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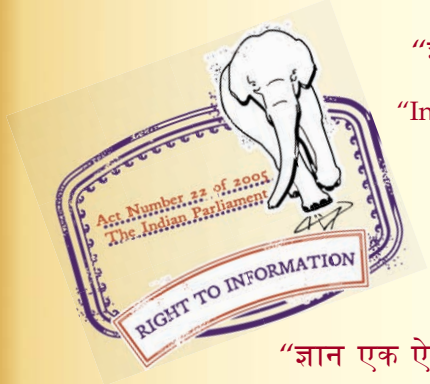
IS 8643 (1977): Dye, methyl violet, for stamp-pad ink [CHD 14: Printing, Inks, Stationary and Allied Products]



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"Knowledge is such a treasure which cannot be stolen"

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IS : 8643 - 1977
(Reaffirmed 2003)

Indian Standard

SPECIFICATION FOR DYE, METHYL VIOLET, FOR STAMP-PAD INK

UDC 667·283·4 : 667·532·032

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

April 1978

Indian Standard

SPECIFICATION FOR DYE, METHYL VIOLET, FOR STAMP-PAD INK

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39-A, Venkatanarayana Road, T. Nagar, Madras 600017

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Indian Standard

SPECIFICATION FOR DYE, METHYL VIOLET, FOR STAMP-PAD INK

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 14 October 1977, after the draft finalized by the Inks and Allied Products Sectional Committee had been approved by the Chemical Division Council.

0.2 The Sectional Committee felt the need of formulating Indian Standards for various dyes used in ink industry. It is expected that this standard will help the producers of stamp-pad ink to procure methyl violet of assured quality from indigenous sources. Formulation of specifications for dyes for writing inks has also been taken up and will form the subject of a separate Indian Standard.

0.3 Since no dependable chemical method for evaluating the purity of the dye is available, spectrophotometric examination of the material has been prescribed.

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 in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for methyl violet dye for use in stamp-pad ink.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given in IS : 4395-1967† in addition to the following shall apply.

*Rules for rounding off numerical values (*revised*).
†Glossary of terms relating to inks and allied industries.

2.1.1 *Approved/Standard Sample* — The sample accepted by the indentor or inspection authority as the basis of supply/manufacture.

3. REQUIREMENTS

3.1 Description — The material shall consist essentially of a complex mixture of *n*-hexa, *n*-penta and *n*-tetra-methyl-*p*-rosaniline hydrochlorides (colour index 42535 B)*. It shall be in the form of a yellowish green powder with metallic lustre. The material shall not be toned with magenta or any other red dye.

3.2 Identification

3.2.1 When dissolved in water, the material shall give a violet solution with a blue tone.

3.2.2 When concentrated sulphuric acid (*see* IS : 266-1961†) is added to the material, it shall turn orange in colour and give a greenish precipitate on dilution with water.

3.2.3 The aqueous solution of the material when made alkaline shall precipitate the dye base giving a colourless supernatant liquid.

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

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in sound, clean, dry tinplate containers with a suitable lever lid or in any other container as agreed to between the purchaser and the supplier.

4.2 Marking — The packages shall be legibly marked with the following information:

a) Description of the material;
b) Tare and net mass;
c) Month and year of manufacture;
d) Manufacturer's name and/or his recognized trade-mark, if any; and
e) Batch number in code or otherwise to enable the lot of manufacture to be traced from records.

*Colour Index, 1957, second edition, published by Society of Dyers and Colourists, UK and American Association of Textile Chemists and Colourists.

†Specification for sulphuric acid (*revised*).

4.2.1 The packages may also be marked with the ISI Certification Mark.

NOTE — The use of ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Mark) 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 standnrd 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 ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

TABLE 1 REQUIREMENTS FOR DYE, METHYL VIOLET, FOR STAMP-PAD INK

(*Clause* 3.3)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST (REF TO CL NO. OF APPENDIX A)
(1)	(2)	(3)	(4)
i)	Volatile matter, percent by mass, *Max*	3·0	A-2
ii)	Water insoluble matter, percent by mass, *Max*	5·0	A-3
iii)	Sulphated ash, percent by mass, *Max*	4·0	A-4
iv)	*p*H (of 1 percent aqueous solution)	4 ± 1	A-5
v)	Acidity	To pass the test	A-6
vi)	Dyeing test	Comparable to approved/standard sample	A-7
vii)	Spectrophotometric examination	Comparable in shape of curve and strength to approved/standard sample	A-8

5. SAMPLING

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

APPENDIX A

(*Clause* 3.3)

METHODS OF TEST FOR DYE, METHYL VIOLET, FOR STAMP-PAD INK

A-1. QUALITY OF REAGENTS

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

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

A-2. DETERMINATION OF VOLATILE MATTER

A-2.0 The volatile matter is determined by Dean and Stark method.

A-2.1 Apparatus

A-2.1.1 *Flask* — Round bottom flask of 500 ml capacity made either of glass or copper, and as shown in Fig. 1. The glass flask may have an interchangeable ground-glass joint at the neck. Alternatively, a cork may be used for connection to the receiver. In case a metal flask is used, care shall be taken to provide airtight connection between the metal flask and the receiver.

A-2.1.2 *Condenser* — A glass, water-cooled, reflux type condenser of the design shown in Fig. 1. The essential dimensions are the external diameters of the inner tube and of the jacket, which shall be 16 to 17 mm and 23 to 25 mm respectively; the length of the cooled section shall be adequate to prevent loss of vapours. The lower end of the inner tube is ground at an angle of approximately 60° to the axis. The drainage tip may finish either in a ground-glass conical joint to fit into the receiver (*see* Fig. 1) or else project 60 mm below the water jacket for fitting into a cork.

A-2.1.3 *Two-Millilitre Receiver* — made of glass and as shown in Fig. 2. The receiver serves to collect and measure the condensed water and to return the carrier liquid to the flask. It may be used with or without standard taper ground-glass joints.

A-2.1.3.1 The graduated portion of the receiver shall have a capacity of 2 ml when filled to the highest graduation mark. The scale shall cover the range of 0·1 to 2 ml and shall be divided into intervals of 0·05 ml. The graduation marks corresponding to 0·5 ml, 1·0 ml, 1·5 ml and 2·0 ml shall be numbered. The numbered graduation marks shall

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

be carried completely round the tube. The graduation marks corresponding to 0·15 ml, 0·25 ml, 0·35 ml and so on up to and including 1·95 ml, shall be carried half-way round the tube. The remaining graduation marks shall be intermediate in length and shall project equally at each end beyond the shortest graduation marks. The error at any point on the scale shall not exceed $\pm$ 0·02 ml and the difference between the errors at any two points shall not exceed 0·02 ml.

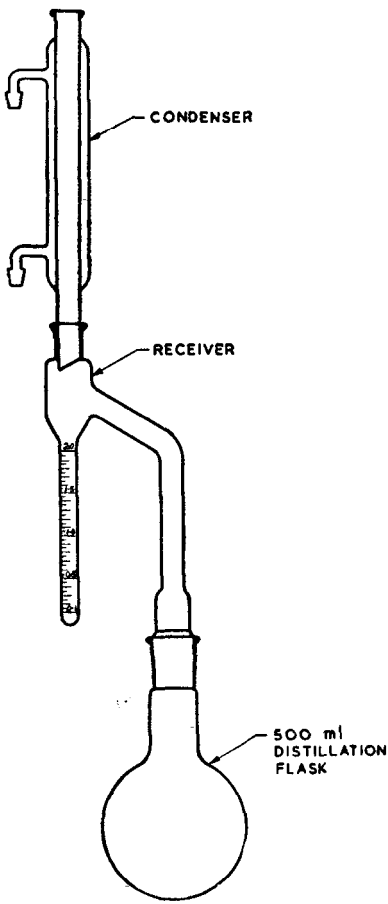
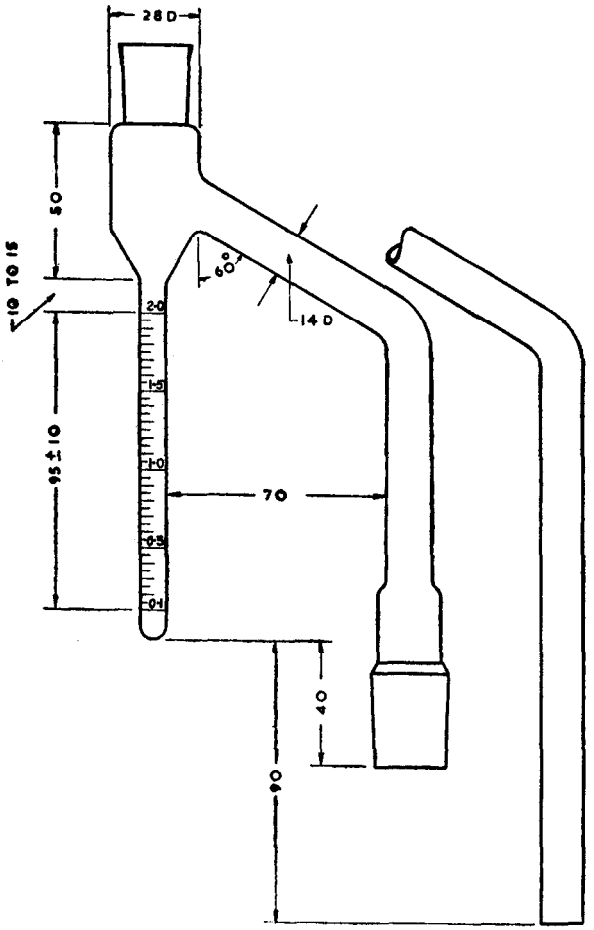


FIG. 1 ASSEMBLY OF APPARATUS



All dimensions in millimetres.

FIG. 2 2-ml RECEIVER

A-2.1.4 *Graduated Measuring Cylinder* — of 100 ml capacity (*see* IS : 878-1956*).

A-2.2 Carrier Liquid — toluene (*see* IS : 1839-1961†).

*Specification for graduated measuring cylinders.
†Specification for toluene, reagent grade.

A-2.3 Procedure

A-2.3.1 Weigh accurately, as quickly as possible, at least 10 g of the material directly into the flask and add 100 ml of the carrier liquid. Fill the receiver with the carrier liquid. Assemble the apparatus as shown in Fig. 1. If ground-glass joints are not used, connect the apparatus with tight-fitting corks and insert the condenser in the receiver so that its lower end is approximately 25 mm above the bottom of the side tube of the receiver. Insert a loose plug of cotton wool in the top of the condenser tube to prevent the condensation of atmospheric moisture in the condenser tube.

A-2.3.2 Heat the flask and regulate the heating so that the condensate falls from the end of the condenser at the rate of 2 to 5 drops per second. Continue heating the material until the volume of water in the receiver remains constant for 5 minutes. Wash down with carrier liquid any water adhering to the condenser tube. When the volume of water remains constant in the receiver, remove the source of heat from the flask and allow the receiver and contents to cool to room temperature If any droplets of water a re adhering to the sides of the receiver, dislodge them with a fine wire. Record the total volume of water.

A-2.4 Calculation

$$\text{Volatile matter, percent by mass} = 100\,\frac{V}{M}$$

where

V = volume in ml of water collected in the apparatus, and

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

A-3. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure — Dissolve with stirring about 2 g of accurately weighed material in 100 ml of water heated at 90°C. Filter the solution through a weighed filter paper (Whatman No. 1 or equivalent) under vacuum. Wash the residue repeatedly till the filtrate is colourless. Dry the filter paper in an oven at 90 ± 2°C and weigh to constant mass.

A-3.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = 100 \times \frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue, and

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

A-4. DETERMINATION OF SULPHATED ASH

A-4.1 Reagent

A-4.1.1 *Concentrated Sulphuric Acid — See* IS : 266-1961*.

A-4.2 Procedure — Ignite a 7-cm silica crucible in a muffle furnace. Cool in a desiccator and weigh. Place about 2 g of the material in the crucible and weigh accurately. Heat the crucible on a low flame until the material is completely carbonized. Burn off the carbonaceous matter by further heating in the muffle furnace. When the ash is practically white in colour, cool the crucible, add 5 drops of concentrated sulphuric acid and heat on a burner. When all the sulphuric acid has volatilized, cool the crucible in a desiccator and weigh accurately to constant mass.

A-4.3 Calculation

$$\text{Sulphated ash, percent by mass} = 100 \times \frac{M_1}{M}$$

where

M_1 = mass in g of the ignited ash, and

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

A-5. DETERMINATION OF *p*H VALUE

A-5.1 Procedure — Dissolve 0·2 g of the material in 50 ml of freshly boiled and cooled water and determine the *p*H by means of a suitable *p*H meter. Reserve the solution for test under **A-6**.

A-6. TEST FOR ACIDITY

A-6.1 Reagent

A-6.1.1 *Standard Sodium Hydroxide Solution* — 1 N.

A-6.2 Procedure — Fitrate the solution reserved in **A-5.1** with standard sodium hydroxide solution. Stir the solution continuously until the *p*H as recorded by a glass or antimony electrode, is exactly 7·0.

A-6.2.1 The material shall be taken to have satisfied the requirements of the test if not more than 2 ml of the standard sodium hydroxide solution is required for titration.

A-7. DYEING TEST

A-7.1 Reagents

A-7.1.1 *Tannic Acid* — (mordanting agent)

*Specification for sulphuric acid (*revised*).

A-7.1.2 *Antimony Potassium Tartrate (Tartar Emetic)* — (fixing agent)

A-7.1.3 *Glacial Acetic Acid* — 30 percent (*m/m*).

A-7.2 Cotton Hanks — 10, 7·4 tex, bleached.

A-7.3 Mordanting (Process for 10 Hanks)

A-7.3.1 Dissolve 3 g of tannic acid in a little quantity of hot water and add this to the remaining quantity of hot water to make a volume of 2 000 ml.

A-7.3.2 Raise the temperature of the mordanting bath to 80°C and then enter the yarn into the bath and work for half an hour at the same temperature. After that, work the material in the cooling bath and keep it overnight.

A-7.3.3 Next day remove the material from the mordanting bath, squeeze well, make even by stretching it on the hanks and take for fixing.

A-7.4 Fixing

A-7.4.1 *Fixing Bath* — Dissolve 1·5 g of antimony potassium tartrate (tartar emetic) in a little quantity of hot water. Add enough cold water to make a volume of 1 000 ml.

A-7.4.2 Enter the tanned material in tan fixing bath and work at room temperature for half an hour. Remove the material from the fixing bath, wash well with cold water to remove the unfixed tannic acid. Then squeeze it and take it for dyeing without drying the material.

A-7.5 Dissolving the Dye — Make a paste of 0·25 g of the dye with 1 ml of glacial acetic acid and cold water and then dissolve by pouring hot water on to the paste with stirring until the dye goes into solution. Make up the volume to 50 ml.

A-7.6 Dye-Bath — Consisting of 180 ml of water and 1 ml of glacial acetic acid.

A-7.7 Procedure — Work the yarn, mordanted and fixed as in **A-7.3** and **A-7.4**, in the dye-bath (without the addition of dye) for about 10 minutes. After that, add 10 ml of dye solution to the dye-bath and work the hank for 10 minutes at 40 to 50°C. Then add a second lot of 10 ml of the dye solution. (During the dye solution addition, remove the hank from the bath.) Raise the temperature to 90°C and work the hank for 30 minutes. Remove the hank, squeeze it and wash in running water till the outflow is colourless. Then squeeze it dry in air and compare it with a hank dyed in a similar manner with approved/standard dyestuff.

A-8. SPECTROPHOTOMETRIC EXAMINATION

A-8.1 Procedure — Weigh 0·125 g of the dye accurately and dissolve it in water and make up exactly to 250 ml in a volumetric flask. Take 2·0 ml of this solution in a 500-ml of volumetric flask, add 10 ml of dilute acetic acid (25 g/l) and 30 ml of methanol. Make up the volume to 500 ml. Take the readings for absorbance in the entire visible range and plot the curve (in case an automatic recorder is available this curve is automatically plotted). The absorbance maxima shall coincide at 585 $\pm$ 2 nm and shall be recorded.

A-8.2 The material shall be considered to have satisfied the requirements of the test if:

a) the general shape of the curve is similar to that obtained with an approved/standard sample of the material, and

b) the percentage purity of the material as determined by the following formula is within $\pm$ 2 of the approved/standard sample:

$$\text{Purity, percent} = 100 \times \frac{\text{Absorbance of the sample}}{\text{Absorbance of the approved/standard sample}}$$

APPENDIX B

(*Clause* 5.1)

SAMPLING OF DYE, METHYL VIOLET, FOR STAMP-PAD INK

B-1. GENERAL

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected place.

B-1.2 The sampling instrument shall be clean and dry.

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

B-1.4 Samples shall be placed in clean, dry, airtight glass or other suitable containers on which the material has no action.

B-1.5 Each sample container after filling shall be sealed airtight with a stopper, and marked with full particulars of the material (*see* **4.2**) and the date of sampling.

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, 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 for ascertaining the conformity of the material to the requirements of the specification.

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

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Lot Size	No. of Containers to be Chosen
N	n
(1)	(2)
Up to 50	2
51 „ 100	3
101 „ 200	4
201 „ 350	5
351 „ 500	6
501 „ 700	7
701 „ 1 000	8
Above 1 000	10

B-2.3 These containers shall be chosen at random from the lot. For random selection procedures, guidance can be had from IS : 4905-1968*. In case this standard is not available, the following procedure shall be adopted. Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1,2,3..., etc, up to r and so on where r is the integral part of N/n (N and n being lot size and the number of containers to be selected respectively). Every rth container thus counted shall be withdrawn from the lot to give a sample for test.

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. The total quantity of the material drawn from each container shall be sufficient to

*Methods for random sampling.

conduct the tests for all the characteristics given under **3** and shall not exceed 200 g.

B-3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken from each selected container and shall be mixed up well together so as to form a composite sample weighing not less than 150 g. This composite sample shall be divided into three equal parts — one for the purchaser, another for the supplier and the third to be used as referee sample.

B-3.1.3 The remaining portions of the material from each container (after a small quantity needed for formation of the composite sample has been taken out) shall be divided into three equal parts, each part having sufficient quantity of the material to carry out the tests. These parts shall be transferred immediately to thoroughly dried bottles which shall then be sealed airtight with stoppers and labelled with all the particulars of sampling given in **4.2**. The material in each such sealed bottle shall constitute a test sample. These individual samples shall be separated into three identical sets of test samples in such a way that each set has a 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 as a referee sample.

B-3.2 Referee Sample — The referee sample shall consist of the composite sample (*see* **B-3.1.2**) and a set of individual test samples marked for this purpose (*see* **B-3.1.3**), 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.

B-4. NUMBER OF TESTS

B-4.1 Tests for all the requirements laid down in **3.1** to **3.3** shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 A lot shall be declared as conforming to the specification if the test results on the composite sample satisfies all the requirements specified in **3.1** to **3.3**.

INDIAN STANDARDS

ON

INKS AND ALLIED PRODUCTS

IS:

219-1975 Ink-powder and tablets (*second revision*)

220-1972 Ferro-gallo tannate fountain pen ink (0·1 percent iron content) (*second revision*)

221-1977 Ink fluid, blue black for permanent records (*second revision*)

222-1977 Ink fluid, for general purposes (*second revision*)

393-1975 Ink, stamp-pad (*second revision*)

394-1963 Ink, cloth marking (*revised*)

788-1971 Ink, drawing, waterproof, coloured (*first revision*)

789-1971 Ink, drawing, waterproof, black (*first revision*)

1221-1971 Dye based fountain pen inks (*first revision*)

1222-1973 Ink, duplicating for twin cylinder rotary machines (*second revision*)

1234-1957 Ink, stencil, oil base, for marking porous surfaces, colour as required

1333-1973 Ink, duplicating for single drum rotary machines (*first revision*)

1379-1959 Ink, stencil, oil base for marking non-porous surfaces, colour as required

1380-1959 Ink, finger printing, black

1440-1959 Ink, metal stamp, black

1551-1976 Carbon papers, for typewriters (*first revision*)

1581-1975 Ferro-gallo tannate fountain pen ink (0·2 percent iron content) (*first revision*)

2230-1962 Dye, methylene blue, for ink industry

2247-1962 Dye, ink blue, for ink industry

2694-1963 School chalks, moulded, white

3450-1976 Carbon papers, handwriting (*first revision*)

4174-1977 Typewriter ribbons (*first revision*)

4175-1967 Correcting fluid

4222-1967 Coloured chalks, moulded

4395-1967 Glossary of terms relating to inks and allied industries

4747-1977 Pads for rubber stamps (*first revision*)

5086-1969 Stencil paper

5805-1970 Ball point pen ink

6897-1973 Methods of test for tannic acid

6898-1973 Methods of test for gallic acid

8075-1976 Back coated carbon papers for typewriter

8100-1976 Water colours for students

8101-1976 Poster colours

8277-1976 Water based records inks

8279-1976 Hectographic carbon paper

8642-1977 Dyes for water based writing inks

8643-1977 Dye, methyl violet, for stamp-pad ink

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*	*Conversion*
Force	newton	N	1 N = 0·101 972 kgf
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
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2