

DRAFT TANZANIA STANDARD

Light kaolin – Specification

Draft for Stakeholders comments only

TANZANIA BUREAU OF STANDARDS

Foreword

This Draft Tanzania Standard is being developed by the Industrial and Laboratory Chemicals Technical Committee under supervision of the Chemicals Division Standards Committee and it is in accordance with the procedures of the Bureau.

This second edition cancels and replaces the first edition (TZS 506: 1996 *Light kaolin – Specification*) with changes on the test method for heavy metal.

In reporting the result of a test or analysis made in accordance with this Tanzania Standard, if the final value, observed or calculated is to be rounded off, it shall be done in accordance with TZS 4 *Rounding off numerical values*.

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Light kaolin – Specification

1. Scope

This Draft Tanzania Standard prescribes the requirements and the methods of sampling and testing of light kaolin for use in chemical industries. It does not apply for light kaolin used in pharmaceutical industry.

2. Normative references

The following referenced documents are indispensable for the application of this document. The latest edition of the referenced document (including any amendments) applies;

TZS 59 *Water for analytical laboratory use – Specification and test method*

TZS 74 *Sulphuric acid - Specification*

TZS 386 *Sampling of pure technical and analytical sodium hydroxide*

3. Terms and definitions

<<<<<Not applicable>>>>>

4. Requirements

4.1 General requirements

4.1.1 Grades

There shall be four grades of the material as follows

Grade 1 - suitable for textile and paper coating industries

Grade 2 - suitable for rubber Industry

Grade 3 - suitable as filler in the paper industry

Grade 4 - suitable as carrier in insecticide industry

4.1.2 Description

4.1.2.1 The material shall be the beneficiated natural mineral powder consisting essentially of hydrate aluminum silicate. It shall be dry and free from extraneous impurities and grit.

4.1.2.2 The material for use in textile and paper industries, the colour shall be white, and for that used in rubber and insecticides, the colour may be white or cream.

4.1.2.3 The material shall not contain any bluing or whitening substances.

4.1.2.4 For material required for textile industry shall conform to the following requirements;

- a) It shall disintegrate readily and form a smooth cream upon agitation when a lump

of it is placed in water;

- b) It shall be able to make a good fluid mixture when adhesive is added: and
- c) It shall not show any definite structure when viewed under the microscope and shall be free from feldspathic material.

4.2 Specific requirements

The material shall also comply with the requirements laid down in table 1, when tested in accordance with the methods prescribed in annex A.

Table 1 – Specific requirements for light kaolin

S/N	Characteristic	Requirements				Method of test
		Grade 1	Grade 2	Grade 3	Grade 4	
1.	Coarse particles or grit represented as residue on 53-micron sieve percent by mass, <i>max</i>	0.1	1.0	1.0	2.0	A.2
2.	Particles larger than 10 microns in diameter, percent by mass, <i>max</i>	5.0	7.0	20.0	20.0	A.3
3.	Particles smaller than 2 microns in diameter. per cent by mass, <i>min.</i>	62	50	35	35	A.4
4.	Relative density at 22/27 °C	2.5 – 2.9				A.5
5.	Loss on drying, percent by mass, <i>max</i>	6.0	1.0	5.0	6.0	A.6
6.	Loss on ignition, percent by Mass, <i>max</i>	14.0				A.7
7.	Matter soluble in water, percent by mass, <i>max</i>	N/A	0.5	0.5	N/A	A.8
8.	Matter soluble in hydrochloric acid, percent by mass, <i>max</i>	2.5				A.9
9.	Arsenic (as As ₂ O ₃), mg/kg, <i>max</i>	10	10	10	10	A.11
10.	pH value (of aqueous extract)	N/A	N/A	4.5 – 7.5	N/A	A.12
11.	Oil absorption, ml per 100 g, <i>min</i>	N/A	50	N/A	N/A	A.13
12.	Colour reflectance to blue light of wavelength 5 040 Å°, percent	80 - 85	As agreed between the purchaser and the supplier	85 min	N/A	A.14
Note: N/A = "Not applicable; stands for parameters not applicable for particular grade						

5 Packing and labelling

5.1 Packing

The material shall be supplied in sound, clean and dry containers and another requirement as agreed between the purchaser and the supplier.

5.2 Labelling

Each container shall legibly and indelibly label with the information below either in English or Kiswahili or in combination as agreed between the manufacturer and/or the supplier. Any other additional language is optional.

- a) Name and grade/use of the material
- b) Name of the manufacturer and recognized trademark, if any
- c) Net content
- d) Date of manufacture
- e) Batch/Lot identification
- f) Instructions for handling

6 Sampling

The method of drawing representative samples of the material from a lot, the number of tests to be performed and the manner of determining the criteria for conformity of the requirements of this specification shall be as prescribed in annex B.

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Annex A (Normative)

Methods of test for light kaolin

A.1 Quality of reagents

Unless specified otherwise, pure chemicals and distilled water (see TZS 59) shall be employed in tests).

A.2 Coarse particles or grit represented as residue on a 53-micron sieve

A.2.1 Reagents

Tetrasodium pyrophosphate solution - 1%(m/v)

A.2.2 Procedure

Weigh about 200 g of the material and add it to about 1 litre of water in a glass or plastic vessel which is capable of being closed with a cap or bung. The vessel shall be of such a capacity that it is not more than about two-thirds full. Add 50 ml of tetrasodium pyrophosphate solution (this makes the wetting and subsequent dispersal and sieving easier). Close it, shake briefly and allow to stand for about 30 min to allow the material to wet thoroughly. Mount the 53-micron sieve over a sink and pour the slurry carefully from the vessel into the sieve. The passage through the sieve is assisted by impingement of a stream of water directed by hand from a flexible tube attached to the water supply the stream also serves to wash out the last traces of solids from the vessel into the sieve. Wash the residue on the sieve until the washings are clear and contain no solid particles. and then transfer by washing into a porcelain dish. Evaporate the water and transfer the dry residue to a watch glass by brushing with a fine bristles brush

A.2.3 Calculation

$$\text{Coarse particles, percent by mass} = 100 \times \frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue obtained. and

M_2 = mass in g of the material taken for the test.

A.3 Particles larger than 10 microns in diameter

A.3.1 Outline of the method

The method prescribed is the beaker sedimentation method and is useful for measurements in the range 4 to 40 microns It is based on Stokes law which defines the constant velocity reached by a sphere falling without turbulence, through a viscous fluid:

$$V = \frac{GD^2(\Delta d)}{18\eta}$$

where

V = sedimentation velocity in cm/s

G = acceleration due to gravity in cm/sec²

D = diameter of sphere in cm

Δd = difference in density between the sphere and the fluid.

η = viscosity of the fluid in poises (for china clay and water is 1.65 g/m):

Note: η is temperature dependent so that if temperature is kept constant η , Δd and G are constants and V is proportional to the square of the diameter of the particles.

Using this relationship, it is possible to predict the depth of particles, starting in the free surface of the suspension, after a certain time has elapsed. Clay particles are not spherical and particles sizes are expressed as equivalent spherical diameters or stoke diameters

For this test it is Important that two conditions are met

- a) The clay shall be completely dispersed and deflocculated, and
- b) The concentration of the solids in the suspension should not exceed 5 percent by mass.

A.3.2 Reagents

Deflocculating solution - prepared by mixing 10 g of anhydrous sodium carbonate and 5 g of sodium hexametaphosphate in 1 litre of water.

A.3.3 Procedure

Take a glass cylinder having approximate dimensions of 5 cm internal diameter and height of 30 cm, and paint or inscribe a meniscus mark at a height of 20 cm above the internal base. Place into this cylinder 20 ml of the deflocculating solution and 20.0 g of dry sample. Add water to the mark. Set aside the cylinder for about one hour after which close it with a rubber bung and shake vigorously in order to disperse the clay; Place the cylinder on the bench and note the time. After 40 minutes, decant the liquid carefully from the residue (a better alternative is to remove the liquid with a baffled Siphon tube connected to a water jet vacuum pump; this technique leaves the residue and about 1 cm of liquor above it undisturbed). Fill the cylinder again to the mark with water and 5 ml of the deflocculating solution, shake to suspend the particles, and after a further 40 minutes, remove the liquid again. Repeat this process twice to make a total of four sedimentations and decantation.

Wash out the final residue with water into a porcelain dish and dry. Transfer the dry residue with a brush to a watch glass and weigh. This mass in g multiplied by 5 gives the percentage by mass of particles coarser than 10 microns.

NOTE - Strictly these conditions apply to a temperature 290K (16.85 °C) but for coarse particles, temperature fluctuations give rise to comparatively small errors and thermostatic water baths are not essential. Corrections for ambient temperature are given below

Temperature (273°K)	Time at 20 cm depth (minutes)
14	43.75
16	41.5
19	38.5
20	37.5
22	35.75

A.4 Particles smaller than 2 microns in diameter

A.4.1 *Outlines of the method*

The method prescribed is the pipette Andersen method, and is useful for measurements in the range 0.5 to 4 microns. It is based on Stokes law and the principle is the same as described for the beaker method in A.3 1

A.4.2 *Apparatus*

The apparatus consists of a thermostatic water bath, glass cylinders, tarred glass sample dishes and a special pipette. The latter is of 20 ml capacity and is mounted in a holder adjustable in height and in two directions in a horizontal place. Thus, a number of cylinders may be sampled in turn without disturbing their contents. The pipette holder also cranes a probe which when brought into the meniscus of suspensions would dictate the depth of the

pipette tip below the meniscus. Large vertical movements of the pipette are achieved by means of a sliding clamp and fine movements by means of a rack and pinion gear. The pipette has two vacuum services from a single pump which enable samples to be taken either rapidly or slowly. For slow sampling, the filling time shall not be less than 15 seconds

A.4.3 Reagent

Deflocculant solution - prepared by mixing 10 g of anhydrous sodium carbonate and 5 g of sodium hexametaphosphate in 1 litre of water.

A.4.4 Procedure

Take about 5 g of material which need not be dried or weighed accurately, and add to 5 ml of the deflocculant solution in a cylinder of diameter 5 cm to 6 cm. Add water to a total volume of about 250 ml and place the cylinder in a water bath whose liquid depth is such that no part of the suspension column is exposed.

A.4.4.1 Allow a period of one hour to elapse to equalize temperatures and to allow the clay to wet thoroughly. Remove the cylinder, close with a bung and shake vigorously to disperse the soaked clay lumps. Replace the cylinder in the bath and immediately sample with the pipette regardless of the pipette depth. Discharge the 20 ml sample into a tarred glass dish which is placed on a water bath or suitable hot plate and evaporate its contents to dryness without boiling. The mass of the clay from the sample is found to the nearest milligram (A).

A.4.4.2 If the temperature of the bath is 297° K(23.85 °C). Withdraw a further sample slowly 3 hours 39 minutes after the first sample and for this, the pipette is lowered carefully into the liquid to avoid disturbing it. By means of tube globe index, take the sample from a depth of 5.0 cm below the free surface. The mass of solids in this second sample is determined as before (B).

A.4.5 Calculation

Mass B expressed as a percentage of A gives the percentage by mass of particles finer than 2 microns. A small correction shall be applied because the soluble salts of the deflocculant solution are counted erroneously as clay. An empirical correction C shall be applied by running a blank determination on a cylinder made up as usual but containing no clay. The mass of dissolved solids so obtained can be subtracted from A and B.

$$\text{Particles smaller than 2 microns, percent by mass} = \frac{B-C}{A-C} \times 100$$

NOTE - The temperature of 297°K(23.85 °C) has been chosen in the above because the control of temperature is facilitated. If the bath temperature is higher than all likely laboratory ambient temperatures. The times which should elapse between the two samplings at various temperatures for 5 cm depth are given below

Temperature (°K)	Time	
	Hours	Minutes
283	5	14
285	4	56
287	4	40
289	4	26
291	4	13
293	4	10
295	3	49
297	3	39

A.5 Relative density

A.5.1 Apparatus

A.5.1.1 Relative density bottle - 50 ml capacity, having a ground-in capillary stopper

A.5.1.2 Vacuum pump - with vacuum desiccators, capable of giving a vacuum of 10 mm

A.5.2 Procedure

Weigh the empty, dry relative density bottle. Introduce about 10 g of the material as dried in A.6 and weigh again. Nearly fill the relative density bottle with water and do not insert the stopper; Place it on a small tarred evaporating dish and keep the assembly in the exhausted receiver of the vacuum pump overnight (at least 16 h). Fill up the relative density bottle carefully with water and insert the stopper. It is possible that a very small portion of the sample will run out of the sides of the stopper on inserting it. Wash down the particles thus forced out into the tarred dish. Wipe off the excess of water from the relative density bottle, bring it to 300 °K (26.85 °C) and weigh.

A.5.2.1 Evaporate the contents of the tarred dish on a boiling water bath under cover, dry to constant mass at 378°K (104.85 °C) to 383°K (109.85 °C)

A.5.2.2 Empty the bottle, fill it with water and weigh after bringing it to 300°K (26.85 °C)

A.5.3 Calculation

$$\text{Relative Density at } 300/300^{\circ}\text{K} = \frac{M_2 - M_1 - M_4}{(M_5 - M_1) - (M_3 - M_2) - M_4}$$

Where,

M_1 = mass in g of the empty relative density bottle;

M_2 = mass in g of relative density bottle and sample;

M_3 = mass in g of relative density bottle with sample and water;

M_4 = mass in g of residue in tarred dish; and

M_5 = mass in g of relative density bottle and water.

A.6 Loss on drying

A.6.1 Procedure

Weigh about 20 g of the material and heat in a squat weighing bottle at 378°K to 383°K. Cool and weigh till constant mass is obtained. Preserve the sample in desiccators for subsequent tests.

A.6.2 Calculation

$$\text{Loss on drying, percent by mass} = \frac{100 M}{M_1}$$

Where,

M = loss in mass in g, and

M_1 = mass in g of the material taken for the test

A.7 Loss on ignition

A.7.1 Procedure

Weigh about 1 g of the material in a porcelain or silica crucible ignite at about 1273°K (999.85 °C)
Cool and weigh till constant mass is obtained

A.7.2 Calculation

Loss on Ignition, percent by mass = $\frac{100 M}{M_1}$

Where,

M = loss in mass in g, and

M_1 = mass in g of the material taken for the test

A.8 Matter soluble in water

A.8.1 Reagents

A.8.1.1 Rectified spirit – 95% ethanol

A.8.1.2 Bromophenol blue indicator solution - Dissolve 0.1 g of bromophenol blue in 100 ml of rectified spirit

A.8.1.3 Dilute hydrochloric acid - 0.1 mole/litre.

A.8.2 Procedure

Weigh about 10 g of the material in a 250 ml beaker. Add 5 ml of rectified spirit to wet the sample thoroughly. Add 200 ml of water boil the suspension for 5 minutes and allow cooling to room temperature. Add sufficient bromophenol blue indicator to give a visible colour, and then add dilute hydrochloric acid until the blue colour disappears.

NOTE - Bromophenol blue turns yellow at about pH 4 at which point flocculation occurs and clears filtration is obtained.

Transfer the contents of the beaker to a 250 ml volumetric flask, dilute to the mark with water and mix well by shaking. Filter through a filter paper, rejecting the first 50 ml of filtrate. Place 100 ml of the clear filtrate into a tared porcelain dish and evaporate to dryness on a water bath. Dry the residue at 378°K \pm 2°K, cool and weigh till constant mass is obtained.

A.8.3 Calculation

Matter soluble in water, percent by mass = $250 \times \frac{M_1}{M}$

where

M_1 = mass in g of the residue obtained, and
 M = mass in g of the material taken for the test

A.9 Matter soluble in hydrochloric acid

A.9.1 Reagent

Dilute hydrochloric acid - 0.2 mole/litre

A.9.2 Procedure

Weigh about 2 g of the material. Transfer to a round-bottom flask with 100 ml of hydrochloric acid. Fix up a reflux condenser and boil the material on a sand bath for 5 minutes. Cool and filter through a sintered glass crucible No. G4. Wash free from chlorides and evaporate the filtrate to dryness on a water-bath in a silica basin. After gentle heating, cool and weigh to constant mass.

A.9.3 Calculation

Matter soluble in hydrochloric acid, percent by mass = $\frac{100 M}{M_1}$

where

M = mass in g of residue, and
 M_1 = mass in g of the material taken for the test

A.10 Copper

A.10.1 Outline of the method

Copper is determined calorimetrically using sodium diethyldithiocarbamate by visual comparison.

A.10.2 Apparatus

Nessler cylinders - 100 ml capacity

A.10.3 Reagents

A.10.3.1 Concentrated ammonium hydroxide- 18 moles/litre (relative density 0.90)

A.10.3.2 Ammonium Citrate solution - Dissolve 100 g of citric acid in 100 ml of ammonium hydroxide and make up to 200 ml with water.

A.10.3.3 Gum Arabic solution - 5 percent (m/v).

A.10.3.4 Sodium diethyldithiocarbamate solution

Dissolve 10 g of sodium diethyldithiocarbamate in 1000 ml of copper-free water; keep in an amber-coloured bottle and protect from strong light.

A.10.3.5 Standard copper solution - Dissolve 0.3928 g of copper sulphate crystals ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in copper-free water and make up the volume of 1000 ml with copper-free water. Take 100 ml of this solution and further dilute to 1 000 ml with copper-free water. One

milliliter of this solution is equivalent to 0.01 mg of copper oxide (as CuO)

A.10.4 Procedure

A.10.4.1 Prepare sample solution - Weigh accurately 2.0 g of the dry sample and mix with 8 g of anhydrous sodium carbonate and fuse in a platinum crucible until the melt is clear. Leach out the melted mass with water into a porcelain dish and then put the crucible in the same dish. Add about 20 ml of water and heat on a water-bath until the product is disintegrated. Remove the crucible after washing in the dish. Evaporate the contents of the dish to dryness. Add a mixture of 12 ml of concentrated sulphuric acid (see TZS 74, see clause 2) and 2 ml of concentrated nitric acid. Evaporate till fumes of sulphur trioxide appear. Dilute to exactly 500 ml and filter through a filter paper to remove silica. Use this prepared sample solution for the test under A. 13.

NOTE - In case the prepared sample solution is turbid, add few drops of ferric chloride solution (5% (m/v), boil and precipitate twice with ammonium hydroxide solution, and filter.

A.10.4.2 Pipette 25 ml of the prepared sample solution into a small conical flask or beaker; Drop into it a small piece of litmus paper and make the solution just alkaline with ammonium hydroxide. Add 2.5 ml of ammonium hydroxide in excess and heat to boiling. Allow to stand on a water-bath for one hour to ensure complete precipitation of aluminum hydroxide and then filter through filter paper (Whatman No.1 or equivalent) into a Nessler cylinder. Wash the filter paper with two or three small portions of hot water; to the solution in the Nessler cylinder, add 5 ml of ammonium citrate, 5 ml of gum Arabic solution, 10 ml of ammonium hydroxide solution and 10 ml of sodium diethyldithiocarbamate solution in the order mentioned. Dilute to 100 ml mark and mix thoroughly. To the other Nessler cylinder containing an equal aliquot of blank solution carried through the entire analysis in the same manner as the prepared sample solution, add equal amounts of the same reagents, dilute to about 90 ml and mix.

Add to this solution standard copper solution from a 10-ml burette until its colour matches that of the material under test after diluting to the same volume. Mix thoroughly after each addition of standard copper solution

If the colour produced with the prepared sample solution is too deep for comparison, a smaller aliquot of the prepared sample solution from the acid digestion should be used.

A.1 0.5 Calculation

$$\text{Copper (as CuO) percent by mass} = \frac{0.02 V}{M}$$

where

V = Volume in ml of standard copper solution used for the blank; and
 M = mass in g of the dry material taken for the test.

A.11 Arsenic

A.11.1 Sample preparation by microwave digestion method

A.11. 1.2 Reagents

A.11.1.2.1 Conc. Hydrochloric acid (HCl)

A.11.1.2.2 Conc. Nitric acid (HNO₃)

A.11.1.2.3 Conc. Hydrofluoric acid (HF)

A.11.2.3 Procedure

Weigh 0.5 g of the sample into the digestion vessel. Add 3 mL of HCl, 1 mL of HNO₃, and 7 mL of HF. Swirl the mixture gently and wait approximately 15 minutes before closing the vessel.

. Allow any initial reaction to subside before sealing vessel.

- Note:
- 1- This procedure uses Hydrofluoric acid; If it is necessary to complex the residual, Hydrofluoric acid or re-dissolve insoluble fluorides formed, an additional complexation step with boric acid should be used.
 - 2- HF should be added slowly and carefully to the sample
 - 3- The control test should be treated the same as the sample.

A.11.2 Determination

Determine the heavy metal (i.e. Arsenic) content using AAS or ICP methods.

A.12 pH value

Procedure

Weigh about 25 g of the material and transfer it to a 500 ml beaker. Add 100ml of freshly boiled and cooled water. Allow to stand for 30 minutes with occasional stirring. Filter, reject the first 50 ml of the filtrate and collect the remaining filtrate in a beaker. Determine the pH of the solution by means of a suitable pH meter using glass electrodes.

A.13 Oil absorption

A.13.1 Procedure

Place about 2 g of dry material, accurately weighed on a glazed porcelain, ground glass or marble plate. Add linseed oil from a weighed dropping bottle, drop by drop and regularly each drop being mixed thoroughly with the material using a palette knife. Mix the oil thoroughly in the course of 20 minutes into the whole of the material with the palette knife, until a coherent mass is obtained. Weigh the dropping bottle again and determine by difference the weight of oil in grams. Where an approved sample is used for comparison, the oil absorption of the approved sample shall be determined by the same person and at the same time.

A.13.2 Calculation

$$\text{Oil absorption, ml per 100 g} = \frac{108 M}{M_1}$$

where

M = mass in g of the linseed oil absorbed; and

M_1 = mass in g of the dry material taken for the test.

A.14 Colour (reflectance to blue light of wavelength 0.504 μm) 5040 A

A.14.1 Outline of the method

The colour of the sample is compared with that of Lovibond magnesium Oxide block.

A.14.2 Procedure

By means of a suitable arrangement, light falling on a Lovibond magnesium oxide block shall be reflected on a selenium cell connected to a galvanometer. The galvanometric deflection is proportional to light reflected. For comparison, the reading obtained by magnesium oxide block is taken as 100.

Substitute the magnesium oxide block with a block prepared from a sample of the material. Measure the reflectance and report as colour (or reflectance) compared to that of magnesium oxide as 100.

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Annex B (Normative)

Sampling of light kaolin

B.1 General Requirements of sampling

In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed

B.1.1 Samples shall not be taken in an exposed place.

B.1.2 Sampling instrument shall be clean and dry when used.

B.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from contamination.

B.1.4 The sample shall be placed in clean, dry, air-tight glass or other suitable containers.

B.1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B.1.6 Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sample; the date of sampling and other identification particulars.

B.1.7 The samples shall be stored in such a manner that during storage the properties of the material do not get affected.

B.1.8 The sampling shall be done by a person or persons mutually agreed to between the purchaser and the supplier.

B.2 Scale of sampling for material packed in bags

B.2.1 Lot - In a single consignment all the material belonging to the same grade and coming from the same supplier or source shall constitute a lot.

B.2.2 Sub-lot - For the purpose of sampling, all the material in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal weight in accordance with columns 1 and 2 of Table 2.

B.2.3 The number of bags to be sampled from each sub-lot is given in column 3 of table 3. These bags shall be selected at random from the sub-lot. In order to ensure the randomness of selection, random number tables shall be used. For guidance and use of random number tables, TZS 386 may be referred. In the absence of random number tables, the following procedure may be adopted:

Starting from any bag in the lot, count them as 1. 2. 3, etc, up to r and so on. Every r^{th} container thus counted shall be withdrawn, r being the integral part of N/n , where N is the total number of containers in the lot, and n the number of containers to be chosen.

Table 2 - Scale of sampling for bags - (clauses B.2.2 and B.2.3)

Number of bags in the lot	Number of sub-lots	Number of bags to be sampled from each sub-lot
Up to 10	1	5
11 to 200	2	10
201 to 500	3	15
501 to 2 000	4	20
2 001 and above	5	-

B.3 Sampling from wagons

B.3.1 Lot - In a single consignment all the material belonging to the same grade and coming from the same supplier or source shall constitute a lot.

B.3.2 Sub-lot - For the purpose of sampling all the wagons in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal weight in accordance with Table 3.

Table 3 - Scale of sampling for wagons

Weight of the material tonnes	Number of sub-lots	Number of increments from each sub-lot
Up to 200	2	20
201 to 500	3	30
501 to 2 000	4	40
2 001 and above	5	50

B.3.3 A minimum of 25 percent of wagons shall be selected at random from the sub-lot. The corresponding number of increments to be taken from a sub-lot shall be distributed over the selected wagons with a view to determining the number of increments that should be taken from each of the selected wagons in the sub-lot

B.3.4 All the increments taken from the selected wagons in a sub-lot shall be pooled together to constitute a gross sample.

B.4 Preparation of samples

B.4.1 All the Increments from the same sub-lot shall be thoroughly mixed to constitute a gross sample representing the sub-lot the minimum size of the composite sample/gross sample shall be about 3 kg. If the gross sample is less than this, additional number of increments/portions shall be drawn so as to make up the required quantity.

B.4.2 The composite sample/gross sample shall be suitably reduced by the method of coning and quartering or any other suitable method to about 750 g which shall be divided into three portions called laboratory sample, one for the purchaser, another for the supplier and the third as the reference sample, each duly placed in the sample container.

B.5 Number of tests and criteria for conformity

B.5.1 One laboratory sample from each sub-lot shall be tested individually for all the requirements of this specification and the sub-lot shall be considered satisfactory If the laboratory sample satisfies all the requirements of this specification

B.5.2 The lot shall be declared to conform to the requirements of the specification if all the sub-lots (see B.5.1) are found satisfactory.