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Indian Standard

SPECIFICATION FOR PHOSPHATE TREATMENT OF IRON AND STEEL FOR PROTECTION AGAINST CORROSION

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG

NEW DELHI 110002

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Gr 4

Indian Standard

SPECIFICATION FOR PHOSPHATE TREATMENT OF IRON AND STEEL FOR PROTECTION AGAINST CORROSION

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SPECIFICATION FOR PHOSPHATE TREATMENT OF IRON AND STEEL FOR PROTECTION AGAINST CORROSION

$\mathbf{0.} \quad \mathbf{FOREWORD}$

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 May 1966, after the draft finalized by the Metal Standards Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Considerable number of different phosphating processes are now in use and the wide variety of purposes for which they are employed is very large. The present standard has been limited to phosphate treatments for the protection of iron and steel against corrosion, as used for general purposes of industry.

0.3 In the formulation of this standard due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country. This has been met by deriving assistance from the following publications:

- B. S. 1391 : 1952 Performance tests for protective schemes used in the protection of light-gauge steel and wrought iron against corrosion. British Standards Institution.
- B. S. 3189 : 1959 Phosphate treatment of iron and steel for protection against corrosion. British Standards Institution.
- UNITED KINGDOM. MINISTRY OF DEFENCE. DEF-29 Phosphate treatment of iron and steel for protection against corrosion. 1965. Her Majestey's Stationery Office. London.
- Steel structures painting manual, Vol 2. Systems and specifications. 1955. Steel Structures Painting Council, Pittsburgh.
- Metal finishing, guide book directory. 1961. Metals and Plastic Publication Inc, New Jersey.

1. SCOPE

1.1 This standard covers the processing of iron and steel to produce coatings consisting essentially of metal phosphates that are intended to be used in conjunction with organic coatings for protection against corrosion. The standard also covers methods of testing finished coatings. It covers five classes of phosphating processes (both unaccelerted and accelerted as defined in 2.1 to 2.6) which are designated in terms of compositions and weights of the coatings (see 3).

1.1.1 This standard is intended for general purposes of industry and does not cover treatment of ferrous surfaces for deep drawing nor does it deal with phosphating treatment on zinc, cadmium or aluminium for passivation purpose.

1.2 Certain phosphating processes, particularly heavy classes, are likely to alter the dimensions of the components and due allowance for this should be given in the design stage or in selecting the particular phosphating process.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Unaccelerated Process — A process in which the phosphating solution is of the metal phosphate or phosphoric acid type without additions that accelerate the coating formation as given in **2.2**.

2.2 Accelerated Process — A process in which the phosphating solution is of the metal phosphate or phosphoric acid type but contains additions that accelerate the coating formation, such as water-soluble oxidizing agents, as for example, nitrates, nitrites, chlorates and/or hydrogen peroxide.

2.3 Organic Coating — A coating of drying or non-drying oil, grease, lacquer, varnish, paint or any other organic material applied after phosphating to complete the protective scheme.

2.4 Pointage — A conventional industrial measure of the strength of a phosphating solution. The pointage of a phosphating bath is the number of millilitre of N/10 sodium hydroxide solution (4.0 g NaOH per litre) required to neutralize 10 ml of the phosphating solution, using phenolphthalein as indicator.

2.5 Sealing — It is the application of any organic coating to a phosphated surface.

2.6 Staining — It is the application of a colour or dye, dissolved in water or spirit, so as to impregnate a phosphated surface uniformly. Water staining will give limited corrosion resistance property, while

the spirit stain will enhance corrosion resistance due to the film of shellac deposited on the phosphated surface.

3. CLASSIFICATION OF PHOSPHATING PROCESS

3.1 Phosphating processes designated in terms of the compositions and weights of the coatings are classified as follows.

3.1.1 Class Al (Heavy Weight) — processes giving coatings that consist essentially of manganese and/or iron phosphate and have a weight not less than 7.5 g/m^2 of treated surface. This class is normally used where maximum protection is desired, particularly under oil or grease or where resistance to wear is required.

3.1.2 Class A2 (Heavy Weight) — processes giving coatings that consist essentially of zinc phosphate and have a weight not less than 7.5 g/m^a of treated surface. This class is normally used for similar purpose as Class A1 (see 3.1.1) where a good degree of protection is required, coupled with a more rapid speed of processing.

3.1.3 Class B (Medium Weight) — processes giving coatings that consist essentially of zinc or other metal phosphates and have a minimum weight 4.3 g/m^2 of treated surface. This class is normally used for general protective purposes under paint coatings, oil or grease where less severe duty than in heavy weight processes (see 3.1.1 and 3.1.2) is required.

3.1.4 Class C (Light Weight) — processes giving coatings that consist essentially of zinc or other metal phosphates and have a weight 1.1 to 4.3 g/m^2 of treated surface. This class is essentially a pre-treatment for painting and is normally used for steel under paints, varnishes or lacquers of high protective value. It includes most of the spray type accelerated zinc phosphate processes.

3.1.5 Class D (Extra Light Weight) — processes giving coatings that consist essentially of iron phosphate and have a weight 0.3 to 1.1 g/m² of treated surface. This class is used for similar purposes as Class C (see 3.1.4) and includes most of the spray type alkali-metal phosphate processes.

4. PHOSPHATE TREATMENT

4.0 Control — The process, including subsequent sealing, shall be operated in conjunction with an agreed process control that is adequate to ensure uniformity of production and compliance with this standard.

4.1 Process — The process consists of the following stages:

- a) Surface preparation,
- b) Phosphate treatment,

- c) Rinsing,
- d) Drying, and
- e) Sealing.

Consecutive stages shall follow one another without delay. Care shall be taken at all stages to prevent contamination of the surfaces being treated by touching with bare hands, splashing with undesirable liquids or the condensation of moisture on parts after drying.

NOTE — If water standing is resorted to, this should be done after rinsing and should be followed by drying and sealing. Spirit stain will be applied after drying.

4.2 Surface Preparation — Before processing, all scale, rust, grease oil and foreign matter shall be removed from the surface to be treated by a method, or combination of methods, suitable for the particular phosphating process concerned. Where necessary, the cleaned components shall be adequately rinsed in cold or hot water to remove such residues of cleaning materials as might affect the bath or the quality of the coating. A slight film of surface rust, such as may be formed on storing a cleaned dried surface for a short time will not interfere with the production of a sound phosphate coating for unaccelerated processes falling in Classes A1 (see 3.1.1) and A2 (see 3.1.2).

4.3 Phosphate Treatment — The phosphate treatment shall be carried out in accordance with the agreed operating instructions for the process and in such a manner that coating formation is complete, for example, proper attention shall be paid to duration of phosphating, solution temperature, and composition, and avoidance of air locks.

The material used in the construction of the phosphating plant shall be such that they have no adverse effect on the quality of the coating produced.

4.3.1 Coating Weight — The phosphate coating weight, when determined as described in **6.3**, shall be as given in Table 1.

CLASS OF PHOSPHATING	PHOSPHATING	PROCESS	COATING WEIGHT, g/m ²		
		Min	Max		
	A1 and A2		7.5		
	В		4.3	<u> </u>	
	C		1.1	4.3	
	D		0-3	1.1	

TABLE 1 WEIGHT OF PHOSPHATE COATING

4.4 Rinsing — After the phosphate treatment, the components shall be rinsed thoroughly to remove any residues of the phosphating solution as laid down in **4.4.1** depending whether the accelerated process or the unaccelerated process has been used.

4.4.1 Rinsing after Phosphating — After treatment, the components shall be thoroughly rinsed, first in the running water and then in a hot dilute chromate solution as given in Table 2 under a temperature not below 60° C.

4.4.1.1 The chromate rinse shall consist of a solution of chromic acid or alkali metal chromate or alkali metal dichromate, or of a mixture containing approximately equal parts of chromic and phosphoric acids. The concentration of the solution shall be as given in Table 2 depending on the nature of the phosphate coating and of the sealing coat.

NOTE — Where a water rinse is used, to avoid undue accumulation of the treatment chemicals in the rinsed water, the water shall be discarded when its acidity, expressed as ml of N/10 sodium hydroxide solution (4.0 g NaOH per litre) required to neutralize a 50 ml sample, exceeds 1.0 ml. The sample shall be taken after stirring the water and shall be cooled down before titration in which phenol-phthalein shall be used as indicator and the permanent pink colour shall be taken at the end point.

TABLE 2 CONCENTRATIONS OF CHROMATE SOLUTION

(Clauses 4.4.1 and 4.4.1.1)

NATURE OF PHOSPHATE COATING AND OF SEALING COAT		CONCENTRATION IN TERMS OF CrO ₃ * PER 1 000 LITRES	
i)	Phosphate coatings of all classes to	<i>Min</i> kg 0°125	Max kg 0.50
	be sealed with paint, varnish or lacquer		
ii)	Zine phosphate coatings to be sealed with oil or grease	0.125	2.20
iii)	Manganese and/or iron phosphate coatings to be sealed with oil or grease	0.125	5.00

*The substitution of equal weight of phosphoric acid for up to one-half of the chromic acid is permissible.

4.4.1.2 The grades of phosphoric and/or chromic acid used in the preparation of chromate rinse as also the quality of water shall be of such purity that the provision relating to freedom from corrosive residues shall be met (see 6.4).

4.5 Drying — After the final rinsing, the components shall be dried thoroughly (preferably force-dried), special attention being paid to the parts that contain pockets crevices. Precautions shall also be taken to avoid local accumulation of solid residues, such as may be formed on the surface, when hard water is used for rinsing.

4.6 Sealing — After rinsing and drying, the phosphate coating shall be sealed. The organic coating to be applied shall be specified at the time of placing the order or in the relevant specification, or on the drawing governing the manufacture of the components. The sealing shall be carried out with minimum delay, but where the process is to be finished by paint, varnish or lacquer, and an interval is unavoidable between the phosphating and the first stage of sealing, the phosphated components shall be stored under conditions that preclude contamination or condensation of moisture on the surface.

NOTE — The removal of surface dust before sealing is permissible, but this shall be carried out without damage to the phosphate coating.

5. ADDITIONAL STEPS TO BE TAKEN FOR TREATMENT OF CREVICED COMPONENTS, COMPOSITE ARTICLES AND HIGH TENSILE STEELS

5.1 Treatment of Creviced Components — Components with folds, seams or crevices shall receive special attention to ensure the removal of oil or grease before phosphating. Care shall also be taken to ensure that the treatment chemicals are removed from folds, seams or crevices particularly when an accelerated phosphating process has been used.

5.2 Treatment of Composite Articles — In general the advice of supplier of the phosphating material should always be sought regarding the treatment of composite articles because:

- a) contaminatian of the bath due to solution of non-ferrous metals may affect the phosphating reaction, and
- b) the phosphating solution may attack one or more of the component metals unduly, for example, through galvanic corrosion set up by contact of dissimilar metals.

5.2.1 Composite articles made up of ferrous and non-ferrous parts shall normally have their ferrous parts phosphated before assembly into the article. Exceptions are permissible in the case of composite articles containing zinc, provided that the phosphating solution used does not cause an undesirable attack on the zinc-base surface.

The phosphating of composite articles containing copper-base material shall be limited to those cases where the copper-case material does not constitute more than 10 percent of the total surface, and where it is unlikely that the phosphating solution will penetrate the joints between the steel and the copper-base material. Tests shall be carried out to ensure that there is no undesirable attack on the copper-base material and that the phosphating solution does not become unduly contaminated with copper. In addition, checks shall be made that the presence of copper in the phosphate coating has no undesirable effect, such as interfering with the hardening of the paint if paint is to be applied.

5.3 Treatment of High Tensile Steels

5.3.1 In any particular case the precise conditions of heat treatment, that is, the temperatures and duration of heating required for all heat treatment operation depend on the composition and tensile strength of the steel and on the size of the sections. As a general rule expert advice should be sought. A general statement regarding suitable treatments is given in 5.3.2 and 5.3.3.

5.3.2 Steel components having a minimum tensile strength of 100 kgf/mm³ or greater shall be given a suitable stress-relieving treatment before phosphating if the parts contain residual stress that may cause cracks or loss of ductility while phosphating. The parts should be heated for not less than 30 minutes at a temperature of at least 130°C but below the temperature at which perceptible softening of steel will occur. To relieve possible embrittlement during phosphating process such steels shall be given a further heat treatment as given in Table 3, immediately after phosphating, washing and drying and before sealing.

5.3.3 Springs or other components subject to flexing made from steels with a specified minimum tensile strength of 100 kgf/mm² shall not be processed in compression or tension, that is, while the component is "under applied stress. Unless otherwise agreed, the phosphating process used in such cases shall be of the accelerated (but copper-free) type and the pointage of the bath shall not exceed 30.

5.3.3.1 In the case of springs or where soldered joints are employed, it may be necessary to keep the temperature below 130°C. Otherwise the treatment given in Table 3 may prove suitable.

6. INSPECTION AND TESTING

6.1 Freedom from Defects — Phosphated surfaces shall be matt without an excessively crystalline appearance. They shall be free from untreated patches and from flaky and uneven deposits, sometimes caused by excessive sludge in the bath. They shall be free from scratches, pits and residues of the processing solution as it may initiate deterioration of the organic coating or premature corrosion.

SL	Specified Range of Tensile	Conditions of HEAT TREATMENT		
NO. STRENGTH OF STEEL	Tomperature		Minimum Duration of	
	- kgf/ınm [*]	Min °C	Max °C	Heating Hours
(1)	(2)	(3)	(4)	(5)
i)	100 to 140	130	200)
ii)	Over 140	150	200	4

TABLE 3 RECOMMENDED HEAT TREATMENT

6.1.1 Samples of the treated components, or with prior agreement, the test panels that are processed simultaneously with the components shall comply with the requirements of tests given in 6.3 and 6.4. Where the test panels are used, these shall be similar in composition and surface condition to the components and shall be processed simultaneously with them, taking care, where spray-phosphating processes are involved, that the panels are held securely so as to present the maximum surface for processing.

6.2 Visual Tests — Visual tests are of value in checking the presence of phosphate coatings, although they are of limited application to lightweight or extra lightweight coatings, that is, Classes C and D.

6.2.1 Test Procedure — The procedure is as given below:

- a) Rub the particular treated surface with steel wool (Grade 000), Phosphated steel shall have a dull and matt appearance, but untreated steel shall have a bright burnished appearance.
- b) Scrape the surface once with a blunt knife. A phosphated surface shall show a whitish streak.
- c) Scrape a small area with a sharp blade. A phosphated surface shall yield a powder.
- d) Immerse the test panel for a period of 12 hours in a 3 percent sodium chloride solution. The surface of the specimen shall show no signs of corrosion.

6.3 Test for Weight of Coating

6.3.1 The weight of the phosphate coating should be determined by the following method, on samples which have not received any finishing treatment.

6.3.2 The phosphated panel shall be of suitable size (approximately 5 cm \times 10 cm), it shall be numbered before weighing for identification and shall be thoroughly degreased in trichloroethylene vapour or clean

mineral spirits. The panel (after drying if necessary and cooling in a dessicator to room temperature) shall be weighed to the nearest 0.1 mg and this weight shall be recorded as X grammes.

6.3.3 The panel shall then be immersed in a freshly prepared solution of concentrated hydrochloric acid in which antimony trioxide has been dissolved as an inhibitor in the proportion of 20 grammes per 100 ml of acid. The temperature of the stripping bath shall be maintained between 25° C and 35° C. When the coating has dissolved off (a period of 5 minutes is normally sufficient), the component shall be removed from the acid, washed for 30 seconds in running water, and any non-adherent matter shall be removed with a soft wet brush. The panel shall then be dried with a clean absorbent material (lambswool) placed in a dessicator for 10 minutes and re-weighed to the nearest 0.1 mg. This weight shall be recorded as Y grammes.

6.3.4 The steps outlined under **6.3.3** shall be repeated until two successive weighing do not differ by more than 0.5 mg. The final weight Y grammes shall be used to calculate coating weight in accordance with the formulae given under **6.3.6**.

6.3.5 The total panel area shall include both sides of the panel, if both were coated.

6.3.6 The coating weight in g/m^2 shall be calculated from the formulae:

Coating weight =
$$\frac{X-Y}{\text{Total coated panel area (cm2)}} \times 10\,000$$

where

X = initial weight in g, and Y = final weight in g.

6.3.7 A 'control' panel having the same metal composition and of the same dimensions, shall be used and the weight loss of the panel should be applied as correction factor in the coating weight.

6.4 Test for Corrosive Residues

6.4.1 This test is conducted to check the efficiency of the rinsing procedure. Samples of the sealed components or test panels shall be subjected to the following test, at the end of which they shall show no rust or other defect, for example, blistering or flaking of the organic coating. Where the organic coating is a multiple coat paint system, the test shall be made after the first coat has been applied.

6.4.2 This test indicates the presence of harmful water-soluble matter under the paint film, and is particularly valuable for showing contamination by finger marks or through splashing.

6.4.2.1 Test procedure — The samples shall be suspended for 7 days at a temperature between 25° C to 35° C in a closed vessel in which air is kept humid by a layer of water at the bottom of the vessel. At the end of the test the samples shall be removed and inspected immediately for signs of rust or blistering of the paint film.

6.5 Test for Resistance to Corrosion — The resistance of the sealed components to corrosion shall be tested in the laboratory by means of the ARE salt droplet test described in Appendix A.

6.5.1 Preliminary Damage (If the Finished Coating is Paint) — In respect of phosphating components which are subsequently painted, preliminary damage to the paint film is optional. But if such damage is desired, it shall be inflicted by scratching. Two longitudinal scratches shall be made on the lower front surface by means of a sharp tool so as to penetrate the paint film.

6.5.2 The duration of the test period shall depend on the class of phosphate coating and the method of sealing. The following examples given in Table 4 may serve as a guide.

SL No.	ORGANIC COATING	TEST CONDITIONS		
		No. of Spraying	Duration of Test Days	
(1)	(2)	(3).	(4)	
i)	Oils, air drying lacquer, spirit, stain lanoline	1	1	
ii)	Stoving lacquer, grease, mine- ral jelly	3	3	
iii)	Paints, air drying or stoving	6	7	

TABLE 4 TEST CONDITIONS AND DURATION

6.5.3 At the end of the test period, the specimen shall not show signs of corrosion.

APPENDIX A

(Clause 6.5)

ARE SALT DROPLET TEST

A-1. TEST PROCEDURE

A-1.1 The specimens shall be suspended vertically, by means of hooks of glass or other non-corrodible insulator passing through the holes in

two corners, over large dishes partly filled with water. The distance between the surface of the water and the lower edge of the specimens shall be 50 to 75 mm and the specimens shall be one behind the other and all face the same way. They shall be at a convenient distance apart (not more than 25 mm) to prevent touching when being moved and a dummy specimen shall be placed at each end of each row.

A-1.1.1 Once daily, for 5 days a week, all the specimens, including the dummies, shall be removed temporarily from their position on the rack and sprayed individually on both sides with the spray solution (see A-2), using a hand-operated atomizer (see A-3). The aim shall be to cover the surface with discrete droplets, by giving a number of puffs with the nozzle at a suitable distance (150 to 300 mm) from the specimens, the spray being directed in turn, at different parts of the surface. The spraying shall not be so heavy that the droplets coalesce; in order to avoid this it may prove necessary on occasions, where the surface is still wet from the previous spraying to refrain from applying the full weight of spray specified below. It is essential to keep the specimens covered with droplets.

A-1.1.2 Immediately after spraying, the specimens shall be returned to their positions on the rack and the whole collection shall be covered with a box like cover of sheet metal or other suitable material to prevent the drying of the droplets (see Note).

A-1.1.3 The intensity of spraying may be checked by weighing the solution deposited on both sides of a blank ($150 \times 100 \text{ mm}$) bare steel specimen, freshly degreased. The weight should lie between 0.5 and 1.0 g.

A-1.1.4 Any cover shall be suitably protected on the inside to prevent contamination of the specimen.

A-1.1.5 The temperature throughout the test shall preferably be between 25°C and 35°C and the test assembly shall be protected against external sources of heat, tending to produce local variations in temperature.

Nore — The spray may be absorbed by non-glossy paint films; if so, it may be impossible to check that the desired distribution of droplets has been obtained. This difficulty may be overcome by spraying dummy specimens of a material on which the droplets may be seen in such a way as to produce the standard distribution of droplets and then spraying the test specimens in exactly the same way. Steel or glass coated with a hard glossy finish i: suitable for this purpose.

A-2. SPRAY SOLUTION

A-2.1 The spray solution shall be a synthetic sea-water, made by dissolving the following salts in distilled water:

Salt	Concentration
	g/l
Sodium chloride, NaCl	23.0
Sodium sulphate, Na, SO ₄ 10H ₂ O	8.9
Magnesium chloride, MgCl, 6H,O	9.8
Calcium chloride (anhydrous), CaCl ₂	1.2

A-2.1.1 The salts shall be of analytical purity.

A-3, ATOMIZER

A-3.1 Any atomizing nozzle capable of producing fine droplets (not a mist) may be used for the test. The whole apparatus including the container shall be made of non-metallic material, for example, glass or ebonite.

A-4. LOSS IN WEIGHT OF CONTROL SPECIMENS

A-4.1 At the end of the agreed test period, the loss in weight of the control specimens shall be determined after derusting as described in A-4.1.1 and A-4.2.

A-4.1.1 The specimen shall be de-rusted by means of Clarke's solution, which consists of 20 g of antimonious oxide and 50 g of stannous chloride dissolved in one litre of hydrochloric acid (sp gr 1.16). The solution shall be used cold and the specimens kept moving in it until derusting is complete. The specimens shall then be washed in running water, dried and weighed. The figure to be recorded is the difference between the weight of the freshly degreased specimen and the corresponding weight after de-rusting.

It is essential that the specimens be kept moving in the solution so as to ensure rapid reduction by the reagent of ferric chloride produced by solution of the rust; otherwise attack on the steel itself may occur.

in Table 5.	
TABLE 5	AVERAGE LOSS IN WEIGHT
TEST DURATION DAYS	Average Loss in Weight per $150 \times 100 \text{ mm}$ Specimen

 $\overset{\mathbf{g}}{0\cdot 2}$

0•6 1•5 3•8

4.7

A-4.2 The average loss in weight shall be not less than the values given in Table 5.

A-5. INSPECTION

7

A-5.1 At the end of the agreed test period the specimens shall be rinsed in cold water, visually inspected, and allowed to dry at room temperature. Immediately the specimens are dry, a final inspection shall be made. The same procedure shall be followed if failure is suspected at an earlier stage, and if inspection confirms failure, the specimen or specimens concerned may be removed from test.

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Plot No. 62-63, Unit VI, Ganga Nagar, BHUBANESHWAR 751001	40	36	27
Kalaikathir Buildings, 670 Avinashi Road, COIMBATORE 641037	21	01	41
Plot No. 43, Sector 16 A, Mathura Road, FARIDABAD 121001	8-28	88	01
Savitri Complex, 116 G. T. Road, GHAZIABAD 201001	8-71	19	96
53/5 Ward No. 29, R. G. Barua Road, 5th By-lane, GUWAHATI 781003	54	11	37
5-8-58C, L. N. Gupta Marg, Nampally Station Road, HYDERABAD 500001	20	10	83
E-52, Chitaranjan Marg, C-Scheme, JAIPUR 302001	37	29	25
117/418 B, Sarvodaya Nagar, KANPUR 208005	21	68	76
Seth Bhawan, 2nd Floor, Behind Leela Cinema, Naval Kishore Road, LUCKNOW 226001	23	89	23
Patliputra Industrial Estate, PATNA 800013	26	23	05
T. C. No. 14/1421, University P. O. Palayam, THIRUVANANTHAPURAM 695034	6	21	17
NIT Building, Second Floor, Gokulpat Market, NAGPUR 440010	52	51	71
Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005	32	36	35
*Sales Office is at 5 Chowringhee Approach, P. O. Princep Street, CALCUTTA 700072	27	10	85
†Sales Office is at Novelty Chambers, Grant Road, MUMBAI 400007	309	65	28
‡Sales Office is at 'F' Block, Unity Building, Narashimaraja Square, BANGALORE 560002	222	39	71