

Australian Standard[®]

**Recommended practice for inductively
coupled plasma mass spectrometry
(ICP-MS)**

**Part 2: Quantitative determination—
Water**



This Australian Standard® was prepared by Committee CH-016, Spectroscopy. It was approved on behalf of the Council of Standards Australia on 9 October 2009. This Standard was published on 4 November 2009.

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- Australian Institute of Physics
- Australian Nuclear Science & Technology Organisation
- Department of Natural Resources and Water, Qld
- Environmental Laboratory Industry Group
- National Association of Testing Authorities Australia
- National Measurement Institute
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Additional Interests:

- International Precious Metal Institute
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 - Sydney Water Corporation
 - University of Canberra
-

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PREFACE

This Standard was prepared by the Standards Australia Committee CH-016, Spectroscopy.

The objective of this Standard is to describe the procedures for the quantitative determinations of elements in water by ICP-MS.

This Standard is part of a series as follows:

AS

4873 Recommended practice for inductively coupled plasma mass spectrometry (ICP-MS)

4873.1 Part 1: Principles and techniques

4873.2 Part 2: Quantitative determination—Water (this Standard)

The term ‘informative’ has been used in this Standard to define the application of the appendix to which it applies. An ‘informative’ appendix is only for information and guidance.

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STANDARDS AUSTRALIA

Australian Standard

Recommended practice for inductively coupled plasma mass spectrometry (ICP-MS)**Part 2: Quantitative determination—Water****1 SCOPE**

This Standard specifies a method for the analysis of water samples for the following elements: aluminium, antimony, arsenic, barium, beryllium, bismuth, boron, cadmium, caesium, chromium, cobalt, copper, gallium, germanium, gold, lead, lithium, manganese, mercury, molybdenum, nickel, palladium, platinum, rhodium, rubidium, selenium, silver, strontium, tellurium, thorium, thallium, tin, titanium, uranium, vanadium, and zinc.

NOTES:

- 1 Other elements may be determined by this method if adequate performance is demonstrated. Depending on the sample pre-treatment, the method is applicable to the determination of dissolved and total recoverable elements. This Standard should be read in conjunction with AS 4873.1.
- 2 This Standard may be used as a guide for the determination of elements in other matrices provided adequate performance is demonstrated.
- 3 This method is suitable for the determination of elements in aqueous solutions where suspended or particulate material is <0.1% (w/v).
- 4 Water samples containing sediment or suspended particulate matter >0.1% (w/v) should be digested using methods for sediment (e.g. aqua regia digestion) before analysis.
- 5 This method is applicable for the determination of dissolved elements and total recoverable (acid extractable) elements. It is not suitable for the determination of 'total elements' concentrations unless appropriate procedures have been followed to ensure the dissolution of any silicates present.
- 6 This method is suitable for non-saline water. For the analysis of saline water, samples should be diluted to reduce dissolved solids to <0.3%. Several elements are prone to interference from polyatomic ions originating from the matrix and appropriate techniques should be used for the determination of such elements.
- 7 Before any samples are taken, it should be established whether dissolved element concentrations or total recoverable element concentrations, or both, are required.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

- | | |
|--------------|---|
| 1678.8.0.008 | Emergency procedure guide—Transport—Hydrofluoric acid |
| 2508 | Safe storage and handling information card |
| 2830 | Good laboratory practice |
| 2830.1 | Part 1: Chemical analysis |
| 2850 | Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct |
| 4873 | Recommended practice for inductively coupled plasma-mass spectrometry (ICP-MS) |
| 4873.1 | Part 1: Principles and techniques |

AS/NZS

2243 Safety in laboratories

2243.1 Part 1: Planning and operational aspects

2243.2 Part 2: Chemical aspects

5667 Water quality—Sampling (Series)

ISO

3585 Borosilicate glass 3.3—Properties

3696 Water for analytical laboratory use—Specification and test methods

NOTE: Additional information may be found in the documents listed in Appendix E.

3 DEFINITIONS

For the purpose of this Standard the definitions in AS 4873.1 and those below apply.

3.1 Dissolved or filterable elements

Elements determined in a water sample that has been filtered through a specified filter.

NOTE: A 0.45 µm membrane filter is commonly used.

3.2 Total recoverable (or acid extractable) elements

Elements determined in an unfiltered water sample (including particulate matter present in water) following some specified treatment.

3.3 Precision

The closeness of agreement between results obtained by applying the experimental procedure several times under prescribed conditions.

NOTE: The smaller the random part of the errors that affect the result, the more precise is the procedure.

3.4 Repeatability (*r*)

The value at or below which the absolute difference between two single test results obtained with the same method on identical test material, under the same conditions (same operator, same apparatus, same laboratory, and the minimum practical time consistent with separate tests), may be expected to lie with a specific probability.

NOTE: In the absence of other specifications, the probability is 95 percent.

3.5 Reproducibility (*R*)

The value at or below which the absolute difference between two single test results obtained with the same method on identical material obtained by operators in different laboratories may be expected to lie with a specified probability.

NOTE: In the absence of other specifications, the probability is 95 percent.

3.6 Within-laboratory reproducibility (*R_w*)

The value at or below which the absolute differences between two single test results obtained on portions taken from the same test sample, at different times with or without different operators in the same laboratory using the same method, may be expected to lie within a specified probability.

4 PRINCIPLE

A representative portion of a prepared sample is ionized by an inductively coupled plasma. The resulting ions are analysed by a mass spectrometer, which separates and detects ions representative of elements within the sample. The concentrations of various elements in the test sample solution are calculated by comparisons of the net intensity for each analyte element with the net intensity measured for that element in at least one calibration solution.

NOTE: For further details, refer to AS 4873.1.

5 INTERFERENCES

5.1 General

The presence of concomitants in the sample can cause interferences, i.e. systematic errors in the measurements.

Interferences are classified as either spectral or non-spectral. Spectral interferences have an additive influence on the measured intensity. They are caused by isobaric or polyatomic ions having the same mass to charge (m/z) ratio as the analyte ions. Non-spectral interferences, usually called matrix effects, cause enhancement or suppression of the analyte intensity. These effects depend on the elements to be measured and are due to differences between the properties of the test sample solution and those of the calibration solutions.

For further details refer to AS 4873.1.

5.2 Spectral interferences

5.2.1 General

In order to detect spectral interferences, it is recommended that each element concentration be measured using several different isotopes, if available. The results from each isotope of a given element should be similar. If the results are dissimilar, interference is indicated and the use of correction equations or other means may be required. Comparison of results with and without the use of correction equations can reveal the extent of interference. The lower result obtained should be taken as an acceptable estimate of the concentration.

NOTE: If similar results are obtained with and without the use of correction equations, the result that does not involve the use of correction equations is preferred.

If an element has only one isotope, e.g. ^{75}As , spectral interferences cannot be detected by comparison with another isotope. The nature of the sample matrix shall be taken into consideration when deciding the use of correction equations. (See AS 4873.1.)

When correction equations are used, it should be noted that the magnitude of interference depends on several factors including the plasma conditions and the concentration of each interfering element. These factors may be different for each test sample solution.

Individual laboratories should regularly check the validity of interference factors (f) used in correction equations (see AS 4873.1). Interference factors can be expected to differ between instruments.

NOTE: Some examples of spectral interferences are provided in Appendix A.

5.2.2 Isobaric interferences

Isobaric interferences may be addressed by the use of correction equations (see AS 4873.1). The isotopes used for correction shall be determinable without interference and with sufficient precision. Correction equations are usually included in the instrument software.

5.2.3 Polyatomic interferences

Polyatomic ions can be formed from plasma gas components, reagents and sample matrix. Examples of correction equations are given in Table 1. Table 2 provides an indication of the magnitude of interferences likely to be encountered in water analysis.

5.3 Non-spectral interferences

For detailed information on non-spectral interferences see AS 4873.1.

TABLE 1
EXAMPLES OF CORRECTION-EQUATIONS

Element	Isotope	Correction equation	Corrected interference
Arsenic	⁷⁵ As	$I_c(^{75}\text{As}) = ^{75}I - 3.127[^{77}I - 0.815I_c(^{82}\text{Se})]$	⁴⁰ Ar ³⁵ Cl on ⁷⁵ As, ⁷⁷ Se on ⁴⁰ Ar ³⁷ Cl
Barium	¹³⁸ Ba	$I_c(^{138}\text{Ba}) = ^{138}I - 0.0009008I_c(^{139}\text{La}) - 0.002825I_c(^{140}\text{Ce})$	¹³⁸ La and ¹³⁸ Ce on ¹³⁸ Ba
Cadmium	¹¹⁴ Cd	$I_c(^{114}\text{Cd}) = ^{114}I - 0.02684I_c(^{118}\text{Sn})$	¹¹⁴ Sn on ¹¹⁴ Cd
Indium	¹¹⁵ In	$I_c(^{115}\text{In}) = ^{115}I - 0.01486I_c(^{118}\text{Sn})$	¹¹⁵ Sn on ¹¹⁵ In
Lead	²⁰⁶ Pb ²⁰⁷ Pb ²⁰⁸ Pb	$I_c(\text{Pb}) = I_c(^{208}\text{Pb}) + I_c(^{207}\text{Pb}) + I_c(^{206}\text{Pb})$	Variation in natural isotopic composition
Molybdenum	⁹⁸ Mo	$I_c(^{98}\text{Mo}) = ^{98}I - 0.1106I_c(^{101}\text{Ru})$	⁹⁸ Ru on ⁹⁸ Mo
Nickel	⁵⁸ Ni	$I_c(^{58}\text{Ni}) = ^{58}I - 0.04825I_c(^{54}\text{Fe})$	⁵⁸ Fe on ⁵⁸ Ni
Selenium	⁸² Se	$I_c(^{82}\text{Se}) = ^{82}I - 1.009I_c(^{83}\text{Se})$	⁸² Kr on ⁸² Se
Tin	¹²⁰ Sn	$I_c(^{120}\text{Sn}) = ^{120}I - 0.01334I_c(^{125}\text{Te})$	¹²⁰ Te on ¹²⁰ Sn
Tungsten	¹⁸⁴ W	$I_c(^{184}\text{W}) = ^{184}I - 0.001242I_c(^{189}\text{Os})$	¹⁸⁴ Os on ¹⁸⁴ W
Vanadium	⁵¹ V	$I_c(^{51}\text{V}) = ^{51}I - 3.127[^{53}I - 0.1134I_c(^{52}\text{Cr})]$	³⁵ Cl ¹⁶ O on ⁵¹ V, ⁵³ Cr on ³⁷ Cl ¹⁶ O

LEGEND:

I = net intensity at the specified mass

I_c = corrected net intensity for the specified isotope

TABLE 2
IMPORTANT INTERFERENCES IN A SOLUTION OF
Na, K, Ca, Mg, Cl, S, P (each 100 mg/L) and Ba (100 µg/L)

Element	Isotope	Apparent concentration * µg/L	Interference
Arsenic	⁷⁵ As	1	⁴⁰ Ar ³⁵ Cl ⁺
Cobalt	⁵⁹ Co	0.2 to 0.8	⁴³ Ca ¹⁶ O ⁺ , ⁴² Ca ¹⁶ O ¹ H ⁺
Chromium	⁵² Cr	1	³⁵ Cl ¹⁶ O ¹ H ⁺
		1	⁴⁰ Ar ¹² C ⁺
		5	³⁷ Cl ¹⁶ O ⁺ , ⁴⁰ Ar ¹³ C ⁺
Copper	⁶³ Cu	1 to 3	⁴⁰ Ar ²³ Na ⁺
		1 to 2	³¹ P ¹⁶ O ₂ ⁺
	⁶⁵ Cu	2	⁴⁰ Ar ²⁵ Mg ⁺
		2	³¹ P ¹⁷ O ₂ ⁺
		2	³² S ¹⁶ O ₂ ¹ H ⁺ ⁴⁸ Ca ¹⁶ O ¹ H ⁺
Gallium	⁶⁹ Ga	1 to 25	¹³⁸ Ba ²⁺
		0.3	³⁸ Ar ³¹ P ⁺
		1	³⁷ Cl ¹⁶ O ₂ ⁺
	⁷¹ Ga	0.2 to 0.6	⁴⁰ Ar ³¹ P ⁺
Germanium	⁷⁴ Ge	0.3	³⁷ Cl ₂ ⁺
		0.3	⁴⁰ Ar ³⁴ S ⁺
Manganese	⁵⁵ Mn	3	³⁹ K ¹⁶ O ⁺
		3	²³ Na ³² S ⁺

(continued)

TABLE 2 (continued)

Element	Isotope	Apparent concentration * µg/L	Interference
Nickel	⁵⁸ Ni	2.5	⁴⁰ Ca ¹⁸ O ⁺ , ⁴⁴ Ca ¹⁴ N ⁺
	⁶⁰ Ni	3 to 12	⁴⁴ Ca ¹⁶ O ⁺ , ⁴² Ca ¹⁷ O ¹ H ⁺
Selenium	⁷⁷ Se	10	⁴⁰ Ar ³⁷ Cl ⁺
Vanadium	⁵¹ V	1 to 5	³⁵ Cl ¹⁶ O ⁺ , ³⁷ Cl ¹⁴ N ⁺
		1	³⁴ S ¹⁶ O ¹ H ⁺
Zinc	⁶⁴ Zn	7	⁴⁰ Ar ²⁴ Mg ⁺
		3	⁴⁸ Ca ¹⁶ O ⁺
		8	³² S ₂ ⁺ , ³² S ¹⁶ O ₂ ⁺
		1	³¹ P ¹⁶ O ₂ ¹ H ⁺
	⁶⁶ Zn	2	⁴⁰ Ar ²⁶ Mg ⁺
		2	¹³² Ba ²⁺
		5	³² S ³⁴ S ⁺ , ³⁴ S ¹⁶ O ₂ ⁺
		4	³¹ P ³⁵ Cl ⁺
	⁶⁸ Zn	50	³⁶ Ar ³² S ⁺ , ³⁴ S ₂ ⁺ , ³⁴ S ¹⁶ O ₂ ⁺
		4	¹³⁶ Ba ²⁺

* This gives an indication of the apparent concentration of the analyte isotope that may be contributed by the interfering ion or ions. Actual values vary from instrument to instrument and with instrument conditions.

6 REAGENTS

WARNING: SOME REAGENTS AND SOLUTIONS USED IN THIS METHOD MAY CONSTITUTE A HEALTH AND SAFETY HAZARD AND THEIR HANDLING SHOULD BE IN ACCORDANCE WITH AS/NZS 2243, PARTS 1 AND 2, AS 2508 AND AS 2830.1.

6.1 General

All water used in the preparation of solutions, including preparation of test sample solutions, should be Grade 1 water in accordance with ISO 3696. All reagents used to prepare solutions should be of high purity to minimize background intensities due to contaminants and unless otherwise stated, all reagent salts shall be dried in an air oven at 105°C for 2 hours. The concentration of the analyte or interfering substances in the reagents and the water should be negligible compared to the lowest concentration to be determined.

It is good laboratory practice to clean all laboratory ware (including disposable items) used to prepare solutions by soaking (e.g. 12 hours) in nitric acid 5% (6.4) to reduce contaminants.

For preservation and digestion, nitric acid is preferred to minimize interferences by polyatomic ions. For some elements, (see Clause 6.18.1), the use of hydrochloric acid or other preservatives is required.

It is recommended that all solution preparations be carried out in a clean environment.

6.2 Aqua regia

Aqua regia is a mixture of hydrochloric acid and nitric acid. It should be freshly prepared, preferably *in situ*, and not stored. Two solutions may be required as follows:

- (a) Conventional aqua regia is prepared by mixing 3 volumes of hydrochloric acid (6.7) and 1 volume of nitric acid (6.3) with stirring.
- (b) Reverse aqua regia is prepared by mixing 1 volume of hydrochloric acid (6.7) and 3 volumes of nitric acid (6.3) with stirring.

NOTE: Reverse aqua regia introduces less chloride into analytical solutions than conventional aqua regia. Chloride-based interferences are less severe when reverse aqua regia is used.

6.3 Nitric acid, $\rho_{20} \approx 1.4 \text{ g/mL}$ ($\approx 70\% \text{ HNO}_3 \text{ w/w}$)

6.4 Nitric acid 5% v/v

Slowly add 50 mL of nitric acid (6.3) to 800 mL of water, allow to cool to room temperature and dilute to 1 L with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.5 Nitric acid 50% v/v

Slowly add 500 mL of nitric acid (6.3) to 400 mL of water, allow to cool to room temperature and dilute to 1 L with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.6 Hydrobromic acid, concentrated, $\rho_{20} \approx 1.49 \text{ g/mL}$ ($\approx 47\% \text{ HBr w/w}$)

6.7 Hydrochloric acid, $\rho_{20} \approx 1.2 \text{ g/mL}$ (32–35% HCl w/w)

6.8 Hydrochloric acid, 50% v/v

Slowly add 500 mL hydrochloric acid (6.7) to 400 mL of water, allow to cool to room temperature and dilute to 1 L with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.9 Hydrochloric acid $\approx 1.05 \text{ M}$

Slowly add 100 mL concentrated hydrochloric acid (6.7) to about 800 mL of water, allow to cool to room temperature and dilute with water to 1 L. Standardize the solution with 0.1000M sodium carbonate solution with a suitable indicator such as methyl orange.

6.10 Hydrofluoric acid, concentrated $\rho_{20} \approx 1.16 \text{ g/mL}$ ($\approx 48\% \text{ HF w/w}$)

WARNING: EXTREME CARE SHALL BE TAKEN WHEN WORKING WITH THIS ACID, AS SKIN CONTACT CAUSES SEVERE BURNS THAT TAKE SOME TIME TO BECOME OBVIOUS. IN EXTREME CASES SKIN CONTACT MAY BE FATAL. HANDLING SHOULD BE IN ACCORDANCE WITH AS 1678.8.0.008.

6.11 Hydrogen peroxide $\approx 30\% \text{ w/w}$

Particular care should be taken that the hydrogen peroxide is of high purity and intended for trace and analytical work. Other grades of hydrogen peroxide are often stabilized with phosphoric acid or tin compounds and may contain other impurities.

6.12 Orthophosphoric acid, concentrated $\rho_{20} \approx 1.69 \text{ g/mL}$ ($\approx 85\% \text{ H}_3\text{PO}_4 \text{ w/w}$)

6.13 Sodium carbonate 0.1000 M

Dissolve 10.5989 g of anhydrous sodium carbonate in 400 mL of water and dilute to 1 L with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.14 Sodium hydroxide $\approx 20\%$ v/v

Carefully dissolve 20 g of sodium hydroxide pellets in 80 mL of water, allow to cool to room temperature and dilute with water to 100 mL. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15 Calibration stock solutions

6.15.1 General

Both single-element stock solutions and multi-element stock solutions, with adequate specification stating the acid used and the preparation technique, are commercially available. A certificate of analysis detailing concentrations of analyte(s), matrix composition and levels of contaminants, should be provided by the manufacturer. Stock solutions of some elements (e.g. Li and U) may not contain natural ratios of isotopes. Use of such solutions can lead to inaccurate results unless the actual isotope concentrations in the calibration solutions are used in the calculation. Stock solutions should be checked against reference materials.

NOTES:

- 1 While it may be possible for the stock solutions to be kept for longer than 12 months, the laboratory should assure itself of the stability of the solution by carrying out regular checks of the concentration of analyte, e.g. by comparison with certified multi-element or single element solutions, preferably obtained from a different source. The laboratory should have records to confirm the continuing stability of the solutions.
- 2 If stock solutions are prepared directly from metals, care should be taken to ensure that the metal used is free of surface oxidation.
- 3 Where possible, metal strip, wire or granules should be used in preference to powder.
- 4 For suitable storage containers for stock solutions refer to Clause 7.3.

Unless otherwise stated, all reagent salts shall be dried in an air oven at 105°C for 2 hours. Single element calibration stock solutions are prepared as indicated below.

6.15.2 Aluminium (1000 mg/L)

Dissolve 1.0000 g of aluminium metal in a covered 250 mL glass beaker with 20 mL of water and 20 mL of hydrochloric acid (6.7). When reaction has ceased add 10 mL of nitric acid 50% (6.5). After dissolution, add 100 mL of water. Allow the solution to cool, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.3 Antimony (1000 mg/L)

Dissolve 1.1971 g of previously dried antimony(III) oxide, (Sb_2O_3) in a covered PTFE beaker containing 20 mL of water and 5 mL of hydrofluoric acid (6.10). Once the reaction subsides, add 40 mL of nitric acid 50% (6.5) and gently heat to complete dissolution. Allow the solution to cool to room temperature and transfer to a 1 L plastic volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.4 Arsenic (1000 mg/L)

Dissolve 1.3203 g of previously dried arsenic(III) oxide (As_2O_3) in a covered 250 mL beaker with a minimum volume of 20% sodium hydroxide (6.14), then neutralize the solution with dilute nitric acid (6.4). Allow the solution to cool to room temperature, add 20 mL of nitric acid 50% (6.5) and transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.5 Barium (1000 mg/L)

Add 1.4369 g of barium carbonate (BaCO_3) to about 20 mL water in a covered 250 mL beaker and slowly add 10 mL of nitric acid 50% (6.5) with stirring. After complete dissolution, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.6 Beryllium (1000 mg/L)

Dissolve 19.655 g of beryllium sulfate tetrahydrate ($\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$) in a covered 250 mL beaker with 100 mL of water. Transfer to a 1 L volumetric flask, add 20 mL of nitric acid 50% (6.5) and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.7 Bismuth (1000 mg/L)

Dissolve 1.0000 g of bismuth metal in a covered 250 mL beaker with 40 mL of nitric acid 50% (6.5). After dissolution, add 100 mL of water. Allow the solution to cool and then transfer to a 1 L volumetric flask containing about 200 mL of water. Add 100 mL hydrochloric acid (6.7), and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.8 Boron (1000 mg/L)

Dissolve 5.7192 g of boric acid (H_3BO_3) in 100 mL water in a covered 250 mL beaker. After dissolution, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.9 Cadmium (1000 mg/L)

Dissolve 1.0000 g of cadmium metal in a covered 250 mL beaker with 40 mL of nitric acid, 50% (6.5). After dissolution, add 100 mL water. Allow the solution to cool to room temperature, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.10 Caesium (1000 mg/L)

Dissolve 1.2667 g of dried caesium chloride (CsCl) in 100 mL water in a covered 250 mL beaker. After dissolution, transfer to a 1 L volumetric flask, add 10 mL of nitric acid 50% (6.5) and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.11 Cerium (1000 mg/L)

Weigh 1.2284 g of cerium (IV) oxide (CeO_2) into a 250 mL beaker. Add 20 mL of nitric acid 50% (6.5) and swirl to disperse the oxide into the liquid. Cover the beaker with a watch glass and heat to boiling. Carefully add a few drops of hydrogen peroxide (6.11), allow the effervescence to subside, and then repeat the addition of a few drops of hydrogen peroxide. Do not add more hydrogen peroxide until the effervescence from the previous addition has stopped. Continue adding hydrogen peroxide a few drops at a time in this way until a clear, colourless solution is obtained, then continue boiling for a few minutes to destroy any excess hydrogen peroxide. Allow to cool, add 20 mL of nitric acid 50% (6.5), transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.12 Chromium (1000 mg/L)

Dissolve 2.8290 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in 100 mL of water in a covered 250 mL beaker. When completely dissolved, add 10 mL of nitric acid 50% (6.5). Transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.13 Cobalt (1000 mg/L)

Dissolve 1.0000 g of cobalt metal in a covered 250 mL beaker with 40 mL of nitric acid 50% (6.5). After dissolution, add 100 mL water. Allow the solution to cool, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.14 Copper (1000 mg/L)

Dissolve 1.0000 g of copper metal in a covered 250 mL beaker with 40 mL of nitric acid 50% (6.5). After dissolution, add 100 mL water. Allow the solution to cool, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.15 Gallium (1000 mg/L)

Dissolve 1.0000 g of gallium metal in a covered 250 mL beaker with 40 mL of nitric acid 50% (6.5). After dissolution, add 100 mL water. Allow the solution to cool, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.16 Germanium (1000 mg/L)

Dissolve 1.0000 g of germanium metal in a PTFE beaker containing 20 mL of water, 5 mL of nitric acid (6.3) and 5 mL of hydrofluoric acid (6.10). Once the reaction subsides, gently heat to complete dissolution. Allow the solution to cool and then transfer to a 1 L plastic volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.17 Gold (1000 mg/L)

Dissolve 1.0000 g of gold metal in a covered 250 mL beaker with 12 mL of aqua regia (6.2(a)). Warm gently on a hot plate to assist dissolution. After dissolution, evaporate to small volume (≈ 5 mL) add 5 mL hydrochloric acid (6.7) and evaporate again to small volume. Allow the solution to cool, transfer to a 1 L volumetric flask, add 100 mL hydrochloric acid (6.7) and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.18 Lead (1000 mg/L)

Dissolve 1.0000 g of lead metal in a covered 250 mL beaker with 40 mL of nitric acid 50% (6.5). After dissolution, add 100 mL of water. Allow the solution to cool, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.19 Lithium (1000 mg/L)

Add 5.3227 g of lithium carbonate (Li_2CO_3) to about 75 mL water in a covered 250 mL beaker and slowly add 25 mL of nitric acid 50% (6.5) with stirring. After complete dissolution, transfer to a 1 L volumetric flask and fill to the mark with water. Transfer to a suitable container (7.3).

6.15.20 Manganese (1000 mg/L)

Dissolve 1.0000 g of manganese metal in a covered 250 mL beaker with 40 mL of nitric acid 50% (6.5). After dissolution, add 100 mL of water. Allow the solution to cool, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.21 Mercury (1000 mg/L)

Dissolve 1.0798 g of mercury(II) oxide (HgO) in a covered 250 mL beaker with 40 mL of nitric acid 50% (6.5). Allow the solution to cool, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.22 Molybdenum (1000 mg/L)

Dissolve 1.8402 g of ammonium heptamolybdate(VI) tetrahydrate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$) in 100 mL water and 5 mL of hydrofluoric acid (6.10) in a covered 250 mL PTFE beaker. Add 40 mL of nitric acid 50% (6.5), transfer to a 1 L plastic volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.23 Nickel (1000 mg/L)

Dissolve 1.0000 g of nickel metal in a covered 250 mL beaker with 40 mL of nitric acid 50% (6.5). After dissolution, add 100 mL of water. Allow the solution to cool, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.24 Palladium (1000 mg/L)

Dissolve 2.6716 g of ammonium tetrachloropalladate(II) ($(\text{NH}_4)_2\text{PdCl}_4$) in 100 mL water in a covered 250 mL beaker. After dissolution, transfer to a 1 L volumetric flask, add 100 mL hydrochloric acid (6.7) and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.25 Platinum (1000 mg/L)

Dissolve 1.9119 g of ammonium tetrachloroplatinate(II) ($(\text{NH}_4)_2\text{PtCl}_4$) in 100 mL water in a covered 250 mL beaker. After dissolution, transfer to a 1 L volumetric flask, add 100 mL hydrochloric acid (6.7) and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.26 Rubidium (1000 mg/L)

Dissolve 1.4148 g of dried rubidium chloride (RbCl) in 100 mL water in a covered 250 mL beaker. After dissolution, transfer to a 1 L volumetric flask, add 10 mL of nitric acid 50% (6.5) and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.27 Scandium (1000 mg/L)

Dissolve 1.5340 g of scandium oxide (Sc_2O_3) in a covered 250 mL beaker with 20 mL 50% hydrochloric acid (6.8). Warm to dissolve. After dissolution, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.28 Selenium (1000 mg/L)

Dissolve 1.0000 g of selenium in a covered 250 mL beaker with 40 mL of nitric acid 50% (6.5) added incrementally. If the reaction becomes too vigorous, cool the mixture. Warm gently to just below boiling point. After ≈ 45 minutes, all the selenium should have dissolved to a colourless solution. Cool, transfer to a 1 L volumetric flask and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.29 Silver (100 mg/L)

Dissolve 0.1000 g of silver metal in a covered 250 mL beaker with 10 mL of nitric acid 50% (6.5) and then add 100 mL of water. Allow the solution to cool, transfer to 1 L volumetric flask, add 300 mL of concentrated hydrochloric acid (6.7), with stirring and fill to the mark with water. Stopper the flask, mix by inverting and shaking, transfer to a suitable container (7.3).

6.15.30 Strontium (1000 mg/L)

Add 1.6849 g of strontium carbonate (SrCO_3) to 20 mL water in a covered 250 mL beaker and slowly add 10 mL of nitric acid 50% (6.5) with stirring. After complete dissolution, transfer to a 1 L volumetric flask and fill to the mark with water. Transfer to a suitable container (7.3).