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STANDARD

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General rule for test methods of reagents

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Translated

by

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General rule for test methods of reagents

Introduction This Japanese Industrial Standard has been prepared based on **ISO 6353-1**, *Reagents for chemical analysis—Part 1: General test methods*, published in 1982 as the first edition; and in this text, the test methods which were specified in traditional Japanese Industrial Standard have been specified and the International Standard corresponding to them has also been specified in Annex 2. The Nessler methods, however, which have been specified in International Standard to determine ammonium and nitrogen compounds, were modified, and instead indophenol blue methods have been specified.

1 Scope This Japanese Industrial Standard specifies the matters commonly applicable when reagents are to be tested. Concerning the highly purified reagents and biochemical reagents, however, General rule for test methods of highly purified reagents in **JIS K 8007** and General rules for biochemical reagents in **JIS K 8008** apply respectively.

If there is some discrepancy between this Standard and other individual standard, the individual standard shall precede.

- Remarks 1 The methods adopted in **ISO 6353-1** are mentioned in Annex 2.
- 2 Normative references to this Standard are listed in Annex Table 1.
- 3 International standards corresponding to this Standard are listed in Annex Table 2.
- 4 The units and numerical values given in { } in this Standard are based on the traditional units and are appended for informative reference.

2 Definitions, symbols and abbreviations The definitions, symbols and abbreviations in **JIS K 0050** apply, and the rest of terms are as follows.

2.1 Terms for heating and cooling

- (1) **Warming** Heating at 60 °C or less.
- (2) **Igniting** Heating at 650 °C ± 50 °C. When ignition residue (sulfate) is tested, however, heat it at 500 °C ± 50 °C.
- (3) **Heating on water bath** Heating on a water bath under being boiled.
It is also effective to heat by steam at about 100 °C.
- (4) **Heating in water bath** Heating in a water bath under being boiled.
- (5) **Heating on hot plate** Heating on a hot plate such as an electric hot plate.
- (6) **Allowing to cool** Allowing to stand until it gets room temperature.
- (7) **Cooling** To let cool its temperature using such as water or icy water.

2.2 Terms expressing degree of dissolution Follow Table 1.

Table 1 Terms expressing degree of dissolution

Terms	Quantity of solvent needed to dissolve definite amount of solute ⁽¹⁾ ml
Extremely soluble	less than 1
Soluble	1 or over to and excl. 10
Rather soluble	10 or over to and excl. 30
Rather insoluble	30 or over to and excl. 100
Insoluble	100 or over to and excl. 1 000
Extremely insoluble	1 000 or over to and excl. 10 000
Almost not soluble	10 000 or over

Note ⁽¹⁾ At ordinary temperature, it means 1 g for solid substance, and 1 ml for liquid substance.

2.3 Other terms

(1) Purity and concentration

- (a) **Purity** Purity means the content ratio of the substance which is constituting the reagent itself. If necessary, as shown in the next example, test condition, test method, and so on are attached in parentheses, after the value of purity.

Purity (after drying) Purity of sample which was measured after being dried under specified conditions.

Purity (converted as dried substance) Measure purity without drying, obtain separately drying loss, and convert the purity of sample into the purity under being dried making use of the drying loss.

Purity (converted as anhydride) Measure purity without drying, obtain separately water content, and convert the purity of sample into the purity under anhydride making use of the water content.

Purity (GC) Making use of a gas chromatograph, develop the sample content under specified analysis condition, and obtain the purity by means of area percentage method or corrected area percentage method. Purity (HPLC) may be obtained similarly.

- (b) **Concentration** When the main component of liquid sample is contained in solvent, if its specified value is considerably lower than 100 %, the value of concentration could be used for purity (hydrochloric acid, nitric acid, aqueous ammonia, and so on).
- (2) **Solubility** Condition shown when sample is dissolved in solvent. It is expressed as water solubility, hydrochloric-acid solubility, or the like, depending on the kind of employed solvent. For instance, in case of solvent of hydrochloric acid, if its concentration is less than 10 %, it would be expressed as in dilute hydrochloric-acid solubility. As other case than above, there are dilute sulfuric-acid solubility, dilute aqueous ammonia solubility, and so on.
- (3) **Test adaptability** Test results shall be adaptable to the limit specified in test method.
- (4) **Limit criteria of adaptability** This is the criteria specifying the limit of adaptability, for instance, the degree of coloring of sample or turbidity.

- (5) **Solution of sample side** The solution which has been made a definite amount either owing to dissolving sample in solvent, or owing to adding solvent in the sample after being pretreated.
- (6) **Solution of standard side** The solution which has been made a definite amount either owing to dissolving standard solution in solvent or owing to adding solvent after the standard solution was treated similarly to the sample (in some cases, sample may be added).
- (7) **Solution of reference side** The definite amount of solution prepared by adding solvent into the reagent which was taken by the same amount as that used for pretreatment of sample (or standard solution) and then treated similarly.
- (8) **Taking accuracy** Using such as a volumetric flask, transfer pipet, or buret, taking liquid with the accuracy by which the measuring instruments are qualified.
- (9) **About** When "about" is attached before the numeral expressing quantity, that means the number that is near numeral. If a tolerance limit is needed, the range of $\pm 10\%$ of the numeral or the larger one of the rounding-off errors of the numeral shall be adopted.

2.4 Symbols

- (1) **Chemical symbols in test items** In the items listed in quality table or test method, the expression such as chloride (Cl), sulfate (SO_4), or iron (Fe), means that the ingredient that is parenthesized will be tested, and the expression such as heavy metals (as Pb) or bases (as K_2CO_3) means that plural target ingredients will be collectively tested in terms of parenthesized one ingredient.

Remarks : **Symbols employed in test method** The symbols employed in the description of test method and preparation method are as follows.

+	To add
→	To transfer to next procedure
.....	Comparison or result
" " (carry out a times)	To carry out a times procedures set in " "
+ A (→ a ml)	Adding solvent A to make total a ml
B ($b+c$)	Mix b ml of reagent B and c ml of water

2.5 Abbreviation

AgDDTC	Silver N,N -diethyl dithiocarbamate
BANASS	4, 4'-bis (4-amino-1-naphthylazo)-2, 2'-stilbenedisulfonic acid
EDTA2Na	Disodium dihydrogen ethylenediaminetetraacetate dihydrate
HSNN	2-hydroxy-1-(2'-hydroxy-4'-sulfo-1'-naphthylazo)-3-naphthoic acid
NaDDTC	Sodium N,N -diethyldithiocarbamate trihydrate
TMS	Trimethylsilyl
AAS	Atomic absorption spectrometry

FES	Flame photometry
GC	Gas chromatography
HPLC	High-performance liquid chromatography
IR	Infrared spectroscopy
ICP-AES	Inductively coupled plasma atomic emission spectrometry
TLC	Thin layer chromatography

3 General terms The general terms commonly applicable to tests shall conform to **JIS K 0050**, but the rest of the terms be as follows.

3.1 Atomic weight (*AW*) and formula weight (*FW*) Atomic weight shall follow the atomic weight table (relative atomic weight of $^{12}\text{C} = 12$) ⁽²⁾ which is the newest when the Standard was established or revised. Formula weight shall be obtained by summing up atomic weights of constituting atoms, and then rounded off to two places of decimals according to **JIS Z 8401**.

Note ⁽²⁾ This table is the one which has prepared by the Atomic Weight Committee of International Union of Pure and Applied Chemistry (IUPAC), and which was published from Atomic Weight Sub-committee of the Chemical Society of Japan.

3.2 Equivalent (chemical equivalent) and conversion factor In such test method as purity, when they are needed for the calculation of content ratio of target component, they shall be mentioned (sometimes they are mentioned as only informative reference). Calculate the equivalent and conversion factor using the formula weight of the substance (total of the atomic weights), and when the first significant digit of obtained number is any of 1, 2, or 3, round off it to 5 significant figures, and when other case than above is, round off it to 4 significant figures.

3.3 Expression of ratio and of solution concentration Ratio is expressed by any of, % for the mass by percentage, ppm for the mass by part (s) per million, ppb for the mass by part (s) per billion, vol % for the volume by percentage, vol ppm for the volume by part (s) per million, or vol ppb for the volume by part (s) per billion. Solution concentration is expressed by any of, mol/l for mol per litre, mmol/l for millimol per litre, or the like, and when it is expressed with mass (g) of solute ⁽³⁾ contained in 1 l of solution, the symbol like g/l is used.

Note ⁽³⁾ If solute is hydrate, it is judged as follows; for example, barium chloride solution (100 g/l) means 100 g/l as the form of BaCl_2 .

3.4 Specified value Specified value is the quality level to which the products shall conform, and gives upper adaptable limit and lower adaptable limit or the range for adaptability.

In case of purity test, when specified value defines only lower adaptable limit, the upper adaptable limit shall be set as smaller one between 101 % and $[100 + (100 - \text{specified value})]$.

3.5 Measured value Measured value is calculated one more digit than those of specified value, and rounded off to the digits of specified value according to **JIS Z 8401**. This measured value is to be judged whether it falls within the range of specified value.

3.6 Reagents, water, and implements for test The reagents, water, and implements for test shall be those specified in Japanese Industrial Standards, and if there is no specification in Japanese Industrial Standards, the ones specified in individual standard shall be used. Each is detailed respectively as follows.

- (1) **Reagents** Reagents shall be those specified in Japanese Industrial Standards, or the ones with equal or superior quality to. If there is no specification in Japanese Industrial Standards, they shall be specified in individual standard.
- (2) **Water** The water applicable to the tests shall be either the water purified by distillation method or ion-exchange method, or the water purified by the method combined reverse-osmosis method, distillation method or ion-exchange method. If necessary, it shall be specified in individual standard.
- (3) **Carbon dioxide-free water** Place water in a flask, boil it for 15 minutes, as shown in Fig. 1, connect the flask with either a gas washing bottle containing potassium hydroxide solution (250 g/l) or a soda-lime pipe in order to shield carbon dioxide in air, and allow it to cool. Prepare this water when it is needed.

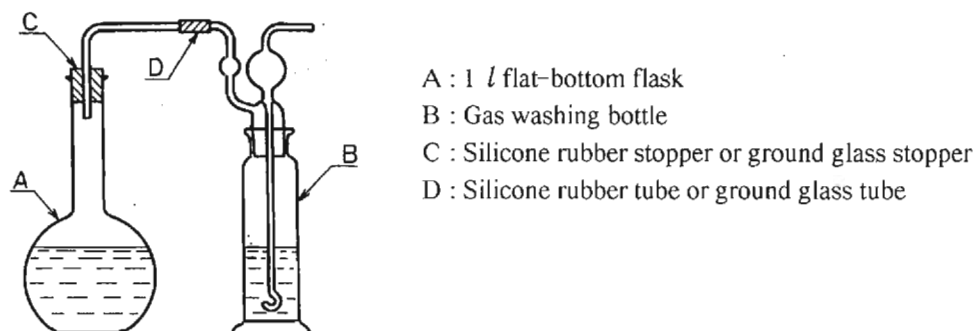


Fig. 1 Example of cooling apparatus of carbon dioxide-free water

- (4) **Dissolved oxygen-free water** Place water in a flask, boil it for 5 minutes, as shown in Fig. 1, connect a gas washing bottle containing alkaline pyrogallol solution ⁽⁴⁾ in order to shield oxygen in air, and allow it to cool. Alternatively, place water in a flask, and aerate nitrogen through water for about 15 minutes. Prepare this water when it is needed.

Note ⁽⁴⁾ **Preparation of alkaline pyrogallol solution** Dissolve 10 g of pyrogallol specified in JIS K 8780 in 80 ml of sodium hydroxide solution (300 g/l), and add sodium hydroxide solution (300 g/l) to make total 100 ml. Prepare this solution when it is needed.

- (5) **Thermometer** Use the glass rod-type thermometer (total immersion) specified in JIS B 7411 which has been calibrated according to JIS Z 8710.

The thermometer having more accuracy is also applicable. When individual standard has each specification about usable thermometer, follow it.

- (6) **Filter paper for washing** Wash two times filter paper (such as 5 grade C) specified in JIS P 3801 with each 50 ml of nitric acid (1+2), then wash two times with each 50 ml of water, take final 20 ml washings into a test tube, add 1 ml of nitric acid (1+2) and 1 ml of silver nitrate solution (20 g/l) into the washings, and confirm that the washings show clarity after 15 minutes standing. If necessary, repeat washing.

- (7) **Desiccator** Employ the desiccator keeping desiccant of silica gel (A type 1 grade) specified in **JIS Z 0701**. Use the silica gel colored with cobalt (II) chloride, and if color is changed, heat it at about 130 °C for regeneration.

Vacuum desiccator means the desiccator with silica gel whose inside pressure was reduced to 2.0 kPa {15 mmHg} or less.

- (8) **Storage container** In order to store solutions for test, employ an airtight container, and in case of glass containers, it shall be a wide-mouthed bottle or narrow-mouthed bottle specified in **JIS R 3503**, and in case of polyethylene bottle, it shall be that specified in **JIS Z 1703** or the equal quality one.

An airtight container is the container which can nearly completely prevent the intrusion of liquid, and deterioration, evaporation, or loss of inside contents.

3.7 Test procedures or the like

- (1) **Environment of testing** Carry out the tests at room temperature.
- (2) **Sampling** Sample shall be taken from the container that represents the lot of products and is newly unsealed.
- (3) **Blank-test procedures for reagents** When test procedures need pretreatment of sample, if the mass of target components, which are contained in reagents for the pretreatment as impurities, exceeds $\frac{1}{30}$ mass of target components contained in consumed amount of reference solution, carry out suitable treatment at standard side on the reagents employed at pretreatment.
- (4) **Titration** Read out the liquid quantity of titrant when titration carried out to the nearest $\frac{1}{10}$ of the smallest graduations of a buret. When the temperature of titrant during titration is out of 20 °C, carry out titration at the same time of standardization; alternatively, when titrant is water solution, the temperature correction of amount of titrant shall be carried out either owing to Table 2 in case of the concentration of 0.1 mol/l or less, or owing to the calculation of correction value according to the following formula in case of over 0.1 mol/l.

Calculation formula Calculation of temperature correction for titrant whose concentration is over 0.1 mol/l.

$$V_t = V_{20} \left[\left[1 + a(t - 20) \right] \times \frac{d_t}{d_{20}} - 1 \right]$$

where, V_t : value for correction of volumeter at t °C which shows V_{20} (ml) at 20 °C (ml)

a : coefficient of volume expansion of glass (0.000 010 K⁻¹)

d_t : density of solution at t °C (g/ml)

d_{20} : density of solution at 20 °C (g/ml)

t : temperature of solution (°C)

Table 2 Correction value at each temperature for 1 000 ml of 0.1 mol/l or less titrant

Temperature °C	Correction value ml	Temperature °C	Correction value ml	Temperature °C	Correction value ml
5	+1.61	17	+0.54	29	− 2.15
6	+1.60	18	+0.37	30	− 2.44
7	+1.57	19	+0.19	31	− 2.73
8	+1.53	20	0.00	32	− 3.03
9	+1.47	21	− 0.20	33	− 3.34
10	+1.40	22	− 0.41	34	− 3.65
11	+1.31	23	− 0.63	35	− 3.97
12	+1.22	24	− 0.86	36	− 4.30
13	+1.11	25	− 1.10	37	− 4.64
14	+0.98	26	− 1.35	38	− 4.98
15	+0.85	27	− 1.61	39	− 5.33
16	+0.70	28	− 1.88		

Remarks : **Way to use Table 2** When the temperature of titrant is other than 20 °C , the volume of titrant is corrected owing to adding the value obtained by multiplying both the correction value (ml) in the Table corresponding to the temperature and the parting ratio [consumed volume (ml)/1 000 ml].

- (5) **Colorimetry and turbidimetry** Carry out colorimetry and turbidimetry with using flat-bottom test tubes shown in Fig. 2 of 5. General test method, stand side by side them of sample side and standard side within 30 seconds after procedures, and compare them by observing either from side or above.
- (6) **Acidity or alkalinity of solution** The acidity or alkalinity of solution is tested with litmus test paper specified in JIS K 9071.
- (7) **Control of pH value of solution** Carry out the control of pH value of solution using a type II pH meter specified in JIS Z 8802.

4 Solutions for test The solution for dilution, reagent solution, reference liquid, indicator, and titrant, which are needed for tests, shall be as follows.

4.1 Solution for dilution When reagent is needed for dilution, as far as the reagents shown in Table 3 is used, the marking as “reagent (a+b)” is adopted. When preparing the concentration other than this table, this preparation method is applicable.

Table 3 Example of solution for dilution

Name	Preparation	Remarks
Aqueous ammonia (2+3)	Aqueous ammonia (JIS K 8085) (28.0 to 30.0 %) 200 ml + water 300 ml	Store in poly-ethylene bottle
Hydrochloric acid (2+1)	Hydrochloric acid (JIS K 8180) (35.0 to 37.0 %) 200 ml + water 100 ml	
Perchloric acid (1+1)	Perchloric acid (JIS K 8223) (60.0 to 62.0 %) 100 ml + water 100 ml	
Hydrogen peroxide (1+9)	Hydrogen peroxide (JIS K 8230) (30.0 to 35.5 %) 10 ml + water 90ml	
Acetic acid (1+1)	Acetic acid (JIS K 8355) (at least 99.5 %) 100 ml + water 100 ml	
Hydrobromic acid (1+1)	Hydrobromic acid (JIS K 8509) (47.0 to 49.0 %) 100 ml + water 100 ml	
Nitric acid (1+2)	Nitric acid (JIS K 8541) (60 to 61 %) 100 ml + water 200 ml	
Hydrofluoric acid (1+1)	Hydrofluoric acid (JIS K 8819) (46.0 to 48.0 %) 100 ml + water 100 ml	Store in poly-ethylene bottle
Hydroiodic acid (1+1)	Hydroiodic acid (JIS K 8917) (55.0 to 58.0 %) 100 ml + water 100 ml	Prepare when it is needed
Sulfuric acid (1+5)	Water 100 ml + sulfuric acid (JIS K 8951) (95.0 % or more) (while stirring and cooling)	
Phosphoric acid (1+1)	Phosphoric acid (JIS K 9005) (at least 85.0 %) 100 ml + water 100 ml	

4.2 Reagent solution In the preparation method, for instance, the expression as “10 g + water (→ 100 ml)” means that “dissolve 10 g in water to make 100 ml solution”.

When preparation of the solution with other concentration than in the table, this preparation method is applicable. The solution not prefixed with name of solvent means water solution.

Table 4 Reagent solution

Name	Concentration (g/l)	Preparation method	Remarks
AgDDTC• chloroform solution		AgDDTC (JIS K 9512) 0.25 g + brucine <i>n</i> -hydrate (JIS K 8832) (as dihydrate) 0.1 g + chloroform (JIS K 8322) 100 ml → stir well. When chloroform dispersed while dissolving, add chloroform to make 100 ml.	Store in a brown colored glass bottle.
EDTA2Na solution	250	EDTA2Na (JIS K 8107) 26.3 g + water 50 ml + aqueous ammonia (2+3) 20 ml → dissolve + water (→ 100 ml)	
EDTA2Na solution (for indophenol blue method)		EDTA2Na 5 g + [sodium hydroxide (JIS K 8576) 1 g + water 60 ml] + water (→ 100 ml)	
NaDDTC solution	40 10 1	NaDDTC (JIS K 8454) 5.3 g + water (→ 100 ml) NaDDTC 1.3 g + water (→ 100 ml) NaDDTC 0.13 g + water (→ 100 ml)	Prepare when it is needed.
Sodium nitrite solution	200	Sodium nitrite (JIS K 8019) 20 g + water (→ 100 ml)	Prepare when it is needed.
Amidosulfuric acid solution	100	Amidosulfuric acid (JIS K 8587) 10 g + water (→ 100 ml)	Prepare when it is needed.
Ammonium amidosulfate solution	350	Ammonium amidosulfate (JIS K 8588) 35 g + water (→ 100 ml)	Prepare when it is needed.
1-amino-2-naphthol-4-sulfonic acid solution		Sodium hydrogensulfite (JIS K 8059) 6 g + sodium sulfite (JIS K 8061) 0.1 g + 1-amino-2-naphthol-4-sulfonic acid (JIS K 8050) 0.05 g + water (→ 50 ml)	Store in a brown colored glass bottle.
Sodium sulfite solution	170 100	Sodium sulfite heptahydrate (JIS K 8060) 34 g + water (→ 100 ml) Sodium sulfite heptahydrate 20 g + water (→ 100 ml)	Prepare when it is needed.
Aluminon solution	1	Aluminon (JIS K 8011) 0.1 g + water (→ 100 ml)	Store in a brown colored glass bottle.
Ammoniacal ammonium chloride buffer solution		Ammonium chloride (JIS K 8116) 7 g + aqueous ammonia 57 ml + water (→ 100 ml)	Store in a polyethylene bottle with airtight stoppering.
Ammoniacal silver nitrate solution		Silver nitrate solution (20 g/l) → add gently aqueous ammonia (2+3) (until once generated precipitation dissolves)	Prepare when it is needed.
Indigo carmine solution	1.8	Indigo carmine (JIS K 8092) 0.18 g (equivalent amount as 100 %) + hydrochloric acid (2+1) 15 ml + water (→ 100 ml)	Store in a brown colored glass bottle. Use up in 30 days.

Table 4 Reagent solution (Continued)

Name	Concentration (g/l)	Preparation method	Remarks
2, 3-indolinedione•sulfuric acid solution		2, 3-indolinedione (JIS K 8089) 0.1 g + sulfuric acid 20 ml	Store in a brown colored glass bottle.
Ethanol	50 (vol%)	Ethanol (99.5) (JIS K 8101) 50 ml + water (→ 100 ml)	
Ethanol (for test of aldehyde and ketone)		Ethanol (99.5) 500 ml + 2, 4-dinitrophenylhydrazine (JIS K 8480) 10 g + hydrochloric acid 0.2 ml → boil for 2 hours with a commonly ground reflux condenser → distill → discard initial 100 ml distillate and use 300 ml intermediate distillate, which must have no coloring (CH_3COCH_3 : about 0.000 1 % or less).	
Ammonium chloride solution	100	Ammonium chloride 10 g + water (→ 100 ml)	
Potassium chloride solution	100	Potassium chloride (JIS K 8121) 10 g + water (→ 100 ml)	
Calcium chloride solution	100	Calcium chloride dihydrate (JIS K 8122) 13.2 g + water (→ 100 ml)	Prepare when it is needed.
Tin (II) chloride solution (for AgDDTC method)		Tin (II) chloride dihydrate (JIS K 8136) 40 g + hydrochloric acid (for arsenic analysis) (→ 100 ml) [Add a couple of grains of tin (JIS K 8580) for storing.] When needed, dilute ten times with water.	Store in a brown colored glass bottle.
Tin (II) chloride solution (for colorimetry for extracted silicate)		Tin (II) chloride dihydrate 1 g + hydrochloric acid (2+1) 9 ml + water (→ 100 ml)	Prepare when it is needed.
Tin (II) chloride solution (for atomic absorption spectrometry of arsenic analysis)		Tin (II) chloride dihydrate 10 g + hydrochloric acid (for arsenic analysis) (→ 100 ml)	Store in a brown colored glass bottle.
Tin (II) chloride solution (for test of arsenic limit)		Tin (II) chloride dihydrate 40 g + hydrochloric acid (for arsenic analysis) 60 ml	Store in a brown colored glass bottle.
Tin (II) chloride solution (for arsenic determination)		Tin (II) chloride dihydrate 4 g + hydrochloric acid (for arsenic analysis) 125 ml + water (→ 250 ml)	Prepare when it is needed.
Tin (II) chloride solution (for phosphoric acid determination)		Tin (II) chloride solution (for test of arsenic limit) 1 ml + sulfuric acid (1+30) (→ 250 ml)	Prepare when it is needed.
Iron (III) chloride solution	100	Iron (III) chloride hexahydrate (JIS K 8142) 16.7 g + hydrochloric acid (2+1) 9 ml + water (→ 100 ml)	Store in a brown colored glass bottle.

Table 4 Reagent solution (Continued)

Name	Concentration (g/l)	Preparation method	Remarks
Sodium chloride solution	100	Sodium chloride (JIS K 8150) 10 g + water (→ 100 ml)	
Barium chloride solution	100	Barium chloride dihydrate (JIS K 8155) 11.7 g + water (→ 100 ml)	
Barium chloride solution (seeding solution)		Preparation of potassium sulfate•dilute ethanol solution Potassium sulfate (JIS K 8962) 0.02 g + water 70 ml → dissolve + ethanol (99.5) (→ 100 ml) Preparation of seeding solution Barium chloride solution (100 g/l) cooled at 10 °C 8 ml + potassium sulfate • dilute ethanol solution 1 ml → shake → use it after exact 1 minute later	
Hydroxylammonium chloride solution	100	Hydroxylammonium chloride (JIS K 8201) 10 g + water (→ 100 ml)	
Hydroxylammonium chloride solution (for iron test)		Hydroxylammonium chloride solution (100 g/l) 100 ml + bathophenanthroline solution 1 ml + ethyl acetate 5 ml → shake violently for 30 s → let it stand → separate water layer" (Repeat until ethyl acetate layer gives no coloring.)	
Hydroxylammonium solution (for copper test)		Hydroxylammonium chloride solution (100 g/l) 100 ml + bathocuproine solution 1 ml + ethyl acetate 5 ml → shake violently for 30 s → let it stand → separate water layer" (Repeat until ethyl acetate layer gives no coloring.)	
Phenylhydrazinium chloride solution	10	Phenylhydrazinium chloride (JIS K 8203) 1 g + water (→ 100 ml)	Store in a brown colored glass bottle.
Basic lead (II) acetate solution		Lead (II) acetate solution (100 g/l) 10 ml + sodium hydroxide solution (100 g/l) (Dissolve once generated precipitate.)	Prepare when it is needed.
Chlorine water	Saturated	Pass chlorine through water until it is saturated.	Store in a brown colored bottle. About 0.6 % at 25 °C . About 0.7 % at 20 °C .
Hydrogen peroxide	3 %	Hydrogen peroxide (JIS K 8230) 10 g + water (→ 100 g)	Prepare when it is needed.
Potassium permanganate solution	50	Potassium permanganate (JIS K 8247) 5 g + water (→ 100 ml) (In case of solution with 1 g/l or less, prepare when it is needed.)	Store in a brown colored glass bottle.

Table 4 Reagent solution (Continued)

Name	Concentration (g/l)	Preparation method	Remarks
Potassium periodate solution	Saturated	Potassium periodate (JIS K 8249) 0.66 g + water (→ 100 ml)	
8-quinolinol solution		8-quinolinol (JIS K 8775) 2 g + acetic acid (1+1) 12 ml + water (→ 100 ml)	Store in a brown colored glass bottle.
Citric acid solution	100	Citric acid monohydrate (JIS K 8283) 10.9 g + water (→ 100 ml)	
Diammonium hydrogen citrate solution	200 100	Diammonium hydrogen citrate (JIS K 8284) 20 g + water (→ 100 ml) Diammonium hydrogen citrate 10 g + water (→ 100 ml)	
Diammonium hydrogen citrate-EDTA2Na solution (for copper test)		Diammonium hydrogen citrate 20 g + EDTA2Na 5 g + water (→ 100 ml) + NaDDTC solution (1 g/l) 1 ml + butyl acetate 10 ml → shake violently for 30 s → let it stand → separate water layer" (Repeat until butyl acetate layer gives no coloring.)	
Acetic acid (for nonaqueous titration)		The following test shall be satisfied. Aniline (JIS K 8042) 1 g + acetic acid (→ 100 ml) (A solution) A solution 25 ml (take accurately) → titrate potentiometrically with 0.1 mol/l perchloric acid (acetic acid solvent) amount of titrant V_1 ml A solution 25 ml (take accurately) + acetic acid 75 ml → titrate potentiometrically with 0.1 mol/l perchloric acid (acetic acid solvent) amount of titrant V_2 ml Difference between V_1 and V_2 shall be 0.1 ml or less.	
Acetic acid—ammonium acetate solution		Acetic acid (1+1) 114 ml + water 800 ml + ammonium acetate (JIS K 8359) 77 g + water (→ 1 l)	
Ammonium acetate solution	250	Ammonium acetate 25 g + water (→ 100 ml)	
Calcium acetate solution	100	Calcium acetate monohydrate (JIS K 8364) 11.1 g + water (→ 100 ml)	Prepare when it is needed.
Sodium acetate solution	200 100	Sodium acetate trihydrate (JIS K 8371) 33.2 g + water (→ 100 ml) Sodium acetate trihydrate 16.6 g + water (→ 100 ml)	

Table 4 Reagent solution (Continued)

Name	Concentration (g/l)	Preparation method	Remarks
Sodium acetate solution (for iron test)		(1) Way to make column Pass sodium chloride solution (100 g/l) through strongly acidic cation-exchange resin (H type) given in (1) of 5.33 at the rate of 5 ml/min to 10 ml/min (until effluent shows neutral by methyl orange test) → pass water at the rate of 5 ml/min to 10 ml/min (until effluent gives almost no chloride reaction) control water level 1 mm to 2 mm upper than upper face of resin layer (While this procedure, liquid level shall be kept not to become lower than the face of resin layer.) (2) Preparation Sodium acetate solution (100 g/l) 200 ml → pass at the rate of 5 ml/min to 10 ml/min using column at (1) (While this procedure, liquid level shall be kept not to become lower than the face of resin layer.) → discard initial 30 ml to 40 ml effluent → employ the effluent thereafter.	
Sodium acetate solution (for copper test)		Sodium acetate solution (100 g/l) 100 ml + bathocuproine • ethanol solution 1 ml + ethyl acetate 5 ml → shake violently for 30 s → let it stand → separate water layer (Repeat until ethyl acetate layer gives no coloring.)	
Lead (II) acetate solution	200	Lead (II) acetate trihydrate (JIS K 8374) 23.4 g + water (→ 100 ml) + acetic acid 0.1 ml To prepare other concentration (A) than above, calculate mass (x) to be taken as follows, and prepare similarly above. $x \text{ (g)} = A \text{ (g/l)} \div 0.86 \div 10$	
Sodium hypochlorite solution	Available chlorine about 1 %	Determine available chlorine in sodium hypochlorite solution (available chlorine 5 to 12 %) when being used, and dilute with water to its available chlorine to be about 1 %. Determination of available chlorine Sodium hypochlorite solution (available chlorine about 5 % to 12 %) 10 ml + water (→ 200 ml) → take its 20 ml → put in a 300 ml Erlenmeyer flask with joint stopper + water 100 ml + potassium iodide (JIS K 8913) 2 g + acetic acid (1+1) 6 ml → stopper and shake → let it stand in the dark place for about 5 min → titrate with 0.1 mol/l sodium thiosulfate solution (indicator : starch solution) Separately, carry out blank test in the same condition, and correct titrant. One millilitre of 0.1 mol/l sodium thiosulfate solution corresponds to 0.003 545 3 g Cl.	Store in cold and dark place, and use it up in 30 days.

Table 4 Reagent solution (Continued)

Name	Concentration (g/l)	Preparation method	Remarks
3, 5-dinitrosalicylic acid solution		3, 5-dinitrosalicylic acid (purity at least 98 %) 1 g + sodium hydroxide (80 g/l) 20 ml + water 50 ml + (+) -sodium tartarate dihydrate (JIS K 8540) 30 g + water (→ 100 ml)	
2, 4-dinitrophenyl-hydrazine•ethanol solution		2, 4-dinitrophenylhydrazine (dried in a desiccator) 0.1 g + hydrochloric acid 3 ml + ethanol (for test of aldehyde and ketone) (→ 50 ml)	Prepare when it is needed.
Dimethylglyoxime•ethanol solution		Dimethylglyoxime (JIS K 8498) 1 g + ethanol (95) (JIS K 8102) (→ 100 ml)	
Oxalic acid solution	40	Oxalic acid dihydrate (JIS K 8519) 5.6 g + water (→ 100 ml)	
Ammonium oxalate solution	40	Ammonium oxalate monohydrate (JIS K 8521) 4.6 g + water (→ 100 ml)	
Bromine water	Saturated	Bromine (JIS K 8529) 3 ml to 4 ml + water (→ 100 ml) → shake violently → let it stand → employ supernatant solution	Store in a brown colored glass bottle. About 3.5 % at 25 °C About 3.6 % at 20 °C
Tartaric acid solution	200	L (+)-tartaric acid (JIS K 8532) 20 g + water (→ 100 ml)	
Potassium nitrate solution	Saturated	Potassium nitrate (JIS K 8548) 33.4 g + water (→ 100 ml)	
Silver nitrate solution	20	Silver nitrate (JIS K 8550) 2 g + water (→ 100 ml)	Store in a brown colored glass bottle.
Magnesium nitrate• ethanol solution		Magnesium nitrate hexahydrate (JIS K 8567) 17 g + ethanol (95) (→ 100 ml)	
Manganese (II) nitrate solution water	50	Manganese (II) nitrate hexahydrate (JIS K 8568) 8 g + water (→ 100 ml)	
Potassium hydroxide solution	200	Potassium hydroxide (JIS K 8574) 23.5 g + water (→ 100 ml) To prepare other concentration (A) than above, calculate mass (x) to be taken as follows, and prepare similarly above. $x \text{ (g)} = A \text{ (g/l)} \div 0.85 \div 10$	Store in a polyethylene bottle.

Table 4 Reagent solution (Continued)

Name	Concentration (g/l)	Preparation method	Remarks
Potassium hydroxide•ethanol solution		Ethanol (for aldehyde and ketone test) 70 ml + water (→ 100 ml) (A solution) potassium hydroxide 11.8 g + A solution (→ 100 ml)	Prepare when it is needed.
Sodium hydroxide solution	300	Sodium hydroxide 31.6 g + water (→ 100 ml) To prepare other concentration (A) than above, calculate mass (x) to be taken as follows, and prepare similarly above. $x \text{ (g)} = A \text{ (g/l)} \div 0.95 \div 10$	Store in a polyethylene bottle.
Ammonium carbonate solution	Saturated	Ammonium carbonate (JIS K 8613) 22 g + water (→ 100 ml)	Store in a polyethylene bottle.
Sodium hydrogencarbonate solution	10	Sodium hydrogencarbonate (JIS K 8622) 1 g + water (→ 100 ml)	
Sodium carbonate solution	100	Sodium carbonate (JIS K 8625) 10 g + water (→ 100 ml)	
Ammonium thiocyanate solution	600	Ammonium thiocyanate (JIS K 9000) 60 g + water (→ 100 ml)	Store in a brown colored glass bottle.
Potassium thiocyanate•1-butanol solution		Potassium thiocyanate (JIS K 9001) 10 g + water 10 ml → warm to 20 °C to 30 °C + 1-butanol (JIS K 8810) (→ 100 ml) → shake → let it stand for one night	
Sodium <i>p</i> -toluenesulfonchloroamide solution	10	Sodium <i>p</i> -toluenesulfonchloroamide trihydrate (JIS K 8318) 1.25 g (equivalent to 12.5 % of available chlorine) + water (→ 100 ml) Formula to obtain available chlorine Available chlorine (%) = purity (%) $\times \frac{35.45}{281.69}$	Prepare when it is needed.
Sodium phenoxide solution		Sodium hydroxide solution (300 g/l) 18 ml → put in a 200 ml beaker → while cooling by cold water + phenol (JIS K 8798) 12.6 g (add gradually) + acetone (JIS K 8034) 4 ml + water (→ 100 ml)	Prepare when it is needed.
Sodium plumbate solution		Sodium hydroxide 50 g + water 100 ml + lead (II) acetate trihydrate 2.5 g + trisodium citrate dihydrate (JIS K 8288) 5 g + water (→ 150 ml)	Store in a polyethylene bottle.
<i>m</i> -phenylenediammonium dichloride solution		<i>m</i> -phenylenediammonium dichloride (purity at least 99 %) 0.5 g + water (→ 100 ml)	

Table 4 Reagent solution (Continued)

Name	Concentration (g/l)	Preparation method	Remarks
Potassium dichromate solution	100	Potassium dichromate (JIS K 8517) 10 g + water (→ 100 ml)	
Ninhydrin • acetone solution	20	Ninhydrin (JIS K 8870) 2 g + acetone (→ 100 ml)	
Ninhydrin • 1-butanol solution	2	Ninhydrin 0.2 g + water-saturated 1-butanol (→ 100 ml)	
Bathocuproine • ethanol solution		Bathocuproine (2, 9-dimethyl-4, 7-diphenyl-1, 10-phenanthroline) (purity at least 99 %) 0.1 g + ethanol (95) (→ 100 ml)	
Bathophenanthroline solution		Bathophenanthroline (4, 7-diphenyl-1, 10-phenanthroline) (purity at least 99 %) 0.1 g + ethanol (99.5) 50 ml + water (→ 100 ml)	
Vanillin • sulfuric acid solution		Vanillin (JIS K 9544) 0.2 g + sulfuric acid 100 ml	Prepare when it needed.
4-pyridine carboxylic acid-pyrazolone solution		3-methyl-1-phenyl-5-pyrazolone (JIS K 9548) 0.3 g + <i>N, N</i> -dimethylformamide (JIS K 8500) 20 ml → dissolve (A solution) Sodium 4-pyridine carboxylate (purity at least 99 %) 2 g + water 70 ml (B solution) A solution + B solution + water (→ 100 ml)	Store in cold and dark place and use it up in 20 days.
1, 10-phenanthroline solution	2	1, 10-phenanthroline chloride monohydrate (JIS K 8202) 0.28 g + ethanol (95) 10 ml + water (→ 100 ml)	Store in a brown colored glass bottle.
Fehling solution		Copper (II) sulfate pentahydrate (JIS K 8983) 34.66 g + water (→ 500 ml) (A solution) (+)-sodium potassium tartarate tetrahydrate (JIS K 8536) 173 g + sodium hydroxide 50 g + water (→ 500 ml) (B solution) When needed, mix A solution and B solution equally.	Store B solution in a polyethylene bottle.
Brucine solution		Brucine <i>n</i> -hydrate 1 g (as dihydrate) + sulfanilic acid (JIS K 8586) 0.1 g + hydrochloric acid (2+1) 5 ml + water (→ 100 ml)	Store in a brown colored glass bottle.
Potassium hexacyanoferrate (II) solution water	50 10	Potassium hexacyanoferrate (II) trihydrate (JIS K 8802) 5.8 g + water (→ 100 ml) Potassium hexacyanoferrate (II) trihydrate 1.2 g + water (→ 100 ml)	Prepare when it is needed.

Table 4 Reagent solution (Continued)

Name	Concentration (g/l)	Preparation method	Remarks
Potassium hexacyanoferrate (III) solution	50	Potassium hexacyanoferrate (III) (JIS K 8801) 5 g + water (→ 100 ml)	Store in a brown colored glass bottle.
Hexamethylenetetramine solution	100	Hexamethylenetetramine (JIS K 8847) 10 g + water (→ 100 ml)	
Sodium pentacyanonitrosylferrate (III) solution	5	Sodium pentacyanonitrosylferrate (III) dihydrate (JIS K 8722) 0.6 g + water (→ 100 ml)	Prepare when it is needed.
Boric acid solution	40	Boric acid (JIS K 8863) 4 g + water 90 ml → dissolve with heating → cool + water (→ 100 ml)	Prepare when it is needed.
Polyvinyl alcohol solution	5	Polyvinyl alcohol (JIS K 9550) 0.5 g + water (→ 100 ml)	
Magnesia mixture		Magnesium chloride hexahydrate (JIS K 8159) 50 g + ammonium chloride 100 g + water 800 ml → dissolve + phenolphthalein solution 3 drops + aqueous ammonia (2+3) (until dark red appears) → let it stand for 2 days and nights → filter → filtrate + water (→ 1 l) + hydrochloric acid (1+10) (control pH 6 to 7)	Prepare when it is needed.
Hexaammonium heptamolybdate solution	100	Hexaammonium heptamolybdate tetrahydrate (JIS K 8905) 21.2 g + warm water 100 ml → dissolve → cool + water (→ 200 ml) (use polyethylene bottle)	Store in a polyethylene bottle.
Hexaammonium heptamolybdate solution (for phosphoric acid determination)		Hexaammonium heptamolybdate tetrahydrate 10.6 g + water 70 ml + aqueous ammonia 7ml → dissolve (never heat) + water (→ 100 ml) → filter → filtrate + water (→ 200 ml) + sulfuric acid (1+5) 10 ml → “transfer into separatory funnel + 1-butanol 30 ml → shake violently for 1 min to 2 min → let it stand → separate 1-butanol layer → separate water layer (preserve) → 1-butanol layer + [wash with sulfuric acid (1+5) 15 ml → remove sulfuric acid layer] (try twice) → 1-butanol layer + tin (I) chloride solution (for phosphoric acid determination) 15 ml → shake for 30 s → let it stand” (If 1-butanol layer shows blue, repeat procedures in “ ” on the preserved water layer.)	Store in a polyethylene bottle.

Table 4 Reagent solution (Continued)

Name	Concentration (g/l)	Preparation method	Remarks
Hexaammonium heptamolybdate-ascorbic acid solution		Hexaammonium heptamolybdate tetrahydrate 6 g + dipotassium bis [(+)-tartarato]diantimonate (III) trihydrate (JIS K 8533) 0.24 g + water 300ml + sulfuric acid (2+1) 120 ml + ammonium amidesulfate 5 g + water (→ 500 ml) (A solution) L (+) -ascorbic acid (JIS K 9502) 7.2 g + water (→ 100 ml) (B solution) When using, mix A and B solution at volumetric rate of 5 : 1	Store B solution in dark place at 0 °C to 10 °C .
Zinc iodite starch solution		Starch (soluble) (JIS K 8659) 2 g + water 20 ml → while agitating → add into 200 ml hot water → boil for about 1 min → cool + zinc chloride (JIS K 8111) 10 g → dissolve + [zinc powder (JIS K 8013) 0.5 g + water 5 ml + iodine 1 g → agitate → filter → filtrate (colorless transparent liquid)] + water (→ 500 ml) → filter Confirmation test This solution 1 ml + water 50 ml + sulfuric acid (1+5) 3 ml.....No blue appears → further, 0.01 mol/l potassium iodate solution 0.05 ml.....Blue appears	Store in a brown colored bottle.
Potassium iodide	200 100	Potassium iodide 20 g + water (→ 100 ml) Potassium iodide 10 g + water (→ 100 ml)	Prepare when it is needed.
Potassium iodide-starch solution		Starch (soluble) 1 g + water 10 ml → while agitating → add it into 200 ml hot water → boil for about 1 min → cool → filter + potassium iodide 1 g (At this time, blue shall not appear.)	Prepare when it is needed.
Sodium sulfide • glycerol solution		Sodium sulfide nonahydrate (JIS K 8949) 5 g + [glycerol (JIS K 8295) 30 ml + water 10 ml] → dissolve → let it stand → employ supernatant solution	Store in cold place and use it up in 3 months.
Sodium sulfide solution	40	Sodium sulfide nonahydrate 1.2 g + water (→ 10 ml)	Prepare when it is needed.
Iron (II) sulfate solution	100	Iron (II) sulfate heptahydrate (JIS K 8978) 18.3 g + sulfuric acid (1+5) 30 ml + water (→ 100 ml)	Prepare when it is needed.
Copper (II) sulfate solution	100	Copper (II) sulfate pentahydrate 15.6 g + water (→ 100 ml)	
Sodium sulfate solution	100	Sodium sulfate (JIS K 8987) 10 g + water (→ 100 ml)	

Table 4 Reagent solution (Concluded)

Name	Concentration (g/l)	Preparation method	Remarks
Manganese (II) sulfate solution		Manganese (II) sulfate pentahydrate (JIS K 8997) 90 g + water 200 ml + phosphoric acid 175 ml + sulfuric acid (1+1) 350 ml + water (→1000ml)	Use it 3 or 4 days later after preparation.
Diammonium hydrogen phosphate solution	130	Diammonium hydrogen phosphate (JIS K 9016) 13 g + water (→ 100 ml)	
Disodium hydrogen phosphate solution	50	Disodium hydrogen phosphate • 12 water (JIS K 9019) 12.6 g + water (→ 100 ml)	
Resorsinol • hydrochloric acid solution		Newly sublimated resorsinol (JIS K 9032) 1 g + hydrochloric acid (→ 100 ml)	Store in a brown colored bottle.

4.3 Reference solution Reference solutions shall be as follows, but such as copper reference solution specified in Japanese Industrial Standard may be also available.

In preparation method, the description such as “reagent 4.40 g + water (→ 1 000 ml) ” means that “Dissolve 4.40 g of reagent in water, transfer it into a 1 000 ml volumetric flask, and add water up to the marked line”, and the description as “stock solution 10 ml” means that 10 ml of stock solution shall be taken using a transfer pipet.

When such treatment as heating is to be carried out, treat it with a beaker, and then transfer it in a volumetric flask. The reference solution, which has been prepared by diluting stock solution, has diluted concentration; therefore be careful about the change of concentration caused by adsorption on the wall of container while it is stored.

- (1) **Reference solution (for general use)** Reference solution (for general use) shall follow Table 5. This can be used for general test as turbidimetry or colorimetry.
- (2) **Reference solution (for atomic absorption spectrometry • flame photometry)** Reference solution (for atomic absorption spectrometry • flame photometry) shall follow Table 6. This can be used for the test by instrumental analysis such as atomic absorption spectrometry or flame photometry.

Table 5 Reference solution (for general use)

Name	Reagents	Preparation		Remarks (⁵)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Zinc reference solution (Zn)	Zinc sulfate heptahydrate (JIS K 8953) $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$	4.4 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	JIS K 0011
Nitrite reference solution (NO_2)	Sodium nitrite (JIS K 8019) NaNO_2	1.50 g (equivalent as 100 %) + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	Prepare stock solution and reference solution when they are needed. JIS K 0032
Acetone reference solution (CH_3COCH_3)	Acetone (JIS K 8034) CH_3COCH_3	1.00 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	Prepare stock solution and reference solution when they are needed.
Sulfite reference solution (SO_3)	Sodium sulfite (JIS K 8061) Na_2SO_3	1.57 g (equivalent as 100 %) + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + sodium hydroxide solution (100 g/l) 1.5 ml + water ($\rightarrow$ 1 000 ml)	Prepare stock solution and reference solution when they are needed.
Aluminum reference solution (Al)	Aluminium potassium sulfate • 12 water (JIS K 8255) $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	17.6 g + hydrochloric acid (2+1) 15 ml + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	Store in a polyethylene bottle.
Antimony reference solution (Sb)	Bis [(+)- tartrato] diantimonate (III) dipotassium trihydrate (JIS K 8533) $\text{C}_8\text{H}_4\text{K}_2\text{O}_{12}\text{Sb}_2 \cdot 3\text{H}_2\text{O}$	2.74 g + tartaric acid solution (200 g/l) 25 ml + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + tartaric acid solution (200 g/l) + water ($\rightarrow$ 1 000 ml)	JIS K 0025
Ammonium reference solution (NH_4)	Ammonium chloride (JIS K 8116) NH_4Cl	2.97 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	
3-methyl-1-butanol reference solution ($\text{C}_5\text{H}_{12}\text{O}$)	3-methyl-1-butanol (JIS K 8051) $\text{C}_5\text{H}_{12}\text{O}$	1.00 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	
Chloride reference solution (Cl)	Sodium chloride (JIS K 8150) NaCl	1.65 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	JIS K 0029

Note (⁵) The standard number in Remarks is the standard number of reference solution stated in Japanese Industrial Standard.

Table 5 Reference solution (for general use) (Continued)

Name	Reagents	Preparation		Remarks (°)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Calcium reference solution (Ca)	Calcium carbonate (JIS K 8617) CaCO_3	2.50 g + water 50 ml + hydrochloric acid (2+1) 15 ml → heat with no boiling (to eliminate carbon dioxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml) [0.1 mgCa/ml] Stock solution 100 ml + water (→ 1 000 ml)	Store in a polyethylene bottle.
Silver reference solution (Ag)	Silver nitrate (JIS K 8550) AgNO_3	1.57 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Store in a brown colored glass bottle.
D (+)- glucose reference solution ($\text{C}_6\text{H}_{12}\text{O}_6$)	D(+)-glucose (JIS K 8824) $\text{C}_6\text{H}_{12}\text{O}_6$	1.00 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	
Silicate reference solution (as SiO_2)	Silicon dioxide (JIS K 8885) SiO_2	0.100 g (strongly heated at 900 °C to 1 000 °C) + sodium carbonate 1 g → fuse in platinum crucible → cool + water (→ 100 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Store in a polyethylene bottle.
Silicon reference solution (Si)	Silicon dioxide (JIS K 8885) SiO_2	0.214 g (strongly heated at 900 °C to 1 000 °C) + sodium carbonate 1 g → fuse in platinum crucible → cool + water (→ 100 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Store in a polyethylene bottle.
Cobalt reference solution (Co)	Diammonium cobalt (II) sulfate hexahydrate (JIS K 8969) $\text{Co}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	6.71 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	JIS K 0014
Cyanide reference solution (CN)	Potassium cyanide (JIS K 8443) KCN	2.50 g (equivalent as 100 %) + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Prepare stock solution and reference solution when it is needed.

Table 5 Reference solution (for general use) (Continued)

Name	Reagents	Preparation		Remarks (°)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Oxalic acid reference solution (C ₂ H ₂ O ₄)	Oxalic acid dihydrate (JIS K 8519) C ₂ H ₂ O ₄ · 2H ₂ O	1.40 g + water (→ 1 000 ml)	[0.1 mg C ₂ H ₂ O ₄ /ml] Stock solution 100 ml + water (→ 1 000 ml)	Prepare stock solution and reference solution when it is needed.
Tartrate reference solution (C ₄ H ₄ O ₆)	(+)-potassium tartrate- water (2/1) (JIS K 8535) 2C ₄ H ₄ O ₆ K ₂ · H ₂ O	1.59 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	
Nitrate reference solution (NO ₃)	Potassium nitrate (JIS K 8548) KNO ₃	1.63 g (dried at 110 °C) + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	JIS K 0031
Mercury reference solution (Hg)	Mercury (II) chloride (JIS K 8139) HgCl ₂	1.35 g + nitric acid(1+2) 25 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid(1+2) 25 ml +water (→ 1 000 ml)	Use up reference solution in one month.
Tin reference solution (Sn)	Tin (II) chloride dihydrate (JIS K 8136) SnCl ₂ · 2H ₂ O	1.90 g (equivalent as 100 %) +hydrochloric acid (2+1) 8 ml + water (→ 1 000 ml)	Stock solution 10 ml+ hydrochloric acid(2+1) 2 ml + water (→ 1 000 ml)	Prepare stock solution and reference solution when it is needed.
Selenium reference solution (Se)	Sodium selenite (JIS K 8036) Na ₂ SeO ₃	2.19 g (equivalent as 100 %) + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	
			[0.1 mgSe/ml] Stock solution 100 ml + water (→ 1 000 ml)	
Carbonate reference solution (CO ₃)	Sodium carbonate (JIS K 8625) Na ₂ CO ₃	1.77 g + water (→ 1 000 ml)	[0.1 mgCO ₃ /ml] Stock solution 100 ml + water (→ 1 000 ml)	
Thiocyanate reference solution (SCN)	Ammonium thiocyanate (JIS K 9000) NH ₄ SCN	1.31 g (equivalent as 100 %) + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Store in a brown colored glass bottle.
Titanium reference solution (Ti)	Titanium (IV) oxide (JIS K 8703) TiO ₂	0.167 g + ammonium sulfate 5 g + sulfuric acid 10 ml → heat to dissolve → cool + water (→ 100 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Prepare reference solution when it is needed.

Table 5 Reference solution (for general use) (Continued)

Name	Reagents	Preparation		Remarks (°)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Nitrogen reference solution (N)	Potassium nitrate (JIS K 8548) KNO_3	7.22 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	
Iron(II) reference solution (Fe^{2+})	Ammonium iron (II) sulfate hexahydrate (JIS K 8979) $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	7.02 g + hydrochloric acid (2+1) 5 ml + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + hydrochloric acid (2+1) 5 ml + water ($\rightarrow$ 1 000 ml)	Prepare stock solution and reference solution when they are used, and store in brown colored glass bottles.
Iron(III) reference solution (Fe^{3+})	Ammonium iron (III) sulfate • 12 water (JIS K 8982) $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	8.63 g + hydrochloric acid (2+1) 3 ml + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + hydrochloric acid (2+1) 3 ml + water ($\rightarrow$ 1 000 ml) [0.001 mgFe/ml] Reference solution 10 ml + hydrochloric acid (2+1) 3 ml + water ($\rightarrow$ 100 ml)	Store in a brown colored glass bottle. JIS K 0016
Copper reference solution (Cu)	Copper (II) sulfate pentahydrate (JIS K 8983) $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$	3.93 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml) [0.1 mgCu/ml] Stock solution 100 ml + water ($\rightarrow$ 1 000 ml)	JIS K 0010
Sodium reference solution (Na)	Sodium chloride (JIS K 8150) NaCl	2.54 g + water ($\rightarrow$ 1 000 ml)	[0.1 mgNa/ml] Stock solution 100 ml + water ($\rightarrow$ 1 000 ml)	Store in a polyethylene bottle.
Lead reference solution (Pb)	Lead (II) nitrate (JIS K 8563) $\text{Pb}(\text{NO}_3)_2$	1.60 g + nitric acid (1+2) 1 ml + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml) [0.1 mgPb/ml] Stock solution 100 ml + water ($\rightarrow$ 1 000 ml)	Prepare reference solution when it is needed. JIS K 0015
Dichromate reference solution (Cr_2O_7)	Potassium dichromate (JIS K 8517) $\text{K}_2\text{Cr}_2\text{O}_7$	1.36 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	

Table 5 Reference solution (for general use) (Continued)

Name	Reagents	Preparation		Remarks (°)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Nickel reference solution (Ni)	Ammonium nickel (II) sulfate hexahydrate (JIS K 8990) $(\text{NH}_4)_2\text{Ni}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$	6.73 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	JIS K 0013
Barium reference solution (Ba)	Barium chloride dihydrate (JIS K 8155) $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$	1.78 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	
Bismuth reference solution (Bi)	Bismuth nitrate pentahydrate (JIS K 8566) $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$	2.32 g + nitric acid (1+2) 10 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 10 ml + water (→ 1 000 ml)	
Arsenic reference solution (As)	Diarsenic trioxide (JIS K 8044) As_2O_3	1.32 g + sodium hydroxide (100 g/l) 6 ml + water 500 ml + hydrochloric acid (1+3) (adjust pH to 3 to 5) + water (→ 1 000 ml)	[0.001 mgAs/ml] Stock solution 25 ml + water (→ 250 ml) This solution 10 ml + water (→ 1 000 ml)	JIS K 0026
Phenol reference solution ($\text{C}_6\text{H}_5\text{OH}$)	Phenol (JIS K 8798) $\text{C}_6\text{H}_5\text{OH}$	1.00 g + water (→ 1000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Prepare stock solution and reference solution when it is needed.
Fluoride reference solution (F)	Sodium fluoride (JIS K 8821) NaF	2.21 g + water (→ 1 000 ml)	[0.1 mgF/ml] Stock solution 100 ml + water (→ 1 000 ml)	Store in a polyethylene bottle. JIS K 0030
Boron reference solution (B)	Boric acid (JIS K 8863) H_3BO_3	5.72 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Prepare reference solution when it is needed.
Formaldehyde reference solution (HCHO)	Formaldehyde liquid (JIS K 8872) HCHO	2.86 g (equivalent as 35 % HCHO) + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Prepare stock solution and reference solution when it is needed.
Magnesium reference solution (Mg)	Magnesium sulfate heptahydrate (JIS K 8995) $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	10.1 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	
			[0.1 mgMg/ml] Stock solution 100 ml + water (→ 1 000 ml)	

Table 5 Reference solution (for general use) (Concluded)

Name	Reagents	Preparation		Remarks (5)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Manganese reference solution (Mn)	Manganese (II) chloride tetrahydrate (JIS K 8160) $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$	3.60 g + hydrochloric acid (2+1) 15 ml + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml) [0.1 mgMn/ml] Stock solution 100 ml + water ($\rightarrow$ 1 000 ml)	JIS K 0027
Molybdenum reference solution (Mo)	Hexaammonium heptamolybdate tetrahydrate (JIS K 8905) $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$	1.84 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	Store in a polyethylene bottle.
Iodide reference solution (I)	Potassium iodide (JIS K 8913) KI	1.31 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	Prepare stock solution and reference solution when it is needed.
Sulfide reference solution (S)	Sodium sulfide nonahydrate (JIS K 8949) $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$	7.49 g (equivalent as 100 %) + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	Prepare stock solution and reference solution when it is needed.
Sulfate reference solution (SO_4)	Potassium sulfate (JIS K 8962) K_2SO_4	1.81 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	(JIS K 0028)
Phosphate reference solution (PO_4)	Potassium dihydrogenphosphate (JIS K 9007) KH_2PO_4	1.43 g + water ($\rightarrow$ 1 000 ml)	Stock solution 10 ml + water ($\rightarrow$ 1 000 ml)	

Table 6 Reference solution (for atomic absorption spectrometry • flame photometry)

Name	Reagents	Preparation		Remarks (°)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Zinc reference solution (Zn)	Zinc (JIS H 2107) Zn (at least 99.9 %)	1.00 g + nitric acid (1+2) 25 ml → heat to dissolve → boil (for removing nitrogen oxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	JIS K 0011
	Zinc sulfate heptahydrate (JIS K 8953) $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$	4.40 g + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
Aluminum reference solution (Al)	Aluminum Al (at least 99.9 %)	1.00 g + hydrochloric acid (2+1) 30 ml → heat to dissolve → cool + water (→ 1 000 ml)	Stock solution 10 ml + hydrochloric acid (2+1) 15 ml + water (→ 1 000 ml)	Store in a polyethylene bottle.
	Aluminum potassium sulfate • 12 water (JIS K 8255) $\text{AlK}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$	1.76 g + water 10 ml + hydrochloric acid (2+1) 15 ml + water (→ 1 000 ml)	Stock solution 10 ml + hydrochloric acid (2+1) 15 ml + water (→ 1 000 ml)	
Antimony reference solution (Sb)	Antimony (Ⅲ) chloride (JIS K 8400) SbCl_3	1.87 g (equivalent as 100 %) + water 20 ml + hydrochloric acid (2+1) 53 ml → heat to dissolve → cool + water (→ 1 000 ml)	Stock solution 10 ml + hydrochloric acid (2+1) 3 ml + water (→ 1 000 ml)	JIS K 0025
Cadmium reference solution (Cd)	Cadmium (JIS H 2113) Cd (at least 99.9 %)	1.00 g + nitric acid (1+2) 25 ml → heat to dissolve → boil (for removing nitrogen oxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	JIS K 0012
	Cadmium sulfate-water (3/8) (JIS K 8961) $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$	2.28 g + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
Potassium reference solution (K)	Potassium chloride (JIS K 8121)	1.91 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Store in a polyethylene bottle.
			[0.1 mgK/ml] Stock solution 100 ml + water (→ 1 000 ml)	

Table 6 Reference solution (for atomic absorption spectrometry • flame photometry) (Continued)

Name	Reagents	Preparation		Remarks ⁽⁵⁾
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Calcium reference solution (Ca)	Calcium carbonate (JIS K 8617) CaCO ₃	2.50 g + water 50 ml + hydrochloric acid (2+1) 15 ml → heat with no boiling (remove carbon dioxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + hydrochloric acid (2+1) 15 ml + water (→ 1 000 ml) [0.1 mgCa/ml] Stock solution 100 ml + hydrochloric acid (2+1) 15 ml + water (→ 1 000 ml)	Store in a polyethylene bottle.
Silver reference solution (Ag)	Silver nitrate (JIS K 8550) AgNO ₃	1.57 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Store in a brown colored glass bottle.
Chromium reference solution (Cr)	Potassium dichromate (JIS K 8517) K ₂ Cr ₂ O ₇	2.83 g + water 50 ml + nitric acid (1+2) 5 ml + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	JIS K 0024
Cobalt reference solution (Co)	Cobalt Co (at least 99.5 %)	1.00 g + nitric acid (1+2) 25 ml → heat to dissolve → boil (for removing nitrogen oxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	JIS K 0014
	Diammonium cobalt (II) sulfate hexahydrate (JIS K 8969) Co(NH ₄) ₂ (SO ₄) ₂ • 6H ₂ O	6.71 g + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
Mercury reference solution (Hg)	Mercury (II) chloride (JIS K 8139) HgCl ₂	1.35 g + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	[0.001 mgHg/ml] Stock solution 25 ml + water (→ 250 ml) This solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Prepare reference solution when it is needed.
Strontium reference solution (Sr)	Strontium nitrate (JIS K 8554) Sr(NO ₃) ₂	2.42 g + water (→ 1 000 ml)	[0.1 mgSr/ml] Stock solution 100 ml + water (→ 1 000 ml)	

Table 6 Reference solution (for atomic absorption spectrometry • flame photometry) (Continued)

Name	Reagents	Preparation		Remarks (5)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Titanium reference solution (Ti)	Titanium (IV) oxide (JIS K 8703) TiO ₂	0.167 g + ammonium sulfate 5 g + sulfuric acid 10 ml → heat to dissolve → cool + water (→ 100 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Prepare reference solution when it is needed.
Iron reference solution (Fe)	Iron Fe (at least 99.5 %)	1.00 g + nitric acid (1+2) 30 ml → heat to dissolve → boil (for removing nitrogen oxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Store in a brown col- ored glass bottle. JIS K 0016
	Ammonium iron (III) sulfate • 12 water (JIS K 8982) FeNH ₄ (SO ₄) ₂ • 12 H ₂ O	8.63 g + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
Copper reference solution (Cu)	Copper (JIS K 8660) Cu (at least 99.9 %)	1.00 g + nitric acid (1+2) 25 ml → heat to dissolve → boil (for removing nitrogen oxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	JIS K 0010
	Copper (II) sulfate pentahydrate (JIS K 8983) CuSO ₄ • 5H ₂ O	3.93 g + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
Sodium reference solution (Na)	Sodium chloride (JIS K 8150) NaCl	2.54 g + water (→ 1 000 ml)	Stock solution 10 ml + water (→ 1 000 ml)	Store in a polyethylene bottle.
			[0.1 mgNa/ml] Stock solution 100 ml + water (→ 1 000 ml)	

Table 6 Reference solution (for atomic absorption spectrometry • flame photometry) (Continued)

Name	Reagents	Preparation		Remarks (°)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Lead reference solution (Pb)	Lead (JIS H 2105) Pb (at least 99.9 %)	1.00 g + nitric acid (1+2) 25 ml → heat to dissolve → boil (for removing nitrogen oxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	JIS K 0015
	Lead (II) nitrate (JIS K 8563) Pb (NO ₃) ₂	1.60 g + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
Nickel reference solution (Ni)	Nickel (JIS H 2104) Ni (at least 99.9 %)	1.00 g + nitric acid (1+2) 25 ml → heat to dissolve → boil (for removing nitrogen oxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	JIS K 0013
	Ammonium nickel (II) sulfate hexahydrate (JIS K 8990) (NH ₄) ₂ Ni(SO ₄) ₂ • 6H ₂ O	6.73 g + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
Barium reference solution (Ba)	Barium chloride dihydrate (JIS K 8155) BaCl ₂ • 2H ₂ O	1.78 g + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
Bismuth reference solution (Bi)	Bismuth Bi (at least 99.9 %)	1.00 g + water 20 ml + nitric acid (1+2) 25 ml → heat to dissolve → boil (for removing nitrogen oxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 1.5 ml + water (→ 1 000 ml)	JIS K 0017
Arsenic reference solution (As)	Diarsenic trioxide (JIS K 8044) As ₂ O ₃	1.32 g + sodium hydroxide solution (100 g/l) 6 ml + water 500 ml + hydrochloric acid (1 + 3) (adjust pH 3 to 5) + water (→ 1 000 ml)	[0.001 mgAs/ml] Stock solution 25 ml + water (→ 250 ml) This solution 10 ml + water (→ 1 000 ml)	JIS K 0026

Table 6 Reference solution (for atomic absorption spectrometry • flame photometry) (Concluded)

Name	Reagents	Preparation		Remarks (5)
		Stock solution (1 mg/ml)	Reference solution (0.01 mg/ml)	
Magnesium reference solution (Mg)	Magnesium (JIS H 2150) Mg (at least 99.9 %)	1.00 g + hydrochloric acid (2 + 1) 23 ml → dissolve + water (→ 1 000 ml)	Stock solution 10 ml + hydrochloric acid (2+1) 15 ml + water	JIS K 0027
	Magnesium sulfate heptahydrate (JIS K 8995) MgSO ₄ • 7H ₂ O	10.1 g + hydrochloric acid (2 + 1) 15 ml + water (→ 1 000 ml)	Stock solution 10 ml + hydrochloric acid (2+1) 15 ml + water	
Manganese reference solution (Mn)	Manganese Mn (at least 99.9 %)	1.00 g + water 20 ml + nitric acid (1 + 2) 25 ml → heat to dissolve → boil (for removing nitrogen oxide) → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
	Potassium permanganate (JIS K 8247) KMnO ₄	2.88 g + water 100 ml + nitric acid (1 + 2) 30 ml + hydrogen peroxide (3 %) 60 ml (add gently to decolor) → boil for about 2 min → cool + water (→ 1 000 ml)	Stock solution 10 ml + nitric acid (1+2) 25 ml + water (→ 1 000 ml)	
	Manganese (II) chloride tetrahydrate (JIS K 8160) MnCl ₂ • 4H ₂ O	3.60 g + hydrochloric acid (2 + 1) 15 ml + water (→ 1 000 ml)	Stock solution 10 ml + hydrochloric acid (2+1) 15 ml + water (→ 1 000 ml)	

4.4 Indicator In preparation method, the description such as “reagent 0.10 g + name of solvent (→ 100 ml)” means that 0.10 g of reagent is dissolved in solvent to make total 100 ml. If it hardly dissolves, reagent shall be ground in an agate mortar, and after adding solvent, dissolve it by heating.

The name of solvent is omitted when other solvent than water was used.

When diluent powder is used, grind well the reagent, and then mix them.

Table 7 Indicator for neutralization titration

Name	Preparation method	Transition interval(pH)	Remarks
Bromophenol blue solution	Bromophenol blue (JIS K 8844) 0.10 g + ethanol (95) (JIS K 8102) 50 ml + water (→ 100 ml)	Yellow 3.0 to 4.6 bluish violet	Store in a brown colored glass bottle
Methyl orange solution	Methyl orange (JIS K 8893) 0.10 g + water (→ 100 ml)	Red 3.1 to 4.4 reddish yellow	Store in a brown colored glass bottle
Alizarin red S solution	Alizarin red S (JIS K 8057) 0.10 g + water (→ 100 ml)	Yellow 3.7 to 5.2 yellowish red	Store in a brown colored glass bottle
Bromocresol green solution	Bromocresol green (JIS K 8840) 0.10 g + ethanol (95) 50 ml + water (→ 100 ml)	Yellow 3.8 to 5.4 blue	Store in a brown colored glass bottle
Methyl red solution	Methyl red (JIS K 8896) 0.10 g + ethanol (95) (→ 100 ml)	Red 4.2 to 6.2 yellow	Store in a brown colored glass bottle
Bromothymol blue solution	Bromothymol blue (JIS K 8842) 0.10 g + ethanol (95) 50 ml + water (→ 100 ml)	Yellow 6.0 to 7.6 blue	Store in a brown colored glass bottle
Phenolphthalein solution	Phenolphthalein (JIS K 8799) 1.0 g + ethanol (95) 90 ml + water (→ 100 ml)	Colorless 7.8 to 10.0 brilliant red	
Thymol blue solution	Thymol blue (JIS K 8643) 0.10 g + ethanol (95) 50 ml + water (→ 100 ml)	Yellow 8.0 to 9.6 blue	Store in a brown colored glass bottle (red 1.2 to 2.8 yellow)
Thymolphthalein solution	Thymolphthalein (JIS K 8642) 0.10 g + ethanol (95) (→ 100 ml)	Colorless 8.6 to 10.5 blue	Store in a brown colored glass bottle
Bromocresol green-methyl red solution	Bromocresol green 0.15 g + methyl red 0.10 g + ethanol (95) (→ 200 ml)	Red via reddish violet (5.0) to green	Store in a brown colored glass bottle
Methyl red-methylene blue solution	Methyl red 0.10 g + methylen blue (JIS K 8897) 0.10 g + ethanol (95) (→ 200 ml)	Reddish violet via grayish blue (5.4) to green	Store in a brown colored glass bottle
Neutral red-bromothymol blue solution	Neutral red 0.10 g + bromothymol blue 0.10 g + ethanol (95) (→ 200 ml)	Red via pale red (7.1) via grayish green to blue	Store in a brown colored glass bottle

Table 8 Solution for precipitation titration, oxidation-reduction titration, and complexometric titration

Name	Preparation method	Remarks
BANASS-brilliant yellow solution	BANASS 0.10 g + brilliant yellow 0.020 g + 0.1 mol/l sodium hydroxide solution 3.0 ml + water 7 ml + methanol (JIS K 8891) (→ 100 ml)	Store in a brown colored glass bottle
HSNN diluent powder	HSNN (JIS K 8776) 0.20 g + potassium sulfate (JIS K 8962) 10 g	Store in a brown colored glass bottle
Uranin solution	Uranin (JIS K 8830) 0.20 g + water (→ 100 ml)	Store in a brown colored glass bottle
Eriochrome black T diluent powder	Eriochrome black T (JIS K 8736) 0.10 g + sodium chloride (JIS K 8150) 10 g	Store in a brown colored glass bottle
Eriochrome black T solution	Eriochrome black T 0.5 g + methanol (→ 100 ml) + hydroxylammonium chloride (JIS K 8201) 0.5 g	Store in a brown colored glass bottle
Xylenol orange solution	Xylenol orange (JIS K 9563) 0.10 g + water (→ 100 ml)	Store in a brown colored glass bottle
Dithizone solution	Dithizone (JIS K 8490) 0.025 g + ethanol (95) (JIS K 8102) (→ 100 ml)	Prepare when it is needed
Iron (III) nitrate solution	Iron (III) nitrate nonahydrate (JIS K 8559) 7 g + sulfuric acid (1+49) (→ 100 ml)	
Starch solution	Starch (soluble) (JIS K 8659) 1.0 g + water 10 ml → add into 200 ml hot water while being agitating → boil for about 1 min → cool	Keep in a cold place and use it up in 10 days
4-(2-pyridylazo) resorcinol solution	4-(2-pyridylazo) resorcinol 0.10 g + ethanol (95) (→ 100 ml)	Store in a brown colored glass bottle
Ferroin solution	Iron (II) sulfate heptahydrate (JIS K 8978) 0.70 g + water 70 ml + 1,10-phenanthroline chloride monohydrate (JIS K 8202) 1.78 g + water (→ 100 ml)	Store in a brown colored glass bottle
Murexide diluent powder	Murexide 0.10 g + potassium sulfate 10 g	Store in a brown colored glass bottle
Potassium iodide-starch test paper	Immerse filter paper in potassium iodide-starch solution, and dry it at ordinary temperature with shielding from light.	Store in a brown colored glass bottle
Ammonium iron (III) sulfate solution	Ammonium iron (III) sulfate • 12 water (JIS K 8982) 10 g + [nitric acid (1+2) 10 ml + water 80 ml]	

4.5 Solution for titration

(1) Zinc solution

(1.1) 0.1 mol/l zinc solution (6.539 g Zn/l)

- (a) **Preparation** Wash required amount of zinc, the reference material for volumetric analysis specified in **JIS K 8005**, with hydrochloric acid (1+3), then water, ethanol (99.5) specified in **JIS K 8101**, and diethyl ether specified in **JIS K 8103**, immediately put it in a desiccator, and allow it to stand for about 12 hours. Weigh its 3.3 g to the nearest 0.1 mg, transfer it into a 300 ml Erlenmeyer flask (⁶), add 25 ml of water and 40 ml of nitric acid (1+2), heat it on a water bath with a condenser to make it dissolve. Then, heat it gently to eliminate nitrogen oxide, allow it to cool, transfer it into a 500 ml volumetric flask, wash the Erlenmeyer flask and condenser used for dissolving with water, put the washings into the above 500 ml volumetric flask, add water up to the marked line, mix them, and store it in an airtight container.

Note (⁶) Employ the Erlenmeyer flask attachable with an interchangeable ground condenser.

- (b) **Calculation** Calculate the factor according to the following formula.

$$f = \frac{a}{3.269\ 5} \times \frac{A}{100}$$

where,

f : factor of 0.1 mol/l zinc solution

a : mass of zinc weighed out (g)

A : purity of zinc (%)

3.269 5: equivalent amount of zinc in 500 ml of 0.1 mol/l zinc solution (g)

(1.2) 0.05 mol/l zinc solution (3.270 g Zn/l)

- (a) **Preparation** Follow (1.1) (a). In this case, weigh 1.7 g of zinc to the nearest of 0.1 mg, and use 25 ml of nitric acid (1+2).

- (b) **Calculation** Calculate the factor according to the following formula.

$$f = \frac{a}{1.634\ 8} \times \frac{A}{100}$$

where,

f : factor of 0.05 mol/l zinc solution

a : mass of zinc weighed out (g)

A : purity of zinc (%)

1.634 8: equivalent amount of zinc in 500 ml of 0.05 mol/l zinc solution (g)

(1.3) 0.01 mol/l zinc solution (0.653 9 g Zn/l)

- (a) **Preparation** Follow (1.1) (a). In this case, weigh 0.33 g of zinc to the nearest of 0.1 mg, and use 25 ml of nitric acid (1+2).

- (b) **Calculation** Calculate the factor according to the following formula.

$$f = \frac{a}{0.326\ 95} \times \frac{A}{100}$$

where, f : factor of 0.01 mol/l zinc solution

a : mass of zinc weighed out (g)

A : purity of zinc (%)

0.326 95: equivalent amount of zinc in 500 ml of 0.01 mol/l zinc solution (g)

(2) **0.5 mol/l sodium nitrite solution** (34.50 g NaNO₂/l)

- (a) **Preparation** Weigh 36 g of sodium nitrite specified in **JIS K 8019**, dissolve it in 1 l water, and store it in an airtight container.

- (b) **Standardization** Place required amount of amidosulfuric acid, the reference material, specified in **JIS K 8005**, for volumetric analysis, into a vacuum desiccator, and dry it for about 48 hours under the inside pressure of the desiccator of 2.0 kPa {15 mmHg} or lower. Weigh its 1.2 g to 1.3 g to the nearest 0.1 mg, transfer it into a 300 ml Erlenmeyer flask with an interchangeable ground joint, and add 50 ml of water to dissolve it. Add 4 ml of sulfuric acid (1+1), and while cooling it at 15 °C or lower, add drop by drop 0.02 mol/l potassium permanganate solution with care until it gets coloring (regard this coloring as standard of the end point of titration that is to be carried out later). "Titrate this solution with the solution prepared at (a), add carefully until colored solution loses the coloring. Then, add a couple of drops of 0.02 mol/l potassium permanganate solution (thereafter add up the amount of titrant, and use it for calculation of titrant)." (Repeat these procedures mentioned in " " until the adding of 0.02 mol/l potassium permanganate solution does not give coloring.) Continuously, titrate it with 0.02 mol/l potassium permanganate solution. Make it an end point when the solution gets the standard coloring.

Remarks: For the purpose of accurate standardization, it is preferable to use 3 ml or less of 0.02 mol/l potassium permanganate solution, so the following method can be employed to eliminate intermediate dropping of 0.02 mol/l potassium permanganate solution; carry out pretest to find approximate amount of titrant of prepared solution, add prepared solution which is 0.1 ml to 0.2 ml more than this amount, and 0.02 mol/l potassium permanganate solution shall be added until it gets standard coloring.

- (c) **Calculation** Calculate the factor according to the following formula.

$$f = \frac{a}{0.048\ 54 \times B} \times \frac{A}{100}$$

where, f : factor of 0.5 mol/l sodium nitrite solution

a : mass of amidosulfuric acid weighed out (g)

A : purity of amidosulfuric acid (%)

0.048 54: mass of amidosulfuric acid corresponding to 1 ml of 0.5 mol/l sodium nitrite solution (g)

$$B: V_1 - \frac{V_2}{10} \text{ [consumed amount of prepared solution corresponding with amidosulfuric acid (ml)]}$$

where,

V_1 : amount of titrant of prepared solution (ml)

V_2 : used amount of 0.02 mol/l potassium permanganate solution (ml)

(3) Disodium dihydrogen ethylenediaminetetraacetate solution (EDTA2Na solution)

(3.1) 0.1 mol/l disodium dihydrogen ethylenediaminetetraacetate solution (0.1 mol/l EDTA2Na solution) (37.22 g $C_{10}H_{14}O_8N_2Na_2 \cdot 2H_2O/l$)

(a) **Preparation** Weigh 38 g of disodium dihydrogen ethylenediaminetetraacetate dihydrate (EDTA2Na) specified in **JIS K 8107**, add 1 l of water to dissolve it, and store it in a polyethylene airtight bottle.

(b) **Standardization** Take accurately 25 ml of 0.1 mol/l zinc solution in a 200 ml conical beaker. After adding 75 ml of water, add 20 ml of the solution prepared at (a) with a buret (use this buret again for titration after pH was adjusted). Then, adjust its pH to be 6 to 8 using sodium hydroxide solution (100 g/l). Add 2 ml of ammoniac ammonium chloride buffer solution and 0.05 g of Eriochrome Black T diluent powder as an indicator, and titrate it with the solution prepared at (a). Make it an end point when the solution turns from red to blue.

(c) **Calculation** Calculate the factor according to the following formula.

$$f_1 = \frac{f_2 \times 25}{V}$$

where,

f_1 : factor of 0.1 mol/l EDTA2Na solution

f_2 : factor of 0.1 mol/l zinc solution

V : volume of 0.1 mol/l EDTA2Na solution needed for titration (ml)

(3.2) 0.05 mol/l disodium dihydrogen ethylenediaminetetraacetate solution (0.05 mol/l EDTA2Na solution) (18.61 g $C_{10}H_{14}O_8N_2Na_2 \cdot 2H_2O/l$)

(a) **Preparation** Follow (3.1) (a). In this case, weigh 18.6 g of EDTA2Na.

(b) **Standardization** Take accurately 25 ml of 0.05 mol/l zinc solution in a 200 ml conical beaker, add 75 ml of water, and adjust its pH to be 6 to 8 using sodium hydroxide solution (100 g/l). Add 2 ml of ammoniac ammonium chloride buffer solution and 0.05 g of Eriochrome Black T diluent powder as an indicator, and titrate it with the solution prepared at (a). Make it an end point when the solution turns from red to blue.

(c) **Calculation** Calculate the factor according to the following formula.

$$f_1 = \frac{f_2 \times 25}{V}$$

where,

f_1 : factor of 0.05 mol/l EDTA2Na solution

f_2 : factor of 0.05 mol/l zinc solution

V : volume of 0.05 mol/l EDTA2Na solution needed for titration (ml)

(3.3) **0.01 mol/l disodium dihydrogen ethylenediaminetetraacetate solution (0.01 mol/l EDTA2Na solution)** (3.722 g $C_{10}H_{14}O_8N_2Na_2 \cdot 2H_2O/l$)

- (a) **Preparation** Follow (3.1) (a). In this case, weigh 3.8 g of EDTA2Na.
- (b) **Standardization** Follow (3.2) (b). In this case, take accurately 25 ml of 0.01 mol/l zinc solution.
- (c) **Calculation** Calculate the factor according to the following formula.

$$f_1 = \frac{f_2 \times 25}{V}$$

where,

f_1 : factor of 0.01 mol/l EDTA2Na solution

f_2 : factor of 0.01 mol/l zinc solution

V : volume of 0.01 mol/l EDTA2Na solution needed for titration (ml)

(3.4) **0.01 mol/l disodium dihydrogen ethylenediaminetetraacetate compound solution (0.01 mol/l EDTA2Na compound solution)**

- (a) **Preparation** Weigh 0.5 g of zinc chloride specified in **JIS K 8111**, 1.0 g of magnesium chloride hexahydrate specified in **JIS K 8159**, and 7.0 g of EDTA2Na, and dissolve them in about 50 ml of water that is free from carbon dioxide. Adjust its pH to be 7 to 8 using sodium hydroxide solution (300 g/l), add carbon-dioxide-free water to make total 1 l, mix them well, and store it in a polyethylene airtight bottle.
- (b) **Standardization** Take accurately 25 ml of 0.01 mol/l zinc solution in a 200 ml conical beaker, add 75 ml of carbon-dioxide-free water, and adjust its pH to be 6 to 8 using sodium hydroxide solution (100 g/l). Add 2 ml of ammoniac ammonium chloride buffer solution and 0.05 g of Eriochrome Black T diluent powder as an indicator, and titrate it with the solution prepared at (a). Make it an end point when the solution turns from red to blue.
- (c) **Calculation** Calculate the factor according to the following formula.

$$f_1 = \frac{f_2 \times 25}{V}$$

where,

f_1 : factor of 0.01 mol/l EDTA2Na compound solution

f_2 : factor of 0.01 mol/l zinc solution

V : volume of 0.01 mol/l EDTA2Na compound solution needed for titration (ml)

(4) **0.05 mol/l magnesium chloride solution** (10.17 g $MgCl_2 \cdot 6H_2O/l$)

- (a) **Preparation** Weigh 10.2 g of magnesium chloride hexahydrate specified in **JIS K 8159**, add 1 l of water to dissolve it, and store it in an airtight container.