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NATIONAL STANDARD OF THE

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GB/T 601-2016

Replacing GB/T 601-2002

Chemical reagent -
Preparations of reference titration solutions

化学试剂标准滴定溶液的制备

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Foreword

This Standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This Standard replaces GB/T 601-2002 *Chemical reagent – Preparations of reference titration solutions*. Compared with GB/T 601-2002, the major technical changes are as follows:

- The relevant contents in general provisions 3.2, 3.6, 3.8, 3.9 and 3.10 have been modified (SEE 3.2, 3.6, 3.8, 3.9 and 3.10 of the present edition; and 3.2, 3.6, 3.8, 3.9 and 3.10 of the 2002 edition);
- "The blank test is performed at the same time" has been added to the method 1 of the reference titration solution of sodium carbonate, and the method 2 (direct preparation with working reference reagent) has been added (SEE 4.4.1.2 and 4.4.2 of the present edition; and 4.4.2 of the 2002 edition);
- The preparation method and calibration method 2 of the reference titration solution of iodine have been modified (SEE 4.9.1 and 4.9.2.2 of the present edition; and 4.9.1 and 4.9.2.2 of the 2002 edition);
- The preparation method of the reference titration solution of sodium thiosulfate has been modified (SEE 4.6.1 of the present edition; and 4.6.1 of the 2002 edition);
- The reference titration solution of sodium oxalate has been added to oxalic acid side by side, and the method 2 (direct preparation of the reference titration solution of sodium oxalate with working reference reagent) has been added (SEE 4.11.1.1 and 4.11.2 of the present edition; and 4.11 of the 2002 edition);
- The method 1 has been added to the reference titration solution of ammonium ferric sulfate (II), and "the blank test is performed at the same time" has been added to the method 2 (SEE 4.13.3.1 and 4.13.3.2 of the present edition; and 4.13 of the 2002 edition);
- The method 2 (direct preparation with working reference reagent) has been added to the reference titration solution of ethylene diamine tetraacetic acid, and the effective number of bits of the molar mass of the working reference reagent zinc oxide has been modified (SEE 4.15.1.2.1, 4.15.1.2.2 and 4.15.2 of the present edition; and 4.15.2.1 and 4.15.2.2 of the 2002 edition);
- Two concentrations of 0.05mol/L and 0.02mol/L have been added to the

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Chemical reagent -

Preparations of reference titration solutions

Warning: Some of the testing procedures specified in this Standard may result in hazardous conditions and the users are responsible for taking appropriate safety and health measures.

1 Scope

This Standard specifies the preparation and calibration methods of the reference titration solutions of chemical reagents.

This Standard applies to the preparation and calibration of the reference titration solutions for determining the purity and impurity content of chemical reagents by titration. Other fields are also available.

2 Normative references

The following documents are essential for the application of this document. For dated references, only the dated editions apply to this document. For undated references, the latest editions (including all amendments) apply to this document.

GB/T 603 *Chemical reagent – Preparations of reagent solutions for use in test methods*

GB/T 606 *Chemical reagent – General method for the determination of water (Karl Fischer method)*

GB/T 6379.6-2009 *Accuracy (trueness and precision) of measurement methods and results – Part 6: Use in practice of accuracy values*

GB/T 6682 *Water for analytical laboratory use – Specification and test methods*

GB/T 9725-2007 *Chemical reagent – General rule for potentiometric titration*

JJG 130 *Liquid-in-glass thermometers for working*

JJG 196-2006 *Working glass container*

JJG 1036 *Electronic balance*

verified by comparison (SEE Appendix C).

3.7 The relative expansion uncertainty of the concentrations of the reference titration solutions in this Standard is not greater than 0.2% (k = 2), and the evaluation method is shown in Appendix D.

3.8 This Standard uses the working reference reagent to calibrate the concentrations of the reference titration solutions. When the accuracy of the concentrations of the reference titration solutions is required to be higher, the standard substance (the expansion uncertainty shall be less than 0.05%) may be used instead of the working reference reagent for calibration or direct preparation, and when calculating the concentrations of the reference titration solutions, its mass fraction is substituted into the calculation formula.

3.9 When the concentration of the reference titration solution is less than or equal to 0.02mol/L (except 0.02mol/L reference titration solutions of ethylene diamine tetraacetic acid and zinc chloride), the reference titration solution with a high concentration shall be diluted with boiling and cooling water (a reference titration solution without non-aqueous solvent) prior to use, and recalibrated if necessary. When it is necessary to use the reference titration solution other than the concentration specified in this Standard, it may be prepared and calibrated by referring to the preparation method of the corresponding reference titration solution in this Standard.

3.10 Storage:

a) Unless otherwise specified, the sealed storage time of the reference titration solutions at 10°C to 30°C is generally not more than 6 months; the sealed storage time of the reference titration solutions of iodine and sodium nitrite [c($NaNO_2$) = 0.1mol/L] is 4 months; and the sealed storage time of the reference titration solutions of perchloric acid, potassium hydroxide-ethanol, and ammonium ferric sulfate (III) is 2 months. Reference titration solutions that exceed the storage time may be used after recalibration.

b) The storage time of the reference titration solution unsealed and used at 10°C to 30°C is generally not more than 2 months (CLOSE immediately after pouring out the solution); for the reference titration solutions of iodine and potassium hydroxide-ethanol, it is generally not more than 1 month; for the reference titration solution of sodium nitrite [c($NaNO_2$) = 0.1mol/L], it is generally not more than 15d; and the reference titration solution of perchloric acid is used on the same day after unsealing.

c) When the standard titration solution shows turbidity, precipitation, color change, etc., it shall be re-prepared.

3.11 The container for storing the reference titration solutions shall not have physical and chemical effects with the solutions, and the thinnest part of the

$$c(1/2\ \mathrm{Br_2}) = \frac{(V_1 - V_2) \times c_1}{V} \quad \cdots\cdots (9)$$

Where:

V_1 – Volume of the reference titration solution of sodium thiosulfate, in milliliter (mL);

V_2 – Volume of the reference titration solution of sodium thiosulfate consumed by blank test, in milliliter (mL);

c_1 – Concentration of the reference titration solution of sodium thiosulfate, in mole per liter (mol/L);

V – Volume of bromine solution, in milliliter (mL).

4.8 Reference titration solution of potassium bromate [c(1/6 $KBrO_3$) = 0.1mol/L]

4.8.1 Preparation

WEIGH 3g of potassium bromate, DISSOLVE it in 1,000mL of water, and SHAKE well.

4.8.2 Calibration

WEIGH 35.00mL to 40.00mL of prepared potassium bromate solution, PLACE it in an iodine flask, ADD 2g of potassium iodide and 5mL of hydrochloric acid solution (20%), SHAKE well, and PLACE it in the dark for 5min. ADD 150mL of water (15°C to 20°C), TITRATE with the reference titration solution of sodium thiosulfate [c($Na_2S_2O_3$) = 0.1mol/L], ADD 2mL of starch indicator solution (10g/L) near the endpoint, and CONTINUE titration until the blue color of the solution disappears. The blank test is performed at the same time.

The concentration of the reference titration solution of potassium bromate [c(1/6 $KBrO_3$)] is calculated according to Formula (10):

$$c(1/6\ \mathrm{KBrO_3}) = \frac{(V_1 - V_2) \times c_1}{V} \quad \cdots\cdots (10)$$

Where:

V_1 – Volume of the reference titration solution of sodium thiosulfate, in milliliter (mL);

V_2 – Volume of the reference titration solution of sodium thiosulfate consumed

300mL of sulfuric acid solution (20%), ADD 700mL of water, and SHAKE well.

4.13.3 Calibration (calibration prior to use)

4.13.3.1 Method 1

WEIGH 0.18g of the working reference reagent, potassium dichromate, dried in an electric oven at 120°C±2°C to a constant-weight, DISSOLVE it in 25mL of water, ADD 10mL of sulfur-phosphorus mixed acid solution, ADD 70mL of water, TITRATE with the prepared ammonium ferric sulfate (II) solution until the orange color disappears, ADD 2 drops of N-phenylanthranilic acid indicator solution (2g/L), and CONTINUE titration until the solution turns from purple to bright green.

The concentration of the reference titration solution of ammonium ferric sulfate (II) {$c[(NH_4)_2 Fe(SO_4)_2]$} is calculated according to Formula (18):

$$c[(NH_4)_2Fe(SO_4)_2]=\frac{m\times 1\,000}{V\times M} \quad \cdots\cdots(18)$$

Where:

m – Mass of potassium dichromate, in gram (g);

V – Volume of ammonium ferric sulfate (II) solution, in milliliter (mL);

M - Molar mass of potassium dichromate, in gram per mole (g/mol) [$M(1/6\ K_2Cr_2O_7) = 49.031$].

4.13.3.2 Method 2

WEIGH 35.00mL to 40.00mL of prepared ammonium ferric sulfate (II) solution, ADD 25mL of anaerobic water, TITRATE with the reference titration solution of potassium permanganate [$c(1/5\ KMnO_4) = 0.1$mol/L] until the solution is pink, and KEEP it for 30s. The blank test is performed at the same time.

The concentration of the reference titration solution of ammonium ferric sulfate (II) {$c[(NH_4)_2 Fe(SO_4)_2]$} is calculated according to Formula (19):

$$c[(NH_4)_2Fe(SO_4)_2]=\frac{(V_1-V_2)\times c_1}{V} \quad \cdots\cdots(19)$$

Where:

V_1 – Volume of the reference titration solution of potassium permanganate, in milliliter (mL);

V_2 – Volume of the reference titration solution of potassium permanganate consumed by blank test, in milliliter (mL);

m – Mass of zinc oxide, in gram (g);

V_1 – Volume of ethylene diamine tetraacetic acid solution, in milliliter (mL);

V_2 – Volume of the ethylene diamine tetraacetic acid solution consumed by blank test, in milliliter (mL);

M - Molar mass of zinc oxide, in gram per mole (g/mol) [M (ZnO) = 81.408].

4.15.1.2.2 Reference titration solution of ethylene diamine tetraacetic acid [c(EDTA) = 0.02mol/L]

WEIGH 0.42g of the working reference reagent, zinc oxide, that has been burnt in a high-temperature furnace at 800°C±50°C to a constant-weight, WET with a small amount of water, ADD 3mL of hydrochloric acid solution (20%) to dissolve, PIPETTE into a 250mL volumetric flask, DILUTE to the scale, and SHAKE well. TAKE 35.00mL to 40.00mL, ADD 70mL of water, USE an ammonia solution (10%) to adjust the pH of the solution to 7 to 8, ADD 10mL of ammonia-ammonium chloride buffer solution A (pH ≈ 10) and 5 drops of chrome black T indicator solution (5g/L), and TITRATE with the prepared ethylene diamine tetraacetic acid solution until the solution turns from purple to pure blue. The blank test is performed at the same time.

The concentration of the reference titration solution of ethylene diamine tetraacetic acid [c(EDTA)] is calculated according to Formula (22):

$$c(\mathrm{EDTA}) = \frac{m \times \left(\frac{V_1}{250}\right) \times 1\,000}{(V_2 - V_3) \times M} \qquad \cdots\cdots (22)$$

Where:

m – Mass of zinc oxide, in gram (g);

V_1 – Volume of zinc oxide solution, in milliliter (mL);

V_2 – Volume of ethylene diamine tetraacetic acid solution, in milliliter (mL);

V_3 – Volume of the ethylene diamine tetraacetic acid solution consumed by blank test, in milliliter (mL);

M - Molar mass of zinc oxide, in gram per mole (g/mol) [M (ZnO) = 81.408].

4.15.2 Method 2

According to the amount specified in Table 15, WEIGH the working reference reagent, ethylene diamine tetraacetic acid, placed in a humidistat with a

$$c = \frac{m \times 1\,000}{(V_1 - V_2) \times M} \qquad \text{(26)}$$

Where:

m – Mass of ethylene diamine tetraacetic acid, in gram (g);

V_1 – Volume of magnesium chloride (or magnesium sulfate) solution, in milliliter (mL);

V_2 – Volume of the magnesium chloride (or magnesium sulfate) solution consumed by blank test, in milliliter (mL);

M - Molar mass of ethylene diamine tetraacetic acid, in gram per mole (g/mol) [M (EDTA) = 372.24].

4.18 Reference titration solution of lead nitrate {$c[Pb(NO_3)_2]$ = 0.05mol/L}

4.18.1 Preparation

WEIGH 17g of lead nitrate, DISSOLVE it in 1,000mL of nitric acid solution (1+2000), and SHAKE well.

4.18.2 Calibration

WEIGH 35.00mL to 40.00mL of prepared lead nitrate solution, ADD 3mL of acetic acid (glacial acetic acid) and 5g of hexamethylenetetramine, ADD 70mL of water and 2 drops of xylenol orange indicator solution (2g/L), and TITRATE with the reference titration solution of ethylene diamine tetraacetic acid [c(EDTA) = 0.05mol/L] until the solution is bright yellow. The blank test is performed at the same time.

The concentration of the reference titration solution of lead nitrate {$c[Pb(NO_3)_2]$} is calculated according to Formula (27):

$$c[\mathrm{Pb(NO_3)_2}] = \frac{(V_1 - V_2) \times c_1}{V} \qquad \text{(27)}$$

Where:

V_1 – Volume of the reference titration solution of ethylene diamine tetraacetic acid, in milliliter (mL);

V_2 – Volume of the reference titration solution of ethylene diamine tetraacetic acid consumed by blank test, in milliliter (mL);

c_1 – Concentration of the reference titration solution of ethylene diamine tetraacetic acid, in mole per liter (mol/L);

$$c(\mathrm{NaCl}) = \frac{m \times 1\ 000}{V \times M} \qquad \cdots\cdots (29)$$

Where:

m – Mass of sodium chloride, in gram (g);

V – Volume of sodium chloride solution, in milliliter (mL);

M – Molar mass of sodium chloride, in gram per mole (g/mol) [M (NaCl) = 58.442].

4.20 Reference titration solution of sodium thiocyanate (or potassium thiocyanate, ammonium thiocyanate) [c(NaSCN) = 0.1mol/L, c(KSCN) = 0.1mol/L, c(NH_4SCN) = 0.1mol/L]

4.20.1 Preparation

WEIGH 8.2g of sodium thiocyanate (or 9.7g of potassium thiocyanate or 7.9g of ammonium thiocyanate), DISSOLVE it in 1,000mL of water, and SHAKE well.

4.20.2 Calibration

4.20.2.1 Method 1

DETERMINE according to the provisions of GB/T 9725-2007. Wherein: WEIGH 0.6g of the working reference reagent, silver nitrate, dried in a sulfuric acid desiccator to a constant-weight, DISSOLVE it in 90mL of water, ADD 10mL of starch solution (10g/L) and 10mL of nitric acid solution (25%), USE the 216-type silver electrode as an indicator electrode and the 217-type double salt bridge saturated calomel electrode as a reference electrode, and TITRATE with the sodium thiocyanate (or potassium thiocyanate or ammonium thiocyanate) solution. CALCULATE V_0 according to the provisions of 6.2.2 of GB/T 9725-2007.

The concentration (c) of the reference titration solution of sodium thiocyanate (or potassium thiocyanate or ammonium thiocyanate) is calculated according to Formula (30):

$$c = \frac{m \times 1\ 000}{V_0 \times M} \qquad \cdots\cdots (30)$$

Where:

m – Mass of silver nitrate, in gram (g);

V_0 – Volume of sodium thiocyanate (or potassium thiocyanate or ammonium blank test is performed at the same time.

The concentration of the reference titration solution of sodium nitrite [$c(NaNO_2)$] is calculated according to Formula (35):

$$c(NaNO_2) = \frac{m \times 1\ 000}{(V_1 - V_2) \times M} \qquad \cdots\cdots (35)$$

Where:

m – Mass of p-aminobenzene sulfonic acid, in gram (g);

V_1 – Volume of sodium nitrite solution, in milliliter (mL);

V_2 – Volume of the sodium nitrite solution consumed by blank test, in milliliter (mL);

M - Molar mass of p-aminobenzene sulfonic acid, in gram per mole (g/mol) {$M[C_6H_4(NH_2)(SO_3H)] = 173.19$}.

4.24 Reference titration solution of perchloric acid [$c(HClO_4)$ = 0.1mol/L]

4.24.1 Preparation

4.24.1.1 Method 1

WEIGH 8.7mL of perchloric acid, INJECT 500mL of acetic acid (glacial acetic acid) under stirring, and MIX well. Dropwise ADD 20mL of acetic anhydride, and STIR until the solution is homogeneous. After cooling, USE an acetic acid (glacial acetic acid) to dilute to 1,000mL.

4.24.1.2 Method 2 [1]

WEIGH 8.7mL of perchloric acid, INJECT 950mL of acetic acid (glacial acetic acid) under stirring, and MIX well. TAKE 5mL, two portions in total. USE pyridine as a solvent, and DETERMINE the mass fraction of water according to the provisions of GB/T 606. CALCULATE the amount of acetic anhydride added in the perchloric acid solution from the average value (w_1) of the results of the two parallel determinations. Dropwise ADD the calculated amount of acetic anhydride, and STIR well. After cooling, USE an acetic acid (glacial acetic acid) to dilute to 1,000mL, and SHAKE well.

The amount (V) of acetic anhydride added in the perchloric acid solution is calculated according to Formula (36):

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[1] The method controls the mass fraction of water in the reference titration solution of perchloric acid to be about 0.05%.

$$c_1(\mathrm{HClO_4}) = \frac{c}{1 + 0.001\,1 \times (t_1 - t)} \quad \cdots\cdots (38)$$

Where:

c – Concentration of the reference titration solution of perchloric acid at the calibration temperature, in mole per liter (mol/L);

t_1 – Temperature of the reference titration solution of perchloric acid when in use, in degrees Celsius (°C);

t_2 – Temperature of the reference titration solution of perchloric acid during calibration, in degrees Celsius (°C);

0.001 1 - The volume expansion coefficient of the reference titration solution of perchloric acid for each change of 1°C, in per degrees Celsius ($°C^{-1}$).

4.25 Reference titration solution of potassium hydroxide-ethanol [c(KOH) = 0.1mol/L]

4.25.1 Preparation

WEIGH about 500g of potassium hydroxide, PLACE it in a beaker, ADD about 420mL of water to dissolve, COOL, PIPETTE into a polyethylene container, and LET it stand. USE a plastic tube to weigh 7mL of the supernatant, and USE ethanol (95%) to dilute to 1,000mL. PLACE in a sealed and dark place for 2d to 4d, and then USE a plastic tube to siphon the supernatant to another polyethylene container (protected from light or USE a dark polyethylene container).

4.25.2 Calibration

WEIGH 0.75g of the working reference reagent, potassium hydrogen phthalate, dried in an electric oven at 105°C to 110°C to a constant-weight, DISSOLVE it in 50mL of carbon dioxide-free water, ADD 2 drops of phenolphthalein indicator solution (10g/L), and TITRATE with the prepared potassium hydroxide-ethanol solution until the solution is pink. The blank test is performed at the same time.

The concentration of the reference titration solution of potassium hydroxide-ethanol [c(KOH)] is calculated according to Formula (39):

$$c(\mathrm{KOH}) = \frac{m \times 1\,000}{(V_1 - V_2) \times M} \quad \cdots\cdots (39)$$

Where:

4.26.2.2 Method 2

WEIGH 35.00mL to 40.00mL of prepared hydrochloric acid-ethanol solution, ADD 2 drops of phenolphthalein indicator solution (10g/L), and TITRATE with the reference titration solution of sodium hydroxide [c(NaOH) = 0.5mol/L] until the solution is pink.

The concentration of the reference titration solution of hydrochloric acid-ethanol [c(HCl)] is calculated according to Formula (41):

$$c(\mathrm{HCl}) = \frac{V_1 \times c_1}{V} \quad \cdots\cdots\cdots\cdots (41)$$

Where:

V_1 – Volume of the reference titration solution of sodium hydroxide, in milliliter (mL);

c_1 – Concentration of the reference titration solution of sodium hydroxide, in mole per liter (mol/L);

V – Volume of hydrochloric acid-ethanol solution, in milliliter (mL).

4.27 Reference titration solution of ammonium ferric sulfate (III) {c[$NH_4Fe(SO_4)_2$] = 0.1mol/L}

4.27.1 Preparation

WEIGH 48g of ammonium ferric sulfate (III) dodecahydrate, ADD 500mL of water, ADD 50mL of sulfuric acid slowly, HEAT to dissolve, COOL, and DILUTE to 1,000mL.

4.27.2 Calibration

WEIGH 35.00mL to 40.00mL of prepared ammonium ferric sulfate (III) solution. ADD 10mL of hydrochloric acid solution (1+1). HEAT to near boiling. Dropwise ADD a stannous chloride solution (400g/L) until the solution is colorless. ADD excessive 1 to 2 drops, COOL, ADD 10mL of saturated solution of mercury chloride, and SHAKE well. LET it stand for 2min to 3min. ADD 10mL of sulfur-phosphorous mixed acid solution (SEE 4.13.1.1). DILUTE to 100mL. ADD 1mL of sodium diphenylamine sulfonate indicator solution (5g/L). TITRATE with the reference titration solution of potassium dichromate [c(1/6 $K_2Cr_2O_7$) = 0.1mol/L] until the solution is purple, and KEEP it for 30s. The blank test is performed at the same time. (COLLECT the waste liquid, and SEE Appendix E for the treatment method.)

The concentration of the reference titration solution of ammonium ferric

Appendix B

(Normative)

Method for determining burette capacity

B.1 Instruments

B.1.1 The analytical balance has a sensitivity of 0.1mg and complies with the provisions of JJG 1036.

B.1.2 The thermometer has a graduation value of 0.1°C and complies with the provisions of JJG 130.

B.1.3 The burette shall comply with the provisions of JJG 196-2006.

B.2 Determining steps

B.2.1 PLACE water in the balance chamber (at room temperature of 20°C±5°C) in advance, and EQUILIBRATE to room temperature.

B.2.2 INSTALL the cleaned burette vertically and securely on the verification stand. FILL the flow port of the burette to about 5mm above the highest mark. Slowly ADJUST to zero.

B.2.3 WEIGH the mass of the light bottle with a cover, m_1.

B.2.4 CONTROL the flow rate to be 6mL/min to 8mL/min. ADJUST the liquid level to the measured graduation line. USE the light bottle above to receive the effluent.

B.2.5 WEIGH the mass of water and light bottle, m_2. MEASURE the temperature of water in the light bottle.

B.2.6 The volume of the burette at 20°C, V_{20}, is calculated according to Formula (B.1):

$$V_{20}=(m_2-m_1)\times K(t) \qquad \text{(B.1)}$$

Where:

m_2 – Mass of water and light bottle, in gram (g);

m_1 – Mass of light bottle, in gram (g);

The blank test of iodine consumed by water is performed at the same time: TAKE 250mL of water (15°C to 20°C), ADD 5mL of the reference titration solution of hydrochloric acid [c(HCl) = 0.1mol/L], ADD 0.05mL to 0.20mL of the reference titration solution of iodine [c(1/2 I_2) = 0.1mol/L] and 2mL of starch indicator solution (10g/L), and TITRATE with the reference titration solution of sodium thiosulfate [c($Na_2S_2O_3$) = 0.1mol/L] until the solution changes from blue to colorless.

The concentration of the reference titration solution of sodium thiosulfate [c($Na_2S_2O_3$)] is calculated according to Formula (C.4):

$$c(\mathrm{Na_2S_2O_3})=\frac{(V_1-V_2)\times c_1}{V_3-V_4} \qquad \cdots\cdots (C.4)$$

Where:

V_1 – Volume of the reference titration solution of iodine, in milliliter (mL);

V_2 - Volume of the reference titration solution of iodine added in the blank test, in milliliter (mL);

c_1 – Concentration of the reference titration solution of iodine, in mole per liter (mol/L);

V_3 – Volume of the reference titration solution of sodium thiosulfate, in milliliter (mL);

V_4 - Volume of the reference titration solution of sodium thiosulfate consumed by blank test, in milliliter (mL).

C.6 Reference titration solution of iodine

WEIGH 35.00mL to 40.00mL of the reference titration solution of iodine [c(1/2 I_2) = 0.1mol/L] to be compared. PLACE it in an iodine flask. ADD 150mL of water (15°C to 20°C). ADD 5mL of the reference titration solution of hydrochloric acid [c(HCl) = 0.1mol/L]. TITRATE with the reference titration solution of sodium thiosulfate [c($Na_2S_2O_3$) = 0.1mol/L]. In the case of near endpoint, ADD 2mL of starch indicator solution (10g/L), and CONTINUE titration until the blue color of the solution disappears.

The blank test of iodine consumed by water is performed at the same time: TAKE 250mL of water (15°C to 20°C), ADD 5mL of the reference titration solution of hydrochloric acid [c(HCl) = 0.1mol/L], ADD 0.05mL to 0.20mL of the reference titration solution of iodine [c(1/2 I_2) = 0.1mol/L] to be compared and 2mL of starch indicator solution (10g/L), and TITRATE with the reference titration solution of sodium thiosulfate [c($Na_2S_2O_3$) = 0.1mol/L] until the blue (mL);

V_2 – Volume of the reference titration solution of sodium thiosulfate consumed by blank test, in milliliter (mL);

c_1 – Concentration of the reference titration solution of sodium thiosulfate, in mole per liter (mol/L);

V – Volume of the reference titration solution of potassium permanganate, in milliliter (mL).

C.8 Reference titration solution of cerous sulfate (or ammonium cerous sulfate)

WEIGH 35.00mL to 40.00mL of the reference titration solution of cerous sulfate [c[Ce(SO$_4$)$_2$] = 0.1mol/L] to be compared. PLACE it in an iodine flask. ADD 2g of potassium iodide and 20mL of sulfuric acid solution (20%), SHAKE well, and LET it stand in the dark for 5min. ADD 150mL of water (15°C to 20°C), and TITRATE with the reference titration solution of sodium thiosulfate [c($Na_2S_2O_3$) = 0.1mol/L]. In the case of near endpoint, ADD 2mL of starch indicator solution (10g/L), and CONTINUE titration until the blue color of the solution disappears. The blank test is performed at the same time.

The concentration of the reference titration solution of cerous sulfate (or ammonium cerous sulfate) {c[Ce(SO$_4$)$_2$]} is calculated according to Formula (C.7):

$$c[\mathrm{Ce(SO_4)_2}]=\frac{(V_1-V_2)\times c_1}{V} \quad \cdots\cdots\cdots\cdots\cdots\cdots (\mathrm{C.7})$$

Where:

V_1 – Volume of the reference titration solution of sodium thiosulfate, in milliliter (mL);

V_2 – Volume of the reference titration solution of sodium thiosulfate consumed by blank test, in milliliter (mL);

c_1 – Concentration of the reference titration solution of sodium thiosulfate, in mole per liter (mol/L);

V – Volume of the reference titration solution of cerous sulfate (or ammonium cerous sulfate), in milliliter (mL).

C.9 Reference titration solution of sodium thiocyanate (or potassium thiocyanate or ammonium thiocyanate)

milliliter (mL);

c_1 – Concentration of the reference titration solution of sodium thiocyanate, in mole per liter (mol/L);

V – Volume of the reference titration solution of silver nitrate, in milliliter (mL).

C.11 Reference titration solution of ethylene diamine tetraacetic acid

WEIGH 35.00mL to 40.00mL of the corresponding concentration of the reference titration solution of zinc chloride, ADD 70mL of water, USE an ammonia solution (10%) to adjust the pH of the solution to 7 to 8, ADD 10mL of ammonia-ammonium chloride buffer solution A (pH ≈ 10) and 5 drops of chrome black T indicator solution (5g/L), and TITRATE with the reference titration solution of ethylene diamine tetraacetic acid to be compared until the solution turns from purple to pure blue. The blank test is performed at the same time.

The concentration of the reference titration solution of ethylene diamine tetraacetic acid [c(EDTA)] is calculated according to Formula (C.10):

$$c(\mathrm{EDTA})=\frac{V_1\times c_1}{V_2-V_3} \qquad \cdots\cdots\cdots\cdots\cdots\cdots (\mathrm{C.10})$$

Where:

V_1 – Volume of the reference titration solution of zinc chloride, in milliliter (mL);

c_1 – Concentration of the reference titration solution of zinc chloride, in mole per liter (mol/L);

V_2 – Volume of the reference titration solution of ethylene diamine tetraacetic acid, in milliliter (mL);

V_3 – Volume of the reference titration solution of ethylene diamine tetraacetic acid consumed by blank test, in milliliter (mL).

C.12 Reference titration solution of zinc chloride

WEIGH 35.00mL to 40.00mL of the corresponding concentration of the reference titration solution of ethylene diamine tetraacetic acid, ADD 70mL of water, ADD 10mL of ammonia-ammonium chloride buffer solution A (pH ≈ 10), and TITRATE with the reference titration solution of zinc chloride to be compared. In the case of near endpoint, ADD 5 drops of chrome black T indicator solution (5g/L), and CONTINUE titration until the solution turns from

Appendix D

(Informative)

Assessment on expansion uncertainty of reference titration solution concentrations

D.1 General rules

During the first preparation of the reference titration solution, the measurement uncertainty shall be assessed. Daily preparation does not have to be assessed each time, but when conditions (such as personnel, measuring instruments, environment, etc.) change, the measurement uncertainty shall be re-assessed.

D.2 Calibration methods for reference titration solutions

D.2.1 Method 1

The concentration of the reference titration solution is calibrated using a working reference reagent. It includes a total of 18 reference titration solutions of sodium hydroxide, hydrochloric acid, sulfuric acid, sodium thiosulfate, iodine, potassium permanganate, ammonium ferric sulfate (II), cerous sulfate, ethylene diamine tetraacetic acid [c(EDTA) = 0.1mol/L, 0.05mol/L] , zinc chloride, magnesium chloride, sodium thiocyanate, silver nitrate, mercury nitrate, sodium nitrite, perchloric acid, potassium hydroxide-ethanol, and hydrochloric acid-ethanol.

This Standard specifies that the concentration of the reference titration solution should be calibrated using a working reference reagent (whose mass fraction is 100%). When the accuracy of the reference titration solution concentration is required to be higher, the secondary purity standard substance or the fixed value standard substance may be used instead of the working reference reagent for calibration, and when the reference titration solution concentration is calculated, the mass fraction is substituted into the calculation formula.

The concentration (c) of the reference titration solution is calculated according to Formula (D.1):

$$c=\frac{m\times w\times 1\,000}{(V_1-V_2)\times M} \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\text{(D.1)}$$

$$c = \frac{\left(\frac{m}{V_3}\right) \times V_4 \times w \times 1\ 000}{(V_1 - V_2) \times M} \qquad \text{(D.3)}$$

Where:

m – Mass of the working reference reagent, in gram (g);

V_3 – Volume of the working reference reagent solution, in milliliter (mL);

V_4 - Volume of the working reference reagent solution measured, in milliliter (mL);

w – Mass fraction of the working reference reagent, %;

V_1 – Volume of the solution calibrated, in milliliter (mL);

V_2 - Volume of the solution calibrated in the blank test, in milliliter (mL);

M – Molar mass of the working reference reagent, in gram per mole (g/mol).

D.2.4 Method 4

The reference titration solution is directly prepared with a working reference reagent. It includes a total of 6 reference titration solutions of sodium carbonate, potassium dichromate, potassium iodate, sodium oxalate, zinc chloride and sodium chloride.

The concentration (c) of the reference titration solution is calculated according to Formula (D.4):

$$c = \frac{m \times w \times 1\ 000}{V \times M} \qquad \text{(D.4)}$$

Where:

m – Mass of the working reference reagent, in gram (g);

w – Mass fraction of the working reference reagent, %;

V - Volume of the reference titration solution, in milliliter (mL);

M – Molar mass of the working reference reagent, in gram per mole (g/mol).

D.3 Assessment on measurement uncertainty of reference titration solution concentrations calibrated with a working reference reagent

reagent mass;

$u_{crel}(w)$ – Relative composite standard uncertainty component of the mass fraction of the working reference reagent;

$u_{crel}(V_1-V_2)$ – Relative composite standard uncertainty component of the difference between the solution volume calibrated and the solution volume calibrated in the blank test;

$u_{crel}(M)$ – Relative composite standard uncertainty component of the molar mass of the working reference reagent;

$u_{rel}(r)$ – Relative standard uncertainty component rounded off by the solution concentration calibrated.

D.3.2.1 Relative standard uncertainty component of the working reference reagent mass [$u_{rel}(m)$]

CALCULATE according to Formula (D.8):

$$u_{rel}(m)=\frac{a}{k\times m} \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\text{(D.8)}$$

Where:

a – Maximum allowable error of the electronic balance, in gram (g);

k – Coverage factor, uniform distribution, $k=\sqrt{3}$;

m – Mass of the working reference reagent, in gram (g).

D.3.2.2 Relative composite standard uncertainty component of the mass fraction of the working reference reagent [$u_{crel}(w)$]

CALCULATE according to Formula (D.9):

$$u_{crel}(w)=\frac{\sqrt{\left(\frac{U}{k}\right)^2+\left(\frac{a}{k_1}\right)^2}}{w} \qquad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\text{(D.9)}$$

Where:

U – Expansion uncertainty of the mass fraction of the working reference reagent, %;

k – Coverage factor (in general, k = 2);

rounding off of the temperature correction value, in milliliter (mL);

V_1 – Solution volume calibrated, in milliliter (mL);

V_2 – Solution volume calibrated in the blank test, in milliliter (mL).

D.3.2.3.1 Standard uncertainty component introduced by the burette capacity [$u_1(V)$]

CALCULATE according to Formula (D.12):

$$u_1(V)=\frac{U}{k} \qquad \cdots\cdots\cdots\cdots (\text{D.12})$$

Where:

U – Expansion uncertainty of the burette capacity (SEE Appendix F for the assessment method), in milliliter (mL);

k – Coverage factor (in general, k = 2).

D.3.2.3.2 Composite standard uncertainty component introduced by the solution volume calibrated determined by interpolation [$u_{2c}(V)$]

CALCULATE according to Formula (D.13):

$$u_{2c}(V)=\sqrt{\left(\frac{a_1}{k_1}\right)^2+\left(\frac{a_2}{k_2}\right)^2} \qquad \cdots\cdots\cdots\cdots (\text{D.13})$$

Where:

a_1 – Greater than and less than half of the correction value difference at the two calibration points of the solution volume calibrated, in milliliter (mL);

k_1 – Triangular distribution, $k=\sqrt{6}$;

a_2 – Half width of the rounding off of the solution volume calibrated determined by interpolation, in milliliter (mL);

k_2 – Uniform distribution, $k=\sqrt{3}$.

D.3.2.3.3 Composite standard uncertainty component introduced by the rounding off of the temperature correction value [$u_{3c}(V)$]

D.3.2.5 Relative standard uncertainty component rounded off by the solution concentration calibrated [$u_{rel}(r)$]

CALCULATE according to Formula (D.16):

$$u_{\mathrm{rel}}(r)=\frac{a}{k\times c} \qquad \cdots\cdots(\text{D.16})$$

Where:

a – Half width of the rounding off of the reference titration solution concentration, in mole per liter (mol/L);

k – Coverage factor, uniform distribution, $k=\sqrt{3}$;

c – Concentration of the reference titration solution, in mole per liter (mol/L).

The above calculation results of $u_{rel}(m)$, $u_{crel}(w)$, $u_{crel}(V_1\text{-}V_2)$, $u_{crel}(M)$ and $u_{rel}(r)$ are substituted into Formula (D.7) to obtain the Class B assessment on relative composite standard uncertainty component of reference titration solution concentrations.

D.3.3 Composite standard uncertainty of the reference titration solution concentration [$u_c(c)$]

CALCULATE according to Formula (D.17):

$$u_{\mathrm{c}}(c)=c\times\sqrt{u_{\mathrm{Arel}}^{2}(c)+u_{\mathrm{cBrel}}^{2}(c)} \qquad \cdots\cdots(\text{D.17})$$

Where:

c – Concentration of the reference titration solution, in mole per liter (mol/L);

$u_{Arel}(c)$ - Class A assessment on relative standard uncertainty component of reference titration solution concentrations;

$u_{cBrel}(c)$ - Class B assessment on relative composite standard uncertainty component of reference titration solution concentrations

D.3.4 Expansion uncertainty of the reference titration solution concentration [U(c)]

CALCULATE according to Formula (D.18):

$$U(c)=k\times u_{\mathrm{c}}(c) \qquad \cdots\cdots(\text{D.18})$$

Appendix F

(Informative)

Assessment on expansion uncertainty of the glass gauge capacity

F.1 Method for determining the glass gauge capacity

DETERMINE according to 7.3.5.1 of JJG 196-2006. The capacity of the glass gauge at a standard temperature of 20°C (V_{20}) is calculated according to Formula (F.1):

$$V_{20} = m \times K(t) \quad \cdots\cdots (F.1)$$

Where:

m – Apparent mass of water contained in the glass gauge to be inspected, in gram (g);

K (t) – Commonly used glass gauge measurement K (t) value, in milliliter per gram (mL/g).

F.2 Assessment on expansion uncertainty of the glass gauge capacity

F.2.1 Class A assessment on relative standard uncertainty component of the capacity of the glass gauge at a standard temperature of 20°C

The Class A relative standard uncertainty component of the capacity of the glass gauge at a standard temperature of 20°C [$u_{Arel}(V_{20})$] is calculated according to Formula (F.2):

$$u_{Arel}(V_{20}) = \frac{s_{rel}}{\sqrt{n}} \quad \cdots\cdots (F.2)$$

Where:

s_{rel} – Relative standard deviation of the glass gauge capacity;

n - Number of verifications for the same capacity.

F.2.2 Class B assessment on relative composite standard uncertainty component of the capacity of the glass gauge at a standard temperature of 20°C

The relative composite standard uncertainty component introduced by the a – Half width of the rounding off of the commonly used glass gauge measurement K (t) value, in milliliter per gram (mL/g);

k – Coverage factor, uniform distribution, $k=\sqrt{3}$;

K (t) – Commonly used glass gauge measurement K (t) value, in milliliter per gram (mL/g).

F.2.2.3 Relative standard uncertainty component introduced by the pure water temperature [$u_{rel}(t)$]

CALCULATE according to Formula (F.6):

$$u_{\mathrm{rel}}(t)=\frac{a}{k\times \mathrm{K}(t)} \qquad \cdots\cdots\cdots\cdots (\mathrm{F.6})$$

Where:

a – K (t) value when the pure water temperature differs by 0.1°C;

k – Coverage factor, uniform distribution, $k=\sqrt{3}$;

K (t) – Commonly used glass gauge measurement K (t) value, in milliliter per gram (mL/g).

F.2.2.4 Relative standard uncertainty component introduced by the rounding off of the glass gauge capacity [$u_{rel}(r)$]

CALCULATE according to Formula (F.7):

$$u_{\mathrm{rel}}(r)=\frac{a}{k\times V_{20}} \qquad \cdots\cdots\cdots\cdots (\mathrm{F.7})$$

Where:

a – Half width of the rounding off of the glass gauge capacity at a standard temperature of 20°C, in milliliter (mL);

k – Coverage factor, uniform distribution, $k=\sqrt{3}$;

V_{20} – Capacity of the glass gauge at a standard temperature of 20°C, in milliliter (mL).

F.2.3 Composite standard uncertainty of the glass gauge capacity at a standard temperature of 20°C [$u_c(V_{20})$]

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