

SPECIFICATION FOR HYDRATED LIME

Revised on 03.04.2024

SPECIFICATIONS FOR HYDRATED LIME

GENERAL

TABLE OF CONTENTS

		PAGE NUMBER
1	Ambient Conditions	6au - 3
2	Suitability for Potable Water	6au - 3
3	Definitions	6au - 3
4	Inspection and Testing	6au - 3
5	Marking	6au - 4
6	Protection during Delivery	6au - 4
7	Storing handling and hauling of Materials	6au -4
8	Manufacturer's Certificate	6au - 4
9	Quality and workmanship	6au - 5
10	Technical Specification	
	1.0 General	6au - 6
	2.0 Packaging	6au - 7
	3.0 Method Of Test	6au -8
	Appendix A – Compliance of Lot	
	A.1 Lot	6au - 8
	A.2 General Requirements Of Sampling	6au - 8
	A.3 Sampling Instruments	6au - 9
	A.4 Scale Of Sampling	6au - 9
	A.5 Preparation Of Sample	6au - 10
	A.6 Number Of Tests	6au -10

Appendix B - Determination of Particle Size

B.1	Apparatus	6au – 10
B.2	Procedure	6au – 10
B.3	Calculation	6au – 11

Appendix C - Determination of Free Water Content in Hydrated Lime

C.1	Principle	6au – 11
C.2	Apparatus	6au – 11
C.3	Procedure	6au – 12
C.4	Calculation	6au – 13

Appendix D - Determination of Available Calcium Hydroxide Content

D.1	Sugar Method	6au – 13
-----	--------------	----------

Appendix E - Determination of Basicity Factor

E.1	Reagents	6au – 15
E.2	Procedure	6au – 15
E.3	Calculation	6au – 15

Appendix F- Determination of Insoluble Matter Including Silicon Dioxide

F.1	Hydrochloric Acid Method	6au – 16
-----	--------------------------	----------

Appendix G- Determination of Magnesium Oxide

G.1	Reagents	6au – 18
G.2	Preparation of Sample Solution	6au – 19
G.3	Procedure	6au – 20
G.4	Calculation	6au – 20

Appendix H- Determination of Heavy Metals

GENERAL

1 Ambient Conditions

All materials shall be in every respect suitable for storage, use and operation in the conditions of temperature and humidity appertaining in Sri Lanka.

The annual average temperature is 35 °C while the relative humidity varies generally from 70% during the day to 90% at night.

2 Suitability for Potable Water

Hydrated Lime will come into contact with potable water shall not constitute a toxic hazard, shall not support microbial growth and shall be suitable for human consumption.

3 Definitions

The definitions given in the relevant standards which are referred to in the specification shall apply for the terms used in this specification.

4 Inspection and Testing

The Manufacturer shall supply, furnish and prepare the necessary test samples of materials and supply the labour facilities and appliances for such testing as may be required to be carried out on his premises according to this specification. If there are no facilities at his own works for making the prescribed tests the Contractor shall bear the cost of carrying out the tests elsewhere.

The Engineer and nominated Inspection Agency shall have full access to all parts of the plant that are concerned with the testing, furnishing or preparation of materials for the performance and testing of work under this Specification.

The Contractor shall furnish the Engineer with reasonable facilities and space (without charge) for the inspection, testing and obtaining of such information, as he desires regarding the character of material in use and the progress and manner of the work.

Further all materials shall be tested to the appropriate tests at the manufacturer's premises and shall be supported by a test certificate from the manufacturer.

Engineer will appoint an inspection team comprising of one Engineer and one Chemist of NWSDB. The manufacturer should provide Laboratory facilities and other chemicals, supporting staff and perform the test in presence of this inspection team. The certificate of acceptance of goods issued by the inspection team is compulsory to deliver materials from the manufacturer's plant.

Contractor is held responsible for the assuring quality of the goods supplied by him until the final delivery point. Hydrated Lime is tested at the Employer's final delivery point by the Engineer and goods shall conform to the standard for the acceptance.

5 Marking

All markings shall be legible and durable unless otherwise specified and shall be as specified in this specification.

6. Protection During Delivery

The contractor shall provide protection to the approval of the Engineer, prior to the materials leaving the place of manufacture and shall maintain such protection until the items reach their destination in order to guard effectively against damage during transit and storage and the ingress of foreign matter.

All details of the proposed arrangement for such protection shall be submitted at the time of tendering.

The cost of providing protection shall be included in the unit prices tendered in the Bills of Quantities.

7. Storing, Handling and Hauling of Materials.

All materials shall be stored in an approved location and in such a manner as to preserve their quality and condition.

Storage shall be in accordance with the manufacturers recommendation and shall be stored in a dry place with a proper packing.

Materials and components shall be handled in such a manner as to avoid any damage or contamination and in accordance with all applicable recommendations of the manufacturers.

The contractor shall give instructions to the shipper on precautions to be taken in the handling of materials during loading, towage delivery and unloading and shall give particulars of these instructions to the purchaser.

8. Manufacturer's Certificate

The Contractor shall supply to the Engineer a certificate stating that hydrated lime supplied has been subjected to the tests laid down herein and conforms in all respects to this Specification or such other Specification which has been submitted to and approved by the Engineer. Testing at Manufacturers factory by the Engineer will perform as specified.

9. Quality and Workmanship

The Bidder shall provide ISO 9001 : 2015 Quality Management System requirement certificate for Quality Assurance for the goods manufacturing factory from an accredited agency for Hydrated Lime. Accredited Agency shall be a member of International Accredited Forum (IAF) and shall have the authority for the accreditation of mentioned goods in their scope of accreditation. Scope of the production shall be clearly specified in the certificate. Manufacturer shall maintain the validity of this certificate during the contract Period. If the supply is made from several factories, ISO 9001 : 2015 certificates for quality management system requirement shall be submitted for each factory.

TECHNICAL SPECIFICATION FOR HYDRATED LIME

1.0 GENERAL

Hydrated Lime shall be in the form of a fine white powder which is free from lumps and hard caking. It shall be substantially free from foreign mater and Core. Hydrated Lime shall conform to the requirements given in Table 1 in SLS 1577 : 2017 given bellow.

Note : Core is the fraction of limestone which has resisted dissociation in the kiln. It is the results of incomplete calcinations or under burning.

Hydrated lime shall not contain any impurities and additives in such concentrations that are capable of producing deleterious or injurious effects on the health of those consuming water that has been treated with lime.

Hydrated lime shall be of Food Grade quality and supplier shall provide documentary evidence of compliance with the International Standards to this effect.

Hydrated lime shall also conform to the requirements given in Table 1 when test in accordance with the relevant methods given in column 4 of the table.

Table 1 – Requirements for hydrated lime

SI No.(1)	Characteristics (2)	Requirement (3)	Method of test (4)
i)	Sampling		Appendix A
ii)	Particle size distribution		
	a) Particles passing through 600-µm sieve, per cent by mass, min.	99.5	Appendix B
	b) Particles passing through 75-µm sieve, per cent by mass, min.	98.0	
iii)	Free water content, per cent by mass, max.	2.0	Appendix C
iv)	Available Calcium hydroxide content (dry basis), per cent by mass,min.	90.0	Appendix D
v)	*Basicity factor, min.	0.72	Appendix E
vi)	Insoluble matter including Silicon dioxide (dry basis) per cent by mass, max.	10	Appendix F

vii)	Magnesium (as MgO), per cent by mass, max.	1.0	Appendix G
viii)	Manganese (asMn)mg/kg,max.	25	Appendix H
ix)	Lead (asPb) mg/kg,max.	10	Appendix H
x)	Cadmium as (Cd) mg/kg,max.	2.0	Appendix H
xi)	Chromium (as Cr) mg/kg,max.	10	Appendix H
xii)	Arsenic (as As) mg/kg,max.	4	Appendix H
xiii)	Mercury (as Hg) mg/kg,max.	0.2	Appendix H

* The basicity factor of lime or limestone product is a measure of available alkalinity. It represents the grams of Calcium oxide equivalent per gram of lime or limestone product and may be used for comparing the relative neutralizing values.

2.0 PACKAGING

Hydrated Lime shall be packed in gunny bags having two polythene inner liners as per the standard SLS 1577 :2017 and shall be free from dirt, any foreign matters likely cause decomposition of the material.

2.1 Marking.

Each package shall be legibly and indelibly marked with the following information.

- (a) Name of the product
- (b) Name and Address of the manufacturer
- (c) Trade mark, if any
- (d) Net mass of the Contents, in Kilograms :
- (e) The words “keep away from heat and moisture” :
- (f) Date of manufacture & Date of expiry.
- (g) Batch or code Number
- (h) The words “National Water Supply and Drainage Board” or NWSDB”
- (i) Contract No.

3. METHOD OF TEST

The materials shall be tested by the appropriate methods prescribed in Appendix B to H of SLS 1577 : 2017 which are given below. If manufacturer uses different test method, those test methods shall be submitted to verify whether those test methods shall comply with the SL 1577 : 2017.

APPENDIX A

COMPLIANCE OF LOT

A.1 LOT

A.1.1 In any consignment all the packages containing the same quantity of Hydrated Lime of one batch of manufacture or supply shall constitute a lot.

A.2 GENERAL REQUIREMENTS OF SAMPLING

A.2.1 in drawing, handling and preparation of sample the following precautions shall be observed :

- A.2.1.1** Sampling and preparation of sample shall be conducted as expeditiously as possible in order to avoid undue exposure of the material to the air.
- A.2.1.2** Samples shall not be taken from broken packages.
- A.2.1.3** Sampling instrument shall be clean and dry when used.
- A.2.1.4** Samples shall be placed in clean, dry and air-tight glass or other suitable containers.
- A.2.1.5** The material being sampled, the sampling instruments and the containers for samples shall be protected from adventitious contamination.
- A.2.1.6** The sample containers shall be sealed air-tight after filling and marked with necessary details of sampling.
- A.2.1.7** Samples shall be stored in the shade.

A.3 SAMPLING INSTRUMENTS

A.3.1 A sampling tube having a core diameter of not less than 25 mm shall be used.

A.4 SCALE OF SAMPLING

A.4.1 Each lot shall be tested separately for ascertaining its conformity to the requirements of this specification.

A.4.2 The number of packages to be selected from a lot shall be in accordance with Table 2.

Table 2 – Scale of Sampling

Number of Packages in the lot	Number of Packages to be selected
Up to 08	02
09 to 27	03
28 to 64	04
65 to 100	05
101 to 300	06
301 to 500	07
501 to 800	08
801 to 1300	09
1301 to above	10

A.4.3 The packages shall be selected at random. In order to ensure randomness of selection random number tables as given in SLS 428 shall be used.

A.5 PREPARATION OF SAMPLE

- A.5.1** A representative sample of material shall be obtained from each package selected as in A.4.2 in accordance with method given in A.5.2.
- A.5.2** The sampling tube shall be inserted into a package being sampled so that it will take a core material from substantially the entire length of the package.
- A.5.3** The material obtained from each package shall be thoroughly mixed and reduced by coning and quartering to obtain a test sample of not less than 150g.

A.6 NUMBER OF TESTS

- A.6.1** Each package selected as in A.4.2 & A.4.3 shall be examined for packing and marking requirements. This may be done at the place of sampling.
- A.6.2** The sample prepared as in A.5.3 shall be tested for requirements specified in 1.0 of this specification.

APPENDIX B

DETERMINATION OF PARTICLE SIZE

B.1 APPARATUS

- B.1.1** *Test sieves*, with nominal aperture 600- μm and 75- μm conforming to SLS 124.

B.2 PROCEDURE

- B.2.1** Weigh, to the nearest 0.1g, about 100g of the sample of hydrated lime and place on a 600- μm sieve, which is rested above a 75- μm sieve. Wash the material through the sieves by means of a stream of water from a faucet. Use a piece of rubber tubing attached to the water faucet for the washing. The velocity of the water may be increased by pressing the tubing, but shall not cause any splashing of the sample over the sides of the sieve. Continue the washing until the water coming through the sieve is clear, but in no case should the washing be continued for more than 30 minutes. Take care not to let water accumulate on the 75- μm sieve, as the openings will be clogged and the operation cannot be completed in 30 minutes. Dry the residues on both sieves in an atmosphere free from carbon dioxide at a temperature between 100 °C and 120 °C, and weigh. Continue the drying and weighing operation until the

deference between two successive weighings not more than 0.01 g.

B.3 CALCULATION

B.3.1 Calculate the percentage residue retained on each sieves, based on the original mass of the sample. The mass of the material retained on the 600- μm sieve shall be added to the mass of the material on the 75- μm sieve to obtain the mass of the material retained on the 75- μm sieve.

B.3.2 Calculate the percentage of particles passing through on each sieve, based on the residue retained on each sieve.

APPENDIX C

DETERMINATION OF FREE WATER CONTENT IN HYDRATED LIME

C.1 PRINCIPLE

The free water in hydrate lime is moisture attached to the product, which is released from the sample at a temperature of 115 °C to 120 °C. The loss in weight of the sample is equal to the free moisture of the hydrated lime. It distinguishes from the hydroxyl water that is chemically bound to the lime and which cannot be liberated except at higher temperatures.

C.2 APPARATUS

C.2.1 Sample flask E, illustrated in **Figure 1**, consists of a 50 mL flat-bottom, glass-stoppered flask, supplied with a ground glass joint and solid ground glass stopper. The flask shall be fitted with an interchangeable hollow ground-glass stopper, equipped with two glass entry tubes for conducting the dry air over the sample.

C.2.2 Purifying train (See figure 1), located outside the oven F for conducting the dry air over the samples, shall consist of a series of scrubbers and absorption bulbs to remove Carbon dioxide and moisture from the air. The apparatus are arranged in the following order starting from the air source.

C.2.3 Soda-lime tower A, at the air inlet to remove Carbon dioxide from the air.

C.2.4 Bottle B, containing lime water to show when the soda lime is exhausted.

C.2.5 Fleming jar C, containing Sulphuric acid to remove water from the air.

C.2.6 Absorption bulb D, filled with Magnesium perchlorate to complete the drying of the air.

C.2.7 Sample flask E

C.2.8 Drying oven F

C.2.9 Absorption bulb G, also filled with Magnesium perchlorate and located on the exit side of the sample bulb as a protective barrier against atmospheric moisture.

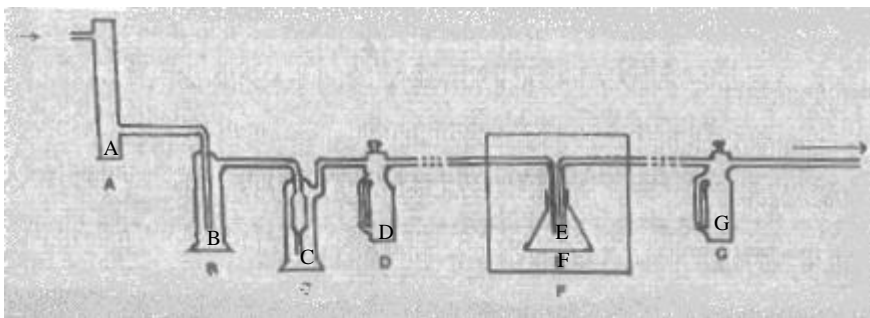


Figure 1 – Apparatus for free moisture in hydrated lime key

A Soda-Lime Tower at inlet to remove Carbon dioxide

B Bottle containing lime water to show when soda lime tower is exhausted

C Fleming jar containing Sulphuric acid to remove water from the air.

D Absorption bulb filled with Magnesium perchlorate to complete the drying of the air.

E 50mL Sample flask

D Drying oven operating at 120 °C

G Absorption bulb filled with Magnesium perchlorate to prevent moisture backup into sample.

C.3 PROCEDURE

Weight 2.5 g to 3 g of the prepared sample, and using glazed paper folded in the shape of a funnel, transfer it rapidly into the previously weighed bottle and immediately restopper it.

Insert the bottle into the 120 °C oven and quickly exchange stoppers. Connect the sample bottle to the purifying train by means of flexible tubing and pass a slow current of dry Carbon dioxide free air through the apparatus for 2 h. Disconnect the sample bottle from the train, remove it from the oven with another quick exchange of stoppers, and place it in a desiccator to cool. When cold, remove it to the balance case for several minutes before weighing it, and just before weighing, lift the stopper slightly for an instant to relieve any vacuum that may exist in the bottle. The loss in mass of the sample represents “free water” loss as 120 °C. Use a bottle similar to the one containing the sample as a counterpoise in all weighings unless a single-pan balance is used.

C.4 CALCULATION

Calculate per cent “free moisture” in the sample as follows:

$$\text{Free moisture content, per cent by mass} = \frac{(A-B)}{C} \times 100$$

Where,

- A** is the mass, in g, of sample flask and sample;
- B** is the mass, in g, of sample flask after drying; and
- A** is the mass, in g, of sample;

APPENDIX D

DETERMINATION OF AVAILABLE CALCIUM HYDROXIDE CONTENT

D.1 SUGAR METHOD

D.1.1 PRINCIPLE

Sucrose is added to accelerate the dissolution of Calcium hydroxide. The resulting alkaline solution is titrated with Hydrochloric acid and the amount of acid consumed is used to calculate the percentage of available Calcium hydroxide.

D.1.2 REAGENTS

D.1.2.1 *Rectified spirit*, conforming to **SLS 351**

D.1.2.2 *Hydrochloric acid*, standard volumetric solution, 0.1 mol/L

D.1.2.3 *Sodium hydroxide*, standard volumetric solution, 0.1 mol/L

D.1.2.4 *Sugar solution*, approximately 15 per cent sucrose (m/V) in Carbon dioxide-free water.

D.1.2.5 *Phenolphthalein indicator solution*, dissolve 0.5 g of phenolphthalein in 100 mL of rectified spirit.

D.1.3 PROCEDURE

D.1.3.1 Weigh to the nearest 0.001 g about 1 g of the finely powdered material into a 500-mL volumetric flask or pyrex glass bottle and wet it with the minimum amount of rectified spirit (**D.1.2.1**). Shake with about 400 mL of sugar solution (**D.1.2.4**) for 3 hours mechanically. Dilute to volume. If use pyrex glass bottle transfer the solution to 500 ml volumetric flask and dilute up to the 500 mL volume. Filter through a coarse, dry filter paper (Whatman No.40 or its equivalent) into a dry flask. Discard the first 25 mL of the filtrate. Pipette out 50 mL from the filtrate into another flask to which 50 mL of Hydrochloric acid (**D.1.2.2**) is added and titrate with Sodium hydroxide solution (**D.1.2.3**) using phenolphthalein (**D.1.2.5**) as indicator.

D.1.3.2 Run a blank using 50 mL of sugar solution and 50 mL of Hydrochloric acid.

D.1.4 CALCULATION

Calcium hydroxide content, per cent by mass =
$$\frac{37.04 (V_1 - V_2) C}{m} \times \frac{100}{(100-M)}$$

(on dry basis)

Where,

V₁ is the volume, in mL, of Sodium hydroxide solution (**D.1.2.3**) used in blank;

V₂ is the volume, in mL, of Sodium hydroxide solution used with the material;

C is the concentration of the Sodium hydroxide solution in mol/L;

m is the mass, in g, of the material taken for the test; and

M is the per cent, by mass of moisture content determined in Appendix C.

APPENDIX E

DETERMINATION OF BASICITY FACTOR

E.1 REAGENTS

E.1.1 *Sulphuric acid*, standard volumetric solution, 0.250 mol/L

E.1.2 *Potassium hydroxide*, standard volumetric solution, 0.500 mol/L

E.2 PROCEDURE

E.2.1 Weigh to the nearest 0.001 g about 1 g of the sample which has been ground to pass a 150- μ m sieve (conforming to **SLS 124**) and transfer to a 500-mL Erlenmeyer flask containing about 20 mL of cold water. Add 100 mL of Sulphuric acid from a pipette (**E.1.1**) and stopper with a two-hole rubber stopper. Place the flask on a hot plate and boil for 15 minutes. (Glass beads may be added to prevent bumping). Remove the flask from the hot plate and cool in water. Add several drops of phenolphthalein indicator solution and titrate the excess acid with potassium hydroxide solution (**E.1.2**).

E.3 CALCULATION

$$\text{Basicity factor} = \frac{(V_1 C_1 - (V_2 C_2 / 2)) \times 0.056}{m}$$

Where,

V_1 is the volume, in mL, of Sulphuric acid solution (**E.1.1**) required for titration of the sample;

V_2 is the volume, in mL, of Potassium hydroxide solution (**E.1.2**) required for titration of the excess:

C_1 is the Concentration, in mol/L, of Sulphuric acid solution;

C_2 is the Concentration, in mol/L, of Potassium hydroxide solution; and

m is the mass, in g, of sample.

APPENDIX F

DETERMINATION OF INSOLUBLE MATTER INCLUDING SILICON DIOXIDE

F.1 HYDROCHLORIC ACID METHOD

F.1.1 Principle

This test method is based on a double evaporation to dryness of the Hydrochloric acid solution of the lime sample to convert silicon dioxide (SiO_2) to the insoluble form. After dissolution in Hydrochloric acid, the silica is dehydrated by a double evaporation to dryness. After each dehydration, the dry salts are redissolved with dilute Hydrochloric acid, the solution is filtered and the siliceous residue and other insoluble matter separated.

F.1.2 Procedure

F.1.2.1 Weigh 0.5 g of hydrated lime (See **Note 1**) and ignite in a covered platinum crucible in an electric muffle (See **Note 2**) at 950 °C for 15 min or longer to effect complete decomposition. Transfer to an evaporating dish, preferably of Platinum (See **Note 3**), containing about 10 mL of water, mix to a thin slurry, add 5 mL to 10 mL of Hydrochloric acid, and digest with the aid of gentle heat and agitation until solution is complete (See **Note 4**).

NOTES

1. Due to the rapidity with which hydrated lime absorb water and carbon dioxide from the air, samples should be protected in tightly stoppered containers at all times. Samples for analysis are to be weighed quickly and the sample container re-stoppered immediately after the sample has been removed.
2. Ignition of the sample in an electric muffle is far superior to flame Ignition. However, if an electric muffle is not available, flame ignition and the blast lamp may be used.
3. If a platinum dish is not available, porcelain may be used. A glass container positively should not be used.
4. Alternatively, the loss on ignition (LOI) can be determined first, using 0.5 g of sample. The insoluble matter including silicon dioxide can then be assayed using the ignited product that remains in the LOI crucible.

F.1.2.2 Evaporate the solution to dryness on a steam bath. When dry or nearly so, cover the dish and place it in an air bath or drying oven or on a metal triangle resting on a hot

plate. Heat for 1 h at 100 °C, remove the dish from the heat, and allow the dish and contents to cool slightly.

F.1.2.3 Drench the cooled mass with 20 mL (1+1) Hydrochloric acid and place on the water bath for 10 min. Filter the mixture containing the insoluble residue through a retentive filter of suitable size. Wash filter thoroughly with warm, diluted (5+95) Hydrochloric acid and then twice hot water. Reserve the paper and residue.

F.1.2.4 Evaporate the filtrate to dryness, dehydrate and extract the residue with hydrochloric acid as before, but this time heat the acidified solution for 1 to 2 min. Filter through a second and smaller piece of retentive filter paper and wash as before. Combine the two wet papers containing the separated residues and transfer to a weighed platinum crucible.

F.1.2.5 Char carefully without allowing the paper to inflame, and then ignite at 1000 °C for 30 min in an electric muffle furnace (See **Note 2** in **F.1.2.1**) . Cool in a desiccator and weigh.

F.1.3 CALCULATON

Insoluble matter including Silicon dioxide, per cent by mass = $\frac{A}{B} \times \frac{100}{(100-M)}$

Where,

A is the mass, in g, of ignited residue;

B is the mass, in g, of original sample; and

M is the per cent, by mass of moisture content determined in Appendix C.

APPENDIX G

DETERMINATION OF MAGNESIUM OXIDE

G.1 REAGENTS

G.1.1 Concentrated Hydrochloric acid

G.1.2 Standard Disodium ethylene diamine tetra-acetate dihydrate (EDTA) solution

Weigh 7.44 g of Disodium ethylene diamine tetra-acetate dihydrate and dissolve in water. Make up the Volume to two litres. Standardize the solution against standard Calcium chloride solution according to the procedure given in **G.1.8**

G.1.3 Standard Calcium chloride solution

Dry about 2 g of Calcium carbonate at 120 °C to constant mass. Dissolve 1.00 g of the dried Calcium carbonate in minimum quantity of concentrated Hydrochloric acid and then make up the volume to one litre. One millilitre of this solution is equivalent to 0.00056 g of Calcium oxide.

G.1.4 Eriochrome black 'T' indicator, (for calcium and magnesium)

Dissolve 0.1 g of the dye in 20 mL of Methanol. This solution shall be prepared freshly every week.

G.1.5 Murexide indicator (for Calcium alone)

Grind 0.2 g of murexide with 10 g of Sodium chloride until the mixture is homogeneous.

G.1.6 Buffer solution

Dissolve 67.5 g of Ammonium chloride in a mixture of 520 mL of Ammonium hydroxide (relative density 0.90) and 250 mL of water. Dissolve a mixture of 0.931 g of Disodium ethylene diamine tetra-acetate dihydrate and 0.616 g of Magnesium sulphate heptahydrate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in 50 mL of water, combine both the solutions and make up to one litre. 5mL of buffer solution added to 50 mL of deionized water should not consume more than one drop of EDTA solution to change to distinct blue with eriochrome black 'T' indicator.

G.1.7 Standard Sodium hydroxide solution, 5 M

G.1.8 Standardization of disodium ethylene diamine tetra-acetate dihydrate (EDTA) Solution

- a) With murexide or calcein indicator

Pipette out 50 mL of standard Calcium chloride solution (see **Note 1**) in to a 250-mL conical flask and add 1 mL of standard Sodium hydroxide solution to adjust the pH of the solution to about 12. Add 0.1 to 0.2 g of murexide indicator (see **Note 2**) and titrate the solution against standard EDTA solution until the colour changes from pink to purple.

- b) With eriochrome black 'T' indicator

Pipette out 50 mL of standard Calcium chloride solution (see **Note 1**) in to a 250-mL conical flask and add 5 mL of buffer solution to adjust the pH of the solution to approximately 10. Add 5 drops of eriochrome black 'T' indicator and titrate against standard EDTA solution until the wine red colour changes to pure blue. (The volume of the solution to be titrated against standard EDTA solution should be approximately 50 mL and the strength of the standard EDTA solution should be adjusted in such a way that almost 50 mL of it is required for the titration.)

NOTES

- 1. The aliquot for titration shall be neutral so that by adding standard Sodium hydroxide solution or buffer solution the desired pH could be adjusted in the solution.*
- 2. If calcein indicator is to be used in place of murexide indicator, add 0.07g of calcein. The colour change in this case will be from greenish yellow fluorescence to brown.*

G.2 PREPARATION OF SAMPLE SOLUTION

Dissolve about 2.5 g of the material, accurately weighed, in a limited quantity of concentrated Hydrochloric acid and make up the volume to 1000 mL.

G.3 PROCEDURE

G.3.1 Pipette out 10 mL of the prepared sample solution (**G.2**) in a 250 mL conical flask and dilute with 40 mL of water. Add 1.5 mL of standard Sodium hydroxide solution followed by a small quantity (0.1 g to 0.2 g) of murexide indicator (or 0.07 g of calcein indicator). Titrate against standard EDTA solution till the colour changes from wine red to purple. (In the case of calcein indicator the colour change will be from greenish yellow fluorescence to brown) Note the burette reading. (V_1)

G.3.2 Pipette out 10 mL of the prepared sample solution (**G.2**) in a 250-mL conical flask and dilute with 35 mL of water. Add 5 mL of buffer solution and 5 drops of erichrome black 'T' indicator. Titrate against standard EDTA solution till the wine red colour of the solution changes to blue.

G.4 CALCULATION

Calculate the per cent Magnesium oxide content in the sample as follows.

$$\text{Magnesium oxide (as MgO),} \\ \text{per cent by mass} = \frac{7190 (V_2A_2 - V_1A_1)}{m}$$

Where,

V_2 is the Volume, in ml, of standard EDTA solution used in **G.3.2**;

A_2 is the mass, in g, of Calcium oxide equivalent to one millilitre of standard EDTA solution as obtained in **G.1.8 (b)**;

V_1 is the volume, in ml, of standard EDTA solution used in **G.3.1**;

A_1 is the mass, in g, of Calcium oxide equivalent to one millilitre of standard EDTA solution as obtained in **G.1.8 (a)**;and

m is the mass, in g, of the material in **G.2**.

APPENDIX H

DETERMINATION OF HEAVY METALS

Atomic Absorption Spectroscopy (**AAS**) methodology or Inductively Coupled Plasma Mass Spectrometry (**ICP-MS**) shall be used for the determination of heavy metals.