

SRI LANKA STANDARD 1247 : 2003
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**SPECIFICATION FOR
BLENDED HYDRAULIC CEMENTS**

SRI LANKA STANDARDS INSTITUTION

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BLENDED HYDRAULIC CEMENTS**

SLS 1247 : 2003

Gr. 12

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**SRI LANKA STANDARD
SPECIFICATION FOR BLENDED HYDRAULIC CEMENTS**

FOREWORD

This standard was approved by the Sectoral Committee on Building and Construction Materials and was authorized for adoption and publication as a Sri Lanka Standard by the Council of the Sri Lanka Standards Institution on 2003 –06-26

Blended hydraulic cements (Portland Pozzolona Cement / Portland Slag Cement) normally produce less heat of hydration and offer greater resistance to the effect of aggressive environment than normal Portland cement. Moreover, it reduces the leaching of calcium hydroxide liberated during the setting and hydration of cement. **It is particularly useful in marine and hydraulic structures and large mass concrete structures.** Blended cement can generally be used wherever Ordinary Portland Cement is usable under normal conditions. However, it should be appreciated that all Pozzolanas need not necessarily contribute to strength at early ages. In view of this fact, this specification has been prepared to enable manufacturers to produce Blended Cement equivalent to Ordinary Portland Cement on the basis of compressive strength at 3 days and 28 days

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test method or observation, shall be rounded off in accordance with **CS 102**. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this standard.

In the preparation of this standard the assistance derived from the publications of the American Society for Testing and Materials, the British Standards Institution, and the Bureau of Indian Standards is gratefully acknowledged.

1. SCOPE

This Sri Lanka standard for Blended hydraulic cements covers the requirements for composition, manufacture, chemical and physical properties and for packaging and marking.

This specification pertains to one strength class of Blended hydraulic cements using natural or artificial Pozzolanic material or slag with Ordinary Portland Cement or Portland Cement Clinker.

2. REFERENCES

- CS 102 Presentation of numerical values
- SLS 107 Ordinary Portland Cement, Part 1 : Requirements, Part 2 : Test methods.
- SLS 428 Random sampling methods.
- SLS 1144 Ready-mixed Concrete
- ISO 565 Test sieves – Metal wire cloth, perforated metal plate electroformed sheet – nominal sizes of openings.
- BSEN 196 Method of testing cement, Part 21 : Determination of the chloride, carbon dioxide and alkali content of cement

3. DEFINITIONS

For the purposes of this standard, the following definitions shall apply.

3.1 blended hydraulic cement : A Hydraulic Cement consisting of two or more inorganic constituents (at least one of which is not Portland Cement or Portland Cement clinker) which separately or in combination contribute to the strength gaining properties of the cement, (made with or without other constituents, by intergrinding or other blending)

3.2 main constituent : Specially selected inorganic material in a proportion exceeding 5 per cent by mass related to the sum of all main and minor additional constituents (see **5.1**).

3.3 manufacturer : The establishment responsible for the quality of cement packed under the brand name of that establishment and shall include any of the following categories :

- i) Cement manufactured and packed in Sri Lanka;
- ii) Cement imported in bulk form and packed in Sri Lanka ; and
- iii) Cement imported in any other form under a Sri Lankan brand name.

3.4 minor additional constituent : Specially selected inorganic material used in a proportion not exceeding a total of 5 per cent by mass related to the sum of all main and minor additional constituents. (see **5.2**).

3.5 reactive calcium oxide (CaO) : That fraction of the calcium oxide which under normal hardening conditions can form calcium silicate hydrates or calcium aluminate hydrates

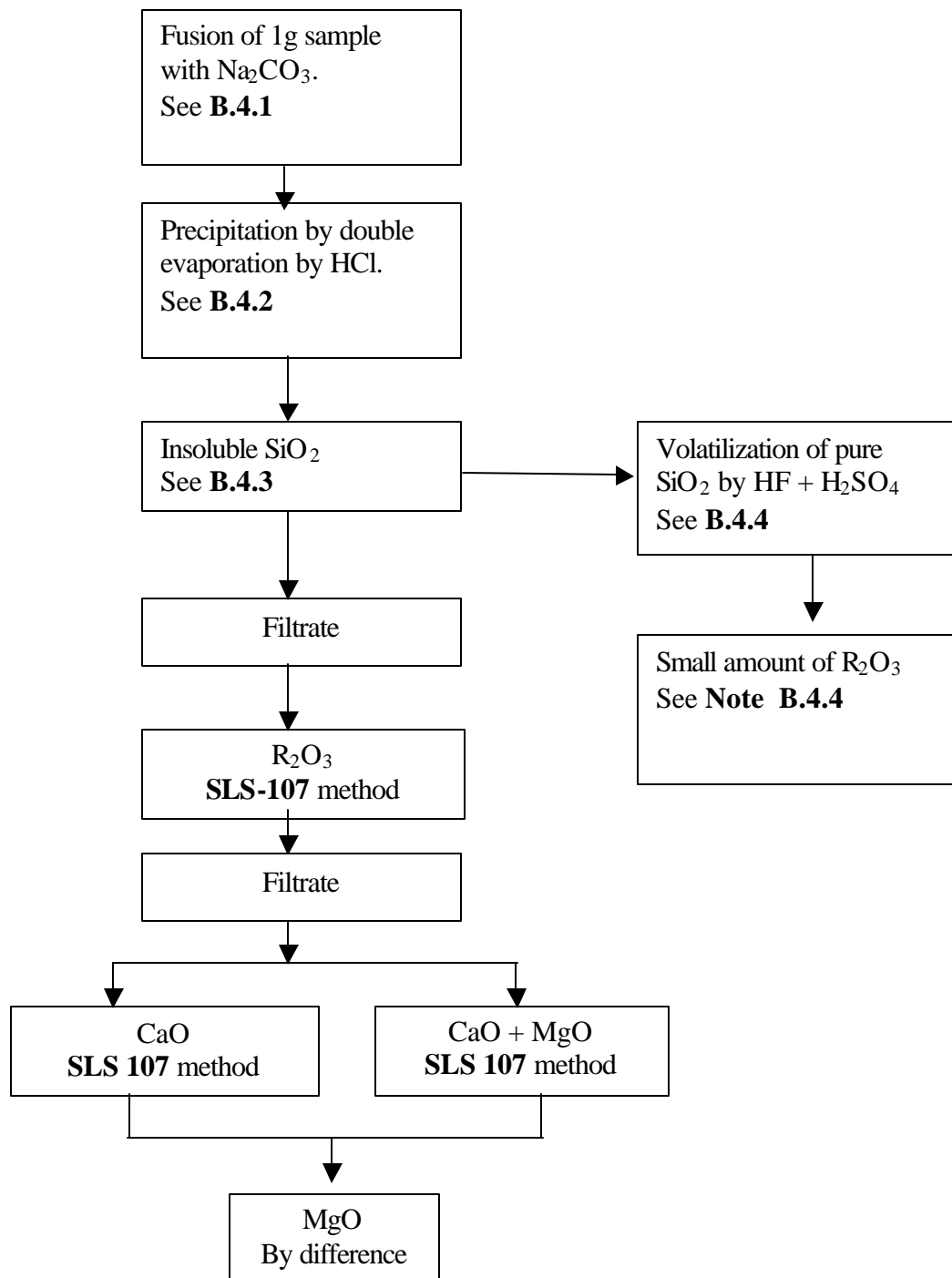
NOTE

*To evaluate this fraction the total calcium oxide content (See **Figure 1**) is reduced by the fraction corresponding to calcium carbonate (CaCO_3), based on the measured carbon dioxide (CO_2) content (See **Appendix A**) and the fraction corresponding to calcium sulphate (CaSO_4), based on the sulphate content measured relative to SO_3 content (see **SLS 107 : Part 2 : 2002**) after subtraction of the SO_3 taken up by alkalis.*

3.6 reactive silicon dioxide (SiO_2) : That fraction of the silicon dioxide which is soluble after treatment with hydrochloric acid (HCl) and with boiling potassium hydroxide (KOH) solution.

NOTE

*The quantity of reactive silicon dioxide is determined by subtracting from the total silicon dioxide content (See **Appendix B**) that fraction contained in the residue insoluble in hydrochloric acid and potassium hydroxide (see **Appendix C**).*

**NOTE**

If the insoluble residue (Test method SLS 107) is less than 1.5 percent, the determination of the main constituents by SLS 107 method shall apply, without fusion.

FIGURE 1 - Analysis of the main constituents

4. COMPOSITION AND MANUFACTURE

4.1 Blended hydraulic cement consists of an intimate and uniform blend of ordinary Portland cement and fine Pozzolanic material / Slag, and it is produced either by intergrinding Portland cement clinker, Pozzolanic material / Slag and gypsum or by blending ordinary Portland cement and fine Pozzolanic material / Slag. The constituents of the above mineral additions are shown in Table I.

4.2 Blended cements shall be either Type 1 or Type 2 depending on the main constituents used for blending with Portland cement or Portland cement clinker (See Table 1)

TABLE 1 – Composition of the blended cement

Designation and Type	Main constituents (% by mass)			Minor additional constituent (% by mass)
	Clinker	Pozzolanic material	Blast furnaces slag	
Portland Pozzolanic Cement (Type 1)	65 to 94	6 to 35	---	0 to 5
Portland Slag Cement (Type 2)	65 to 94	---	6 to 35	0 to 5

5. CONSTITUENTS

5.1 Main constituents

5.1.1 *Portland cement clinker*

Portland cement clinker is made by sintering a precisely specified mixture of raw materials (raw meal, paste or slurry) containing elements, usually expressed as oxides, CaO, SiO₂, Al₂O₃, Fe₂O₃ and small quantities of other materials. The raw meal, paste or slurry is finely divided, intimately mixed and therefore homogeneous.

Portland cement clinker is a hydraulic material which shall consist of at least two-thirds by mass of calcium silicates (3CaO . SiO₂ and 2CaO. SiO₂), the remainder consisting of aluminium and iron containing clinker phases and other compounds. The ratio by mass (CaO) / (SiO₂) shall be not less than 2.0. The content of magnesium oxide (MgO) (see Figure 1) shall not exceed 5.0 per cent by mass.

5.1.2 *Granulated blastfurnace slag*

Granulated blastfurnace slag is made by rapid cooling of a slag melt of suitable composition, as obtained by smelting iron ore in a blastfurnace and contains at least two-thirds by mass of glassy slag and possesses hydraulic properties when suitably activated.

Granulated blastfurnace slag shall consist of at least two-thirds by mass of the sum of calcium oxide (CaO), magnesium oxide (MgO) and silicon dioxide (SiO₂). The remainder contains aluminium oxide (Al₂O₃) together with small amounts of other compounds. The ratio by mass (CaO + MgO) / (SiO₂) shall exceed 1.0.

5.1.3 *Pozzolan materials*

5.1.3.1 General

Pozzolan materials are natural substances of siliceous or silico-aluminous composition or a combination thereof. Although fly ash has Pozzolan properties, it is specified in a separate clause (see 5.1.4).

Pozzolan materials do not harden in themselves when mixed with water but, when finely ground and in the presence of water, they react at normal ambient temperature with dissolved calcium hydroxide (Ca(OH)₂) to form strength-developing calcium silicate and calcium aluminate compounds. These compounds are similar to those which are formed in the hardening of hydraulic materials. Pozzolanas consist essentially of reactive silicon dioxide (SiO₂) and aluminium oxide (Al₂O₃). The remainder contains iron oxide (Fe₂O₃) and other oxides. The proportion of reactive calcium oxide for hardening is negligible. The reactive silicon dioxide content shall be not less than 25.0 per cent by mass when determined in accordance with the test method given in **Appendix B**.

Pozzolan materials shall be correctly prepared, i.e. selected, homogenized, dried, or heat treated and comminuted, depending on their state of production or delivery.

5.1.3.2 Natural Pozzolana

Natural Pozzolanas are usually materials of volcanic origin or sedimentary rocks with suitable chemical and mineralogical composition and shall conform to **5.1.3.1**.

5.1.3.3 Natural calcined Pozzolanas

Natural calcined pozzolanas are materials of volcanic origin, clays, shales or sedimentary rocks, activated by thermal treatment and shall conform to **5.1.3.1**.

5.1.3.4 Paddy husk ash

Paddy husk, a waste of the rice milling industry, which contains amorphous SiO₂ embedded in the body of the husk, on combustion under controlled burning, generates about 15 to 25 per cent of its mass as ash (Paddy husk ash). The ash shall contain at least 85 per cent by mass of amorphous silicon dioxide when determined in accordance with **Appendix B**.

5.1.3.5 Burnt shale

Burnt shale, specifically burnt oil shale, is produced in a special kiln at temperatures of approximately 800 °C. Owing to the composition of the natural material and the production process, burnt shale contains clinker phases, mainly dicalcium silicate and monocalcium aluminate. It also contains, besides small amounts of free calcium oxide and calcium sulphate, larger proportions of Pozzolanically reacting oxides, especially silicon dioxide. Consequently, in a finely ground state burnt shale shows pronounced hydraulic properties like Portland Cement and in addition Pozzolanic properties.

Adequately ground burnt shale shall have a compressive strength of at least 25.0 N/mm² at 28 days when tested in accordance with **SLS 107 : Part 2 : 2002** for cement. The test mortar shall be prepared with finely ground burnt shale only instead of cement. The mortar specimens shall be demoulded 48 h after preparation and cured in a moist atmosphere of relative humidity of at least 90 per cent until tested.

The expansion (soundness) of burnt shale shall not exceed 10 mm when tested by the Le Chatellier's method specified in the **SLS 107 : Part 2 : 2002** for cement, using a mixture of 30 per cent by mass of ground burnt shale and 70 per cent by mass of Portland cement conforming to **SLS 107**.

NOTE

If the sulphate content measured relative to SO₃ content of the burnt shale exceeds the permissible upper limit for the sulphate content of the cement then this has to be taken into account for the manufacture of the cement by appropriately reducing the calcium sulphate-containing constituents.

5.1.4 Fly ashes

5.1.4.1 General

Fly ash is obtained by electrostatic or mechanical precipitation of dust-like particles from the flue gases from furnaces fired with pulverized coal. Fly ash obtained by other methods shall not be used in cement that conforms to this standard.

Fly ash may be siliceous or calcareous in nature. The former has Pozzolanic properties; the latter may have, in addition hydraulic properties. The loss on ignition of fly ash determined in accordance with the method given in **SLS 107: Part 2 : 2002** for cement but using an ignition time of 1 h for fly ash, shall not exceed 5.0 per cent by mass.

Fly ash with a loss on ignition of 5.0 per cent to 7.0 per cent by mass may also be accepted, provided that particular requirements for durability and for compatibility with admixtures are met (see **SLS 1144 : Part 1**). Loss on ignition shall be determined in accordance with the method specified in **SLS 107** except that fly ash will be used instead of cement.

Siliceous fly ash is a fine powder of mostly spherical particles having Pozzolanic properties. It consists essentially of reactive silicon dioxide (SiO_2) and aluminium oxide (Al_2O_3). The remainder contains iron oxide (Fe_2O_3) and other compounds.

The content of reactive calcium oxide shall be less than 10.0 per cent by mass, and the content of free calcium oxide, as determined by the method described in Appendix D shall not exceed 1.0 per cent by mass. Fly ash having a free calcium oxide content higher than 1.0 per cent by mass but less than 2.5 per cent by mass is also acceptable provided that the requirement on expansion (soundness) does not exceed 10 mm when tested by the Le Chatellier's method specified in **SLS 107 : Part 2 : 2002** for cement using a mixture of 30 per cent by mass of siliceous fly ash and 70 per cent by mass of Portland cement conforming to **SLS 107**. The reactive silicon dioxide content shall be not less than 25.0 per cent by mass.

5.1.4.2 Calcareous fly ash

Calcareous fly ash is a fine powder, having hydraulic and/or pozzolanic properties. It consists essentially of reactive calcium oxide (CaO), reactive silicon dioxide (SiO_2) and aluminium oxide (Al_2O_3). The remainder contains iron oxide (Fe_2O_3) and other compounds. The content of reactive calcium oxide shall be not less than 10.0 per cent by mass. Calcareous fly ash containing between 10.0 per cent and 15.0 per cent by mass of reactive calcium oxide shall contain not less than 25.0 per cent by mass of reactive silicon dioxide.

Adequately ground calcareous fly ash containing more than 15.0 per cent by mass of reactive calcium oxide, shall have a compressive strength of at least 10.0 N/mm^2 at 28 days when tested in accordance with **SLS 107 : Part 2 : 2002** for cement. Before testing, the fly ash shall be ground and the fineness, expressed as the proportion by mass of the ash retained when wet sieved on a $40 \mu\text{m}$ mesh sieve, shall be between 10 per cent and 30 per cent by mass. The test mortar shall be prepared with ground calcareous fly ash only instead of cement. The mortar specimens shall be demoulded 48 h after preparation and then cured in a moist atmosphere of relative humidity of at least 90 per cent until tested.

The expansion (soundness) of calcareous fly ash shall not exceed 10 mm when tested by the Le Chatellier's method specified in **SLS 107 : Part 2 : 2002** using a mixture of 30 per cent by mass of calcareous fly ash ground as described above and 70 per cent by mass of Portland cement conforming to **SLS 107**.

NOTE

If the sulphate content measured relative to SO_3 content of the fly ash exceeds the permissible upper limit for the sulphate content of the cement then this has to be taken into account for the manufacture of the cement by appropriately reducing the calcium sulphate-containing constituents.

5.2 Minor additional constituents

Minor additional constituents are specially selected, inorganic natural mineral materials, inorganic mineral materials derived from the clinker production process or constituents as specified in 5.1 unless they are included as main constituents in the cement.

Minor additional constituents, after appropriate preparation and on account of their particle size distribution, improve the physical properties of the cement (such as workability or water retention). They can be inert or have slightly hydraulic, latent hydraulic or pozzolanic properties. However, no requirements are set for them in this respect.

Minor additional constituents shall be correctly prepared, i.e selected, homogenized, dried and comminuted depending on their state of production or delivery. They shall not increase the water demand of the cement appreciably, impair the resistance of the concrete or mortar to deterioration in any way or reduce the corrosion protection of the reinforcement.

NOTE

Information on the minor additional constituents in the cement should be available from the manufacturer on request.

5.3 Calcium sulphate

Calcium sulphate is added to the other constituents of cement during its manufacture to control setting.

Calcium sulphate can be gypsum (calcium sulphate dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2} \text{H}_2\text{O}$), or anhydrite (anhydrous calcium sulphate, CaSO_4) or any mixture of them. Gypsum and anhydrite are found naturally. Calcium sulphate is also available as a by-product of certain industrial processes.

5.4 Additives

Additives for the purpose of this standard are constituents not covered in 5.1 to 5.3 which are added to improve the manufacture or the properties of the cement.

The total quantity of additives shall not exceed 1.0 per cent by mass of the cement (except for pigments). The quantity of organic additives on a dry basis shall not exceed 0.5 per cent by mass of the cement.

These additives shall not promote corrosion of the reinforcement in concrete or impair the properties of the cement or of the concrete or mortar made from the cement.

Admixtures for concrete, mortar or grouts in dry form shall not be incorporated in blended cement.

6. MECHANICAL , PHYSICAL AND CHEMICAL REQUIREMENTS

6.1 Mechanical requirements

6.1.1 *Compressive strength*

The average compressive strength of three mortar cubes prepared, stored and tested as in the method described in **6.0** of **SLS 107 : Part 2 : 2002** shall not be less than 20.0 N/mm² at 3 days and 40.0 N/mm² at 28 days. The compressive strength of mortar cubes at 28 days shall be higher than that at 3 days and shall not exceed 62.5 N/mm².

6.2 Physical requirements

6.2.1 *Setting time*

The setting time of cement paste of standard consistency as determined by the method described in **SLS 107 : Part 2 : 2002** shall be :

- a) Initial setting time not less than 45 min.; and
- b) Final setting time, not more than 10 h.

6.2.2 *Soundness*

6.2.2.1 The test method for soundness depends on the magnesia content of the cement. Hence prior to testing for soundness, magnesia content shall be determined in accordance with the appropriate method given in Figure 1.

For cement with magnesia content not more than 3 per cent, **6.2.2.2** shall apply. For other cement **6.2.2.3** shall apply.

6.2.2.2 The cement shall not have an expansion of more than 10 mm when tested for soundness by the Le Chatellier's method described in **9** and **10** of **SLS 107 : Part 2 : 2002** for cement.

If the cement fails to comply with this requirement, a further test shall be made in the manner described. For this test, another portion of the same sample shall be used after it has been aerated by being spread out to a depth of 70 mm to 80 mm at a relative humidity of 50 per cent to 80 per cent for a total period of 7 days. The expansion of this aerated sub sample shall not exceed 5 mm.

6.2.2.3 The cement shall when tested for soundness by the autoclave test described in **10** of **SLS 107 : Part 2 : 2002** and shall not have an expansion of more than 0.8 per cent.

6.2.2.4 Soundness of constituents of blended cement shall conform to limits specified in **5**, when tested as specified.

6.3 Chemical requirements

6.3.1 Blended cement

The properties of the blended cement shall conform to the requirements listed in Table 2 when tested in accordance with the standards referred in the table.

6.3.2 Constituents

The constituents shall conform to chemical requirements specified in 5, when tested in accordance with the relevant method.

TABLE 2 - Chemical requirements

Property	Reference to test method	Cement type	Requirements Per cent by mass of the final cement
Sulphate content (as SO ₃)	SLS 107 : Part 2	Type 1 Type 2	3.5
Chloride content	SLS 107 : Part 2	Type 1 Type 2	0.10

7 MARKING

7.1 Blended hydraulic cement manufactured in compliance with this standard shall be marked in relation to the product (either on the bag or, when supplied in bulk, on the manufacturer's certificate, the delivery note or the invoice) legibly and indelibly with the following particulars:

- a) name, address, trade mark or other means of identification of the manufacturer (see **3.3**);
- b) type of Blended hydraulic cement, that is : Portland Pozzolanic cement or Portland slag cement
- c) country in which cement is produced;
- d) the net mass of the contents in kg. (See Note in **8**) ;
- e) the date of packing; and
- f) range for loss on ignition for fly ash, see **5.1.4.1** (applicable only if loss on ignition limit exceeds 5.0 per cent)

NOTES

- 1) Item b) shall appear in Sinhala, Tamil, and English.
- 2) Attention is drawn to the certification marking facilities offered by the Sri Lanka Standards Institution. See the inside back cover of this standard.

8. DELIVERY AND PACKAGING

The cement shall be supplied in bulk, or packed in bags of sufficient strength and construction to prevent damage or deterioration of cement, during normal handling.

Any container used for bulk supply shall have an airtight fully enclosed body robust enough to prevent spillage of cement, and a special facility for dustless discharge such as air slide, pneumatic discharger or spiral conveyor.

In order to assess whether cement bags are of sufficient strength, the drop test on bags consisting of Blended hydraulic cement, as delivered, shall be carried out as described in **11 of SLS 107 : Part 2 : 2002**. Each Blended hydraulic cement bag shall be capable of sustaining 10 drops without failure, to pass the drop test.

When Blended hydraulic cement is issued in bag form, the net mass of each bag shall be at least 50.0 kg. The net mass of the bag of cement shall be determined from its gross mass and the mass of package. The nominal mass of the empty bag shall be marked (to the nearest gram) on the package, where facilities exist for such marking. If mass of an empty bag is not displayed on the bag, mass should be determined by weighing 10 empty bags used for the same batch. These empty bags should be supplied by the manufacturer. (See **3.3**)

The bags of Blended hydraulic cement not in good condition, due to causes such as moisture patches, torn bags, burst stitches, spilling cement or exudation of cement dust, at the time of inspection, shall be rejected.

NOTE

To protect cement from premature hydration after delivery, bulk silos should be waterproof and internal condensation should be minimized. Paper bags should be stored clear of the ground, not more than eight bags high and protected by a waterproof structure. As significant strength losses begin after 4 weeks to 6 weeks of storage in bags in normal conditions, and considerably sooner under adverse weather conditions or high humidity, deliveries should be controlled and used in order of receipt.

9. MANUFACTURER'S CERTIFICATE

The manufacturer shall be satisfied that the Blended hydraulic cement at the time of its delivery complies with the requirements of this standard (see **Notes 1 & 2**). The manufacturer on request shall forward a certificate to this effect to the purchaser or his representative. The certificate shall include the following details in respect of the Blended hydraulic cement delivered.

- a) The percentage of Ordinary Portland cement or Portland cement clinker, pozzolanic material and minor additional constituents.

b) The results of tests on:

- i) compressive strength at 3 days and 28 days (which may be provided after delivery);
- ii) setting times;
- iii) soundness;
- iv) sulphate content;
- v) chloride content; and
- vi) chemical composition of major constituents.

NOTES

1. In case of imported cement, time of delivery means the time of delivery to a Port in Sri Lanka.

2. If the time between the sampling and the delivery of bagged cement to a Port in Sri Lanka exceeds two weeks, the compressive strength of the cement (at 3 days and 28 days) may be reduced during transport and / or storage. In case of doubt, cement should be re-tested at the time of delivery.

10. INDEPENDENT TESTS

10.1 If the purchaser or his representative requires independent tests, they shall be carried out in accordance with this standard on the written instructions of the purchaser or his representative.

10.2 The manufacturer/vendor shall supply, free of charge, the cement required for testing. Unless otherwise specified in the enquiry and order, the cost of the tests shall be borne as follows:

- a) By the manufacturer/vendor if the results show that the Blended hydraulic cement does not comply with the requirements of this standard; and
- b) By the purchaser if the results show that the Blended hydraulic cement complies with the requirements of this standard.

NOTE

List of testing authorities acceptable to the Sri Lanka Standards Institution is available on request.

11. SAMPLING AND ACCEPTANCE

such an agreement is not possible or for the purpose of independently assessing the conformity with this standard, sampling of cement and criteria of conformity shall be in accordance with Appendix E.

APPENDIX A

DETERMINATION OF CARBON DIOXIDE CONTENT

A.1 Principle

The cement sample is treated with phosphoric acid to decompose the carbonate present. The carbon dioxide liberated is entrained in a current of carbon dioxide-free gas or air through a series of absorption tubes. The first two removes hydrogen sulphide and water and the following then absorb carbon dioxide. Two absorption tubes, each containing a granular absorption for carbon dioxide and anhydrous magnesium perchlorate to retain the water formed during the absorption reaction are weighed to determine the mass of carbon dioxide released.

A.2 Reagents

A.2.1 Copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)

A.2.2 Copper sulphate solution, saturated

A.2.3 Absorbent for hydrogen sulphide

Place a weighed quantity of dried pumice stone with a grain size between 1.2 mm and 2.4 mm into a flat dish and cover with a volume of saturated copper sulphate solution so that the mass of the copper sulphate solution is approximately half that of the pumice stone. Evaporate the mixture to dryness, while stirring frequently with a glass rod. Dry the contents of the dish for at least 5 h in an oven at a temperature of 155 ± 5 °C. Allow the solid mixture to cool in a desiccator and store in an airtight bottle.

A.2.4 Absorbent for water, anhydrous magnesium perchlorate ($\text{Mg}(\text{ClO}_4)_2$) with a particle size between 0.6 mm and 1.2 mm.

A.2.5 Absorbent for carbon dioxide, synthetic silicates with a particle size between 0.6 mm to 1.2 mm impregnated with sodium hydroxide (NaOH).

A.2.6 Concentrated phosphoric acid (H_3PO_4)

A.2.7 Concentrated sulphuric acid (H_2SO_4)

A.3 Apparatus

A.3.1 Figure 2 shows a typical piece of apparatus which can be fitted with either a cylindrical pressure container, a small electrical compressor or a suitable suction pump which will ensure an even flow of gas or air.

The gas (air or nitrogen) entering the apparatus should have previously had its carbon dioxide removed by first being passed through an absorbent tube or tower containing the carbon dioxide absorbent (A.2.5). The apparatus consist of a 100 ml reaction flask (A) fitted with a three-neck adaptor. Neck B₁ is connected to a dropping funnel (O), neck B₂ to a connecting tube and neck C

to a water cooled condenser. The funnel on to B₁ and the connecting tube onto B₂ are joined together by means of a Y-piece (P), so that the carbon dioxide-free air can flow either through the connecting tube or the funnel by means of a Mohr clip (N). After the condenser (L), the gas is passed through concentrated sulphuric acid (D), then through absorption tubes containing the absorbent for hydrogen sulphide (A.2.3) (E) for water (A.2.4) (F) and subsequently through two absorption tubes (G,H) which can be weighed and which are three-quarters filled with the absorbent for carbon dioxide (A.2.5) and a quarter with the absorbent for water (A.2.4). The absorbent for carbon dioxide (A.2.5) is placed upstream of the absorbent for water (A.2.4) with respect to the gas flow. Absorption tube (H) is followed by an additional absorption tube (I), which also contains the absorbent for carbon dioxide and water, which is fitted in order to protect absorption tube (H) against penetration by carbon dioxide and water from the air.

The absorption tubes (G) and (H) which are to be weighed may have, for example, the following sizes.

External distance between branches 45mm, internal diameter 20 mm, distance between the lower part of the tube and the upper part of the ground section 75mm, and tube wall thickness 1.5mm.

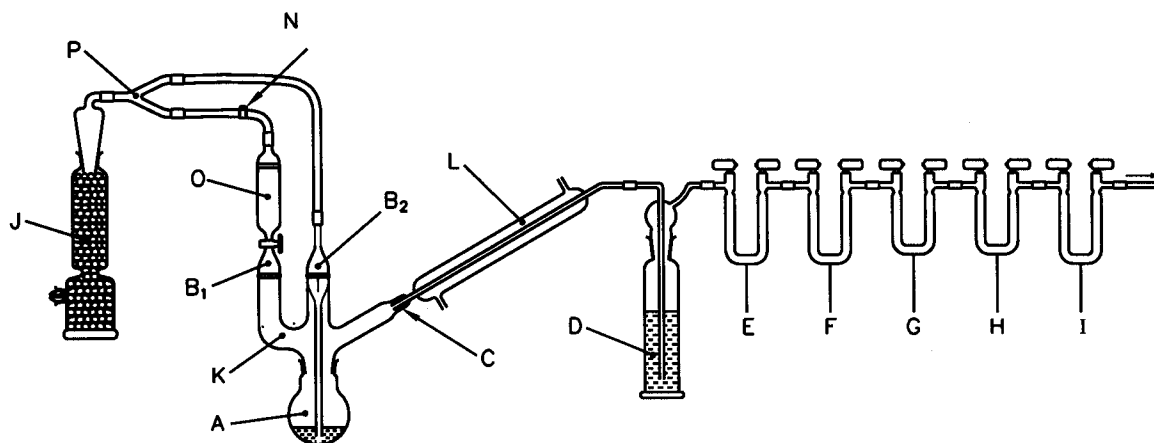
A.3.2 Balance, capable of weighing to the nearest 0.000 1 g.

A.3.3 Electric oven, which can be set at 105 ± 5 °C and at 155 ± 5 °C.

A.3.4 Desiccator, containing anhydrous magnesium perchlorate Mg(ClO₄)₂.

A.4 Procedure

Weigh 1 ± 0.05 g of cement and place it in a dry 100 ml distillation flask. Connect the flask to the apparatus (A.3.1) as shown in Figure 2, but without the two absorption tubes (G) and (H). Pass a current of carbon dioxide-free gas through the apparatus for 15 min at approximately 3 bubbles per second (bubble counter) via the connecting tube onto B₂ (branch onto B₁, Mohr clip closed). Release the Mohr clip and remove the gas supply from the funnel (O). Add 30 ml concentrated phosphoric acid into the dropping funnel and reconnected the gas supply to fill the funnel (O).



Key

A	100 ml distillation flask
B ₁	Dropping funnel connector
B ₂	Connecting tube connector
C	Condenser connector
D	Wash bottle with concentrated sulphuric acid (A.2.7)
E	Absorption tube with absorbent for hydrogen sulphide (A.2.3)
F	Absorption tube with magnesium perchlorate absorbent for water (A.2.4)
G, H and I	Absorption tubes with absorbents for carbon dioxide (A.2.5) and for water (A.2.4)
J	Absorption tower containing carbon dioxide absorbent (A.2.5)
K	Three-armed still head
L	Condenser
N	Mohr clip
O	Dropping funnel
P	Y-piece

FIGURE 2 - Apparatus for the determination of the carbon dioxide content

Condition the closed absorption tubes (G) and (H) for 15 min in the balance case in order to achieve temperature equilibrium. Then weigh each tube separately. Shut off the flow of gas and attach the tubes to the apparatus as shown in Figure 2.

Wear protective gloves when carrying out this operation.

Then reopen the gas flow. After 10 min, close absorption tubes (G) and (H), remove them, place them in the balance case for 15 min and then weigh them separately. Repeat the passage of gas, removal and weighing of absorption tubes (G) and (H) for as long as is required for the results of two successive weighing of a tube not to differ by more than 0.0005 g.

If the change in mass of the absorption tubes (G) and (H) remains greater than 0.0005 g, renew the absorbents in tubes (E) and (F).

Attach the weighed absorption tubes (G) and (H) to the apparatus, as shown in Figure 2.

Open the funnel tap and allow the phosphoric acid to drop into the distillation flask (A). After the reaction has ceased, heat the contents of the flask to boiling and boil gently for 5 min. Maintain the gas flow through the apparatus until the flask has cooled to room temperature.

Close absorption tubes (G) and (H), remove them and place them in the balance case for 15 min and then weigh them separately. The increase in mass of each tube is used for the calculation of the carbon dioxide content (A.5).

The carbon dioxide is practically completely absorbed by tube (G). If the increase in mass of tube (H) exceeds 0.000 5 g, renew the absorbent in tube (G) and start the test again.

A.5 Expression of results

Calculate the carbon dioxide content (in percent) of the cement from the formula:

$$\text{CO}_2 = \frac{m_2 + m_3}{m_1} \times 100$$

Where m_1 is the mass of cement test portion;
 m_2 is the increase in mass of tube G after absorption;
 m_3 is the increase in mass of tube H after absorption.

The mean of the two results shall be rounded to the nearest 0.01 percent
If the carbon dioxide content calculated from equation is less than 0.5 percent, repeat the determination with a cement sample weighing 2g.

A.6 Repeatability and reproducibility

The standard deviation of repeatability is 0.07 per cent

The standard deviation of reproducibility is 0.10 per cent

APPENDIX B DETERMINATION OF TOTAL SILICA (SiO₂)

B.1 Principle of the test

This test method is based on the sodium carbonate fusion followed by double evaporation to dryness of the hydrochloric acid solution of the fusion product to convert silicon dioxide (SiO₂) to the insoluble form. The solution is filtered and the insoluble siliceous residue is ignited and weighed. Silicon dioxide is volatilized by hydrofluoric acid and the loss of mass is reported as pure SiO₂.

B.2 Reagents

Use only reagents analytical grade and distilled water, or water of equal purity.

B.2.1 Sodium carbonate (Na₂CO₃)

B.2.2 Hydrochloric acid (Specific gravity 1.19)

B.2.3 Hydrochloric acid (1 + 1)

B.2.4 Hydrochloric acid (1 + 3)

B.2.5 Hydrochloric acid (1 + 99)

B.2.6 Sulphuric acid (1 + 1)

B.2.7 Hydrofluoric acid (HF)

B.3 Apparatus**B.3.1** Balance, capable of weighing to an accuracy of 0.0001 g. or better.**B.3.2** Desiccators, containing a drying agent example silica gel.**B.3.3** Covered Platinum crucible, of capacity about 20 ml to 30 ml**B.3.4** 300 ml casserole**B.3.5** Steam bath**B.4 Procedure****B.4.1** Weigh a quantity of the ignited sample equivalent to 0.5 of the as-received sample calculated as follows:

$$M = [0.5 (100.00 - l)]/100$$

Where, M = mass of ignited sample in grams ; and
 l = loss of ignition , per cent

The ignited material from the loss on ignition determination (the method given in **SLS 107: Part 2 : 2002** for content may be followed) may be used for the sample. Thoroughly mix the sample with 4 g. to 6 g of Na_2CO_3 by grinding in an agate mortar. Place a thin layer of Na_2CO_3 on the bottom of the platinum crucible add the cement - Na_2CO_3 mixture and cover the mixture with a thin layer of Na_2CO_3 . Place the covered crucible over a moderately low flame and increase the flame gradually to a maximum (approximately 1100 °C) and maintain this temperature until the mass is quiescent (about 45 min). Remove the burner, lay aside the cover of the crucible, grasp the crucible with tongs, and slowly rotate the crucible so that the molten contents spread over the sides and solidify as a thin shell on interior. Set the crucible cover aside to cool. Rinse off the outside of the crucible and place the crucible on its side in a 300 ml casserole about one third full of water. Warm the casserole and stir until the cake in the crucible disintegrates and can be removed easily. By means of a glass rod, lift the crucible out of the liquid, rinsing it thoroughly with water, Rinse the cover and crucible with HCl (1+3): then add the rinse to the casserole. Very slowly and cautiously add 20 ml of HCl (specific gravity 1.19) to the covered casserole. Remove the cover and rinse. If any gritty particles are present, the fusion is incomplete and the test must be repeated, using a new sample.

NOTE

Subsequent steps of the test method shall be followed exactly for accurate results.

B.4.2 Evaporate the solution to dryness on a steam bath (there is no longer a gelatinous appearance). Without heating the residue any further, treat it with 5 ml to 10 ml of HCl, wait at least 2 min, and then add an equal amount of water. Cover the dish and digest for 10 min on the steam bath or a hot plate. Dilute the solution with an equal volume of hot water, immediately filter through medium-textured paper and wash the separated SiO_2 thoroughly with hot HCl (1+99), then with hot water. Reserve the residue.

Again evaporate the filtrate to dryness, and bake the residue in an oven for 1 h at 105 °C to 100°C, to cool, add 10 ml to 15 ml of HCl (1+1) and digest on the steam bath or hot plate for 10 min. Dilute with an equal volume of water, filter immediately on a fresh filter paper, and wash the small SiO₂ residue thoroughly as described in the above paragraph.

B.4.3 Transfer the filter papers and residue to a weighed platinum crucible, dry and ignite, at first slowly until the carbon of the paper is completely consumed without inflaming, and finally at 1100 °C to 1200 °C for 1 h. Cool in a desiccator and weigh (m_1). Reignite to constant mass.

B.4.4 Treat the SiO₂ thus obtained, which will contain small amounts of impurities, in the crucible with 1 ml or 2 ml of water, 2 drops of H₂SO₄ (1+1), and about 10 ml of HF, and evaporate cautiously to dryness. Finally, heat the small residue at 1050 °C to 1100 °C for 5 min, cool in a desiccator, and weigh (m_2). The difference between this mass and the mass previously obtained represents the mass of SiO₂.

NOTE

Consider the weighed residue remaining after the volatilization of SiO₂ as combined aluminum and ferric oxides and add it to the result obtained in the determination of the ammonium hydroxide group.

B.5 Calculation

Calculate the content (per cent) of the silicon dioxide (per cent) in the sample from the following equation:

$$\text{SiO}_2 = \{m_1 - m_2\} 200$$

Where,

SiO₂ is the content of the silicon dioxide (per cent)

m_1 , m_2 are the masses of the 1st and 2nd precipitates (g) as per **B.4.3** and **B.4.4** respectively.

B.6 Repeatability and reproducibility

The standard deviation of repeatability is 0.07 per cent

The standard deviation of reproducibility is 0.10 per cent

APPENDIX C

DETERMINATION OF RESIDUE INSOLUBLE IN HYDROCHLORIC ACID AND POTASSIUM HYDROXIDE

C.1 Principle

This is a conventional method in which the insoluble residue in cements is obtained by treating with a hydrochloric acid solution. The residue from this treatment is then treated with a boiling solution of potassium hydroxide. The residue is determined gravimetrically after ignition.

C.2 Procedure

To (1 + 0.05) g of cement (m_1), placed in a porcelain dish, add 25 ml of cold water and disperse using a glass-stirring rod. Add 40 ml of concentrated hydrochloric acid. Heat the solution gently and crush the sample with the flattened end of a glass-stirring rod until decomposition of the cement is complete. Evaporate to dryness on a water bath. Repeat the operation twice more with 20 ml concentrated hydrochloric acid.

Treat the residue from the last evaporation with 100 ml of dilute hydrochloric acid (1+3). Re-heat, filter through a medium filter paper and wash with almost boiling water at least ten times until free from Cl^- ions, tested by the silver nitrate test. (Add several drop of silver nitrate solution, check the absence of turbidity of precipitate in the solution. If present, continue washing while carrying out periodic checks until the silver nitrate test is negative.)

Transfer the filter and its contents to a 250 ml conical flask fitted with a bulb condenser and add 100 ml of the potassium hydroxide solution (Dissolve 250 g. KOH in water and make up to 1000 ml). Leave to stand for 16h at room temperature and then boil the solution under reflux for 4 h.

Filter on a medium filter paper and wash with water then with 100ml of hydrochloric acid (1 + 9) and finally with almost boiling water until free from Cl^- ions, tested by the silver nitrate test.

Ignite at 975 ± 25 °C for 15 minutes then check for constant mass. In general, an ignition period of 30 min is sufficient for obtaining constant mass (m_2).

C.3 Expression of results

The insoluble residue is calculated in percent from the formula:

$$\text{Insoluble residue} = \frac{m_2}{m_1} \times 100$$

Where: m_1 is the mass of the test portion, in grams;
 m_2 is the mass of the ignited insoluble residue, in grams.

C.4 Repeatability and reproducibility

The standard deviation for repeatability is 0.15 per cent

The standard deviation for reproducibility is 0.18 per cent

APPENDIX D
DETERMINATION OF FREE CALCIUM OXIDE CONTENT

D.1 Reagents

Use only reagents of analytical grade and distilled water, or water of equal purity.

- D.1.1** Butanoic acid, 3-oxo-ethyl ester (= ethyl acetoacetate)
- D.1.2** Butane 2 -ol
- D.1.3** Propane 2-ol
- D.1.4** Indicator (0.1 g of bromophenol blue in 100 ml of ethanol)
- D.1.5** Hydrochloric acid (0.100 M)

D.2 Apparatus

- D.2.1** Balance, capable of weighing to an accuracy of 0.0001 g or better
- D.2.2** Test sieve, with 0.063 mm sieve cloth conforming to **ISO 565** test sieve
- D.2.3** Mortar and pestle, or similar equipment for grinding
- D.2.4** Desiccator, containing a drying agent, e.g. silica gel
- D.2.5** Flask, 250 ml.
- D.2.6** Spiral reflux condenser
- D.2.7** Absorption tube, filled with sodium hydroxide on an inorganic carrier (to protect the contents of the flask and the condenser from reacting with atmospheric carbon dioxide).
- D.2.8** Filter crucible, with pore sizes of 0.004 mm to 0.010 mm.

NOTE

Alternatively also a filter funnel, in which a filter paper with fine pores of a diameter of approximately 0.002 mm and a filter paper with medium pores of a diameter of approximately 0.007 mm can be placed may be used.

D.3 Procedure

D.3.1 Preparation of sample

Subdivide the laboratory sample, prepared in accordance with Appendix **A** of **SLS 107 : Part 1: 2002** by a suitable method to obtain a sub sample of about 100 g. Pass this sub sample through the test sieve (**D.2.2**). Grind any residue in the mortar (**D.2.3**) until all the sub sample passes through the sieve completely. Homogenize the total sub sample and place it in the desiccator (**D.2.4**) until tested.

D.3.2 Determination

Place a weighed portion of 1.0 g to 1.5 g of the subsample prepared in accordance with **D.3.1** into the 250 ml flask (**D.2.5**) and add a mixture of 12 ml butanoic acid, 3-oxo-ethyl ester (**D.1.1**) and 80 ml butan-2-ol (**D.1.2**). Fit the flask with the spiral reflux condenser (**D.2.6**) and the absorption tube (**D.2.7**) and boil for 1 h. Filter the warm mixture through the filter crucible (**D.2.8**). Wash the residue with propan-2-ol (**D.1.3**) until the filtrate reaches a volume of 50 ml. If the filtrate is cloudy, reject it and repeat the extraction with a new weighed portion of the subsample.

Add a few drops of bromophenol blue indicator (**D.1.4**) to the filtrate and titrate with hydrochloric acid (**D.1.5**) until the colour changes to yellow. Record the volume V of hydrochloric acid used.

D.3.3 Calculations

The free calcium oxide content (M_{CaO}), expressed as a percentage by mass of the dry fly ash, shall be calculated from the following equation :

$$M_{CaO} = \frac{28.04 \times C \times V}{m \times 1000}$$

where

C is the concentration in mol/l of the hydrochloric acid solution ;
V is the volume in ml of hydrochloric acid solution used for titration; and
m is the weighed proportion in g. of the dried fly ash

D.4 Results

The mean value of two determinations, calculated to two decimal places and expressed to one decimal place, shall be taken as the free calcium oxide content of the sample.

D.5 Repeatability and reproducibility

The standard deviation for repeatability is 0.02 per cent by mass (provisional value).

The standard deviation for reproducibility is 0.04 per cent by mass (provisional value).

APPENDIX E SAMPLING AND CRITERIA FOR CONFORMITY

The sampling scheme given in this Appendix should be applied where compliance of a lot to the requirements of this specification is to be assessed based on statistical sampling and inspection.

Where compliance with this specification is to be assured based on manufacture's control systems coupled with type testing and check tests or any other procedure, appropriate schemes of sampling and any other inspection procedure should be adopted.

E.1 Lot

In any consignment, all the packages of cement or a quantity of bulk cement belonging to one batch of manufacture or supply shall constitute a lot.

E.2 General requirements of sampling

E.2.1 Precautions shall be taken to transfer the sample into a clean, dry and air tight containers and marked with necessary details of sampling.

E.2.2 Sampling instruments shall be clean and dry when used.

E.3 Sampling instruments

A sampling tube or an appropriate instrument shall be used (see Figure 3).

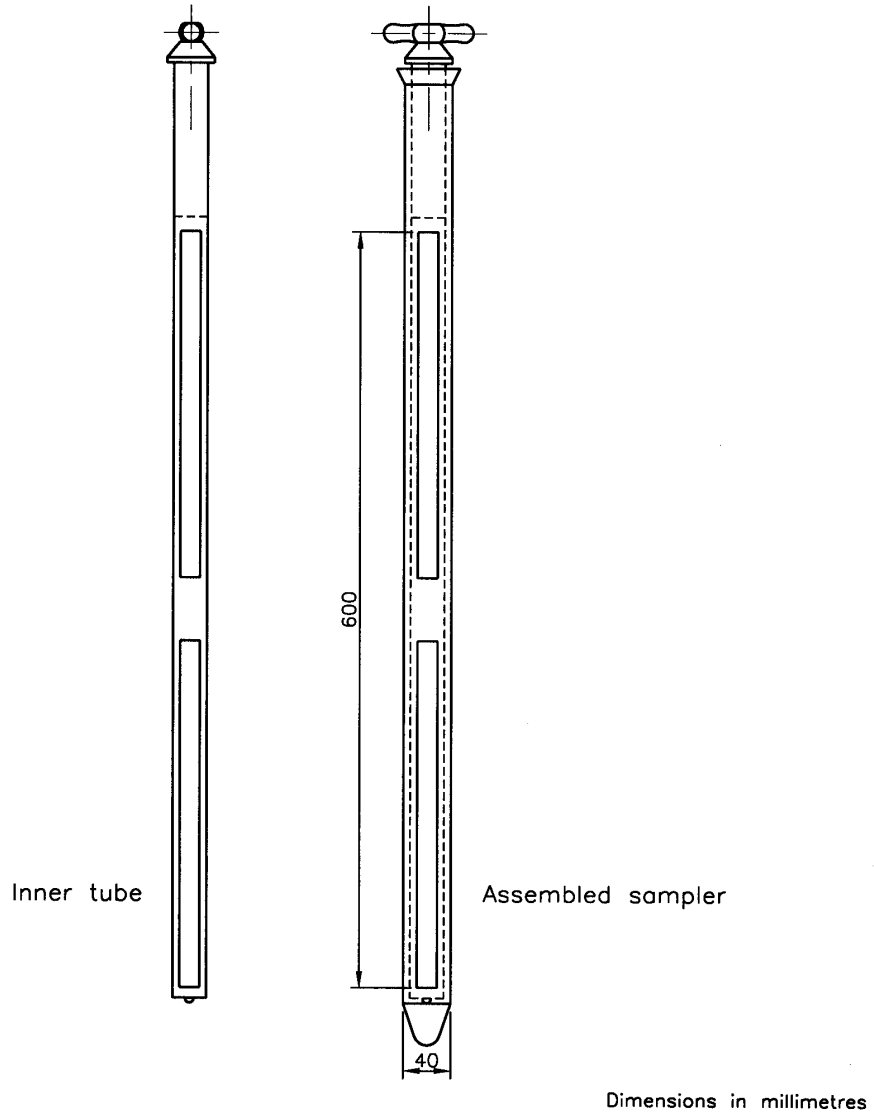


FIGURE 3 - Typical sampling tube

E.4 Scale of sampling

Samples shall be tested for each lot for ascertaining its conformity to the requirements of this specification

E.4.1 *Sampling from bags*

E.4.1.1 The number of bags to be selected from the lot shall be in accordance with Table 3.

TABLE 3 : Scale of sampling

Lot size	Sample size
number of bags	
Up to 150	5
151 to 300	10
301 to 600	15
601 to 800	20
801 to 1000	25
1001 and above	30

These bags shall be chosen at random, from the lot. To ensure the randomness of selection a method given in **SLS 428** shall be used. Alternatively all the bags in the lot may be arranged in a serial order and starting from any bag, Every r^{th} bag be selected till the requisite number is obtained, r being the integral part of N/n where N is the lot-size (number of bags in a lot) and n is the sample size (number of bags selected).

E.4.1.2 Preparation of sample

Equal quantities of cement shall be taken from each bag selected as in E.4.1.1 and mixed together to form a composite sample of approximately 10 kg.

E.4.2 *Sampling from bulk container*

E.4.2.1 A gross sample shall be taken from the lot by taking increments at regular intervals when the cement is being charged into the container or being discharged from the container. The number of increments shall be such that one increment is taken for 10 tonnes of cement. Each increment shall weigh about 2 kg. All the increments taken as above shall be mixed together to form a composite sample of cement.

E.4.3 Sampling from wagon/truck

E.4.3.1 The mass of the gross sample to be drawn from the lot shall depend on the lot size.

E.4.3.2 A sufficient number of increments shall be drawn from evenly distributed points of each wagon/truck to obtain a gross sample of 2 kg. (approximately) per 10 tonnes of cement in the lot or part of it. The increments taken as above shall be mixed together to form a composite sample.

NOTE

1. Increments shall be taken by using a sampling tube or appropriate sampling instrument.

2. The minimum mass of the gross sample to be drawn shall be 10 kg. Sufficient number of increments shall be taken when the lot size is less than 50 tonnes.

E.4.4 Preparation of laboratory sample

The composite sample prepared as in **E.4.2.1** or **E.4.3.2** shall be reduced to obtain the laboratory sample of 10 kg.

NOTE

Heaping into a cone and quartering method shall be applied for reducing the size of the sample.

E.5 Number of tests

E.5.1 Each bag selected as in **E.4.1.1** shall be inspected for marking and packaging requirements. (see **7** and **8**)

E.5.2 In case of packaged material, the composite sample prepared as in **E.4.1.2** shall be tested for all the requirements (see **6**) given in the standard.

In case of bulk material, the laboratory sample prepared in accordance with **E.4.4** shall be tested for all the requirements (see **6**) given in this standard.

NOTE

Tests shall be carried out as prescribed in this standard.

E.6 Criteria for conformity

A lot shall be declared as conforming to the requirements of this specification if the following conditions are specified.

E.6.1 Each bag inspected as in **E.5.1** satisfies the relevant requirements.

E.6.2 The test results on each composite sample or the laboratory sample when tested as in

E.5.2 satisfy the relevant requirements.

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