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Replacing HG/T 3696.2-2002

Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis – Part 2: Preparations of standard solutions for impurity

无机化工产品 化学分析用标准溶液、制剂及制品的制备 –
第 2 部分：杂质标准溶液的制备

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Foreword

HG/T 3696 “Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis – Part 2: Preparations of standard solutions for impurity” is divided into the following parts:

- Part 1: Preparations of standard volumetric solutions;

- Part 2: Preparations of standard solutions for impurity;

- Part 3: Preparation of reagent solutions.

This part is part 2 of HG/T 3696.

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

This part replaces HG/T 3696.2-2002 “Inorganic chemicals for industrial use - Preparations of standard solutions for impurity for chemical analysis”. As compared with HG/T 3696.2-2002, the main technical changes of this part in addition to editorial changes are as follows:

- CHANGE the name of this part in accordance with the requirements of GB/T 1.1-2009 into “Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis – Part 2: Preparations of standard solutions for impurity”;

- ADD the requirements for the required standard and reagent solutions (SEE 3.3);

- ADD chapter 4 “Warning” (SEE 4);

- MODIFY the name of Table 1 into “Preparation of standard stock solution for metallic element impurity” (SEE 5.1; clause 4.1 of 2002 version);

- ADD the method of using high purity magnesium to prepare magnesium impurity standard solution (SEE Table 1 of 5.1);

- ADD the method of using stannous chloride to prepare tin impurity standard solution (SEE Table 1 of 5.1);

- DELETE the preparation of the ferrous impurity standard solutions (SEE Table 1 of 4.1 in 2002 version);

- MODIFY the name of Table 2 into “Preparation of standard stock solution of non-metallic element impurity” (SEE 5.2; 4.2 of 2002 version);

- ADD the preparation of standard solutions of iodine, fluorine and bromine in Table 2 as well as another preparation method of sulfur and chlorine impurity standard solutions (SEE Table 2 of 5.2);

Inorganic chemicals for industrial use - Preparations of standard and reagent solutions for chemical analysis - Part 2: Preparations of standard solutions for impurity

1 Scope

This part specifies the preparation method of impurity standard solution for chemical analysis of inorganic chemical products.

This part is applicable to the preparation of the standard solution of a certain element, ion or molecule of a precise amount in a certain volume as required for the determination of the impurity content in the inorganic chemical product analysis. It may also be used for chemical analysis of other chemical products.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this Standard.

GB/T 6682-2008 Water for analytical laboratory use – Specification and test methods

HG/T 3696.1 Inorganic chemicals for industrial use – Preparations of standard and reagent solutions for chemical analysis – Part 1: Preparations of standard volumetric solutions

HG/T 3696.3 Inorganic chemicals for industrial use – Preparations of standard and reagent solutions for chemical analysis – Part 3: Preparation of reagent solutions

3 General provisions

3.1 The water used in this part shall comply with the level III water requirements as specified in GB/T 6682-2008, unless otherwise specified.

3.2 The purity of the reagents used in this part shall be above analytical pure. The readily oxidized metal shall, before use, be subjected to surface treatment.

3.3 The standard and reagent solutions as required by the Appendixes of this part shall, unless otherwise specified, be prepared in accordance with the provisions of HG/T 3696.1 and HG/T 3696.3.

3.4 When diluting the impurity standard solution, it shall use accurate measuring device (pipette, volumetric flask) to perform integral multiple dilution.

AND the volume of the standard solution measured each time shall be not less than 1.00 mL.

3.5 The standard solution for impurity determination shall, unless otherwise specified, be generally stored at room temperature for 12 months; AND it shall be prepared again when there is turbidity, precipitation, or color change.

3.6 The methods for the preparation of the impurity standard solutions as specified in this part are in alphabetical order.

3.7 The volume of the prepared stock solution in this part is for reference only; AND the user may adjust appropriately the preparation volume in accordance with the use amount, BUT it shall be at least not less than 100 mL.

4 Warning

Some of the reagents used in the preparation process of this part are toxic or corrosive, AND the operator must be careful! Any splashes on the skin must be flushed with water immediately, AND medical attention shall be sought immediately if it is serious.

5 Preparation method

5.1 Preparation of standard solution of metallic element impurity

Unless otherwise specified, this impurity standard solution (SEE Table 1) shall be stored in a suitable plastic bottle.

Table 1 -- Preparation of standard stock solution of metallic element impurity

Name	Solution concentration	Preparation method
Palladium (Pd)	1 mL solution contains 1 mg of palladium (Pd)	WEIGH 0.833 g of palladium chloride which is dried in advance at 105 °C ~ 110 °C for 1 h; ADD 15 ml of 1 +4 hydrochloric acid solution to dissolve it; TRANSFER all of it into a 500 ml volumetric flask; DILUTE it to the mark; SHAKE to make it uniform.
Barium (Ba)	1 mL solution contains 1 mg of barium (Ba)	1. WEIGH 0.889 g of barium chloride ($BaCl_2 \cdot 2H_2O$); DISSOLVE it in water; TRANSFER all of it into a 500 ml volumetric flask; DILUTE it to the mark; SHAKE to make it uniform. 2. WEIGH 0.718 g of barium carbonate which was dried at 110 °C to constant mass; ADD 1 + 4 hydrochloric acid solution to dissolve it; TRANSFER all of it into a 500 ml volumetric flask; DILUTE it to the mark; SHAKE to make it uniform.
Bismuth (Bi)	1 mL solution contains 1 mg of bismuth (Bi)	1. WEIGH 1.616 g of bismuth nitrate [$Bi(NO_3)_3 \cdot 5H_2O$]; ADD 50 ml of 1 +4 hydrochloric acid solution to dissolve it; TRANSFER all of it into a 500 ml volumetric flask; DILUTE it to the mark; SHAKE to make it uniform. 2. WEIGH 0.100 g of metal bismuth; DISSOLVE it in 6 mL of nitric acid; BOIL it to remove nitrogen dioxide gas; TRANSFER all of it into a 100 ml volumetric flask; DILUTE it to the mark; SHAKE to

Appendix A

(Informative)

Calibration of sulfur impurity standard solution

As for the sulfur (S) impurity standard solution which is prepared using the method 2 of Table 2 in clause 5.2, it may be calibrated using the following method if necessary:

ADD 10 mL of 1 mol/L zinc acetate solution into 250 mL iodine flask; PIPETTE 10.00 mL of sulfur [sulfide (by S)] and 20.00 mL of iodine standard solution [c ($1/2I_2$) ≈0.1 mol/L]; USE water to dilute it to 60 mL; ADD 5 mL of (1 + 5) sulfuric acid solution; tightly APPLY the water seal; SHAKE it uniformly; PLACE it in the dark for 5 min; USE the sodium thiosulfate standard titration solution [c ($Na_2S_2O_3$) ≈0.1 mol/L] to titrate the solution until it is in light yellow; ADD 1 mL of starch indicator solution; CONTINUE titration until the blue color disappears. Meanwhile USE 10 mL of water to substitute the sulfur impurity standard solution to make blank test.

The sulfur (S) content in the sulfur impurity standard solution is calculated by the mass concentration ρ of sulfur (S), it is expressed in (mg/mL), AND it is calculated in accordance with the formula (A.1):

$$\rho=\frac{(V_0-V_1)cM}{10} \quad \text{(A.1)}$$

Where:

V_1 – The volume value of the sodium thiosulfate standard titration solution as consumed to titrate the sulfur impurity standard solution, in the unit of milliliters (mL);

V_0 – The volume value of the sodium thiosulfate standard titration solution as consumed to titrate the blank reagent solution, in the unit of milliliters (mL);

c - The exact value of the concentration of sodium thiosulfate standard titration solution ($Na_2S_2O_3$), in moles per liter (mol/L);

M – Molar mass value of sulfur (1/2S), in grams per mole (g/mol) (M = 16.03).

Appendix B

(Normative)

Methods for determination of the content of two solutions

B.1 Determination of hexafluorosilicate (fluorosilicic acid)

WEIGH 3 g of hexafluorosilicate (fluorosilicic acid), accurate to 0.0002 g; PLACE it into the polyethylene cup; ADD 100 mL of water, 10 mL of saturated potassium chloride solution, and 3 drops of 10 g/L phenolphthalein indicator solution; COOL it to 0 °C; USE the sodium hydroxide standard titration solution [c (NaOH) $\approx$ 1 mol/L] to titrate it to pink; AND it will be deemed as end point if the color does not fade within 15 s; RECORD the volume of sodium hydroxide standard titration solution (V_1). And then HEAT it to 80 °C; CONTINUE using the sodium hydroxide standard titration solution [c (NaOH) $\approx$ 1 mol/L] to titrate it until the solution is in stable pink; RECORD the volume of sodium hydroxide standard titration solution (V_2).

The content of hexafluorosilicate is calculated by the mass fraction w_1 of hexafluorosilicate (H_2SiF_6), the result is expressed in w_1, AND it is calculated in accordance with the formula (B.1)

$$w_1 = \frac{(V_2 - V_1) \times 10^{-3} cM}{m} \times 100 \quad \text{(B.1)}$$

Where:

V_1 - The value of the volume of sodium hydroxide standard titration solution as consumed to titrate to the first end point, in milliliters (mL);

V_2 - The value of the volume of sodium hydroxide standard titration solution as consumed to titrate to the second end point, in milliliters (mL);

c – The accurate value of the concentration of the sodium hydroxide standard titration solution (NaOH), in moles per liter (mol/L);

m – The value of the mass of the sample, in grams (g);

M – The value of the molar mass of hexafluorosilicate (1/4 H_2SiF_6), in grams per mole (g/mol) (M = 36.02).

B.2 Determination of 30% hydrogen peroxide content

WEIGH 1.8 mL (about 2 g) of 30% hydrogen peroxide; INJECT it into a stoppered conical flask; WEIGH it, accurate to 0.0002 g. TRANSFER it into a 250 mL volumetric flask; DILUTE it to the mark; SHAKE it uniformly; MEASURE 25.00 mL of it; ADD 10 mL of 20% sulfuric acid; USE the potassium