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मानक

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Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

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“Step Out From the Old to the New”

IS 2316 (1990): Methods of preparation of standard solutions for colorimetric and volumetric analysis [CHD 1: Inorganic Chemicals]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
कैलोरीमितीय और आयतनमितीय विश्लेषणों के लिए
मानक घोल तैयार करने की पद्धति
(दूसरा पुनरीक्षण)

Indian Standard

METHODS OF PREPARATION OF STANDARD
SOLUTIONS FOR COLORIMETRIC AND
VOLUMETRIC ANALYSIS

(Second Revision)

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FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 28 February 1990, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

This standard was first issued in 1963 and revised in 1968. In this second revision, more standard solutions have been incorporated. New salts have been prescribed in many cases deleting the conventional salts for preparing the standard solutions.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF PREPARATION OF STANDARD SOLUTIONS FOR COLORIMETRIC AND VOLUMETRIC ANALYSIS

(Second Revision)

1 SCOPE

1.1 This standard prescribes the methods of preparation of standard solutions for the common colorimetric and volumetric determinations.

2 REFERENCES

2.1 The following Indian Standards have been referred to in this standard :

IS No.	Title
1070 : 1977	Water for general laboratory use (second revision)
2263 : 1979	Methods of preparation of indication solution for volumetric analysis (first revision)

3 GENERAL

3.1 Solutions for colorimetric determinations are

given in Section 1 and solutions for volumetric determinations are given in Section 2 and 3. Section 2 deals with primary standards which require no further standardization while Section 3 deals with secondary standards which require standardization before use. All volumetric titrations shall be carried out in duplicate.

3.2 Unless specified otherwise, all chemicals which do not contain water of crystallization shall be dried in an oven at $105 \pm 2^\circ\text{C}$ until free from moisture.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, chemicals of analytical reagent grade and distilled water (see IS 1070 : 1977) shall be employed in preparing solutions and in tests.

SECTION 1 COLORIMETRIC SOLUTIONS

5 STANDARD ALUMINIUM SOLUTION

5.1 Preparation

Dissolve 1.000 g of pure aluminium foil in 40 ml of (1 : 1) hydrochloric acid and dilute to 100 ml with water. One ml of this solution contains 1.0 mg of aluminium (as Al).

6 STANDARD AMMONIUM SOLUTION

6.1 Keep overnight about 5 g of pure ammonium chloride (NH_4Cl) in a vacuum dessicator using calcium chloride as dessicant.

6.1.1 Preparation

Dissolve 3.1409 g of dried material in water and dilute to 1 000 ml. One ml of this solution contains 1.0 mg of ammonia (as NH_3).

6.1.2 A solution containing 3.8190 g of dried material per litre contains 1.0 mg ammonical nitrogen (as N) per ml.

7 STANDARD ANTIMONY SOLUTION

7.1 Preparation

Dissolve 2.7427 g of potassium antimonyl tartrate [$\text{K}(\text{SbO})\text{C}_4\text{H}_4\text{O}_6 \cdot \frac{1}{2}\text{H}_2\text{O}$] in 300 ml of 10

percent sulphuric acid. Dilute to 1 000 ml with 10 percent sulphuric acid. One ml of this solution contains 1.0 mg of antimony (as Sb).

8 STANDARD ARSENIC SOLUTION

8.1 Preparation

Dissolve 1.3203 g of arsenious trioxide (As_2O_3) in minimum volume of dilute sodium hydroxide solution containing 4 g of sodium hydroxide in 100 ml of water. Neutralize with dilute sulphuric acid. Dilute with water containing 10 ml concentrated sulphuric acid to 1 000 ml. One ml of this solution contains 1.0 mg of arsenic (as As).

9 STANDARD BARIUM SOLUTION

9.1 Preparation

Dissolve 1.7787 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of barium (as Ba).

10 STANDARD BISMUTH SOLUTION

10.1 Preparation

Dissolve 1.000 g of bismuth metal in nitric acid. Add 50 ml of 1 : 1 sulphuric acid and evaporate

to dense white fumes. Cool and dilute to 1 000 ml with water. One millilitre of this solution contains 1.0 mg of bismuth (as Bi).

11 STANDARD BORON SOLUTION

11.1 Preparation

Dissolve 5.719 9 g of boric acid (H_3BO_3), previously dried to constant mass at $105 \pm 5^\circ C$, in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of Boron (as B); store in a polyethylene bottle.

12 STANDARD CADMIUM SOLUTION

12.1 Preparation

Dissolve 1.000 g of pure cadmium metal chippings in minimum volume of dilute nitric acid. Add 50 ml of 1 : 1 sulphuric acid and evaporate to dense white fumes. Dilute to 1 000 ml with water. One millilitre of this solution contains 1.0 mg of cadmium (as Cd).

13 STANDARD CHLORIDE SOLUTION

13.1 Preparation

Dissolve 1.648 5 g of sodium chloride in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of chloride (as Cl).

14 STANDARD CHROMIUM SOLUTION

14.1 Preparation

Dry about 4 g of pure potassium dichromate ($K_2Cr_2O_7$) at $110^\circ \pm 5^\circ C$ to constant mass. Dissolve 2.828 9 g of dried material in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of chromium (as Cr^{+6}).

15 STANDARD COBALT SOLUTION

15.1 Preparation

Dissolve 1.000 g of pure cobalt metal in minimum volume of 1 : 1 nitric acid. Add 40 ml of 1 : 1 sulphuric acid. Heat to dense white fumes. Dilute to 1 000 ml with water. One millilitre of this solution contains 1.0 mg of cobalt (as Co).

15.2 Alternatively, dissolve 4.769 8 g of cobalt sulphate ($CoSO_4 \cdot 7H_2O$) in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of cobalt (as Co).

16 STANDARD COPPER SOLUTION

16.1 Preparation

Clean the surface of an unenamelled copper wire with a sand paper. Dissolve 1.000 g of copper wire in minimum quantity of dilute nitric acid solution. Add about 40 ml of 1 : 1 sulphuric acid

and evaporate to dense white fumes. Dilute to 1 000 ml with water. One millilitre of this solution contains 1.0 mg of copper (as Cu).

16.2 Alternatively, dissolve 3.929 1 g of cupric sulphate ($CuSO_4 \cdot 5H_2O$) in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of copper (as Cu).

17 STANDARD IODINE SOLUTION

17.1 Preparation

Dissolve 1.308 1 g of potassium iodide (KI), previously dried at $110 \pm 5^\circ C$ to constant mass, in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of iodine (as I).

18 STANDARD IRON SOLUTION

18.1 Preparation

Dissolve 7.021 5 g of ammonium ferrous sulphate [$(NH_4)_2SO_4 \cdot FeSO_4 \cdot 6H_2O$] in 200 ml water containing 10 ml concentrated sulphuric acid. Dilute with water to 1 000 ml. One millilitre of this solution contains 1.0 mg of iron (as Fe^{2+}).

19 STANDARD LEAD SOLUTION

19.1 Preparation

Dissolve 1.598 5 g of lead nitrate [$Pb(NO_3)_2$] in 200 ml of 1 percent nitric acid solution. Dilute to 1 000 ml with 1 percent nitric acid. One millilitre of this solution contains 1.0 mg of lead (as Pb).

19.2 Alternatively dissolve 1 000 g of pure lead in 40 ml of 1 : 1 nitric acid. Dilute to 1 litre with water. One millilitre of this solution contains 1.0 mg of lead (as Pb).

19.3 If lead is to be determined by turbidimetric procedure, weigh 1.835 8 g of lead acetate [$Pb(CH_3CO_2)_2 \cdot 3H_2O$] and dilute to 1 000 ml with water. One millilitre of this solution contains 1.0 mg of lead (as Pb).

20 STANDARD MAGNESIUM SOLUTION

20.1 Preparation

Dissolve 1.000 g of magnesium turnings or 1.658 3 g of magnesium oxide (MgO) in minimum amount of dilute hydrochloric acid and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of magnesium (as Mg).

21 STANDARD MANGANESE SOLUTION

21.1 Preparation

Dissolve 1.000 g pure manganese metal in 30 ml of 1 : 1 nitric acid. Heat to expel brown fumes.

Cool and dilute with water to 1 000 ml. One millilitre of this solution contains 1.0 mg of manganese (as Mn).

21.2 Alternatively heat about 5 g of manganese sulphate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$) at $500^\circ \pm 10^\circ\text{C}$ to constant mass. Then dissolve 2.748 5 g of anhydrous material in 50 ml of 10 percent nitric acid. Dilute to 1 000 ml with water. One millilitre of this solution contains 1.0 mg of manganese (as Mn).

22 STANDARD MOLYBDENUM SOLUTION

22.1 Preparation

Dry about 2 g of molybdenum trioxide (MoO_3) at $105 \pm 5^\circ\text{C}$ to constant mass and cool. Dissolve 1.500 3 g of the dried material in a few ml of dilute sodium hydroxide solution. Dilute with water, render slightly acidic with dilute hydrochloric acid and make up to 1 000 ml. One millilitre of this solution contains 1.0 mg of molybdenum (as Mo).

23 STANDARD NICKEL SOLUTION

23.1 Preparation

Dissolve 1.000 g of 99.9 percent pure nickel in 30 ml of 1:1 nitric acid. Boil to expel brown fumes. Cool and dilute to 1000 ml with water. One millilitre of this solution contains 1.0 mg of nickel (as Ni).

23.2 Alternatively dissolve 6.728 8 g of nickel ammonium sulphate [$\text{NiSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of nickel (as Ni).

24 STANDARD NITRATE SOLUTION

24.1 Preparation

Dry about 2 g of potassium nitrate (KNO_3) to $105 \pm 5^\circ\text{C}$ to constant mass and cool. Dissolve 1.630 6 g of dried material in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of nitrate (as NO_3).

25 STANDARD NITRITE SOLUTION

25.1 Preparation

Dissolve 1.499 7 g of sodium nitrite (NaNO_2) in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of nitrite (as NO_2).

26 STANDARD PHOSPHATE SOLUTION

26.1 Preparation

Dry about 3 g of potassium dihydrogen phosphate (KH_2PO_4) at $105 \pm 5^\circ\text{C}$ to constant mass and

cool. Dissolve 1.432 9 g of dried material in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of phosphate (as PO_4).

27 STANDARD POTASSIUM SOLUTION

27.1 Preparation

Dry about 3 g of potassium chloride (KCl) at $105 \pm 5^\circ\text{C}$ to constant mass and cool. Dissolve 1.906 8 g of dried material in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of potassium (as K).

28 STANDARD SODIUM SOLUTION

28.1 Preparation

Dry about 4 g of sodium chloride (NaCl) at $500 \pm 20^\circ\text{C}$ in a platinum crucible to constant mass and cool. Dissolve 2.542 1 g of dried material in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of sodium (as Na).

29 STANDARD SILICA SOLUTION

29.1 Preparation

Fuse 0.500 0 g of pure precipitated silica (SiO_2) previously dried at $110 \pm 5^\circ\text{C}$ with 5 g anhydrous sodium carbonate in a platinum crucible for one hour and leach the melt with water. Filter and dilute to 500 ml with water. Draw a 50 ml aliquot acidify with hydrochloric acid and determine silica content gravimetrically. Suppose silica content (as SiO_2), percent by mass in precipitated silica is X , then one millilitre of this solution contains $1.0 \times X/100$ mg of silica (SiO_2). Store in a polyethylene bottle.

30 STANDARD SILVER SOLUTION

30.1 Preparation

Dissolve 1.000 g of 99.99 percent pure silver foil in about 30 ml of 1:1 nitric acid. Warm to expel brown fumes. Dilute to 1 000 ml with water and store in amber coloured bottle. One millilitre of this solution contains 1.0 mg of silver (as Ag).

30.2 Alternatively dissolve 1.574 8 g of silver nitrate (AgNO_3) in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of silver (as Ag).

NOTES

1 Care should be taken that there should be minimum exposure of silver metal or its salts to direct sunlight during weighing.

2 All apparatus should be carefully washed with chloride free distilled water before use.

31 STANDARD SULPHATE SOLUTION

31.1 Preparation

Ignite about 5 g of sodium sulphate (Na_2SO_4) at $500 \pm 20^\circ\text{C}$ to constant mass. Take 1.478 7 g of

the anhydrous material and dissolve in water and dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of sulphate (as SO_4).

32 STANDARD SULPHIDE SOLUTION

32.1 Preparation

Dissolve about 15 g of sodium sulphide ($\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$) in water and dilute to 500 ml in a volumetric flask. Pipette out 25 ml of this solution in a conical flask and add 50 ml of 0.1 N iodine solution to it. Keep for 5 minutes and titrate with 0.1 N sodium thiosulphate using starch as indicator to a colourless end point. Similarly titrate 50 ml of blank iodine solution with sodium thiosulphate. The difference of two readings gives the volume of 0.1 N iodine equivalent to sulphide content in 25 ml solution. 1 ml of 0.1 N iodine solution consumed corresponds to 1.603 mg of sulphide. Thus this solution (A) contains $1.603 \times V/25$ mg sulphide per millilitre. Dilute, in volumetric flask, a volume of sodium sulphide solution containing 1 g of sulphide to 1 000 ml with water. One millilitre of this solution contains 1.0 mg of sulphide (as S).

NOTE — This solution is unstable and shall be prepared fresh before use.

33 STANDARD TIN SOLUTION

33.1 Preparation

Dissolve 1.000 g of 99.95 percent pure metallic tin in 100 ml of 1:1 hydrochloric acid and dilute with the same acid to 1 000 ml. One millilitre of this solution contains 1.0 mg of tin (as Sn).

34 STANDARD TITANIUM SOLUTION

34.1 Preparation

Weigh 7.3965 g of potassium titanyl oxalate [$\text{K}_2\text{OTi}(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$] into a Kjeldahl flask, add 8 g of ammonium sulphate and 50 ml of concentrated sulphuric acid. Gradually heat the contents and boil for 10 minutes. Cool, pour the solution into 500 ml dilute (4 N) sulphuric acid and dilute to 1 000 ml. Aliquot of the solution may be estimated for TiO_2 , if necessary. One millilitre of this solution contains 1.0 mg of titanium (as Ti).

35 STANDARD VANADIUM SOLUTION

35.1 Preparation

Keep over night about 5 g of ammonium vanadate (NH_4VO_3) in a vacuum desiccator using calcium chloride as desiccant.

Dissolve 2.2963 g of ammonium vanadate in minimum content of concentrated nitric acid. Heat, cool and add 10 ml concentrated nitric acid and then dilute to 1 000 ml. One millilitre of this solution contains 1.0 mg of vanadium (as V).

36 STANDARD ZINC SOLUTION

36.1 Preparation

Dissolve 1.000 g of 99.95 percent pure metallic zinc in 40 ml of 1:1 hydrochloric acid and dilute to 1 000 ml with water. One millilitre of this solution contains 1.0 mg of zinc (as Zn).

SECTION 2 PRIMARY STANDARD VOLUMETRIC SOLUTIONS

37 STANDARD ARSENIOS OXIDE (OR SODIUM ARSENITE SOLUTION)

37.1 Preparation of 0.1 N Solution

Dissolve 4.946 g of pure arsenious oxide (As_2O_3) in 30-40 ml of warm water containing 2-3 g of sodium hydroxide. Dilute the solution to about 250 ml. Neutralize the excess alkali with dilute sulphuric acid (approximately 2 N) until a few drops of phenolphthalein are just decolorized. Add 500 ml of a 5 percent solution of sodium bicarbonate. If the colour returns, add a few drops of sulphuric acid to decolorize it. Dilute the solution with water to 1 000 ml.

38 STANDARD CALCIUM SOLUTION

38.1 Preparation of 0.5 N Solution

Dissolve 2.5022 g of pure calcium carbonate in 50 ml of water containing 10.6 ml of hydrochloric

acid. When the effervescence ceases, boil the solution to remove dissolved carbon dioxide. Cool to room temperature and dilute to 1 000 ml with water.

39 STANDARD OXALIC ACID

39.1 Preparation of 0.1 N Solution

Dissolve 6.3033 g of crystallized oxalic acid (dihydrate) in about 200 ml of water and dilute to 1 000 ml.

40 STANDARD POTASSIUM BROMATE SOLUTION

40.1 Preparation of 0.1 N Solution

Dry about 4 g of potassium bromate (KBrO_3) at $150 \pm 5^\circ\text{C}$ to constant mass and cool. Dissolve 2.7833 g of the dried material in water and dilute to 1 000 ml.

41 STANDARD POTASSIUM DICHROMATE SOLUTION**41.1 Preparation of 0.1 N Solution**

Carefully pulverise about 6 g of potassium dichromate ($K_2Cr_2O_7$). Dry at $120 \pm 5^\circ C$ to constant weight and cool. Dissolve 4.9031 g of dried material in water and dilute to 1 000 ml.

42 STANDARD POTASSIUM HYDROGEN PHTHALATE SOLUTION**42.1 Preparation of 0.1 N Solution**

Dry about 25 g of potassium hydrogen phthalate ($KHC_8H_4O_4$) at $105 \pm 5^\circ C$ for 2 hours and cool. Weigh 20.4223 g of dried material and dissolve in carbon dioxide free from water and dilute to 1 000 ml.

43 STANDARD POTASSIUM IODATE SOLUTION**43.1 Preparation of 0.1 N Solution**

Dry about 5 g of potassium iodate (KIO_3) at $105 \pm 5^\circ C$ to constant mass and cool. Dissolve 3.5667 g of dried material in water and dilute to 1 000 ml.

44 STANDARD SODIUM CARBONATE SOLUTION**44.1 Preparation of 0.1 N Solution**

Dry about 10 g of sodium carbonate (Na_2CO_3) at $260 \pm 10^\circ C$ for 1 hour and cool. Weigh 5.2994 g of dried material, dissolve in water and dilute to 1 000 ml.

45 STANDARD SODIUM CHLORIDE SOLUTION**45.1 Preparation of 0.1 N Solution**

The sodium chloride may be further purified (if necessary) by precipitating the sodium chloride from a nearly saturated solution by passing hydrogen chloride gas into the solution through inverted funnel. The precipitated sodium chloride is collected on a sintered crucible or other suitable filter medium and dried. About 7.8 g of this material is transferred to platinum crucible and ignited at $500 \pm 20^\circ C$. 5.8443 g of ignited and cooled material is then dissolved in water and diluted to 1 000 ml.

46 STANDARD SODIUM OXALATE SOLUTION**46.1 Preparation of 0.1 N Solution**

Dry sodium oxalate ($C_2Na_2O_4$) at $100 \pm 5^\circ C$ to constant mass and cool. Dissolve 6.7000 g of dried material in water and dilute to 1 000 ml.

SECTION 3 SECONDARY STANDARD VOLUMETRIC SOLUTIONS**47 STANDARD THIOCYANATE SOLUTION****47.1 Preparation of Approximate 0.1 N Solution**

Dissolve about 8 g of ammonium thiocyanate (NH_4CNS) or 10 g of potassium thiocyanate ($KCNS$) in water and dilute to 1 000 ml. Standardize the solution as follows:

Pipette 25 ml of standard 0.1 N silver nitrate solution (see 54) into a 250-ml conical flask, add 5 ml of 6 N nitric acid (1:3) and 1 ml of ferric alum indicator solution (see IS 2263: 1979). Titrate with ammonium thiocyanate or potassium thiocyanate solution from a burette. At first a white precipitate is produced imparting a milky appearance to the solution, and as each drop of thiocyanate fills in, it produces a pink colour, which quickly disappears on shaking. As the end point approaches, the precipitate becomes flocculent and settles easily. The end point is reached when one drop of the thiocyanate solution produces a faint brown colour, which no longer disappears on vigorous shaking. (It is essential to shake vigorously during the

titration in order to obtain correct results). Repeat the titration with two other 25 ml portions of silver nitrate solution. Individual titrations should agree within 0.1 ml. Determine the reagent blank. Calculate the normality of the thiocyanate solution on the basis of normality of standard silver nitrate solution.

NOTE — The strength of the standard solution thus prepared remains the same for a long period if evaporation is prevented.

48 STANDARD CERIC SULPHATE SOLUTION**48.1 Preparation of Approximate 0.1 N Solution**

Weigh out 36 g of pure ceric sulphate [$Ce(SO_4)_2$] into a 600-ml beaker, add 56 ml of 1:1 sulphuric acid and stir with frequent additions of water and gentle warming until the material is dissolved. Cool the solution and dilute with water to 1 000 ml in a volumetric flask.

48.2 Alternatively, weigh out 66 g of pure ceric ammonium sulphate [$Ce(SO_4)_2 \cdot 2(NH_4)_2SO_4$] into a solution prepared by adding

28 ml of sulphuric acid (sp. gr. 1.84) to 500 ml of water. Stir the mixture until the material is dissolved. Transfer to a 1 000-ml volumetric flask and make up to the mark with water. Standardize the solution as follows.

48.2.1 With Arsenious Oxide

Weigh accurately 0.10 to 0.15 g of arsenious oxide (As_2O_3), previously dried at 100°C for 1 hour into a 250-ml conical flask. Add 15 ml of sodium hydroxide solution (2 N) and heat gently to hasten solution. Cool to room temperature and add 25 ml of sulphuric acid (1 : 3). Now dilute to 100 ml with water and add 3 drops of osmium tetroxide solution (0.01 N prepared by dissolving 0.255 g of osmium tetroxide in 100 ml of 0.1 N sulphuric acid) as a catalyst (see Note) and one drop of ferroin indicator solution. Finally titrate this solution with ceric sulphate solution until the orange colour changes sharply to colourless or pale blue. Calculate the normality of the ceric sulphate solution on the basis of arsenious oxide taken. Each 4.946 mg of As_2O_3 is equivalent to 1 ml of 0.1 N ceric sulphate.

NOTE — In place of osmium tetroxide, iodine monochloride (prepared by adding 25 ml of 0.04 M potassium iodide solution and 40 ml of 12 M hydrochloric acid to 20 ml of 0.025 M potassium iodate solution) may be used as a catalyst as follows:

Dissolve the arsenious oxide as described above and add 20 ml of concentrated hydrochloric acid. Dilute to 100 ml and add 2.5 ml of iodine monochloride solution and one drop of ferroin indicator. Titrate until the orange colour begins to fade and returns only slowly after the addition of each drop of ceric sulphate solution. Now heat the solution to about 50°C and continue the addition of ceric sulphate solution drop by drop until the colour is completely discharged and does not return within one minute. Calculate the normality of the ceric sulphate solution on the basis of arsenious oxide taken.

48.2.2 With Sodium Oxalate

Weigh accurately 0.15 to 0.20 g of sodium oxalate ($Na_2C_2O_4$) into a 250-ml beaker. Add 75 ml of water, 20 ml of 12 N-hydrochloric acid and 1.5 ml of iodine monochloride solution as catalyst (see Note above). Heat to about 50°C and titrate with ceric sulphate solution potentiometrically using platinum electrode or visually with 2 to 3 drops of ferroin indicator solution, until the solution remains pale blue for at least one minute. Do not allow the temperature to fall below 45°C. Calculate the normality of ceric sulphate solution on the basis of sodium oxalate taken.

48.2.3 With Sodium Thiosulphate

Transfer a 25 ml portion of standardized 0.1 N sodium thiosulphate solution into a 500-ml conical flask. Add 0.5 g of potassium iodide, 20 ml of 0.2 percent freshly prepared starch solution, dilute to 50 ml and titrate with ceric sulphate solution till the blue colour persists for 1 minute.

Repeat the titration with two other similar portions of standardized 0.1 N sodium thiosulphate solution. Calculate the normality of the ceric sulphate solution on the basis of normality of standard sodium thiosulphate solution.

49 STANDARD DISODIUM ETHYLENE DIAMINE, TETRAACETATE DIHYDRATE (EDTA) SOLUTION

49.1 Preparation of Approximate 0.05 M Solution (0.1 N)

Dissolve 18.6119 g of disodium ethylene diamine tetraacetate dihydrate [$(OOCCH_2)_2NH_2CH_2CH_2NH_2(CH_2COO)_2Na_2 \cdot 2H_2O$] in water and dilute to 1 000 ml. Store in a polyethylene bottle. Standardize the solution as follows:

Transfer 10 ml of the standard 0.1 N zinc sulphate solution, prepared by dissolving 28.756 g of zinc sulphate in water and made up to 1 litre. Add 50 ml of buffer solution (67.5 g of ammonium chloride dissolved in 570 ml of ammonium hydroxide and diluted to 1 litre). Titrate it against the EDTA solution till the final blue colour is obtained.

50 STANDARD FERROUS AMMONIUM SULPHATE SOLUTION

50.1 Preparation of Approximate 0.1 N Solution

Transfer about 40 g of ferrous ammonium sulphate crystals [$FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$] to a 1 000-ml volumetric flask. Shake until the material has dissolved in water containing 20 ml concentrated sulphuric acid and make up to the mark with water. Standardize the solution as follows:

Transfer 25 ml of the solution to a 250-ml conical flask, add 25 ml of 1 N sulphuric acid, and titrate with standard 0.1 N potassium permanganate (see 53) solution to the appearance of permanent pink colour.

Alternatively, pipette 25 ml of the solution to a conical flask, add 5 ml of phosphoric acid (sp. gr. 1.7), 0.3 ml of 0.01 M solution of the sodium salt of diphenylamine sulphuric acid, and dilute to about 300 ml. Titrate with standard 0.1 N potassium dichromate solution (see 40) slowly while stirring until the original pure green colour change to a greenish grey. At this point, add the dichromate one drop at a time until the tinge of purple colour appears and remains on stirring. From the total volume of dichromate used, subtract 0.05 ml as a blank on the indicator and calculate the normality of ferrous ammonium sulphate solution on the basis of standard potassium dichromate solution.

51 STANDARD HYDROCHLORIC ACID**51.1 Preparation of Approximate 0.1 N Solution**

Transfer by means of graduated cylinder or a burette 9 ml of concentrated hydrochloric acid (HCl) into a 1 000-ml volumetric flask containing about 500 ml of water cool and dilute to 1 000 ml. Standardize the solution as follows:

Transfer 25 ml of standard 0.1 N sodium carbonate solution (see 43) to 250-ml conical flask, dilute to 50-75 ml with water, and add 2 drops of methyl orange indicator (see IS 2263 : 1979). Titrate the sodium carbonate solution against the hydrochloric acid to be standardized, continue the addition until the colour due to methyl orange becomes a very faint yellow. Boil the solution and cool again. Continue the titration by adding the acid dropwise and carefully until the colour of the methyl orange becomes orange or faint pink. The titration is repeated with two other portions of sodium carbonate solution. Individual titration should agree within 0.1 ml. Calculate the normality of the hydrochloric acid on the basis of standard carbonate solution.

52 STANDARD IODINE SOLUTION**52.1 Preparation of Approximate 0.1 N Solution**

Transfer 12.7 g of resublimed iodine (I_2) to a 250-ml beaker containing 25 g of iodate free potassium iodide and 25 ml of water. Stir until all iodine is dissolved and dilute with water to 1 000 ml. Preserve in an amber-coloured glass stoppered bottle in a cool place. Standardize the solution as follows:

Transfer 25 ml of iodine solution to 250-ml conical flask and dilute to about 100 ml with water. Titrate against standard 0.1 N sodium thiosulphate solution from a burette until the colour is reduced to pale yellow. Add 2 ml of starch solution and continue titration until the solution is just colourless. Repeat the titration with two more 25 ml portions of iodine solution. The titration should agree within 0.1 ml. Calculate the normality of the iodine solution on the basis of standard sodium thiosulphate solution.

NOTE — The strength of iodine solution deteriorates on keeping. It shall always be standardized before use.

53 STANDARD POTASSIUM FERROCYANIDE SOLUTION**53.1 Preparation of Approximate 0.05 M Solution**

Dissolve 21.125 g of potassium ferrocyanide [$K_4Fe(CN)_6 \cdot 3H_2O$] and 0.5 g of pure potassium ferrocyanide in previously boiled water and dilute to 1 000 ml. The solution shall be allowed to

stand for four weeks before using. Standardize the solution as follows:

Take into a 400-ml beaker about 0.35 g accurately weighed metallic zinc (99.95 percent pure) cover with water and add 10 ml concentrated hydrochloric acid to dissolve. Add 15 ml of ammonia, neutralize with hydrochloric acid and add 3 ml excess. Dilute to about 200 ml with water. Heat to 70°C approximately, add 2 drops of diphenylamine indicator solution. While stirring vigorously titrate with potassium ferrocyanide to the colour change from purple to persistent yellowish green. Repeat two more titrations. Calculate zinc equivalent of potassium ferrocyanide solution and then calculate the normality.

NOTE — The true end point is sharp, persistent change from purple to a yellowish green. Initially a blue colour is developed after addition of a few drops of potassium ferrocyanide solution. About 0.5-1.0 ml before end point, the colour changes to purple. After this, titration should be performed dropwise for a sharp end point.

54 STANDARD POTASSIUM PERMANGANATE SOLUTION**54.1 Preparation of Approximate 0.1 N Solution**

Carefully dissolve 3.2 g of potassium permanganate ($KMnO_4$) in water and dilute to 1 000 ml. Allow to stand for 2-3 days in an amber-coloured glass stoppered bottle. Filter off any manganate hydroxide through glass wool. Store in an amber-coloured glass-stoppered bottle. Standardize the solution as follows:

Pipette out 25 ml of 0.1 N sodium oxalate solution (see 45) and add 50 ml of 10 percent sulphuric acid. Add 20 ml of potassium permanganate solution from a burette while shaking till the solution is decolourized and then heat to $55 \pm 5^\circ C$. At this temperature complete the titration stirring gently with a thermometer and allowing time for each drop to be decolourized before adding the rest. A faint pink colour which persists for 30 second after the addition of last drop shall be the end point. Repeat the titration with two more volumes of sodium oxalate solution. Run a blank determination using 50 ml of 10 percent sulphuric acid. Calculate the normality of potassium permanganate based on the strength of standard sodium oxalate solution.

55 STANDARD SILVER NITRATE SOLUTION**55.1 Preparation of Approximate 0.1 N Solution**

Dissolve 16.9873 g of silver nitrate ($AgNO_3$) in water and dilute to 1 000 ml. All apparatus should be thoroughly washed with absolutely chloride free distilled water before use. Store in an amber

coloured glass-stoppered bottle. Standardize the solution as follows:

Pipette out 25 ml of standard 0.1 N sodium chloride solution (*see 27*) into a 250-ml conical flask resting on a white porcelain tile. Add a few drops of potassium chromate indicator solution (*see IS 2263 : 1979* 'Methods of preparation of indicator solutions') and titrate against silver nitrate until a faint but distinct reddish-brown colour persists after vigorous shaking.

Determine the indicator blank correction by adding 1 ml of the indicator to a volume of water equal to the final volume in the titration and then silver nitrate solution until the colour of the blank matches that of the solution titrated. The indicator blank correction should not amount to more than 0.03 to 0.1 ml of silver nitrate.

Repeat the titration with two further 25 ml portions of sodium chloride solution. The titrations should agree within 0.1 ml. Calculate the normality of silver nitrate solution on the basis of normality of sodium chloride.

56 STANDARD SODIUM HYDROXIDE SOLUTION

56.1 Preparation of Approximate 0.1 N Solution

Dissolve 5 g of sodium hydroxide (NaOH) in 100 ml of boiled and carbon-dioxide free water in an Erlenmeyer flask. Add about 0.5 g of barium hydroxide and dilute to 1 000 ml with water. Store in a polyethylene bottle. Standardize this solution as follows:

Determine the normality by titrating 25 ml of this solution against 0.1 N standard potassium hydrogen phthalate solution (*see 41*) using phenolphthalein as indicator.

Repeat the titration with two further 25 ml portions of sodium hydroxide solution. The various titrations should agree within 0.1 ml.

Calculate the normality of sodium hydroxide solution on the basis of standard potassium hydrogen phthalate solution.

57 STANDARD SODIUM THIOSULPHATE SOLUTION

57.1 Preparation of Approximate 0.1 N Solution

Weigh 25 g of sodium thiosulphate crystals ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$), dissolve in water and dilute 1 000 ml. Standardize the solution as follows:

Transfer 25 ml of the standard 0.1 N potassium dichromate solution (*see 40*) into a 250-ml conical flask. Add 2 g of potassium iodide and 8 ml of concentrated hydrochloric acid. Mix thoroughly and titrate with sodium thiosulphate, swirling the liquid constantly until the brown colour changes to yellowish green. Add 2 ml of starch solution and continue the titration until the colour changes sharply from blue to light green. Calculate the normality of sodium thiosulphate solution on the basis of normality of potassium dichromate.

58 STANDARD SULPHURIC ACID

58.1 Preparation of Approximate 0.1 N Solution

Transfer 3 ml of sulphuric acid (sp gr 1.84) into a 1 000-ml volumetric flask containing about 500 ml of water. When cold, dilute to 1 000 ml. Standardize the solution as follows:

Transfer 25 ml of standard 0.1 N sodium hydroxide solution (*see 55*) into 250-ml conical flask. Titrate with sulphuric acid solution using phenolphthalein indicator. Repeat the titration with two more 25 ml portions of sodium hydroxide solution. The various titration should agree within 0.1 ml. Calculate the normality of sulphuric acid solution on the basis of standard sodium hydroxide solution.

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