification of recovered Sr

Same as 3.1.5[*334]. However, note that as the oxalate method does not remove Ca, quantification by gravimetric analysis is not possible. In addition, since the amount of Sr carrier to be added is larger than with other methods, attention should also be paid to the aliquot volume, dilution factor, and concentration of the calibration solution. Further, (c) when performing atomic absorption spectrometry, it is necessary to add lanthanum nitrate solution (0.2 w/v%).

[*332] It is better to add 5 mL of a Ca carrier solution (Ca2+: 50 mg/mL) to samples with low Ca content such as milled rice and tea leaves.

[*333] The standard amount of sodium carbonate to be added is 10 g per 10 g of ash, up to a maximum of 30 g. The standard beaker size used for producing oxalate precipitation is a 1 L beaker for 10 g to 20 g of ash, a 2 L beaker for 30 g to 40 g of ash, and a 3 L beaker for 50 g or more of ash. The standard amount of oxalic acid to be added is 30 g for fish and skim milk, 20 g for seaweed and dry milk, and 10 g for others. For samples with a high Ca content (approximately 3 g or more), procedures 5.3.2 2) to 34) for seawater samples should be applied.

[*334] In the case of ash samples, the Sr concentration (C_{sr}) in the sample is expressed in mg/g ash, and the test quantity (V) is expressed in W and in g ash.

Chapter 7 Measurement

When using an LBC, ^{90}Sr is quantified by separating its daughter nuclide ^{90}Y, which is in radioactive equilibrium with ^{90}Sr, and measuring the activity of ^{90}Y. ^{89}Sr is quantified by measuring the radioactivity of strontium carbonate, separating ^{90}Y from the strontium carbonate, measuring the radioactivity of ^{90}Y, calculating the (^{90}Sr+^{90}Y) count from the obtained ^{90}Sr, and subtracting the (^{90}Sr+^{90}Y) count from the strontium carbonate count. When using an LSC, ^{89}Sr is quantified by measuring it immediately after scavenging. ^{90}Sr is measured after ^{90}Y growth and quantified by subtracting the ^{89}Sr count from the obtained count.

7.1 Devices, equipment, and reagents

(1) Devices

1) Water purification system
2) Hot plate: can be set to approximately 80°C to 250°C. A sand bath type is also acceptable.
3) Water bath
4) pH meter
5) Infrared lamp
6) Aspirator: Effective exhaust speed: approximately 12 L/min to 15 L/min, ultimate pressure: approximately 6.6 x 10^3 Pa
7) Electric furnace: Temperature can be set to 450°C to 600°C, temperature rise time to 3 hours, and holding time to 3 to 48 hours.
8) Electric dryer: Temperature can be set from room temperature to approximately 120°C
9) LBC
10) LSC

(2) Devices

1) Volumetric pipette (or micropipette): 5 mL, 10 mL
2) Graduated cylinder (or dispenser)
3) Evaporating dish
4) Beaker: 100 mL
5) Watch glass: 75 mm diameter
6) Filter paper
   - No. 5A: 11 cm diameter
   - No. 5C: 24 mm diameter
7) Funnel: 65 mm diameter x 8 mm x 150 mm
8) Suction flask: 1 L, borosilicate glass
9) Separable filter assembly: Glass, see Figure 7.1 below.
10) Sample dish: 1 inch diameter x 6.2 mm stainless steel
11) Starch paste
12) Desiccator: Vacuum type is preferred. A desiccant type may be used.
13) Filter paper holder: See Figure 7.3 below.
14) Volumetric flask: 100 mL, 1 L

(3) Reagents

Reagents that conform to Japanese Industrial Standards (JIS) should be used, and for those not specified in the standards, the highest possible purity should be selected. The weight and volume figures in the text are merely illustrative of the percentage of preparation and may be adjusted as necessary. The reagent prepared by mixing reagent volume a and water volume b is described as

the reagent name (a+b)[*335].

1) Pure water: Water purified by ion exchange, etc.[*336]
2) Hydrochloric acid (HCl)[*337,*338,*339]: Special grade reagent
   - Hydrochloric acid (1+1): Mix one part of hydrochloric acid with one part of pure water by volume.
   - Hydrochloric acid (1+5): Mix one part of hydrochloric acid with five parts of pure water by volume.
3) Oxalic acid dihydrate ($H_2C_2O_4 \cdot 2H_2O$)[*338,*339]: Special grade reagent
4) Nitric acid (HNO_3)[*337,*338,*339]: Special grade reagent, mass fraction 60% to 61%
   - Nitric acid (1+1): Mix one part of nitric acid to one part of pure water by volume.
   - Aqua regia: Mix three parts of hydrochloric acid with one part of nitric acid by volume. Prepare immediately before use.
5) Aqueous ammonia (NH3)[*337,*338,*339]: Special grade reagent
   - Aqueous ammonia (1+1): Mix one part of aqueous ammonia with one part of pure water by volume.
   - Aqueous ammonia (1+100): Mix one part of aqueous ammonia with one hundred parts of pure water by volume.
   - Aqueous ammonia (1+500):Mix one part of aqueous ammonia with five hundred parts of pure water by volume.
6) Yttrium oxide (Y_2O_3)[*338]: Mass fraction of 99.99% or higher
   - Y carrier solution (Y^{3+}: 10 mg/mL): Dissolve 1.270 g of yttrium oxide in 50 mL of nitric acid (1+1) while heating. Allow it to cool, then transfer it to a 100 mL volumetric flask, fill to the mark with pure water, and mix well.
   - Y carrier solution (Y^{3+}: 1 mg/mL): Dissolve 1.270 g of yttrium oxide in 50 mL of nitric acid (1+1) while heating. Allow it to cool, then transfer it to a 1 L volumetric flask, fill to the

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[*335] JIS K 0050:2019 "General rules for chemical analysis," states that it is applicable only to reagents with a purity, molar concentration and density described in "reagents that can be expressed by mixing ratio with water."

[*336] All water used for analysis shall be pure water. For the quantitative analysis of elements using ICP-AES or atomic absorption spectrometry, as described in JIS K 0116: 2014 "General rules for atomic emission spectrometry" and JIS K 0121:2006 "General rules for atomic absorption spectrometry," use A3 or A4 pure water (resistivity 1 MΩ•cm or more, etc.) as specified in JIS K 0557. Further, it is preferable to use water of higher purity (ultrapure water: specific resistivity of 18.2 MΩ•cm or more).

[*337] Class 3 substances in the Ordinance on Prevention of Hazards due to Specified Chemical Substances (substances that can cause acute poisoning due to mass leakage): It is necessary to take measures to reduce the exposure concentration, such as installing a local exhaust ventilation system.

[*338] Substances subject to hazardous substances labeling under the Industrial Safety and Health Law (those that may cause danger or health impairment to workers): Labeling, issuance of Safety Data Sheets, and risk assessment (risk reduction measures, etc.) are obligatory, and workers must be informed and educated regarding these matters.

[*339] Substances covered by the Poisonous and Deleterious Substances Control Act (those with a high risk of causing damage to health due to acute toxicity): Storage and display areas must be labeled as "non-medicinal poisonous substances" and "non-medicinal deleterious substances," and necessary measures must be taken to prevent theft, loss, leakage, etc.

mark with pure water, and mix well.

7) ^{90}Sr standard solution[*340]
8) ^{89}Sr standard solution[*340]
9) Electrolytic iron powder: Mass fraction 95.0% or more, particle size (150 μm passage) 95% or more
   - Iron(III) chloride–Y mixed carrier solution ((Fe^{3+}: 1 mg + Y^{3+}: 10 μg)/mL): Accurately weigh 1.000 g of electrolytic iron powder, dissolve it in aqua regia, allow the solution to cool, transfer the solution to a 1 L volumetric flask, add exactly 1 mL of Y carrier solution (Y^{3+}: 10 mg/mL), fill to the mark with pure water, and mix well.
10) Ammonium chloride (NH_4Cl)[*338]: Special grade reagent
11) Ammonium carbonate ($(NH_4)_2CO_3$): Special grade reagent
    - Ammonium carbonate solution (saturated): Place 200 g of ammonium carbonate and 250 mL of pure water in a well-sealed container, shake thoroughly, and allow to stand. Use the supernatant after the undissolved ammonium carbonate has settled. After use, it can be used as a saturated solution by adding pure water and shaking as long as the solid ammonium carbonate remains at the bottom of the container.
12) Ethanol (C_2H_5OH)[*338,*341]: Special grade reagent
13) Collodion[*338,*341,*342]: Grade 1 reagent, nitrocellulose (pyroxylin) 5.0% to 5.5%
    - Collodion-ethanol solution: Mix twenty parts of ethanol with one part of collodion by volume.
14) Phenolphthalein: Special grade reagent, transition range: pH 7.8 to pH 10.0
    - Phenolphthalein solution: Place 0.5 g of phenolphthalein in a 500 mL beaker, dissolve in 250 mL of ethanol, and add pure water to make 500 mL.

[*340] The Act on the Regulation of Radioisotopes, etc. applies. These substances are subject to permission or notification for their use, notification for businesses engaging in sale or lease, and permission for businesses engaging in disposal. On March 18, 2020, Nuclear Regulation Authority Notification No. 6, "On the Specification of Quantities of Radioisotopes Emitting Radiation, etc." set forth the minimum (notification) quantities for each radionuclide. Facilities using radioisotopes must calculate, for each radionuclide in their possession, the ratio of the activity held (Bq) to the notification quantity (Bq), and ensure that the sum of these ratios for all radionuclides does not exceed 1. In June 2005, the Enforcement Order of the Act on the Regulation of Radioisotopes, etc. introduced the concept of a minimum quantity for each nuclide. If the amount handled is below the minimum quantity, use is permitted outside controlled areas. For practical application, refer to the "Safety Handling Manual for the Use of Unsealed Radioisotopes Below the Minimum Quantity" published by the Japan Radioisotope Association. (https://www.jrias.or.jp/report/cat1/308.html)

[*341] Classified as a Class 4 hazardous material (flammable liquid) by the Fire Service Act. Appropriate precautions are required, such as keeping the substance away from open flames or other sources of ignition, storing and using it at temperatures below its flash point, keeping hazardous materials of Class 1 and Class 5 away from it, preventing the generation of static electricity, and not filling containers to their full capacity when transferring it to smaller containers (to avoid the risk of container damage due to thermal expansion).

[*342] Class 2 organic solvents under the Ordinance on Prevention of Organic Solvent Poisoning: Measures must be taken to reduce airborne concentrations, such as installing devices that enclose sources of organic solvent vapors and providing local exhaust ventilation systems in the workplace.

7.2 ^{90}Y separation (Milking)

From the strontium carbonate precipitate or the Sr-containing solution, for which the recovery rate has been determined according to the analytical procedures in each chapter, separate ^{90}Y using the iron(III) hydroxide coprecipitation method or the yttrium oxalate precipitation method, respectively.

Dissolve the strontium carbonate precipitate[*343] in hydrochloric acid (1+1), transfer the solution to a 100 mL beaker, and add pure water to adjust the volume to approximately 50 mL.

Transfer the solution containing Sr to a 100 mL beaker and increase its concentration on a hot plate until the volume is approximately 30 to 40 mL.

7.2.1 Iron (III) hydroxide coprecipitation method

1) Add exactly 5 mL of an iron(III) chloride-Y mixed carrier solution ((Fe^{3+}: 1 mg+Y3+: 10 μg)/mL) to the sample solution.
2) Add 1 g of ammonium chloride and 0.5 mL of phenolphthalein solution, then heat it to drive off carbon dioxide.
3) Add aqueous ammonia[*344] gradually to the solution to precipitate iron(III) hydroxide until the solution turns red.
4) Add 1 mL of aqueous ammonia and heat to mature the precipitate.
5) Quickly filter with filter paper (No.5A) (record the date and time as the "milking date"), and wash the precipitate with warm aqueous ammonia (1+500).
6) Combine the filtrate and washings, acidify the solution with hydrochloric acid, and store it.[*345]
7) Place the beaker used for filtration in step 5) under the funnel, dissolve the precipitate completely with warm hydrochloric acid (1+5), and wash with warm hydrochloric acid (1+5) until no yellow Fe(III) color is observed on the filter paper. Then wash with 40 mL of warm pure water.
8) Perform steps 2) to 7) again, and then perform steps 2) to 4).

7.2.2 Yttrium oxalate precipitation method

1) Add exactly 10 mL of Y carrier solution (Y^{3+}: 1 mg/mL) and 10 g of ammonium chloride to the sample solution.
2) Heat to remove carbon dioxide, add aqueous ammonia little by little, and precipitate yttrium hydroxide to a pH of approximately 8[*344]. Continue heating for an additional approximately 10 minutes to mature the precipitate.
3) Quickly filter with filter paper (No.5A) (record the date and time as the "milking date"), and wash the precipitate with warm aqueous ammonia (1+500).
4) Combine the filtrate and washings, acidify the solution with hydrochloric acid, and store it.[*345]
5) Place the beaker used for filtration in step 3) under the funnel, dissolve the precipitate completely with a small amount of hydrochloric acid (1+1), and wash the filter paper with 50 mL of warm pure water.
6) Add 1 g of ammonium chloride to the sample solution obtained in step 5), and repeat steps

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[*343] When also quantifying ^{89}Sr, follow 7.4.2.

[*344] If carbon dioxide is contained in aqueous ammonia, Sr precipitates as carbonate and is filtered together with iron hydroxide precipitation. Therefore, use unopened aqueous ammonia containing little or no CO_2.

[*345] If left for more than two weeks, milking can be performed again.

2) through 5).

7) Warm the sample solution in a water bath, and add 2 g of oxalic acid dihydrate.
8) Add aqueous ammonia (1+1) and adjust the pH to 1.5 using a pH meter to precipitate yttrium oxalate.
9) After heating in a water bath for approximately 10 minutes, leave at room temperature for approximately 20 minutes.
10) Filter and mount the precipitate according to 7.3. Wash the precipitate with pure water and ethanol.
11) After measuring the radioactivity, place the precipitate with the filter paper in a porcelain crucible and heat at 800 °C for approximately 30 minutes.
12) It is weighed as yttrium oxide (Y_2O_3)[*346], and the recovery rate of Y carrier is determined.

7.3 Sample Mounting for Radioactivity Measurement

The chemical forms of the measurement samples in this method are iron(III) hydroxide, yttrium oxalate, and strontium carbonate.. A separable filter assembly such as that shown in Figure 7.1 is usually used to separate these precipitates, and the precipitate collected on the filter paper is mounted as a sample for radioactivity measurement.

1) Filter paper (No. 5C) is placed on the filter plate and clamped with the filter tube, and the precipitate is collected by suction filtration.
2) Any precipitate attached to the inner wall of the filter tube is washed down with aqueous ammonia (1+100) or ethanol.
3) For iron(III) hydroxide, the entire filter paper is affixed to a sample dish (e.g. stainless steel), then dried with an infrared lamp, then two or three drops of a collodion–ethanol solution (1:20, v/v) are added, and the dish is dried again (see Figure 7.2).
4) For yttrium oxalate and strontium carbonate, after drying the precipitate at 110°C, the filter paper with the precipitate is secured using a plastic filter paper holder (see Figure 7.3).

[*346] The ratio of the atomic weight of Y to the formula weight of yttrium oxide is 0.787.

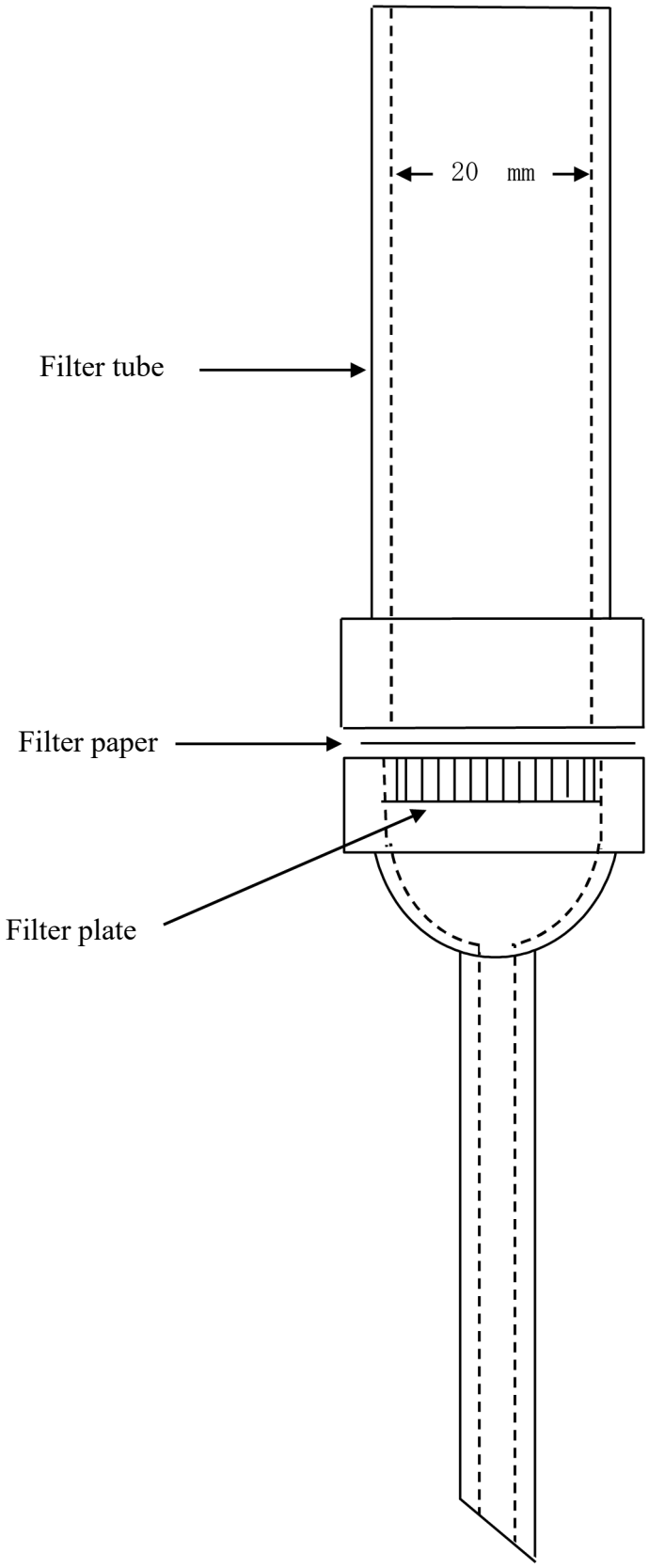


Figure 7.1 Separable filter assembly (example)

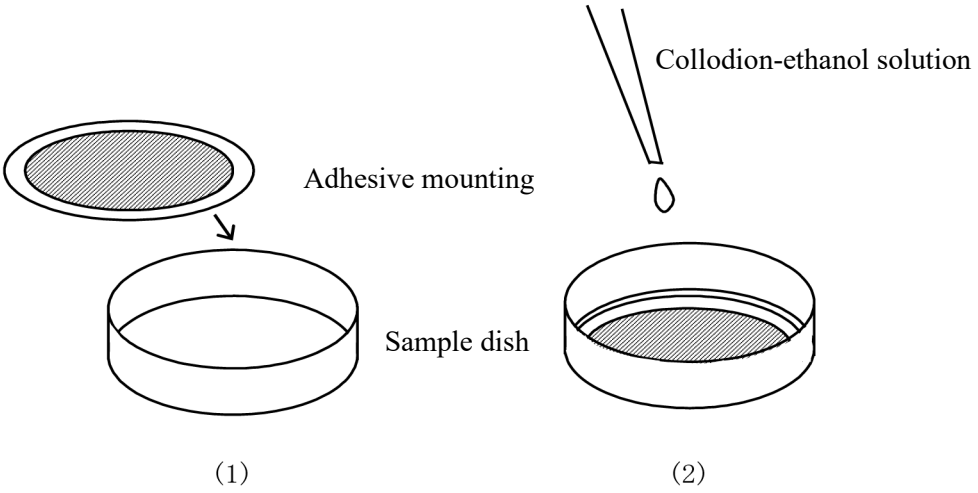


Figure 7.2 Sample dish method

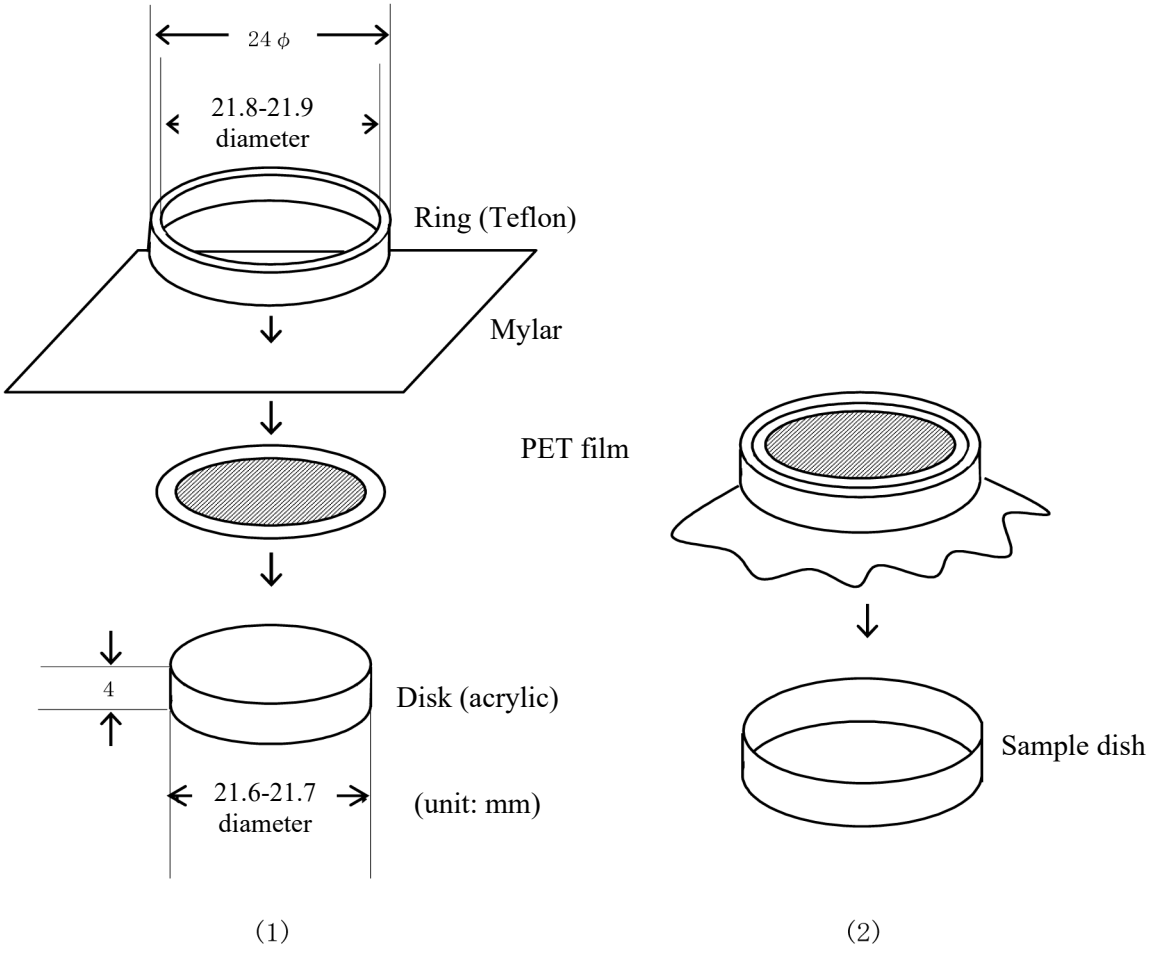


Figure 7.3 Method using a filter paper holder

7.4 Procedure for measuring radioactivity using an LBC

Measurements shall be made in the following order:

1) Record the model and name of the device used and the form of the measurement sample.
2) Using a suitable sealed β-ray check source (e.g. ^{137}Cs)[*347], verify that the device is operating properly.
3) Measure the background for t_{b1} minutes.
4) Measure the sample for t_s minutes.
5) After completing the series of measurements, measure the background for t_{b2} minutes again. The sum of the counts obtained in this step and in step 3) is taken as the background count N_b. Further, the sum of t_{b1} and t_{b2} is defined as the background measurement time (t_b).
6) Subtract the background counting rate from the sample counting rate to obtain the net counting rate r_1 (cpm) and its counting uncertainty σ_{r1} using the following equations:

$$r_1 \pm \sigma_{r1} = \left(\frac{N_s}{t_s} - \frac{N_b}{t_b}\right) \pm \sqrt{\frac{N_s}{t_s^2} + \frac{N_b}{t_b^2}}$$

N_s : Sample count
t_s : Sample counting time (min)
N_b : Background count
t_b : Background counting time (min)

7.4.1 Quantification of ^{90}Sr

7.4.1.1 Determination of the counting efficiency of ^{90}Y

Place a fixed amount of ^{90}Sr standard solution into several 100 mL beakers, separate ^{90}Y using the iron(III) hydroxide coprecipitation method 7.2.1 or the yttrium oxalate precipitation method 7.2.2, and measure its activity. The counting efficiency ε (%) is determined by dividing the ^{90}Y (cps) counting rate by the amount of ^{90}Sr (Bq) collected (Table 7.1). The counting efficiency is taken as the mean of the values obtained for several measurements.[*348]

Table 7.1 Counting efficiency ε for ^{90}Y in the iron(III) hydroxide coprecipitation method

(Example for 1 inch diameter, thin-window detector)

No.	Counting efficiency ε (%)
1	27.049
2	27.237
3	26.711
Average efficiency	26.999

7.4.1.2 Measurement of ^{90}Y samples

1) Measure ^{90}Y of the samples obtained using the Iron (III) hydroxide coprecipitation method described in 7.2.1 or the yttrium oxalate precipitation method described in 7.2.2 in

[*347] A constant count rate is to be checked continuously, so the shape of the β source and the radionuclide used are not critical. What matters is not which β source is used, but that the same β source is used consistently for the evaluation.

[*348] With the yttrium oxalate precipitation method, vary the amount of Y carrier and obtain several data points (for example, Y^{3+}: 6, 8, and 10 mg).

accordance with 7.4, and use $r_1 \pm \sigma_{r1}$ (cpm) for the counting rate. [*349]

2) Record the time t_1 (hour) from the date and time when the separation of ^{90}Y was performed (milking date and time) to the date and time when measurement of the sample was started.
3) The count rate $r_n \pm \sigma_{rn}$ (cpm) of ^{90}Y at the time of scavenging is calculated from t_1 using the following equation.[*349]

$$r_n \pm \sigma_{r_n} = \frac{r_1 \pm \sigma_{r_1}}{e^{-\lambda_2 \times t_1}} \times \frac{1}{(1 - e^{-\lambda_2 \times t_2})} \times \frac{100}{Y_Y}$$

Y_Y : Recovery rate (%) of Y carrier[*350]
t_1 : Time from the milking date and time to the measurement start date and time (hour)
t_2 : Time from scavenging date and time to milking date and time (hour)
λ_2 : Decay constant of ^{90}Y[*351]

7.4.1.3 Calculation of ^{90}Sr activity in samples

The ^{90}Sr activity $B_{90} \pm \sigma_{B90}$(Bq) contained in the sample is calculated using the following equation:

$$B_{90} \pm \sigma_{B_{90}} = \left(r_n \pm \sigma_{r_n}\right) \times \frac{100}{\varepsilon} \times \frac{1}{60} \times \frac{100}{Y_{Sr}}$$

$r_n \pm \sigma_{rn}$: Count rate of ^{90}Y at the scavenging time (cpm):
ε : ^{90}Y counting efficiency (ε)
Y_{Sr} : Chemical yield of Sr (%):

7.4.1.4 Calculation of radioactivity concentration by sample type

(1) Fallout samples

- The activity of ^{90}Sr in the fallout per 1 km^2 is calculated using the following equation:

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{10^6}{S} \times \frac{1}{10^6}$$

A : ^{90}Sr activity per km^2 (MBq/km^2)
S : Water receiving area (m^2)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

(2) Airborne dust samples

- The activity of ^{90}Sr in the airborne dust per 1 m^3 of air is calculated using the following equation:

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{1}{V_s} \times 10^3$$

A : Activity of ^{90}Sr per m^3 (mBq/m^3)

[*349] To confirm that the radioactivity of the measurement sample decays in accordance with the half-life of ^{90}Y (64.05 hours), the sample is measured once a day for three or four days. If the removal of ^{90}Sr is incomplete and the decay does not follow the half-life of 64.05 hours, the milking must be repeated. In actual environmental samples, the evaluation needs to take counting errors into account.

[*350] With the 7.4.1.1 iron(III) hydroxide coprecipitation method, the recovery rate of ^{90}Y is assumed to be 100 % for the calculation.

[*351] $\lambda_2 = 0.693/T_{90Y}$, where 0.693 is an approximation to the natural logarithm of 2 (ln 2), and T_{90Y} is the half-life of ^{90}Y. The unit of the half-life must be consistent with those of t_1 and t_2.

V_s : Air volume sucked in (m^3)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

(3) Inland water samples

- The activity of ^{90}Sr per L of inland water sample is calculated using the following equation:

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{1}{V_s} \times 10^3$$

A : Activity of ^{90}Sr per L (mBq/L)
V_s : Sample quantity (liter)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

- The activity of ^{90}Sr per 1 kg of inland water sample is calculated using the following equation.

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{1}{W_s} \times 10^3$$

A : Activity of ^{90}Sr per kg (mBq/kg)
W_s : Sample quantity (kg)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

(4) Seawater samples

- The activity of ^{90}Sr per L of seawater sample is calculated using the following equation:

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{1}{V_s} \times 10^3$$

A : Activity of ^{90}Sr per L (mBq/L)
V_s : Sample quantity (liter)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

- The activity of ^{90}Sr per 1 kg of seawater sample is calculated using the following equation.

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{1}{W_s} \times 10^3$$

A : Activity of ^{90}Sr per kg (mBq/kg)
W_s : Sample quantity (kg)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

(5) Soil and sea bottom sediment samples

- The activity of ^{90}Sr per kg of air-dried fine soil is calculated using the following equation.

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{10^3}{W_s}$$

A : Activity of ^{90}Sr per kg of air-dried fine soil (Bq/kg)
W_s : Sample quantity (g)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

- The ^{90}Sr activity[*352] in soil per 1 km^2 is calculated using the following equation:

$$A' \pm \sigma_{A'} = (A \pm \sigma A) \times \frac{R}{100} \times W_f \times \frac{10^{10}}{S} \times \frac{1}{10^6}$$

A' : ^{90}Sr activity per km^2 (MBq/km^2)

[*352] The activity of ^{90}Sr per 1 km^2 of soil at the sampling depth is determined, and the amount accumulated per 1 km^2 of soil is used. Not applicable to sea bottom sediment, etc.

A : Activity of ^{90}Sr per kg of air-dried fine soil (Bq/kg)
R : Dry rate (%)[*353]
W_f : Total weight of collected soil (kg)
S : Sampling area (cm^2):

(6) Ash samples

- The activity of ^{90}Sr per kg of fresh mass is calculated using the following equation:

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{R}{100} \times \frac{10^3}{W_s}$$

A : Activity of ^{90}Sr per 1 kg fresh mass (Bq/kg raw)
R : Total ash (%)
W_s : Sample quantity (g)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

- When monitoring the concentration of radioactive substances contained in a unit volume of liquid food (milk, etc.), the ^{90}Sr activity per 1 L is calculated using the following equation:

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{C}{W_s}$$

A : Activity of ^{90}Sr per L (Bq/L):
C : Total ash (g/L)
W_s : Sample quantity (g)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

- When monitoring the concentration of radioactive substances contained in the daily diet rather than a single sample, the intake of ^{90}Sr activity per person per day in the daily diet is calculated using the following formula.

$$A \pm \sigma_A = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{C}{W_s}$$

A : ^{90}Sr activity intake per person per day (Bq/(person•day))
C : Amount of ash per person per day (g/(person•day))
W_s : Sample quantity (g)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

- When monitoring the relative content of radioactive material to Ca, the Sr unit is calculated using the following equation:

$$SU \pm \sigma_{SU} = \left(B_{90} \pm \sigma_{B_{90}}\right) \times \frac{100}{W_s \times R_{Ca}}$$

SU : Sr unit
W_s : Sample quantity (g)
R_{Ca} : Amount of Ca in ash (% in ash)
$B_{90} \pm \sigma_{B90}$: Activity of ^{90}Sr contained in the sample (Bq)

7.4.1.5 Decay correction to the sampling date (reference date for decay correction)

To obtain the ^{90}Sr activity (concentration) on the sampling date (reference date for decay correction), calculate $B_{90ref} \pm \sigma_{B90ref}$ (Bq) using the following equation:

The correction may be applied either after obtaining $B_{90} \pm \sigma_{B90}$ in 7.4.1.3, or after obtaining $A \pm \sigma_A$ or $A' \pm \sigma_A$ in 7.4.1.4.

[*353] The dry rate is obtained from the dry mass determined by drying the collected soil at 105°C to 110°

$$B_{90\mathrm{ref}} \pm \sigma_{\mathrm{B}_{90\mathrm{ref}}} = \frac{B_{90} \pm \sigma_{\mathrm{B}_{90}}}{e^{-\lambda_1 \times t_3}}$$

t_3 : Time interval from the scavenging date and time to the sampling date (reference date for decay correction)
(the time unit must be consistent with that used for the half-life)

$B_{90} \pm \sigma_{\mathrm{B}90}$: Activity of ^{90}Sr contained in the sample (Bq)
or $A \pm \sigma_{\mathrm{A}}$, $A' \pm \sigma_{\mathrm{A}'}$

λ_1 : Decay constant of ^{90}Sr[*354]

[*354] $\lambda_1 = 0.693/T_{90\mathrm{Sr}}$, where 0.693 is the approximation of the natural logarithm of 2 (ln 2), and $T_{90\mathrm{Sr}}$ is the half-life of ^{90}Sr. The unit of the half-life must be consistent with that of t_3.

7.4.2 Quantification of ^{89}Sr

7.4.2.1 Determination of the counting efficiency of (^{90}Sr+^{90}Y)

1) Place a fixed amount of ^{90}Sr standard solution into eight 100 mL beakers.
2) Add exactly 3, 5, 7, 9, 11, 13, 16, and 20 mL each of Sr carrier solution (Sr^{2+}: 5 mg/mL).
3) Add pure water to make approximately 50 mL, and add aqueous ammonia (1+1) to adjust the pH to approximately 9.
4) Add 5 mL of a saturated solution of ammonium carbonate to precipitate strontium carbonate.
5) Furthermore, heat on a hot plate for approximately 5 minutes to age the precipitate, and then allow to cool at room temperature.
6) Using a separable filter assembly of the same diameter as that used for actual samples (see Figure 7.1) and filter paper (No. 5C) of known mass, collect the precipitate by suction filtration.
8) Wash the precipitate with aqueous ammonia (1+100) and ethanol.
9) Dry the precipitate on the filter paper at 110°C for 1 hour, and allow to cool in a desiccator.
10) Weight the precipitate and determine the recovery rate of the Sr carrier.[*355]
11) Fix the precipitate with the filter paper using a filter paper holder (see Figure 7.3).
12) After standing for at least 2 weeks, measure the radioactivity according to 7.4.
13) From the ^{90}Sr activity (Bq)[*356] taken in step 1), the Sr carrier recovery rate determined in step 10), and the counting rate (cpm) of (^{90}Sr + ^{90}Y) obtained in step 12), determine the counting efficiency ε_1 (%) for (^{89}Sr + ^{90}Y).
14) Prepare a counting-efficiency curve showing the relationship with the mass of strontium carbonate (see Figure 7.4).

7.4.2.2 Determination of the counting efficiency for ^{89}Sr

Carry out the same procedure as in 7.4.2.1, using a ^{89}Sr standard solution. However, the radioactivity of the precipitate may be measured immediately and does not need to be left for two weeks.

Prepare a counting-efficiency curve showing the relationship between the mass of strontium carbonate and the counting efficiency ε_2 (%) for ^{89}Sr (see Figure 7.5).

[*355] Atomic weight of Sr/formula weight of strontium carbonate = 0.594.

[*356] Note that ^{90}Y is not included.

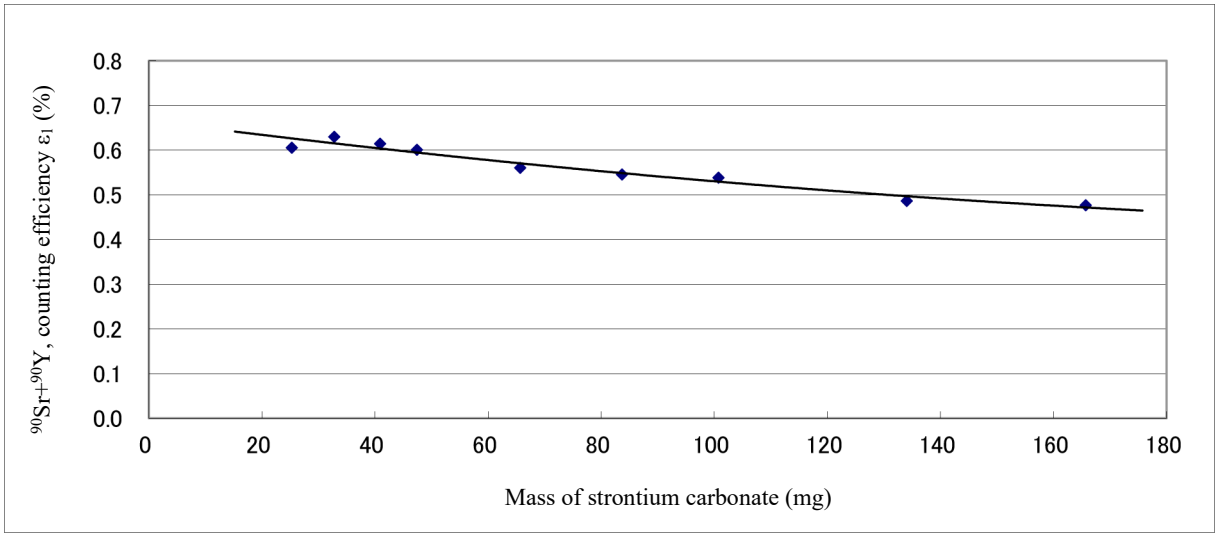


Figure 7.4 Relationship between the mass of strontium carbonate and the counting efficiency ε_1 of ($^{90}Sr + ^{90}Y$)
(example using a 1-inch thin-window detector)

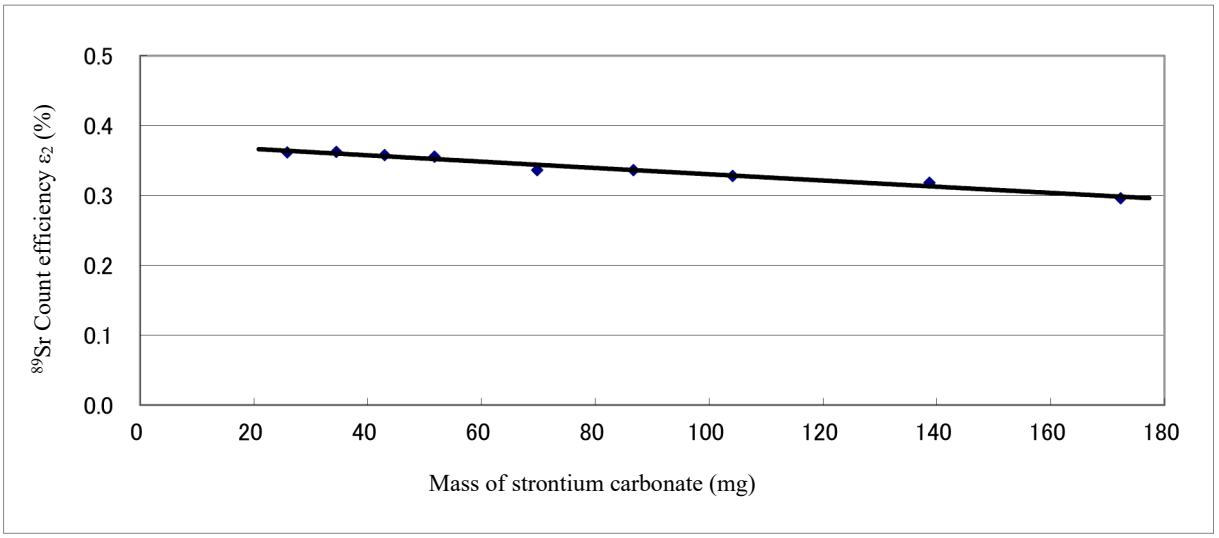


Figure 7.5 Relationship between the mass of strontium carbonate and the counting efficiency ε_2 for ^{89}Sr
(example using a 1-inch thin-window detector)

7.4.2.3 Calculation of ^{89}Sr in the sample [*357]

1) Measure the strontium carbonate sample that has been left to stand for at least two weeks in accordance with 7.4, and use $r_T \pm \sigma_{rT}$ (cpm) as the net counting rate for ($^{90}Sr + ^{90}Y + ^{89}Sr$).
2) Quantify the ^{90}Sr activity $B_{90} \pm \sigma_{B90}$ (Bq) in the strontium carbonate sample in accordance with 7.2.1.
3) Calculate the contribution of ($^{90}Sr + ^{90}Y$) to $r_T \pm \sigma_{rT}$, i.e. $r_{90} \pm \sigma_{r90}$ (cpm), using the following equation:

[*357] The detectable level of ^{89}Sr depends on the amount of ^{90}Sr in the sample, because in the course of calculating the activity the contribution derived from the ^{90}Sr activity is subtracted. If the ^{90}Sr activity concentration is assumed to be near the detection level of ^{90}Sr, then the detection level of ^{89}Sr is approximately three times that of ^{90}Sr.

$$r_{90} \pm \sigma_{r_{90}} = \left(B_{90} \pm \sigma_{B_{90}}\right) \times 60 \times \frac{\varepsilon_1}{100} \times \frac{Y_{Sr}}{100}$$

ε_1 : Counting efficiency (%) of (^{90}Sr + ^{90}Y)
Y_{Sr} : Recovery rate of Sr (%)

4) Calculate the contribution of ^{89}Sr to $r_T \pm \sigma_{rT}$, i.e. $r_{89} \pm \sigma_{r89}$ (cpm), using the following equation:

$$r_{89} \pm \sigma_{r_{89}} = \left(r_T \pm \sigma_{r_T}\right) - \left(r_{90} \pm \sigma_{r_{90}}\right) = (r_T - r_{90}) \pm \sqrt{{\sigma_{r_T}}^2 + {\sigma_{r_{90}}}^2}$$

5) Calculate the $_{89}$Sr activity in the sample, $B_{89} \pm \sigma_{B89}$ (Bq), using the following equation:

$$B_{89} \pm \sigma_{B_{89}} = \left(r_{89} \pm \sigma_{r_{89}}\right) \times \frac{100}{\varepsilon_2} \times \frac{1}{60} \times \frac{100}{Y_{Sr}}$$

ε_2 : Counting efficiency of ^{89}Sr (%)
Y_{Sr} : Recovery rate of Sr (%)

7.4.2.4 Calculation of activity concentration by sample type

(1) Fallout samples

- The ^{89}Sr activity in fallout per 1 km² is calculated by:

$$A \pm \sigma_A = \left(B_{89} \pm \sigma_{B_{89}}\right) \times \frac{10^6}{S} \times \frac{1}{10^6}$$

A : ^{89}Sr activity per 1 km² (MBq/km²)
S : Water receiving area (m^2)
$B_{89} \pm \sigma_{B89}$: ^{89}Sr activity contained in the sample (Bq)

(2) Airborne dust samples

- The ^{89}Sr activity in airborne dust per 1 m³ of air is calculated by:

$$A \pm \sigma_A = \left(B_{89} \pm \sigma_{B_{89}}\right) \times \frac{1}{V} \times 10^3$$

A : ^{89}Sr activity per 1 m³ (mBq/m³)
V : Air volume sucked in (m^3)
$B_{89} \pm \sigma_{B89}$: ^{89}Sr activity contained in the sample (Bq)

(3) Inland water samples

- The ^{89}Sr activity per 1 L of inland water is calculated by:

$$A \pm \sigma_A = \left(B_{89} \pm \sigma_{B_{89}}\right) \times \frac{1}{V} \times 10^3$$

A : Radioactivity of ^{89}Sr per L (mBq/L):
V : Sample quantity (liter)
$B_{89} \pm \sigma_{B89}$: ^{89}Sr activity contained in the sample (Bq)

- The activity of ^{89}Sr per 1 kg of inland water sample is calculated using the following equation.

$$A \pm \sigma_A = \left(B_{89} \pm \sigma_{B_{89}}\right) \times \frac{1}{W_s} \times 10^3$$

A : Activity of ^{89}Sr per kg (mBq/kg)
W_s : Sample quantity (kg)
$B_{89} \pm \sigma_{B89}$: ^{89}Sr activity contained in the sample (Bq)

(4) Seawater samples

- The activity of ^{89}Sr per L of seawater sample is calculated using the following equation:

$$A \pm \sigma_{\mathrm{A}} = \left(B_{89} \pm \sigma_{\mathrm{B}_{89}}\right) \times \frac{1}{V} \times 10^3$$

A : Radioactivity of ^{89}Sr per L (mBq/L):
V : Sample quantity (liter)
$B_{89} \pm \sigma_{B89}$: ^{89}Sr activity contained in the sample (Bq)

- The activity of ^{89}Sr per 1 kg of seawater sample is calculated using the following equation.

$$A \pm \sigma_{\mathrm{A}} = \left(B_{89} \pm \sigma_{\mathrm{B}_{89}}\right) \times \frac{1}{W_{\mathrm{s}}} \times 10^3$$

A : ^{89}Sr activity per L (mBq/kg)
W_{s} : Sample quantity (kg)
$B_{89} \pm \sigma_{B89}$: ^{89}Sr activity contained in the sample (Bq)

(5) Soil and sea bottom sediment samples

- The activity of 89Sr per kg of air-dried fine soil is calculated using the following equation.

$$A \pm \sigma_{\mathrm{A}} = \left(B_{89} \pm \sigma_{\mathrm{B}_{89}}\right) \times \frac{10^3}{W_{\mathrm{s}}}$$

A : Activity of ^{89}Sr per kg (Bq/kg)
W_{s} : Sample quantity (g)
$B_{89} \pm \sigma_{B89}$: ^{89}Sr activity contained in the sample (Bq)

- The ^{89}Sr activity[*358] in soil per 1 km^2 is calculated using the following equation:

$$A' = (A \pm \sigma_{\mathrm{A}}) \times \frac{R}{100} \times W_{\mathrm{f}} \times \frac{10^{10}}{S} \times \frac{1}{10^6}$$

A ' : ^{89}Sr activity per km^2 (MBq/km^2)
A : Activity of ^{89}Sr per kg of air-dried fine soil (Bq/kg)
R : Dry rate (%)[*359]
W_{f} : Total weight of collected soil (kg)
S : Sampling area (cm^2):

(6) Ash samples

- The activity of ^{89}Sr per kg of fresh mass is calculated using the following equation:

$$A \pm \sigma_{\mathrm{A}} = \left(B_{89} \pm \sigma_{\mathrm{B}_{89}}\right) \times \frac{R}{100} \times \frac{10^3}{W_{\mathrm{s}}}$$

A : Activity of ^{89}Sr per kg (Bq/kg)
R : Total ash (%)
W_{s} : Sample quantity (g)
$B_{89} \pm \sigma_{B89}$: ^{89}Sr activity contained in the sample (Bq)

- When monitoring the concentration of radioactive substances contained in a unit volume of liquid food (milk, etc.), the radioactivity of ^{89}Sr per 1 L of milk is calculated using the following equation:

[*358] The activity of ^{89}Sr per 1 km^2 of soil at the sampling depth is determined, and the amount accumulated per 1 km^2 of soil is used. Not applicable to sea bottom sediment, etc.

[*359] The dry rate is obtained from the dry mass determined by drying the collected soil at 105°C to 110°. R = (dry mass (g)/collected soil mass (g)) x 100

$$A \pm \sigma_{\mathrm{A}} = \left(B_{89} \pm \sigma_{\mathrm{B}_{89}}\right) \times \frac{C}{W_{\mathrm{s}}}$$

A : Radioactivity of ^{89}Sr per L (Bq/L):
C : Quantity of ash per L (g/L):
W_{s} : Sample quantity (g)
$B_{89} \pm \sigma_{\mathrm{B89}}$: ^{89}Sr activity contained in the sample (Bq)

- When monitoring the concentration of radioactive substances contained in food consumed daily, rather than a single sample, the ^{89}Sr activity per person per day in food consumed daily is calculated using the following equation:

$$A \pm \sigma_{\mathrm{A}} = \left(B_{89} \pm \sigma_{\mathrm{B}_{89}}\right) \times \frac{C}{W_{\mathrm{s}}}$$

A : ^{89}Sr intake per person per day (Bq/(person•day))
C : Amount of ash per person per day (g/(person•day))
W_{s} : Sample quantity (g)
$B_{89} \pm \sigma_{\mathrm{B89}}$: ^{89}Sr activity contained in the sample (Bq)

7.5 Procedure for measuring radioactivity using an LSC

Radioactivity is measured and calculated from the sample solution after scavenging using an LSC. For details, follow the Series of Environmental Radioactivity Measuring Methods No. 23, "AI-LSC Radionuclide Analysis using Liquid Scintillation Counter."

7.6 Uncertainty

Uncertainty of measurement is used as an indicator of the reliability of a measurement result. It is defined as follows. Measurement uncertainty is a parameter associated with a measurement result that characterizes the variation in a value, which can reasonably be attributed to the quantity being measured.[*360] It is important to note that measurement uncertainty is a value that represents the potential variability of a set of measurements, not the variability of each measurement result.

In conventional radioactive Sr analysis, it was a common practice to determine and report only the statistical uncertainty (counting error) of counts in radioactivity measurements. Although counting uncertainty is dominant in the analysis of low-level environmental samples for Sr, uncertainty factors exist in the entire radioactive Sr analysis process, including pretreatment and measuring device calibration, etc. Therefore, evaluation of uncertainty is required at each step. By evaluating the uncertainty, it is possible to judge the agreement/disagreement of the radioactive Sr analysis results of each analytical laboratory regarding the skill test and compare laboratories. Furthermore, it is also possible to improve the quality of analysis results by extracting the process with the largest uncertainty and reducing the uncertainty.

Refer to Explanation C for details regarding the method used for uncertainty evaluation in radioactive Sr analysis.

7.7 Lower detection limit

The lower detection limit in radionuclide analysis varies depending on the analytical sample quantity, recovery rate, measurement time, background count rate, etc., and it shall be set according to the purpose of analysis and measurement. The determination of whether radioactivity is detected or not is generally made based on whether or not the net counting rate exceeds three times the statistical uncertainty of the net counting rate. Calculating the lower detection limit provides an indication of the radioactivity level at which measurement is possible. Environmental

[*360] JIS Z 8404-1: Uncertainty of measurement–Part 1, JIS Z 8404-1:2018 (2018)

radioactivity is often weak and undetectable, so it is advisable to obtain the lower detection limit when measuring analytical samples. Note that radioactivity will most likely be detected if it exceeds the lower detection limit, and if it is at the limit or lower, it is not necessarily undetectable and may still be detected.

The calculation methods used for the lower detection limit include Cooper's method[*361], Kaiser's method[*362, *363], and the evaluation method according to the international standard ISO 11929[*364], etc. In the concept for each lower detection limit, the distribution of measured values must be considered. When measurement of a measurement target is repeated a sufficient number of times, the measured value is expressed by a mathematical model called a normal distribution or a Gaussian distribution with the mean as the top as shown in Figure 7.6.

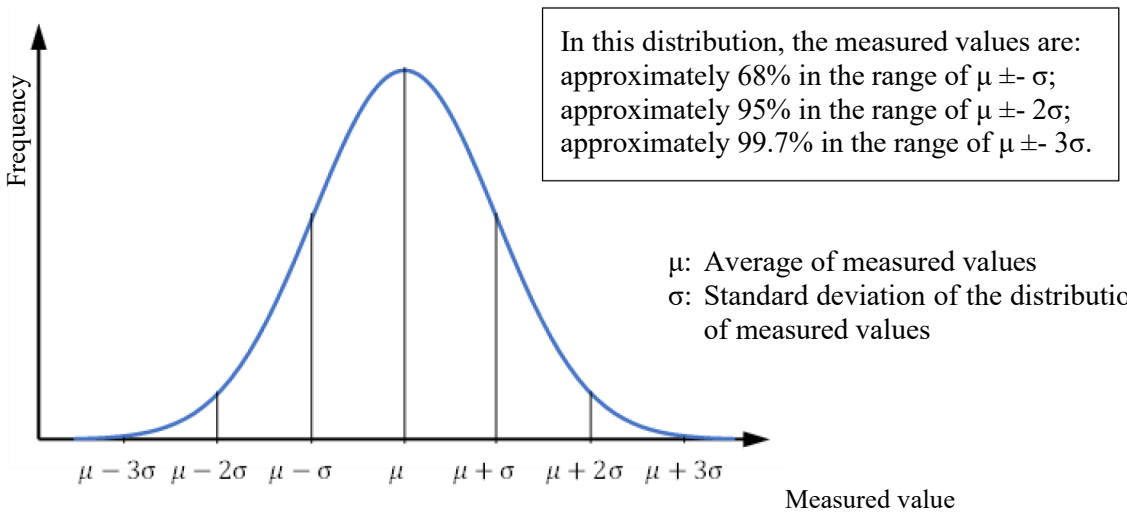


Figure 7.6 Distribution of measured values

In this measurement method, the Kaiser method and the evaluation method according to ISO 11929 are described. Up to now, the Kaiser method has been used for the derivation of the lower detection limit, as it can be derived relatively easily using the sample measurement time, background measurement time, and background counting rate. However, from the perspective of unifying the international comparison of national measurement standards, the general uncertainty of radiation measurement and the related decision threshold and detection limit are specified by ISO 11929 and have begun to be used for evaluation. The calculation method used for the lower detection limit for each measurement is to be selected by the user, but it is necessary to clearly state which method was used for the evaluation as necessary. Specific calculation examples are described in Explanation D.

[*361] J.A. Cooper: Factors determining the ultimate detection sensitivity of Ge (Li) gamma-ray spectrometers, Nuclear Instruments and Methods, 82,273-277(1970)

[*362] H.Kaiser: Zum Problem der Nachweisgrenze, Z.Analyt.Chem., 209,1-18(1965)

[*363] IUPAC Analytical Chemistry Division Commission on Spectrochemical and Other Optical Procedures for Analysis: Nomenclature, Symbols, Units and their Usage in Spectrochemical Analysis-III. Analytical Flame Spectroscopy and Associated Non-Flame Procedures, Pure & Appl. Chem., 45,105-123(1976)

[*364] ISO 11929-4:2022, Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 4:Guidelines to applications. ISO 11929-4:2022(2022)

Chapter 8 Quality Assurance

In addition to conducting analyses and measurements in accordance with the procedures described in this method, it is extremely important to ensure traceability to national standards, etc. and to conduct daily inspections to ensure the integrity of devices. Continuous implementation of these practices will demonstrate to third parties that the quality of analysis results is assured. To guarantee the quality of analysis results, it is desirable to follow the concept of ISO/IEC 17025, which can be broadly classified into internal quality control conducted by analyzers/measurers and external quality control in which analyzers/measurers participate in proficiency tests conducted by external organizations.

8.1 Internal quality control

8.1.1 Ensuring traceability

In radioactive Sr analysis, radioactivity (Bq) and weight (kg) are considered to be traceable. The traceability of the former is required for an LBC calibrated using a standard source, and the traceability of the latter should be ensured for electronic balances used in the preparation of measurement samples. When users prepare efficient radiation sources, they can obtain standard solution sources traceable to national standards, with calibration certificates issued by calibration laboratories under the calibration laboratories registration system based on the Measurement Act. For electronic balances, calibration services are provided by the respective manufacturers, and maintenance and inspection, including the issuance of calibration certificates, should be performed. Calibration of devices should be carried out at an appropriate frequency, and calibration expiration dates should be established and controlled. During the period of validity, daily inspections, as described in 8.1.2, should be performed to verify that no changes have occurred in devices that may affect analysis results.

Traceability should also be ensured for records, etc. accompanying analysis results. Specifically, it is important that collection record sheets in which the date and place of sample collection and unique numbers (analysis numbers, etc.) that identify analyzed samples are recorded, and various records and forms generated during analyses and measurement operations, are managed in a seamlessly linked manner.

8.1.2 Daily inspection

The inspection items and inspection methods for devices used for radioactive Sr analysis and β-ray measurement are described below. To confirm that devices are sound and can be used, it is necessary to establish acceptance criteria for each inspection item. It is recommended that provisional criteria be established using the initial data obtained experimentally, and once sufficient data has been accumulated, the acceptance criteria should be re-established[*365] through statistical processing. Because it is sufficient to confirm that no changes affecting analysis results have occurred in the device, a different method may be used to confirm this. Additionally, the daily inspection date, person in charge, and inspection results should be recorded.

If the daily inspection results do not meet the acceptance criteria, use of a device should be suspended, and the cause of the problem should be investigated and resolved. If the cause is not eliminated via re-measurement by the user or by resetting the device, inspection or repair by the

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[*365] It is convenient to determine the mean value and standard deviation, and use the range $\pm k\sigma$(k = 2 or 3) around the mean value as the acceptance standard. In addition, if device maintenance is performed or if repair, relocation, or changes to the standard source, etc. occur, confirm whether the acceptance criteria should be revised.

manufacturer should be performed.

(1) LBC

Periodically inspect the LBC and confirm through daily inspection that no changes have occurred in the device that will affect analysis results obtained before the next periodic inspection. In terms of frequency, it is recommended that this test be conducted every time a measurement is performed.

- Counting efficiency

  If a standard source containing a certain amount of radioactivity is measured under the same conditions and the obtained counts (or count rates) are within the acceptable standard, it can be judged that counting efficiency has not changed enough to affect the results. When checking changes in counting efficiency, it is necessary to consider the decay of the radioactive nuclides contained in the standard source.

- Installation environment

  Ensure that the LBC is installed in a location that has appropriate climate control and confirm that the temperature and humidity during use are maintained in an appropriate state. The room temperature in the installation environment should be approximately 23˚C to 25˚C, with a variation range of ±2˚C or less, and relative humidity should be kept at 30% to 80%.

(2) Electronic balance

Inspect the electronic balance periodically and confirm through daily inspection that no changes have occurred in the device that will affect analysis results obtained before the next periodic inspection. It is desirable to conduct pre-use inspections before each use and periodic inspections on a monthly basis.

- Pre-use inspection

  This inspection is to be performed before using the balance. Balances with built-in weights should be calibrated internally in advance. Conduct a pre-use inspection of the electronic balance using a weight[*366] that is close to the weight of the sample to be weighed (or at the lower limit of the usable range), and confirm that the weighing result is within the permissible standard.[*367] It is even better if the same inspection is also performed after using the electronic balance.

- Periodic inspection

  Check the sensitivity of the electronic balance using a weight close to the maximum weight that can be weighed by the electronic balance to be used. Balances with built-in weights should be calibrated internally in advance. The grade and acceptance criteria of weights used may conform to those employed in pre-use inspection. This inspection should be performed at regular intervals, and it is recommended that it be performed approximately once a month.

- Installation environment

  The electronic balance is susceptible to the effects of temperature changes, static electricity, and vibration, so be sure to install it in a location that has appropriate climate control and confirm that the temperature and humidity during use are maintained in an appropriate state. Additionally, it should be used on a stable table, and use in locations exposed to direct sunlight or wind or in high-traffic areas should be avoided. The ideal

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[*366] Weights with calibration certificates (OIML (JIS) standard F2 or higher) are preferred.

[*367] Although the tolerance criteria must be determined based on weighing uncertainty, minimum weighing value, and safety factor, an initial control range of 0.5% may be set (e.g., for a 10 g weight, the tolerance criteria will be 9.95 g to 10.05 g.

temperature and humidity during use are a room temperature of approximately 18˚C to 22˚C and a relative humidity of 45% to 60%.

(3) ICP-AES

Periodically inspect the ICP-AES and confirm through daily inspection that no changes that will affect analysis results have occurred in the device before the next periodic inspection. In terms of frequency, it is recommended that this test be conducted every time a measurement is performed.

- Measurement of a solution of known concentration

  If a solution of known concentration is measured under the same conditions and the obtained measurement result is within acceptable standards, it can be judged that no change in concentration has occurred that would affect results.

(4) Atomic absorption spectrometer

Periodically inspect the atomic absorption spectrometer and confirm through daily inspection that no changes have occurred in the device that will affect analysis results before the next periodic inspection. In terms of frequency, it is recommended that this test be conducted every time a measurement is performed.

- Measurement of a solution of known concentration

  If a solution of known concentration is measured under the same conditions and the obtained measurement result is within acceptable standards, it can be judged that no change in concentration has occurred that would affect results.

(5) LSC

Periodically inspect the LSC to ensure that no changes have occurred in the device that will affect analysis results. In terms of frequency, it is recommended that this test be conducted every time a measurement is performed.

- Counting efficiency

  If a standard source containing a certain amount of radioactivity is measured under the same conditions and the obtained counts (or count rates) are within the acceptable standard, it can be judged that counting efficiency has not changed enough to affect the results. When checking changes in counting efficiency, it is necessary to consider the decay of the radioactive nuclides contained in the standard source.

- Cooling performance

  An LSC equipped with a cooler maintains a constant temperature inside the measuring device. Maintaining a low temperature in the measuring device makes it is possible to reduce incidental coincidence counts caused by dark current (dark noise) generated in the photomultiplier tube and suppress changes in quenching caused by temperature changes in the measurement sample. The set temperature should be approximately 15°C (refer to the device's operation manual for the set temperature), and should be controlled within a fluctuation range of ± 3°C.

  The use of a thermometer with a sensor is convenient for controlling the temperature inside the measuring device. Checking the temperature without opening and closing the measuring device can suppress temperature changes inside the device and reduce the influence of photoluminescence caused by fluorescent molecules and visible light.

- Installation environment

  Install the LSC in a place where appropriate air-conditioning management is possible, and confirm that the installation environment is maintained. The room temperature should be approximately 23°C to 25°C, with a variation range of ± 2°C or less, and relative

humidity should be kept at 30% to 80%. In addition, although the quality of analysis results is not directly affected , when installing an LCS that is extremely heavy due to lead shielding it is essential that the load-bearing capacity of the floor is sufficient for the weight of the device.

8.2 External quality control

8.2.1 Interlaboratory comparison

The validity of the measurement and analysis process can be confirmed by conducting an inter-comparison analysis with another laboratory (preferably an ISO/IEC 17025-accredited laboratory) using the same sample to confirm that there is no significant difference between the results of the two analyses.

8.2.2 Proficiency test

The proficiency of the testing laboratory an be objectively demonstrated by participation in a proficiency test conducted by an external organization (preferably one with ISO/IEC 17043 accreditation) and comparison of the analysis results of the tested product with the added value (assigned value). In the case of the proficiency test based on ISO/IEC 17043, performance evaluation methods for participating laboratories include z-scores and E_n scores, etc.v

Explanation

Explanation A Study on column size for the ion exchange method (from revision 4 in 2003)

The ion exchange method described in Radioactive Sr Analysis (Revision 3, 1983) can be applied to samples containing less than 1 g of Ca. However, as shown in Table A.1, the amount of Ca in target samples is often greater than 1 g and less than or equal to 5 g. Therefore, in order to make the method applicable to a wider range of samples, we examined column dimensions for ion-exchange resin that allow handling of up to 5 g of Ca. For samples containing more than 5 g of Ca, it was decided that this should be addressed by increasing the number of columns used.

Table A.1 Amounts of Ca, etc. in samples for environmental radioactivity surveys

Sample name	Sample quantity	Ca (g/analytical sample)	Sr (mg/analytical sample)
Fish (whole)	1 kg (fresh)	2 to 19	7 to 100
Seawater	40 L	17	140 to 330
Powdered milk	1 kg (fresh)	4 to 13	2 to 5
Sea bottom sediment	100 g (dry soil)	(in some samples ≥ 5 g)	3 to 400
Fish (muscle)	1 kg (fresh)	0.05 to 4	0.1 to 17
Shellfish	1 kg (fresh)	0.05 to 4	2 to 30
Spinach	1 kg (fresh)	0.2 to 17	0.4 to 5
Seaweed	1 kg (fresh)	0.6 to 1.6	40 to 130
Milk	1 L	0.9 to 1.3	<0.1
Daily diet	1 person, 1 day	0.2 to 1.1	0.6 to 7
Inland water	100 L	(approximately 1)	1 to 50
Soil	100 g (dry soil)	0 to 1	0.1 to 40
Tea	200 g (dry matter)	0.1 to 0.9	0.3 to 3
Radish	1 kg (fresh)	0.1 to 0.4	0.3 to 2
Polished rice	1 kg (fresh)	0.02 to 0.06	<0.1
Fallout	1-month sample	—	—

*Samples names are listed in descending order of Ca content. Of the approximately 1,500 samples analyzed annually, approximately 97% contain 5 g or less of Ca.

A.1 Setting the conditions for column operation

Solutions (500 mL each) were prepared containing 0.5 g, 3 g, 4 g, or 5 g of Ca, together with 50 mg of Sr, 50 mg of Ba, 50 mg of Bi, and 50 mg of Pb, in a hydrochloric acid (1+23) medium. Then, under the experimental conditions described below, elution curves were obtained. Ba, Bi, and Pb were added to investigate the effect of the naturally occurring radionuclides ^{226}Ra, ^{210}Bi, and ^{210}Pb on the analytical results, and to determine their decontamination factors.

(1) Experimental conditions

1) Strongly acidic cation-exchange resin A : Crosslinking degree 8%, 100 to 200 mesh, H^+ form
2) Column size and flow rate : 3 cm ID × 26 cm Length, approximately 5 mL/min
3) Eluents : Eluent A: 1000 mL of ammonium acetate (15.4 w/v%)–methanol mixture (1:1, v/v), Eluent B: 500 mL of ammonium acetate solution (15.4 w/v%), Hydrochloric acid (1+2): 1750 mL

(2) Experimental results and discussion

As an example, the elution curve for 4 g Ca is shown in Figure A.1. Additionally, the Sr recovery rate and decontamination factor, etc., in the effluent of Sr eluent B are shown in Table A.2.

In all cases of Ca loading, the recovery rate of Sr was 90% or higher, and the maximum amount of Ca as an impurity was 0.4 mg. This amount of Ca corresponds to approximately 1 mg as carbonate and gives a positive error of approximately 1% in the recovery of Sr. However, considering the counting uncertainty of radioactivity measurements and other factors, this is not regarded as problematic here.

The Ba and Pb decontamination factor was more than 8×10^3. Although the Bi decontamination factor is one order of magnitude lower, it is removed in the subsequent iron (III) hydroxide coprecipitation process, and the decontamination factor in all processes is over 10^3. Each element's decontamination factor is sufficient considering the ^{226}Ra, ^{210}Bi and ^{210}Pb contained in the analytical sample.

Table A.2 Sr recovery rate, Ca content and the Ba, Bi and Pb decontamination factors in the effluent of eluent B

Test sample solution - Ca in sample -	Sr recovery rate (%)	Ca content (mg)	Decontamination factor		
			Ba	Bi	Pb
0.5g	91	0.06	4×10^5 or more	2×10^2	8×10^3 or more
3g	97	0.2	5×10^4	2×10^3	8×10^3 or more
4g	95	0.4	2×10^4	2×10^3	8×10^3 or more
5g	90	0.2	2×10^4	5×10^3	8×10^3 or more

1) Volume of eluent A

In the experiment, 1000 mL of eluent A was used. In the present analytical method, this volume is increased to 1100 mL in order to ensure more reliable removal of Ca.

2) Volume of eluent B

Approximately 1.0 mg of Sr was present in the first fraction of hydrochloric acid (1+2). Therefore, while eluent B was 500 mL in the experiment, it was 600 mL in this measurement method to increase the recovery rate and not to be affected by ^{226}Ra.

A.2 Experiments using another strongly acidic cation exchange resin

For strongly acidic cation-exchange resin B, which is another strongly acidic cation-exchange resin with the same nominal specifications but supplied by a different manufacturer, we examined whether each element could be separated under the same conditions.

(1) Experimental results and discussion

As an example, the elution curve obtained when the Ca content was 4 g is shown in Figure A.2.

Comparing Figure A.1 (experiment using strongly acidic cation-exchange resin A) and Figure A.2 (experiment using strongly acidic cation-exchange resin B), the elution behavior of the elements is different—for example, Ba appears in the effluent of eluent B in Figure A.2. Therefore, when strongly acidic cation-exchange resin B or any other resin different from resin A is used, it is necessary to determine the elution curves in advance and then establish appropriate analytical conditions.

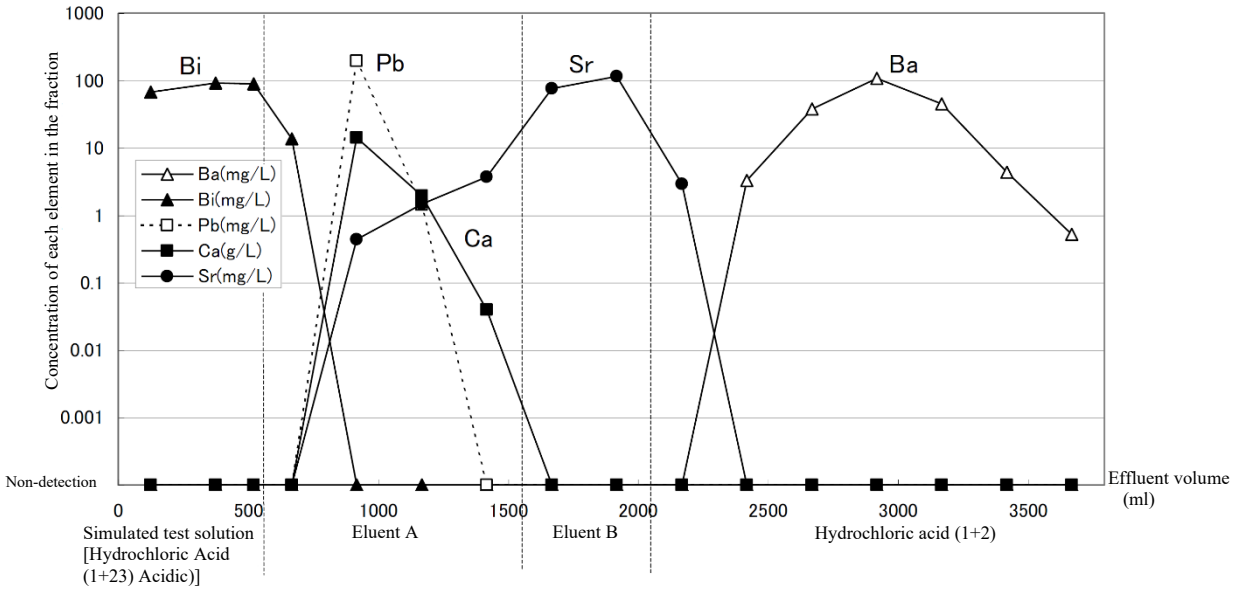


Figure A.1 Elution curves obtained with strongly acidic cation-exchange resin A (Ca content: 4 g)

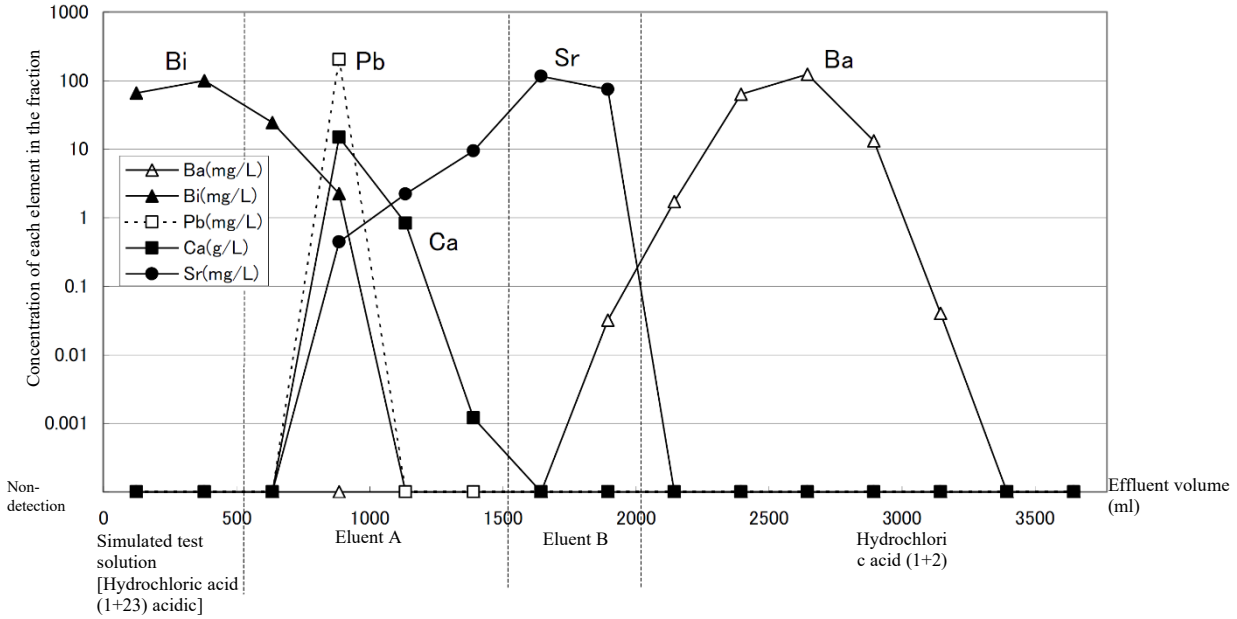


Figure A.2 Elution curves obtained with strongly acidic cation-exchange resin B (Ca content: 4 g)

Explanation B Preliminary Concentration of Seawater Samples ((from revision 4 in 2003)

When large volumes of seawater are analyzed by the ion-exchange method, separation of Mg (approximately 1.3 g/L) and Ca (approximately 420 mg/L) from Sr (approximately 8 mg/L) in seawater would require a long, large column, and performing such an operation is realistically difficult.

Therefore, as a method for analyzing 40 L of seawater, a two-stage column procedure was adopted: in the first-stage column, most of the Mg is removed, and in the second-stage column, a 3 cm ID × 26 cm Length column—the same as that used for other samples—is employed.

This section describes the elution curves and related data for the first-stage column.

B.1 Preliminary experiment (relationship with hydrochloric acid concentration)

To determine the optimum acid concentration of the seawater sample to be passed through the column, elution curves were obtained by varying the concentration of hydrochloric acid in the seawater sample.

(1) Experimental conditions

1) Strongly acidic cation exchange resin : Crosslinking degree 8%, 100 to 200 mesh, H^+ form
2) Column size and flow rate : 3 cm ID × 26 cm Length, approximately 3 mL/min
3) Amount of seawater sample : 5 L
4) Hydrochloric acid concentration in seawater samples : Hydrochloric acid (1+23), (1+120), (1+600), and neutral
5) Eluents : Hydrochloric acid (1+2) 1 L

(2) Experimental results and discussion

Table B.1 Sr recovery rate and related data in the hydrochloric acid (1+2) fraction

Hydrochloric acid concentration	Sr recovery rate (%)	Ca remaining (%)	Mg remaining (%)
Hydrochloric acid (1+23) acidic	57	36	15
Hydrochloric acid (1+120) acidic	89	56	21
Hydrochloric acid (1+600) acidic	98	60	23
Neutral	97	61	23

The Sr recovery rates in neutral or acidic hydrochloric acid (1+600) were 97% or higher. In practice, when seawater samples are collected, hydrochloric acid (1+1) or nitric acid (1+1) is usually added at 2 mL per 1 L of seawater (The Series of Environmental Radioactivity Measuring Methods No. 16 "Environmental Sampling Method"). As a result, the sample becomes approximately (1+1000) acidic, and can be passed through the column without further adjustment.

B.2 Elution curves for the first-stage column

Based on the above experimental results, the seawater sample (40 L) was adjusted to hydrochloric acid (1+600) acidity, and the experiment was carried out under the following

conditions.

(1) Experimental conditions

1) Strongly acidic cation exchange resin : Crosslinking degree 8%, 100 to 200 mesh, H^+ form
2) Column size and flow rate : 9 cm ID × 26 cm length, approximately 30 mL/min
3) Seawater sample : 40 L, adjusted to hydrochloric acid (1+600) acidity
4) Eluents : Eluent A: 2.50 L of ammonium acetate (15.4 w/v%)–methanol mixture (1:1, v/v), Hydrochloric acid (1+2): 6.00 L

(2) Experimental results and discussion

The elution curves for Sr, Ca, and Mg are shown in Figure B.1. The recovery rate of Sr in the hydrochloric acid (1+2) eluent was 97%, and the residual fractions (amounts remaining) of Ca and Mg were 30% (approximately 5 g) and 0.3% (approximately 0.2 g), respectively.

Thereafter, Sr is separated and purified by the revised ion exchange method described in Explanation A.

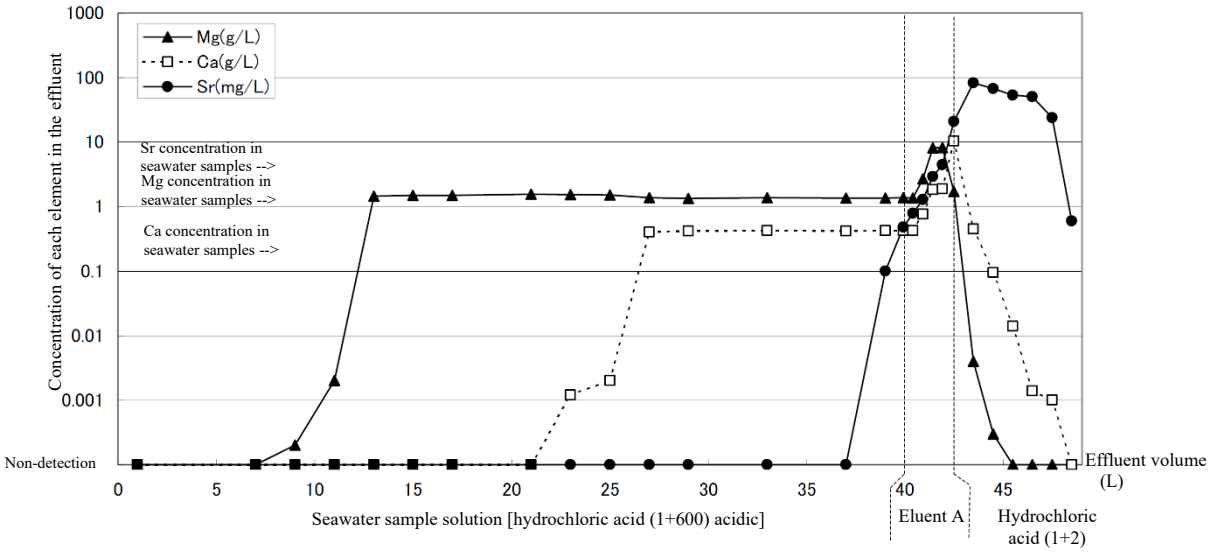


Figure B.1 Elution curves of Sr, Ca, and Mg in the preliminary concentration of seawater samples

Explanation C Example of uncertainty evaluation

Regarding how the reliability of measurement results is evaluated and expressed, traditionally several differing concepts existed across countries and technical fields, leading to confusion. Consequently, the International Committee for Weights and Measures (CIPM) raised this issue in the late 1970s. This spurred work, involving several international organizations concerned with metrology, to establish a unified method for the evaluation and expression of the reliability of measurement results. The outcome of this work was the "Guide to the Expression of Uncertainty in Measurement" (hereinafter referred to as "GUM"), published in 1993 under the joint auspices of seven international organizations, including the International Organization for Standardization (ISO). is the default. GUM adopted the position that quantities that are fundamentally unknowable, such as "true value" and "error," should not be introduced into the evaluation procedure. To distinguish it from similar conventional concepts, "uncertainty," which represents the dispersion of measurement results, was introduced as a new indicator of the reliability of a measurement result. Due to the high technical rationale and consistency recognized in the GUM concept of uncertainty evaluation, "uncertainty" has rapidly become widely used in various situations where the statement of measurement reliability is critical.

In environmental radiation monitoring, as the obtained measurement results are widely referenced both domestically and internationally, the need for them to be traceable to appropriate metrological standards has grown. Establishing the internationally agreed upon metrological traceability requires several essential elements, such as an uninterrupted chain of calibrations. The evaluation of measurement uncertainty is one of these elements, and the calculation and expression of results must be carried out based on the agreed method—specifically, the aforementioned GUM. Furthermore, in the proficiency test conducted by the International Atomic Energy Agency (hereinafter, IAEA), participating analytical laboratories are required to report measurement results including their uncertainty. Using "uncertainty," which serves as an internationally common indicator of the reliability of measurement results, ensures the international consistency, reliability, and comparability of the results. Moreover, upper limits that take uncertainty into consideration are sometimes compared with the standard value to demonstrate with high reliability that the radioactivity concentration in environmental samples does not exceed the standard value.

Thus, the evaluation of measurement uncertainty is widely established as the standard method for objectively demonstrating the reliability of environmental radiation monitoring. It is desirable for domestic analytical laboratories and other organizations to adopt this approach to secure the reliability of their own measurement results.

This explanation aims to enable individual analytical laboratories to understand the methods for evaluating various uncertainty factors and to evaluate the factors that should be considered. To this end, it describes the procedure for evaluating the uncertainty of each factor in ^{90}Sr analysis. Since the uncertainty factors are countless, including those with small contributions, it is difficult to estimate the uncertainty by considering all factors. Therefore, in practical application, it is not always necessary to evaluate all factors; and it is practical to focus on evaluating factors that make a significant contribution to the overall uncertainty. For reference, the uncertainty evaluation procedure and calculation examples for the actual analysis and measurement process are provided here in order to aid in the consideration of factors that should be taken into account.

C.1 Fundamentals of uncertainty evaluation

The measured quantity that is ultimately targeted in radionuclide analysis is the radioactivity concentration. However, regardless of how accurately the measurement is performed and how all biases of the measured quantity are corrected, the measurement result is an estimate, and not the true value of the measured quantity itself. Measurement uncertainty is evaluated as a parameter that expresses the reliability of this estimate. The values of all input quantities used in the calculation of the radioactivity concentration during the process from chemical analysis to radiation measurement are also estimates. The measurement uncertainty of the radioactivity concentration, which is the measured quantity, is calculated by combining the uncertainties of each estimate (input value).

The radioactivity concentration is calculated as a function of input values such as the mass of the sample used, the recovery rate, the count of the radiation emitted by the radionuclide in the measurement sample, the detector counting efficiency, the measurement time, and other correction factors.

$$A = f(x_1, x_2, x_3, \cdots, x_n) \qquad \text{(C. 1)}$$

A: Value of radioactivity concentration

$f(x_1,x_2,x_3,\cdots,x_n)$: Function that represents calculations required to derive the radioactivity concentration

The combined standard uncertainty of the radioactivity concentration to be determined is evaluated as the combined uncertainty of all input values involved in the derivation of the radioactivity concentration. If all input values are independent (uncorrelated), the uncertainty of each are combined according to the following equation:

$$u(A) = \sqrt{\sum_{i=1}^{n} \{c_i u(x_i)\}^2} \qquad \text{(C. 2)}$$

$u(A)$: Combined standard uncertainty of value A of the radioactivity concentration

$u(x_i)$: Standard uncertainty of input value x_i

c_i : Sensitivity coefficient of value A of the radioactivity concentration with respect to input value x_i, given by the differential coefficient ($\partial f/\partial$xi)

In equation (C.2), c_i (sensitivity coefficient) represents how much value A of the radioactivity concentration changes with respective changes in input values x_1, x_2, x_3, ⋯, x_n. For example, the change in value A of the radioactivity concentration caused by small change Δx_i in input value x_i is given by $(\Delta A)_i = c_i \Delta x_i$. The change in value A of the radioactivity concentration that reflects the standard uncertainty of input value x_i will be $|c_i|u(x_i)$.

Suppose that functional quantity f is $m < n$.

$$f(x_1, x_2, x_3, \cdots, x_n) = \frac{x_1 \cdot x_2 \cdot x_3 \cdot \cdots \cdot x_m}{x_{m+1} \cdot x_{m+2} \cdot x_{m+3} \cdot \cdots \cdot x_n} \qquad \text{(C. 3)}$$

When expressed only by multiplication and division of x_1, x_2, x_3, ⋯, x_n, as shown in, equation (C.2) is equivalent to the following equation:

$$\frac{u(A)}{A} = \sqrt{\sum_{i=1}^{n} \left\{\frac{u(x_i)}{x_i}\right\}^2} \qquad \text{(C. 4)}$$

$\frac{u(A)}{A}$: Relative combined standard uncertainty of value A of the radioactivity concentration

$\frac{u(x_i)}{x_i}$: Relative standard uncertainty of input value x_i

Therefore, the relative standard uncertainty of value *A* of the radioactivity concentration can be determined by calculating the square root of the sum of the squares of the relative standard uncertainties of the respective input values. In this explanation, equation (C.2) and equation (C.4) are used to evaluate measurement uncertainty.

C.2 Uncertainty evaluation procedure

The procedure for evaluating uncertainty is explained by taking the case of analyzing an ash sample using the ion exchange method as an example. Assuming that the amount of Sr is quantified using ICP-AES and the net count rate of ^{90}Y is measured using an LBC, the uncertainty of the radioactivity concentration per 1 kg of ash sample is calculated as follows.

(1) Organize uncertainty factors based on the calculation formula for the ^{90}Sr radioactivity concentration (hereinafter referred to as the "model equation").

Value A of the radioactivity concentration of ^{90}Sr is equal to the activity concentration of ^{90}Y of the daughter nuclide in radiative equilibrium, and is calculated using the following model equation.

$$A = \frac{r_{\mathrm{n}} \times 100}{W_{\mathrm{s}} \times Y_{\mathrm{Sr}} \times \varepsilon} \qquad (\mathrm{C}.5)$$

A : Radioactivity concentration of ^{90}Sr in the sample (Bq/kg ash)
W_s : Sample quantity (kg ash)
Y_{Sr} : Recovery rate (%)
ε : Counting efficiency of ^{90}Y (cps/Bq)
r_{n} : ^{90}Y net counting rate (cps)

Various uncertainty factors are conceivable for each item. Table C.1 shows an example of an uncertainty budget sheet created to organize the uncertainty factors. Note that there may be other factors that affect the measurement results in addition to those listed in Table C.1.

(2) Evaluate uncertainty for individual uncertainty factors.

Methods used for evaluating uncertainty are classified into two types: Type-A evaluation and Type-B evaluation.

Type-A evaluation is a method used for uncertainty evaluation based on the statistical analysis of a series of input values. For each factor, the experimental standard deviation is obtained from repeated measurements and is used as the standard uncertainty.

Type-B evaluation is a method employed for evaluating uncertainty by means other than the statistical analysis of a series of input values. Here, each factor is evaluated by scientific judgement based on all available information regarding possible variability. Available information includes the following:

- Manufacturer's specifications
- Data contained in calibration or other certificates
- Uncertainty assigned to reference data taken from a handbook
- General knowledge or experience of the behaviors and properties of materials and measuring devices

Not all uncertainty factors contribute considerably to the uncertainty of the measurement result being evaluated. After evaluating each uncertainty and combining uncertainties to determine their contribution to the overall uncertainty, items that contribute little can be omitted from evaluation.

(3) Combine all individual uncertainties to obtain the resultant uncertainty of measurements.

The relative combined standard uncertainty of the radioactivity concentration is obtained by calculating the square root of the sum of the squares of the relative standard uncertainties of the respective input values. The combined standard uncertainty of the radioactivity concentration is obtained by multiplying the relative combined standard uncertainty by the radioactivity concentration. The combined standard uncertainty represents the standard deviation associated with the measurement result. If it is necessary to express an interval that is expected to contain the majority of the distribution of values attributable to the measured value, coverage factor k is multiplied by the combined standard uncertainty to obtain expanded uncertainty. k is determined

from the level of confidence and *t*-distribution[*368] and is generally chosen between 2 to 3. In the measurement of the radioactivity concentration, the distribution of measurement results can be regarded as a normal distribution; therefore, $k = 2$ when the level of confidence is approximately 95%. When reporting the uncertainty together with the measurement result, it is necessary to clearly indicate the type of uncertainty (combined standard uncertainty or expanded uncertainty $k = 2$ to 3)) listed alongside the measurement value.

In the calculation example in Table C.1, the overall relative combined standard uncertainty is 20% (two significant figures), but the breakdown shows that the contribution of the uncertainty due to counting statistics (u_{11}) accounts for the majority of the contribution, and the contribution of other items is almost negligible.

This indicates that if the analysis and measurement conditions are the same as the calculation example, and the ^{90}Sr radioactivity concentration is equal to or less than the same level, it is acceptable as a matter of practicality to adopt only the uncertainty due to counting statistics (u_{11}) as the overall relative combined standard uncertainty. The relative combined standard uncertainty is obtained by calculating the square root of the sum of the squares of the relative standard uncertainties of each factor. At that time, the influence of uncertainty factors that contribute 1/10 or less compared to the factor with the largest contribution on the final calculation result is minimal and as a matter of practicality can be disregarded. For example, when combining a relative standard uncertainty of 10% and a relative standard uncertainty of 1%, $\sqrt{10^2 + 1^2}$=10.05 % is obtained. Since the number of significant figures for uncertainty is usually one to two figures, the result is hardly affected.

What is important in the evaluation of uncertainty is to reliably evaluate the uncertainty factors with large contributions. It is not realistic to try to evaluate all uncertainty factors, including those with small contributions. In the case of radiation measurement, the uncertainty factors with large contributions are the uncertainty due to counting statistics and the uncertainty of the assigned value of the standard source, etc. In most cases, as a matter of practicality evaluating just these factors is sufficient.

Note that it is not necessary to evaluate all uncertainty factors in Table C.1 for every measurement. Items that vary with each measurement, such as the uncertainty due to counting statistics, must be evaluated every time, but other items may be evaluated when a device is replaced, maintenance or inspection are performed, or a reagent lot is changed, etc. In this case, the relative combined standard uncertainty of the measurement is obtained by combining the uncertainty due to counting statistics (which varies with each measurement) and the other uncertainties (which do not vary with each measurement).

[*368] Annex G of the following document provides more information (Table G.2 of Annex G gives the t-distribution table). Hidetaka Imai et al.: Guide to the Expression of Uncertainty in Measurement [GUM] Handbook, published by the Japanese Standards Association: ISBN:9784542307056 (2018)

Table C.1 Uncertainty budget (example)

Uncertainty factors *1	Relative standard uncertainty	Calculation example *2	Notes
C.3.1 Sample quantity (Ws)	$\frac{u(W_s)}{W_s}$	**0.045%**	Relative standard uncertainty of (1)
(1) Weighing	u_1	0.045%	
C.3.2 Recovery (Y_{Sr})	$\frac{u(Y_{Sr})}{Y_{Sr}}$	**1.5%**	Combine the relative combined standard uncertainty of (2) through (4) and the relative standard uncertainty of (6).
(2) Sr amount added as a carrier	u_2	1.2%	Combined standard uncertainty of u_2 through u_4 = 0.60 mg Relative combined standard uncertainty of (2) through (4) = 0.60 mg/62.06 mg = **0.97%**
(3) Stable Sr concentration in the sample	u_3	0.71%	
(4) Sample quantity	u_4	0.045%	
(6) Sr amount obtained by recovery measurement	u_5	1.1%	
C.3.3 ^{90}Y counting efficiency (ε)	$\frac{u(\varepsilon)}{\varepsilon}$	**1.4%**	Combine the relative standard uncertainties of (1) through (5).
(1) Standard source	u_6	0.65%	
(2) Preparation of calibration sources	u_7	0.19%	
(3) Counting statistics	u_8	0.54%	Can be omitted when evaluating the uncertainty of (4) calibration formula fitting.
(4) Calibration formula fitting	u_9	1.1%	
(5) Decay correction	u_{10}	0.0046%	It is negligibly small and does not necessarily need to be assessed.
C.3.4 ^{90}Y net counting rate (r_n)	$\frac{u(r_n)}{r_n}$	**20%**	Combine the relative standard uncertainties of (1) through (3).
(1) Counting statistics	u_{11}	20%	Evaluate at each measurement
(2) Variations in the measurement system	u_{12}	0.35%	
(3) Decay correction	u_{13}	0.0030%	It is negligibly small and does not necessarily need to be assessed.
Relative combined standard uncertainty*3	$\frac{u(A)}{A}$	**20%**	Evaluate at each measurement

*1 The numbers preceding each uncertainty factor correspond to those referred to in section C.3.

*2 In the calculation example in the table, for convenience intermediate results are shown as rounded values (rounded in the table, but not in the actual calculation).

*3 Relative combined standard uncertainty values must have at most two significant figures.

[How to examine the factors to be considered]

- This explanation intentionally contains factors with small contributions as well for the mastery of evaluation methods for various factors. However, in actual evaluation of uncertainties, it is better to give top priority to the uncertainty due to counting statistics as the relative standard uncertainty with a large contribution among all factors "C. 3.4 (1)."
- In order to reliably evaluate uncertainty factors with large contributions, it is advisable to evaluate additional factors that are likely to contain a large uncertainty while using the uncertainty of each factor in this table as a reference, to combine them with the uncertainty due to counting statistics, and then determine whether the uncertainty factor should be considered.
- Items other than the uncertainty due to counting statistics can be evaluated when a device is replaced, maintenance or inspection are performed, or a reagent lot is changed, etc.

C.3 Example of uncertainty calculation procedures

This section describes as a reference how to calculate each factor shown in Table C.1 using the case in C.2.

Intermediate results obtained in the process of calculation of each uncertainty are not rounded. Rather, only the final result obtained when determining the relative combined standard uncertainty is rounded [*369].

Value A of the radioactivity concentration of ^{90}Sr is calculated using the following model equation according to equation (C.5).

$$A = \frac{r_{\mathrm{n}} \times 100}{W_{\mathrm{s}} \times Y_{\mathrm{Sr}} \times \varepsilon}$$

A : ^{90}Sr radioactivity concentration in the sample (Bq/kg ash)
W_{s} : Sample quantity (kg ash)
Y_{Sr} : Recovery rate (%)
ε : Counting efficiency of ^{90}Y (cps/Bq)
r_{n} : ^{90}Y net counting rate (cps)

The relative combined standard uncertainty of value A of the radioactivity concentration of ^{90}Sr is given by combining the relative standard uncertainty in each parameter (sample quantity W_{s}, recovery Y_{Sr}, ^{90}Y counting efficiency ε, ^{90}Y net counting rate r_{n}) in the model equation.

Here, the combined standard uncertainty of A is defined as $u(A)$, while the relative combined standard uncertainty is defined as $\frac{u(A)}{A}$. Other parameters are also similarly defined.

When the relative standard uncertainties $(\frac{u(x_i)}{x_i})$ in the input quantities x_1, x_2, x_3, ⋯, x_n in equation (C.4) shown above are substituted into the parameters in the model equation, the equation is expressed as follows.

$$\frac{u(A)}{A} = \sqrt{\left(\frac{u(W_{\mathrm{s}})}{W_{\mathrm{s}}}\right)^2 + \left(\frac{u(Y_{\mathrm{Sr}})}{Y_{\mathrm{Sr}}}\right)^2 + \left(\frac{u(\varepsilon)}{\varepsilon}\right)^2 + \left(\frac{u(r_{\mathrm{n}})}{r_{\mathrm{n}}}\right)^2} \qquad \text{(C. 6)}$$

To calculate $\frac{u(A)}{A}$, the relative standard uncertainties are calculated in C.3.1 through C.3.4 respectively in the order of sample quantity W_{s}, recovery Y_{Sr}, ^{90}Y counting efficiency ε, and ^{90}Y net counting rate r_{n}. Then they are combined using equation (C.6) in C.3.5.

[*369] In the calculation example in this explanation, for convenience intermediate results are shown as rounded values (rounded in the table, but not in the actual calculation).

C.3.1 Uncertainty in sample quantity W_s $(\frac{u(W_s)}{W_s})$

When ash sample is used, the uncertainty in sample quantity W_s is considered to be caused by the uncertainty in weighing. The relative standard uncertainty in weighing is defined as u_1.

(1) Uncertainty in weighing (u_1)

The method for calculating the uncertainty in weighing differs depending on whether the uncertainty is stated on the calibration certificate for the electronic balance used.

The uneven distribution of radionuclides in a sample has an impact on the measured value, but it is difficult to estimate the uncertainty. Therefore, although the homogeneity of a sample does not need to be evaluated, the sample should be thoroughly mixed and made uniform before weighing.

a) When uncertainty is stated on the calibration certificate

The uncertainty stated on the calibration certificate of the electronic balance is used. If the stated uncertainty is not relative, the relative standard uncertainty in weighing (u_1) is calculated using the following equation.

$$u_1 = \frac{u(W)}{W} \times 100\ (\%) \qquad (C.7)$$

$u(W)$: Uncertainty in weighing value (g)
W: Weighing value (g)

b) When uncertainty is not stated on the calibration certificate

If the calibration certificate of the electronic balance does not state the uncertainty, the relative standard uncertainty in weighing (u_1) is determined by combining the relative standard uncertainties of the uncertainty associated with rounding of the weighing value ($u_{1.1}$) and the uncertainty associated with weighing repeatability ($u_{1.2}$). Only the uncertainty in weighing is obtained by combining them, but the measurement uncertainty at calibration is negligibly small and does not need to be taken into account. However, if there are other significant uncertainty factors, they should also be factored in.

1) Uncertainty associated with rounding of the weighing value ($u_{1.1}$)

When the reading limit (minimum scale) of the electronic balance is l, the readings distribute rectangularly with upper and lower limits of $\pm\, l/2$ at the zero point and the weighing value, respectively. When these two distributions are combined, a triangular distribution with upper and lower limits of $\pm\, l$ is obtained, and $u_{1.1}$ is calculated using the following equation.

$$u_{1.1} = \frac{l}{\sqrt{6} \times W} \times 100\ (\%) \qquad (C.8)$$

l : Reading limit (g)
W : Weighing value (g)

2) Uncertainty associated with weighing repeatability ($u_{1.2}$)

The measurement is repeated about 10 times using counter weights of a mass similar to the sample quantity, and the mean value and standard deviation of the measurements are calculated. When the mean value of the measurements of n times is used as the sample quantity, $u_{1.2}$ is calculated using the following equation.

$$u_{1.2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100\ (\%) \qquad \text{(C. 9)}$$

$\bar{W}$: Mean value of repeated measurements (g)
W_{STD} : Standard deviation of repeated measurements (g)
n : Number of measurements of the sample (when the mean value is used as the sample quantity)

3) Combining uncertainties in weighing

The uncertainties $u_{1.1}$ and $u_{1.2}$ are combined to obtain u_1.

$$u_1 = \sqrt{(u_{1.1})^2 + (u_{1.2})^2} \qquad \text{(C. 10)}$$

[Calculation example]

Calculate $u_{1.1}$ using equation (C.8) when an electronic balance with a reading limit of 0.01 g is used and the weighing value is 20.10 g.

$$u_{1.1} = \frac{l}{\sqrt{6} \times W} \times 100 = \frac{0.01}{\sqrt{6} \times 20.10} \times 100 = 0.020\ \%$$

Calculate $u_{1.2}$ using equation (C.9) when the mean value of repeated measurements is 20.100 g, the standard deviation is 0.008 g, and the number of sample measurements is 1.

$$u_{1.2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.008}{20.100} \times \frac{1}{\sqrt{1}} \times 100 = 0.040\ \%$$

Calculate u_1 using equation (C.10).

$$u_1 = \frac{u(W)}{W} = \sqrt{(u_{1.1})^2 + (u_{1.2})^2} = \sqrt{0.020^2 + 0.040^2} = 0.045\%$$

C.3.2 Uncertainty in recovery Y_{Sr} ($\frac{u(Y_{\mathrm{Sr}})}{Y_{\mathrm{Sr}}}$)

The recovery rate Y_{Sr} is calculated using the following equation.

$$Y_{\mathrm{Sr}} = \frac{W_2}{W_1 + C_{\mathrm{st}} \times W_{\mathrm{s}}} \times 100\ (\%) \qquad \text{(C. 11)}$$

W_1 : Sr amount added as a carrier (mg)
C_{st} : Stable Sr concentration in the sample (mg/g)
W_{s} : Sample quantity (g)
W_2 : Stable Sr amount obtained by recovery measurement (mg)

Here, the relative standard uncertainties in W_1, C_{st}, W_{s}, and W_2 are defined as follows.

$\frac{u(W_1)}{W_1} (= u_2)$: Relative standard uncertainty in W_1

$\frac{u(C_{\mathrm{st}})}{C_{\mathrm{st}}} (= u_3)$: Relative standard uncertainty in C_{st}

$\frac{u(W_{\mathrm{s}})}{W_{\mathrm{s}}} (= u_4)$: Relative standard uncertainty in W_{s}

$\frac{u(W_2)}{W_2} (= u_5)$: Relative standard uncertainty in W_2

If $W_1 + C_{\mathrm{st}} \times W_{\mathrm{s}} = Z$, equation (C.11) is expressed as follows.

$$Y_{Sr} = \frac{W_2}{W_1 + C_{st} \times W_s} = \frac{W_2}{Z}$$

Therefore, $\frac{u(Y_{Sr})}{Y_{Sr}}$ is calculated using the following equation, which combines $\frac{u(Z)}{Z}$ and $\frac{u(W_2)}{W_2}$ (= u_5).

$$\frac{u(Y_{Sr})}{Y_{Sr}} = \sqrt{\left(\frac{u(Z)}{Z}\right)^2 + \left(\frac{u(W_2)}{W_2}\right)^2} \qquad \text{(C. 12)}$$

Here, to obtain $\frac{u(Y_{Sr})}{Y_{Sr}}$, $\frac{u(Z)}{Z}$ is calculated in (1) through (5), $\frac{u(W_2)}{W_2}$ (= u_5) is calculated in (6), and then $\frac{u(Z)}{Z}$ and $\frac{u(W_2)}{W_2}$ are combined in (7).

(1) Uncertainty in Z ($\frac{u(Z)}{Z}$)

The equation Z (= $W_1 + C_{st} \times W_s$) has a multiplication part ($C_{st} \times W_s$) and a sum part ($W_1 + C_{st} \times W_s$). The combined standard uncertainty of the multiplication part is given by the square root of the sum of squares of the relative standard uncertainties in C_{st} and W_s according to equation (C.4), and the combined standard uncertainty of the sum part is given by the square root of the sum of squares of the standard uncertainties in W_1 and $C_{st} \times W_s$ according to equation (C.2).

First, the combined standard uncertainty in the multiplication part ($C_{st} \times W_s$) is calculated as follows.

$$(C_{st} \pm u(C_{st})) \times (W_s \pm u(W_s))$$

$$= (C_{st} \times W_s) \pm (C_{st} \times W_s) \times \sqrt{\left(\frac{u(C_{st})}{C_{st}}\right)^2 + \left(\frac{u(W_s)}{W_s}\right)^2}$$

$$= (C_{st} \times W_s) \pm \left(\sqrt{\left({C_{st}}^2 \times {W_s}^2 \times \left(\frac{u(C_{st})}{C_{st}}\right)^2\right) + \left({C_{st}}^2 \times {W_s}^2 \times \left(\frac{u(W_s)}{W_s}\right)^2\right)}\right)$$

$$= (C_{st} \times W_s) \pm \left(\sqrt{{W_s}^2 \times (u(C_{st}))^2 + {C_{st}}^2 \times (u(W_s))^2}\right)$$

Next, the combined standard uncertainties in W_1 and $C_{st} \times W_s$ are combined.

$$(W_1 \pm (u(W_1)) + \left((C_{st} \times W_s) \pm \left(\sqrt{{W_s}^2 \times (u(C_{st}))^2 + {C_{st}}^2 \times (u(W_s))^2}\right)\right)$$

$$= \left(W_1 + (C_{st} \times W_s)\right) \pm \left(\sqrt{(u(W_1))^2 + \left(\sqrt{{W_s}^2 \times (u(C_{st}))^2 + {C_{st}}^2 \times (u(W_s))^2}\right)^2}\right)$$

$$= \left(W_1 + (C_{st} \times W_s)\right) \pm \sqrt{(u(W_1))^2 + {W_s}^2 \times (u(C_{st}))^2 + {C_{st}}^2 \times (u(W_s))^2}$$

Therefore, $u(Z)$ is calculated using the following equation.

$$u(Z) = \sqrt{(u(W_1))^2 + {W_s}^2 \times (u(C_{st}))^2 + {C_{st}}^2 \times (u(W_s))^2} \qquad \text{(C. 13)}$$

Next, Z is calculated, and $u(Z)$ is divided by Z to obtain $\frac{u(Z)}{Z}$.

In the following calculation example, uncertainty in the case where analysis is conducted under the following conditions is calculated.

W_1 = 50.00 mg
C_{st} = 0.60 mg/g ash
W_s = 20.10 g

When Z is calculated from the above values, it is as follows.

$$Z = W_1 + C_{st} \times W_s = 50.00 \text{ mg} + 0.60 \text{ mg/g ash} \times 20.10 \text{ g} = 62.06 \text{ mg}$$

(2) Uncertainty in Sr amount added as a carrier (u_2)

The Sr carrier solution is prepared by dissolving strontium nitrate in pure water. Therefore, u_2 is given by combining the relative standard uncertainties of the uncertainty in the purity of strontium nitrate ($u_{2.1}$), the uncertainty in the weighing of strontium nitrate ($u_{2.2}$), the uncertainty in diluting the Sr carrier solution ($u_{2.3}$), and the uncertainty in adding Sr carrier solution ($u_{2.4}$).

1) Uncertainty in the purity of strontium nitrate ($u_{2.1}$)

If the purity of strontium nitrate is assumed to have a concentration range with the center value R% as indicated on the reagent bottle, and the maximum value is 1 (100%) and the distance between the center value and the minimum value is the same as that between the maximum value and the center value, the following can be applied.

Maximum value: 1

Center value: R

Minimum value: R - (1 - R) = 2R - 1

Purity tolerance: $\frac{1-(2R-1)}{2}$

When it is assumed that the uncertainty within the above range distributes rectangularly, $u_{2.1}$ is calculated using the following equation.

$$u_{2.1} = \frac{1-(2R-1)}{2\sqrt{3} \times R} \times 100 \ (\%) \qquad \text{(C. 14)}$$

[Calculation example]

Calculate $u_{2.1}$ using equation (C.14) when the purity of strontium nitrate is 98% (= 0.98).

$$u_{2.1} = \frac{1-(2R-1)}{2\sqrt{3} \times R} \times 100 = \frac{1-(2 \times 0.98-1)}{2\sqrt{3} \times 0.98} \times 100 = \ 1.2 \ \%$$

2) Uncertainty in the weighing of strontium nitrate ($u_{2.2}$)

As is the case with u_1 described above, the method for calculating the uncertainty in weighing differs depending on whether the uncertainty is stated in the calibration certificate for the electronic balance. If it is not stated, $u_{2.2}$ can be determined by combining the relative standard uncertainties of the uncertainty associated with rounding of the weighing value of the balance ($u_{2.2.1}$) and the uncertainty associated with repeatability ($u_{2.2.2}$).

[Calculation example]

Calculate $u_{2.2.1}$ using equation (C.8) when an electronic balance with a reading limit of 0.01 g is used and the weighing value is 24.15 g.

$$u_{2.2.1} = \frac{l}{\sqrt{6} \times W} \times 100 = \frac{0.01}{\sqrt{6} \times 24.15} \times 100 = \ 0.017\%$$

Calculate $u_{2.2.2}$ using equation (C.9) when the mean value of repeated measurements is 24.150 g, the standard deviation is 0.007 g, and the number of sample measurements is 1.

$$u_{2.2.2} = \frac{W_{STD}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.007}{24.150} \times \frac{1}{\sqrt{1}} \times 100 = 0.029 \ \%$$

Combine $u_{2.2.1}$ and $u_{2.2.2}$ using equation (C.10) to obtain $u_{2.2.}$

$$u_{2.2} = \sqrt{(u_{2.2.1})^2 + (u_{2.2.2})^2} = \sqrt{0.017^2 + 0.029^2} = 0.034\%$$

3) Uncertainty in diluting Sr carrier solution ($u_{2.3}$)

A volumetric flask is used for diluting Sr carrier solution. Therefore, $u_{2.3}$ can be determined by combining the relative standard uncertainties of the uncertainty in the volumetric flask volume ($u_{2.3.1}$) and the uncertainty in the volumetric flask repeatability ($u_{2.3.2}$).

3-1) Uncertainty in the volumetric flask volume ($u_{2.3.1}$)

The tolerance range of the volumetric flask volume distributes rectangularly, and therefore, $u_{2.3.1}$ is calculated using the following equation.

$$u_{2.3.1} = \frac{V_i}{\sqrt{3} \times V} \times 100\ (\%) \qquad \text{(C. 15)}$$

V : Volumetric flask volume (mL)
V_i : Tolerance of volumetric flask

3-2) Uncertainty in the volumetric flask repeatability ($u_{2.3.2}$)

The mass of the pure water determined using a volumetric flask is measured repeatedly about ten times with an electronic balance to calculate the mean value and standard deviation. When the mean value of the measurements of n times is adopted, $u_{2.3.2}$ is calculated using the following equation.

$$u_{2.3.2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100\ (\%) \qquad \text{(C. 16)}$$

$\bar{W}$: Mean value of repeated weighing (g)
W_{STD} : Standard deviation in repeated weighing (g)
n : Number of repetitions

3-3) Combining uncertainties in diluting Sr carrier solution

Uncertainties of $u_{2.3.1}$ and $u_{2.3.2}$ are combined to obtain $u_{2.3}$.

$$u_{2.3} = \sqrt{(u_{2.3.1})^2 + (u_{2.3.2})^2} \qquad \text{(C. 17)}$$

[Calculation example]

Calculate $u_{2.3.1}$ using equation (C.15) when a 1 L (= 1000 mL) volumetric flask with a tolerance of 0.4 mL is used.

$$u_{2.3.1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.4}{\sqrt{3} \times 1000} \times 100 = 0.023\ \%$$

Calculate $u_{2.3.2}$ using equation (C.16) when the mean value of repeated measurements is 1000.080 g, the standard deviation is 0.170 g, and the number of sample measurements is 1.

$$u_{2.3.2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.170}{1000.080} \times \frac{1}{\sqrt{1}} \times 100 = 0.017\ \%$$

Combine $u_{2.3.1}$ and $u_{2.3.2}$ using equation (C.17) to obtain $u_{2.3.}$

$$u_{2.3} = \sqrt{(u_{2.3.1})^2 + (u_{2.3.2})^2} = \sqrt{0.023^2 + 0.017^2} = 0.029\ \%$$

4) Uncertainty in adding Sr carrier solution ($u_{2.4}$)

A volumetric pipette is used to add Sr carrier solution. Therefore, $u_{2.4}$ can be determined by combining the relative standard uncertainties of the uncertainty in the volumetric pipette volume

($u_{2.4.1}$) and the uncertainty in the volumetric pipette repeatability ($u_{2.4.2}$).

4-1) Uncertainty in the volumetric pipette volume ($u_{2.4.1}$)

The tolerance range of the volumetric pipette volume distributes rectangularly, and therefore, $u_{2.4.1}$ is calculated using the following equation.

$$u_{2.4.1} = \frac{V_i}{\sqrt{3} \times V} \times 100\ (\%) \qquad \text{(C. 18)}$$

V : Volumetric pipette volume (mL)

V_i : Volumetric pipette tolerance

4-2) Uncertainty in the volumetric pipette repeatability ($u_{2.4.2}$)

The mass of the pure water determined using a volumetric pipette is measured repeatedly about 10 times with an electronic balance to calculate the mean value and standard deviation. When the mean value of the measurements of n times is adopted, $u_{2.4.2}$ is calculated using the following equation.

$$u_{2.4.2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100\ (\%) \qquad \text{(C. 19)}$$

$\bar{W}$: Mean value of repeated weighing (g)

W_{STD} : Standard deviation in repeated weighing (g)

n : Number of repetitions

4-3) Combining uncertainties in adding Sr carrier solution ($u_{2.4}$)

$u_{2.4.1}$ and $u_{2.4.2}$ are combined to obtain $u_{2.4}$.

$$u_{2.4} = \sqrt{(u_{2.4.1})^2 + (u_{2.4.2})^2} \qquad \text{(C. 20)}$$

[Calculation example]

Calculate $u_{2.4.1}$ using equation (C.18) when a 5 mL volumetric pipette with a tolerance of 0.015 mL is used.

$$u_{2.4.1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.015}{\sqrt{3} \times 5} \times 100 = 0.17\ \%$$

Calculate $u_{2.4.2}$ using equation (C.19) when the mean value of repeated measurements is 5.003 g, the standard deviation is 0.004 g, and the number of sample measurements is 1.

$$u_{2.4.2} = \frac{V_{\mathrm{STD}}}{\bar{V}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.004}{5.003} \times \frac{1}{\sqrt{1}} \times 100 = 0.080\ \%$$

Combine $u_{2.4.1}$ and $u_{2.4.2}$ using equation (C.20) to obtain $u_{2.4.}$

$$u_{2.4} = \sqrt{(u_{2.4.1})^2 + (u_{2.4.2})^2} = \sqrt{0.17^2 + 0.080^2} = 0.19\ \%$$

5) Combining uncertainties in the amount of Sr added as a carrier (u_2)

Uncertainties of $u_{2.1}$ through $u_{2.4}$ are combined to obtain u_2.

$$u_2 = \frac{u(W_1)}{W_1} = \sqrt{(u_{2.1})^2 + (u_{2.2})^2 + (u_{2.3})^2 + (u_{2.4})^2} \qquad \text{(C. 21)}$$

[Calculation example]

Combine $u_{2.1}$ through $u_{2.4}$ using equation (C.21) to obtain u_2.

$$u_2 = \frac{u(W_1)}{W_1} = \sqrt{(u_{2.1})^2 + (u_{2.2})^2 + (u_{2.3})^2 + (u_{2.4})^2}$$
$$= \sqrt{1.2^2 + 0.034^2 + 0.029^2 + 0.19^2} = 1.2\%$$

(3) Uncertainty in the stable Sr concentration in the sample (u_3)

The following describes how to calculate uncertainty in the case where the stable Sr concentration (C_{st}) in the sample is quantified using ICP-AES. The uncertainty of u_3 is given by combining the relative standard uncertainties of the uncertainty in weighing the stable Sr measurement sample ($u_{3.1}$), the uncertainty in diluting the stable Sr measurement sample solution ($u_{3.2}$), the uncertainty in the calibration curve ($u_{3.3}$), and the uncertainty in the calibration curve solution ($u_{3.4}$).

1) Uncertainty in weighing the stable Sr measurement sample ($u_{3.1}$)

As is the case with u_1 described above, the method for calculating the uncertainty in weighing differs depending on whether the uncertainty is stated in the calibration certificate for the electronic balance. If it is not stated, $u_{3.1}$ can be determined by combining the relative standard uncertainties of the uncertainty associated with rounding of the weighing value of the balance ($u_{3.1.1}$) and the uncertainty associated with weighing repeatability ($u_{3.1.2}$).

[Calculation example]

Calculate $u_{3.1.1}$ using equation (C.8) when an electronic balance with a reading limit of 0.00001 g is used and the weighing value is 1.00066 g.

$$u_{3.1.1} = \frac{l}{\sqrt{6} \times W} \times 100 = \frac{0.00001}{\sqrt{6} \times 1.00066} \times 100 = 0.00041\ \%$$

Calculate $u_{3.1.2}$ using equation (C.9) when the mean value of repeated measurements is 1.000660 g, the standard deviation is 0.000087 g, and the number of sample measurements is 1.

$$u_{3.1.2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.000087}{1.000660} \times 100 \times \frac{1}{\sqrt{1}} = 0.0087\ \%$$

Combine $u_{3.1.1}$ and $u_{3.1.2}$ using equation (C.10) to obtain $u_{3.1.}$

$$u_{3.1} = \sqrt{(u_{3.1.1})^2 + (u_{3.1.2})^2} = \sqrt{0.00041^2 + 0.0087^2} = 0.0087\ \%$$

2) Uncertainty in diluting the stable Sr measurement sample solution ($u_{3.2}$)

Volumetric flasks and a volumetric pipette are used to dilute the stable Sr measurement sample solution. Therefore, as is the case with $u_{2.3}$ and $u_{2.4}$ described above, $u_{3.2}$ is given by combining the relative standard uncertainties of the uncertainty in the volumetric flask volume ($u_{3.2.a1}$, $u_{3.2.b1}$) and the uncertainty in the volumetric flask repeatability ($u_{3.2.a2}$, $u_{3.2.b2}$), the uncertainty in the volumetric pipette volume ($u_{3.2.c1}$), and the uncertainty in the volumetric pipette repeatability ($u_{3.2.c2}$).

[Calculation example]

This example shows how to calculate $u_{3.2}$ when 5 mL of sample solution is taken out of the sample solution in a 100 mL volumetric flask with a volumetric pipette and transferred to a 50 mL volumetric flask for dilution. This case uses two types of volumetric flask, 100 mL and 50 mL, and a 5 mL volumetric pipette. Therefore, $u_{3.2}$ can be given by combining the relative standard uncertainties of the uncertainty in the 100 mL volumetric flask ($u_{3.2.a}$) and the uncertainty in the 50 mL volumetric flask ($u_{3.2.b}$) and the uncertainty in the 5 mL volumetric pipette ($u_{3.2.c}$).

a) Uncertainty in the volumetric flasks ($u_{3.2.a}$, $u_{3.2.b}$)

First, calculate $u_{3.2.a}$. Calculate $u_{3.2.a1}$ using equation (C.15) when a 100 mL volumetric flask with a tolerance of 0.1 mL is used.

$$u_{3.2.a1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.1}{\sqrt{3} \times 100} \times 100 = 0.058\ \%$$

Calculate $u_{3.2.a2}$ using equation (C.16) when the mean value of repeated measurements is 100.002 g, the standard deviation is 0.059 g, and the number of sample measurements is 1.

$$u_{3.2.a2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.059}{100.002} \times \frac{1}{\sqrt{1}} \times 100 = 0.059\ \%$$

Combine $u_{3.2.a1}$ and $u_{3.2.a2}$ using equation (C.17) to obtain $u_{3.2.a}$.

$$u_{3.2.a} = \sqrt{(u_{3.2.a1})^2 + (u_{3.3.a2})^2} = \sqrt{0.058^2 + 0.059^2} = 0.083\ \%$$

Next, calculate $u_{3.2.b}$. Calculate $u_{3.2.b1}$ using equation (C.15) when a 50 mL volumetric flask with a tolerance of 0.06 mL is used.

$$u_{3.2.b1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.06}{\sqrt{3} \times 50} \times 100 = 0.069\ \%$$

Calculate $u_{3.2.b2}$ using equation (C.16) when the mean value of repeated measurements is 49.995 g, the standard deviation is 0.034 g, and the number of sample measurements is 1.

$$u_{3.2.b2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.034}{49.995} \times \frac{1}{\sqrt{1}} \times 100 = 0.068\ \%$$

Combine $u_{3.2.b1}$ and $u_{3.2.b2}$ using equation (C.17) to obtain $u_{3.2.b}$.

$$u_{3.2.b} = \sqrt{(u_{3.2.b1})^2 + (u_{3.2.b2})^2} = \sqrt{0.069^2 + 0.068^2} = 0.097\ \%$$

b) Uncertainty in the volumetric pipette ($u_{3.2.c}$)

Calculate $u_{3.2.c1}$ using equation (C.18) when a 5 mL volumetric pipette with a tolerance of 0.015 mL is used.

$$u_{3.2.c1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.015}{\sqrt{3} \times 5} \times 100 = 0.17\ \%$$

Calculate $u_{3.2.c2}$ using equation (C.19) when the mean value of repeated measurements is 5.003 g, the standard deviation is 0.004 g, and the number of sample measurements is 1.

$$u_{3.2.c2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.004}{5.003} \times \frac{1}{\sqrt{1}} \times 100 = 0.080\ \%$$

Combine $u_{3.2.c1}$ and $u_{3.2.c2}$ using equation (C.20) to obtain $u_{3.2.c}$.

$$u_{3.2.c} = \sqrt{(u_{3.2.c1})^2 + (u_{3.2.c2})^2} = \sqrt{0.17^2 + 0.080^2} = 0.19\ \%$$

c) Combining uncertainties in dilution ($u_{3.2}$)

Combine $u_{3.2.a}$, $u_{3.2.b}$, $u_{3.2.c}$ to obtain $u_{3.2}$.

$$u_{3.2} = \sqrt{(u_{3.2.a})^2 + (u_{3.2.b})^2 + (u_{3.2.c})^2} = \sqrt{0.083^2 + 0.097^2 + 0.19^2} = 0.23\ \%$$

3) Uncertainty in the calibration curve ($u_{3.3}$)

The following is the calculation of uncertainty in the case where a calibration curve is plotted by the least-squares method from the measurement results of standard solutions of n concentrations and a linear equation of $y = ax + b$ is obtained. Strictly speaking, uncertainty should be evaluated at each measurement. However, the impact of the difference in the uncertainty of calibration curves on the final uncertainty is negligibly small, so evaluation for each measurement is not necessary unless device adjustment is performed or a condition is changed.

If the standard uncertainty in the calibration curve is defined as $u(C_f)$, the value of u (C_f) to be used is given as follows: calculate the difference between estimated intensity y_{est}, which is obtained by substituting concentration x to $y = ax + b$, and actual intensity y_i, which is obtained by measuring the standard solutions; and divide the largest value of the obtained differences by gradient a[*370].

$$u(C_f) = \frac{|\mathrm{MAX}(y_i - y_{est})|}{a} \qquad \text{(C. 22)}$$

Y_i : Actual intensity obtained by measuring the standard solutions

y_{est} : Estimate of intensity y obtained by substituting the concentration x into $y = ax + b$

Therefore, $u_{3.3}$ is calculated using the following equation.

$$u_{3.3} = \frac{u(C_f)}{C_f} \times 100\ (\%) \qquad \text{(C. 23)}$$

C_f: Concentration of the measurement sample

[Calculation example]

Assume that five standard solutions with concentrations ranging from 0 to 0.5 mg/L are used to obtain the results shown in the following table, and that the linear equation is $y = 6.4317x + 0.0128$.

Table C.2 Calibration curve measurement results

	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5
x: Concentration	0	0.01	0.05	0.1	0.5
y_i: Intensity	0.0163	0.0778	0.337	0.648	3.23
y_{est}	0.01280	0.07712	0.3344	0.6560	3.229
$y_i - y_{est}$	0.00350	0.00068	0.0026	-0.0080	0.001

[*370] This evaluation method is simple but overestimates the uncertainty. For more appropriate uncertainty assessment, refer to the following document that contains other methods.
National Institute of Technology and Evaluation International Accreditation Japan: JCSS 校正における測定不確かさの評価内挿校正式による不確かさの見積もり, JCG200S21-02 (2022).

Calculate $u(C_f)$ using equation (C.22). For $y_i - y_{est}$, use the value of Standard 4 in which the difference between the calibration curve and the actual measurement point is the largest.

$$u(C_f) = \frac{|\mathrm{MAX}(y_i - y_{est})|}{a} = \frac{|-0.0080|}{6.4317} = 0.00124$$

Calculate $u_{3.3}$ using equation (C.23) when the concentration of measured sample is 0.25 mg/L.

$$u_{3.3} = \frac{u(C_f)}{C_f} \times 100 = \frac{0.00124}{0.25} \times 100 = 0.50\ \%$$

4) Uncertainty in the calibration curve solution ($u_{3.4}$)

The calibration curve solution is prepared by diluting the Sr standard solution. Therefore, $u_{3.4}$ can be determined by combining the relative standard uncertainties of the uncertainty in the Sr standard solution ($u_{3.4.1}$) and the uncertainty in diluting the calibration curve solution ($u_{3.4.2}$).

4-1) Uncertainty in Sr standard solution ($u_{3.4.1}$)

Use the expanded uncertainty specified on the certificate of analysis for the Sr standard solution. When the expanded uncertainty specified on the certificate of analysis of the Sr standard solution is U% and the inclusion factor is k, $u_{3.4.1}$ is calculated using the following equation.

$$u_{3.4.1} = \frac{U}{k}\ (\%) \qquad \text{(C. 24)}$$

4-2) Uncertainty in diluting the calibration curve solution ($u_{3.4.2}$)

A volumetric flask and a volumetric pipette are used to prepare a calibration curve solution from the Sr standard solution. If the dilution method differs depending on the concentration, the uncertainty of the concentration at which the uncertainty is the greatest is used. Therefore, as is the case with $u_{2.3}$ and $u_{2.4}$ in the above, $u_{3.4.2}$ is determined by combining the relative standard uncertainties of the uncertainty in the volumetric flask volume ($u_{3.4.2.a1}$, $u_{3.4.2.b1}$, $u_{3.4.2.c1}$) and the uncertainty in the volumetric flask repeatability ($u_{3.4.2.a2}$, $u_{3.4.2.b2}$, $u_{3.4.2.c2}$), the uncertainty in the volumetric pipette volume ($u_{3.4.2.d1}$, $u_{3.4.2.e1}$, $u_{3.4.2.f1}$), and the uncertainty in the volumetric pipette repeatability ($u_{3.4.2.d2}$, $u_{3.4.2.e2}$, $u_{3.4.2.f2}$).

4-3) Combining uncertainties in the calibration curve solution ($u_{3.4}$)

$u_{3.4.1}$ and $u_{3.4.2}$ are combined to obtain $u_{3.4}$.

$$u_{3.4} = \sqrt{(u_{3.4.1})^2 + (u_{3.4.2})^2} \qquad \text{(C. 25)}$$

[Calculation example]

Calculate $u_{3.4.1}$ using equation (C.24) when an expanded uncertainty U of 0.3% and an inclusion coefficient k of 2 are given on the certificate of analysis for the Sr standard solution.

$$u_{3.4.1} = \frac{U}{k} = \frac{0.3}{2} = 0.15\ \%$$

This example shows how to calculate $u_{3.4.2}$ when 1000 mg/L Sr standard solution is diluted as shown in Table C.3 to prepare 0.01 mg/L Sr standard solution.

Table C.3 Calibration curve solution dilution method

	Concentration before dilution	volumetric pipette to be used	Volumetric flask to be used	Concentration after dilution
1	1000 mg/L	5 mL	500 mL	10 mg/L
2	10 mg/L	10 mL	100 mL	1 mg/L
3	1 mg/L	2 mL	200 mL	0.01 mg/L

This case uses three types of volumetric flask, 500 mL, 100 mL and 200 mL, and three types of volumetric pipettes, 5 mL, 10 mL, and 2 mL. Therefore, $u_{3.4.2}$ is determined by combining the relative standard uncertainties of the uncertainty in the 500 mL volumetric flask ($u_{3.4.2.a}$), the uncertainty in the 100 mL volumetric flask ($u_{3.4.2.b}$), the uncertainty in the 200 mL volumetric flask ($u_{3.4.2.c}$), the uncertainty in the 5 mL volumetric pipette ($u_{3.4.2.d}$), the uncertainty in the 10 mL volumetric pipette ($u_{3.4.2.e}$), and the uncertainty in the 2 mL volumetric pipette ($u_{3.4.2.f}$).

a) Uncertainty in the volumetric flasks ($u_{3.4.2.a}$, $u_{3.4.2.b}$, $u_{3.4.2.c}$)

First, calculate $u_{3.4.2.a}$. Calculate $u_{3.4.2.a1}$ using equation (C.15) when a 500 mL volumetric flask with a tolerance of 0.25 mL is used.

$$u_{3.4.2.a1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.25}{\sqrt{3} \times 500} \times 100 = 0.029\ \%$$

Calculate $u_{3.4.2.a2}$ using equation (C.16) when the mean value of repeated measurements is 500.014 g, the standard deviation is 0.012 g, and the number of sample measurements is 1.

$$u_{3.4.2.a2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.012}{500.014} \times \frac{1}{\sqrt{1}} \times 100 = 0.0024\ \%$$

Combine $u_{3.4.2.a1}$ and $u_{3.4.2.a2}$ using equation (C.17) to obtain $u_{3.4.2.a}$.

$$u_{3.4.2.a} = \sqrt{(u_{3.4.2.a1})^2 + (u_{3.4.2.a2})^2} = \sqrt{0.029^2 + 0.0024^2} = 0.029\ \%$$

Next, calculate $u_{3.4.2.b}$. Calculate $u_{3.4.2.b1}$ using equation (C.15) when a 100 mL volumetric flask with a tolerance of 0.1 mL is used.

$$u_{3.4.2.b1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.1}{\sqrt{3} \times 100} \times 100 = 0.058\ \%$$

Calculate $u_{3.4.2.b2}$ using equation (C.16) when the mean value of repeated measurements is 100.002 g, the standard deviation is 0.059 g, and the number of sample measurements is 1.

$$u_{3.4.2.b2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.059}{100.002} \times \frac{1}{\sqrt{1}} \times 100 = 0.059\ \%$$

Combine $u_{3.4.2.b1}$ and $u_{3.4.2.b2}$ using equation (C.17) to obtain $u_{3.4.2.b}$.

$$u_{3.4.2.b} = \sqrt{(u_{3.4.2.b1})^2 + (u_{3.4.2.b2})^2} = \sqrt{0.058^2 + 0.059^2} = 0.083\ \%$$

Lastly, calculate $u_{3.4.2.c}$. Calculate $u_{3.4.2.c1}$ using equation (C.15) when a 200 mL volumetric flask with a tolerance of 0.15 mL is used.

$$u_{3.4.2.c1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.15}{\sqrt{3} \times 200} \times 100 = 0.043\ \%$$

Calculate $u_{3.4.2.c2}$ using equation (C.16) when the mean value of repeated measurements is 200.015 g, the standard deviation is 0.011 g, and the number of sample measurements is 1.

$$u_{3.4.2.c2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.011}{200.015} \times \frac{1}{\sqrt{1}} \times 100 = 0.0055\%$$

Combine $u_{3.4.2.c1}$ and $u_{3.4.2.c2}$ using equation (C.17) to obtain $u_{3.4.2.c}$.

$$u_{3.4.2.c} = \sqrt{(u_{3.4.2.c1})^2 + (u_{3.4.2.c2})^2} = \sqrt{0.043^2 + 0.0055^2} = 0.043\ \%$$

b) Uncertainty in the volumetric pipettes ($u_{3.4.2.d}$, $u_{3.4.2.e}$, $u_{3.4.2.f}$)

First, calculate $u_{3.4.2.d}$. Calculate $u_{3.4.2.d1}$ using equation (C.18) when a 5 mL volumetric pipette with a tolerance of 0.015 mL is used.

$$u_{3.4.2.d1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.015}{\sqrt{3} \times 5} \times 100 = 0.17\ \%$$

Calculate $u_{3.4.2.d2}$ using equation (C.19) when the mean value of repeated measurements is 5.003 g, the standard deviation is 0.004 g, and the number of sample measurements is 1.

$$u_{3.4.2.d2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.004}{5.003} \times \frac{1}{\sqrt{1}} \times 100 = 0.080\ \%$$

Combine $u_{3.4.2.d1}$ and $u_{3.4.2.d2}$ using equation (C.20) to obtain $u_{3.4.2.d}$.

$$u_{3.4.2.d} = \sqrt{(u_{3.4.2.d1})^2 + (u_{3.4.2.d2})^2} = \sqrt{0.17^2 + 0.080^2} = 0.19\ \%$$

Next, calculate $u_{3.4.2.e}$. Calculate $u_{3.4.2.e1}$ using equation (C.18) when a 10 mL volumetric pipette with a tolerance of 0.02 mL is used.

$$u_{3.4.2.e1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.02}{\sqrt{3} \times 10} \times 100 = 0.12\ \%$$

Calculate $u_{3.4.2.e2}$ using equation (C.19) when the mean value of repeated measurements is 10.013 g, the standard deviation is 0.007 g, and the number of sample measurements is 1.

$$u_{3.4.2.e2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.007}{10.013} \times \frac{1}{\sqrt{1}} \times 100 = 0.070\ \%$$

Combine $u_{3.4.2.e1}$ and $u_{3.4.2.e2}$ using equation (C.20) to obtain $u_{3.4.2.e}$.

$$u_{3.4.2.e} = \sqrt{(u_{3.4.2.e1})^2 + (u_{3.4.2.e2})^2} = \sqrt{0.12^2 + 0.070^2} = 0.14\ \%$$

Lastly, calculate $u_{3.4.2.f}$. Calculate $u_{3.4.2.f1}$ using equation (C.18) when a 2 mL volumetric pipette with a tolerance of 0.01 mL is used.

$$u_{3.4.2.f1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.01}{\sqrt{3} \times 2} \times 100 = 0.29\ \%$$

Calculate $u_{3.4.2.f2}$ using equation (C.19) when the mean value of repeated measurements is 2.007 g, the standard deviation is 0.003 g, and the number of sample measurements is 1.

$$u_{3.4.2.f2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.003}{2.007} \times \frac{1}{\sqrt{1}} \times 100 = 0.15\ \%$$

Combine $u_{3.4.2.f1}$ and $u_{3.4.2.f2}$ using equation (C.20) to obtain $u_{3.4.2.f}$.

$$u_{3.4.2.f} = \sqrt{(u_{3.4.2.f1})^2 + (u_{3.4.2.f2})^2} = \sqrt{0.29^2 + 0.15^2} = 0.33\ \%$$

Combine $u_{3.4.2.a}$, $u_{3.2.4.b}$, $u_{3.4.2.c}$, $u_{3.4.2.d}$, $u_{3.4.2.e}$, and $u_{3.4.2.f}$ to obtain $u_{3.4.2}$.

$$u_{3.4.2} = \sqrt{(u_{3.4.2.a})^2 + (u_{3.4.2.b})^2 + (u_{3.4.2.c})^2 + (u_{3.4.2.d})^2 + (u_{3.4.2.e})^2 + (u_{3.4.2.f})^2}$$

$$= \sqrt{0.029^2 + 0.083^2 + 0.043^2 + 0.19^2 + 0.14^2 + 0.33^2} = 0.42\ \%$$

c) Combining uncertainties in dilution ($u_{3.4}$)

Calculate $u_{3.4}$ using equation (C.25).

$$u_{3.4} = \sqrt{(u_{3.4.1})^2 + (u_{3.4.2})^2} = \sqrt{0.15^2 + 0.42^2} = 0.45\ \%$$

5) Combining uncertainties in the stable Sr amount in the sample (u_3)

Uncertainties of $u_{3.1}$ through $u_{3.4}$ are combined to obtain u_3.

$$u_3 = \sqrt{(u_{3.1})^2 + (u_{3.2})^2 + (u_{3.3})^2 + (u_{3.4})^2} \qquad \text{(C. 26)}$$

[Calculation example]

Combine $u_{3.1}$ through $u_{3.4}$ using equation (C.26) to obtain u_3.

$$u_3 = \sqrt{(u_{3.1})^2 + (u_{3.2})^2 + (u_{3.3})^2 + (u_{3.4})^2}$$
$$= \sqrt{0.0087^2 + 0.23^2 + 0.50^2 + 0.45^2} = 0.71\ \%$$

(4) Uncertainty in sample quantity (u_4)

As is the case with u_1 described above, the method for calculating the uncertainty in weighing differs depending on whether the uncertainty is stated in the calibration certificate for the electronic balance. If it is not stated, u_4 can be determined by combining the relative standard uncertainties of the uncertainty associated with rounding of the weighing value of the balance ($u_{4.1}$) and the uncertainty associated with repeatability ($u_{4.2}$).

[Calculation example]

Calculate $u_{4.1}$ using equation (C.8) when an electronic balance with a reading limit of 0.01 g is used and the weighing value is 20.10 g.

$$u_{4.1} = \frac{l}{\sqrt{6} \times W} \times 100 = \frac{0.01}{\sqrt{6} \times 20.10} \times 100 = 0.020\ \%$$

Calculate $u_{4.2}$ using equation (C.9) when the mean value of repeated measurements is 20.100 g, the standard deviation is 0.008 g, and the number of sample measurements is 1.

$$u_{4.2} = \frac{W_{\mathrm{STD}}}{\overline{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.008}{20.100} \times \frac{1}{\sqrt{1}} \times 100 = 0.040\ \%$$

Combine $u_{4.1}$ and $u_{4.2}$ using equation (C.10) to obtain u_4.

$$u_4 = \sqrt{(u_{4.1})^2 + (u_{4.2})^2} = \sqrt{0.020^2 + 0.040^2} = 0.045\%$$

(5) Calculating relative combined standard uncertainty in Z

As described above, it is necessary to calculate $u(Z)$ in order to obtain $\frac{u(Z)}{Z}$. Therefore, first, $u(W_1)$, $u(C_{\mathrm{st}})$ and $u(W_{\mathrm{s}})$ are calculated by multiplying u_2, u_3 and u_4 by respective input values. Next, after $u(Z)$ is obtained using equation (C.13), $\frac{u(Z)}{Z}$ is calculated.

[Calculation example]

The values of u_2, u_3 and u_4 obtained in (2) to (4) are as follows.

$$u_2 = \frac{u(W_1)}{W_1} = 1.2\ \% = 0.012$$
$$u_3 = \frac{u(C_{\mathrm{st}})}{C_{\mathrm{st}}} = 0.71\ \% = 0.0071$$
$$u_4 = \frac{u(W_{\mathrm{s}})}{W_{\mathrm{s}}} = 0.045\ \% = 0.00045$$

First, calculate the standard uncertainty of each from the relative standard uncertainty. Calculations are based on the analytical conditions described above: W_1 = 50.00 mg, C_{st} = 0.60 mg/g ash, and W_{s} = 20.10 g.

$$u(W_1) = W_1 \times u_2 = W_1 \times \frac{u(W_1)}{W_1} = 50.00\ \mathrm{mg} \times 0.012 = 0.60\ \mathrm{mg}$$

$$u(C_{\mathrm{st}}) = C_{\mathrm{st}} \times u_3 = C_{\mathrm{st}} \times \frac{u(C_{\mathrm{st}})}{C_{\mathrm{st}}} = 0.60\ \mathrm{mg/g} = 0.60\ \mathrm{mg/g\ ash} \times 0.0071 = 0.0043\ \mathrm{mg/g\ ash}$$

$$u(W_{\mathrm{s}}) = W_{\mathrm{s}} \times u_4 = W_{\mathrm{s}} \times \frac{u(W_{\mathrm{s}})}{W_{\mathrm{s}}} = 20.10\ \mathrm{g} \times 0.00045 = 0.0090\ \mathrm{g}$$

Next, calculate $u(Z)$ using equation (C.13).

$$u(Z) = \sqrt{\left(u(W_1)\right)^2 + {W_s}^2 \times \left(u(C_{st})\right)^2 + {C_{st}}^2 \times \left(u(W_s)\right)^2}$$

$$= \sqrt{0.60^2 + 20.10^2 \times 0.0043^2 + 0.60^2 \times 0.0090^2} = 0.61 \text{ mg}$$

Lastly, determine $\frac{u(Z)}{Z}$ using $Z = 62.06$ mg.

$$\frac{u(Z)}{Z} = \frac{0.61}{62.06} \times 100 = 0.98\ \%$$

(6) Uncertainty in the Sr amount obtained by recovery measurement (u_5)

The recovery of ^{90}Sr, which is the nuclide to be measured, is assumed to be the same as that of stable Sr, and is analyzed assuming that there are no errors due to systematic or accidental effects. There are various methods for determining recovery in Sr analysis. The uncertainty described here is the uncertainty in the case where the stable Sr amount in an assay sample is quantified using ICP-AES.

The recovery is determined in accordance with the following procedures. A specified amount of stable Sr is added as a carrier when pretreating the radioactivity assay sample, and the recovery rate measurement sample is taken from the sample solution after chemical separation is completed.

The measurement sample is diluted and the stable Sr amount is quantified using ICP-AES, which is u_2. The stable Sr amount added as the carrier and the total stable Sr amount in the sample are compared to obtain the recovery rate.

Therefore, u_5 is determined by combining the relative standard uncertainties of the uncertainty in diluting the recovery measurement sample solution ($u_{5.1}$), the uncertainty in the calibration curve ($u_{5.2}$), and the uncertainty in the calibration curve solution ($u_{5.3}$).

1) Uncertainty in diluting the recovery measurement sample solution ($u_{5.1}$)

Volumetric flasks and volumetric pipettes are used to dilute the recovery measurement sample solution. Therefore, as is the case with $u_{2.3}$ and $u_{2.4}$ in the above, $u_{5.1}$ is determined by combining the relative standard uncertainties of the uncertainty in the volumetric flask volume ($u_{5.1.a1}$, $u_{5.1.b1}$, $u_{5.1.c1}$) and the uncertainty in the volumetric flask repeatability ($u_{5.1.a2}$, $u_{5.1.b2}$, $u_{5.1.c2}$), uncertainty in the volumetric pipette volume ($u_{5.1.d1}$, $u_{5.1.e1}$), and the uncertainty in the volumetric pipette repeatability ($u_{5.1.d2}$, $u_{5.1.e2}$).

[Calculation example]

This example shows how to calculate $u_{5.1}$ when sample in a 100 mL volumetric flask is diluted at a ratio of 1:2500. This case uses a 100 mL volumetric flask, two 50 mL volumetric flasks, and two 1 mL volumetric pipettes. To determine $u_{5.1}$, combine the relative standard uncertainties of the uncertainty in the 100 mL volumetric flask ($u_{5.1.a}$), the uncertainty in the 50 mL volumetric flasks ($u_{5.1.b}$, $u_{5.1.c}$) and the uncertainty in the 1 mL volumetric pipettes ($u_{5.1.d}$, $u_{5.1.e}$).

a) Uncertainty in the volumetric flasks ($u_{5.1.a}$, $u_{5.1.b}$, $u_{5.1.c}$)

First, calculate $u_{5.1.a}$. Calculate $u_{5.1.a1}$ using equation (C.15) when a 100 mL volumetric flask with a tolerance of 0.1 mL is used.

$$u_{5.1.a1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.1}{\sqrt{3} \times 100} \times 100 = 0.058\ \%$$

Calculate $u_{5.1.a2}$ using equation (C.16) when the mean value of repeated measurements is 100.002 g, the standard deviation is 0.059 g, and the number of sample measurements is 1.

$$u_{5.1.a2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.059}{100.002} \times \frac{1}{\sqrt{1}} \times 100 = 0.059\ \%$$

Combine $u_{5.1.a1}$ and $u_{5.1.a2}$ using equation (C.17) to obtain $u_{5.1.a}$.

$$u_{5.1.a} = \sqrt{(u_{5.1.a1})^2 + (u_{5.1.a2})^2} = \sqrt{0.058^2 + 0.059^2} = 0.083\ \%$$

Next, calculate $u_{5.1.b}$. Calculate $u_{5.1.b1}$ using equation (C.15) when a 50 mL volumetric flask with a tolerance of 0.06 mL is used.

$$u_{5.1.b1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.06}{\sqrt{3} \times 50} \times 100 = 0.069\ \%$$

Calculate $u_{5.1.b2}$ using equation (C.16) when the mean value of repeated measurements is 49.995 g, the standard deviation is 0.034 g, and the number of sample measurements is 1.

$$u_{5.1.b2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.034}{49.995} \times \frac{1}{\sqrt{1}} \times 100 = 0.068\ \%$$

Combine $u_{5.1.b1}$ and $u_{5.1.b2}$ using equation (C.17) to obtain $u_{5.1.b}$.

$$u_{5.1.b} = \sqrt{(u_{5.1.b1})^2 + (u_{5.1.b2})^2} = \sqrt{0.069^2 + 0.068^2} = 0.097\ \%$$

The value of $u_{5.1.c}$ is the same as that of $u_{5.1.b}$.

$$u_{5.1.c} = u_{5.1.b} = 0.097\ \%$$

b) Uncertainty in volumetric pipettes ($u_{5.1.d}$, $u_{5.1.e}$)

First, calculate $u_{5.1.d}$. Calculate $u_{5.1.d1}$ using equation (C.18) when a 1 mL volumetric pipette with a tolerance of 0.008 mL is used.

$$u_{5.1.d1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.008}{\sqrt{3} \times 1} \times 100 = 0.46\ \%$$

Calculate $u_{5.1.d2}$ using equation (C.19) when the mean value of repeated measurements is 1.001 g, the standard deviation is 0.003 g, and the number of sample measurements is 1.

$$u_{5.1.d2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.003}{1.001} \times \frac{1}{\sqrt{1}} \times 100 = 0.30\ \%$$

Combine $u_{5.1.d1}$ and $u_{5.1.d2}$ using equation (C.20) to obtain $u_{5.1.d}$.

$$u_{5.1.d} = \sqrt{(u_{5.1.d1})^2 + (u_{5.1.d2})^2} = \sqrt{0.46^2 + 0.30^2} = 0.55\ \%$$

The value of $u_{5.1.e}$ is the same as that of $u_{5.1.d}$.

$$u_{5.1.e} = u_{5.1.d} = 0.55\ \%$$

c) Combining uncertainties in dilution ($u_{5.1}$)

Combine $u_{5.1.a}$, $u_{5.1.b}$, $u_{5.1.c}$, $u_{5.1.d}$, and $u_{5.1.e}$ to obtain $u_{5.1}$.

$$u_{5.1} = \sqrt{(u_{5.1.a})^2 + (u_{5.1.b})^2 + (u_{5.1.c})^2 + (u_{5.1.d})^2 + (u_{5.1.e})^2}$$
$$= \sqrt{0.083^2 + 0.097^2 + 0.097^2 + 0.55^2 + 0.55^2} = 0.79\ \%$$

2) Uncertainty in the calibration curve ($u_{5.2}$)

The uncertainty is calculated in the same manner as $u_{3.3}$ described above. Strictly speaking, uncertainty should be evaluated at each measurement. However, the impact of the difference in the uncertainty of calibration curves on the final uncertainty is negligibly small, so evaluation for each measurement is not necessary unless device adjustment is performed or a condition is changed.

[Calculation example]

When the same calibration curve as $u_{3.3}$ described above is used, the obtained combined standard uncertainty of the calibration curve is $u(C_f) = 0.00124$. Calculate $u_{5.2}$ using equation

(*C*.23) when the measured value is 0.18 mg/L[*371].

$$u_{5.2} = \frac{u(C_f)}{C_f} \times 100 = \frac{0.00124}{0.18} \times 100 = 0.69\ \%$$

3) Uncertainty in the calibration curve solution ($u_{5.3}$)

As is the case with $u_{3.4}$ described above, the uncertainty is determined by combining the relative standard uncertainties of the uncertainty in the Sr standard solution ($u_{5.3.1}$) and the uncertainty in diluting the calibration curve solution ($u_{5.3.2}$).

[Calculation example]

When the same calibration solution as that for $u_{3.4}$ is used, the value of $u_{5.3}$ is the same as that of $u_{3.4}$.

$$u_{5.3} = \sqrt{(u_{5.3.1})^2 + (u_{5.3.2})^2}$$

$$= u_{3.4} = \sqrt{(u_{3.4.1})^2 + (u_{3.4.2})^2} = \sqrt{0.15^2 + 0.42^2} = 0.45\ \%$$

4) Combining uncertainties in the Sr amount obtained by recovery measurement (u_5)

The uncertainties of $u_{5.1}$ through $u_{5.3}$ are combined to obtain u_5.

$$u_5 = \sqrt{(u_{5.1})^2 + (u_{5.2})^2 + (u_{5.3})^2} \qquad (C.\ 27)$$

[Calculation example]

Combine $u_{5.1}$ through $u_{5.3}$ using equation (C.27) to obtain u_5. $\left(= \frac{u(W_2)}{W_2}\right)$

$$u_5 = \sqrt{(u_{5.1})^2 + (u_{5.2})^2 + (u_{5.3})^2} = \sqrt{0.79^2 + 0.69^2 + 0.45^2} = 1.1\ \%$$

(7) Combining uncertainties in recovery Y_{Sr} $\left(\frac{u(Y_{Sr})}{Y_{Sr}}\right)$

Combine $\frac{u(Z)}{Z}$ and u_5 $\left(= \frac{u(W_2)}{W_2}\right)$ obtained in (5) and (6) using equation (C.12) to obtain $\frac{u(Y_{Sr})}{Y_{Sr}}$.

[Calculation example]

In (5) and (6), $\frac{u(Z)}{Z}$ and $\frac{u(W_2)}{W_2}$ are determined as follows.

$$\frac{u(Z)}{Z} = 0.98\ \%$$

$$\frac{u(W_2)}{W_2} = 1.1\ \%$$

Calculate $\frac{u(Y_{Sr})}{Y_{Sr}}$ using equation (C.12).

$$\frac{u(Y_{Sr})}{Y_{Sr}} = \sqrt{\left(\frac{u(Z)}{Z}\right)^2 + \left(\frac{u(W_2)}{W_2}\right)^2} = \sqrt{0.98^2 + 1.1^2} = 1.5\ \%$$

[*371] Strictly speaking, when the same calibration curve as the one used for $u_{3.3}$ or the Sr standard solution prepared for $u_{3.3}$ is used, the correlation between the uncertainties in the concentration should be taken into account. However, it is not factored in because its impact is small.

C.3.3 Uncertainty in ^{90}Y counting efficiency $\varepsilon(\frac{u(\varepsilon)}{\varepsilon})$

In the measurement of Sr, uncertainties to be taken into account are the uncertainty in the standard source used for calibration (counting efficiency ε), the uncertainty associated with preparation of calibration sources, and the uncertainty associated with measurement of calibration sources. Therefore, $\frac{u(\varepsilon)}{\varepsilon}$ is determined by combining the relative standard uncertainties of the uncertainty in the standard source (u_6), the uncertainty associated with the preparation of calibration sources (u_7), the uncertainty due to counting statistics (u_8), the uncertainty associated with the calibration formula fitting (u_9) and the uncertainty due to decay correction (u_{10}).

(1) Uncertainty in the standard source (u_6)

The uncertainty to be used is the uncertainty stated on the certificate of analysis for the standard source used for calibration.

(2) Uncertainty associated with the preparation of calibration sources (u_7)

To prepare calibration sources, a fixed quantity of the ^{90}Sr standard source is separated with a volumetric pipette to prepare multiple efficiency measurement samples (calibration sources). Therefore, u_7 can be determined by combining the relative standard uncertainties of the uncertainty in weighing ($u_{7.1}$) and the uncertainty in the volumetric pipette ($u_{7.2}$).

1) Uncertainty in weighing ($u_{7.1}$)

As is the case with u_1 described above, the method for calculating the uncertainty in weighing differs depending on whether the uncertainty is stated in the calibration certificate for the electronic balance. If it is not stated, the uncertainty can be determined by combining the relative standard uncertainties of the uncertainty associated with rounding of the weighing value ($u_{7.1.1}$) and the uncertainty associated with the weighing repeatability ($u_{7.1.2}$).

2) Uncertainty in the volumetric pipette ($u_{7.2}$)

As is the case with $u_{2.4}$ described above, the uncertainty in the volumetric pipette is determined by combining the relative standard uncertainties of the uncertainty in the volumetric pipette volume ($u_{7.2.1}$) and the uncertainty associated with repeatability ($u_{7.2.2}$).

3) Combining uncertainties associated with the preparation of calibration sources (u_7)

Uncertainties of $u_{7.1.1}$, $u_{7.1.2}$, $u_{7.2.1}$, and $u_{7.2.2}$ are combined to obtain u_7.

$$u_7 = \sqrt{(u_{7.1.1})^2 + (u_{7.1.2})^2 + (u_{7.2.1})^2 + (u_{7.2.2})^2} \qquad \text{(C. 28)}$$

[Calculation example]

Calculate $u_{7.1.1}$ using equation (C.8) when an electronic balance with a reading limit of 0.00001 g is used and the weighing value is 1.00066 g.

$$u_{7.1.1} = \frac{l}{\sqrt{6} \times W} \times 100 = \frac{0.00001}{\sqrt{6} \times 1.00066} \times 100 = 0.00041\ \%$$

Calculate $u_{7.1.2}$ using equation (C.9) when the mean value of repeated measurements is 1.000660 g, the standard deviation is 0.000089 g, and the number of sample measurements is 1.

$$u_{7.1.2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.000089}{1.000660} \times \frac{1}{\sqrt{1}} \times 100 = 0.0089\ \%$$

Calculate $u_{7.2.1}$ using equation (C.18) when a 5 mL volumetric pipette with a tolerance of 0.015 mL is used.

$$u_{7.2.1} = \frac{V_i}{\sqrt{3} \times V} \times 100 = \frac{0.015}{\sqrt{3} \times 5} \times 100 = 0.17\ \%$$

Calculate $u_{7.2.2}$ using equation (C.19) when the mean value of repeated measurements is 5.003 g, the standard deviation is 0.004 g, and the number of sample measurements is 1.

$$u_{7.2.2} = \frac{W_{\mathrm{STD}}}{\bar{W}} \times \frac{1}{\sqrt{n}} \times 100 = \frac{0.004}{5.003} \times \frac{1}{\sqrt{1}} \times 100 = 0.080\ \%$$

Combine $u_{7.1.1}$, $u_{7.1.2}$, $u_{7.2.1}$, and $u_{7.2.2}$ using equation (C.28) to obtain u_7.

$$u_7 = \sqrt{(u_{7.1.1})^2 + (u_{7.1.2})^2 + (u_{7.2.1})^2 + (u_{7.2.2})^2}$$
$$= \sqrt{0.00041^2 + 0.0089^2 + 0.17^2 + 0.080^2} = 0.19\ \%$$

(3) Uncertainty due to counting statistics (u_8)

The relative standard uncertainty is calculated from the count obtained by measuring the calibration source and the uncertainty due to counting statistics. The relative standard uncertainty (u_8) is determined for all the prepared calibration sources, and the largest one (u_{8max}) is set as u_8. However, this may be omitted because the uncertainty due to counting statistics is factored in when determining the "coefficient of variation" in calibration formula fitting (u_9) in the following paragraph (4).

$$u_8 = \frac{\sigma_{\mathrm{source}}}{N_{\mathrm{source}}} \times 100 = \frac{\sqrt{N_{\mathrm{source}}}}{N_{\mathrm{source}}} \times 100(\%) \qquad \text{(C. 29)}$$

N_{source}: Count obtained through the calibration source measurement

σ_{source}: Uncertainty due to counting statistics

[Calculation example]

Assume that the following result is obtained by measuring ***n*** calibration sources.

Table C.4 Calibration sources measurement result

Calibration source	Count obtained through calibration source measurement	Uncertainty due to counting statistics	Relative standard uncertainty (u_8)(%)
1	40479	201.19	0.50
2	35167	187.53	0.53
3	35373	188.08	0.53
4	40711	201.77	0.50
5	34748	186.41	0.54
⋮	⋮	⋮	⋮
n	40448	201.12	0.50

*When more than one source is used for calibration, use the maximum value of all the relative standard uncertainties.

For example, u_8 of calibration source 5 is obtained from equation (C.29) as follows.

$$u_8 = \frac{\sigma_{\mathrm{source}}}{N_{\mathrm{source}}} \times 100 = \frac{\sqrt{N_{\mathrm{source}}}}{N_{\mathrm{source}}} \times 100 = \frac{\sqrt{34748}}{34748} \times 100 = 0.54\ (\%)$$

When u_8 is calculated in this way, the value of u_8 in Table C.4 is given. The maximum of them is adopted as $u_{8\ \max}$ as follows.

$u_{8\ \max} = u_8 = 0.54\%$

(4) Uncertainty associated with calibration formula fitting (u_9)

Based on the calibration data of the standard sources, all the coefficients of variation (f) relative to actual measurements are calculated, and the maximum of them (f_{max}) is adopted as u_9. The coefficient of variation relative to actual measurement value is calculated using the following equation.

$$f = \frac{|\text{Actual measurement - Median}|}{\text{Actual measurement}} \times 100\ (\%) \qquad \text{(C.30)}$$

$u_9 = f_{max}$

f_{max}: Maximum value of the coefficients of variation relative to actual measurements

[Calculation example]

Assume that efficiency calibration in a windowed detector is performed using ^{90}Y calibration sources, and that the following results are obtained.

Table C.5 Calibration data of the calibration sources

^{90}Y calibration source	Median	Actual measurement	Coefficient of variation relative to actual measurement (u_9) (%)
1	0.26877	0.26844	0.12
2		0.26950	0.27
3		0.27029	0.56
4		0.26852	0.093
5		0.26877	0
⋮		⋮	⋮
n		0.26579	1.1

*When more than one source is used for calibration, use the maximum value of all the relative standard uncertainties.

For example, u_9 of the ^{90}Y calibration source n is obtained from equation (C.30) as follows.

$$u_9 = \frac{|\text{Actual measurement - Median}|}{\text{Actual measurement}} \times 100 = \frac{|0.26579 - 0.26877|}{0.26579} \times 100 = 1.1\ (\%)$$

When u_9 is calculated in this way, the value of u_9 in Table C.5 is given. The maximum of them is adopted as u_{9max} as follows.

$u_{9\,max} = f_{max} = 1.1\%$

(5) Uncertainty due to decay correction (u_{10})

The decay correction factors based on the elapsed time from the reference date and time are calculated for both half-life and half-life + uncertainty, and the relative value of the difference between them is adopted as u_{10}. Since the uncertainty due to decay correction is negligibly small, it is not necessary to evaluate it in actual operation.

$$DF_1 = \left(\frac{1}{2}\right)^{\frac{t_1}{T_{90Sr}}} \times \left(\frac{1}{2}\right)^{\frac{t_2}{T_{90Y}}} \quad \text{(C. 31)}$$

$$DF_2 = \left(\frac{1}{2}\right)^{\frac{t_1}{T_{90Sr}+\sigma}} \times \left(\frac{1}{2}\right)^{\frac{t_2}{T_{90Y}+\sigma}} \quad \text{(C. 32)}$$

$$u_{10} = \frac{|DF_2 - DF_1|}{DF_1} \times 100\ (\%) \quad \text{(C. 33)}$$

t_1 : Elapsed time from the reference date of the ^{90}Sr standard source to the preparation of the calibration source (separation of ^{90}Y)

t_2 : Elapsed time from the preparation of the calibration source (separation of ^{90}Y) to the start of measurement

T_{90Sr} : ^{90}Sr half-life

$T_{90Sr} + \sigma$: ^{90}Sr half-life + uncertainty

T_{90Y} : *90Y* half-life

$T_{90Y} + \sigma$: *90Y* half-life + uncertainty

DF_1 : Decay correction factor for T_{90Sr} and T_{90Y}

DF_2 : Decay correction factor for $T_{90Sr} + \sigma$ and $T_{90Y} + \sigma$

[Calculation example]

Table C.6 shows the half-lives of ^{90}Y and ^{90}Sr and uncertainties in them. The Evaluated Nuclear Structure Data File (ENSDF) (as of February 2025) was used as the nuclear data.

Table C.6 Half-lives and uncertainties in them

Nuclide	Half-life	Uncertainty in half-life
^{90}Y	64.05 hours	0.05 hours
^{90}Sr	28.91 years	0.03 years

Calculate DF_1 and DF_2 using equations (C. 31) and (C. 32) assuming that the elapsed time from the reference date of the ^{90}Sr standard source to the preparation of the calibration source is 60 days and that the elapsed time from the preparation of the calibration source to the start of measurement is 5 hours.

$$DF_1 = \left(\frac{1}{2}\right)^{\frac{t_1}{T_{90Sr}}} \times \left(\frac{1}{2}\right)^{\frac{t_2}{T_{90Y}}} = \left(\frac{1}{2}\right)^{\frac{60\div 365.25}{28.91}} \times \left(\frac{1}{2}\right)^{\frac{5}{64.05}}$$

$$= 0.996069 \times 0.947328 = 0.943604$$

$$DF_2 = \left(\frac{1}{2}\right)^{\frac{t_1}{T_{90Sr}+\sigma}} \times \left(\frac{1}{2}\right)^{\frac{t_2}{T_{90Y}+\sigma}} = \left(\frac{1}{2}\right)^{\frac{60\div 365.25}{28.91+0.03}} \times \left(\frac{1}{2}\right)^{\frac{5}{64.05+0.05}}$$

$$= 0.996073 \times 0.947368 = 0.943648$$

Calculate u_{10} using equation (C.33).

$$u_{10} = \frac{|DF_2 - DF_1|}{DF_1} \times 100 = \frac{|0.943648 - 0.943604|}{0.943604} \times 100 = 0.0046\ \%$$

(6) Combining uncertainties in ^{90}Y counting efficiency ε $(\frac{u(\varepsilon)}{\varepsilon})$

Uncertainties of u_6, u_7, u_8, u_9, and u_{10}.are combined to obtain $\frac{u(\varepsilon)}{\varepsilon}$.

$$\frac{u(\varepsilon)}{\varepsilon} = \sqrt{(u_6)^2 + (u_7)^2 + (u_8)^2 + (u_9)^2 + (u_{10})^2} \quad \text{(C. 34)}$$

[Calculation example]

The values of u_6, u_7, u_8, u_9, and u_{10} obtained in (1) to (5) are as follows. Here, a relative standard uncertainty of 0.65% is given on the certificate of analysis of the standard source used for calibration.

Standard source	:$u_6 = 0.65\%$
Preparation of calibration sources	:$u_7 = 0.19\%$
Counting statistics	:$u_8 = 0.54\%$
Calibration formula fitting	:$u_9 = 1.1\%$
Decay correction	:$u_{10} = 0.0046\%$

Combine them using equation (C.34) to obtain $\frac{u(\varepsilon)}{\varepsilon}$.

$$\frac{u(\varepsilon)}{\varepsilon} = \sqrt{0.65^2 + 0.19^2 + 0.54^2 + 1.1^2 + 0.0046^2} = 1.4\ \%$$

C.3.4 Uncertainty in ^{90}Y net counting rate r_n ($\frac{u(r_n)}{r_n}$)

To calculate the radioactive concentration in the measurement of Sr, net counting rate r_n, which is obtained by subtracting the background counting rate from the sample counting rate, is corrected for the detector counting efficiency and the half-life. Therefore, $\frac{u(r_n)}{r_n}$ can be determined by combining the relative standard uncertainties of the uncertainty due to counting statistics (u_{11}), uncertainty due to variations in the measurement system (u_{12}), and the uncertainty due to decay correction (u_{13}).

(1) Uncertainty due to counting statistics (u_{11})

The uncertainty is calculated in the same manner as u_8 described above. For the calculation of the uncertainty, the net counting rate of an unknown sample is used.

$$u_{11} = \frac{\sigma_{r_n}}{r_n} \times 100\ (\%) \qquad \text{(C. 35)}$$

r_n : Net counting rate of unknown sample

σ_{rn} : Statistical uncertainty of the net counting rate

[Calculation example]

The statistical uncertainty u_{11} of a net counting rate of 0.0094 ± 0.0019 cps is calculated using equation (C.35) while assuming a measurement time of 60 minutes, an unknown sample count of 40, and a background count of 6.

$$u_{11} = \frac{\sigma_{r_n}}{r_n} \times 100 = \frac{0.0019}{0.0094} \times 100 = 20\ \%$$

(2) Uncertainty due to variations in the measurement system (u_{12})

The uncertainty due to variations in the measurement system can be estimated by determining the stability of the measuring device, that is, the tolerance limits of the variation range.

For example, when the beta-ray source for checking is measured under a certain condition for daily accuracy control, tolerance limits of ± R are set for the variation range such as the following and the values are used for calculating the uncertainty: that the difference between the obtained counting rate and the value on the certificate of analysis should be within a certain range, or that the difference between the obtained counting rate and the average value of the beta-ray source for checking over the past year should be up to three times the standard deviation. The uncertainty of u_{12} distributes rectangularly whose upper and lower limits are the tolerance limits of the variation range, and therefore can be calculated using the following equation.

$$u_{12} = \frac{R}{\sqrt{3}} \times 100\ (\%) \qquad \text{(C. 36)}$$

R: Upper tolerance limit for the variation range

[Calculation example]

Calculate u_{12} from equation (C. 36) when tolerance limits for the variation range of ± 0.6% is obtained by measuring the beta-ray source for checking.

$$u_{12} = \frac{R}{\sqrt{3}} \times 100 \ = \frac{0.006}{\sqrt{3}} \times 100 = 0.35\ \%$$

(3) Uncertainty due to decay correction (u_{13})

As is the case with u_{10} described above, the decay correction factors based on the elapsed time from the reference date and time are calculated for both half-life and half-life + uncertainty, and the relative value of the difference between them is adopted as u_{13}. Since the uncertainty due to decay correction is negligibly small, it is not necessary to evaluate it in actual operation.

$$DF_1 = \left(\frac{1}{2}\right)^{\frac{t_1}{T_{90\mathrm{Sr}}}} \times \left(1 - \left(\frac{1}{2}\right)^{\frac{t_2}{T_{90\mathrm{Y}}}}\right) \times \left(\frac{1}{2}\right)^{\frac{t_3}{T_{90\mathrm{Y}}}} \qquad \text{(C. 37)}$$

$$DF_2 = \left(\frac{1}{2}\right)^{\frac{t_1}{T_{90\mathrm{Sr}}+\sigma}} \times \left(1 - \left(\frac{1}{2}\right)^{\frac{t_2}{T_{90\mathrm{Y}}+\sigma}}\right) \times \left(\frac{1}{2}\right)^{\frac{t_3}{T_{90\mathrm{Y}}+\sigma}} \qquad \text{(C. 38)}$$

$$u_{13} = \frac{|DF_2 - DF_1|}{DF_1} \times 100\ (\%) \qquad \text{(C. 39)}$$

t_1 : Elapsed time from the sampling date (decay correction reference date) to scavenging

t_2 : Elapsed time from scavenging to milking

t_3 : Elapsed time from milking to the start of measurement

$T_{90\mathrm{Sr}}$: ^{90}Sr half-life

$T_{90\mathrm{Sr}} + \sigma$: ^{90}Sr half-life + uncertainty

$T_{90\mathrm{Y}}$: ^{90}Y half-life

$T_{90\mathrm{Y}} + \sigma$: ^{90}Y half-life + uncertainty

DF_1 : Decay correction factor for $T_{90\mathrm{Sr}}$ and $T_{90\mathrm{Y}}$

DF_2 : Decay correction factor for $T_{90\mathrm{Sr}} + \sigma$ and $T_{90\mathrm{Y}} + \sigma$

[Calculation example]

Calculate DF_1 and DF_2 using equations (C.37) and (C.38) assuming that the elapsed time from the reference date to scavenging is 60 days, that from scavenging to milking is 14 days, and that from milking to the start of measurement is 5 hours.

$$DF_1 = \left(\frac{1}{2}\right)^{\frac{t_1}{T_{90\mathrm{Sr}}}} \times \left(1 - \left(\frac{1}{2}\right)^{\frac{t_2}{T_{90\mathrm{Y}}}}\right) \times \left(\frac{1}{2}\right)^{\frac{t_3}{T_{90\mathrm{Y}}}}$$

$$= \left(\frac{1}{2}\right)^{\frac{60\div365.25}{28.91}} \times \left(1 - \left(\frac{1}{2}\right)^{\frac{14\times24}{64.05}}\right) \times \left(\frac{1}{2}\right)^{\frac{5}{64.05}}$$

$$= 0.996069 \times 0.973647 \times 0.947328 = 0.918737$$

$$DF_2 = \left(\frac{1}{2}\right)^{\frac{t_1}{T_{90\mathrm{Sr}}+\sigma}} \times \left(1 - \left(\frac{1}{2}\right)^{\frac{t_2}{T_{90\mathrm{Y}}+\sigma}}\right) \times \left(\frac{1}{2}\right)^{\frac{t_3}{T_{90\mathrm{Y}}+\sigma}}$$

$$= \left(\frac{1}{2}\right)^{\frac{60\div365.25}{28.91+0.03}} \times \left(1 - \left(\frac{1}{2}\right)^{\frac{14\times24}{64.05+0.05}}\right) \times \left(\frac{1}{2}\right)^{\frac{5}{64.05+0.05}}$$

$$= 0.996073 \times 0.973572 \times 0.947368 = 0.918709$$

Calculate u_{13} using equation (C.39).

$$u_{13} = \frac{|DF_2 - DF_1|}{DF_1} \times 100 = \frac{|0.918709 - 0.918737|}{0.918737} \times 100 \ = 0.0030\ \%$$

(4) Combining uncertainties in ^{90}Y net counting rate r_n $\left(\frac{u(r_\mathrm{n})}{r_\mathrm{n}}\right)$

Uncertainties of u_{11} through u_{13} obtained in (1) to (3) are combined to obtain $\frac{u(r_\mathrm{n})}{r_\mathrm{n}}$.

$$\frac{u(r_\mathrm{n})}{r_\mathrm{n}} = \sqrt{(u_{11})^2 + (u_{12})^2 + (u_{13})^2} \qquad \text{(C. 40)}$$

[Calculation example]

The values of u_{11}, u_{12} and u_{13} obtained in (1) to (3) are as follows.

Counting statistics :u_{11} = 20%

Variations in the measurement system :u_{12} = 0.35%

Decay correction :u_{13} = 0.0030%

Combine them using equation (C.40) to obtain $\frac{u(r_\mathrm{n})}{r_\mathrm{n}}$.

$$\frac{u(r_\mathrm{n})}{r_\mathrm{n}} = \sqrt{20^2 + 0.35^2 + 0.0030^2} = 20\ \%$$

C.3.5 Uncertainty in ^{90}Sr radioactive concentration A ($\frac{u(A)}{A}$)

The relative standard uncertainties obtained in C.3.1 to C.3.4 are combined using equation (C.6) to obtain $\frac{u(A)}{A}$.

[Calculation example]

The relative standard uncertainties determined in C.3.1 to C.3.4 are as follows.

$$\frac{u(W_s)}{W_s} = 0.045\ \%$$

$$\frac{u(Y_{Sr})}{Y_{Sr}} = 1.5\ \%$$

$$\frac{u(\varepsilon)}{\varepsilon} = 1.4\%$$

$$\frac{u(r_n)}{r_n} = 20\ \%$$

Combine them using equation (C.6) to obtain $\frac{u(A)}{A}$.

$$\frac{u(A)}{A} = \sqrt{\left(\frac{u(W_s)}{W_s}\right)^2 + \left(\frac{u(Y_{Sr})}{Y_{Sr}}\right)^2 + \left(\frac{u(\varepsilon)}{\varepsilon}\right)^2 + \left(\frac{u(r_n)}{r_n}\right)^2}$$

$$= \sqrt{0.045^2 + 1.5^2 + 1.4^2 + 20^2} = 20\ \%$$

In the calculation example this time, the uncertainty factor which has a large contribution was the uncertainty due to counting statistics. In the measurement of environmental samples, ^{90}Sr is often detected at low concentrations, and the uncertainty due to counting statistics often makes the largest contribution. The magnitude of the contribution of each uncertainty factor should be examined, and it is desirable to consider whether the uncertainty in the factor with a large contribution can be reduced by reviewing the analysis and measurement conditions. In the case herein, since the uncertainty due to counting statistics has the largest contribution, it can be reduced by lengthening the measurement time. The calculation example for this measuring method intentionally also includes explanations of factors with small contributions in order to assist in learning how to evaluate various factors. However, in actual evaluation of uncertainties, it is better to first give top priority to the uncertainty associated with the net counting rate of ^{90}Y. In order to reliably evaluate uncertainty factors with large contributions, it is advisable to evaluate additional factors that are likely to contain a large uncertainty while using the uncertainty of each factor in this calculation example as a reference, to combine them with the uncertainty due to counting statistics, and then determine whether the uncertainty factor should be considered.

Explanation D Lower detection limit calculation example

D.1 Lower detection limit according to ISO 11929

D.1.1 Concept

Regarding the lower detection limit, ISO 11929 introduces the concept of type I and type II errors for the distributions of both background and measured values to define the decision threshold and lower detection limit.

In addition, it is designed to handle uncertainties in the standard deviation of each distribution. The Kaiser method uses the uncertainty due to counting statistics for the standard deviation of measured value distributions, whereas the lower detection limit according to ISO 11929 uses the combined standard uncertainty in the measured quantity for the standard deviation of distributions in line with the ISO/IEC Guide 98-3:2008 “Guide to the expression of uncertainty in measurement” (GUM)[*372].

D.1.2 Decision threshold (A^*)

Value A^* is the value at which physical effects are determined to be present, and it is defined by the distribution of blank measurements and expressed by the following equation:

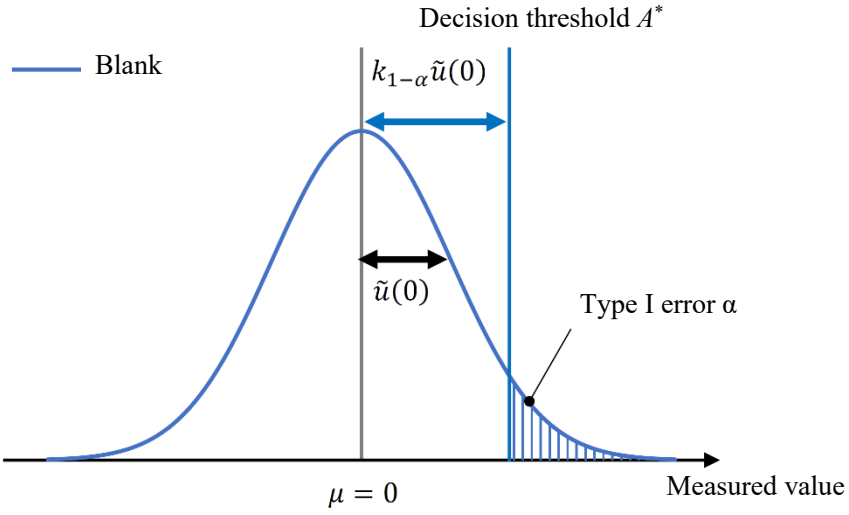
$$A^* = k_{1-\alpha} \times \tilde{u}(0) \qquad (D.1)$$

where $k_{1-\alpha}$: Coefficient for avoiding type I errors (false positive)

$\tilde{u}(0)$: Standard uncertainty in the measured value when the primary estimate is zero

The measured values in the measurement of a background that does not contain the target radionuclide at all distribute (μ= 0) as shown in Figure D.1. In this distribution, the upper limit of the range within which the reliability is 1-α (risk rate α) is adopted as the decision threshold, and measured values that exceed the upper limit are considered to be type I errors that are significantly different from the blank measurement and physical effects are present. In this case, the probability of type I errors is α.

[*372] 今井秀孝他: 測定における不確かさの表現のガイド[GUM]ハンドブック, published by the Japanese Standards Association
ISBN:9784542307056(2018)



ũ(0): Uncertainty in the measured value when the primary estimate is zero
$k_{1\text{-}\alpha}$: Coefficient for avoiding type I errors (false positive)

Figure D.1 Decision threshold

D.1.3 Lower detection limit ($A^{\#}$)

This value determines that a measured value is derived not only from a blank but also from the sample, and is defined as follows.

$$A^{\#} = A^{*} + k_{1-\beta} \times \tilde{u}(A^{\#}) \qquad \text{(D. 2)}$$

where $k_{1\text{-}\beta}$: Coefficient for avoiding type II errors (false negative)
ũ($A^{\#}$): Standard uncertainty in the measured value when the primary estimate is $A^{\#}$

In measurement of the sample, when the primary estimate of the measured quantity is larger than but close to the decision threshold, the measured values distribute as shown in Figure D.2, and the distribution overlaps with the distribution of the blank measurement.

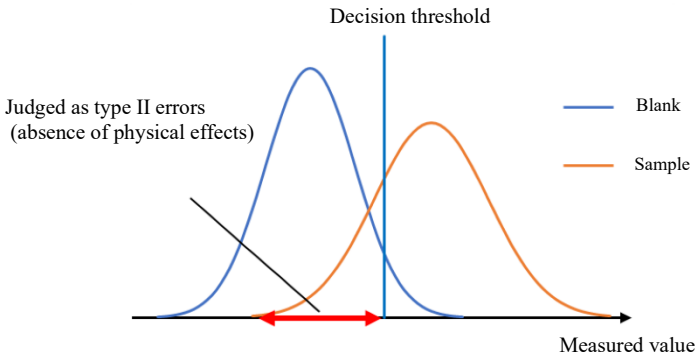
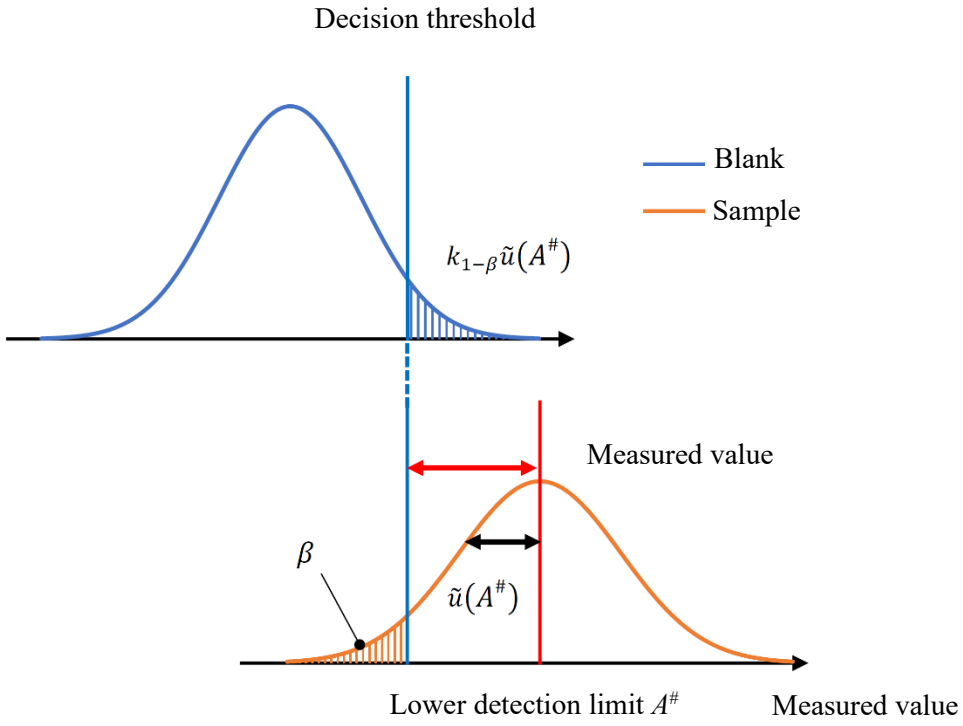


Figure D.2 Distribution of the measured values when the primary estimate of the measured quantity is close to the decision threshold

In this case, the measured values within the section shown in red in Figure D.2 are below the decision threshold, and it is judged that physical effects are absent. Therefore, in the distribution of the sample measurement, a section with a power of 1 - β is provided, and the primary estimate of the measured quantity when the lower limit of the section matches the decision threshold is set as the lower detection limit as shown in Figure D.3. In this case, the probability of type II errors is β.



$\tilde{u}(a^{\#})$: Standard uncertainty in the measured value when the primary estimate is $A^{\#}$
$k_{1-\beta}$: Coefficient for avoiding type II errors (false negative)

Figure D.3 Lower detection limit

The lower detection limit is compared with guideline value A_r to evaluate the measurement procedure. When $A^{\#} < A_r$, the measurement procedure is evaluated as adequate. When $A^{\#} > A_r$, it is necessary to lower the lower detection limit by, for example, lengthening the measurement time. Guideline value A_r for general measurement adopted in ISO 11929-4:2022 is 3 Bq. (For cases where the background count is small, it is set at 0.1 Bq.)

Here, guideline value A that is specified in ISO 11929-1:2019 is the value that corresponds to scientific, legal or other requirements regarding detection ability, and is also the value compared with the lower detection limit to evaluate the measurement procedure[*373]. Figure D.4 summarizes the concepts of the decision threshold and the lower detection limit in one diagram.

[*373] For example, the guideline value is given as activity, specific activity, radioactivity concentration, surface concentration of radioactivity or dose rate. By comparing the lower detection limit with the guideline value, whether the measurement procedure meets the requirements defined by the guideline value, and therefore whether the measurement procedure is suitable for the measurement purpose, can be determined. The measurement procedure meets the requirements when the lower detection limit is smaller than the guideline value. The guideline value should not be confused with other values specified in compliance requirements, regulations or restrictions.

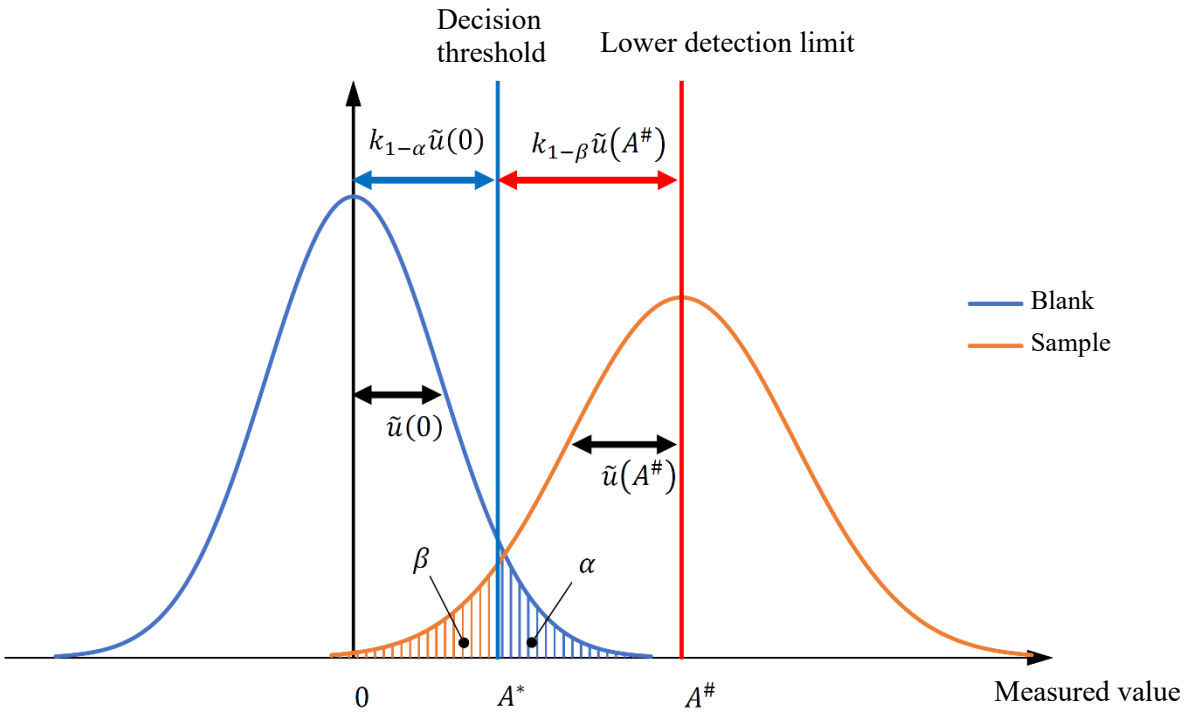


Figure D.4 Concept of lower detection limit according to ISO 11929

D.1.4 Lower detection limit calculation example according to ISO 11929

The decision threshold and lower detection limit are derived using the model equations described in ISO 11929-4[*374].

(1) General formula for determining radioactivity from a count value

$$A = r_n \times w = (r_g - r_0) \times w \quad \text{(D. 3)}$$

A : Measured quantity (radioactivity concentration)

r_n : Net counting rate

w : Conversion factor from counting rate to radioactivity. When the detection efficiency is ε, the chemical recovery is Y, the sample quantity (kg, L, etc.) is W_s, and the decay correction factor is DF,

$$w = \frac{1}{\varepsilon \times Y \times W_s \times DF}$$

is true.

However, W_s is not necessary when the measured quantity A is determined as the radioactivity.

r_g : Sample measurement counting rate When the sample measurement count value is N_g and the measurement time is t_g, $r_g = \frac{N_g}{t_g}$ is true.

r_0 : Background measurement counting rate When the background measurement counting rate is N_0 and the measurement time is t_0, $r_0 = \frac{N_0}{t_0}$ is true.

In this case, when the standard uncertainties of r_n and w are respectively $u(r_n)$ and $u(w)$, the uncertainty in A, which is $u(A)$, is expressed as follows.

$$u(A) = r_n \times w \times \sqrt{\left(\frac{u(r_n)}{r_n}\right)^2 + \left(\frac{u(w)}{w}\right)^2} \quad \text{(D. 4)}$$

$$= \sqrt{w^2 u^2(r_n) + r_n^2\, u^2(w)} \quad \text{(D. 5)}$$

Then, from (D.3), $r_n = \frac{A}{w}$ and $u(r_n) = \sqrt{\frac{r_g}{t_g} + \frac{r_0}{t_0}}$ are true, and they are substituted into equation (D.5).

$$u(A) = \sqrt{w^2 \left(\frac{r_g}{t_g} + \frac{r_0}{t_0}\right) + A^2 \frac{u^2(w)}{w^2}} \quad \text{(D. 6)}$$

The value of $\frac{u(w)}{w}$ represents the relative combined standard uncertainty $u_{rel}(w)$ excluding the uncertainty associated with measurement counting.

In order to calculate the decision threshold and lower detection limit in accordance with ISO 11929, it is necessary to obtain the function for the uncertainty in the measured value using the primary estimate of the measured quantity $\tilde{A}$ as variable $\tilde{u}(\tilde{A})$. From equation (D.3), the following equation is expressed using the primary estimate of $\tilde{A}$.

$$r_g = \frac{\tilde{A}}{w} + r_0 \quad \text{(D. 7)}$$

When it is substituted into equation (D.6),

[*374] ISO 11929-4:2022, Determination of the characteristic limits (decision threshold, detection limit and limits of the coverage interval) for measurements of ionizing radiation — Fundamentals and application — Part 4:Guidelines to applications. ISO 11929-4:2022 (2022)

$$\tilde{u}(\tilde{A}) = \sqrt{w^2\left(\frac{\left(\frac{\tilde{A}}{w} + r_0\right)}{t_g} + \frac{r_0}{t_0}\right) + \tilde{A}^2\frac{u^2(w)}{w^2}} \qquad \text{(D. 8)}$$

is obtained.

(2) Decision threshold

The primary estimate of $\tilde{A} = 0$ is substituted into equation (D.8), and the result is substituted in $\tilde{u}(0)$ of equation (D.1).

$$A^* = k_{1-\alpha} \times w \times \sqrt{\frac{N_0}{t_0 t_g} + \frac{N_0}{t_0^2}} \qquad \text{(D. 9)}$$

A^* : Decision threshold (Bq or Bq/kg, Bq/L, etc.)
$k_{1-\alpha}$: Coefficient for avoiding type I errors (false positive)

(3) Lower detection limit

When the lower detection limit is set to $A^{\#}$, $A^{\#}$ is substituted into equation (D.8), and the result is substituted for $\tilde{u}(A^{\#})$ in equation (D.2).

$$A^{\#} = A^* + k_{1-\beta}\sqrt{w^2\left(\frac{\left(\frac{A^{\#}}{w} + r_0\right)}{t_g} + \frac{r_0}{t_0}\right) + A^{\#2}\frac{u^2(w)}{w^2}}$$

$$(A^{\#} - A^*)^2 = k_{1-\beta}^2\left[w^2\left(\frac{\left(\frac{A^{\#}}{w} + r_0\right)}{t_g} + \frac{r_0}{t_0}\right) + A^{\#2}\frac{u^2(w)}{w^2}\right]$$

$$\left(1 - k_{1-\beta}^2 \times u_{\mathrm{rel}}^2(w)\right)A^{\#2} - 2A^*A^{\#} + A^{*2} = k_{1-\beta}^2 w^2\left(\frac{A^{\#}}{w t_g} + \frac{r_0}{t_g} + \frac{r_0}{t_0}\right) \qquad \text{(D. 10)}$$

Here, when $k_{1-\alpha} = k_{1-\beta} = k$, from equation (D.9)

$$A^{*2} = k^2 \times w^2\left(\frac{r_0}{t_g} + \frac{r_0}{t_0}\right)$$

is true. Therefore, this expression is substituted into equation (D.10).

$$\left(1 - k^2 \times u_{\mathrm{rel}}^2(w)\right)A^{\#2} - 2A^*A^{\#} + A^{*2} = \frac{k^2 w}{t_g}A^{\#} + A^{*2}$$

$$\left(1 - k^2 \times u_{\mathrm{rel}}^2(w)\right)A^{\#2} - \left(2A^* + \frac{k^2 w}{t_g}\right)A^{\#} = 0$$

The lower detection limit is obtained by solving the quadratic equation.

$$A^{\#} = \frac{2 \times A^* + \frac{k^2 \times w}{t_g}}{1 - k^2 \times u_{\mathrm{rel}}^2(w)} \qquad \text{(D. 11)}$$

$A^{\#}$: Lower detection limit (Bq or Bq/kg, Bq/L, etc.)
k : Inclusion factor, $k = k_{1-\alpha} = k_{1-\beta}$
$k_{1-\alpha}$: Coefficient for avoiding type I errors (false positive)
$k_{1-\beta}$: Coefficient for avoiding type II errors (false negative)

In general, $\alpha = \beta = 0.05$ (5%)
(Inclusion factor $k_{1-\alpha} = k_{1-\beta} \approx 1.645$) is adopted.

$u_{rel}(w)$: Relative standard uncertainty in the conversion factor w
Relative standard uncertainty combining all measurement uncertainties excluding the uncertainty associated with measurement counting

D.2 Lower detection limit using the Kaiser method

In the sample measurement, when the sample does not contain the target radionuclide at all, the background counting rate distributes as shown in Figure D.5. In this case, when the counting rate is r_n and the uncertainty due to counting statistics is σ, $r_{DL} = k\sigma$ holds true, and the lower detection limit is defined as the detectable limit of the counting rate.

$$r_{DL} = k\sigma \qquad \text{(D.12)}$$

where r_{DL} : Detectable limit of the counting rate
σ : Uncertainty due to counting statistics
k : Inclusion factor (generally set as 3)

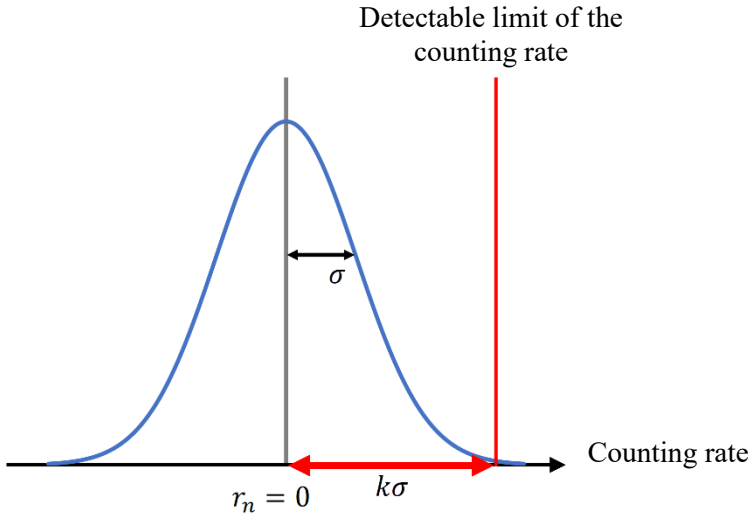


Figure D.5 Schematic diagram of the lower detection limit using the Kaiser method

Now, the following definitions are adopted.

r_g : Sample counting rate (cps)
r_0 : Background counting rate (cps)
t_g : Sample measurement time (sec)
t_0 : Background measurement time (sec)

In this case, r_{DL} and σ are expressed by the following equations.

$$r_{DL} = r_g - r_0 \qquad \text{(D. 13)}$$

$$\sigma^2 = \frac{r_g}{t_g} + \frac{r_0}{t_0} \qquad \text{(D. 14)}$$

Equation (D.13) is converted to $r_g = r_{DL} + r_0$, and is substituted together with equation (D.14) into equation (D.12). Then, the quadratic equation is solved for r_{DL} to obtain the detectable limit of the counting rate.

$$r_{DL} = \frac{\frac{k^2}{t_g} + k\sqrt{(\frac{k}{t_g})^2 + 4r_0(\frac{1}{t_g} + \frac{1}{t_0})}}{2} \qquad \text{(D. 15)}$$

The detectable limit of the counting rate is converted to the radioactivity or radioactive concentration, and that is adopted as the lower detection limit.

D.3 Lower detection limit calculation example

This section shows a calculation example of the lower detection limit using the Kaiser method and ISO 11929 using the following measurement conditions.

○ Measurement conditions

Assay sample	: Ash
Analysis method	: Ion exchange method
Sample quantity W_s	:15.01 g
Measurement time t_g and t_0	: 60 minutes
Chemical recovery Y_{Sr}	:81.2%
Counting efficiency ε	:27.1%
Uncertainty in weighing u_{rel}(*weighting*)	:0.043%
Uncertainty associated with the carrier u_{rel} (*carrier*)	:0.13%
Uncertainty in recovery u_{rel} (*yield*)	:3.7%
Uncertainty associated with measurement device calibration u_{rel} (*calibration*)	:3.2%

Decay correction is not conducted.

Sample and background counts: See Table D.1.

Table D.1 Sample and background counts

Number of measurements	BG1 count	Sample count	BG2 Count
1	11	437	7

The background counting rate r_0(cpm) and the sample counting rate r_g(cpm) are as follows.

$$r_0 = \frac{11+7}{60\times 2} = 0.15$$

$$r_g = \frac{437}{60} = 7.28$$

Therefore, the net counting rate r_n(cpm) and its uncertainty $u(r_n)$ are as follows.

$$r_n = (r_g - r_0) = 7.28 - 0.15 = 7.13$$

$$u(r_n) = \sqrt{\frac{r_g}{t_g}+\frac{r_0}{t_0}} = \sqrt{\frac{7.28}{60}+\frac{0.15}{60\times 2}} = 0.35$$

Next, the conversion coefficient w and the uncertainty $u(w)$ are calculated to obtain the relative standard uncertainty $u_{rel}(w)$.

$$w = \frac{1}{\varepsilon \times Y_{Sr} \times W_s} = \frac{1}{\frac{27.1}{100}\times\frac{81.2}{100}\times\frac{15.01}{1000}} = 303$$

$$u(w) = w\sqrt{u_{rel}^2(weighting)+u_{rel}^2(carrier)+u_{rel}^2(yield)+u_{rel}^2(calibration)}$$

$$= 303\times\sqrt{\left(\frac{0.043}{100}\right)^2+\left(\frac{0.13}{100}\right)^2+\left(\frac{3.7}{100}\right)^2+\left(\frac{3.2}{100}\right)^2} = 15$$

$$u_{\mathrm{rel}}(w) = \frac{u(w)}{w} = \frac{15}{303} = 0.050$$

Next, the radioactivity concentration A (Bq/kg ash) and the combined standard uncertainty $u(A)$ are calculated[*375].

$$A = \frac{r_{\mathrm{n}} \times w}{60} = \frac{7.13 \times 303}{60} = 36.0$$

$$u(A) = A \times \sqrt{\left(\frac{u(r_{\mathrm{n}})}{r_{\mathrm{n}}}\right)^2 + \left(\frac{u(w)}{w}\right)^2} = 36 \times \sqrt{\left(\frac{0.35}{7.13}\right)^2 + \left(\frac{15}{303}\right)^2} = 2.5$$

Then, the lower detection limit is calculated using the Kaiser method. In the calculation, the detectable counting rate r_{DL}(cpm) is obtained using equation (D.15).

$$r_{DL} = \frac{\frac{k^2}{t_{\mathrm{g}}} + k\sqrt{(\frac{k}{t_{\mathrm{g}}})^2 + 4r_0(\frac{1}{t_{\mathrm{g}}} + \frac{1}{t_0})}}{2}$$

$$= \frac{\frac{3^2}{60} + 3\sqrt{(\frac{3}{60})^2 + 4 \times 0.15 \times (\frac{1}{60} + \frac{1}{60 \times 2})}}{2} = 0.273$$

Lastly, the value is converted to radioactive concentration by using the sample quantity and counting efficiency to obtain the lower detection limit$A^{\#}_{\mathrm{Kaiser}}$ [Bq/kg ash].

$$A^{\#}_{\mathrm{Kaiser}} = \frac{r_{DL}}{W_{\mathrm{s}} \times \varepsilon \times Y_{\mathrm{Sr}} \times 60} = \frac{0.273}{\frac{15.01}{1000} \times \frac{27.1}{100} \times \frac{81.2}{100} \times 60} = 1.378$$

However, the lower detection limit according to ISO 11929 $A^{\#}_{\mathrm{ISO11929}}$(Bq/kg ash) is calculated using equations (D.9) and (D.11) together with the decision threshold.

$$A^{*} = k_{1-\alpha} \times w \times \sqrt{\frac{N_0}{t_0 \times 60 \times t_{\mathrm{g}} \times 60} + \frac{N_0}{t_0 \times 60 \times t_0 \times 60}}$$

$$= 1.645 \times 303 \times \sqrt{\frac{11+7}{(60 \times 2 \times 60) \times (60 \times 60)} + \frac{11+7}{(60 \times 2 \times 60) \times (60 \times 2 \times 60)}}$$

$$= 0.5083$$

$$A^{\#}_{\mathrm{ISO11929}} = \frac{2 \times A^{*} + \frac{k^2 \times w}{t_{\mathrm{g}} \times 60}}{1 - k^2 \times u^2_{\mathrm{rel}}(w)} = \frac{2 \times 0.5083 + \frac{1.645^2 \times 303}{60 \times 60}}{1 - 1.645^2 \times 0.04911^2} = 1.252$$

Table D.2 summarizes the above results. For convenience of calculation, half-life correction was not performed for the sample measurement.

[*375] When the unit of the obtained counting rate is cpm, convert it to radioactivity (Bq) by dividing it by 60.

Table D.2 Lower detection limit by parameter and calculation method

Parameter	Kaiser method	ISO 11929
Radioactivity concentration A (Bq/kg ash)	36.0	
Combined standard uncertainty $u(A)$ (Bq/kg ash)	2.5	
Decision threshold A^* (Bq/kg ash)	1.378	0.5083
Lower detection limit $A^\#$ (Bq/kg ash)		1.252
Ratio to Kaiser (%)		-10

Information

Information A Solid Phase Extraction

This paper introduces solid phase extraction, which has been used as a pretreatment for LSC measurement and mass spectrometry in analytical and research institutes in Japan and overseas. The filler used for solid phase extraction is a resin functionalized with 4,4'(5')-di-tert-butyldicyclohexano-18-crown-6 (crown ether) 1-octanol (hereinafter referred to as "crown ether resin"), and it selectively extracts Sr. Although other alkali metals and alkaline earth metals (especially Ca) have considerably lower affinity to resin than Sr, samples with a high concentration of alkali metals and alkaline earth metals may cause a decrease in the recovery of Sr.[*376] Depending on the sample[*377], these elements should be separated by ion exchange or other method before solid phase extraction. Furthermore, soil samples contain a large amount of Pb, and the affinity of Pb for resin is quite high. Therefore, Pb is separated by the ion exchange method .or other method before solid phase extraction.

Section A.1 in this paper describes a standard pretreatment method using cartridge type[*378] columns packed with crown ether resin. In addition, Section A.2 describes the pretreatment of water samples reported by Tazoe[*379] et al. as a practical example of the combination of ion exchange and solid phase extraction.

For details on the method for measuring samples pretreated by solid phase extraction, refer to the Series of Environmental Radioactivity Measuring Methods (No.23) "AI-LSC Radionuclide Analysis using Liquid Scintillation Counter" and Infomation B Mass Spectrometry in this measuring method.

A.1 Analytical procedure

1) Insert columns with funnels[*380] into the suction manifold to which a pump is connected, and set containers (beakers or centrifuge tubes) to receive waste liquid at the outlets of the columns (Figure A.1).
2) Place 8 mol/L nitric acid of an amount three times the amount of resin, into the funnel, and add suction with the pump while adjusting the flow rate to 1 to 2 mL/min to clean the column. Discard the effluent.

[*376] For details on the impact of alkali metal and alkaline earth metal concentrations on the recovery, refer to the manufacturer's technical data. For example, recovery by crown ether resins made by a manufacturer declines when Ca and Na exceed 0.5 mol/L and K exceeds 0.01 mol/L.

[*377] For instance, milk samples contain high levels of Ca, which may reduce recovery.

[*378] Shows how to operate packed columns. When a column is filled with a purchased resin, the liquid running through it flows down naturally.

[*379] H.Tazoe et al.: Rapid Chemical Separation Protocol for Optimized ^{90}Sr Determination, Radiation Environment and Medicine, 11 (1), 7 – 15 (2022)

[*380] The resin amount of the cartridge to be used should be adjusted depending on the sample. For example, one cartridge column filled with 2 mL resin is used for the analysis of inland water samples (1 L); one cartridge column filled with 2 mL resin and one cartridge column filled with 1 mL resin are connected in series for the analysis of ash samples (200 g animal sample (muscle, etc.), 100 mL milk sample, and 5 g to 10 g plant sample): and two cartridge columns filled with 2 mL resin are connected in series for the analysis of seawater samples (1 L to 2 L) and soil samples (50 g).

3) Dry and solidify the pretreated[*381] sample solution on a hot plate.
4) Dissolve the dehydrated sample in about 10 mL of 8 mol/L nitric acid to make the assay sample.
5) Place the assay sample into the funnel, and apply suction with the pump while adjusting the flow rate to 1 to 2 mL/min to allow Sr be adsorbed in the column. Discard the effluent.
6) Pass 5 mL of 8 mol/L nitric acid through the column while cleaning the funnel with it. Discard the effluent.
7) Place 8 mol/L nitric acid of an amount three times the amount of resin, into the funnel, and apply suction with the pump while adjusting the flow rate to 1 to 2 mL/min to clean the column. Discard the effluent.
8) Place a mixed solution of 3 mol/L nitric acid and 0.05 mol/L oxalic acid of an amount three times the amount of resin into the funnel, and apply suction with the pump while adjusting the flow rate to 1 to 2 mL/min to wash away impurities. Discard the effluent.
9) Place 8 mol/L nitric acid of an amount three times the amount of resin, into the funnel, and apply suction with the pump while adjusting the flow rate to 1 to 2 mL/min to wash away impurities. Discard the effluent.
10) Replace the receiving container with a new one.
11) Place 0.05 mol/L nitric acid of an amount five times the amount of resin into the funnel, and apply suction with the pump while adjusting the flow rate to 1 mL/min to elute and recover Sr.



Information figure A.1 Schematic diagram of a solid phase extraction system

[*381] Perform the pretreatment (evaporation and ion exchange) described for this measuring method. You may also use the manufacturer's standard protocol. For the analysis sample quantity, refer to the manufacturer's standard protocol.

A.2 Practical example of the combination of ion exchange and solid phase extraction

1) Fill the polyethylene column with 5 mL of cation exchange resin.
2) Pass 30 mL of 8 mol/L nitric acid through the column using a tube pump. Discard the effluent.
3) Pass 30 mL of 0.05 mol/L nitric acid through the column using a tube pump. Discard the effluent.
4) Pass 20 mL of 4 mol/L nitric acid through the column using a tube pump. Discard the effluent.
5) Add nitric acid to a 1 L water sample until a concentration of 0.1 mol/L is achieved, and pass the entire amount through the column using a Peristar pump at a flow rate of 60 mL/min. Discard the effluent.
6) Pass 30 mL of 3 mol/L nitric acid through the column using a tube pump to elute and recover Sr.
7) Fill the polyethylene column with 2 mL of crown ether resin.
8) Pass 10 mL of 0.05 mol/L nitric acid through the column. Discard the effluent.
9) Pass 10 mL of 4 mol/L nitric acid through the column. Discard the effluent.
10) Pass the entire amount of solution recovered in 6) through the column. Discard the effluent.
11) Pass 10 mL of 8 mol/L nitric acid through the column to clean it. Discard the effluent.
12) Pass 10 mL of 0.05 mol/L nitric acid through the column to elute and recover Sr.

Information B Mass Spectrometry

For the analysis of ^{90}Sr, radiochemical analysis has been used for a long time. After radionuclides are separated in radiochemical analysis, the beta rays of ^{90}Sr and its progeny nuclide ^{90}Y are measured using radiation detectors such as an LBC, LSC, or a low background beta-ray scintillation spectrometer. The reason for this is that the lower detection limit can be lowered to environmental levels by using a large number of samples for analysis. However, this analytical method uses a large amount of reagents, causing a high environmental load, and the load on the analysis worker is also high because of the long work process and dangerous reagents used. The analysis period for analysis of environmental level samples is at least one month and generally about two months because it includes a period during which samples are left to stand until ^{90}Sr and ^{90}Y reach a state of radioactive equilibrium.

Radionuclide analysis using mass spectrometry is considered to be more advantageous for radionuclides with a long half-life (around 100 years or more) than radiochemical analysis, and has been widely used as a general method for measuring the isotopic ratio of U, etc. For the above reason, in measurement of environmental level samples for the analysis of ^{90}Sr, whose half-life is about 30 years, mass spectrometry has been considered to be less advantageous than radiochemical analysis. However, studies on ^{90}Sr analysis using mass spectrometry have been progressing since the Fukushima Daiichi Nuclear Power Plant accident. Such efforts have led to the development of analytical methods that require small samples and a shorter analysis period, and this can also be applied to environmental level samples, and now it is possible to select a method according to the purpose of the analysis.

The following section introduces the analysis of ^{90}Sr using mass spectrometry.

B.1 Quantitative analysis of ^{90}Sr using an inductively coupled plasma mass spectrometer

The inductively coupled plasma mass spectrometer (hereinafter referred to as "ICP-MS") is a device that introduces ions generated by inductively coupled plasma into the mass analyzer to perform qualitative and quantitative analysis based on the mass numbers of the ions and the ion intensities. This device enables quantification in low concentrations with an order of ppt and is widely used for elemental analysis together with ICP-AES. A device that adopts a quadrupole mass spectrometer (hereinafter referred to as "QMS") as its mass analyzer is widely available, and most of the studies on ^{90}Sr analysis by ICP-MS also use QMS as the mass analyzer. Some studies use a triple quadrupole ICP-MS with two quadrupole mass filters to improve the abundance sensitivity. In general, the “abundance sensitivity ” represents the ability to discriminate the abundance of a chemical species or isotope with a mass number different by 1 from that of a chemical species or isotope with a mass number of m. Abundance sensitivity in the analysis of ^{90}Sr using mass spectrometry is often expressed as follows since there is the problem that the peak of mass number 88 overlaps the peak of mass number 90.

Abundance sensitivity = Ion strength of mass number 90/Ion strength of mass number 88

That is, it shows the ^{90}Sr/^{88}Sr ratio and is a numerical value representing the ability to distinguish ^{90}Sr, which has a high abundance, from ^{88}Sr, which has a low abundance. The smaller the numerical value, the higher the ability to discriminate between ^{88}Sr and ^{90}Sr.

The approximate abundance ratios of Sr isotopes are as follows: ^{88}Sr (83%), ^{86}Sr (10%), and ^{87}Sr (7%). The isotopic ratio of ^{90}Sr, a radioisotope, against ^{88}Sr varies depending on the abundance of ^{90}Sr and the amount of stable Sr in the sample. To analyze ^{90}Sr in environmental samples with a high reliability, an abundance sensitivity of ^{90}Sr/^{88}Sr ratio ranging from 1×10^{-8} to 1×10^{-12} is required.

The following section introduces an outline of ^{90}Sr analysis using ICP-MS.

In analyzing ^{90}Sr in an environmental sample using ICP-MS, two points should be noted. The first point is the interference due to zirconium 90 (^{90}Zr), an isobar, which is contained in the sample. The second point is the increase in the background due to peak tailing of ^{88}Sr, which has the highest isotopic abundance among stable isotopes of Sr. These factors lower analytical precision and increase the lower detection limit.

Methods that have been developed to address the interference by ^{90}Zr, an isobar in the sample, are chemical separation using crown ether resin, and oxidation of $^{90}Zr^+$ in a collision/reaction cell with oxygen.

The increase in the background due to the peak tailing of ^{88}Sr can be mitigated by using an ICP-MS system with good abundance sensitivity. Although ICP-QMS cannot analyze ^{90}Sr in soil samples with a $^{90}Sr/^{88}Sr$ ratio of less than 10^{-9}, the use of triple quadrupole ICP-MS enables the analysis of ultra-trace amounts of ^{90}Sr in soil samples with a ^{90}Sr-^{88}Sr ratio of about 5×10^{-12}[*382].

The following section introduces a study on ^{90}Sr analysis using ICP-MS.

In order to establish rapid and daily radiation monitoring of ^{90}Sr in water samples, Tazoe et al.[*383] developed an analytical method that uses cation exchange resin and crown ether resin for pretreatment. With this method, the sample solution pretreated by the combination of ion exchange and solid phase extraction, which was introduced in Information A, is directly measured using the ICP-MS. In the pretreatment process, a sample of about 1 L is enriched to 10 mL, and ^{90}Zr, an elements causing isobaric interference, and $^{74}Ge^{16}O^+$ and $^{74}Se^{16}O^+$, elements causing polyatomic interference, are removed. This method is applicable to water samples containing up to 300 mg of calcium carbonate, and can satisfy a lower detection limit of 5 Bq/kg by using a triple quadrupole ICP-MS with an abundance sensitivity of 1×10^{-10} or less. This is also a rapid analytical method that requires only 3 hours for the processing of 10 samples.

Yanagisawa[*384] et al. developed a method that couples isotope dilution (hereinafter referred to as "ID") with online solid-phase extraction-ICP-QMS. Since the ICP-QMS system is equipped with solid-phase extraction columns for Sr isolation, this method can quantify ^{90}Sr in water samples by simply adding a ^{86}Sr concentrated solution to the sample and introducing it into the device. With this method, a certain amount of a ^{86}Sr concentrated solution with known isotopic ratios (^{84}Sr : ^{86}Sr : ^{87}Sr : ^{88}Sr = 0.0008 : 0.9702 : 0.0078 : 0.0212) is added to a sample, and the ^{88}Sr-^{86}Sr and $^{90}Sr/^{88}Sr$ ratios in the sample before and after the addition of the ^{86}Sr concentrated solution are measured to quantify ^{90}Sr. Using the ID method, this method does not require the preparation of a calibration curve, thus shortening the time for the analysis process. In addition, although it takes a long time to analyze a number of samples, this method has an advantage of being unlikely to be affected by signal drift over time because the sample spiked with ^{86}Sr concentrated solution also functions as an internal standard. When the sample quantity is 10 mL, the lower detection limit is 5.6 Bq/L, and the abundance sensitivity ($^{90}Sr/^{88}Sr$) is 6×10^{-9}.

[*382] T. Ohno et al.: Determination of strontium 90 in environmental samples by triple quadrupole ICP-MS and its application to Fukushima soil samples, Journal of Analytical Atomic Spectrometry, 33,1081 – 1085 (2018)

[*383] H.Tazoe et al.: Rapid Chemical Separation Protocol for Optimized ^{90}Sr Determination, Radiation Environment and Medicine, 11 (1), 7 – 15 (2022)

[*384] K.Yanagisawa et al.:Online solid-phase extraction-inductive coupled plasma-quadrupole mass spectrometric quantification of ^{90}Sr using $^{88}Sr/^{86}Sr$ isotope dilution method, Talanta, 244,123442(2022).

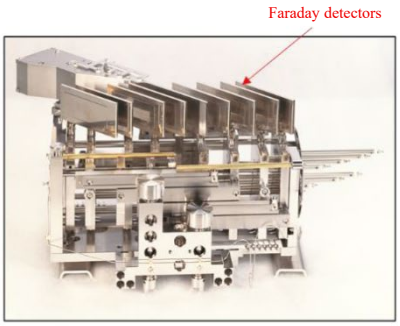
B.2 Quantification of ^{90}Sr by TIMS

The thermal ionization mass spectrometer (hereinafter referred to as "TIMS") is a system for measuring isotopic ratios, and it has been used to measure the isotopic ratios of various metal elements i.e. as a tool for analyzing U enrichment (^{235}U/^{238}U) in nuclear fuel in the nuclear field, and for determining age in the geochemistry field. It is also used for measuring isotopic ratios between ^{87}Sr, a radiogenic Sr isotope, and ^{86}Sr, a stable Sr isotope.

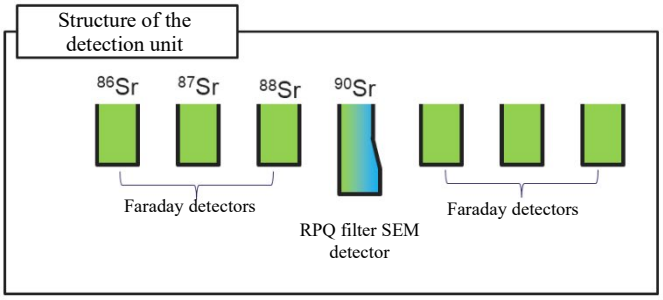
The main components of a TIMS system are the ionizing unit, mass separation unit, and detection unit. The ionizing unit adopts the thermal ionization method, and ionizes the sample applied on a rhenium or tungsten filament by heating the filament with an electric current. The acceleration voltage at the ion source is maintained at about 10 kV, and the generated ion beams are introduced into the mass separator. The application of the sample to the filament is an operation in which about 1 μL of the sample is dropped onto a filament with a width of several millimeters, which requires a skilled operator.

The mass separation unit has a fan-shaped magnetic field, and by selecting an appropriate magnetic field strength, it separates ion beams according to the mass-to-charge ratio and introduces them into the detector.

The detection unit has several Faraday cup detectors (6 or 8 pieces) with a high signal output stability. When the Faraday cup detectors[*385] receive the ion beam with the target mass-to-charge ratio, a faint signal is generated. The faint signal is amplified by an amplifier and output as an electric signal. If a secondary electron multiplier tube detector[*386] (hereinafter referred to as “SEM detector”) with a higher sensitivity than Faraday detectors is installed in the center, the analysis sensitivity is further improved. However, if a Daly detector[*386], which is less degradable and shows little change in the background and multiplication factor over time is installed in the center as well, the precision of analysis will be further improved. That is, this method can perform coincidence counting using multiple detectors, and due to being equipped with detectors with different sensitivities, such as the combination of Faraday detectors and SEM detectors, it can achieve a wide dynamic range. Furthermore, if a quadrupole energy mass filter (hereinafter referred to as an RPQ filter) is installed in the upstream of the SEM detector, the scattered ion beams that are the cause of the ^{88}Sr peak tailing can be removed, and the abundance sensitivity will be improved as a result. In the ^{90}Sr analysis, the use of a TIMS system equipped with an RPQ filter and SEM detector will enable the sensitivity of the analysis to be increased.



Information Figure B.1 External view of detection unit



Information Figure B.2 Schematic diagram of the structure of the detection unit

[*385] 山本康太, 浅沼尚: 特集: 定量分析・同位体分析のためのイオン検出器. ファラデーイオン検出器, Journal of the Mass Spectrometry Society of Japan, 69 (6), 160-165 (2021)

[*386] 山下修司,平田岳史: 特集: 定量分析・同位体分析のためのイオン検出器. 電子増倍管とデイリー検出器, Journal of the Mass Spectrometry Society of Japan, 69 (6), 166-170 (2021)

In analyzing ^{90}Sr in an environmental sample using TIMS, two points should be noted. As is the case with the quantification of ^{90}Sr using ICP-MS, attention should be paid to the interference by the isobar ^{90}Zr and the increase in the background due to the peak tailing of ^{88}Sr.

To address the interference by ^{90}Zr, an isobar contained in the sample, as in the analysis using ICP-MS, chemical separation with solid phase extraction resin using N,N,N',N'-tetra-n-octyldiglycolamide, which adsorbs Zr, and chemical separation with crown ether resin, which selectively adsorbs Sr, are used. There is also a method unique to TIMS which controls the temperature of the filament coated with the sample to suppress the ionization of Zr and thereby reduces the interference. This method heats the filament at a temperature at which only Sr is ionized and evaporated from the filament while taking advantage of the fact that the ionization potential of Sr (5.7 eV) is lower than that of Zr (6.6 eV), thereby reducing the interference by Zr. The evaporation temperature of Sr is about 1500°C to 1600°C, and the evaporation temperature of Zr is about 1790°C. Therefore, when the filament heating temperature is controlled to below 1650°C, the interference by Zr can be reduced[*387].

The increase in the background due to the peak tailing of ^{88}Sr can be mitigated by using a TIMS system with improved abundance sensitivity due to the adoption of an RPQ filter.

The following introduces a study on ^{90}Sr analysis using TIMS.

In the IAEA proficiency test, Kavasi[*388] et al. reported values obtained using TIMS that were good results, thus validating the ^{90}Sr analytical method that uses TIMS.

Aoki[*389] et al. developed a technology that quantifies ultra-trace amounts of ^{90}Sr in microscale samples using TIMS. This method uses ID-TE-TIMS, which combines ID and total evaporation (hereinafter referred to as "TE"), and the major feature of the method is that ^{90}Sr can be quantified only by TIMS. In addition to the filament heating temperature control and the installation of the RPQ filter, they made a correction to compensate for the dark current of the SEM detector, and successfully quantified 9.8×10^{-19} g (5.0 μBq) of ^{90}Sr in 0 to 300 mg/L stable Sr. Although it depends on the stable Sr concentration in 1 μL of sample, the lower detection limit ranged from 6.15×10^{-20} to 3.90×10^{-19} g (from 0.31 to 1.95 μBq). This method was validated through the spike and recovery test and a comparison with the official method (radiochemical analysis). The results of both tests were good, showing that it is a highly reliable analytical method. In addition, they also conducted the analysis of ^{90}Sr in biological samples, which had been considered necessary but not performed because radiochemical analysis could not be used due to the small amount of samples, and succeeded in determining ^{90}Sr in the teeth of rats.

[*387] C. Ito et al.: Isotope Dilution – Total Evaporation – Thermal Ionization Mass Spectrometric Direct Determination of Radioactive Strontium-90 in Microdrop Samples. Analytical Chemistry, 92, 16058−16065 (2020)

[*388] N.Kavasi et al.:Accurate and precise determination of ^{90}Sr at femtogram level in IAEA proficiency test using Thermal Ionization Mass Spectrometry, Scientific Reports, 9,16532(2019)

[*389] J.Aoki et al.: Direct Quantification of Attogram Levels of Strontium-90 in Microscale Biosamples Using Isotope Dilution-Thermal Ionization Mass Spectrometry Assisted by Quadrupole Energy Filtering, Analytical Chemistry, 95, 4932−4939 (2023)

Appendixes

Appendix A　Nuclear Data and Decay Diagrams of ^{90}Sr and Others

Nuclear data extracted from a reliable nuclear data collection should be used for the calculation of radioactivity, and the nuclear data used should be indicated together with the analysis results.

In this measuring method, the nuclear data of ^{89}Sr, ^{90}Sr and ^{90}Y was obtained from the Evaluated Nuclear Structure Data File (ENSDF) managed by the National Nuclear Data Center (NNDC) of the United States, and Annex A.1 shows the data. Figure A.1 shows the decay diagrams of ^{89}Sr and ^{90}Sr.

Annex A.1　Nuclear data

Nuclide	Half-life	Maximum energy of beta rays (MeV)	Decay pattern (Release rate (%))
^{89}Sr	50.563 ± 0.025 days	1.5004 ± 0.0024	β^- (100)
^{90}Sr	28.91 ± 0.03 years	0.5459 ± 0.0014	β^- (100)
^{90}Y	64.05 ± 0.05 hours	2.2785 ± 0.0016	β^- (100)

*　The source of the nuclear data is ENSDF (as of February 2025). The half-life and beta ray maximum energy values are described with uncertainties.

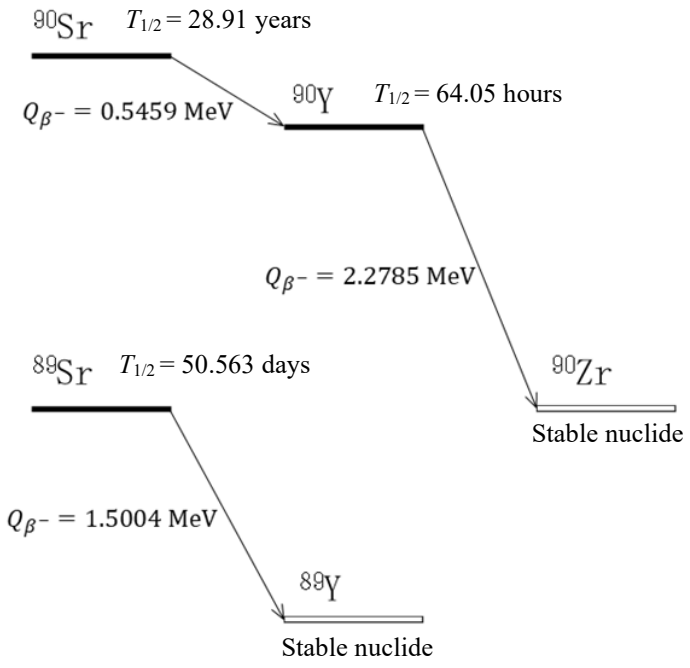


Figure A.1　Decay diagrams of ^{89}Sr and ^{90}Sr

Appendix B Explanation of Terms

Term	Explanation
Ion exchange resin	An insoluble synthetic resin with an ion exchange group. It is used for chemically separating metal ions in the sample solution by taking advantage of their property of strongly attaching to or detaching from the ion exchange resin depending on the chemical properties of the sample solution.
Primary estimate	A mathematically estimated value with the highest probability. It corresponds to "primary estimate" in ISO11929.
^{90}Y (Yttrium 90)	A radioactive nuclide formed when ^{90}Sr undergoes beta minus decay. It is a radioisotope that emits only beta rays. It decays by beta minus emission to ^{90}Zr and has a half-life of 64.05 hours. The maximum beta ray energy is 2.2785 MeV.
Liquid scintillation counter	Abbreviated to LSC. This device uses a photomultiplier tube to count the light pulses (Cerenkov light, fluorescence of scintillators excited by radiation) of the sample to be measured. It is widely used for measuring low-energy beta ray emitting nuclides because it can detect alpha and beta rays emitted from a sample in all solid angle (4π) directions and there is no self-absorption by the sample. There are models that have a lead shield around the detector and adopt a incidence circuit to reduce the background counting rate and models that are equipped with a guard counter to remove the impact of external radiation.
Coprecipitation	Process in which a substance of a concentration at which it is not precipitated on its own by a precipitant due to the amount being very small is made to precipitate together with a carrier. By adding another substance (carrier) that is precipitated by the same precipitant, they are precipitated together. This method is used to chemically separate ultra-trace amounts of substances like radioactive isotopes.
Counting rate	Count value per unit of time. Usually expressed in count per minute (cpm) or count per second (cps).
Atomic absorption spectrometry	Abbreviated to AAS. When an element to be measured is atomized by spraying a sample into the frame or heating it in a graphite furnace, and light with a wavelength specific to the element to be measured is made to penetrate through the atomized sample, the ground state atoms absorb the light and transition to an excited state. This method measures the concentration of the element from the absorption rate of the light (absorbance).
Conditioning	To control devices, reagents, conditions, etc. to the optimum state before starting measurement or analysis operations, etc. In this analysis book, conditioning refers to a kind of pretreatment performed before using ion exchange resin for the purpose of removing impurities from the resin.
Scavenging	In chemical analysis, this refers to the operation of separating and removing unnecessary elements. A carrier added for separation and removal is called a scavenger.

Term	Explanation
	With this measuring method, the operation to remove ^{90}Y formed from ^{90}Sr in the sample is called scavenging. This operation is conducted to precisely calculate the proportion of ^{90}Y formed during the time between the scavenging and the milking.
Sr (Strontium)	Strontium has 4 stable isotopes (mass numbers of 84, 86, 87 and 88) and more than 20 radioisotopes. The most important radioisotopes for environmental radiation monitoring are ^{89}Sr and ^{90}Sr.
^{89}Sr (Strontium 89) ^{90}Sr (Strontium 90)	They are produced by nuclear fission of uranium and plutonium during the operation of nuclear reactors, and nuclear tests, etc. Both nuclides emit beta rays when undergoing beta minus decay. The half-life of ^{89}Sr is 50.563 days, the maximum energy of beta rays is 1.5004 MeV, and it decays to ^{89}Y by emitting beta rays. The half-life of ^{90}Sr is 28.91 years, the maximum energy of beta rays is 0.5459 MeV, and it decays to ^{90}Y by emitting beta rays. Using the radiative equilibrium relationship between ^{90}Sr and ^{90}Y, beta rays from ^{90}Y are measured to quantify ^{90}Sr.
Carrier	A large amount of substance added to precipitate radioactive nuclides that are very small in atomic number in the precipitation method, etc. In this analysis book, carrier refers to a stable isotope added to separate or extract radionuclides. The Sr carrier added with this measuring method also has the purpose of calculating the recovery for correcting the amount of radioactive Sr lost in the analysis process.
Low background 2π gas flow counter	Abbreviated to LBC. This device efficiently measures alpha and beta rays. The device has a lead shield around its detectors and performs anti-coincidence counting using two detectors, a center counter and a guard counter, thereby reduces the background noise. As the detectors, GM counters are mainly used, and in such cases, Q gas is used as the counter gas. In recent years, various detectors have been developed including proportional counter tubes using PR gas as the counter gas, a type that combines different detectors, and detectors that are not the gas flow-type. In actual operations, be sure to check the gas type, applied voltage, etc. with the specifications of the detector to be used.
Fuming nitric acid	Brown concentrated nitric acid solution that contains nitrogen dioxide. It is called fuming nitric acid because it generates red or white fumes. It is more irritating and oxidizing than general nitric acid, and therefore is more dangerous and must be handled very carefully.
Radiochemical analysis	An analytical method in which radionuclides in a sample are separated and purified, and their radioactivity is measured to determine the amounts of the radionuclides.
Radioactive equilibrium	A state in which the relationship between the radioactivity of a parent radionuclide and that of a progeny nuclide produced by the decay of the radionuclide reach equilibrium at a certain rate over

Term	Explanation
	time. The state is classified into permanent or transient equilibrium according to the ratio of their half-lives. The equilibrium relationship between ^{90}Sr and ^{90}Y is a permanent one, so when a sufficient time, in terms of the half-life of the progeny nuclide ^{90}Y, elapses, the radioactivity of the two nuclides becomes equal.
Milking	An operation to repeatedly separate and extract a short-lived progeny nuclide from a parent nuclide with a long half-life while they are in a radioactive equilibrium state. Even after the progeny nuclide is separated from the parent nuclide, the parent nuclide continues to generate the progeny nuclide. Therefore, the progeny nuclide can be separated and extracted repeatedly as many times as possible as time passes. This operation is likened to milking dairy cows so is called milking.
Inductively coupled plasma atomic/optical emission spectroscopy	Abbreviated to ICP-AES or ICP-OES. This emission spectroscopic analysis uses inductively coupled plasma (ICP) as the light source. The sample solution is atomized and introduced into the argon plasma, and the light emitted by the element when it is excited and then returns to the ground state is dispersed. The element is qualitatively determined by the wavelength and quantitatively determined by the radiant intensity.
Elution	In this document, this refers to the operation of eluting a target component from the components adsorbed in an ion exchange resin. A sample solution is passed through a column filled with ion exchange resin to exchange and adsorb various ions, and then a solvent called an eluent is passed through the column. The ions in the eluent exchange (adsorption and desorption) on the exchange group, and the target component is separated.
Elution curve	Curve in which the amount of eluent is plotted on the horizontal axis and the concentration of the compounds (or ions) eluted is plotted on the vertical axis for the substances separated by the column. The curve is used to determine whether two or more compounds (or ions) are sufficiently separated.
Beta minus (β^-) decay	A process in which one nuclide converts into another by releasing an electron (e^-). Since one neutron in the atomic nucleus converts to a proton, the mass number of the nuclide remains the same, but the atomic number increases by one. The energy of the released electron is distributed continuously over a range. The energy of beta rays is not constant because of the distribution of the energy and neutrino that are released at the same time.
Beta ray	A type of radiation, also called a high-speed electron, that is released from a nucleus during beta decay. There are two types: negatively charged beta minus (β^-) rays and positively charged beta plus (β^+) rays.

Term	Explanation
	The energy is distributed continuously from zero to the maximum energy specific to the nuclide.
PR gas	A mixture of argon (90%) and methane (10%) gases used in measuring devices (proportional counter tubes) for measuring beta rays.
Q gas	A mixture of helium (99%) and isobutane (1%) gases that is used as a counter gas in measuring devices (GM counter tubes) for the analysis of ^{90}Sr.

Appendix C Results of the Cross-check Analysis, etc.

This section shows the result of the cross-check analysis (conversion of the unit of radioactivity from pCi to Bq) performed at the time of the third revision in 1983 and that of the verification test of the ion exchange method improved at the time of the forth revision in 2003.

1. Result of the cross-check analysis (excerpt from revision 3 in 1983)

1.1 Cross check samples

(1) Seawater : About 115 L of seawater was collected on the Pacific Coast, and 580 mL of nitric acid was added to the sample immediately after it was collected. Then the sample was spiked with 20 Bq of ^{90}Sr in the laboratory and mixed with a propeller agitator for about 2 hours. The sample was divided into 20 L portions and distributed to analytical organizations.

(2) Soil : Three soil samples collected at three locations were dried at about 105°C, pulverized, and sieved through a 297 μm sieve. Then they were each thoroughly mixed with a blender and divided into 100 g or 200 g portions and distributed to analytical organizations.

(3) Skim milk : One bag (25 kg) of a commercial product was purchased and incinerated at 500°C in an electric furnace. The obtained ash was pulverized, mixed thoroughly with a V-blender, divided into 50 g portions, and distributed to analytical organizations.

1.2 Participants (analytical organizations)

College of Science and Engineering, Aoyama Gakuin University
Ibaraki Environmental Technology Center (currently Ibaraki Prefecture Environmental Radiation Monitoring Center)
Hydrographic Department, Maritime Safety Agency, Ministry of Transport (currently Hydrographic and Oceanographic Department, Japan Coast Guard, the Ministry of Land, Infrastructure, Transport and Tourism)
Shizuoka Prefectural Environmental Radiation Monitoring Center
Tokai Plant, Power Reactor and Nuclear Fuel Development Corporation
(currently Japan Nuclear Fuel Cycle Engineering Laboratories, Japan Atomic Energy Agency)
Tokai Laboratory, Japan Atomic Energy Research Institute
(currently Nuclear Science Research Institute, Japan Atomic Energy Agency)
Japan Analysis Center (currently Japan Chemical Analysis Center)
Livestock Experiment Station, the Ministry of Agriculture, Forestry and Fisheries
(currently Institute of Livestock and Grassland Science, National Agriculture and Food Research Organization)

1.3 Analysis result

The error (±) of the measured values shows the uncertainty due to counting statistics (counting error) in the radioactivity measurement.

(1) Seawater

Annex C.1 Seawater sample analysis result

Analysis method	Analytical organization	Sample quantity	Measured ^{90}Sr value (Bq/L)
Fuming nitric acid method	g	5 L	0.15 ± 0.011, 0.17 ± 0.011
Oxalate method	b	10 L	0.17 ± 0.004, 0.17 ± 0.004
	e	10 L	0.17 ± 0.007
	g	10 L	0.15 ± 0.004

*In consideration of counting errors, none of the measured values should be rejected.

(2) Soil

Annex C.2 Soil sample analysis result

Analysis method	Analytical organization	Sample quantity	Measured ^{90}Sr value (Bq/kg)		
			Sample A	Sample B	Sample C
Fuming nitric acid method	g	100 g	5.9 ± 0.26 5.5 ± 0.26 5.7 ± 0.26	2.6 ± 0.19 2.7 ± 0.19 2.6 ± 0.19	1.0 ± 0.15 1.0 ± 0.15 1.0 ± 0.15
Ion exchange method	a	100 g	4.6 ± 0.33	3.1 ± 0.22	1.0 ± 0.15
	b	100 g			1.1 ± 0.07 1.1 ± 0.07
	g	100 g	5.8 ± 0.26	2.4 ± 0.19	0.96 ± 0.15
Oxalate method	b	100 g	5.9 ± 0.19 6.0 ± 0.19	3.0 ± 0.15 2.8 ± 0.11	0.96 ± 0.07 1.1 ± 0.07
	e	100 g	5.5 ± 0.52	2.7 ± 0.33	0.96 ± 0.26

Although the number of measured values and the number of repeated measurement values are small, there were no values that should be rejected in terms of the three sigma (3σ) rule, and the results are summarized for each analysis method as shown in Annex C.3.

Annex C.3 Results for soil samples obtained by each analytical method (summary)

	Statistical item		Sample A	Sample B	Sample C
Fuming nitric acid method	n=3	$\bar{X}$	5.7	2.6	1.0
		σ	0.20	0.06	0.02
		CV(%)	3.6	2.2	2.1
Ion exchange method	n=4	$\bar{X}$			1.0
		σ			0.06
		CV(%)			6.0
Oxalate method	n=3	$\bar{X}$	5.8	2.8	1.0
		σ	0.25	0.13	0.09
		CV(%)	4.3	4.6	8.4

(3) Skim milk (ash)

Annex C.4 Skim milk sample analysis results and the results from each analytical method (summary)

Analysis method	Analytical organization	Sample quantity	^{90}Sr (Bq/g ash)	Mean value, etc.	
				By analytical organization	By analytical method
Fuming nitric acid method	g	20 g	0.019 ± 0.0011 0.019 ± 0.0011 0.019 ± 0.0011 0.020 ± 0.0011	n :4 $\bar{X}$:0.019 σ :0.0007 CV(%) :3.7	
Ion exchange method	b	10 g	0.019 ± 0.0011 0.020 ± 0.0015 0.019 ± 0.0011 0.020 ± 0.0015	n :4 $\bar{X}$:0.020 σ :0.0006 CV(%) :3.2	n :13 $\bar{X}$:0.020 σ :0.0017 CV(%) :8.4
	d	10 g	0.020 ± 0.0022 0.017 ± 0.0019 0.023 ± 0.0022 0.019 ± 0.0022 0.021 ± 0.0022	n :5 $\bar{X}$:0.020 σ :0.0021 CV(%) :10.6	
	g	10 g	0.019 ± 0.0026 0.023 ± 0.0030 0.019 ± 0.0019 0.022 ± 0.0022	n :4 $\bar{X}$:0.021 σ :0.0021 CV(%) :9.3	

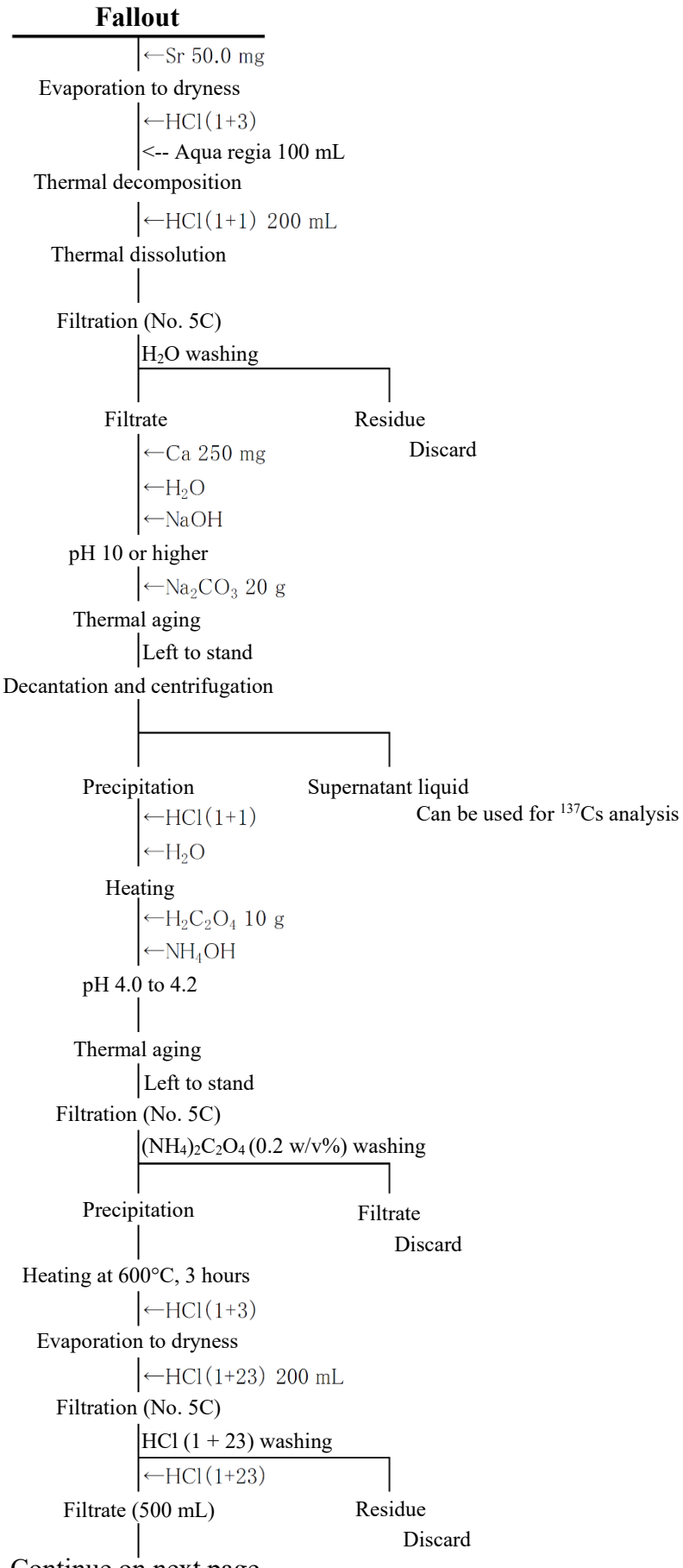
2. Results of the validation of ^{90}Sr determination using this measuring method's ion exchange method (excerpt from revision 4 in 2003)

Two types of samples for process control that are used in regular work at Japan Chemical Analysis Center (ash and soil samples) were used to determine the ^{90}Sr in them. The table below shows the obtained ^{90}Sr values together with the standard values for process control with which the obtained Sr recoveries and ^{90}Sr values should be compared. These standard values are the results obtained by the conventional method that combines the fuming nitric acid method and ion exchange method. The ash sample of 18.5 g was analyzed in order to confirm the Sr recovery in the assay sample containing 5 g of Ca. The analysis results agreed well with the reference value.

Annex C.5 Results in ash and soil process control samples using the ion exchange method

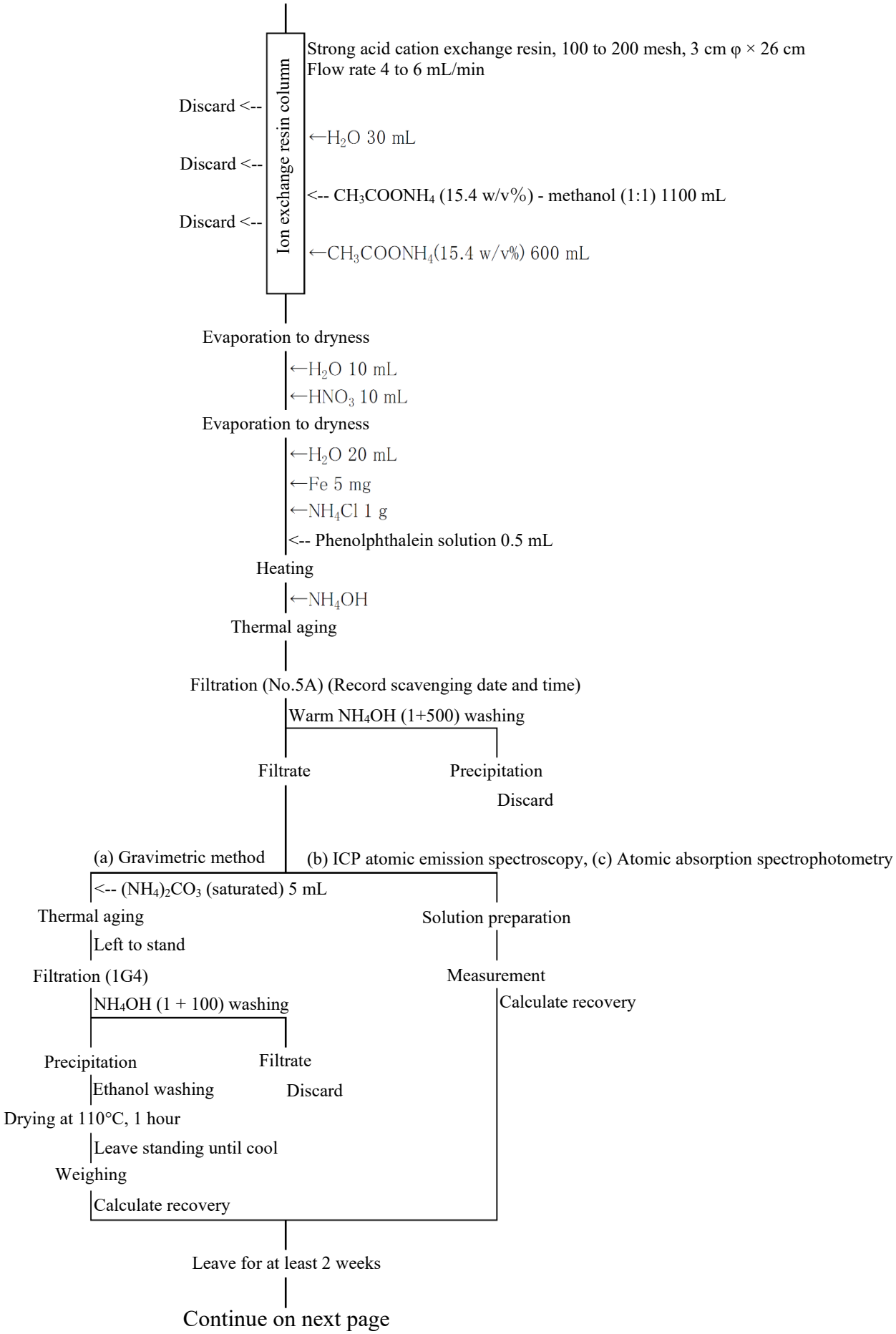
Sample	Sample quantity analyzed (g)	Sr recovery (%)	^{90}Sr concentration (mBq/g ash or dry soil)	
			Analysis result	Process control standard value
Ash	10	97	159 ± 4.6	163 ± 5.0
		98	157 ± 4.6	
		97	164 ± 4.7	
		Mean	160 ± 3.6	
	18.5	82	166 ± 3.7	
		86	159 ± 3.5	
		84	160 ± 3.6	
		Mean	162 ± 3.8	
Soil	100	90	2.0 ± 0.18	2.1 ± 0.17
		93	2.5 ± 0.20	
		91	2.4 ± 0.20	
		Mean	2.3 ± 0.26	

Appendix D Analysis Flows



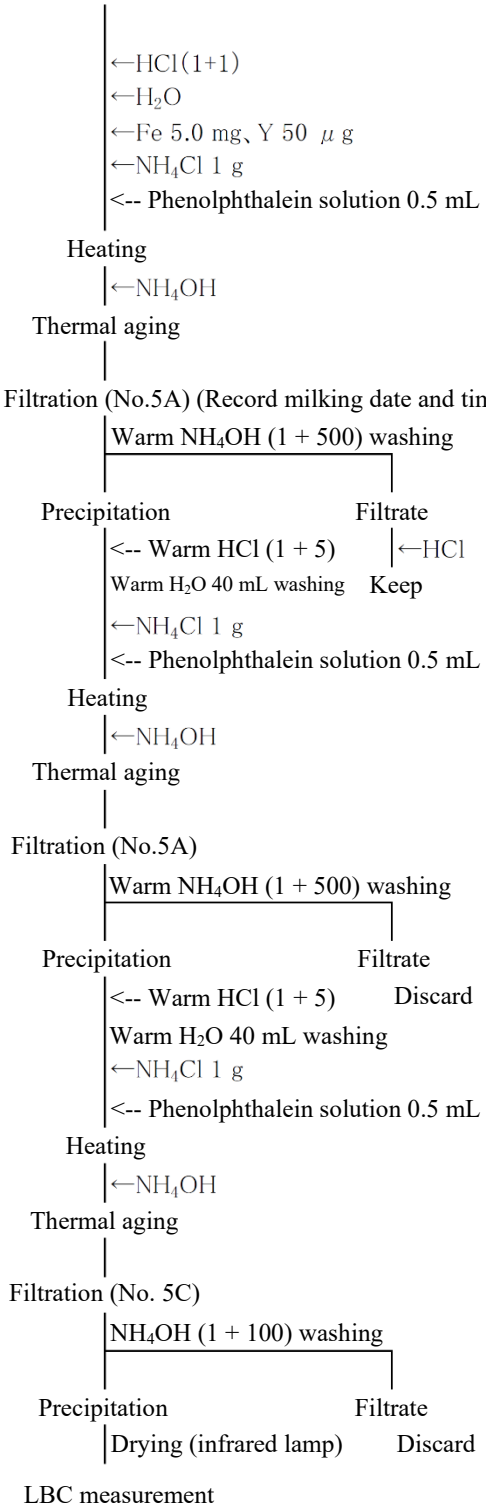
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Fallout (evaporation)—Ion exchange method (1/3)

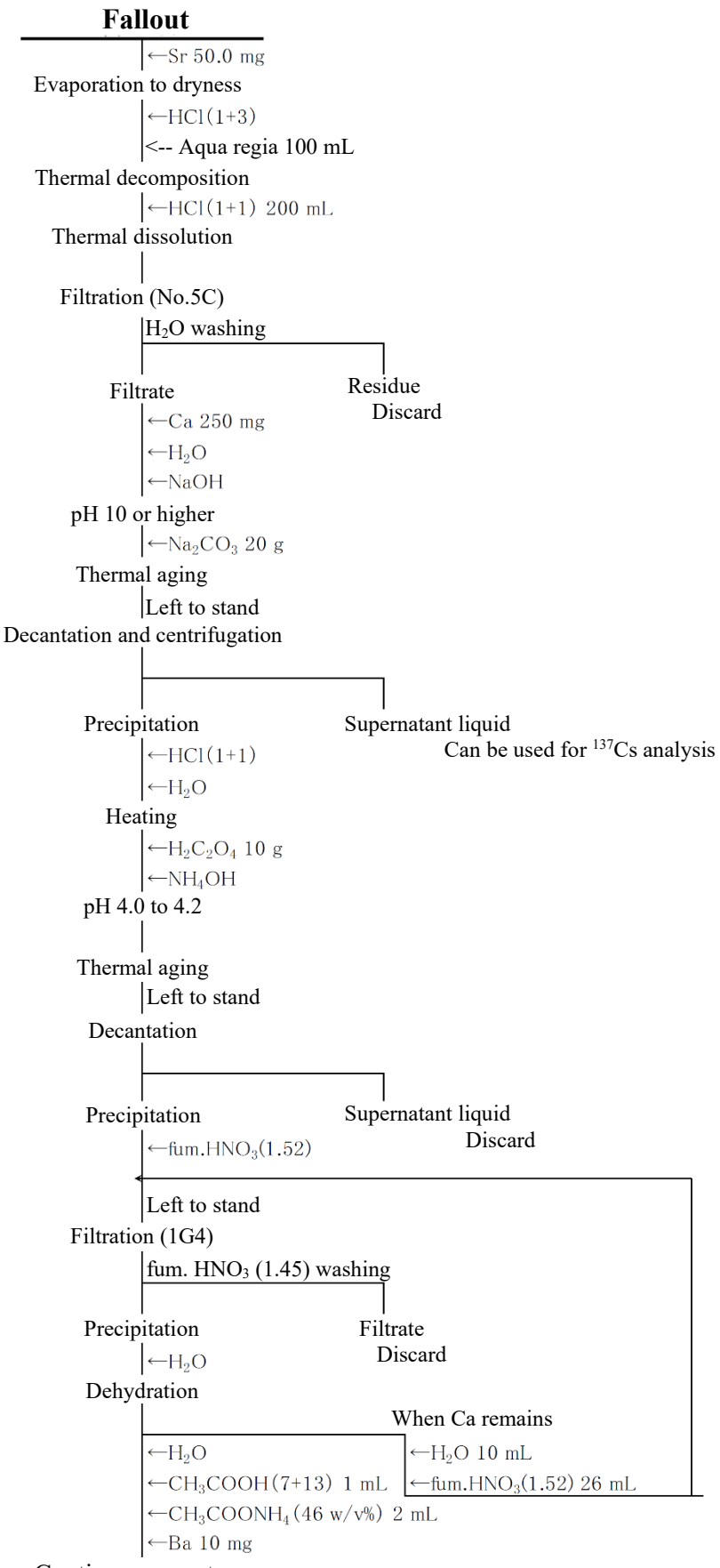


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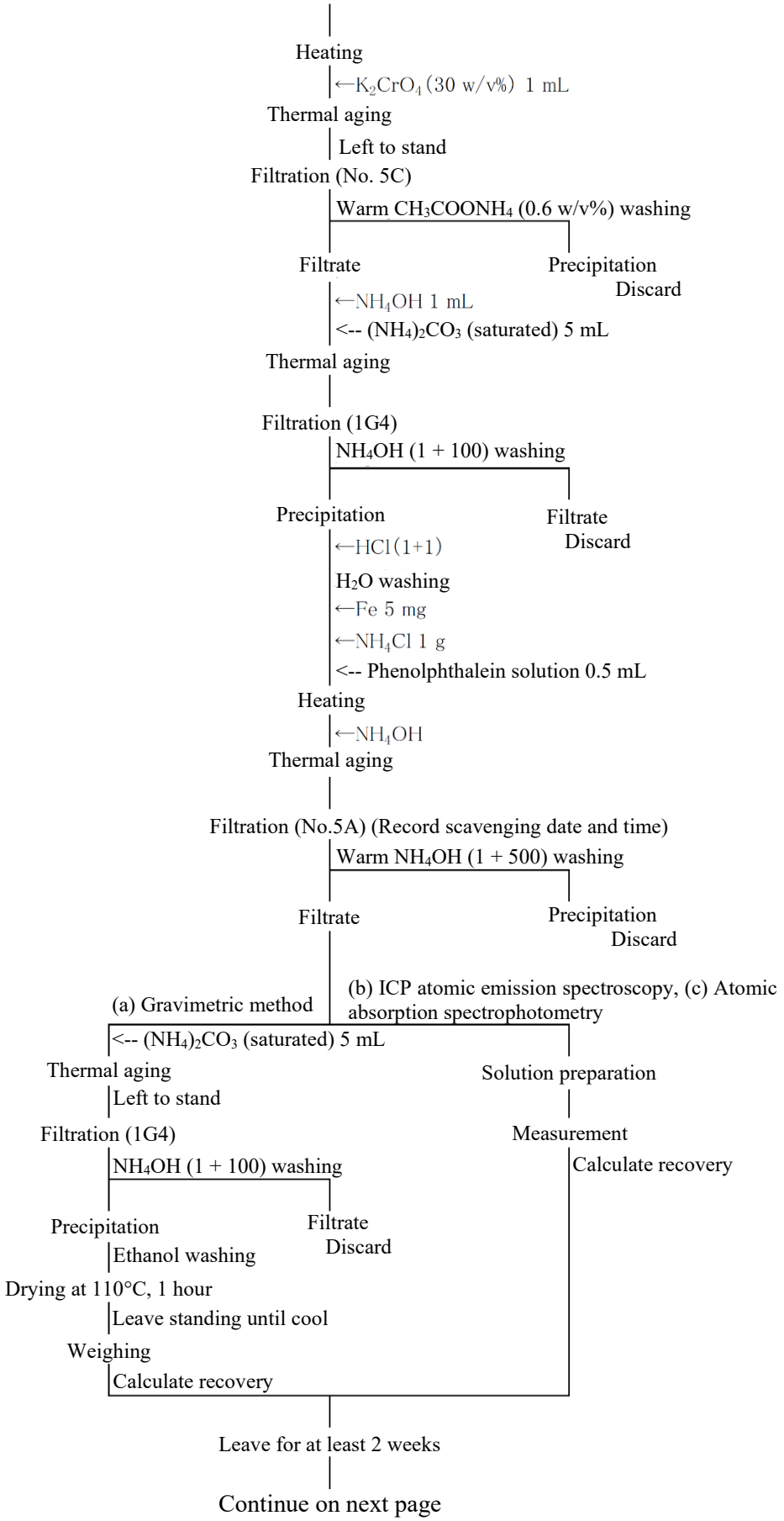
Fallout (evaporation)—Ion exchange method (2/3)



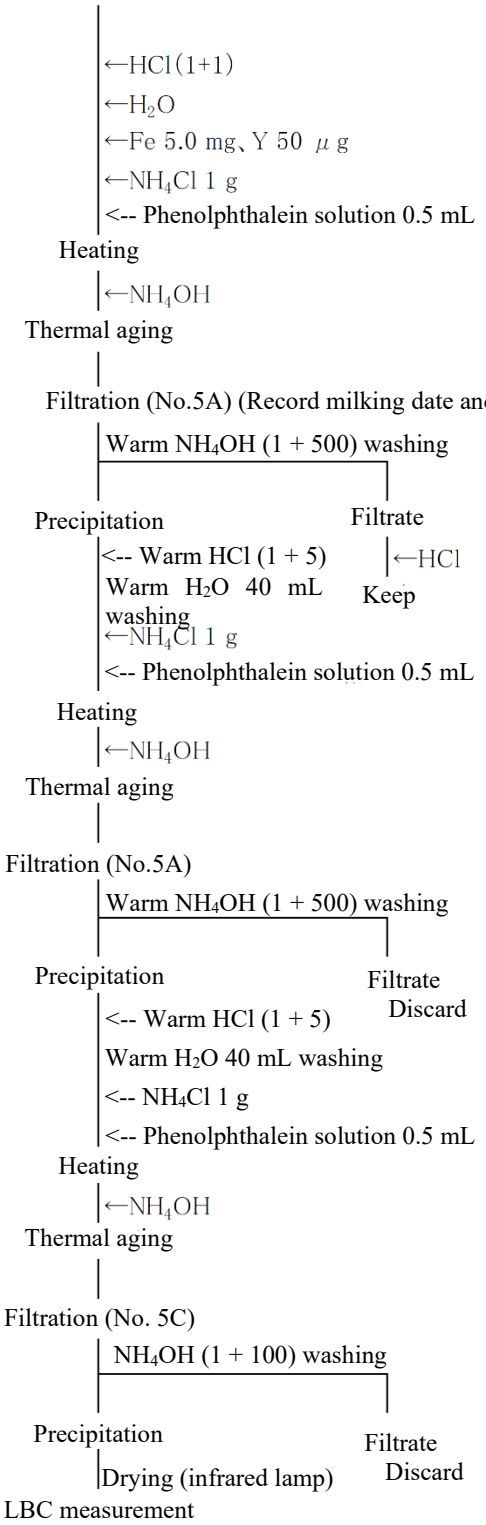
Fallout (evaporation)—Ion exchange method (3/3)



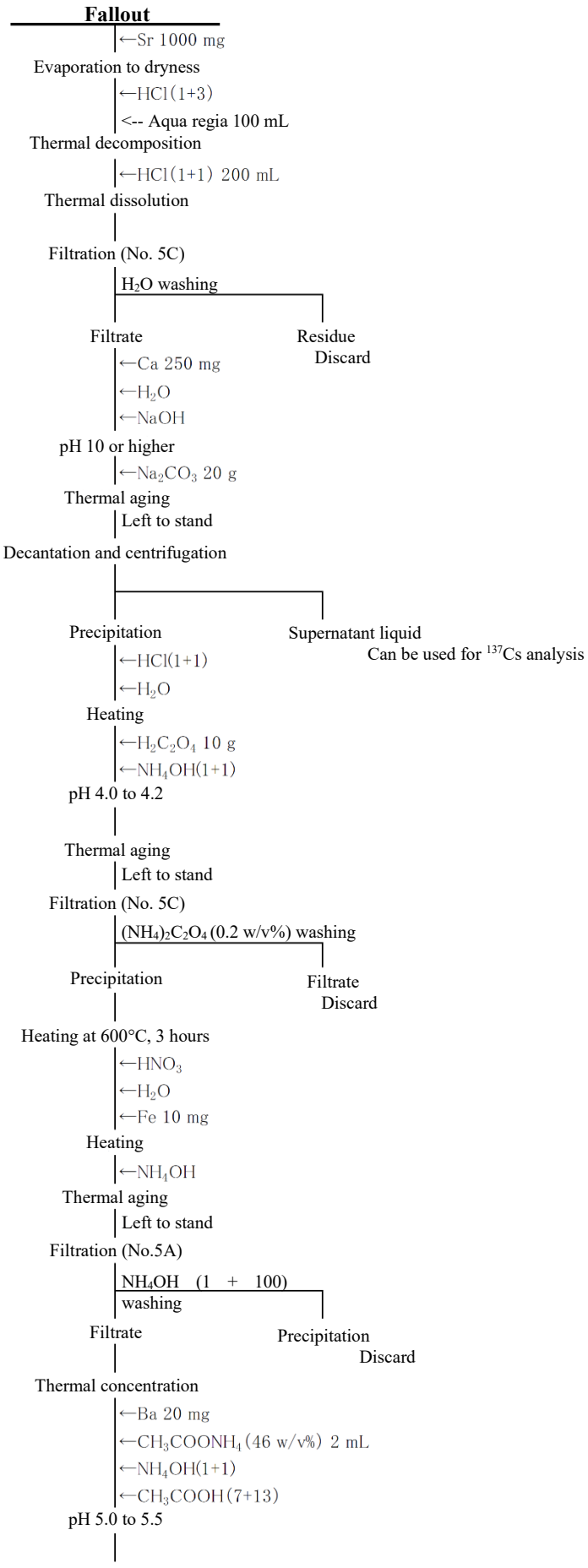
Fallout (evaporation)—Fuming nitric acid method (1/3)



Fallout (evaporation)—Fuming nitric acid method (2/3)

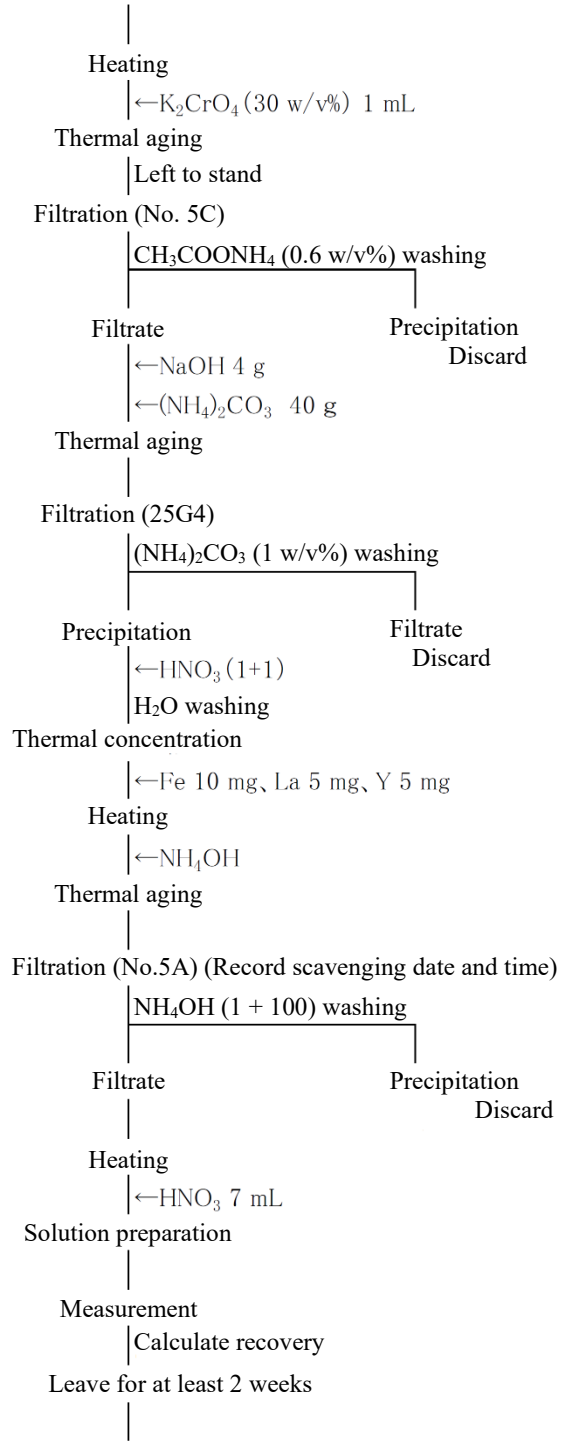


Fallout (evaporation)—Fuming nitric acid method (3/3)



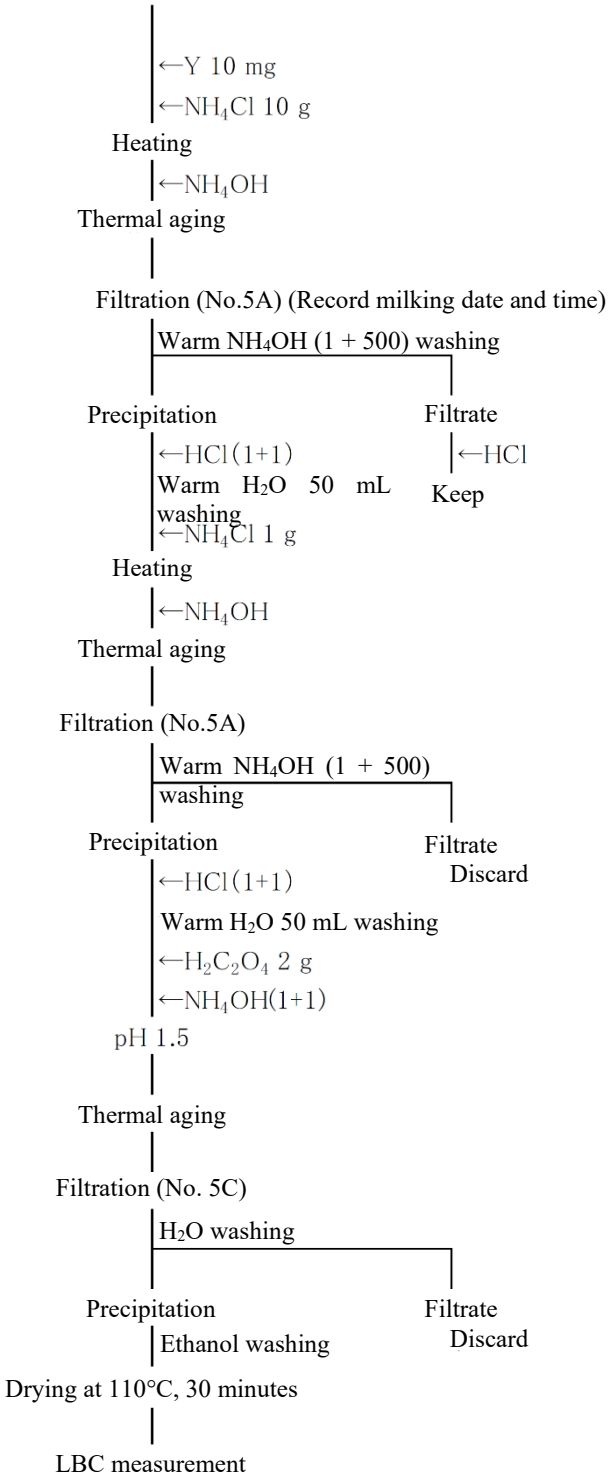
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Fallout (evaporation)—Oxalate method (1/3)

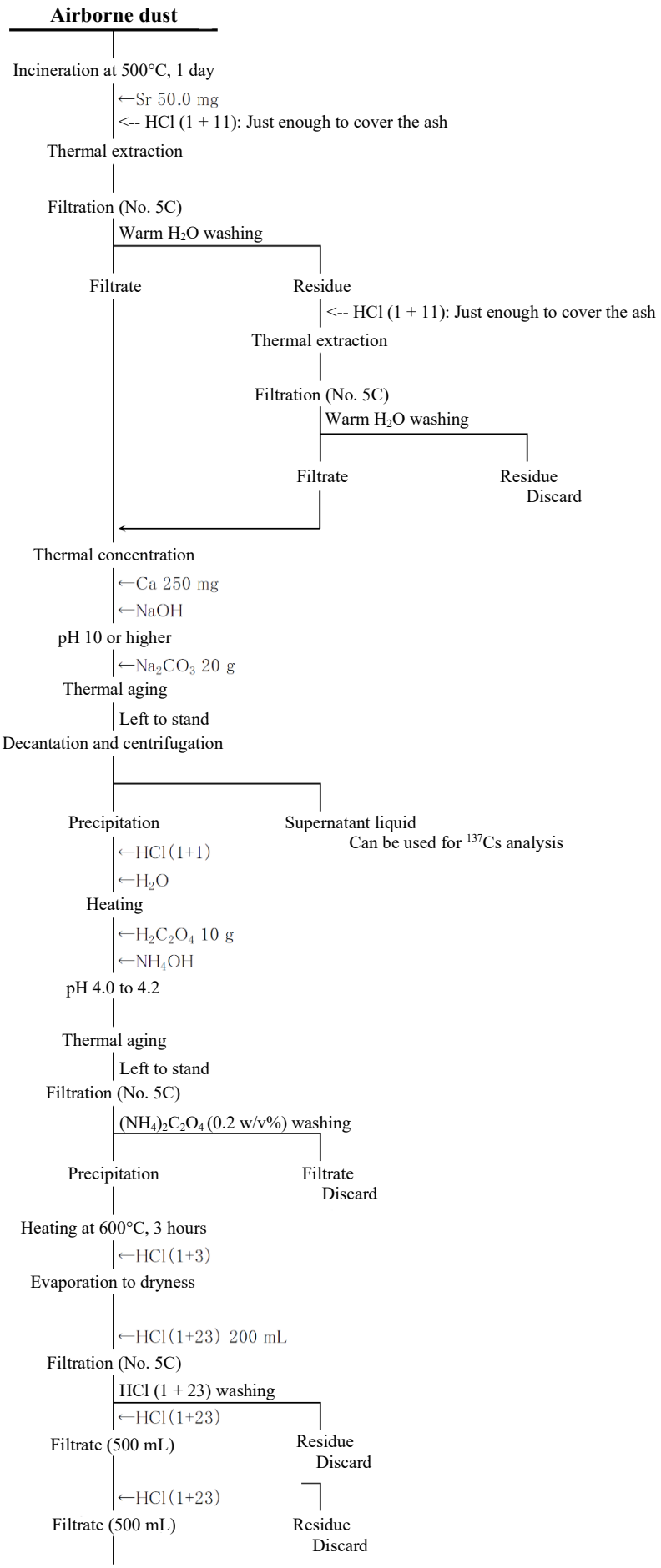


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Fallout (evaporation)—Oxalate method (2/3)

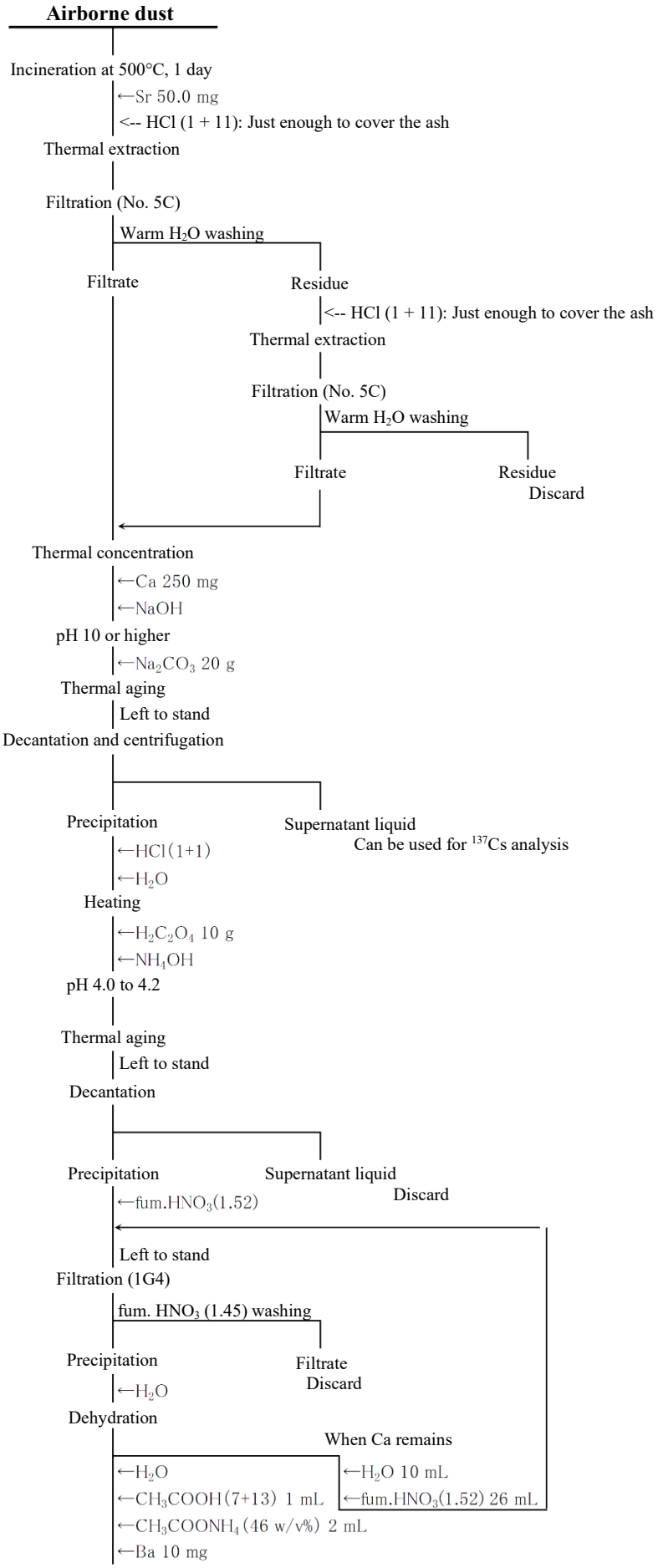


Fallout (evaporation)—Oxalate method (3/3)



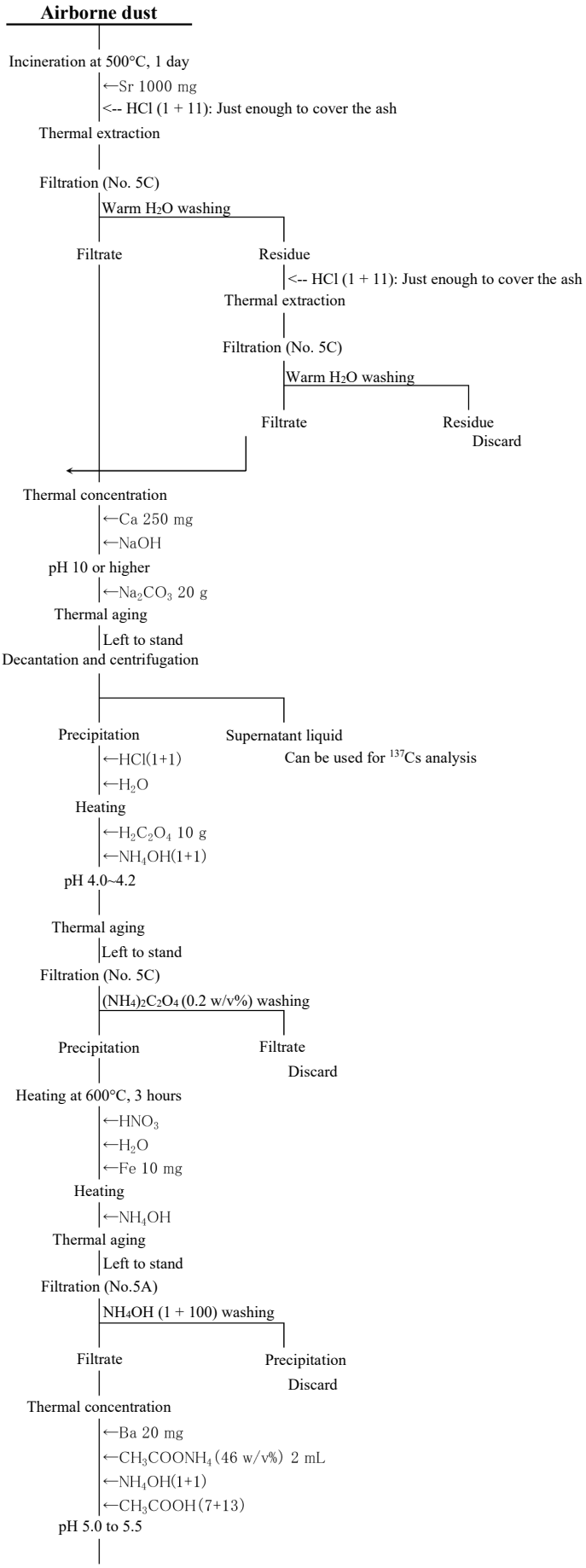
To Fallout (evaporation)—Ion exchange method (2/3)

Airborne dust—Ion exchange method



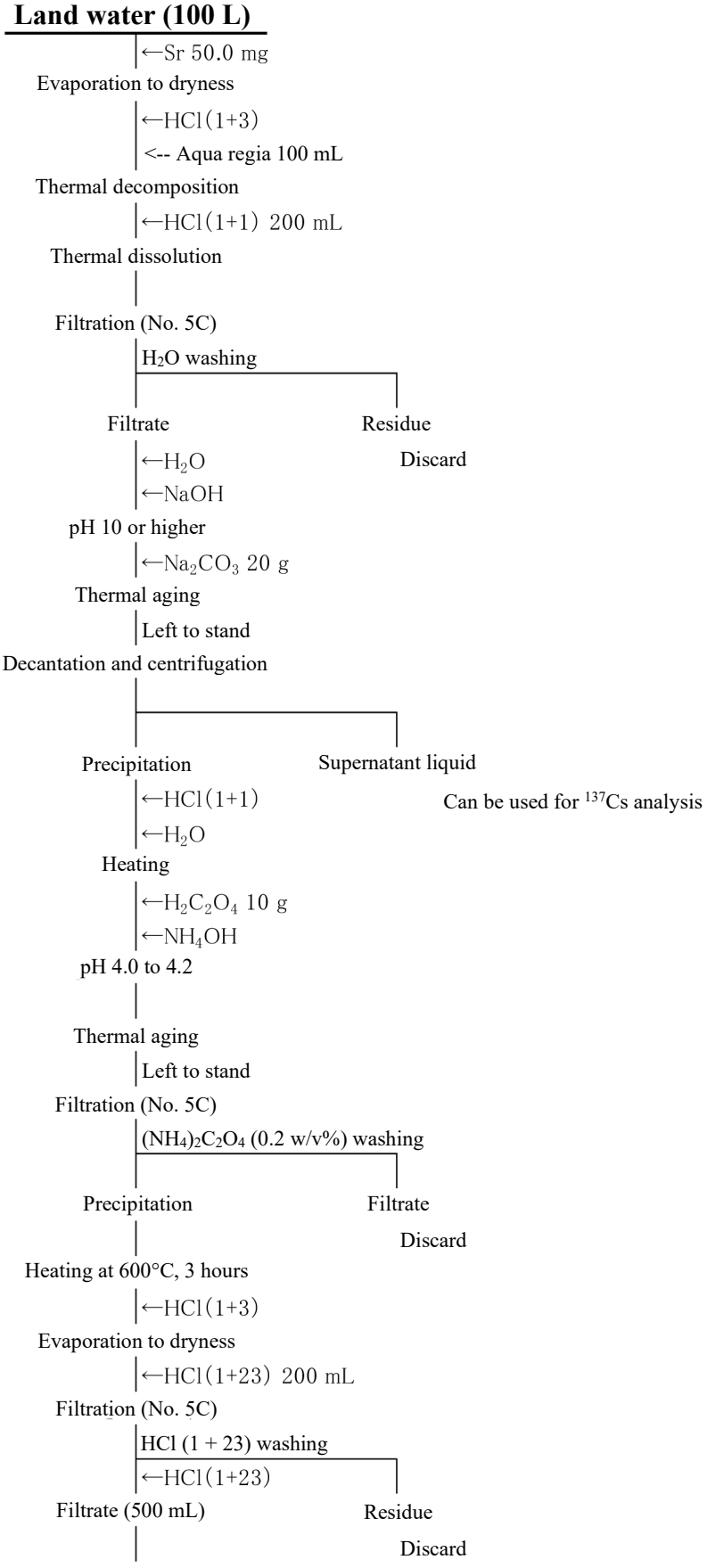
To Fallout (evaporation)—Fuming nitric acid method (2/3)

Airborne dust—Fuming nitric acid method



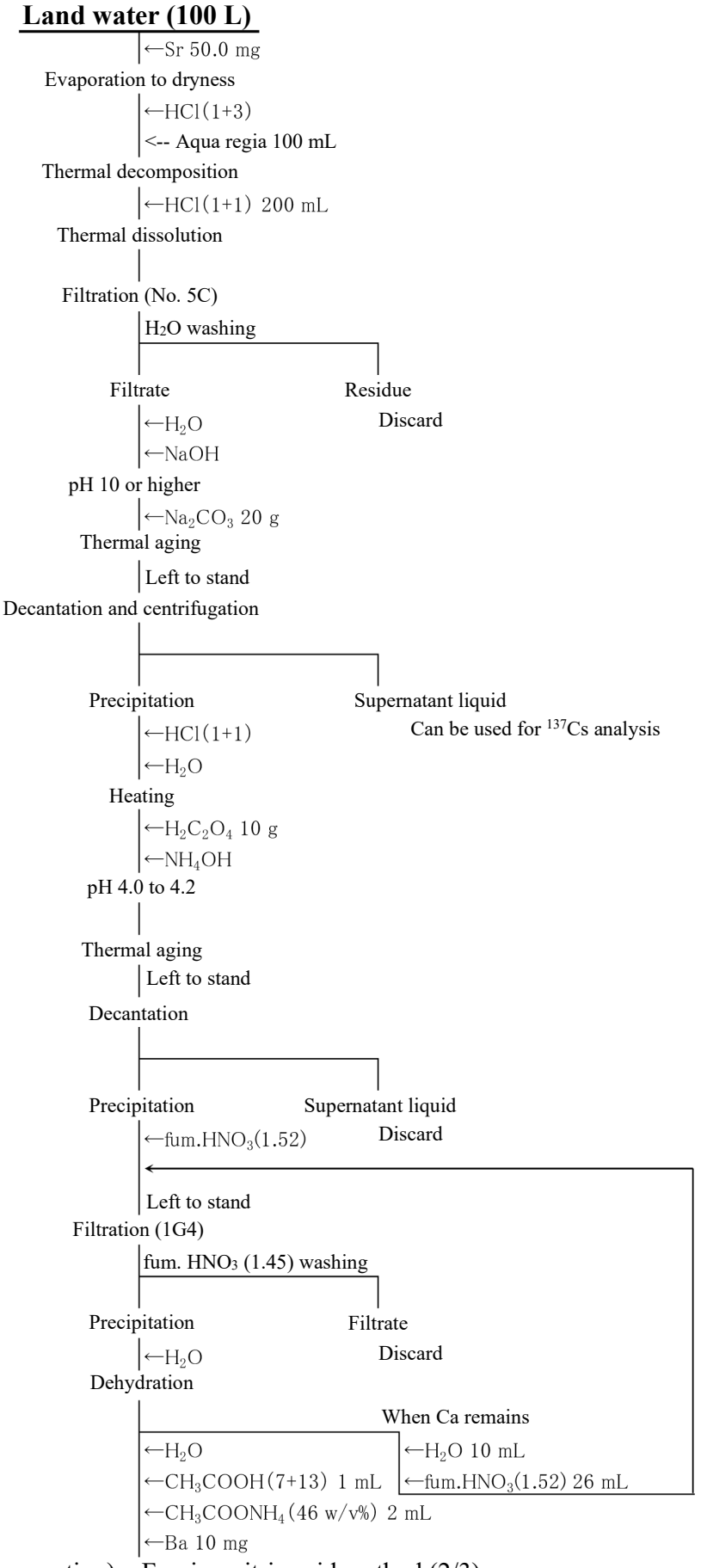
To Fallout (evaporation)—Oxalate method (2/3)

Airborne dust—Oxalate method



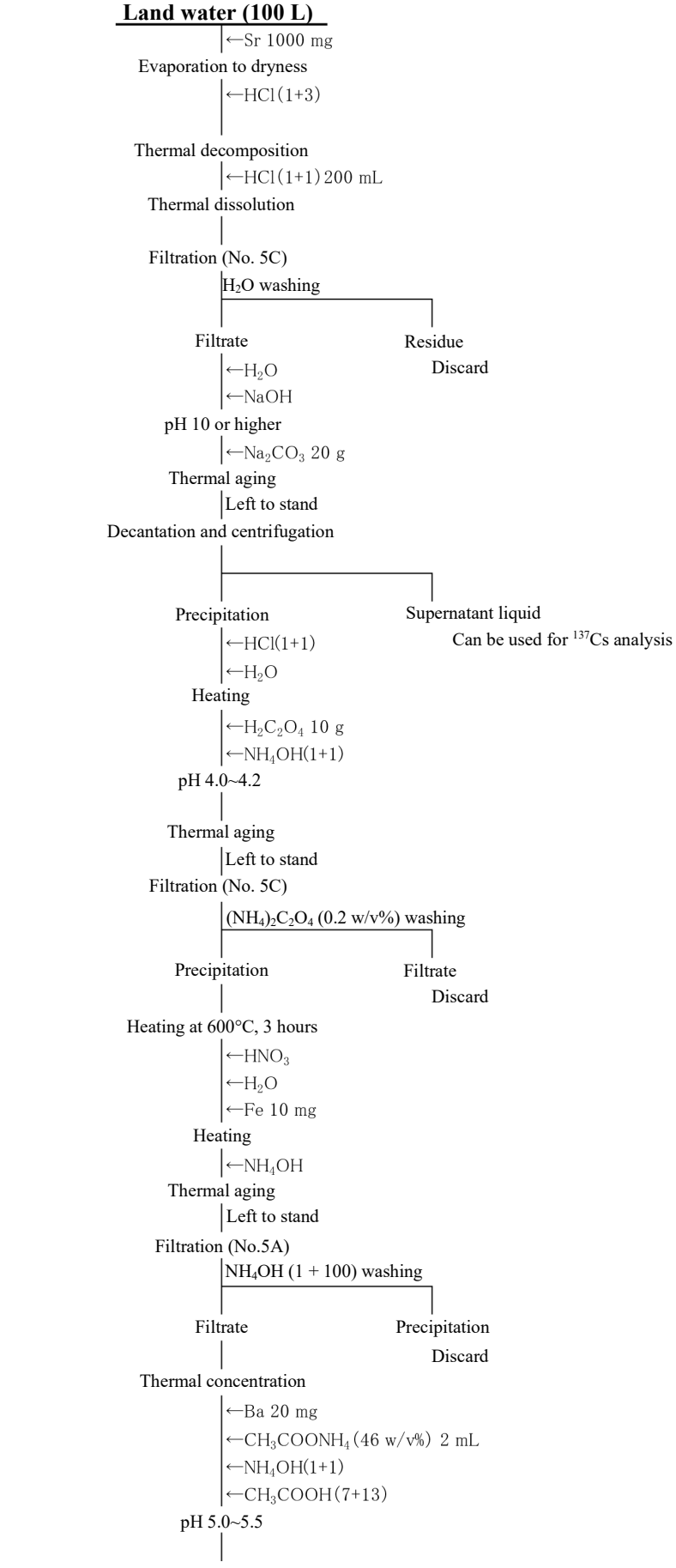
To Fallout (evaporation)—Ion exchange method (2/3)

Land water—Ion exchange method



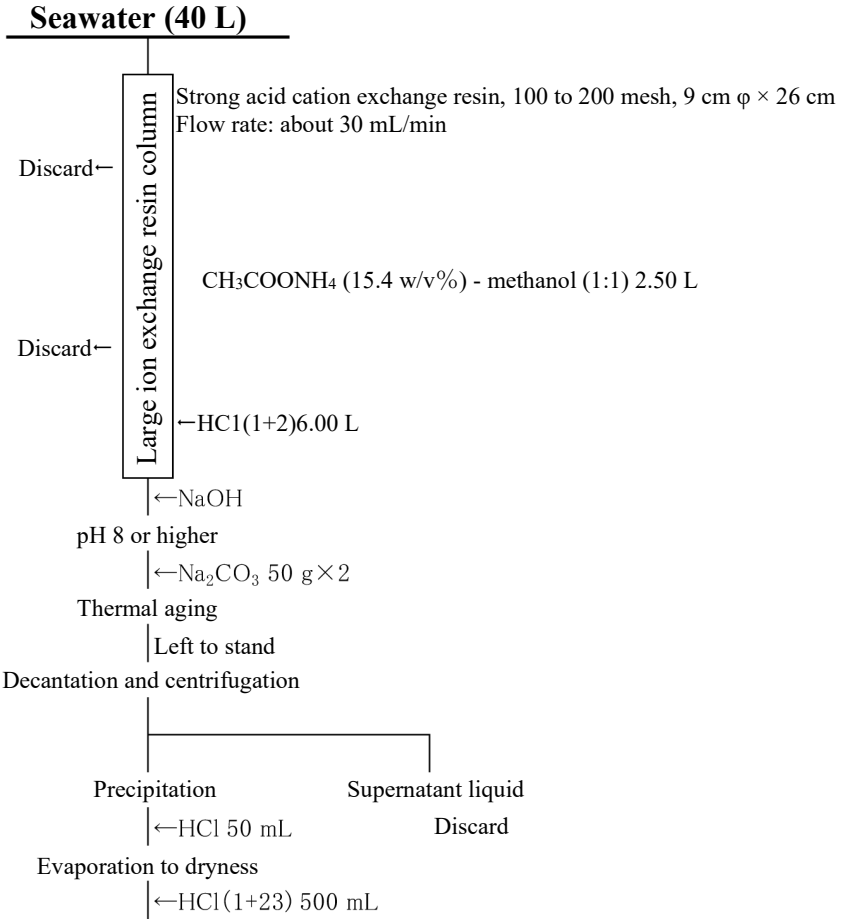
To Fallout (evaporation)—Fuming nitric acid method (2/3)

Land water—Fuming nitric acid method



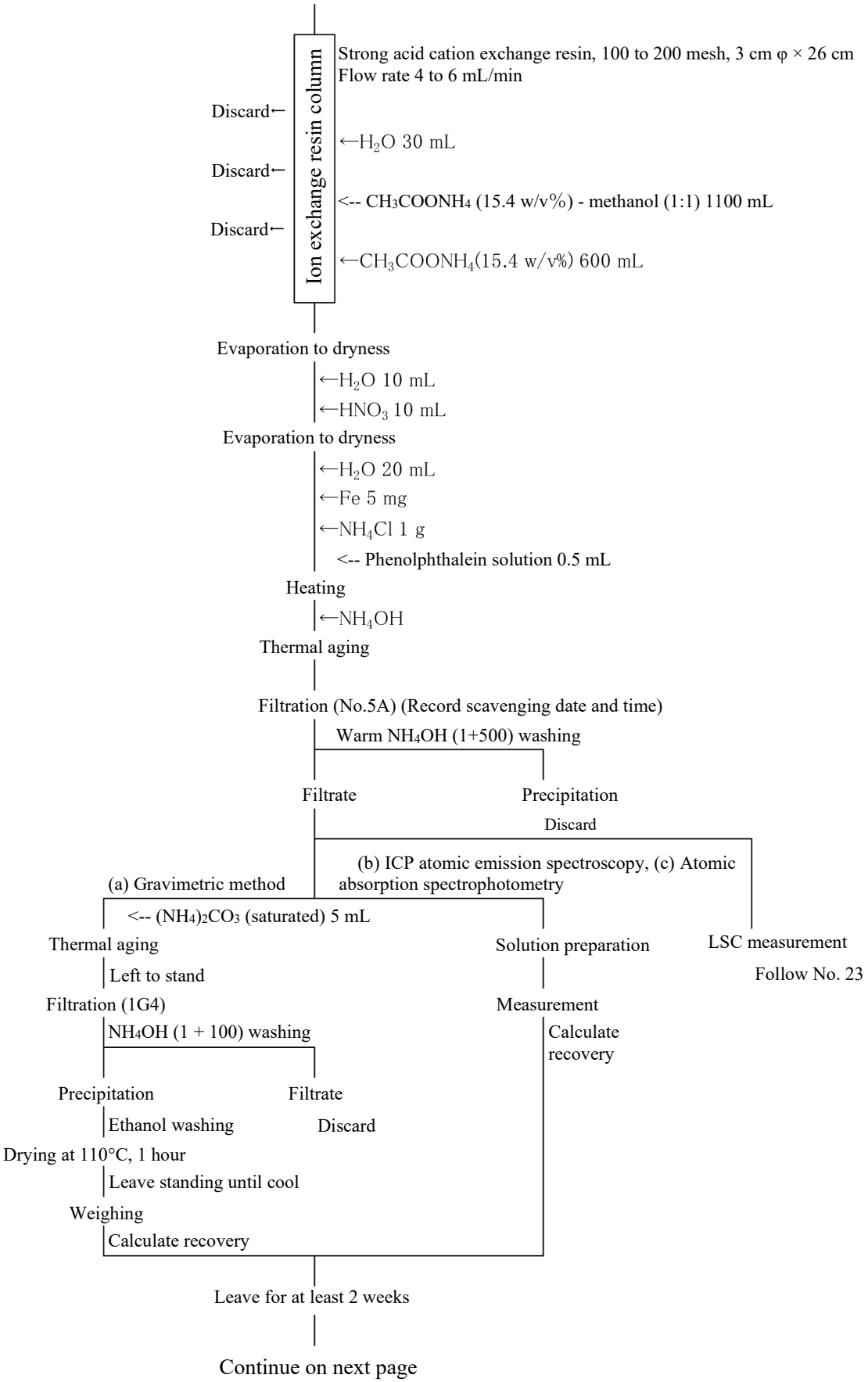
To Fallout (evaporation)—Oxalate method (2/3)

Land water—Oxalate method



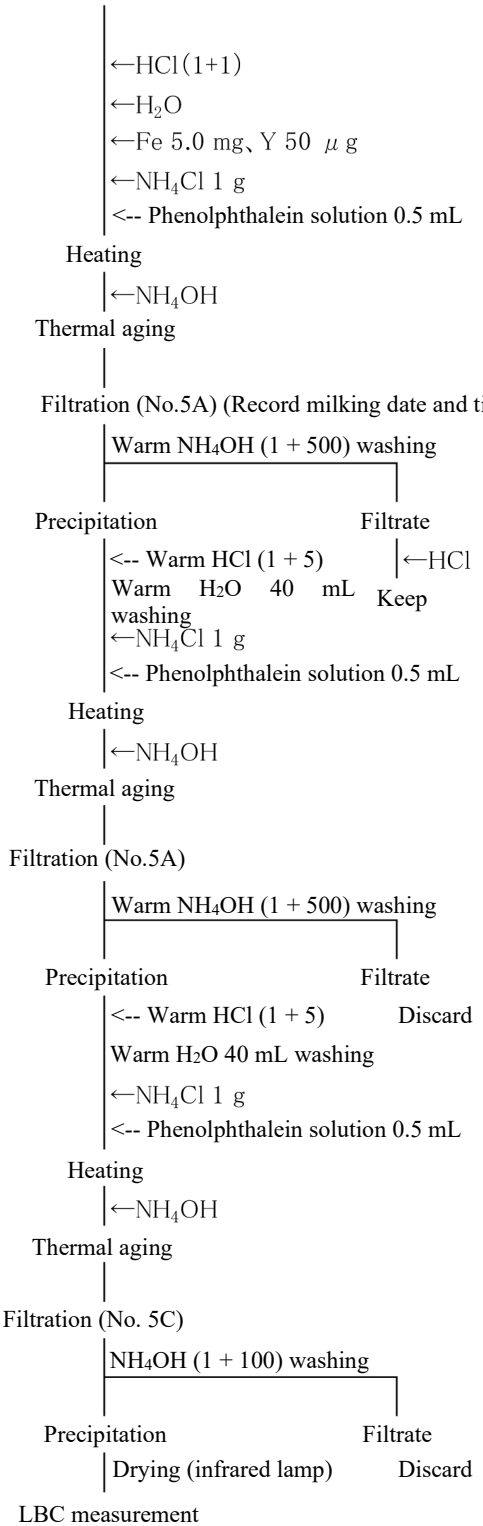
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Seawater—Ion exchange method (1/3)

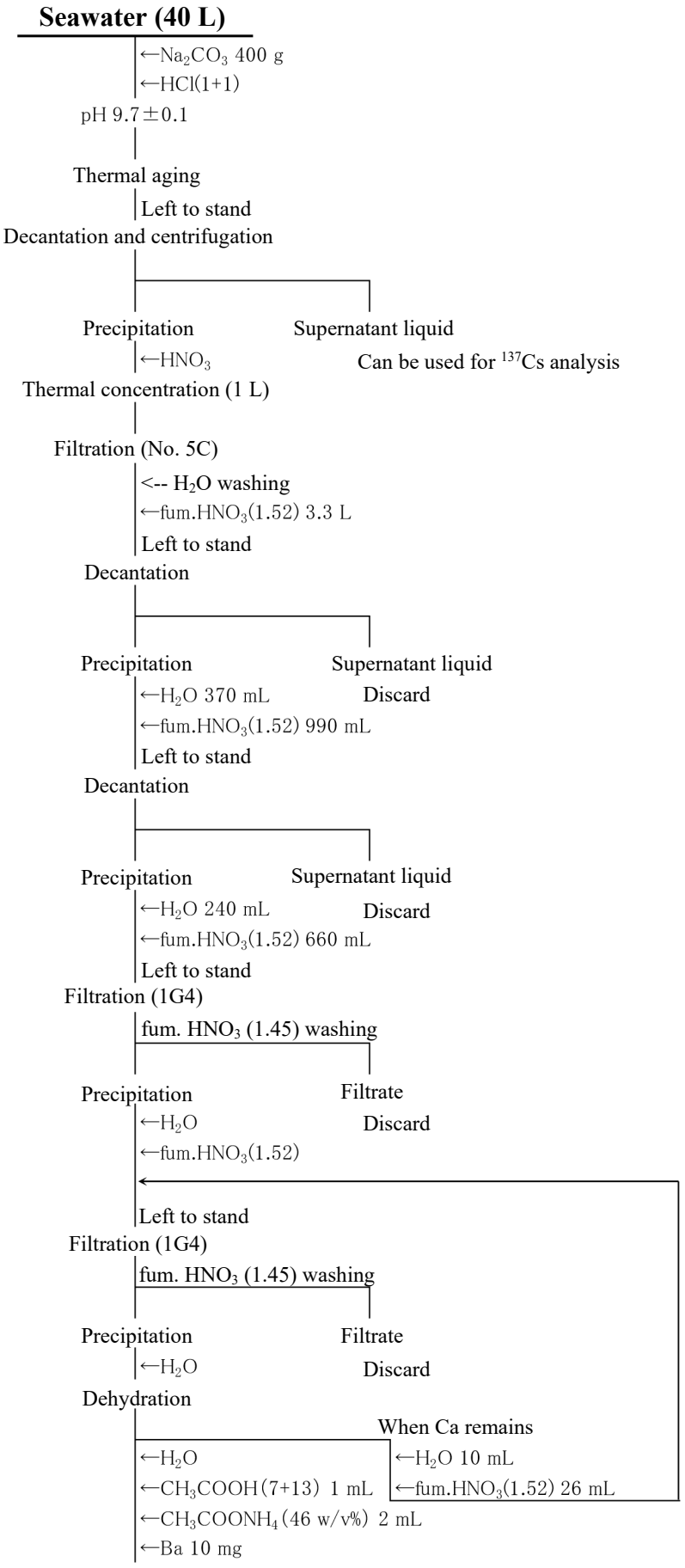


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Seawater—Ion exchange method (2/3)

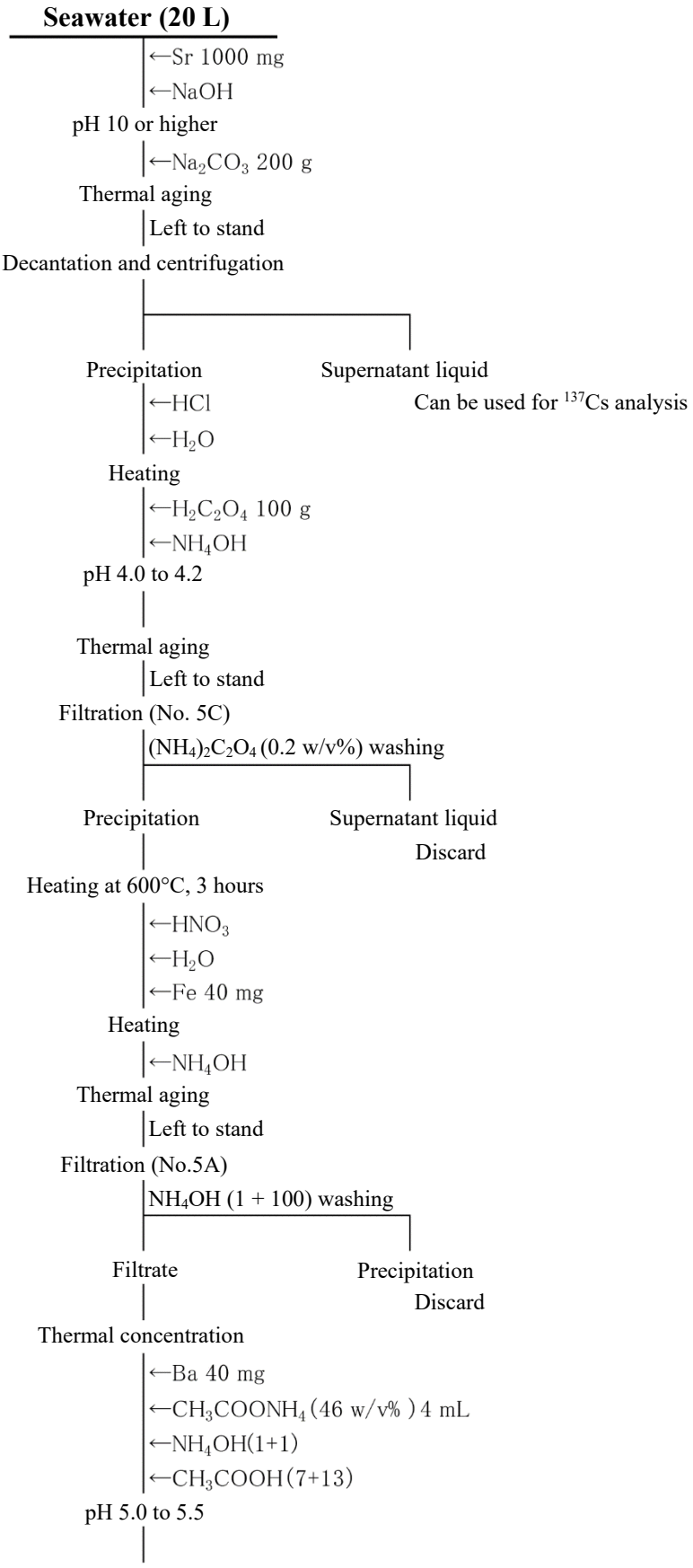


Seawater—Ion exchange method (3/3)



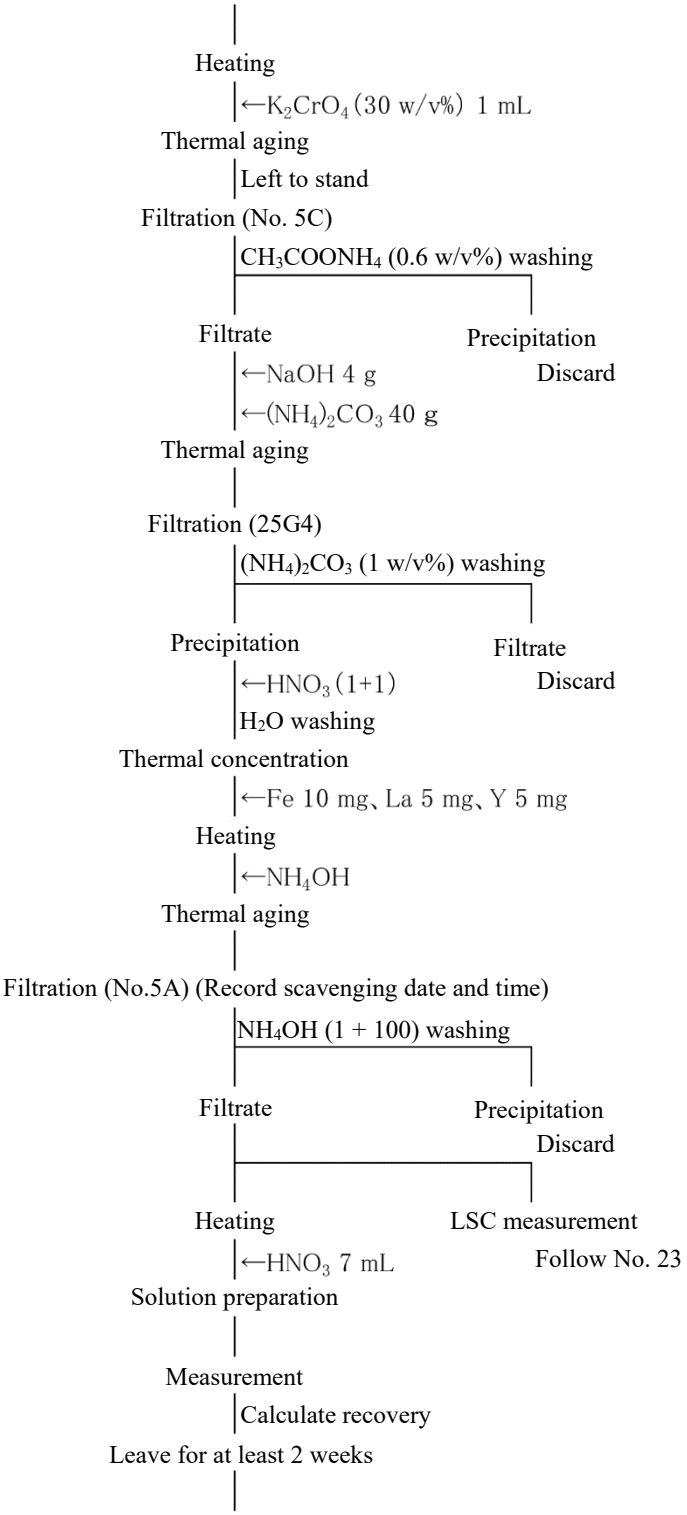
To Fallout (evaporation)—Fuming nitric acid method (2/3)

Seawater—Fuming nitric acid method



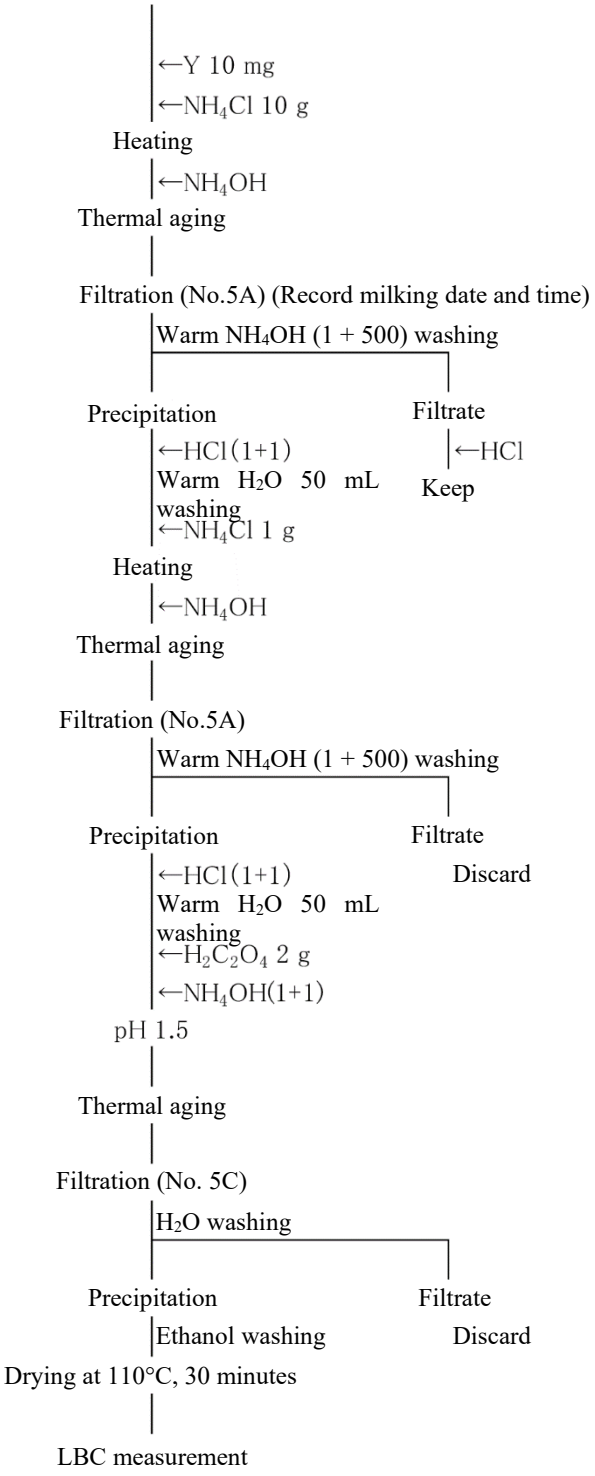
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Seawater—Oxalate method (1/3)

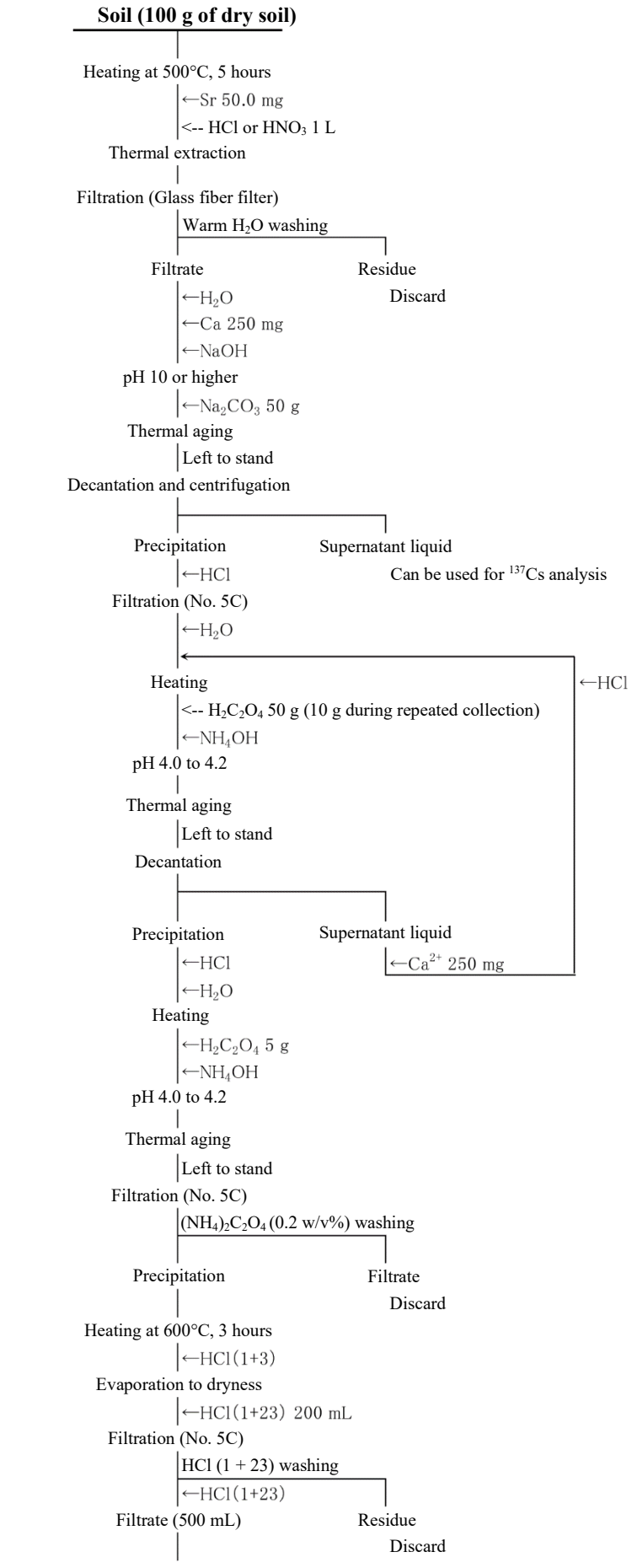


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Seawater—Oxalate method (2/3)

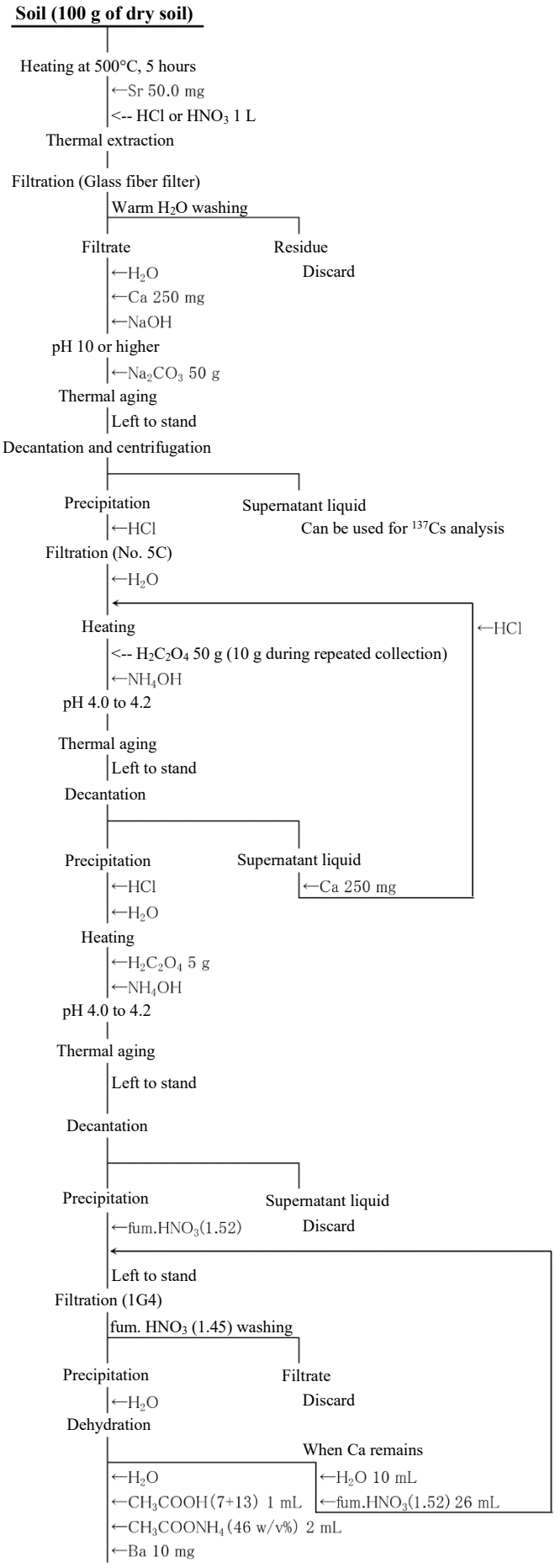


Seawater—Oxalate method (3/3)



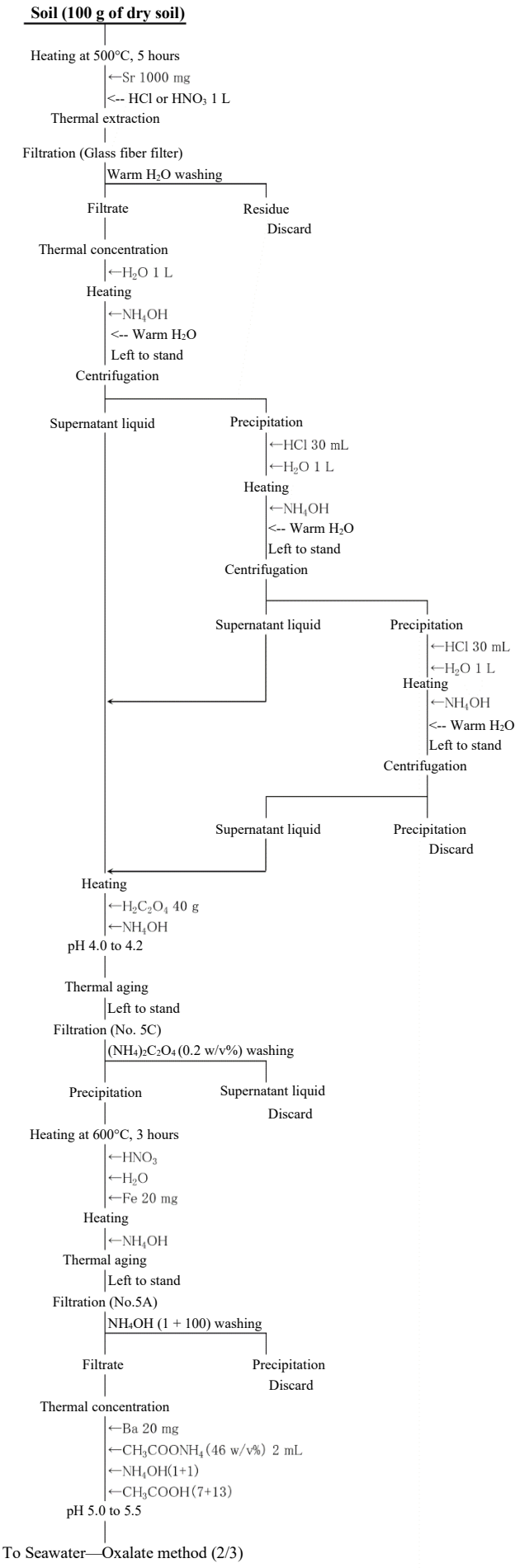
To Seawater—Ion exchange method (2/3)

Soil—Ion exchange method



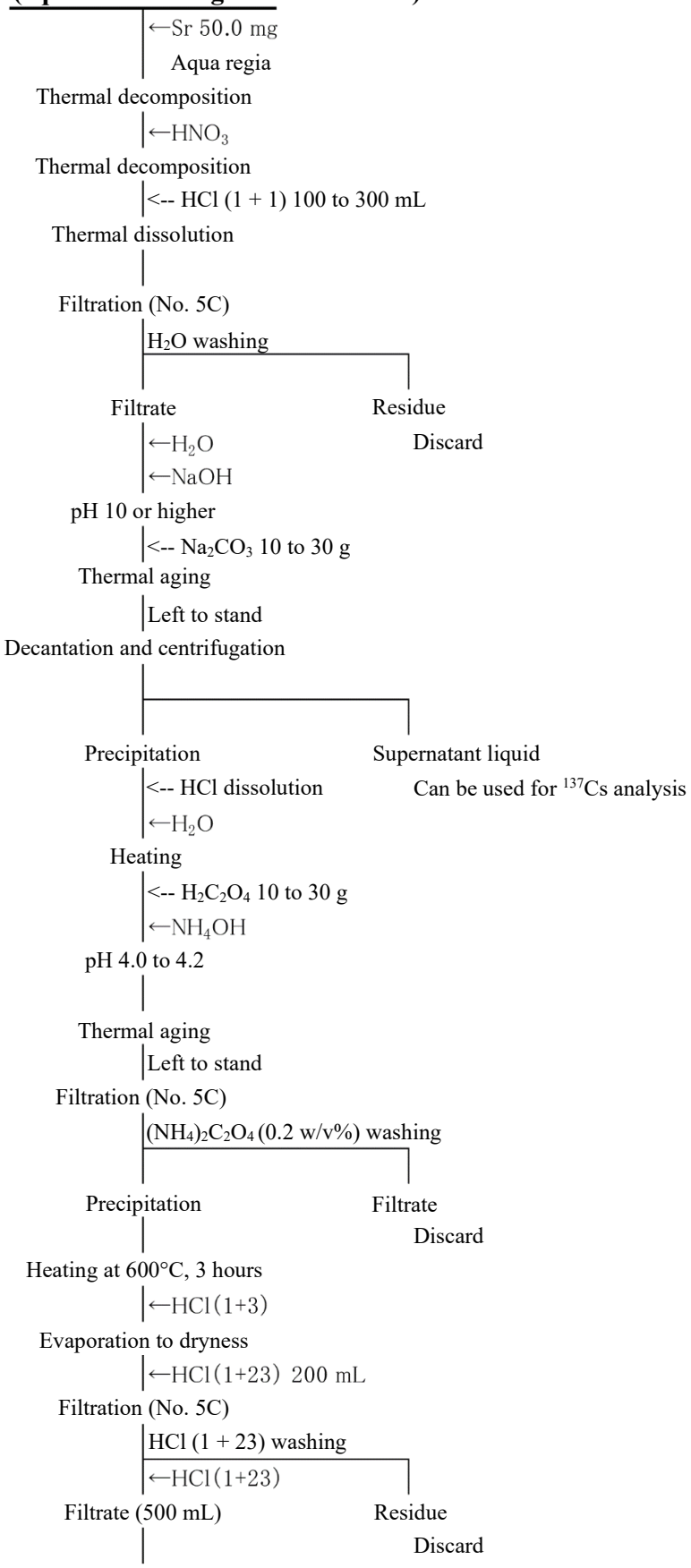
To Fallout (evaporation)—Fuming nitric acid method (2/3)

Soil—Fuming nitric acid method



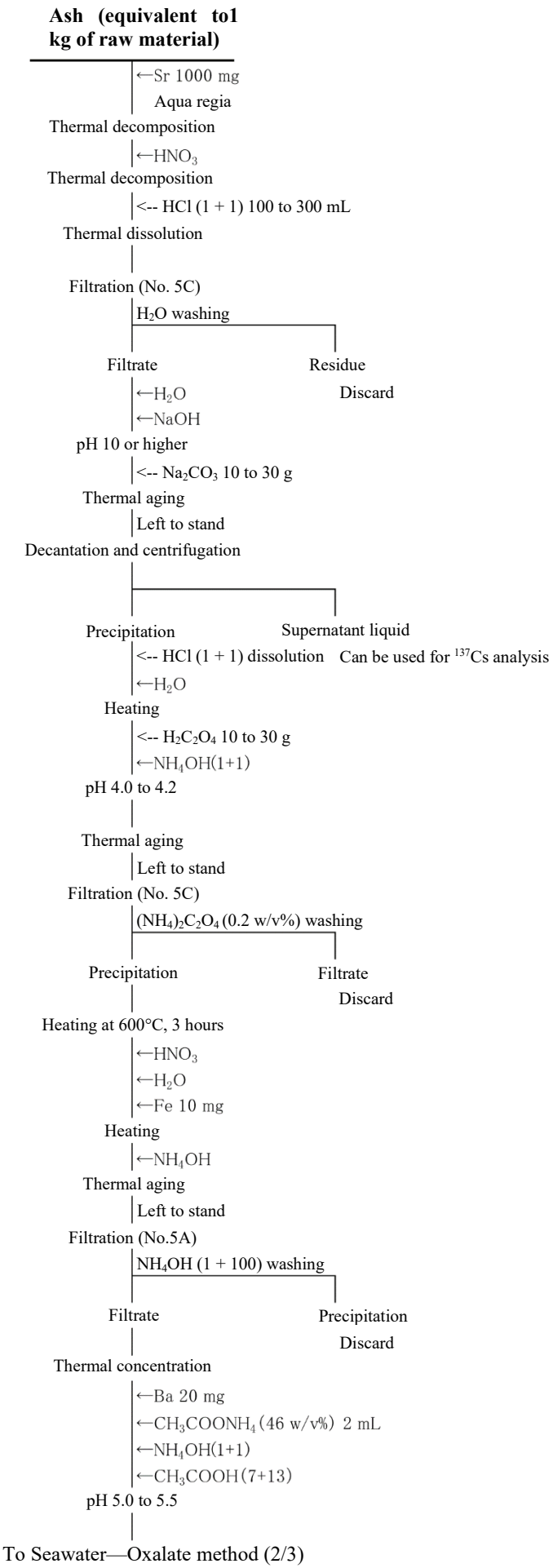
Soil—Oxalate method

Ash (equivalent to1 kg of raw material)



To Seawater—Ion exchange method (2/3)

Ash—Ion exchange method



Ash—Oxalate method

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23) J.Aoki et al.: Direct Quantification of Attogram Levels of Strontium-90 in Microscale Biosamples Using Isotope Dilution-Thermal Ionization Mass Spectrometry Assisted by Quadrupole Energy Filtering, Analytical Chemistry, 95, 4932−4939 (2023)
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Revision History

Established in 1960

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