भारतीय मानक
Indian Standard

IS 10350 : 2020

हेयर डाई पाउडर — विशिष्ट

(तीसरा पुनरीक्षण)

Powder Hair Dyes — Specification

(Third Revision)

ICS 71.100.70

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Cosmetics Sectional Committee, PCD 19

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

In general, hair dyes may be broadly classified as, powder hair dyes and liquid/gel/cream hair dyes. Liquid/ gel/ cream hair dyes, however, may be further classified into the four types, namely:

a) oxidation hair dyes (liquid/ gel/ cream);
b) lead salt-based hair darkeners;
c) emulsion type hair dyes; and
d) vegetable based hair dyes.

The requirements pertaining to liquid/gel/cream hair dyes are covered in IS 8481 : 2019 'Oxidation hair dyes "liquid/gel/cream" — Specification (*fourth revision*)'. Emulsion type hair dyes are covered in IS 15205 : 2002 'Oxidation hair dyes (emulsion type) — Specification'.

This standard covers the requirements for powder hair dyes/colours, which contain synthetic dye ingredients. A powder hair dye may contain an arylamine, for example, *p*-phenylenediamine (PPD) or its analogue, salt or related compound or direct synthetic colour as the active ingredient and solid peroxide as the oxidizing agent. Powder hair dyes containing only direct dyes may not contain oxidizing agent.

This standard was first published in 1982. After gaining the experience on the subject, the Committee revised this standard in 1993 incorporating two types of powder hair dyes based on shades in vogue in the country. In addition, the lower limit for dye content was considered essential in order to safeguard consumer's interest and get him his money's worth whereas an upper limit was fixed to allow only a safe dye concentration in the ready to use product in the market. A new requirement for active matter in the dye ready for use, prepared after recommended dilution with water as per manufacturer's instructions was also added. The marking clause was modified to include expiry date, declaration of ingredients, batch number, manufacturing date and minimum PPD content for dyes in powder form to be declared.

In the second revision (1999), the requirements of active matter as PPD content had been modified for both Type 1 and 2 of powder hair dyes as well as for dye ready for use. The test method for dye content was also modified.

In this revision, following major changes have been carried out:

a) A new type of powder hair dyes, namely, Type 3 (others) has been included to provide flexibility to the manufacturers to introduce new shades which could be formulated with nil or significantly lower levels of PPD and/or using other permissible dye intermediates.
b) The marking clause has been harmonized with Rule 148 of the Drugs and Cosmetics Rules, 1945.
c) In the marking clause, cautions/warnings related to PPD content in the product have been aligned with IS 4707 (Part 2). Further, 12 local languages for primary patch test instructions have been listed [**5.3.2(d)**].
d) A new method for estimation of PPD content in salt form (Annex C) has been incorporated.
e) HPLC method for estimation of PPD (Annex D) has been incorporated as alternate method.
f) A new Annex E for estimation of picramic acid by HPLC method has been incorporated.

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 '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.

Indian Standard

POWDER HAIR DYES — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for powder hair dyes.

2 REFERENCES

The following Indian Standards contain provisions, which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
2088 : 1983	Methods for determination of arsenic (*second revision*)
3958 : 1984	Methods of sampling cosmetics (*first revision*)
4011 : 2018	Methods of test for safety evaluation of cosmetics (*third revision*)
4707	Classification of cosmetic raw materials and adjuncts
(Part 1) : 2017	Colourants (*third revision*)
(Part 2) : 2017	List of raw materials generally not recognized as safe for use in cosmetics (*fourth revision*)

3 TYPES

There shall be three types of the powder hair dyes, namely:

a) *Type 1* — Black;
b) *Type 2* — Brown; and
c) *Type 3* — Others (covering entire range of fashion hair colours like black, brown, red, blonde, purple etc.).

4 REQUIREMENTS

4.1 The powder hair dye shall be fine and free flowing.

4.2 Ingredients

Unless specified otherwise all the raw materials, used in the manufacture of powder hair dye shall conform to the requirements prescribed in the relevant Indian Standards where such standards exist.

4.3 Ingredients of dye and developer shall comply with the provisions of IS 4707 (Part 1) and IS 4707 (Part 2) subject to the provisions of *The Drugs and Cosmetics Act*, 1940 and Rules, 1945 framed thereunder.

4.4 For safety evaluation of novel ingredients used in formulation of powder hair dye, it shall comply to IS 4011.

4.5 The powder hair dye shall also comply with the requirements given in Table 1 when tested as prescribed in column 6 of Table 1.

4.6 Dye Ready for Use

The dye ready for use is prepared after mixing the powder hair dye with water or solution as recommended or provided by the manufacturer. The dye ready for use shall comply with the requirement given in Table 2 when tested as prescribed in column 6 of Table 2.

4.6.1 The procedure for calculation of *p*-phenylenediamine (PPD) content in dye ready for use is as follows:

If PPD content in powder hair dye is x percent and manufacturer recommends that 1 part of dye may be mixed with y part of water or diluent supplied by the manufacturer then:

$$\text{PPD content in the dye ready for use} = \frac{x}{y+1}$$

5 PACKING AND MARKING

5.1 Packing

The powder hair dye shall be packed in suitable containers.

5.2 Storage

Material shall be stored such that it is protected from heat and sunlight.

5.3 Marking

5.3.1 Each container and the carton containing the material shall be legibly marked with the following information:

a) Name of the material;
b) Shade of powder hair dye;
c) Name and address of the manufacturer;
d) Net content;
e) Month and year of manufacture (MM/YY);
f) Use before……. (Month and year MM/YY, or months/years from the date of manufacture) to be declared by the manufacturer;
g) Declaration 'Maximum arylamine (PPD) content after dilution as per manufacturer's instructions for use' (To be declared by manufacturer);
h) Minimum PPD content for dye in powder form;
j) Batch number;
k) List the ingredients (at the time of manufacture) under the title 'Ingredients' as follows:
   1) For ingredients more than 1 percent (by mass or volume) — List the ingredients in decreasing order of percentage.
   2) For ingredients less than 1 percent (by mass or volume) — List the ingredients in any order.

   NOTE — This is exempted in case of pack sizes less than 30 g of solid/semi-solid and 60 ml of liquid.
m) Contains Phenylenediamines, and/or any other ingredient as required to be mentioned by IS 4707 (Part 2);
n) The mixing ratio of the dye-to-water for preparation of dye ready for use to be declared by the manufacturer;
p) Warning "This product shall not be used for dyeing eyelashes or eyebrows as such, a use may cause severe inflammation of the eye or even blindness"
q) Following cautions;
   1) Keep out of reach of children.
   2) This product is not intended for use on persons under the age of 16.

Table 1 Requirements for Powder Hair Dyes

(*Clause* 4.5)

Sl No.	Characteristics	Requirement			Method of Test, Ref to Annex
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	*p*H of 5 percent (m/m) solution in water	6-10	6-10	4-10	A
ii)	Active matter as PPD content, percent by mass (*m/m*), *(including PPD and their derivatives, their salts, m and p-Phenylenediamines, their N-substituted derivatives and their salts, and N-substituted derivatives of o-Phenylenediamine)*	3 - 30	1 - 20	0-30	B (for PPD content)/ C (for PPD content in its salt form) } or D[1]
iii)	Dye ingredients *(includes lower or nil levels of PPD and/or phenylenediamine including their N-substituted derivatives and their salts and/or Toluenediamine and derivatives and their salts, other permissible dye intermediates, couplers and modifiers, aminophenols, resorcinols, picramic acid and its salts and all permitted dye chemicals)*	—	—	Present	B (for PPD content)/ C (for PPD content in its salt form)/ E (for picramic acid)/ F (dye ingredient by TLC) } or D[2]
iv)	Heavy metals (as Pb), parts per million, *Max*	20	20	20	G
v)	Arsenic (as As_2O_3), parts per million, *Max*	2	2	2	H

[1] In case of any dispute, method of test prescribed at Annex B/C shall be the reference method.
[2] In case of any dispute, method of test prescribed at Annex B/C/E/F shall be the reference method.

Table 2 Requirement for Hair Dye Ready for Use

(*Clause* 4.6)

Sl No.	Characteristic	Requirement			Method of Test, Ref to Clause
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
(i)	Calculated active matter (as PPD) in the solution after recommended dilution with water/diluent, percent by mass, *Max*	3	1.5	3	Procedure for calculation as given in **4.6.1**

3) This product contains ingredients which may cause skin irritation in certain cases and so a preliminary test according to the accompanying direction should first be made.

r) Any other information required by statutory authorities.

5.3.2 In addition to the above, the following information shall also be given (in the leaflet which is inserted in the container packing of the dye, or may be printed on the carton itself, as the case may be):

a) Procedure for conducting preliminary test for sensitivity (patch test);

b) Instructions for use (may be given in attached leaflet);

c)  Hair colorants can cause severe allergic reactions. Read and follow instructions. Temporary 'black henna' tattoos may increase your risk of allergy. Do not colour your hair if:

- you have a rash on your face or sensitive, irritated and damaged scalp,
- you have ever experienced any reaction after colouring your hair,
- you have experienced a reaction to a temporary 'black henna' tattoo in the past.

d) Each package shall contain instructions in 12 languages, namely, English, Hindi, Marathi, Gujarati, Punjabi, Bengali, Tamil, Kannada, Malayalam, Telugu, Urdu and Oriya on the outer pack or accompanying leaflet on the following lines for carrying out the test:

'This preparation may cause serious inflammation of the skin in some cases and so a preliminary test should always be carried out to determine whether or not special sensitivity exists. For carrying out the test, cleanse a small area of skin behind the ear or upon the inner surface of the forearm, using either soap or water or alcohol. Apply a small quantity of the hair dye as prepared for use to the area and allow it to dry. After 24 h, wash the area gently with soap and water. If no irritation or inflammation is apparent, it may be assumed that no hypersensitivity to the dye exists. The test should, however, be carried out before each and every application.'

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 3958.

6.2 Tests for all the requirements shall be carried out on a composite sample.

6.3 The material shall be taken to have conformed to this standard if the composite sample passes all the tests.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water [*see* IS 1070 : 1992 Reagent grade water (*third revision*)] shall be employed in the tests.

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

ANNEX A

[*Clause* 4.5, and *Table* 1, *Sl No*. (i)]

DETERMINATION OF *p*H

A-1 APPARATUS

A *p*H meter preferably equipped with glass electrode.

A-2 PROCEDURE

Make 5 percent solution (*m/m*) of powder hair dyes at 27 ± 2°C. Determine its *p*H after stabilization using the *p*H meter.

ANNEX B

[*Clause* 4.5, *Table* 1, *Sl No*. (ii), (iii)]

DETERMINATION OF ARYLAMINE CONTENT

B-1 OUTLINE OF THE METHOD

This method estimates arylamine as diacetyl derivative of arylamine.

B-2 APPARATUS

B-2.1 Continuous Extraction Apparatus, as in Fig. 1.

B-2.2 G4 Sintered Glass Crucible

B-2.3 Beaker, 250 ml capacity.

B-3 REAGENTS

B-3.1 Chloroform, reagent grade.

B-3.2 Acetic Anhydride, analytical reagent grade.

B-4 PROCEDURE

Transfer accurately weighed quantity (1 to 2 g) of hair dye, so as to contain 0.1 to 0.3 g PPD, to the inner tube of the continuous extractor, previously charged with chloroform. Take 60 ml chloroform in the flask and completely extract the dye. About 5 h extractions is sufficient. Remove the flask and transfer chloroform extract to a 250 ml beaker, rinsing the flask with few small portions of chloroform. Evaporate chloroform to about 25 ml and add 1 ml acetic anhydride slowly, with stirring. Let it stand for 1 h and filter on a weighed G4 sintered glass crucible. Wash the beaker and precipitate with three or four 5 ml portions of chloroform. Carefully remove last traces of precipitate from the beaker. Dry to constant mass at 120°C and weigh the precipitate of diacetyl-*p*-phenylenediamine.

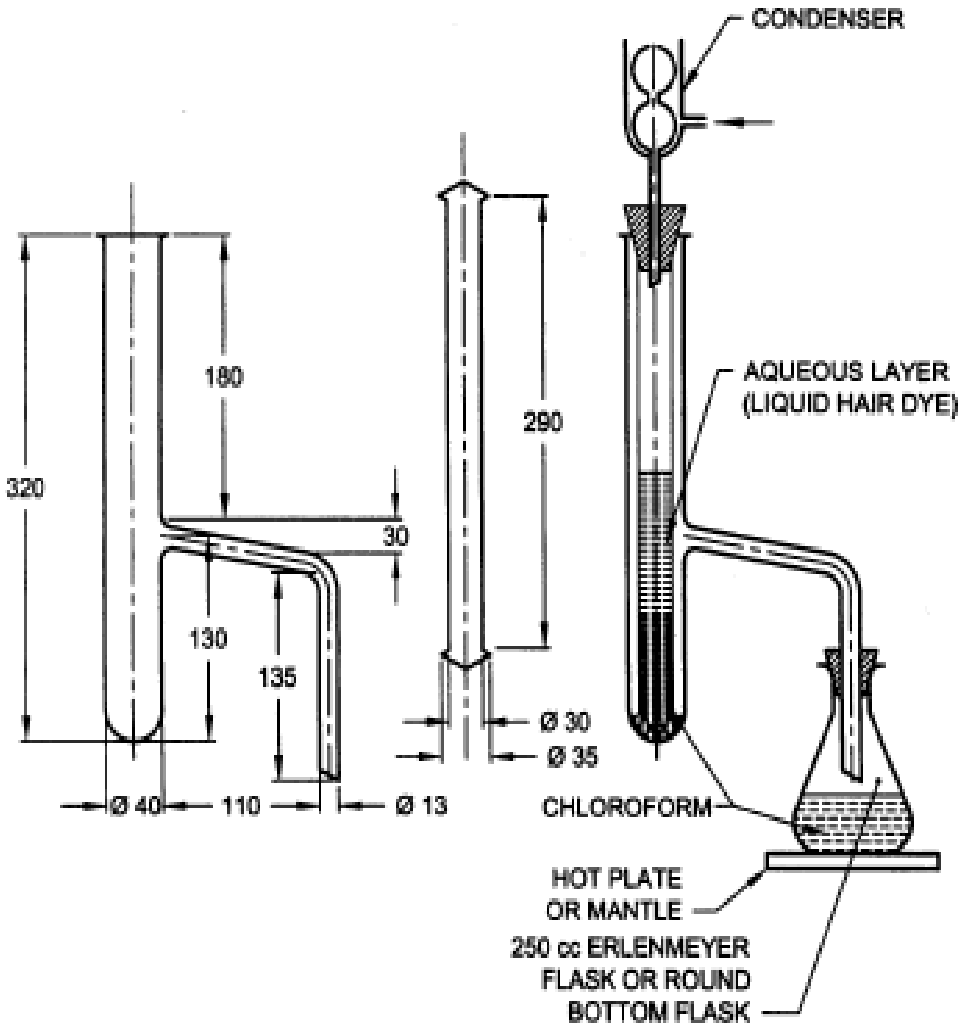
B-5 CALCULATION

$$\text{Arylamine content (as PPD)} = \frac{m \times 0.5626 \times 100}{M}$$

Where,

m = mass in g, of the precipitate; and

M = mass in g, of the hair dye taken for extraction.



All dimensions in millimetres.

FIG. 1 CONTINUOUS EXTRATION APPRATUS

ANNEX C

[*Clause* 4.5, *Table* 1, *Sl No*. (ii), (iii)]

DETERMINATION OF PPD CONTENT IN SALT FORM

C-1 OUTLINE OF THE METHOD

This method estimates the PPD as diacetyl derivative of *p*-phenylenediamine from a product if it is present in its bound state as a salt.

C-2 APPARATUS

C-2.1 Flat Bottom Flask with Stopper, 250 ml.

C-2.2 Beaker, 500 ml capacity.

C-2.3 G4 Sintered Glass Crucible

C-2.4 Whatman Filterpaper-40

C-2.5 Measuring Cylinder, 10 ml.

C-2.6 Magnetic Stirrer

C-3 REAGENTS

C-3.1 Chloroform, reagent grade

C-3.2 Ammonia Solution, 30 percent

C-3.3 Anhydrous Sodium Sulphate

C-4 PROCEDURE

Accurately weigh sufficient product containing 0.3-0.5 g of p-phenylenediamine salt in 250 ml flat bottom flask with stopper, add 10 ml of ammonia solution (**C-3.2**) and 100 ml of chloroform. Stir for 30 min using a magnetic stirrer at the temperature of 27 ± 2°C. Filter the chloroform layer through anhydrous sodium sulphate layer using Whatman filter paper 40. Add 50 ml of chloroform to the residue and stir it on magnetic stirrer for 10 min. Filter through anhydrous sodium sulphate.

Tare a 500 ml beaker and glass rod to be used for evaporation along with the G4 sintered glass crucible. Collect the chloroform extracts into the tarred beaker and evaporate over a water bath to a volume of about 25 ml. Cool the extract to room temperature, add 2 ml of acetic anhydride slowly while stirring. Allow this mixture to stand in a dark place for half an hour. Filter the precipitate through previously weighed G4 sintered glass crucible. Wash the precipitate with chloroform. Dry the set at 120°C to constant mass and weigh.

C-5 CALCULATION

$$\text{PPD, percent by mass} = \frac{m \times 0.5626 \times 100}{M}$$

Where,

m = mass in g, of the precipitate; and

M = mass in g, of the hair dye taken for extraction.

ANNEX D

[*Clause* 4.5, *Table* 1, *Sl No.* (ii) *and* (iii)]

IDENTIFICATION AND ESTIMATION OF DYE INGREDIENTS BY HPLC

D-1 OUTLINE OF THE METHOD

This method estimates the PPD content and other precursors, couplers and colourants using high performance liquid chromatography.

D-2 APPARATUS

D-2.1 HPLC with UV Detector

D-2.2 Volumetric Flask , 100 ml, 50 ml and 25 ml.

D-2.3 Glass Rod

D-2.4 Nylon Syringe Filter (0.45µ)

D-2.5 Ultrasonic Bath

D-3 REAGENTS AND CHEMICALS

D-3.1 Water, HPLC grade

D-3.2 Ammonium Acetate, analytical reagent grade

D-3.3 Methanol, HPLC grade

D-3.4 Ascorbic Acid (Vitamin C), reagent grade

D-3.5 Monoethanolamine or Ammonia, analytical reagent grade

D-4 CHROMATOGRAPHIC CONDITIONS

Column	C18 AQ (150 mm × 4.6 mm × 5 µm)
Flow rate	0.8 ml/min
Injection volume	20 µl
Column oven temperature	25ºC
Wavelength	280 nm

D-5 PREPARATION OF MOBILE PHASE

D-5.1 Preparation of 0.02 M Ammonium Acetate Solution

Weigh accurately and transfer 1.541 g of ammonium acetate to 1 000 ml water and sonicate to dissolve. Filter through 0.45 µ nylon filter.

D-5.2 Preparation of Mobile Phase

Mix 0.02 M ammonium acetate and methanol in the ratio of 83 : 17 (*v/v*) and sonicate for 15 min.

D-6 PREPARATION OF DILUENT

D-6.1 Preparation of Diluent

Mix, water and methanol in the ratio of 80 : 20 (*v/v*). Add 0.1 g of ascorbic acid and sonicate to dissolve.

NOTE — Use freshly prepared diluent for analysis.

D-6.2 Preparation of Methanolic Monoethanolamine (MEA) or Ethanolamine Solution (20 Percent, *v/v*)

Transfer accurately 20 ml of ethanolamine into a 100 ml volumetric flask and make up to volume with diluent and mix well.

D-7 PREPARATION OF BLANK SOLUTION

Transfer accurately 1 ml of 20 percent methanolic ethanolamine to 25 ml volumetric flask. Make up to the volume with diluent and mix well.

D-8 PREPARATION OF STANDARD STOCK SOLUTION

D-8.1 Preparation of Standard Stock Solution for Determination of Active Matter as PPD Content

D-8.1.1 *Preparation of Standard Stock Solution 'A'*

Weigh accurately and transfer 0.176 g of PPD into 50 ml volumetric flask, add about 35 ml of diluent, mix. Add 2 ml of 20 percent methanolic ethanolamine (**D-5.2**), sonicate to dissolve. Make up to the volume with diluent, mix well.

D-8.1.2 *Preparation of Standard Solution (about 700 ppm)*

Pipette out 5 ml from standard stock solution 'A' (**D-7.1.1**) and transfer to 25 ml volumetric flask. Make up to volume with diluent and mix well.

NOTES:

1 Use freshly prepared standard solution for analysis.

2 Make necessary dilutions of the above solutions if required, to achieve concentrations in the same range as expected in the sample.

D-9 PREPARATION OF SAMPLE SOLUTION

Weigh accurately about 1.2 g of powder sample and transfer to an appropriate volumetric flask, add diluent to about 70 percent of total volume of the volumetric flask, mix well. Add 20 percent MEA solution about 4 percent of total volume of volumetric flask

and sonicate for about 15-20 min with intermittent swirling. Cool the solution at room temperature and make up the volume with diluent. Mix well, keep the solution standing still for 5 min then filter the supernatant through nylon syringe filter and inject into the HPLC system.

If required, make necessary dilutions of the above solution to achieve the final concentration equal to the standard concentration using diluent.

NOTES:

1 Ascorbic acid peak may undergo oxidation, hence, it may be observed that the area of ascorbic acid peak gradually decreases or vanishes in the standard and sample solution. Since it is blank (diluent) peak, it need not be integrated in standard and in sample solutions.

2 Alternative validated test conditions and standard/sample preparation steps are acceptable in order to improve/ achieve clear separation and/or quantification when several precursors/couplers/colorants are present. Responsibility of changes in test conditions lies with manufacturer provided suitably validated for type and concentration of dyes added in compliance with IS 4707 (Part 1 and 2) to suit separation and identification of probable dyes added and approved in formulation.

D-10 PROCEDURE

D-10.1 Saturate the column before analysis for about 60 min to achieve the stable baseline. Inject the freshly prepared solution as per following sequence outlined below.

Sl No.	*Name of Solution*	*Number of Replicates*
(1)	(2)	(3)
i)	Blank solution	01
ii)	Standard solution	03
iii)	Sample solution – 1	01
iv)	Sample solution – 2	01
v)	Sample solution – 3	01

D-10.2 Guide retention time and relative retention time as follows:

Name of Peak	*Guide Retention Time (RT)*	*Guide Relative Retention Time (RRT)*
Ascorbic acid	1.99	----
PPD	3.06	1.54

D-11 CALCULATIONS

PPD, percent by mass =

$$\frac{\text{Sample area} \times \text{Standard weight (g)} \times \text{Sample dilution} \times \text{Percent purity of standard}}{\text{Standard area} \times \text{Standard ailution} \times \text{Sample weight (g)}}$$

ANNEX E

[*Clause* 4.5, *Table* 1, *Sl No.* (iii)]

IDENTIFICATION AND ESTIMATION OF PICRAMIC ACID BY HPLC

E-1 OUTLINE OF THE METHOD

This method estimates the picramic acid content using high performance liquid chromatography.

E-2 CHROMATOGRAPHIC CONDITIONS

Column	C18 AQ (250 mm × 4.6 mm × 5 µ particle size)
Flow rate	1.5 ml/min
Injection volume	20 µl
Column oven temperature	27 ± 2ºC
Wavelength	272 nm
Run time	15 min

E-3 PREPARATION OF MOBILE PHASE

E-3.1 Preparation of 0.01 M of Sodium Butane Sulphonate

Dissolve 1.6017 g of sodium butane sulphonate in 1 000 ml water.

E-3.2 Solution A

Take 1 000 ml of 0.01 M sodium butane sulphonate and add 4 ml of formic acid. Mix the solution A properly.

E-3.3 Solution B

Acetonitrile (100 percent, HPLC grade)

E-3.4 Mobile Phase

Mix Solution A and Solution B in the ratio 70 : 30. Filter, degas and use as the mobile phase.

E-4 PREPARATION OF DILUENT

Mobile phase A : Methanol :: 80 : 20

E-5 PREPARATION OF SOLUTIONS

E-5.1 Standard Preparation

Weigh accurately and dissolve about 37.5 mg of picramic acid standard into 50 ml volumetric flask, make up the volume with methanol. Further, dilute 1 ml of it into 50 ml volumetric flask and make up the volume with diluent.

E-5.2 Preparation of Sample Solution

Weigh accurately and dissolve about 0.75 g of test sample into 50 ml volumetric flask and make up the volume with methanol. Sonicate the resulting solution, mix well and keep aside for settling. Further dilute 1 ml of it into 50 ml volumetric flask and make up the volume with diluent.

E-6 PROCEDURE

E-6.1 Separately inject equal volume (20 µl) of blank, standard preparation in triplicate and sample preparation each into the chromatograph, record the chromatogram and measure the peak response for picramic acid. Retention time of picramic acid is about 9.0 min.

E-6.2 Evaluate the system suitability parameter from the peak response of picramic acid obtained in the chromatogram of standard solution.

Sl No.	*System Suitability Parameters*	*Acceptance Criteria*
(1)	(2)	(3)
i)	Tailing factor of picramic acid	≤ 1.5
ii)	Theoretical plate of picramic acid	≥ 2 000
iii)	Percent relative standard deviation of peak response of picramic acid.	≤ 2 percent

E-7 CALCULATIONS

Calculate percent of picramic acid in sample using the following formula:

$$\text{Picramic acid content, percent } (w/w) = \frac{A_{SAM} \times D_{STD} \times 100}{A_{STD} \times D_{SAM}}$$

Where,

A_{SAM} = Area of peak response due to picramic acid in sample preparation;

A_{STD} = Mean area of peak responses due to picramic acid in standard preparation;

D_{STD} = Dilution factor of standard preparation (mg/ml);

D_{SAM} = Dilution factor of sample preparation (mg/ml); and

P = Purity of picramic acid Standard.

ANNEX F

[*Clause* 4.5, *Table* 1, *Sl No.* (iii)]

IDENTIFICATION AND ESTIMATION OF DYE INGREDIENT BY TLC

F-1 OUTLINE OF THE METHOD

This method identifies and estimates the oxidative/ sulphated dyes in powder hair dye using TLC.

F-2 APPARATUS

F-2.1 Weighing Balance

F-2.2 TLC Plates, pre-coated silica gel 60 F_{254}, 0.2 mm thickness.

F-2.3 TLC Apparatus/Beakers, 250 ml (narrow).

F-2.4 Iodine Chamber

F-2.5 Test Tubes with Stoppers

F-2.6 Pipette, 10 ml.

F-2.7 Syringe, 5 or 10 µl capacity.

F-2.8 Silicone Wax

F-3 REAGENTS

F-3. 1 Toluene, analytical reagent grade.

F-3.2 Ethyl Acetate, analytical reagent grade.

F-3.3 Methanol, analytical reagent grade.

F-3.4 Sodium Sulphite, analytical reagent grade.

F-3.5 Monoethanolamine (MEA)

F-3.6 Standards, expected dye precursors/ingredients in the respective shade being analyzed.

F-3.7 Test Sample, 1 g of dye being tested.

F-4 PREPARATION OF SOLUTIONS

F-4.1 Mobile Phase

Toluene : Ethyl acetate : Methanol :: 50 : 40 : 10 (*v/v*). Mix these well in the stated dilution and keep stoppered.

F-4.2 Sample Solutions

Weigh accurately 0.05 g of sample from the shade being tested. Add 0.15 g of sodium sulphite, 4 ml of methanol (**F-3.3**) and mix well. Use this solution for spotting. For oxidation dyes in salt form, add 0.1 ml MEA to this solution.

F-4.3 Standard Solutions

Weigh quantity nearly sufficient of the standard dye precursors/ ingredients expected in the respective shade being analyzed, add 0.15 g sodium sulphite and dissolve in 4 ml of methanol (**F-3.3**). Add 0.1 ml MEA to this solution.

Combine 1 ml of each of the standard solutions to prepare the mixed standard solution.

F-5 PROCEDURE

F-5.1 Pour 15-20 ml of mobile phase into the 250 ml narrow beaker (developing chamber) and cover with a suitable petridish using silicone wax as sealant.

F-5.2 Spot 1 µl of the test solution slowly on the TLC plate. Similarly, spot the standard solutions as mentioned above. Allow spots to dry and develop in the developing chamber. Remove the TLC plate when the mobile phase reaches 0.5 cm away from the end of the TLC plate. Allow the plate to dry completely and develop the spots in an Iodine chamber. See and compare the R_f values, colour, shape and size of the sample spots versus the standard spots to identify and determine the presence of dye ingredients.

NOTE — If the concentration of some ingredient is too small for proper identification and characterization the sample and standard concentrations may be increased accordingly.

ANNEX G

[*Clause* 4.5, *Table* 1, *Sl No.* (iv)]

TEST FOR HEAVY METALS

G-1 OUTLINE OF THE METHOD

The colour produced with hydrogen sulphide solution is matched against, that obtained with standard lead solution.

G-2 APPARATUS

G-2.1 Nessler Cylinders, 50-ml capacity.

G-3 REAGENTS

G-3.1 Dilute Hydrochloric Acid, approximately 5 N.

G-3.2 Dilute Acetic Acid, approximately 1 N.

G-3.3 Hydrogen Sulphide Solution, standard.

G-3.4 Standard Lead Solution

Dissolve 1.600 g of lead nitrate in water and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One milliliter of this solution contain 0.01 mg of lead (as Pb).

G-4 PROCEDURE

G-4.1 Weigh about 2.000 g of material in a crucible and heat on a hot plate and then in a muffle furnace to ignite it at 600°C to constant mass. Add 3 ml of dilute hydrochloric acid, warm (wait till no more dissolution occurs) and make up the volume to 100 ml. Filter the solution. Transfer 25 ml of the filtrate into a Nessler's cylinder. In the second Nessler's cylinder, add 2 ml of dilute acetic acid, 1.0 ml of standard lead solution and make up the volume with water to 25 ml.

G-4.2 Add 10 ml of hydrogen sulphide solution to each Nessler cylinder and make up the volume with water to 50 ml. Mix and allow to stand for 10 min. Compare the colour produced in the two Nessler's cylinders. Blank determination without samples are recommended to avoid errors arising out of reagents.

G-5 RESULTS

The sample may be taken to have passed the test, if the colour developed in the sample solution is less than that of standard solution.

ANNEX H

[*Clause* 4.5, *Table* 1, *Sl No.* (v)]

DETERMINATION OF ARSENIC

H-1 OUTLINE OF THE METHOD

Arsenic present in a solution of the material is reduced to arsine, which is made to react with mercuric bromide paper. The stain produced is compared with a standard stain.

H-2 REAGENTS

H-2.1 Mixed Acid

Dilute one volume of concentrated sulphuric acid with four volumes of water. Add 10 g of sodium chloride for each 100 ml of the solution.

H-2.2 Ferric Ammonium Sulphate Solution

Dissolve 64 g of ferric ammonium sulphate in water containing 10 ml of mixed acid and make up to one liter.

H-2.3 Concentrated Hydrochloric Acid [*see* IS 265 : 1993 'Hydrochloric acid — Specification (*fourth revision*)']

H-2.4 Stannous Chloride Solution — Dissolve 80 g of stannous chloride ($SnCl_2$, $2H_2O$) in 100 ml of water containing 5 ml of concentrated hydrochloric acid.

H-3 PROCEDURE

Carry out the test as prescribed in IS 2088, adding into the Gutzeit bottle, 2 ml of ferric ammonium sulphate solution, 0.5 ml of stannous chloride solution and 25 ml of sample solution as prepared in **G-4.1**.

For comparison, prepare a stain using 0.001 mg of arsenic trioxide.

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