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Indian Standard

IS 8481 : 2020

खिजाब (तरल, जैल और क्रीम) — विशिष्टि
(चौथा पुनरीक्षण)

**Oxidation Hair Dyes (Liquid / Gel /
Cream) — Specification**
(Fourth Revision)

ICS 71.100.70

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FOREWORD

This Indian Standard (Fourth 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. The requirements pertaining to powder hair dyes are covered in IS 10350 : 2019 'Powder hair dyes — Specification (Third revision)'.

Liquid/gel/ cream hair dyes, however, may be further classified into the following types:

- a) Oxidation hair dyes (liquid/ gel/ cream);
- b) Lead salt based hair darkeners;
- c) Emulsion type hair dyes; and
- d) Vegetable based hair dyes.

This standard covers only oxidation hair dyes (liquid/gel/cream) based on oxidative hair dyes/precursors and couplers in combination with suitable developers in liquid/gel/cream form. Emulsion type hair dyes are covered in IS 15205 : 2002 'Oxidation hair dyes (emulsion type) — Specification'.

This standard was first published in 1977. In the first revision, two types of oxidation hair dyes, on the basis of shades in vogue in the country were incorporated. In addition, requirement for *p*-phenylenediamine (PPD) was revised, specifying a range, thereby prescribing lower as well as upper limit for PPD content. Fixing of lower limit for PPD content was considered essential in order to safeguard consumer's interest to enable him to get a dye that would perform and is money's worth whereas upper limit was fixed to allow only a safe dye in the market. Also, a new requirement for PPD in the dye, ready for use prepared after formulation with developer as per manufacturer's instructions, was prescribed with a procedure to calculate the same. Marking clause was elaborated, according to which it was made mandatory for the manufacturers to declare PPD content in liquid dye, instructions for preparation of dye ready for use, besides warning, declaration of other relevant information and precautions. "Best use before" was prescribed as a regular requirement and declaration of list of key ingredients on the carton/package of dyes was made compulsory, in line with other cosmetic formulations.

In the second revision, requirement of PPD content in the dye was modified. Accordingly, requirement for calculated active matter was also modified for Type 2. The requirement for assay (as hydrogen peroxide) for developer was also modified keeping in view the latest technological development in the field. Modifications were also made in the marking clause.

In the third revision, a new classification of colours, namely, Type 3 (Others) was added in order to cover an entire range of fashion hair colours like black, brown, red, blonde, purple, etc., which could be formulated with nil or significantly lower levels of PPD and/or by using other permissible dye intermediates (aminophenols, resorcinol, and any other permitted dye chemicals; and their derivatives) as substitutes. The extent of coloration, or colour impact, for the Type 3 range of colours could vary, and could be substantially different from Type 1 and Type 2. The active dye content for Type 3 colours is specified in two parts:

- a) An upper limit active matter as PPD content, percent by mass is fixed to allow only a safe dye in the market; and
- b) Presence of dye ingredients (inclusive of lower or nil levels of PPD and/or phenylenediamine including their N-substituted derivatives and their salts, toluenediamine and derivatives and their salts, other permissible dye intermediates, couplers and modifiers, aminophenols, resorcinols, and all permitted dye chemicals, and their derivatives) is considered essential in order to safeguard consumer's interest to enable him/her to get a dye that would perform and is money's worth.

Secondly, the specification on the residue on evaporation of the developer was deleted in recognition of the latest technological developments in formulating developers using permissible fatty substances and emulsifiers. Lastly, the requirement on the dye-ready-for-use was also modified by replacing the specification on the PPD content

(Continued on third cover)

Indian Standard

OXIDATION HAIR DYES (LIQUID / GEL / CREAM) — SPECIFICATION

(Fourth Revision)

1 SCOPE

This standard prescribes the requirements and methods for sampling and test for oxidation hair dyes (liquid/ gel/ cream).

2 REFERENCES

The following 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:

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

3 TYPES

There are three types of dyes:

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

4 REQUIREMENTS

4.1 Description

The oxidation hair dyes (liquid/ gel/ cream) generally consist of two parts, namely:

- the dye, and

- the developer, which are supplied in separate containers.

4.2 Ingredients

Unless specified otherwise, all the raw materials used in the manufacture of oxidation hair dye, liquid, gel and cream 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 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 oxidation hair dyes (liquid/gel/cream), shall comply to IS 4011.

4.5 Dye

4.5.1 The active ingredient in Type 1 and 2 hair dyes usually contains an arylamine dispersed in a suitable base in an alkaline medium. They may also contain other dye chemicals like *p*-aminophenols, etc., besides arylamines. They may also contain suitable modifiers, such as, resorcinol. Type 3 hair dyes maybe formulated with nil or significantly lower levels of *p*-phenylenediamine (PPD) and/or phenylenediamines including their *N*-substituted derivatives and their salts, toluenediamine and derivatives and their salts, other permissible dye intermediates, couplers and modifiers, aminophenols, resorcinols, and all permitted dye chemicals; and their derivatives.

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

4.6 Developer

4.6.1 The developer is an oxidizing agent, usually a dilute solution of hydrogen peroxide (H₂O₂). It may be formulated using permissible fatty substances and emulsifiers, free from any foreign matter and suitably stabilized.

4.6.2 The developer shall also comply with the requirements given in Table 2 when tested as prescribed in column 4 of Table 2.

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Table 1 Requirements for Dye
(Clause 4.5.2)

SI No.	Characteristics	Requirement			Method of Test, Ref to Annex
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	pH	9.0 - 11.0	9.0 - 11.0	6.0 - 11.0	A
ii)	Active matter as PPD content, percent by mass (<i>m/m</i>), <i>Max</i> (including PPD and their derivatives, their salts, <i>p</i> -phenylenediamines, their <i>N</i> -substituted derivatives and their salts, and <i>N</i> -substituted derivatives of <i>o</i> -Phenylenediamine)	4.0	3.0	3.0	B or D ¹⁾
iii)	Dye ingredients (includes lower or nil levels of PPD and/or phenylenediamine including their <i>N</i> -substituted derivatives and their salts and/or Toluenediamine and derivatives and their salts, other permissible dye intermediates, couplers and modifiers, aminophenols, resorcinols, and all permitted dye chemicals)	—	—	Present	E or D ¹⁾
iv)	Heavy metals (as Pb), parts per million, <i>Max</i>	20	20	20	F
v)	Arsenic (as As ₂ O ₃), parts per million, <i>Max</i>	2	2	2	G

¹⁾ In case of any dispute, method of test prescribed at Annex B/E shall be the reference method.

Table 2 Requirements for the Developer
(Clause 4.6.2)

SI No.	Characteristics	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	pH	1.8 - 4.0	A
ii)	Assay (as H ₂ O ₂), percent by mass (<i>m/m</i>), <i>Max</i>	12	C
iii)	Heavy metals (as Pb), parts per million, <i>Max</i>	20	F
iv)	Arsenic (as As ₂ O ₃), parts per million, <i>Max</i>	2	G

4.7 Dye Ready for Use

4.7.1 The dye ready for use is prepared after mixing the dye content and developer in the proportion recommended by the manufacturer.

4.7.2 The mixing ratio of the dye-to-developer shall be in the range of 1 : 0.5 to 1 : 3.0.

4.8 The procedure for calculation of PPD content in solution after recommended dilution with developer is as follows:

If PPD content in liquid/cream oxidation hair dye is *x* percent and manufacturer recommends that 1 part of dye may be mixed with *y* part of developer, then,

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

4.9 Additional Requirements for ECO-Mark

4.9.1 General Requirements

4.9.1.1 The product shall conform to the requirements for quality, safety and performance prescribed under **4.1** to **4.8**.

4.9.1.2 All the ingredients that go into formulation of cosmetics shall comply with the provisions for IS 4707 (Part 1 and 2).

4.9.1.3 The material used for product packaging shall be recyclable, reusable or biodegradable.

4.9.1.4 The product package shall display a list of ingredients in descending order of quantity present.

4.9.1.5 The product shall not be manufactured from any carcinogenic ingredients.

4.9.1.6 The manufacturer shall produce to BIS the environmental consent clearance from the concerned State Pollution Control Board as per the provisions of *The Water (Prevention and Control of Pollution) Cess Act, 1977* and *Air (Prevention and Control of Pollution) Act, 1981* alongwith the authorization, if required under *The Environment (Protection) Act, 1986* and Rules made thereunder while applying for ECO-Mark. Additionally, provision of *The Drugs and Cosmetics Act, 1940* and the Rules thereunder shall also be complied with.

4.9.1.7 The product package shall be suitably marked that ECO-Mark label is applicable only to the contents, if the product package is not separately covered under the ECO-Mark scheme.

4.9.1.8 The product package shall display in brief the criteria based on which the product has been labeled environment friendly.

4.9.1.9 The material for product packaging shall meet the parameters evolved under the scheme of labelling environment friendly packaging/packaging materials.

4.9.2 Specific Requirements

4.9.2.1 Product shall be dermatologically safe when tested as prescribed in IS 4011.

4.9.2.2 Heavy metals calculated as lead (Pb) and arsenic as (As₂O₃) shall not exceed 20 and 2 ppm, respectively when tested by the respective method prescribed in Indian Standards.

5 PACKING AND MARKING

5.1 Packing

The dye and the developer shall be packed separately in packed in suitable containers. The dye and the developer are packed in the same carton. The individual containers and the carton shall be suitably labelled.

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:

- Name of the material;
- Shade of oxidation hair dye;
- Name and address of the manufacturer;
- Net content;

- Month and year of manufacture (MM/YY);
- Use before..... (Month and year MM/YY, or months/years from the date of manufacture) to be declared by the manufacturer;
- Declaration 'minimum arylamine (PPD) content after dilution as per manufacturer's instructions for use' (To be declared by manufacturer);
- Batch number;
- List the ingredients (at the time of manufacture) under the title 'Ingredients' as follows:

- For ingredients more than 1 percent (by mass or volume) — List the ingredients in decreasing order of percentage.
- 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.

- Contains phenylenediamines, and/or any other ingredient as required to be mentioned by IS 4707 (Part 2);
- The mixing ratio of the dye-to-developer for preparation of dye ready for use to be declared by the manufacturer;
- 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".
- Following cautions;
 - Keep out of reach of children.
 - This product is not intended for use on persons under the age of 16.
 - 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.
- 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 and developer, or may be printed on the carton itself, as the case may be):

- Procedure for conducting preliminary test for sensitivity (patch test);
- Instructions for use (may be given in attached leaflet);



- 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;

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- you have ever experienced any reaction after colouring your hair; and
 - 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.’

5.3.3 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

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

[Clauses 4.5.2 and 4.6.2, Tables 1 and 2, SI No. (i)]

DETERMINATION OF pH

A-1 APPARATUS

A-1.1 pH Meter, preferably equipped with glass electrode.

A-2 PROCEDURE

A-2.1 For Dye — Take 15 ml of the dye and determine its pH at $27 \pm 2^\circ\text{C}$ using the pH meter.

A-2.2 For Developer — Take 15 ml of the developer and determine its pH at $27 \pm 2^\circ\text{C}$ using the pH meter.

ANNEX B

[Clause 4.5.2, Table 1, SI No. (ii)]

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 5ml 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

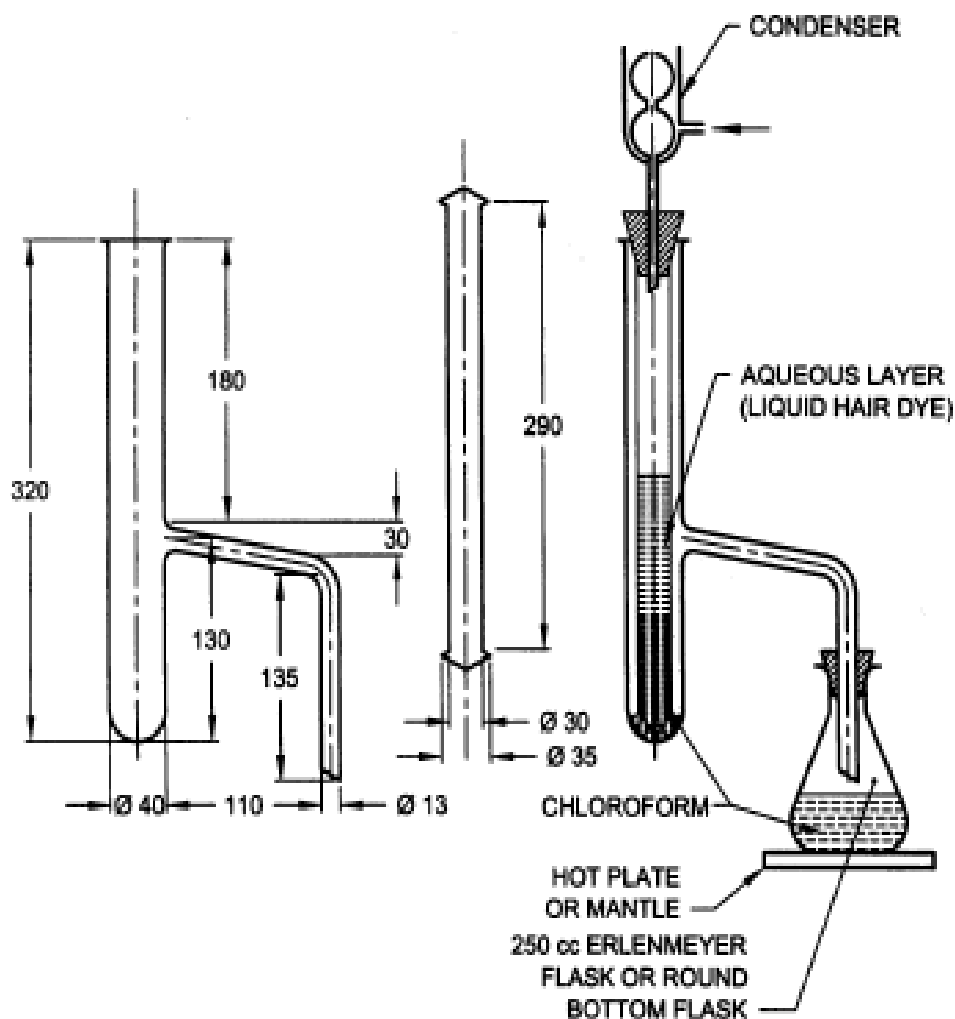
$$\text{Arylamine content (as PPD)} = \frac{m \times 0.5626 \times 100}{M}$$

Where,

m = mass, of the precipitate, in g; and

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

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All dimensions in millimetres.

FIG. 1 CONTINUOUS EXTRACTION APPARATUS

ANNEX C

[Clause 4.6.2, Table 2, Sl No. (ii)]

DETERMINATION OF HYDROGEN PEROXIDE CONTENT

C-1 REAGENTS

C-1.1 Dilute Sulphuric Acid

C-1.2 Potassium Permanganate Solution Freshly Standardized (N/10)

C-2 PROCEDURE

Weigh accurately about 10 g of the developer and dilute to 500 ml. Take 25 ml of the diluted solution in a conical flask, add 5 ml of sulphuric acid and titrate against potassium permanganate solution.

C-3 CALCULATION

$$\text{Assay, percent (m/m)} = \frac{V \times N \times 34.02}{M}$$

Where,

V = volume of potassium permanganate solution required for titration, in ml;

N = normality of potassium permanganate solution; and

M = mass, of developer taken to prepare 500 ml solution, in g.

ANNEX D

[Clause 4.5.2, 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 C18 Column, (150 mm × 4.6 mm × 5 μ)

D-2.5 Nylon Syringe Filter (0.45μ)

D-2.6 Ultrasonic Bath

D-2.7 Ultra Turrax

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 PREPARATION OF MOBILE PHASE

D-4.1 Preparation of 0.02M Ammonium Acetate Solution

Accurately weigh 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-4.2 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-5 PREPARATION OF DILUENT

D-5.1 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-5.2 Preparation of Methanolic Monoethanolamine (MEA) or Ethanolamine Solution (20 percent, v/v)

Accurately transfer 20 ml of Ethanolamine into a 100 ml volumetric flask and make up to volume with diluent and mix well.

D-6 PREPARATION OF BLANK SOLUTION

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

D-7 PREPARATION OF STANDARD STOCK SOLUTION

D-7.1 Preparation of Standard Stock Solution for Determination of Active Matter as PPD Content

D-7.1.1 Preparation of Standard Stock Solution 'A'

Weigh accurately and transfer 0.300 g of PPD into 100 ml volumetric flask, add about 70 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-7.1.2 Preparation of Standard Solution (about 300 ppm)

Pipette out 5 ml from Standard stock solution 'A' (D-7.1.1) and transfer to 50 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-7.2 Preparation of Standard Stock Solution for Identification of Dye Ingredients

Weigh 50 ± 5 mg of the needed reference material into a 50 ml flask, fill up to volume with diluent and mix well. If necessary use an ultra-sonic treatment but only for a few seconds. Prepare one stock solution for each dye present in the sample. Pipette 1 ml of the standard stock solution in a 10 ml flask, fill up to volume with diluent and mix thoroughly.

D-8 PREPARATION OF SAMPLE SOLUTION

Weigh 2.5 g of sample into a 50 ml beaker, add approximately. 15-20 ml diluent and homogenize with Ultra Turrax to obtain a homogeneous mixture.

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Transfer the mixture into a 100 ml flask. The beaker together with the Ultra Turrax will be rinsed thoroughly 2 times with approximately 20 ml solvent to remove sample residues. The rinsing solutions are added to the mixture in the flask to ensure a complete sample transfer. The flask is filled up to approximately 99 ml with diluent. Use Acetonitrile to fill up to the mark to eliminate any evolved foam, mix and shake well and filter the sample solution through a syringe filter and inject into the HPLC system.

D-9 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	25°C
Wavelength	280 nm

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.

<i>Sl No.</i>	<i>Name of Solution</i>	<i>Number of Replicates</i>
(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
vi)	—	—

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

<i>Name of Peak</i>	<i>Guide Retention Time (RT)</i>	<i>Guide Relative Retention Time (RRT)</i>
Ascorbic acid	1.99	—
PPD	3.06	1.54

D-11 CALCULATIONS

Percent content =

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

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

ANNEX E

[Clause 4.5.2, Table 1, Sl No. (iii)]

IDENTIFICATION AND ESTIMATION OF DYE INGREDIENT BY TLC

E-1 OUTLINE OF THE METHOD

This method identifies and estimates the dye/ coupler precursors/ ingredients in hair dye using TLC.

E-2 APPARATUS

E-2.1 Weighing Balance

E-2.2 TLC Plates, precoated silica gel 60 F₂₅₄, 0.2 mm thickness.

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

E-2.4 Iodine Chamber

E-2.5 Test Tubes with Stoppers

E-2.6 Pipette, 10 ml.

E-2.7 Syringe, 5 or 10 ml capacity.

E-2.8 Silicone Wax

E-3 REAGENTS

E-3.1 Toluene, analytical reagent grade.

E-3.2 Ethyl Acetate, analytical reagent grade.

E-3.3 Methanol, analytical reagent grade.

E-3.4 Sodium Sulphite, analytical reagent grade.

E-3.5 Standards, expected dye precursors/ingredients in the respective shade being analyzed.

E-3.6 Test Sample, 1 g of dye being tested.

E-4 PREPARATION OF SOLUTIONS

E-4.1 Mobile Phase — Toluene : Ethyl acetate : Methanol :: 50 : 40 : 10 (v/v). Mix these well in the stated dilution and keep stoppered.

E-4.2 Sample Solutions — Weigh accurately 1 g of sample from the shade being tested. Add a small quantity of sodium sulphite, add 4 ml of methanol (**E-3.3**) and mix well. Use this solution for spotting.

E-4.3 Standard Solutions — Weigh quantity nearly sufficient of the standard dye precursors/ ingredients

expected in the respective shade being analyzed, add sodium sulphite and dissolve in 5 ml of methanol (**E-3.3**) so that the concentration of each dye precursor is equivalent to its concentration in the sample solutions.

E-5 PROCEDURE

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

E-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 F

[*Clauses 4.5.2 and 4.6.2, Table 1, SI No. (iv) and Table 2, SI No. (iii)]*

TEST FOR HEAVY METALS

F-1 OUTLINE OF THE METHOD

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

F-2 APPARATUS

F-2.1 Nessler Cylinders, 50 ml capacity.

F-3 REAGENTS

F-3.1 Dilute Hydrochloric Acid, approximately 5 N.

F-3.2 Dilute Acetic Acid, approximately 1 N.

F-3.3 Hydrogen Sulphide Solution, standard.

F-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).

F-4 PROCEDURE

F-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.

F-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.

F-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.

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ANNEX G

[*Clauses 4.5.2 and 4.6.2, Table 1, SI No. (v) and Table 2, SI No. (iv)]*

DETERMINATION OF ARSENIC

G-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.

G-2 REAGENTS

G-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.

G-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.

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

G-2.4 Stannous Chloride Solution

Dissolve 80 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 100 ml of water containing 5 ml of concentrated hydrochloric acid.

G-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 F-4.1.

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

(Continued from second cover)

in the ready-to-use dye mixture (a metric derived by calculation using the dye-to-developer mixing ratio) with a specification based directly on the recommended dye : developer mixing ratio itself. This change simplified the dye-ready-for-use specification and makes it uniform across the three types of dye classification, namely, Types 1, 2 and 3.

In this revision, following modification have been carried out:

- a) Lower limit for PPD has been removed because of advancements taken place in the field of hair dyes. Presently, with the multiplicity of available ingredients, it is possible to achieve the same end result with respect to colour delivery whilst lowering the overall dye contents as well as individual dye levels.
- 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).
- d) In the TLC method for determination of dye ingredient (Annex E), use of benzene has been replaced with toluene.
- e) HPLC method for estimation of PPD in hair dyes (Annex D) has been modified. The revised method is simplified, explicit and more accurate.

A scheme for labeling environment friendly product as known as ECO-Mark was introduced at the instance of the Ministry of Environment and Forests and Climate Change (MEF&CC), Government of India. The ECO-Mark is being administered by the Bureau of Indian Standards (BIS) under the *Bureau of Indian Standards Act, 1986* as per the Resolution No. 71 dated 21 February 1991 and No. 768 dated 24 August 1992 published in the Gazette of the Government of India. For a product to be eligible for marking with ECO logo it shall also carry the Standard Mark of BIS besides meeting additional environment friendly requirements. For this purpose, the Standard Mark of BIS would be a single mark being a combination of the BIS monogram and the ECO logo. Requirements for ECO friendliness will be additional, manufacturing units will be free to opt for Standard Mark alone also. The additional requirements for ECO-Mark are given at **4.9**.

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.

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