

Reagent preparation procedures
Environmental Health quality control
Laboratory unit, Jimma University



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1. ACIDITY

Titration Method

A) Carbon dioxide-free water: - prepare all stock and standard solutions and dilution water for the standardization procedure with distilled or de-ionized water that has been freshly boiled for 15 min and cooled to room temperature. The final pH of the water should be > 6.0 and its Conductivity should be <2 $\mu\text{mhos/cm}$.

B) Potassium hydrogen phthalate solution:- approximately 0.05N: crush 15 to 20g primary Standard $\text{KHC}_8\text{H}_4\text{O}_4$ to about 100 mesh and dry at 120°C for 2h. Cool in a desiccators. Weigh 10.0+ 0.5g (to the nearest mg), transfer to a 1-L volumetric flask, and dilute to 1000 ml.

C). Standard sodium hydroxide titrant, 0.1N:- prepare approximately 0.1N by dissolving 4g NaOH in CO_2 free water and diluting to 1 liter. Standardize by titrating 40.00 mL $\text{KHC}_8\text{H}_4\text{O}_4$ solution using a 25-mL burette. Titrate to the inflection point, which should be close to pH 8.7. Calculate the normality of NaOH:-

$$\text{Normality} = \frac{A \times B}{204.2 \times C}$$

Were:

A: g $\text{KHC}_8\text{H}_4\text{O}_4$, Weighed In to 1-L Flask

B: mL $\text{KHC}_8\text{H}_4\text{O}_4$ solution taken for titration and

C: mL NaOH solution used.

Use the measured normality in further calculations or adjust to 0.1 N; 1mL=5.00mg

D). Standard sodium hydroxide titrant, 0.02N:- Dilute 200mL 0.1N NaOH to 1000mL and store in a polyolefin bottle protected from atmospheric CO_2 by a soda lime tube or tight cap. 1mL = 100mg CaCO_3 .

E). Hydrogen peroxide, H_2O_2 30%.

F). Bromophenol blue indicator solution, pH 3.7 indicator:- Dissolve 100 mg bromophenol blue, sodium salt, in 100ml water.

G). Meta cresol purple indicator solution, pH 8.3:- Dissolve 100mg metacresol purple in 100ml water.

H).Methyl orange indicator solution: - dissolve 500 mg methyl-orange powder in distilled water and dilute to 1L

I). Phenolphthalein indicator solution, alcoholic, PH = 8.3 indicator:- Dissolve 8g phenolphthalein in 500 mL 95 % ethyl or isopropyl alcohol and add 500mL distilled water. If necessary add 0.02 N NaOH drop wise until a faint pink color appears.

J). Sodium thiosulfate, 0.1N:- dissolve 25g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ and dilute to 1000 mL with distilled water.

2. ALKALINITY

Titration Method

A. Standard sulfuric acid or hydrochloric acid, 0.1N: Dilute 2.8mL Conc. H_2SO_4 (Specific gravity 1.834-1.836, 96-99% W/W H_2SO_4) or 8.3mL ConC. Hcl (Specific gravity 1.174-1.189, 36-37% w/w HCl) to 1000mL. Standardize against the standard NaoH Solution prepared for Acidity determination.

B. Standard sulfuric acid or hydrochloric acid, 0.02N: Dilute 200.00mL 0.1000N standard Acid to 1000mL with distilled water.

C. Mixed bromocresol green –Methyl red indicator solution: Dissolve 0.02g methyl red and 0.1g bromocresol green in 100ml 95% ethyl alcohol.

D. Phenolphthalein solution, alcoholic ph 8.3 indicator: same as for acidity

E. Sodium thiosulfate, 0.1N. look on reagent preparation for acidity test

3. TOTAL HARDNESS TEST

EDTA Titrimetric Method

A. Buffer solution: Dissolve 16.9g ammonium chloride (NH_4Cl) in 143 mL of conc. Ammonium hydroxide (NH_4OH). Add 1.25g magnesium salt of EDTA and dilute to 250mL with distilled water. Store in a plastic or borosilicate glass container for no longer than 1 month. Stopper tightly to prevent loss of ammonia or pick up of CO_2 . Dispense by means of a bulb-operated pipette.

B. Complexing agents:

Inhibitor I - Sodium cyanide powder

Inhibitor II- Dissolve 5.0g sodium sulfide anhydride ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$) or 3.7g $\text{Na}_2\text{S}\cdot 5\text{H}_2\text{O}$ in 100 mL distilled water.

Inhibitor III - Dissolve 4.5g hydroxylamine hydrochloride in 100 mL of 95 % ethyl alcohol.

C. Indicators:

Liquid indicator: Dissolve 0.5g Eriochrome Black T in 100g triethanolamine or 2-methoxymethanol. Add 2drops per 50mL solution to be titrated.

Solid indicator: mixture-weigh separately 0.5g Eriochrome black T dye and 100g sodium chloride. Place in a mortar and grind together with a pestle until the dark dye is uniformly distributed through the white salt. Store in a tightly stopper bottle.

D. Standard EDTA titrant, 0.01M (0.02N) :

Weigh 3.723g analytical reagent-grade disodium ethylene diaminetetraacetate dehydrate, dissolve in distilled water and dilute to 1000mL. Standardize against standard calcium solution and store in a polyethylene bottle. Compensate for gradual deterioration by periodic re-standardization and by using a suitable correction factor.

E. Standard Calcium Solution:

Weigh 1.000g anhydrous CaCO_3 powder into a 500-mL Erlenmeyer flask. Place a funnel in the flask neck and add, a little at a time, 1+1 HCl until all the CaCO_3 , has dissolved. Add 200 mL distilled water and boil for a few minutes to expel CO_2 cool, add a few drops of methyl red indicator and adjust to the intermediate orange color by adding 3N NH_4OH or 1+1 HCl as required. Transfer quantitatively and dilute to 1000 mL with distilled water, 1mL = 1.00mg CaCO_3

F. Sodium Hydroxide: NaOH, 0.1N

4. CALCIUM HARDNESS

EDTA Titrimetric Method

A. Standard CaCO_3 Solution: Weigh 1.000 g anhydrous CaCO_3 into 500 mL Erlenmeyer flask. Place a funnel in the flask neck and add a little at a time 1 + 1 HCl until all the CaCO_3 has dissolved. Add 200 ml distilled water and boil for a few minutes to expel CO_2 . Cool and adjust pH to a value of 5.0 using 3N NH_4OH or 1 + 1 HCl as required.

Transfer to a 1 liter volumetric flask and bring up to volume.

- B. Buffer Solution:** Dissolve 16.9 g ammonium chloride (NH_4Cl) in 143 ml concentrated ammonium hydroxide; add 1.170 g of disodium ethylene diamine tetracetate dihydrate and 780 mg of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ and dilute to 250 ml with distilled water. Keep in the hood in a tightly stoppered plastic or resistant glass container.
- C. EDTA Titrant, approx. 0.01 M (1 ml = 1 mg hardness as CaCO_3):** Dissolve 3.723 g disodium ethylene diamine tetraacetate dihydrate (a.k.a. Titra Ver Hardness Reagent or Ethylenediaminetetraacetic Acid, Disodium Salt, Dihydrate) in distilled water and dilute to 1 liter.
- D. EBT indicator:** Dissolve 0.5 g of Eriochrome Black T and 4.5 g hydroxylamine hydrochloride in 5% ethyl alcohol.

E. Sodium Hydroxide, 1N

- F. Indicators:** Murexide ammonium purpurate or calcon (Eriochrome blue black R) solid

Indicator mixture:

Weigh separately: 100g sodium chloride and either 0.2g murexide or 0.2g calcon place the NaCl and the indicator in a mortar and grind and the indicator in a mortar and grind together with a pestle until the dye is uniformly distributed through white salt. Store in a tightly stoppered bottle.

5. CHLORIDE TEST

Argentometric Method

- A. Potassium chromate indicator solution:** Dissolve 50g K_2CrO_4 in a little distilled water. Add AgNO_3 solution until a definite a red precipitate is formed. Let stand 12h filter and dilute to 1L with distilled water.
- B. Standard silver nitrate titrant, 0.0141N:** Dissolve 2.395g AgNO_3 in distilled water and dilute to 1000mL. Standardize against 0.0141N NaCl solution.
1.00mL = 500 μgCl^- , store in a brown bottle.
- C. Standard sodium chloride. 0.0141N:** Dissolve 824g NaCl (dried at 140°C) in distilled water and dilute to 1000 mL, 1.00mL=600 $\mu\text{g cl}^-$.
- C. Special reagents for removal of interference:**

- I. Aluminum hydroxide suspension:** dissolve 125g Aluminum potassium sulfate or aluminum ammonium sulfate, in 1L distilled water. Warm to 60°C and add 55mL conc. NH₄OH slowly with stirring. Let stand about 1hr transfer to a large bottle, and wash precipitate by successive additions, with thorough mixing and decanting with distilled water, until free from chloride.
- II. Phenolphthalein in indicator solution.**
- III. Sodium hydroxide 1N.**
- IV. Sulfuric acid, 1N.**
- V. Hydrogen peroxide, 30%.**

6. AMMONIA NITROGEN

Direct Nesslerization Method

- A. Zinc sulfate solution:** Dissolve 100g ZnSO₄·7H₂O and dilute to 1L with water.
- B. Stabilize reagent (Rochelle salt solution):** Dissolve 50g potassium sodium tartarate tetrahydrat in 100 mL distilled water. Remove ammonia usually present in the salt by boiling off 30 mL of solution. After cooling, dilute to 100 mL.
- C. Nessler reagent:** Dissolve 100g HgI₂ and 70g KI in a small quantity of water and add this mixture slowly, with stirring, to a cool solution of 160g NaOH dissolved in 500mL water. Dilute to 1L. Store in rubber stoppered borosilicate glassware and out of sunlight to maintain reagent stability for up a year under normal laboratory conditions. Check reagent to make sure that it yields the characteristic color with 0.1mg NH₃-N/L with in 10min after addition and does not produce a precipitate with small amounts of ammonia with in 2h. (Caution: Toxic care to avoid ingestion).
- D. Stock ammonium solution:** Dissolve 3.819g anhydrous NH₄Cl, dried at 100 °c In water and dilute to 1000mL, 1mL = 1.00mgN = 1.22mg NH₃.
- E. Standard ammonium solution:** Dilute 10mL stock ammonium solution to 100mL with water, 1.00mL = 10.00 µg N= 12.2µg NH₃.
- F. 6N NaOH solution**

7. NITRITE NITROGEN

Diazotization Method

- A. Nitrite-free water:** use nitrite - free water in making all reagents and dilutions
- B. Sulfanilamide reagent:** Dissolve 5g sulfanilamide in a mixture of 50mL conc. HCl and about 300 mL water. Dilute to 500mL with water. The solution is stable for many months.
- C. N-(1-naphthyl-) ethylenediamine dihydrochloride solution:** Dissolve 0.5g NED dihydrochloride in 500mL water. Store in dark bottle. Replace monthly or immediately when it develops a strong brown color.
- D. Hydrochloric acid, Hcl, 1+3:** Use water for dilution.
- E. Stock Nitrite solution:** Dissolve 1.232g NaNO₂ in water and dilute to 1000mL 1.00mL = 250µgN. Preserve with 1mL CHCl₃.
- F. Intermediate nitrite solution:** Dilute 50mL stock nitrite solution to 250mL with distilled water, 1.00mL= 50.0µg N prepare daily.
- G. Standard nitrite solution:** dilute 10,00mL intermediate NO₂ - solution to 1000mL with water 1:00mL =0.500µg N prepare daily.

8. NITRATE NITROGEN

Phenoldisulfonic Acid Method

- A. Standards silver sulfate solution:** Dissolve 4.40g silver sulfate free from nitrate in distilled water and dilute to 1000 mL ,1.00 mL = 1.00 mg /L
- B. Phenoldisulfonic acid reagent:** Dissolve 25g pure white phenol in 150 mL conc. H₂SO₄. Add 75 mL fuming H₂SO₄ (15% free SO₃) stir well and heat for 2 hour on a hot water bath.
- C. Ammonium hydroxide Conc:** if this can not be used, prepare 12N KOH solution by dissolving 673 g KOH in distilled water and diluting to 1 liter
- D. EDTA reagent:** Rub 50 gm disodium ethylendeiamine tetracetate dehydrate with 20mL distilled water to form a thoroughly weight paste add 60 ml concentrated ammonium hydroxide(NH₄OH) and mix well to dissolve the paste.
- E. Stock nitrate solution:** dissolve 9.7218g anhydrous potassium nitrate and dilute

to 1000 mL with distilled water 1 mL = 100 µg N.

F. Standard Nitrate solution: Evaporate 50.0 mL stock nitrate solution to dryness on a steam or water bath dissolve the residue by rubbing with 2.0 mL phenoldisulfonic acid reagent, and dilute to 500 mL with distilled water,

1.00 mL = 10.0 µg N = 44.3 µg NO₃

G. Reagent for treatment of unusual interference:

a. Aluminum hydroxide suspension-prepares as in for chloride determination but wash K₂CO₃ free of ammonia, chloride nitrite, and nitrate

b. Sulfuric acid 1N dilutes cautiously 28 mL conc. H₂SO₄ to 1L with distilled water.

c. Potassium permanganate 0.1N: dissolve 0.316g KMNO₄ in distilled water and dilute to 100 mL.

d. Dilute hydrogen peroxide solution: dilute 10 mL of 30% hydrogen peroxide to 100 mL with distilled water.

e. Sodium hydroxide 1N: dissolve 40g NaOH and dilute to 1 Liter with distilled water.

9. FLUORIDE

Alizarin Photometric Method

A. Stock fluoride solution: Dissolve 0.2210 g anhydrous sodium fluoride, NaF, in distilled water and dilute to 1000mL, 1.00ml = 100µgF⁻.

B. Standard fluoride solution: Dilute 100 mL stock fluoride solution to 1000 mL with distilled water, 1.00ml = 10.0. µgF⁻.

C. Alizarin red solution: Dissolve 0.75 g alizarin reds in distilled water and dilute to 1 liter. If insoluble material is present, filter protect from direct sun light.

D. Zirconyl acid reagent: Dissolve 133mg zirconyl chloride octahydrate, ZrOCl₂·8H₂O, in about 25 ml distilled water. Add 350 ml conc H₂SO₄ slowly, and dilute to 500 ml with distilled water. Cool to room temperature and after one hour the reagent is ready to use.

E. Sodium arsenite solution: Dissolve 5.0 g NaAsO₂ and dilute to 1L with distilled water. (Caution: Toxic – avoid ingestion).

10. PHOSPHATE

Stannous Chloride Method

- A. **Phenolphthalein indicator** aqueous solution
- B. **Strong-acid solution:** slowly add 300 mL – ConC. H_2SO_4 to about 600mL distilled water. When cool, add 4.0 mL conc HNO_3 and dilute to 1L.
- C. **Ammonium molybdate reagent:** Dissolve 25g $(NH_4)_6 MO_7O_{24} \cdot (4H_2O)$ in 1750 mL distilled water. Cautiously add 280 mL ConC. H_2SO_4 to 400 ml distilled water. Cool, and molybdate solution, and dilute to 11.
- D. **Stannous chloride reagent:** Dissolve 2.5g fresh $SnCl_2 \cdot 2H_2O$ in 100 mL glycerol. Heat in a water bath and stir with a glass rod to hasten dissolution. This reagent is stable and requires neither preservatives nor special storage.
- E. **Stock phosphate solution:** dissolve in distilled water 0.7165g anhydrous $KH_2 PO_4$ and dilute to 1000 mL; 1.00mL = 500 ug PO_4^{3-}
- F. **Standard phosphate solution** – Dilute 10mL stock to 11 with distilled water, 1mL = 5.0ug PO_4^{3-} .

11. IRON

Phenanthroline Method

- A. **Concentrated hydrochloric acid:** The acid must contain less than 0.00005% iron.
- B. **Ammonium acetate buffer solution:** Dissolve 250g ammonium acetate $[NH_4CH_3COO]$ in 150mL deionized water. Add 700mL glacial acetic acid and bring up to 1 L with de ionized water.
- C. **Hydroxylamine solution:** Dissolve 10g $NH_2OH \cdot HCl$ in 100mL of de ionized water.
- D. **0.1 N Potassium Permanganate solution:** Dissolve 3.2g of potassium permanganate $[KMnO_4]$ in 1L of deionized water.
- E. **Phenanthroline solution:** Dissolve 1.0g of 1,10-phenanthroline monohydrate $[C_{12}H_8N_2(H_2O)]$ in 1L of de ionized water that contains 20 drops of concentrated hydrochloric acid by stirring and heating to 80°C.

Note: The stoichiometric ratio for the complex is 3 moles of phenanthroline per mole of Fe^{2+} , or 10.65g phenanthroline per g of Fe^{2+} . This implies that 1mL (1 mg phenanthroline) of this reagent is sufficient for about 100 μg of Fe. However, in order to get rapid, complete complexation, excess phenanthroline is required and in practice 1mL of this solution suffices for no more than 2.5 μg of Fe^{2+} .

F. Stock Iron Solution: Slowly add 20mL of concentrated sulfuric acid to 50mL of deionized water and dissolve 1.404g of ferrous ammonium sulfate $[\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2(\text{H}_2\text{O})_6]$ in the acid/water mixture. Add 0.1 N potassium permanganate drop wise until a faint pink color persists. Dilute to 1L with de ionized water. Note, 1.00mL=200 μg Fe.

G. Standard Iron Solution: Prepare this solution daily. Using a volumetric pipette, pipette 50mL of the stock iron solution into a 1L volumetric flask and dilute to 1L with deionized water. Note, 1.00mL=10 μg Fe.

12. MANGANESE

Persulfate Method

A. Special reagent: Dissolve 75g HgSO_4 in 400 mL Conc HNO_3 and 200 mL distilled water. Add 200mL 85% phosphoric acid, and 35 mg silver nitrate. Dilute the cooled solution to 1L.

B. Ammonium Per sulfate, $(\text{NH}_4)_2\text{S}_2\text{O}_8$, solid.

C. Stock manganese solution: Weigh 0.308g dry manganese sulphatemonohydrate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$). Carefully transfer the weighed comical to a 250 mL beaker and dissolve in 100mL distilled water. Cautiously add 1.0 mL concentrated sulfuric acid with constant mixing. Transfer the acid solution to a 1L volumetric flask & dilute to volume. Stopper & mix thoroughly. 1 mL = 100 μg Mn.

D. Standard manganese solution: with a volumetric pipet, measure 10 mL stock manganese solution into a 100 mL volumetric flask. Dilute to the mL mark with distilled water. Stopper and mix thoroughly. Prepare on the day of use 1mL = 10 μg Mn.

E. Hydrogen Peroxide H_2O_2 , 30%.

13. BIOLOGICAL OXYGEN DEMAND (BOD)

The Azide Modification of the Winkler Method

A. Manganese sulfate: Dissolve 480 gm $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ 400 gm $\text{MnSO}_4 \cdot 2\text{H}_2\text{O}$ or

364g $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ in distilled water filter and dilute to 1L. MnSO_4 solution should not give a color with starch when added to an acidified KI solution.

- B. Alkali-iodide-azide:** Dissolve 500g NaOH (or 700g KOH) and 235 g NaI (or 100g KI) in distilled water and dilute to 1L Add 10g sodium azide dissolved in 40 mL distilled water potassium and sodium salts may be used interchangeably. This reagent should not give a colour with starch solution when diluted and acidified.
- C. Sulfuric acid: H_2SO_4 conc.:** One millimeter is equivalent to about 3ml alkali Iodideazide reagent.
- D. Starch Indicator:** Dissolve 2g of starch and 0.2g salicylic acid in 100mL of hot deionized water.
- E. Sodium thiosulfate, 0.025(m) N:** Dissolve 6.205g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ in deionized water, add 0.4g NaOH and dilute to 1L. (Note: Sodium thiosulfate absorbs and loses water readily, so this solution should be standardized against potassium bi-iodate.). Add 1.5 mL 6N NaOH or 0.4g solid NaOH and dilute to 1000 mL. Standardize with bi - iodate.
- F. Standard potassium bi - iodate solution 0.0021 M. 0.025N:** Dissolve 0.8124g $\text{KH}(\text{IO}_3)_2$ in distilled water and dilute to 1000 mL .Standardization=dissolve approximately 2g KI, Free from iodate, in an Erlenmeyer flask with 100 to 150 mL distilled water. Add 1 mL 6N H_2SO_4 or a few drops of conc. H_2SO_4 and 20 mL of standard bi - iodate solution. dilute to 100 mL and titrate the liberated iodate with thiosulfate titrant adding starch toward end of titration, when a pale straw color is reached adjust the $\text{Na}_2\text{S}_2\text{O}_3$ solution to 0.025 N if necessary.
- G. Potassium fluoride solution:** dissolve 40g $\text{KF} \cdot 2\text{H}_2\text{O}$ in distilled water and dilute to 100 mL.
- H. Phosphate buffer solution:** Dissolve 8.5g KH_2PO_4 , 21.75 g K_2HPO_4 , 33.4g $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$, and 1.7g NH_4Cl in about 500 mL distilled water and dilute to 1L. The PH should be 7.2 without further adjustment. Discard reagent (or any of the following reagent) if there is any sign of biological growth in the stock bottle.
- I. Magnesium sulfate solution:** Dissolve 22.5g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ in distilled water and dilute to 1L.
- J. Calcium chloride solution:** dissolve 27.5g CaCl_2 in distilled water and dilute to 1L.
- I. Ferric Chloride solution:** dissolve 0.25g $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ in distilled water and dilute to 1L

M. Acid and alkali solutions, IN: for neutralization of caustic or acidic waste sample.

N. Sodium sulfite solution 0.025N: Dissolve 1.575g Na₂SO₃ in 1000 mL distilled water.

This solution is not stable; prepare on the day of use.

O. Nitrification inhibitor: 2-chloro-6- (trichloro methyl) pyridine.

P. Glucose-glutamic acid solution: Dry reagent-grade glucose and reagent-grade glutamic acid at 103OC for 1h. Add 0.150 g glucose and 0.150g glutamic acid to distilled water, dissolve and dilute to 1 liter. Prepare fresh immediately before use.

14. CHEMICAL OXYGEN DEMAND (COD)

Open Reflux Method, Titrimetric Method

A. Standard potassium dichromate solution, 0.0417 M (0.25N): dissolve 12.259g K₂Cr₂O₇ primary standard grade previously dried at 103OC for 2h in distilled water and dilute 100mL

B. Sulfuric acid reagent: Add Ag₂SO₄ at the rate of 5.5g Ag SO₄ per Kg H₂SO₄ Let stand 1 to 2 days to dissolve Ag₂SO₄ per kg H₂SO₄ let 1 to 2 days to dissolve Ag₂SO₄.

C. Ferroin indicator solution: dissolve 1.485g 1,10-phenanthrolina monohydrate and 0.695g FeSO₄. 7H₂O in distilled water and dilute to 100mL.

D. Standard ferrous ammonium sulfate (FAS) titrant approximately 0.25M (0.25N):
Dissolve 98g Fe (NH₄)₂ (SO₄)₂ 6H₂O in distilled water. Add 20 mL Conc. H₂SO₄ Cool, and dilute to 1000 mL. Standardize daily against K₂Cr₂O₇ solution as follows:- Dilute 10.0 mL standard K₂Cr₂O₇ to about 100 mL. Add 30 mL Concentrated H₂SO₄ and cool. Titrate with FAS titrant using 0.10 to 0.15 mL (2 to 3 drops) Ferroin indicatory

E. Mercuric sulfate, crystals or powder. (extra pure)

F. Sulfamic acid

G. Potassium hydrogen phthalate (KHP) standard: Lightly crush and then dry KHP to constant weight at 120OC. Dissolve 0.425 g in distilled water and dilute to 1000 mL. KHP has a theoretical COD of 1.176 mg O₂/mg this solution has a theoretical COD of 5000g O₂/mL. This solution is stable when refrigerated for up to 3 months.

15.SULFATE

Gravimetric Method with Ignition of Residual

- A. **Methyl red indicator solution:** dissolve 0.1 g methyl red sodium salt in distilled water and dilute to 100 mL
- B. **Hydrochloric acid HCl 1+1**
- C. **Barium chloride solution:** dissolve 100g $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in 1L distilled water. Filter through a membrane filter or hard-finish filter paper prior use 1 mL is capable of precipitating approximately 40mg SO_4
- D. **Silver nitrate - nitric acid reagent:** dissolve 8.5 g AgNO_3 and 0.5 mL Conc. HNO_3 in 500 mL distilled water.

16.Chromium

Diphenyl Carbazide Method

- A. **Stock chromium solution:** Dissolve 141.1 mg $\text{K}_2\text{Cr}_2\text{O}_7$ in water & dilute to 1L.
1mL = 50.0 $\mu\text{g Cr}$.
- B. **Standard chromium solution:** Dilute 10.0 mL stock chromium solution to 1000 mL
1.00mL = 5.00 $\mu\text{g Cr}$.
- C. **Nitric acid, HNO_3 conc.**
- D. **Sulfuric acid. H_2SO_4 1+1**
- E. **Methyl orange indicator solution**
- F. **Hydrogen peroxide H_2O_2 , 30%.**
- G. **Redistilled water:** - Distilled water re-distilled in all-glass apparatus.
- H. **NH_4OH , Conc.**
- I. **Potassium permanganate solution:** dissolve 4g KMnO_4 in 100 mL water.
- J. **Sodium azide solution:** Dissolve 0.5g NaN_3 in 100 mL water
- K. **Diphenylcarbazide solution:** dissolve 250 mg 1.5 - diphenylcarbazide in 50 mL acetone.
Store in a brown bottle discard when solution becomes discolored.
- l. **Sulfuric acid. H_2SO_4 0.2N:** dilute 17 mL 6N H_2SO_4 to 500 mL with water.
- M. **Chloroform – chemically pure.**
- N. **Cupferron solution:** Dissolve 5g $\text{C}_6\text{H}_5\text{N}(\text{NO})\text{ONH}_4$ in 96 mL water