1. **PURPOSE:**

To lay down a procedure for preparation and standardization of Normal solutions.

1. **SCOPE:**

This procedure is applicable for preparation and standardization of Normal solutions in quality control department at Discovery.

1. **RESPONSIBILITY:**
   1. Analyst-QC is responsible to follow this SOP.
   2. Head-QC/Designee is responsible for ensuring implementation of this SOP.
   3. Head-QA/Designee is responsible for monitoring overall compliance of this SOP.
2. **DEFINITIONS:**
   1. **Normality (N):** A reference solution in which the concentration is stated with regard to the number of gram equivalent weights present per liter of solution.
   2. **Molarity (M):** A unit of concentration expressing the number of moles of solute dissolved per liter of solvent. M = N/2
   3. **Primary solution:** Primary standard solution can be prepared by weighing directly a sufficient pure compound and dissolved to a measured volumeto the reagent.
   4. **Secondary solution:** Secondary standard solution is a solution whose concentration is determined by reference to a primary standard.
3. **PROCEDURE:**
   1. **General guidelines**
      1. Volumetric solutions are divided into primary solutions and secondary solutions.
      2. Prepare volumetric solutions according to the individual procedures as given in this document.
      3. Paste the “Normal solution label” (QC049-FM093) on the bottle and store at appropriate place.
      4. Standardize these solutions after preparation on the predetermined schedule whenever required in duplicate. Record the value as an average of 2 observations, which are very close values.
      5. The average value shall be reported by rounding up to 4 digits after the decimal.
      6. Record the calculations, weights and normalities in respective preparation and standardization records.
      7. Before using any volumetric solution, check visually that the contents of the bottle are clear and if contamination or abnormal change is observed, fresh solution are to be prepared.
      8. Volumetric solutions should not differ from the prescribed strength by more than the 10%. Where the strength falls above 10% the solution should be replaced.
      9. During the standardisation, the difference between two trails of standardisation should not differ by 0.5% for volumetric solution having strengths more than 0.5 Normality or Morality.
      10. During the standardization the difference between two trails of standardisation should not differ by 1.0 % for volumetric solution having strengths less than or equal to 0.5 Normality or Morality.
      11. All reagent bottles / chemicals should be properly labeled and stored in identified areas or shelves.
      12. Labels should indicate the date on which the reagents were opened/prepared and standardized.
      13. Spoiled and disfigured labels should be replaced.
      14. All volumetric solutions shall be used before 30 days from the date of preparation.
      15. All volumetric solutions shall be re standardized once in a week.
      16. All the reagents shall be dried at 110°C for 2 hours before performing the standardization.
      17. **Numbering of volumetric solution:**

**Batch Number : X/Z/MM/YY/NN/A**

X indicates Volumetric solution name

Z indicates Normality of solution.

MM indicates Month (01 for January, 02 for February and so on)

YY indicates last two digits of the year.

NNN indicates Sequential serial number starting from ‘001’

A indicates Sequential serial number starting from ‘1’.

**HCL/1.0036N/04/16/001/1**

**HCL/1.0019N/04/16/001/2……4**

* 1. **Preparation and standardization of 0.1N ammonium thiocyanate:** 
     1. **Preparation:** 
        1. Take a 1000 mL cleaned and dried volumetric flask containing about 500 mL of water.
        2. Add slowly 8.0gr Ammonium thiocyanate into above flask and shake well.
        3. Then make up to 1000 mL with water.
        4. Transfer the solution into amber colored bottle and then paste the label with details.
        5. Re-standardization should perform for every Week.
        6. Solution should be used with in 1 month from date of preparation.
     2. **Standardisation:**
        1. Weigh accurately about 0.5 of Silver nitrate in to 250 ml conical flask
        2. add 50 mL of water, 2 ml freshly prepared ferric sulphate solution and 2 ml of Nitric Acid, mix the content well
        3. Titrate against with 0.1 N Ammonium thiocyanate solution
        4. Almost colourless to Red – Brown colour is end the point.
        5. Note down the final burette readings. Perform the same analysis in duplicate and report the average result.
     3. **Calculation:**

Normality of 0.1 N Ammonium thiocyanate =

* 1. **Preparation and standardization of** 0.1 N IODINE**:** 
     1. **Preparation:** 
        1. Take a 1000 mL cleaned and dried volumetric flask containing about 500 mL of water.
        2. Add slowly Add 14 gms of Iodine and 36 gr of potassium iodine into above flask
        3. Then make up to 1000 mL with water.
        4. Transfer the solution into amber colored bottle and then paste the label with details.
        5. Re-standardization should perform for every Week.
        6. Solution should be used with in 1 month from date of preparation.
     2. **Standardisation:**
        1. Take 25 ml of 0.1N Iodine Solution into 250 mL stopper conical flask.
        2. Add 100 mL of water, 1 ml of 1N HCL mix well.
        3. Titrate against with 0.1 N Sodium Thio-Sulphate until solution has a pale yellow color and Add 2 ml of starch and continue the titration until the solution is colorless
        4. Note down the final burette readings. Perform the same analysis in duplicate and report the average result.
     3. **Calculation:**
  2. **PREPARATION AND STANDARDIZATION OF 0.1 N PERCHLORIC ACID:** 
     1. **Preparation:** 
        1. Take a 1000 mL cleaned and dried volumetric flask containing about 500 mL of Anhydrous glacial acetic acid.
        2. Add slowly 8.5 ml of Perchloric acid (70%) of Perchloric acid and 21 ml of acetic anhydride shake well and cool the room temperature.
        3. Then make up to 1000 mL with glacial acetic acid.
        4. Allow the prepared solution to stand for one day for excess acetic anhydride to be combined and carried out the determination of water.
        5. If the water content exceeds 0.05%, add 5-10 ml acetic anhydride.
        6. If the solution contains no titrable water add sufficient water to obtain content water between 0.02% to 0.05% of water.
        7. Transfer the solution into amber colour bottles and paste the label with details.
        8. Re Standardization should be performed for every week.
        9. Solution should be used with in 1 month from date of preparation
     2. **Standardisation:**
        1. Take 0.50gm of Potassium hydrogen phthalate (previously dried at 1200C for two hrs) into a 250 mL conical flask
        2. Add 50 mL of glacial acetic acid in flask mix the contents well.
        3. Add 0.1 ml of crystal violet solution
        4. Titrate against with 0.1N Perchloric acid.
        5. Violet colour to emerald –green is the end point.
        6. Note down the final burette readings. Perform the same analysis in duplicate and report the average result.
     3. **Calculation:**

Normality of 0.1 N Perchloric acid =

* 1. **Preparation and standardization of :** 
     1. **Preparation:** 
        1. Take a 1000 mL cleaned and dried volumetric flask containing about 500 mL of water.
     2. **Standardisation:**
        1. Weigh accurately about 0.5 of Silver nitrate in to 250 ml conical flask
     3. **Calculation:**
  2. **Preparation and Standardization of 1.0 N NaOH Solution:** 
     1. **Preparation:**

Weigh accurately 42 gm of sodium hydroxide pellets (AR grade) and transfer quantitatively in to a 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q/ HPLC grade water. After complete dissolution of the pellets in water, make up to the mark with distilled/Milli-Q /HPLC grade water.

* + 1. **Standardization:**

Weigh accurately 3.0 gm of previously dried potassium hydrogen phthalate (LR/AR grade) in 250 ml conical flask. Then add 50 ml of water to dissolve. Add 3 drops of phenolphthalein indicator and titrate against 1.0 N NaOH to pink colour end point from colourless. At the end point, note down the titre value. Repeat the titration in the same manner. The standardization details shall be entered in format No. QC049-FM094.

* 1. **Preparation and Standardization of 0.1 N NaOH Solution:** 
     1. **Preparation:**

Weigh accurately 4.2 gm of sodium hydroxide pellets (AR grade) and transfer quantitatively in to a 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q / HPLC grade water. After complete dissolution of the pellets in water and make up to the mark with distilled/Milli-Q / HPLC grade water.

* + 1. **Standardization:**

Weigh accurately 0.3 gm of previously dried potassium hydrogen phthalate (LR/AR grade) in 250 ml conical flask. Then add 50 ml of water to dissolve. Add 3 drops of phenolphthalein indicator and titrate against 0.1 N NaOH to pink colour end point from colourless. At the end point, note down the titre value. Repeat the titration in the same manner. The s standardisation details shall be entered in format No. QC049-FM111.

* 1. **Preparation and Standardization of 1.0 N HCl Solution:** 
     1. **Preparation:**

Take 88 ml of Hydrochloric acid (LR/AR grade) in 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q/HPLC grade water and make up to the mark with distilled/Milli-Q/HPLC grade water.

* + 1. **Standardization:**

Weigh accurately 1 gm of previously dried Sodium carbonate (LR/AR grade) and transfer into 250 ml conical flask containing 50 ml of water. Add 5 drops of Methyl Orange indicator and titrate against 1.0 N HCl to pink colour end point from yellow colour. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM095.

* 1. **Preparation and Standardisation of 0.1 N HCl Solution:**
     1. **Preparation:**

Take 8.8 ml of Hydrochloric acid (LR/AR grade) in 1000 ml volumetric flask containing 500 ml of distilled water and then make up to the mark with distilled water

* + 1. **Standardisation:**

Weigh accurately 0.5 gm of Sodium carbonate (LR/AR grade) and transfer into 250 ml conical flask containing 50 ml of water. Add 5 drops of Methyl Orange indicator and titrate against 0.1 N HCl to pink colour end point from yellow colour. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM096.

* 1. **Preparation and Standardisation of 1.0 N Sulphuric acid Solution:** 
     1. **Preparation:**

Take 28 ml of sulphuric acid (LR/AR grade) in 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q / HPLC grade water and then make up to the mark with distilled /Milli-Q/HPLC grade water.

* + 1. **Standardisation:**

Weigh accurately 1.5 gm of previously dried Sodium carbonate (LR/AR grade) and transfer into 250 ml conical flask containing 50 ml of water. Add 5 drops of Methyl Orange indicator and titrate against 1.0 N Sulphuric acid to pink colour end point from yellow colour. At the end point note, down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM097.

Note: Cool the volumetric flask which contains water before adding Sulphuric acid in preparation procedure.

* 1. **Preparation and Standardisation of 0.1 N Sulphuric acid Solution:** 
     1. **Preparation:**

Take 2.8 ml of Sulphuric acid (LR/AR grade) in 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q / HPLC grade water and then make up to the mark with distilled/Milli-Q / HPLC grade water

* + 1. **Standardisation:**

Weigh accurately 0.15 gm of Sodium carbonate (AR grade) and transfer into 250 ml conical flask containing 50 ml of water. Add 5 drops of Methyl Orange indicator and titrate against 0.1 N Sulphuric acid to pink colour end point from yellow colour. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM098.

**Note:** Cool volumetric flask containing water before adding Sulphuric acid in preparation procedure.

* 1. **Preparation and Standardisation of 0.1 N Ammonium thiocyanate solution:** 
     1. **Preparation:**

Take 7.6 gm of Ammonium thiocyanate (LR/AR grade) in 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q / HPLC grade water. After dissolving in water make up to the mark with distilled/Milli-Q / HPLC grade water to produce 1000 ml. Store the solution for 24 Hrs and then standardize it.

* + 1. **Standardisation:**

Take 25 ml of 0.1 N Silver Nitrate solution in to a 250 ml conical flask. Add 5 ml of concentrated Nitric acid and FAS indicator (Ferric ammonium sulphate solution (10 % of w/v 3ml)). Titrate with Ammonium thiocyanate solution to reddish brown colour end point. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM105.

* 1. **Preparation and Standardisation of 0.1 N Silver Nitrate Solution:** 
     1. **Preparation:**

Weigh accurately 17.5 gm of Silver Nitrate (LR/AR grade) and transfer in 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q / HPLC grade water and make up to the mark with distilled/Milli-Q / HPLC grade water. Store the solution for 24 Hrs and then standardize it.

* + 1. **Standardisation:**

Weigh accurately about 0.1 gm of Sodium Chloride (LR/AR grade) and transfer into 250 ml conical flask, add 5 ml of water and dissolve it. Then add 5 ml of Acetic acid and 50 ml of methanol and 0.15 ml Eosin solution and titrate with silver nitrate solution. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM099.

* 1. **Preparation and Standardisation of 0.1 N Sodium thiosulphate Solution:** 
     1. **Preparation:**

Weigh and transfer 26.0 gm of Sodium thiosulphate (LR/AR grade) and 0.2 gm of Sodium Carbonate into 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q/ HPLC grade water and makeup to the mark with dist/Milli-Q / HPLC grade water. Store the solution for 24 Hrs and then standardize it.

* + 1. **Standardisation:**

Weigh and transfer 0.2 gm of previously dried Potassium Iodate into 250 ml volumetric flask and make up to the mark with water. Pipette out 50 ml of this solution into 250 conical flask. Add 2 gm of potassium Iodide (KI) and add 3 ml of 2 M HCl. Titrate with the Sodium thiosulphate solution. Using starch solution added towards end of the titration until the blue colour is discharged. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM100.

* 1. **Preparation and Standardisation of 0.1 N Perchloric acid Solution:** 
     1. **Preparation:**

Take accurately 8.6 ml of Perchloric acid (LR/AR grade) in 1000 ml volumetric flask containing 500 ml of Glacial Acetic Acid (LR/AR grade). Add 20 ml of Acetic Anhydride (LR/AR Grade) and make up to the mark with Acetic acid. Store the solution for 24 Hrs.Check the water content of the solution. It should be in between 0.02% to 0.05%. If water content exceeds 0.05%, there is more acetic anhydride. If the solution contains no titratable water, add sufficient water to obtain water content between 0.02% and 0.05%. Allow to stand for 1 day, and again titrate the water content. The solution so obtained contains water content between 0.02% and 0.05%, indicating freedom from acetic anhydride. Standardize the solution as follows.

* + 1. **Standardisation:**

Weigh accurately 0.3 gm of previously dried potassium hydrogen phthalate (LR/AR grade) and transfer into 250 ml conical flask containing 50 ml glacial acetic acid. Add 5 drops of crystal violet indicator and titrate against 0.1 N Perchloric acid to emerald green colour end point from blue colour. At the end point, note down the titre value.

Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM104.

Note:

Perchloric acid and acetic acid added slowly individually at 20°C only.

* 1. **Preparation and Standardisation of 0.1 M Edetate Disodium (EDTA) Solution:** 
     1. **Preparation:**

Weigh and transfer 37.2 gm of Edetate Disodium (EDTA Disodium salt) (LR/AR grade) into 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q / HPLC grade water and makeup to the mark with distilled/Milli-Q / HPLC grade water.

* + 1. **Standardisation:**

Weigh accurately 0.2 gm of Magnesium sulphate heptahydrate (LR/AR grade) and transfer into 250 ml conical flask containing 50 ml water. Add 5 ml of ammonia-ammonium chloride buffer and 2 ml eriochrome black-T indicator and titrate against 0.1 M Edetate disodium solution to blue colour end point. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM101.

**Preparation of ammonia-ammonium chloride buffer:**

Dissolve 67.5 gm of ammonium chloride in 760 ml of ammonium hydroxide (Liq. ammonia) and dilute to 1000 ml with water.

* 1. **Preparation and Standardisation of 0.05 M Edetate Disodium Solution:** 
     1. **Preparation:**

Weigh and transfer 18.6 gm of Edetate Disodium (LR/AR grade) into 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q / HPLC grade water and makeup to the mark with distilled/Milli-Q / HPLC grade water.

* + 1. **Standardisation:**

Weigh accurately 0.1 gm of Magnesium sulphate heptahydrate (LR/AR grade) and transfer into 250 ml conical flask containing 50 ml water. Add 5 ml of ammonia-ammonium chloride buffer and 2 ml eriochrome black-T indicator and titrate against 0.05 M Edetate disodium solution to blue colour end point. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM102.

**Preparation of ammonia-ammonium chloride buffer:**

Dissolve 67.5 gm of ammonium chloride in 760 ml of ammonium hydroxide (Liq. ammonia) and dilute to 1000 ml with distilled/Milli-Q / HPLC grade water.

* 1. **Preparation and Standardisation of 0.01 M Edetate Disodium Solution:** 
     1. **Preparation:**

Weigh and transfer 3.8 gm of Edetate Disodium (LR/AR grade) into 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q / HPLC grade water and makeup to the mark with distilled/Milli-Q / HPLC grade water.

* + 1. **Standardisation:**

Weigh accurately 0.05 gm of Magnesium sulphate heptahydrate (LR/AR grade) and transfer into 250 ml conical flask containing 50 ml water. Add 5 ml of ammonia-ammonium chloride buffer and 2 ml eriochrome black-T indicator and titrate against 0.01 M Edetate disodium solution to blue colour end point. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM103.

**Preparation of ammonia-ammonium chloride buffer:**

Dissolve 67.5 gm of ammonium chloride in 760 ml of ammonium hydroxide (Liq. ammonia) and dilute to 1000 ml with distilled/Milli-Q / HPLC grade water.

* 1. **Preparation and Standardisation of 0.1 N Iodine Solution:** 
     1. **Preparation:**

Dissolve 14 gm of Iodine in a solution of 36 gm of potassium iodide in 100 ml of water and dilute to 1000 ml with distilled/Milli-Q / HPLC grade water.

* + 1. **Standardisation:**

Take 25 ml of 0.1 N Iodine solution in to a 250 ml conical flask. Add 1 ml of 1 M Hydrochloric acid and swirl gently to mix the contents. Titrate against 0.1 N sodium thiosulfate solution until the solution has a pale yellow colour. Add 2 ml of starch solution and continue titrating until the solution is colourless. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM106.

* 1. **Preparation and Standardisation of 0.1 N Potassium dichromate Solution:** 
     1. **Preparation:**

Dissolve 5 gm of Potassium dichromate in 1000 ml of distilled/Milli-Q / HPLC grade water.

* + 1. **Standardisation:**

Take 25 ml of above prepared solution in to a 250 ml conical flask. Add 2 gm of potassium iodide, dilute with 25 ml of water and 5 ml of Hydrochloric acid, allow to stand for 10 minutes in a dark place. Titrate against 0.1 N sodium thiosulfate solution until the solution has a pale yellow colour. Add 2 ml of starch solution and continue titrating until the solution is colourless. At the end point note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM107.

* 1. **Preparation and Standardisation of 0.1 N Potassium permanganate Solution:** 
     1. **Preparation:**

Dissolve 3.3 gm of Potassium permanganate in 1000 ml of distilled/Milli-Q / HPLC grade water. Boil the solution for 15 minutes and allow to stand for two days and filter through glass wool.

* + 1. **Standardisation:**

Take 25 ml of above prepared solution in to a 250 ml conical flask. Add 2 gm of potassium iodide, dilute with 25 ml of water and 5ml of Hydrochloric acid, allow to stand for 10 minutes in a dark place. Titrate against 0.1 N sodium thiosulfate solution until the solution has a pale yellow colour. Add 2 ml of starch solution and continue titrating until the solution is colourless. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM108.

* 1. **Preparation and Standardisation of 0.1 N KCl solution:** 
     1. **Preparation:**

Take 7.5 gm of Potassium chloride (LR/AR grade) in 1000 ml volumetric flask containing 500 ml of distilled / Milli-Q/HPLC grade water. After dissolving in water, make up to the mark with distilled / Milli-Q/ HPLC grade water to produce 1000 ml.

* + 1. **Standardisation:**

Take 25 ml of 0.1 N Silver Nitrate solution in to a 250 ml conical flask. Add 5 ml of concentrated Nitric acid and Ferric ammonium sulphate solution (10 % of w/v 3ml). Titrate with Potassium chloride solution to reddish brown colour end point. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM109.

* 1. **Preparation and Standardisation of 1.0 N KOH Solution:** 
     1. **Preparation:**

Weigh accurately 68 gm of potassium hydroxide pellets (85%) (AR grade) and transfer quantitatively in to a 1000 ml volumetric flask containing 500 ml of distilled/Milli-Q / HPLC grade water. After complete dissolution of the pellets in water and make up to the mark with distilled/Milli-Q / HPLC grade water.

* + 1. **Standardisation:**

Weigh accurately 3.0 gm of previously dried potassium hydrogen phthalate (LR/AR grade) in 250 ml conical flask. Then add 50 ml of water to dissolve. Add 3 drops of phenolphthalein indicator and titrate against 1.0 N KOH to pink colour end point from colourless. At the end point, note down the titre value. Repeat the titration in the same manner. The standardisation details shall be entered in format No. QC049-FM110.

1. **FORMATS / ANNEXURE(S):**
   1. Volumetric solution label - QC049-FM093
   2. Preparation and Standardisation of 1.0 N NaOH Solution - QC049-FM094
   3. Preparation and Standardisation of 1.0 N HCl Solution **-** QC049-FM095
   4. Preparation and Standardisation of 0.1 N HCl Solution **-** QC049-FM096
   5. Preparation and Standardisation of 1.0 N Sulphuric acid Solution - QC049-FM097
   6. Preparation and Standardisation of 0.1 N Sulphuric acid Solution - QC049-FM098
   7. Preparation and Standardisation of 0.1 N Silver Nitrate Solution - QC049-FM099
   8. Preparation and Standardisation of 0.1 N Sodium thiosulphate Solution - QC049-FM100
   9. Preparation and Standardisation of 0.1 M Edetate Disodium Solution - QC049-FM101
   10. Preparation and Standardisation of 0.05 M Edetate Disodium Solution - QC049-FM102
   11. Preparation and Standardisation of 0.01 M Edetate Disodium Solution - QC049-FM103
   12. Preparation and Standardisation of 0.1 N Perchloric acid Solution - QC049-FM104
   13. Preparation and Standardisation of 0.1 N Ammonium thiocyanate solution - QC049-FM105
   14. Preparation and Standardisation of 0.1 N Iodine Solution - QC049-FM106
   15. Preparation and Standardisation of 0.1 N Potassium dichromate Solution - QC049-FM107
   16. Preparation and Standardisation of 0.1 N Potassium permanganate Solution - QC049-FM108
   17. Preparation and Standardisation of 0.1 N KCl solution - QC049-FM109
   18. Preparation and Standardisation of 1.0 N KOH Solution - QC049-FM110
   19. Preparation and Standardisation of 0.1 N NaOH Solution - QC049-FM111
2. **CHANGE HISTORY:**

| **Revision No.** | **Effective Date** | **Details of Revision** | **Ref CCF No.** |
| --- | --- | --- | --- |
| 00 | 01.01.2017 | New SOP prepared. | -- |