PRODUCT MANUAL FOR MICROFINE ORDINARY PORTLAND CEMENT ACCORDING TO IS 16993: 2018

This Product Manual shall be used as reference material by all Regional/Branch Offices & licensees to ensure coherence of practice and transparency in operation of certification under Scheme-I of Bureau of Indian Standards (Conformity Assessment) Regulations, 2018 for various products. The document may also be used by prospective applicants desirous of obtaining BIS certification licence/certificate.

1	**Product**	:	IS 16993 : 2018
	Title	:	MICROFINE ORDINARY PORTLAND CEMENT
	No. of Amendment	:	1
2	**Sampling Guidelines:**		
a)	**Raw material**	:	Clinker – IS 16353 : 2016 Ordinary Portland Cement – IS 269 : 2015
b)	**Grouping guidelines**	:	Please refer ANNEX – A
c)	**Sample Size**	:	For Physical test – 9 kg, For Chemical test – 1 kg
3	**List of Test Equipment**	:	Please refer ANNEX – B
4	**Scheme of Inspection and Testing**	:	Please refer ANNEX – C
5	**Possible tests in a day:**		
	a) Loss of ignition (Clause 5.1, Table 1) b) Insoluble residue (Clause 5.1, Table 1) c) Particle size (Clause 6, Table 2) d) Setting time (Clause 6, Table 2) e) Bleeding test at 3 h from the time of mixing (Clause 6, Table 2)		
6	**Scope of the licence :**		
	"Licence is granted to use Standard Mark on Microfine Ordinary Portland Cement and /or Microfine Sulphate Resisting Portland cement as per IS 16993 : 2018"		

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ANNEX - A

GROUPING GUIDELINES

1. IS 16993: 2008 covers the requirements for Microfine Cement (Microfine Ordinary Portland Cement as well as Microfine Sulphate Resisting Portland Cement).

2. The additional requirements for Microfine Sulphate Resisting Portland Cement are also given in Table 1under clause 5.1.

3. Considering the above, following grouping guidelines is developed for GoL/CSoL:

   a) Sample of each type of Microfine Cement, i.e Microfine Ordinary Portland Cement and Microfine Sulphate Resisting Portland Cement shall be drawn to cover that variety in the scope of the licence.

   b) However, if sample of Microfine Sulphate Resisting Portland Cement is tested for all requirements, then Microfine Portland cement may also be covered in the scope of the licence.

4. The firm shall declare the variety they intend to cover in the licence. The scope of the Licence may be restricted on the Manufacturing and Testing capabilities of the Manufacturer.

5. During the operation of the licence, BO shall ensure that all the varieties covered in the Licence are tested in rotation to the extent possible.

ANNEX- B
List of Test Equipment

Major test equipment required to test as per the Indian Standard

S. No.	Tests used in with Clause Reference	Test Equipment
1	Fineness (BET Method- Nitrogen absorption) Clause 6, Table 2	Apparatus as per clause 4.1 and Fig 2 of IS 11578 : 1986
2	Particle size Clause 6 Table 2	Laser diffraction PSD analyser
3	Setting time Clause 6, Table 2	Vicat apparatus
		Vicat moulds
		Consistency needle
		Initial and Final setting time needles
4	Bleeding test at 3 h from the time of mixing Clause 6, Table 2	Marsh funnel as per Fig of IS 16993 : 2018
		Measuring jars and measuring cylinders
		Stop watch
		Steel scale
		Rotational viscometer
5	Compressive strength Clause 6 Table 2	Vibration machine with timer & cube mould fitting assembly 12000 ± 400 vibration per min.
		Compressive Strength machine
		Poking Rod , Petroleum Jelly
		Planetary mixer
		Jolting apparatus
		Scraper and demoulding device
		Proving ring with all accessories suitable for calibration of CST machine
		Tachometer
		Moulds as per 4.2 of IS 10078 (Prism moulds)
		Gauging trowel (210 ± 10 g) Gauging plate- stainless steel(non-perforated)
		Standard sand (as per IS 650)
		Curing tank of appropriate size with water circulation arrangement
		Graduated glass cylinders 150 to 200 ml
		Humidity chamber with temperature & RH Control 27 ± 2° C, RH 90 to 100 %
6	**IS 16993 : 2018**	**General Equipments for Cement testing**
	To control humidity & temperature in lab	1. Humidity chamber with temperature & RH control 27 ± 2 °C, RH 90 to 100 % 2. Suitable arrangement to demonstrate maintenance of temperature of 27 ± 2 ° C & RH 65 ± 5% constantly

	For cement Sampling cement	Mixing trays – Adequate size including trays of 24 partitions for keeping hourly samples
	To measure temperature	Thermometers
	Lab ball mill (motorized)	To grind the clinker and gypsum sample in lab ball mill for testing
	To weigh the material	Platform type balance Electrical balance Weight box with weights (1 mg - 500 g)
7	**General test equipments for chemical testing Clause 5.1 , Table 1 of IS 16993 : 2018**	Heater and hot plate
		Muffle furnace with thermostatic control
		Wet and dry bulb assembly/humidity meter
		Thermometer
		Distillation Assembly
		Crucible: Platinum or Porcelain / silica
		Filter paper (No- 1, 40, 41, 42)
		Desiccators with cover & Desiccant
		Water bath
		Oven
		Sulphide Sulphur apparatus
		Flame photometer
		Bunsen burner
		Standard cement samples
		pH meter/paper
		Glassware - volumetric flask -0-250 ml, beaker 0-250 ml, measuring cylinder 0-50,100, 500, 1000 ml, burette 0-25/50 ml, conical flasks- 0-250 ml, pipette 0-5,10, 25, 50 ml Erlenmeyer flask Gas generating flask, etc
		All chemicals required for complete chemical analysis of ultrafine ground granulated blast furnace slag testing as per IS 4032
		Tongs including platinum tipped tong
		Wire gauge with asbestos sheet at the middle
		Washing bottle

8	**Testing of clinker and Ordinary Portland Cement (Clause 4 of IS 16993 : 2018)**	
	Fineness	Blaine's apparatus variable flow type with permeability cell and perforated metal disc
		Stop watch with start-stop mechanism
		Mercury for calibration
		Balance, Standard weights
		Standard Cement
		Manometer liquid (di-butyl phthalate or light mineral oil.)
		Mercury of reagent grade or better, Pyknometer
		Circular discs of filter paper of medium porosity (mean pore diameter 7 μ).
		Le-Chatelier's flask
		Constant temperature water bath to maintain temp. within ± 0.1 ° C
	Soundness by Autoclave	Auto clave machine with thermostatic control to maintain pressure of 2.1 MPa for 3 hrs, pressure to be attained within 1-1 ¼ hrs; L-Shape thermometer LC 1 °C
		Pressure gauge 0-42 kg/cm^2 LC = 0.4 kg/cm^2
		Humidity chamber with temperature & RH control 27 ± 2 °C, RH 90 to 100 %
		Standard bar 308 mm, max
		Bar moulds 25 x 25 x 282 mm
		Length comparator with dial gauge
		Mineral oil for covering moulds
	Soundness by Le-chatelier Method	Le-Chatelier's water bath preferably with thermostatic control raising temperature from 27 ± 2 ^{0} C to boiling in 27 ± 3 minutes
		Le-Chatelier's moulds with weights and cover glasses minimum 8 nos.
		Humidity chamber with temperature & RH control 27 ± 2 ^{0} C, RH 90 to 100 %
		Steel scale 12" (304.8 mm)
	Setting time	Vicat apparatus
		Needle for Consistency, IST& FST testing
		Moulds
		Stop Watch
		Balance – 1000 g ± 0.1 g and Standard Weights 1 mg to 500 gm
		Gauging trowel of weight 210 ± 10 g
	Compressive Strength	Vibration machine with timer & cube mould fitting assembly 12000 ± 400 vibration per min.
		Compressive Strength machine
		Poking Rod , Petroleum Jelly
		Proving ring with all accessories suitable for calibration of CST machine

		Tachometer
		Cube Moulds 70.6 × 70.6 mm, Poking rod
		Gauging trowel (210 ± 10 g) gauging plate, stainless steel(non-perforated)
		Standard sand (as per IS 650)
		Curing tank of appropriate size with water circulation arrangement
		Graduated glass cylinders 150 to 200 ml
		Humidity chamber with temperature & RH Control 27 ± 2° C, RH 90 to 100 %
	Transverse Strength Test	Moulds
		Planetary Mixer, Standard Sand
		Jolting Apparatus, Scraper, Demolding device as per IS 4031 (Part 8)

The above list is indicative only and may not be treated as exhaustive.

ANNEX - C

Scheme of Inspection and Testing

1. LABORATORY - A laboratory shall be maintained which shall be suitably equipped (as per the requirement given in column 2 of Table 1) and staffed, where different tests given in the specification shall be carried out in accordance with the methods given in the specification.

1.1 The manufacturer shall prepare a calibration plan for the test equipment. However calibration of following test equipments shall be carried out at a frequency shown against each and record of same shall be kept.

Sl. No.	TEST EQUIPMENT	FREQUENCY OF CALIBRATION
1	Laser diffraction PSD analyser	Daily with licensee's own Standard cement sample and monthly with standard cement samples supplied by NCCBM.
2	Compressive strength Testing machine	Once in a month with Licensee's own Proving Ring and the Proving Ring shall be Calibrated once in two years from a NPL/NABL Accredited Calibrating body or NPL or NPL accredited Proving Ring manufacturer.
3	Vibration machine	Once in a month by licensee's own Tachometer. The tachometer shall be calibrated once in a year from NPL/NABL accredited outside agency.

2. TEST RECORDS – The manufacturer shall maintain test records for the tests carried out to establish conformity.

3. LABELLING AND MARKING – As per the requirements of IS 16993 : 2018

4. CONTROL UNIT – Entire quantity of Microfine Ordinary Portland Cement produced in a week shall constitute a control unit.

5. **LEVELS OF CONTROL -** The tests as indicated in column 1 of Table 1 and the levels of control in column 3 of Table 1, shall be carried out on the whole production of the factory which is covered by this plan and appropriate records maintained in accordance with paragraph 2 above.

5.1 All the production which conforms to the Indian Standard and covered by the licence should be marked with Standard Mark.

5.2 PACKING – Packing of Microfine Ordinary Portland Cement shall be done as per clause 9 of IS 16993 : 2018.

5.3 WEIGHMENT – One filled bag from each nozzle shall be taken at random twice in each shift of operation and weight checked in case of electronic packers with recorders. In all other

cases one filled bag from each nozzle shall be checked once in two hours. The bag shall be so chosen for weighment such that bags from each nozzle are taken for weighment. The weighing and packing machines shall be adjusted as and when necessary in such a way that net quantity of each bag shall be in accordance with the tolerances given clause 9.1.1 of IS 16993 : 2018. Such adjustments for each nozzle shall be recorded.

5.3.1 For packing of Microfine Ordinary Portland Cement in bulk cement terminal weighment of hourly check of mass of drums also shall be done in addition to weighment of bags mentioned in para 5.3 above. The records of weighments shall be maintained.

6. REJECTIONS – Disposal of non-conforming product shall be done in such a way so as to ensure that there is no violation of provisions of BIS Act, 2016.

TABLE 1

(1)				(2)	(3)			
Test Details				Test equipment requirement R: required (or) S: Sub-contracting permitted	Levels of Control			
Cl.	Requirement	Test Methods			Number of sample	Frequency		Remarks
		Clause	Reference			Grinding stage	Packing stage	
4	**Manufacture**							
	Clinker	4	IS 16353	S	One	One sample form each lot received shall be tested for assessment of conformity. Further testing is not required if lot is received is ISI marked.		
	Ordinary Portland cement	4	IS 269	S	One	Further testing is not required if lot is received is with manufacturers test certificate or ISI marked.		
5.1 & Table 1	**Chemical requirements**							
i)	$\frac{CaO + 0.7\,SO_3}{2.8\,SiO_2 + 1.2\,Ai_2O_3 + 0.65Fe_2O_3}$	5.1	IS 4032	R	One	Daily Composite sample	Weekly composite sample	-
ii)	Ratio of percentage of alumina to that of iron oxide	5.1	IS 4032	R	One	Daily Composite sample	Weekly composite sample	-
iii)	Insoluble residue	5.1	IS 4032	R	One	Daily Composite sample	Weekly composite sample	-
iv)	Magnesia	5.1	IS 4032	R	One	Daily Composite sample	Weekly composite sample	-
v)	Total Sulphur as SO_3	5.1	IS 4032	R	One	Daily Composite sample	Weekly composite sample	-
vi)	Loss on ignition	5.1	IS 4032	R	One	Daily Composite sample	Weekly composite sample	-
vii)	Chloride content	5.1	IS 4032	R	One	-	Weekly composite sample	Additional sample may be tested in case of change of source of raw material.
viii)	Alkali content	5.1	IS 4032	R	One	Daily Composite sample	Weekly composite sample	As per mutual agreement between purchaser and supplier
ix)	**Additional requirements for microfine sulphate resisting Portland cement**							
a)	Tricalcium aluminate (C_3A)	5.1	IS 4032	R	One	Daily Composite	Weekly composite	-
b)	C_2AF+2C_2A	5.1	IS 4032	R	One	Daily Composite	Weekly composite	-

6 & Table 2	**Physical requirements**							
i)	Fineness BET Method	6	IS 11578	S	-	As per agreement between the purchaser and manufacturer		
ii)	Particle size , µm a) D_{50} b) D_{95}	6	Laser diffraction PSD analyser	R	One	Hourly sample	Daily composite sample	-
iii)	Setting time	6 and Annex-B	IS 16693	R	One	Daily Composite sample	Weekly Composite sample	-
iv)	Bleeding test at 3 h from the time of mixing	6 and Annex-C	IS 16693	R	One	One sample per shift (Composite sample)	Daily Composite sample	-
v)	Compressive Strength	6	IS 4031 (Part 8)	R	One	Daily Composite sample	Weekly Composite sample	-

Note-1: Composite sample shall be made out of hourly samples for the required period (Pl see IS 3535 Methods of sampling hydraulic cements). If slag is manufactured using same raw materials from more than one grinding mill, sample from each mill shall be tested for fineness as per the above table. For all other parameters composite samples from all the mills shall be tested. If slag is manufactured using different source of raw materials from more than one grinding mill, sample from each mill shall be tested for all requirements as per the above table.

Note 2 : For manufacturing units where there is no packing silo and slag is packed directly from grinding mill, the frequency of tests specified for grinding stage would apply for the various tests to be carried out on samples taken from packing spouts along with weekly chloride content test.

Note- 3 : Sub-contracting is permitted to a laboratory recognized by the Bureau or Government laboratories empanelled by the Bureau.

Note- 4 : Levels of control given in column 3 are only recommendatory in nature. The manufacturer may define the control unit/batch/lot and submit his own levels of control in column 3 with proper justification for approval by BO Head.