IS 2529 (1983): Magnesium Oxide for Cosmetic Industry [PCD 19: Cosmetics]

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Indian Standard

SPECIFICATION FOR MAGNESIUM OXIDE FOR COSMETIC INDUSTRY

(Second Revision)

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Indian Standard

SPECIFICATION FOR MAGNESIUM OXIDE FOR COSMETIC INDUSTRY

(Second Revision)

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Indian Standard

SPECIFICATION FOR MAGNESIUM OXIDE FOR COSMETIC INDUSTRY

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 27 May 1983, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1963 and subsequently revised in 1977. Initially, while reviewing this standard the Sectional Committee had agreed to indicate separately the essential and optional requirements. Subsequently, this was formed non-implementable for certification and, therefore, in this revision only a single set of requirements has been stipulated.

0.3 Magnesium oxide is used in anti-perspirants to overcome skin irritation and corrosion of fabrics in contact with the skin, due to liberation of acidity from astringents. It is also used in dusting powders.

0.4 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 or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value inis standard.

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1. SCOPE

1.1 This standard prscribes requirements and methods of sampling and test for magnesium oxide for cosmetic industry.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of either bulk powder known as light magnesium oxide, or as a relatively dense powder known as heavy magnesium oxide. It shall be white. The material shall be practically insoluble in water and alcohol but shall be soluble in dilute acids.

2.1.1 The material shall be odourless and shall not develop any disagreeable smell when suspended in distilled water.

2.2 The material shall also comply with the requirements given in Table 1 when tested according to methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR MAGNESIUM OXIDE FOR COSMETIC INDUSTRY

No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Magnesium (as MgO), percent by mass of the ignited residue, *Min*	96·0	A-2
ii)	Heavy metals (as Pb), ppm, *Max*	20	A-3
iii)	Arsenic (as AS_2O_3), ppm, *Max*	2	A-4
iv)	Bulk density	As agreed to between the purchaser and the supplier	A-5
v)	Matter insoluble in dilute hydrochloric acid, percent by mass, *Max*	0·1	A-6
vi)	Matter soluble in water, percent by mass, *Max*	2·0	A-7
vii)	Loss on ignition, percent by mass, *Max*	6·0	A-8
viii)	Calcium (as CaO), percent by mass of the ignited residue, *Max*	1·0	A-9
ix)	Alkali carbonates	To pass test	A-10
x)	Iron (as Fe), percent by mass, *Max*	0·05	A-11

3. PACKING AND MARKING

3.1 The material shall be packed in well-closed containers as agreed to between the purchaser and the supplier.

3.2 The packages shall be securely closed and legibly marked with the following information:

a) Name of material;

b) Manufacturer's or supplier's name;

c) Mass of the material in the package;

d) Recognized trade-mark, if any; and

e) Batch number to enable the lot of manufacture to be traced back from records.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative test samples of the material shall be prepared as prescribed in Appendix B.

4.2 Number of Tests

4.2.1 Tests for the determination of magnesium shall be conducted on each of the individual samples constituting the set of test samples (*see* **B-3.1.1**).

4.2.2 Tests for the remaining characteristics, namely, bulk density, matter insoluble in dilute hydrochloric acid, matter soluble in water, loss on ignition, calcium, alkali carbonates, heavy metals, iron and arsenic shall be conducted on the composite sample (*see* **B-3.1.2**).

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The mean and the range for the test results for magnesium shall be calculated as follows:

Mean ($\bar{X}$) = the sum of the test results divided by the number of test results, and

Range (R) = the difference between the maximum and the minimum values of the test results.

4.3.1.1 The lot shall be declared to have satisfied the requirement for magnesium if $\bar{X}$ — 0·6 R is equal to or greater than 96·0.

4.3.2 *For Composite Sample* — The test results on the composite sample shall meet the corresponding requirements specified in **2** and in Tables 1 and 2.

4.3.3 The lot shall be declared as conforming to the requirements of this specification if **4.3.1** and **4.3.2** are satisfied; otherwise not.

APPENDIX A

(*Clause* 2.2)

ANALYSIS OF MAGNESIUM OXIDE FOR COSMETIC INDUSTRY

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affects the results of analysis.

A-2. DETERMINATION OF MAGNESIUM

A-2.1 Reagents

A-2.1.1 *Standard Sulphuric Acid* — 1 N.

A-2.1.2 *Standard Sodium Hydroxide Solution* — 1 N, freshly standardized.

A-2.1.3 *Methyl Orange Indicator Solution* — Dissolve 0·01 g of methyl orange in 100 ml of water.

A-2.2 Procedure — Weigh accurately about 1 g of the freshly ignited material and transfer it to a 250-ml conical flask. Add into the flask about 20 ml of water and transfer with a pipette 25 ml of standard sulphuric acid. Cover the flask with a watch-glass and stir the flask carefully. Wash down the watch-glass and the sides of the conical flask with water and titrate the solution in the flask with standard sodium hydroxide solution using methyl orange indicator. Carry out a blank determination using the same quantities of the reagents.

*Specification for water for general laboratory use (*second revision*).

A-2.3 Calculation

$$\text{Magnesium (as MgO), percent by mass of the ignited residue} = \frac{2{\cdot}016\,(V_1 - V_2)\,N}{M} - 0{\cdot}719\,A$$

where

V_1 = volume in ml of standard sodium hydroxide solution required for titration in the blank determination,

V_2 = volume in ml of standard sodium hydroxide solution required for titration in the test with the material,

N = normality of standard sodium hydroxide solution,

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

A = percentage by mass of calcium (as CaO) as obtained in **A-9.3**.

A-3. TEST FOR HEAVY METALS

A-3.1 Apparatus

A-3.1.1 *Nessler Cylinders* — 50-ml capacity.

A-3.2 Reagents

A-3.2.1 *Concentrated Hydrochloric Acid — see* IS : 265-1976*.

A-3.2.2 *Ammonium Chloride*

A-3.2.3 *Acetic Acid* — 1 N.

A-3.2.4 *Standard Lead Solution* — Dissolve 1·60 g of lead nitrate in water, add one millilitre of concentrated nitric acid (*see* IS : 264-1976†) and make up the volume to 1 000 ml. Pipette out 10 ml of this solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0·01 mg of lead (as Pb).

A-3.2.5 *Hydrogen Sulphide Solution* — freshly prepared, saturated solution.

A-3.3 Procedure — Mix 2·000 g of the material with 10 ml of water and add just sufficient concentrated hydrochloric acid to dissolve the material. Evaporate to dryness on a steam-bath and dissolve the residue in about 30 ml of water. Transfer the solution to a Nessler cylinder, add 1 g of ammonium chloride and 1 ml of acetic acid. Carry out a control test another Nessler cylinder using 4 ml of standard lead

*Specification for hydrochloric acid (*second revision*).
†Specification for nitric acid (*second revision*).

solution in place of the material, and 1 g of ammonium chloride and 1 ml of acetic acid. To each Nessler cylinder add 10 ml of hydrogen sulphide solution, dilute to the mark and shake well. Compare the intensity of colour produced in the two cylinders.

A-3.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour obtained with the material is not greater than that obtained in the control test.

A-4. TEST FOR ARSENIC

A-4.1 Preparation of Solution — Dissolve 1·000 g of the material in 5 ml of dilute hydrochloric acid (approximately 5 N) and dilute with water to 15 ml.

A-4.2 Carry out the test for arsenic as prescribed in IS : 2088-1971*, using for comparison a stain obtained with 0·002 mg of arsenic trioxide (as AS_2O_3).

A-5. DETERMINATION OF BULK DENSITY

A-5.1 Apparatus

A-5.1.1 Assemble the apparatus as shown in Fig. 1. The base of measuring cylinder *A* shall be ground flat and the empty measuring cylinder *A* together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250 ml with an error, if any, of less than one millilitre. The distance between zero and 250-ml graduation on the measuring cylinder *A* shall be not less than 220 mm and not more than 240 ml. The distance between the flat-ground part of the base of measuring cylinder *A* and the rubber base pad *B*, when the measuring cylinder *A* is raised to full height shall be 25 ± 2mm.

A-5.1.2 *Rubber Base Pad* — The rubber base pad *B* shall have a shore hardness of 42 to 50.

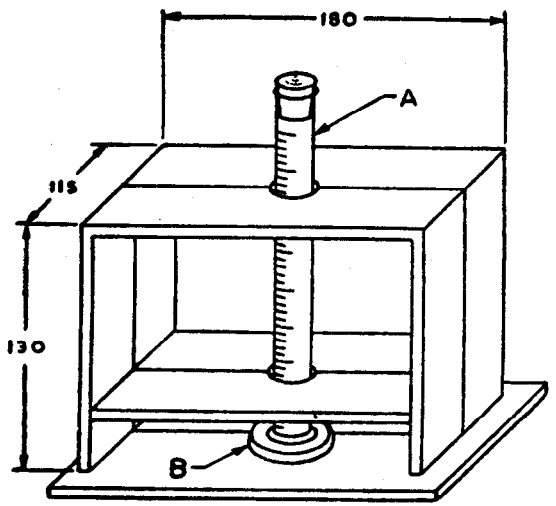
A-5.1.3 *Balance* — Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to less than 0·1 g.

A-5.2 Procedure

A-5.2.1 Sieve about 40 g of the material through 250-micron IS Sieve on to a tared glazed paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder which should be held at 45° to the vertical, without knocking or squeezing. Assemble the apparatus as shown in Fig. 1. With the thumb and four fingers of one hand, gently grasp the upper part of the cylinder, and within one second left it about 25 mm (taking care not to jerk the cylinder by knocking it

*Methods for the determination of arsenic (*first revision*).

against the upper stop) and let it drop. Note the volume after dropping it once. Continue lifting and droping untll 50 complete drops have been given to the cylinder. During this operation give a gentle turn of about 10° in the clockwise direction to the cylinder after every two drops. As soon as 50 drops are completed, raise the cylinder to eye level and read the volume of the material.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF BULK DENSITY

A-5.3 Calculation

Bulk density, g/ml:

a) After one tap $= \frac{M}{V_1}$

b) After 50 taps $= \frac{M}{V_{50}}$

where

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

V_1 = volume in ml of the material after one tap, and

V_{50} = volume in ml of the material after 50 taps.

A-6. DETERMINATION OF MATTER INSOLUBLE IN DILUTE HYDROCHLORIC ACID

A-6.1 Reagent

A-6.1.1 *Concertnated Hydrochloric Acid — see* IS : 265-1976*.

A-6.2 Procedure — Dissolve about 5 g of the material, accurately weighed, in a mixture of 100 ml of water and 25 ml of concentrated hydrochloric acid. Boil the solution for 5 minutes, filter any undissolved matter through an ashless filter paper and wash well with water until the last washing is free from chloride. Dry the residue and ignite at a temperature of 700° to 800°C for about 15 minutes. Cool in a desiccator and weigh.

A-6.3 Calculation

$$\text{Matter insoluble in dilute hydrochloric acid} = \frac{100\ M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

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

A-7. DETERMINATION OF MATTER SOLUBLE IN WATER

A-7.1 Procedure — Boil about 2 g of the material, accurately weighed, with about 50 ml of water for 5 minutes in a covered beaker and filter while hot. Wash the filter three or four times and collect the washings with the filtrate. Cool and dilute the solution to 100 ml. Evaporate 25 ml of the filtrate to dryness and dry at 105° ± 2°C for one hour. Preserve the remaining solution for test in **A-10.**

A-7.2 Calculation

$$\text{Matter soluble in water, percent by mass} = \frac{400\ M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

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

A-8. DETERMINATION OF LOSS ON IGNITION

A-8.1 Procedure — Weigh accurately about 0·5 g of the material and ignite it to constant weight in a covered platinum crucible.

*Specification for hydrochloric acid (*second revision*).

A-8.2 Calculation

$$\text{Loss on ignition, percent by mass} = \frac{100\ M_1}{M_2}$$

where

M_1 = loss in mass in g, and

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

A-9. DETERMINATION OF CALCIUM

A-9.1 Reagents

A-9.1.1 *Concentrated Hydrochloric Acid — see* IS : 265-1976*

A-9.1.2 *Rectified Spirit* — 95 percent by volume.

A-9.1.3 *Sulphuric Acid* — 25 percent (*m/v*).

A-9.2 Procedure — Dissolve about 0·5 g of freshly ignited material accurately weighed, in concentrated hydrochloric acid added in small portions. Filter, if necessary, add to the filtrate 100 ml of rectified spirit and 40 ml of sulphuric acid and let stand overnight. If crystals of magnesium sulphate separate out, warm the mixture to about 50°C to dissolve them. Filter, wash with a mixture of two volumes of rectified spirit and one volume of sulphuric acid and ignite. Weigh the residue.

A-9.3 Calculation

$$\text{Calcium (as Cao), percent by mass of the ignited residue} = \frac{41{\cdot}18\ A}{M}$$

where

A = mass in g of the residue, and

M = mass in g of the ignited material taken for the test.

A-10. TEST FOR ALKALI CARBONATES

A-10.1 Reagents

A-10.1.1 *Standard Hydrochloric Acid* — 0·1 N.

A-10.1.2 *Methyl Red Indicator Solution* — Dissolve 0·1 g of methyl red in 100 ml of rectified spirit (*see* IS : 323-1959†).

*Specification for hydrochloric acid (*second revision*).

†Specification for rectified spirit (*revised*).

A-10.2 Procedure —Pipette exactly 50 ml of the solution preserved in **A-7.1** into a conical flask. Titrate with standard hydrochloric acid, using methyl red indicator.

A-10.2.1 The material shall be taken to have passed the test if not more than 1·0 ml of standard hydrochloric acid is required for the titration.

A-11. TEST FOR IRON

A-11.1 Apparatus

A-11.1.1 *Nessler Cylinders* — 50-ml capacity.

A-11.2 Reagents

A-11.2.1 *Concentrated Nitric Acid — see* IS : 264-1976*.

A-11.2.2 *Ammonium Persulphate*

A-11.2.3 *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up to 100 ml and shake vigorously until the solution is clear.

A-11.2.4 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$FeSO_4 (NH_4)_2 SO_4 \cdot 6H_2O$] in 10 ml of dilute sulphuric acid (10 percent by volume) and dilute with water to 1 000 ml. Pipette out 10 ml of the solution and dilute with water to make up the volume to 100 ml. One millilitre of this solution contain 0·01 mg of iron (as Fe).

A-11.3 Procedure — Boil 0·100 g of the material with 5 ml of a mixture of one volume of concentrated nitric acid and nine volumes of water for one minute. Cool, transfer the solution to a Nessler cylinder add 50 mg of ammonium pursulphate and 15 ml of butanolic potassium thiocyanate solution. Carry out a control test in another Nessler cylinder with 5 ml of standard iron solution in exactly the same way as above.

A-11.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour obtained in the test with the material is not greater than that in the control test.

*Specification for nitric acid (*second revision*).

APPENDIX B

(*Clauses* 4.1, 4.2.1 *and* 4.2.2)

SAMPLING OF MAGNESIUM OXIDE FOR COSMETIC INDUSTRY

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 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 The sampling instrument shall be clean and dry.

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

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry air-tight glass or other suitable containers.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling, the date of sampling, and the year of manufacture of the material.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

B-2.1.1 Samples shall be tested from each lot separately for ascertaining conformity of the material to the requirements of this specification.

B-2.2 The number of containers (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be as given in Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(*Clauses* B-2.2 *and* B-3.1.1)

LOT SIZE (N)	NUMBER OF CONTAINERS TO BE SELECTED (n)
(1)	(2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 and above	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables *see* IS : 4905-1968* shall be used. In case such tables are not available, the following procedure may be adopted:

Starting from any container, count them as 1, 2, 3 ..... r and so on in a systematic manner, where r is the integral part of N/n. Every rth container thus counted shall be withdrawn from the lot, till the requisite sample is obtained.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (*see* Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **2** and shall be not less than 250 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantities shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 0·5 kg. This sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.1.3 The remaining portion of the material from each container (after the quantity needed for the formation of composite sample has been taken) shall be divided into three equal parts, each part weighing

*Methods for random sampling.

not less than 100 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed air-tight with stoppers and labelled with all the particulars of sampling given in **B-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used for the referee.

B-3.2 Referee Sample — The referee sample shall consist of a composite sample (*see* **B-3.1.2**) and a set of individual samples (*see* **B-3.1.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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INDIAN STANDARDS

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Chemicals, organic (miscellaneous)
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Coal carbonization products
Coated fabrics
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Dental materials
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Dye intermediates
Electroplating chemicals
Explosive and pyrotechnicals
Fertilizers
Fillers, stoppers and putties materi
Footwear
Glass and glassware
Industrial gases
Inks and allied products
Laboratory glassware thermometers and related apparatus
Lac and lac products
Leather, leather goods and leather dressings
Linters and allied products
Lubricants and related products
Oil pastes
Oils and fats, oleaginous seeds and fruits
Painters' materials (miscellaneous)
Paper and its products
Paper and pulp board packaging materials
Perfumery materials, natural and synthetic
Petroleum and petroleum products
Photographic chemicals
Pigments and extenders
Plastics
Polishes
Printing inks
Ready mixed paints and enamels
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Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones : 26 60 21, 27 01 31 **Telegrams : Manaksanstha**

Regional Offices :		Telephone
Western : Novelty Chambers, Grant Road	BOMBAY 400007	89 65 28
Eastern : 5 Chowringhee Approach	CALCUTTA 700072	27 50 90
Southern : C.I.T. Campus, Adyar	MADRAS 600113	41 24 42
Northern : B69, Phase VII	S.A.S. NAGAR (MOHALI) 160051	8 78 26

Branch Offices :		
'Pushpak' Nurmohamed Shaikh Marg, Khanpur	AHMADABAD 380001	2 03 91
'F' Block, Unity Bldg, Narasimharaja Square	BANGALORE 560002	22 48 05
Gangotri Complex, Bhadbhada Road, T.T. Nagar	BHOPAL 462003	6 27 16
22E Kalpana Area	BHUBANESHWAR 751014	5 36 27
5-8-56C, L.N. Gupta Marg	HYDERABAD 500001	22 10 83
R 14 Yudhister Marg, C Scheme	JAIPUR 302005	6 98 32
117/418 B Sarvodaya Nagar	KANPUR 208005	4 72 92
Patliputra Industrial Estate	PATNA 300013	6 28 08
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27

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