IS 220 (1988): Fountain Pen Ink - Ferro-gallo Tannate (0.1 Percent Iron Content) [CHD 14: Printing, Inks, Stationary and Allied Products]

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IS 220 : 1988
Reaffirmed 2010

Indian Standard
FOUNTAIN PEN INK—FERRO-GALLO TANNATE (0.1 PERCENT IRON CONTENT) SPECIFICATION
(Third Revision)

UDC 667.485

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

September **1989** **Price Group 4**

Inks and Allied Products Sectional Committee, CDC 13

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards on 21 November 1988, after the draft finalized by the Inks and Allied Products Sectional Committee had been approved by the Chemical Division Council.

The original standard was published in 1950 which covered iron based ink of blue-black colour only. It was revised in 1959 to include iron based inks of different colours such as blue, brown and black. As a result of experience gained through the years a test for sedimentation was included in the second revision in 1972.

The present revision had been necessitated due to the change in availability of raw materials and as such the requirement of Iodine absorption has been deleted. The value of total solids has been modified and the value of sedimentation has been quantified. A method for determination of sedimentation has been included.

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, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised)*'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 2 AUGUST 2006
TO
IS 220 : 1988 FOUNTAIN PEN INK FERRO-GALLO TANNATE (0.1 PERCENT IRON CONTENT)

(Third Revision)

(*First cover page*) — Insert the Hindi Title as follows:

‘फाउन्टेन पैन की फैरो-गैलो स्याही (0.1 प्रतिशत लौह अंश)
(तीसरा पुनरीक्षण)’

(CHD 14)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 NOVEMBER 2004
TO
IS 220 : 1988 FOUNTAIN PEN INK— FERRO-GALLO TANNATE (0.1 PERCENT IRON CONTENT) — SPECIFICATION

(Third Revision)

[*Page* 2, *Table* 1, *Sl No.* (ii), *col* 3] — Substitute '0.05' *for* '0.1'.

(CHD 14)

Reprography Unit, BIS, New Delhi, India

Indian Standard

FOUNTAIN PEN INK – FERRO-GALLO TANNATE (0.1 PERCENT IRON CONTENT) SPECIFICATION

(*Third Revision*)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and tests for ferro-gallo tannate fountain pen inks containing not less than 0.1 percent of iron.

2 REFERENCES

The Indian Standards listed in Annex A are necessary adjuncts to this standard.

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 4395 : 1987, shall apply.

4 REQUIREMENTS

4.1 General

The ink shall be suitable for use in fountain pens. It shall be quick drying and shall flow easily from the pen. It shall not feather on paper having one minute cobb test value equivalent to cream wove/cream laid paper (*see* IS 1848 : 1981) or smudge or be sticky after drying on the writing paper.

4.2 The ink shall also comply with the requirements given in Table 1.

4.3 Keeping Quality

The ink shall write well with a fountain pen without clogging for a minimum period of two years from the date of manufacture.

5 PACKING AND MARKING

5.1 Packing

Unless otherwise agreed to between the purchaser and the supplier, the material shall be filled in glass bottles conforming to IS 1945: 1986.

5.2 Marking

Each bottle shall be marked with the following information:

a) Name of the material followed by the words 'iron based' in parantheses;

b) Colour of the material;

c) Volume, in ml, in the bottle;

d) Manufacturer's name and/or his recognized trade-mark, if any; and

e) Identification in code or otherwise to enable the lot and date of manufacture to be traced back from records.

5.2.1 The bottles may also be marked with the Standard Mark.

6 SAMPLING

6.1 The method of drawing representative samples of the material from a lot, number of tests to be performed and the criteria for finding the conformity of the material to the requirements of this specification shall be as prescribed in Annex K.

7 TEST METHODS

7.1 Test shall be conducted as prescribed in col 4 of Table 1.

7.2 Quality of Reagents

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.

Table 1 Requirements for Ferro-Gallo Tannate Fountain Pen Ink (0.1 Percent Iron Content)

(*Clause* 4.2 *and* 7.1)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Total solids, g per 100 ml, *Max*	3.0	B
ii)	Iron content (as Fe), g per 100 ml, *Min*	0.1	C
iii)	Ratio of ethyl acetate extract to iron content, *Min*	3.0	D
iv)	Sedimentation, g/100 ml, *Max*	0.05	E
v)	Corrosion, percent by mass, *Max*	5	F
vi)	Hue ami intensity of colour	Close match to the reference ink	G
vii)	Penetration	Not more than the reference ink	
viii)	Permanence	Not less than the reference ink	
ix)	Freedom from clogging	To pass test	H
x)	Stability	To pass test	J

ANNEX A

(*Clause* 2.1)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
IS 262 : 1982	Specification for ferrous sulphate, heptahydrate (*second revision)*
IS 265 : 1987	Specification for hydrochloric acid (*third revision)*
IS 323 : 1959	Specification for rectified spirit (*revised)*
IS 324 : 1959	Specification for ordinary denatured spirit (*revised)*
IS 517 : 1967	Specification for methanol (methyl alcohol) (*second revision)*
IS 538 : 1968	Specification for phenol (carbolic acid) (*first revision)*
IS 1070 : 1977	Specification for water for general laboratory use (*second revision)*
IS 1388 : 1959	Specification for reagent bottles
IS 1848 : 1981	Specification for writing and printing papers (*second revision)*
IS 1945 : 1986	Specification for glass bottles for fluid inks (*first revision)*
IS 4395 : 1987	Glossary of terms relating to ink and allied industries *(first revision)*
IS 4905 : 1968	Method for random sampling
IS 8642 : 1977	Specification for dyes for water-based writing inks.

ANNEX B

(*Table* 1)

DETERMINATION OF TOTAL SOLIDS

B-0 GENERAL

B-0.1 Outline of the Method

The material is evaporated to dryness and the residue weighed till constant weight.

B-1 APPARATUS

Hot Air Oven, able to maintain 105 ± 2°C.

Steam-bath

Porcelain, Silica or Platinum Dish

Analytical Balance, with sensitivity of 1 mg.

B-2 PROCEDURE

B-2.1 Evaporate on a steam-bath 10 ml of ink in a tared porcelain, silica or platinum dish Dry in an air-oven maintained at 105 ± 2°C. Cool the residue in a desiccator and weigh. Repeat the operation till constant mass is obtained. Reserve the residue for determination of iron contents.

B-2.2 Calculation

$$\text{Total solids, percent } (m/v) = 100 \times \frac{M}{V}$$

where

M = mass in g of the residue after drying, and

V = volume in ml of the ink taken for the test.

ANNEX C

(*Table* 1)

DETERMINATION OF IRON CONTENT

C-1 REAGENTS

Standard Sodium Thiosulphate Solution, approximately 0.025 *N*.

Dilute Hydrochloric Acid, approximately 5 N.

Chlorine Water, saturated.

Potassium Iodide Solution, 20 percent (*m/v*), freshly made.

Starch Indicator Solution

Triturate 5 g of starch and 0.01 g of mercuric iodide with 30 ml of cold water and slowly pour it with stirring into 1 litre of boiling water. Boil for 3 minutes. Allow the solution to cool and decant off the supernatant clear liquid.

C-2 PROCEDURE

Ignite the residue as obtained in **B-2.1** at a temperature below 800°C to burn off the organic matter. Guard against overheating which would produce unremovable iron stains or iron particles of difficult solubility. Add 5 ml of dilute hydrochloric acid to the ash, cover with a watch glass and heat on a steam bath till all the iron has dissolved. Add 2 ml of chlorine water and evaporate to dryness. Dissolve the iron salts in 1 ml of dilute hydrochloric acid and dilute to 20 ml with water. Filter and wash, if necessary. Collect the filtrate and washings in an iodine flask. Add 10 ml of potassium iodide solution, stopper the flask, seal the space between the stopper and the neck of the flask with solution of potassium iodide. Place the stoppered flask in a water-bath maintained at about 55°C for one and a half hours and then cool rapidly with cold Water to room temperature. Make up the solution to exactly 100 ml and titrate an aliquot (say, a ml) of the solution with standard thiosulphate solution using starch solution as indicator. Make a blank experiment on the reagents and apply the necessary correction.

C-3 CALCULATION

$$\text{Iron (as Fe), g per 100 ml} = V \times N \times 0{\cdot}05584 \times \frac{1\,000}{a}$$

where

V = volume in ml of the standard sodium thiosulphate solution used,

N = normality of the standard thiosulphate solution, and

a = volume in ml of solution used for the titration.

ANNEX D

(*Table* 1)

DETERMINATION OF RATIO OF ETHYL ACETATE EXTRACT TO IRON CONTENT

D-1 REAGENTS

Concentrated Hydrochloric Acid — *See* IS 265 : 1976.

Potassium Chloride Solution — 16 percent *(m/v)*.

Ethyl Acetate

D-2 PROCEDURE

Evaporate 100 ml of ink in a porcelain basin over steam-bath to a volume of about 10 ml. Transfer it to a 25-ml volumetric flask. Wash the basin with water carefully and transfer it to the volumetric flask, make up the volume to 25 ml and shake. Pipette out 10 ml of this concentrated ink and add 10 ml of concentrated hydrochloric acid keeping the receptacle in cold water. Shake the mixture carefully with 50 ml of ethyl acetate in a separating funnel kept in ice cold water, and separate the ethyl acetate extract from the aqueous solution. Repeat this operation thrice with the same volume of ethyl acetate added each time. Collect the ethyl acetate extract together and shake successively with 10 ml of potassium chloride solution till the ethyl acetate layer is free from iron salts. (This can be found out by taking a drop of potassium chloride extract and treating with a drop of potassium thiocyanate solution. Red colour will indicate the presence of iron.) If necessary, filter the ethyl acetate extract and wash the filter paper with ethyl acetate. Evaporate nearly to dryness in vacuum the original extract together with the washings, the temperature not exceeding 60°C. Transfer carefully the residue from the distilling flask to a tared porcelain dish, washing the last traces with ethyl acetate and evaporate to dryness first at a temperature not exceeding 60°C and afterwards at 100°C in a vacuum desiccator or vacuum oven till the mass is constant.

D-3 CALCULATION

$$\text{Ratio of ethyl acetate extract of iron content} = \frac{2{\cdot}5\,A}{B}$$

where

A = mass in g of the residue obtained, and

B = iron content as obtained in **C-3.**

ANNEX E

(*Table* 1)

SEDIMENTATION

E-1 PROCEDURE

Dry a 10-ml or 25-ml capacity glass centrifuge tube and weigh it accurately. Mix the ink sample and take 10 ml or 25 ml in the centrifuge tube with the help of a pipette and centrifuge for 5 minutes at not less than 1 400 rpm. Take out the tube and drain off the supernatant liquid. Add fresh quantity of 10-ml or 25-ml of water into the tube and centrifuge again. Drain out the liquid again, wipe off the adhering ink inside the tube with the help of filter paper. Dry the tube with the sediment at 100 ± 2°C in hot air oven, cool and weigh. Repeat the process of heating, cooling and weighing till constant mass is obtained.

E-2 CALCULATION

$$\text{Sediment, g/100 ml} = \frac{B-A}{V} \times 100$$

where

B = mass, in g, of the sediment and the centrifuge tube;

A = mass, in g, of the centrifuge tube; and

V = volume, in ml, of the ink sample taken for the test.

ANNEX F

(*Table* 1)

DETERMINATION OF CORROSION

F-1 APPARATUS

Brass Piece

Containing approximately 60 percent copper and 40 percent zinc, having the dimensions approximately 25 mm × 25 mm × 0.3 mm. All the surfaces of the brass piece shall be smooth and well polished.

F-2 PROCEDURE

Thoroughly clean the brass piece with ethyl alcohol and ether. Dry at 105 ± 5°C and weigh accurately to the nearest milligram. Suspend the brass piece by means of a silk thread in 50 ml of ink contained in a beaker. Keep it completely immersed and without touching the beaker for 7 days at room temperature. The beaker shall be suitably covered during all this period. After 7 days, remove the brass piece, wash with water and wipe with soft lint-free cloth. Rinse with alcohol and dry as before to constant mass.

F-3 CALCULATION

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

where

M_1 = loss in mass in g of the brass piece, and

M = mass in g of the brass piece taken for the test.

ANNEX G

(*Table* 1)

TEST FOR HUE AND INTENSITY OF COLOUR, PENETRATION AND PERMANENCE

G-1 PREPERATION OF INK STRIPES

G-1.1 Place a sheet of paper having one minute cobb test value equivalent to cream wove/cream laid paper (*see* IS 1848 : 1981) with the felt side upwards and slightly stretched on a piece of board or glass plate with a smooth surface. Adjust the board or glass plate so that the surface of the paper makes an angle of 45° with the vertical. Draw up 1 ml of ink in a 1-ml pipette and allow the nozzle just to touch the paper near the top edge, the pipette remaining vertical. Release the ink carefully to form a perfect stripe of uniform width across the paper.

G-1.2 The stripe shall not be wavy and there shall be no feathering. The intensity of the stripe shall be uniform throughout and not variable at different places.

NOTE — When arranging the sheet of paper for making the stripes it is desirable to place it so that the ink will flow in the machine direction. There is a slight but definite difference in the arrangement of the fibres in the machine direction and at right angles to it. If a piece of paper about 25 mm square is laid on water, in a moment or two it starts to curl up on two opposite edges and thus makes a shallow trough. The axis on the trough is parallel to the machine direction of the paper. If the ink flows across the machine direction the paper will become wrinkled across the stripes, these will then be unevenly coloured.

G-1.3 Dry the stripe for 15 minutes and remove it from the board. Cut off a strip of paper about 4 cm in width from the bottom of the sheet at right angles to the stripe and reject it. Cut the remaining paper into strips each 5 cm wide and at right angles to the stripe.

G-1.4 Prepare similar stripes with the reference ink (*see* Annex M) on the same sheet.

G-1.5 Dilute separately the ink under test and reference ink with an equal volume of water and repeat the experiment.

G-2 PROCEDURE FOR TESTING HUE, INTENSITY OF COLOUR PENETRATION

G-2.1 Compare the stripes prepared with ink under test and with the reference ink immediately after drying and also after one week's exposure to diffused daylight.

G-2.2 The stripes prepared with the ink under test shall show a close match with those prepared with the reference ink as regards the general form, hue, intensity of colour and penetration when examined from front and back of the paper.

G 3 PROCEDURE FOR TESTING PERMANENCE

G-3.0 Test prescribed in **G-3.1** and **G-3.2** shall be conducted after at least seven days of preparing the stripes.

G-3.1 Ultra-Violet Ray Exposure

Place the papers containing the stripes from ink under test and the reference ink at a distance of 25 cm from an ultra-violet lamp (*see* **G-3.1.1**), normal to the rays, and expose for a total period of 27 hours. Expose the papers containing the stripes from diluted ink and diluted reference ink (*see* **G-1.5**) in the same manner for a total period of 27 hours.

G-3.1.1 The ultra-violet lamp shall be 125 of watts and of long wave UV region, chiefly at 3 655A°.

G-3.1.2 The darkness (intensity) of the stripes from the ink and the diluted ink shall be at least equal to that of the stripes from the reference ink and the diluted reference ink, respectively.

G-3.2 Water and Alcohol Immersion

Immerse vertically the papers containing stripes from the ink and the reference ink for 1 hour in water and examine them at the end of period. Repeat the experiment using rectified spirit *(see* IS 323 : 1959) or methyl alcohol *(see* IS 517 : 1967) or denatured spirit (*see* IS 324 : 1959).

G-3.2.1 Repeat the above test with diluted ink and diluted reference ink.

G-3.2.2 The stripes from the ink and the diluted ink shall not show a greater degree of fading than those from the reference ink and the diluted reference ink, respectively.

ANNEX H

(Table 1)

TEST FOR FREEDOM FROM CLOGGING

H-1 PROCEDURE

H-1.1 Fill a fountain pen which is known to be in good working condition with the ink. Replace the cap and allow it to remain in vertical condition with nib upwards for five days at ambient room temperature. After this period, write with the pen on writing paper.

H-1.2 The ink shall be regarded as free from clogging effect if the pen starts to write without undue pressure and without stopping or other impairment in the ink flow.

ANNEX J

(Table 1)

TEST FOR STABILITY

J-1 APPARATUS

Glass Beaker, Unlipped, 100 ml capacity.

J-2 PROCEDURE

J-2.1 Take 50 ml of the ink in an unlipped beaker of 100 ml capacity, Mark the level of the liquid, cover it with a watch glass and leave for 15 days in a place uncontaminated by dust and chemical fumes. Examine for moulds and ilakes after this period.

J-2.2 Make up the volume with water and stir. Allow to stand for 24 hours and filter. The ink shall remain non-gelatinous.

ANNEX K

(*Clause* 6.1)

SAMPLING AND CRITERIA FOR CONFORMITY

K-1 GENERAL REQUIREMENTS OF SAMPLING

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

K-1.1 Samples shall be drawn from original unopened bottles.

K-1.2 Samples shall not be taken in an exposed place.

K-1.3 Unless otherwise stated in any test method, sample ink from each of the selected bottles shall be drawn after shaking and then allowing the fluid to come to rest.

K-1.4 Each of the sample containers shall be marked with full details of sampling and the month and year of the manufacturer of the material.

K-2 SCALE OF SAMPLING

K-2.1 All the bottles in a single consignment of the material and containing ink of the same type, colour and composition shall be grouped together to form a lot.

K-2.2 Samples of ferro-glass tannate fountain pen ink shall be tested from each lot separately for ascertaining the conformity of the material to the requirements of the specification.

K-2.3 The number of bottles (*n*) to be chosen from the lot (*N*) depends upon the size of the lot and shall be in accordance with Table 2.

K-2.4 These bottles shall be chosen at random from the lot. In order to ensure randomness of selection some random number table may be used. For guidance to random selection procedures, reference may be made to IS 4905 : 1968.

Table 2 Number of Bottles to be Selected for Sampling

(*Clause* K-2.3)

Lot Size *N*	No. of Bottles to be Chosen *n*
Up to 25	3
26 to 50	4
51 to 100	5
101 to 300	7
301 to 500	9
501 and above	12

K-3 NUMBER OF TESTS

K-3.1 Ink from each of the selected bottle shall be tested for general requirements (*see* **3.1**) and sediment.

K-3.2 For testing all the other characteristics, a composite sample shall be prepared by mixing equal volume (about 40 ml) of ink from each of the selected bottles. From this composite sample, required quantity of ink shall be drawn for conducting various tests prescribed in Table 1, excluding test for sediment.

K-4 CRITERIA FOR CONFORMITY

The lot shall be declared to have complied with the requirements of the specification if all the individual samples (**K-3.1**) and the composite sample (**K-3.2**) pass the relevant tests.

ANNEX M

(*Clause* G-1.4)

METHOD OF PREPARATION OF REFERENCE INK

M-1 COMPOSITION

Reference ink to be used shall be prepared according to the following formula:

Tannic acid (*see* Note 1)	4.0 g
Gallic acid (*see* Note 1)	1.5 g
Ferrous sulphate crystals (*see* IS 262 : 1982 and Note 2)	5.5 g
Concentrated hydrochloric acid (*see* IS 265 : 1987)	5.0 g
Dye, ink blue (*see* IS 8642 : 1977)	5.0 g
Provisional dye (for inks other than blue black) (*see* Note 3)	As advised by supplier
Phenol (*see* IS 538 : 1968)	
Distilled water	To make up the total volume to one litre

NOTES

1 Tannic acid and gallic acid shall conform to the following requirements:

Material	*Characteristic*	*Requirement*
a) Tannic acid	1) Tannic acid, percent by mass of the dried material, *Min*	95
	2) Loss on drying at 100°C, percent by mass, *Max*	12
	3) Ash, percent by mass, *Max*	0.2
	4) Insoluble matter, percent by mass, *Max*	0.2
b) Gallic acid	1) Gallic acid, percent by mass of the dried material, *Min*	88
	2) Loss on drying at 100°C, percent by mass, *Max*	10
	3) Ash, percent by mass, *Max*	0.2
	4) Insoluble matter, percent by mass, *Max*	0.2

2 It is important that the ferrous sulphate crystals conform to the formula $FeSO_4$, $7H_2O$. When exposed to the air, ferrous sulphate crystals lose part of their water of crystallization and are no longer correctly represented by the formula $FeSO_4$, $7H_2O$. The loss in weight may be as much as 20 percent of the weight of crystals that have the correct formula. Ink made with this weathered salt is likely to deposit sediment in a short time.

3 In case of inks other than blue black, for the purpose of easy comparison it is necessary to add the same percentage of the same dye as is added to the ink under test. Therefore, the same quantity of the dyestuff as advised by the ink manufacturer should be added to the base of reference ink prepared in **M-1.1**. In case the manufacturer does not wish to declare the exact dyes used by him, he may supply along with the tender sample, sufficient amount of dye (or mixture of dyes) with the directions regarding the mass that is to be dissolved in one litre of base.

M-2 PROCEDURE

M-2.1 Dissolve gallic acid and tannic acid in 300 ml of water heated to about 60°C in a beaker. Filter the solution while hot into one-litre flask, washing the filter paper and the beaker with hot water. Separately dissolve ferrous sulphate in minimum quantity of water in a beaker and add concentrated hydrochloric acid and then mix the two solutions. Dissolve in another beaker phenol in small quantity of water and transfer it to the solution. Cool the mixture to room temperature and add to it a solution of the dye. Shake the flask to mix the dye solution thoroughly and then add water to bring the total volume to one litre. Allow it to stand overnight and then filter through a Buchner filter.

NOTE — In order to exclude air, the bottles should be filled as completely as possible.

M-2.1.1 The reference ink shall not be used for more than one month after the date of preparation and shall be stored in amber-coloured reagent bottles (*see* IS 1388 : 1959).

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Revision of Indian Standards

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