5. CHAPTER V

PREPARATION OF SOLUTIONS

Use accurately calibrated equipment, which means meets the TS specifications.

Working temperature of standard solution should approximate that of its temperature during preparation and standardisation. If temperature correction necessary, sufficient accuracy may be obtained by use of Tables.

Table 5.1. Temperature corrections for volume of aqueous solutions (AOAC, 1990).

	Correction ml at						
Volume of standard solution (ml)	8°C	12 °C	16 °C	20 °C	24 °C	28 °C	30 °C
10	0.01	0.01	0.01	0.00	0.00	-0.02	-0.02
20	0.03	0.02	0.01	0.00	-0.02	-0.03	-0.03
25	0.03	0.03	0.02	0.00	-0.02	-0.04	-0.05
30	0.04	0.03	0.02	0.00	-0.02	-0.05	-0.07
40	0.06	0.04	0.03	0.00	-0.03	-0.07	-0.09
50	0.07	0.06	0.03	0.00	-0.04	-0.09	-0.12

5.1. Preparations of Indicators

5.1.1. Phenolphthalein

1 % phenolphthalein (w/w) solution is prepared by using alcohol.

5.1.2. Methylene Blue

1 % methylene blue (w/w) solution is prepared by using water.

5.1.3. Methylene Orange

0.1 % methylene orange (w/w) solution is prepared by using water.

5.1.4. Ferric Alum

Saturated solution of $FeNH_4(SO_4)_2.12H_2O$ in water.

5.1.5. Potassium Chromate

5 % potassium chromate, K_2CrO_4, (w/w) solution is prepared by using water.

5.1.6. Methylene Red

1 % methylene red solution is prepared by using water.

5.2. Standard Solution of Hydrochloric Acid, HCl

Table 5.2. Approximate volumes of 36.5–38 % HCl required to make 10 liter standard solution.

Approximate normality	ml HCl to be diluted to 10 liter
0.01	8.6
0.02	17.2
0.10	86.0
0.50	430.1
1.00	860.1

Standardisation of HCl Solutions

Titrate 40 ml against alkaline solution (NaOH) of same concentration as acid being standardised in 300 ml flask that has been swept free from CO_2, using CO_2-free water and 3 drops of phenolphthalein as indicator.

Normality = (ml standard alkali * normality of alkali) / ml HCl

If more concentrated than desired, dilute solution to required normality value by following formula:

$$V_1 = V_2 * N_2 / N_1$$

where N_2 and V_2 represent normality and volume of stock solution and V_1 volume of stock solution should be diluted to obtain desired normality, N_1.

Check the normality of final solution by titration as above.

5.3. Preparation of Standard Sodium Hydroxide Solution, NaOH

To 1 part NaOH (reagent quality containing < 5 % Na_2CO_3) in flask add 1 part water and swirl until solution is complete.

Table 5.3 Approximate volumes of NaOH solution necessary to make 10 liter standard solution.

Approximate normality	ml NaOH to be diluted to 10 liter
0.01	8.6
0.02	17.2
0.10	86.0
0.50	430.1
1.00	860.1

Add required volume of NaOH solution (1+1) to 10 liter CO_2-free water. Check normality, as in standardisation section, which should be slightly high and adjust to desired concentration by following formula

$$V_1 = V_2 * N_2 / N_1$$

where N_2 and V_2 represent normality and volume of stock solution and V_1 volume of stock solution should be diluted to obtain desired normality, N_1.

Check the normality of final solution by titration as above.

Standardisation of NaOH Solution

Accurately weigh enough dried acid potassium phthalate, $KHC_8H_4O_4$, to titrate 40 ml and transfer to 300 ml flask that has been swept free from CO_2. Add 50 ml cool CO_2-free water. Stopper flask and swirl gently until sample dissolved. Titrate to pH 8.6 with solution being standardised, taking 3 drops of phenolphthalein as indicator. Determine volume NaOH required to produce end point of blank by matching color in another flask containing 3 drops of phenolphthalein and same volume of CO_2-free water. Subtract volume required from that used in first titration and calculate normality.

Normality = g $KHC_8H_4O_4$ * 1000/ml NaOH * 204.229

5.4. Preparation of Standard Sulphuric Acid Solution, H_2SO_4

Table 5.4. Approximate volumes of 95–98 % H_2SO_4 required to make 10 liter standard solution.

Approximate normality	ml H_2SO_4 to be diluted to 10 liter
0.01	2.8
0.02	5.6
0.10	27.7
0.50	138.1
1.00	276.1

Standardisation of H_2SO_4 Solutions

Standardisation of H_2SO_4 can be carried out as in HCl (Section 5.2)

5.5. Standard Solution of Ammonium and Potassium Thiocyanates

Prepare 0.1 N solution from reagents free from Cl; using 7.612 g NH_4SCN in 1 liter or 9.718 g KSCN in 1 liter.

5.6. Standard Solution of Sodium Thiosulphate

Dissolve 25 g $NaS_2O_3.5H_2O$ in 1 liter of water. Boil gently 5 min. transfer while hot to storage bottle previously cleaned with hot chromic acid cleaning solution and rinsed with warm boiled water.

5.7. Standard Solution of Silver Nitrate

Dissolve slightly more than theoretical weigh of $AgNO_3$ (equivalent weight, 169.87) in halogen-free water, and dilute to volume.

Standardisation by Mohr Method:

Potassium chloride: Recrystallize KCl 3 times from water, dry at 110 °C, and than heat at 500 °C to constant weight. Equivalent weight KCl = 74.555.

Potassium chromate: 5 % solution of K_2CrO_4 in water.

Accurately weigh enough KCl to yield titration of 40 ml (0.3 g for 0.1 N solution) and transfer to 250 ml Erlenmeyer with 40 ml water. Add 1 ml K_2CrO_4 solution and titrate with $AgNO_3$ until first perceptible pale-red-brown appears. From titration volume subtract ml of the $AgNO_3$ solution required to produce end point color in 75 ml water containing 1 ml K_2CrO_4 solution. From the volume of $AgNO_3$ calculate normality:

Normality = g KCl * 1000 / ml $AgNO_3$ * 74.555

5.8. Standard Solution of Iodine

Dissolve weighed amounts of I (127.1 g/L) and KI, in proportion of 20 g KI to 13 g I in 50 ml water, when I dissolves, transfer solution to volumetric flask. Dilute to volume with water and mix thoroughly. Store in dark brown bottle in dark place.

5.9. Standard Solution of Alcoholic Potassium Hydroxide

Preparation of 0.5 N KOH: Dissolve 28.055 g KOH in 1 liter of alcohol.

5.10. Standard Solution of Sodium Carbonate

Preparation of 0.5 M $NaCO_3$: Dissolve 5.3 g $NaCO_3$ in water and dilute to 100 ml.

5.11. Standard Solution of Boric Acid

Preparation of 0.2 M H_3BO_3: Dry H_3BO_3 to constant weight in desicator over $CaCl_2$. Dissolve 21.405 g H_3BO_3 in water and dilute to 1 liter.

5.12. Standard Solutions of Fehling's

I: Dissolve 69.278 g copper sulphate $CuSO_4.5H_2O$ in water make up to 1 liter.

II: Dissolve 100 g NaOH and 346 g sodium potassium tartrate in water and make up to 1 liter.

5.13. Standard Starch Solution

Add desired powdered starch into volumetric flask. Add some water. Place the volumetric flask into water bath at 80 °C and swirl frequently until starch dissolved. Cool the flask to 20 °C and make up to desired volume.