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Draft Indian Standard

AUTO-OXIDATION HAIR DYES/COLOURS — SPECIFICATION

(First Revision)

(ICS No. 71.100.70)

Cosmetics Sectional Committee
PCD 19

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FOREWORD

(Formal clauses will be added later)

Hair dyes are cosmetic products intended for imparting colour to hair to produce varying shades either for grey coverage or simply to change the original colour of hair to make a fashion statement. Hair dyes may be broadly classified as Natural and Synthetic based on their Origin, as Permanent/Demi-permanent, Semi-Permanent and Temporary based on their mechanism of deposition on hair and their subsequent resistance to wash out. Another way of classification is based on the physical form of the final product offering. Bureau of Indian Standards has prescribed the following standards Hair Colours/Dyes.

I. Oxidative : Permanent/Demi-permanent

- a. IS 10350 : 2019 ‘Powder Hair Dyes — Specification (*Third Revision*)’.
- b. IS 8481 : 2019 ‘Oxidation Hair Dyes, Liquid, Gel and Cream — Specification (*Fourth Revision*)’]
- c. IS 15205 : 2005 Oxidation Hair Dyes (Emulsion Type) — Specification

II. Vegetable Based Hair Dyes

- a. IS 11142 : 2019 Specification for Henna Powder (*Second Revision*)

IS 15205 was first drafted to regulate the number of emulsion products that appeared in the market that delivered colour slowly over a period of 2-3 days following repeated applications. Since these products were in an emulsion form the standard was classified as “*Emulsion Type*”. It is important to note however that the colour delivery was primarily an outcome of oxidation of the dye precursor in the formulation, primarily PPD (p-Phenylenediamine) by air without the use of an external hydrogen peroxide source which is necessary for all other oxidative hair dyes/colours. There have been several advancements in technology in the past few years enabling a faster delivery using air oxidation thereby reducing time required to obtain the final colour without having to wait for 2-3 days and also avoiding multiple applications of the product. A more apt

classification for such products that is also a globally accepted nomenclature is Auto-Oxidation dyes.

It is therefore the aim of this revision to only relook at the specifications prescribed and suitably modify the same in light of the technological advances in this field but also to change the name of the standard to AUTO-OXIDATION HAIR DYES — SPECIFICATION from the earlier OXIDATION HAIR DYES (EMULSION TYPE) — SPECIFICATION.

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This specification covers only Oxidation Hair Dyes/Colours containing *p*-phenylenediamine (PPD) and/or other suitable dye precursors and couplers which are allowed for use as per the latest amendments of IS 4707 Part 2 as active ingredients.

This standard was first published in 2002. In this revision, following modification have been carried out:

- i) The name of the standard has been changed to AUTO-OXIDATION HAIR DYES/COLOURS — SPECIFICATION from the earlier OXIDATION HAIR DYES (EMULSION TYPE) — SPECIFICATION.
- ii) Addition column for Type 3 has been incorporated in Table 1, Requirements for Auto-Oxidation Hair Dyes/Colours Clause 4.5 to allow for products with newer technologies
- iii) The requirement of compliance of Auto-Oxidation Hair Dyes/Colours to IS 4011 for use of novel ingredients has also been incorporated.
- iv) The marking clause has been harmonized with Rule 34 of the Cosmetics Rules, 2020
- v) In the marking clause, cautions/warnings related to PPD content in the product have been aligned with IS 4707 (Part 2)
- vi) A list of languages in which the instructions for conducting hypersensitivity test have been provided
- vii) HPLC method for estimation of PPD in hair dyes (Annex D) has been incorporated.

Ideal properties of Auto-Oxidation Hair Dyes/Colours have been listed in Annex A for guidance only.

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*, 2016 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**.

The composition of the Committee, responsible for the formulation of this standard is given at Annex B (will be added later).

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 : 2022 'Rules for rounding off numerical values (*second revision*)'. 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

This draft standard prescribes the requirement and method of sampling and test for Auto-Oxidation Hair Dyes/Colours

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:

<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 : 1997	Methods of Test for Safety Evaluation of Cosmetics (<i>second revision</i>)
4707	Classification of Cosmetic Raw Materials and Adjuncts
(Part 1) : 2020	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>)
16913 : 2018	Methods of Test For Cosmetics — Determination of Heavy Metals (Arsenic, Cadmium, Lead and Mercury) by Atomic Absorption Spectrometry (AAS)

3 TYPES

3.1 Auto-oxidation Hair Dyes/Colours are broadly classified in three categories depending upon the colour, which they impart to the hair after application of dye:

- a) Type 1 – Black,
- b) Type 2 – Brown, and
- c) Type 3 – Others

4 REQUIREMENTS

4.1 Description —

The Auto-Oxidation Hair Dyes/Colours are ready to use products which do not require mixing with developer as oxidation to deliver the colour happens with oxygen from air. They can be in the form of lotion, cream, gels, pastes or liquids formulated with suitable product characteristics to facilitate ease of application and delivery when used as per instructions though Type 1 and 2 are essentially emulsion type.

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 the dye 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, 2020 framed thereunder.

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

4.5 The Auto-Oxidation Hair Dyes/Colours shall also comply with the requirements given in Table 1 when tested as prescribed in col 6 of Table 1.

Table 1 Requirements for Auto-Oxidation Hair Dyes/Colours
(Clause 4.5)

Sl. No.	Characteristics	Requirement			Method of Test, Ref to Annex/IS
		Type 1	Type 2	Type 3	
(1)	(2)	(3)	(4)	(5)	(6)
i)	pH (As Such)	7 – 9	7 – 9	6 – 10.5	B
ii)	Active matter as PPD content, percent by mass (w/w), (including p-Phenylenediamines and their derivatives, their salts, m and p-Phenylenediamines, their N-substituted derivatives and their salts, and N-substituted derivatives of o-Phenylenediamine with the exception of those that are listed in Annex A of IS 4707 Part 2)	4.0 (Max)	3.0 (Max)	4.0 (Max)	C 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, and all permitted dye chemicals; and their derivatives with exception of those compounds and derivatives listed in Annex A of IS 4707 Part 2)	-	-	Present	D ²

iv)	Total fatty matter, percent by mass, <i>Min</i>	5	5	5 for emulsions 0 for non-emulsions	E
v)	Specific Gravity, kg/m ³	0.90-1.1	0.90-1.1	0.90-1.1	IS 4730
vi)	Heavy metals (as Pb), parts per million, <i>Max</i>	20	20	20	F or IS 16913
vii)	Arsenic (as As ₂ O ₃), parts per million, <i>Max</i>	2	2	2	G or IS 16913
viii)	Mercury, parts per million, <i>Max</i>	1	1	1	IS 16913

NOTE ¹In case of any dispute, method of test prescribed at Annex C shall be the reference method.

²In case there is no PPD then this test needs to show presence of other dyes

4.6 Additional Requirements for ECO-Mark

4.6.1 General Requirements

4.6.1.1 The product shall conform to the requirements for quality, safety and performance prescribed under **4.1** to **4.5**.

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

4.6.1.3 The product package shall display a list of ingredients in descending order of quantity present as prescribed in Rule 148 of the Cosmetics Rules, 2020.

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

4.6.1.5 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 & 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.

4.6.1.6 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.

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

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

5 PACKING AND MARKING

5.1 Packing

The dye shall be filled in suitable containers like amber bottles, tubes etc. that are capable of protecting the product adequately during the shelf life. These products need to be suitably labelled as per requirements of different regulations as applicable.

5.2 Storage

The material shall be stored such that it is protected from direct heat and sunlight.

5.3 Marking

5.3.1 The bottles/ tubes/other suitable containers and the carton containing the material shall be legibly marked with the following information:

- a) Name of the material;
- b) Shade/Type of Auto-Oxidation hair dye/colour;
- 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 'arylamine (*p*-phenylenediamine) not more than 4 percent for Type 1 and Type 3 and not more than 3 percent for Type 2'
- h) Batch number;
- i) 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.

- j) Contains Phenylenediamines, and/or any other ingredient as required to be mentioned by IS 4707 Part 2;
- k) 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"
- l) Following Cautions;
 1. Keep out of reach of children.
 2. This product is not intended for use on persons under the age of 16.

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.

m) 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 48hrs, 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 The product may also be marked with the Standard Mark.

5.3.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations made thereunder. The details of conditions under which the license for use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

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

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

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

6 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

(Foreword)

(Informative)

IDEAL PROPERTIES OF OXIDATION HAIR DYE (EMULSION TYPE)

A The Auto-Oxidation Dye/Colour should

A.1 On visual inspection, product should be homogeneous and uniform in texture and should not show excessive signs or oxidation except on the top portion of the contents. There should not be any sign of separation.

A.2 It should be formulated with a suitable viscosity for ease of spreading and application and yet preventing undue dripping off onto skin.

A.3 It should impart the desired colour on hair for a long lasting effect

A.4 It should rinse out easily and should not have any residual tackiness and should not affect the texture of hair adversely

A.5 It may have an agreeable odour/perfume

A.6 The product should be formulated and packaged in a manner to ensure performance for the declared shelf life

ANNEX B

[*Clauses 4.5, Tables 1, SI No. (i)*]

DETERMINATION OF pH

B-1 APPARATUS

B-1.1 pH Meter —Equipped with glass electrode.

B-2 PROCEDURE

Take 50 ml of the dye and determine its pH at $27 \pm 2^\circ\text{C}$ using the pH meter.

ANNEX C

[*Clauses 4.5, Tables 1, SI No. (ii)*]

DETERMINATION OF ARYLAMINE CONTENT

C-1 REAGENTS

C-1.1 Sodium Hypochlorite Solution — 5 percent (available chlorine NLT 3.5 percent).

C-1.2 Sodium Arsenite Solution — 10 percent, dissolve 10 g of sodium arsenite (reagent grade) in 100 ml of water or dissolve by heating 8.5 g of arsenous oxide and 1.5 g of sodium hydroxide in 100 ml water.

C-1.3 Sodium Carbonate

C-1.4 Chloroform

C-1.5 Potassium Iodide

C-1.6 Hydrochloric Acid Concentrated — 38 percent (*w/v*).

C-1.7 Sodium Thiosulphate Solution — 0.1 N.

C-1.8 Starch Solution

Triturate 5 g of starch, add 0.01 g of mercuric iodide in 30 ml of cold water and slowly pour it with stirring into one litre of boiling water. Boil for 3 min, allow to cool and decant off the supernatant liquid for test.

C-2 PROCEDURE

C-2.1 Preparation of Sample Solution

Weigh accurately about 10 g of dye sample and dilute to 250 ml using water in a standard volumetric flask such that 10 ml of the solution contains about 0.01 g of *p*-phenylenediamine.

C-2.2 Add to a separating funnel containing 5 ml of sodium hypochlorite solution and 1 g of sodium carbonate, 10 ml of the sample solution. Gently swirl the separating funnel during addition so that the solution is thoroughly mixed (insufficient sodium hypochlorite is indicated by the presence of brown colour). If brown colour appears, repeat the operation by using larger quantity of sodium hypochlorite. Stopper the separating funnel and shake for 10s. Add 10 ml of sodium arsenite solution, stopper and shake again.

C-2.3 Extract the dichlorimide with two 25 ml portions of chloroform and collect combined extracts in a second separating funnel. Wash the extracts with two 10 ml portions of water and transfer into an iodine flask. Make additional chloroform extractions, if necessary, wash with water and combine with the major portion (any emulsion formed during extraction may be broken by adding a few drops of alcohol). Add 50 ml of water containing 1 g of potassium iodide and 3 ml of hydrochloric acid to the iodine flask. Stopper the flask and shake vigorously at intervals during titration. The iodine in the chloroform acts as an indicator. Towards the end of the titration, add starch solution for final end point.

C-3 CALCULATION

$$\text{Total active matter (dye content), percent by mass} = \frac{45.05 \times V \times N}{M}$$

where

V = volume of sodium thiosulphate required to titrate 10 ml of sample solution,
 N = normality of sodium thiosulphate solution, and
 M = mass of dye taken to prepare 250 ml of sample solution.

ANNEX D

[*Clauses 4.5, Tables 1, SI No. (ii)*]

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-3.6 Acetonitrile

D-4 PREPARATION OF MOBILE PHASE

D-4.1 Preparation of 0.02 M Ammonium Acetate Solution — Accurately weigh and transfer 1.541 g of Ammonium Acetate to 1000 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. 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

¹Ascorbic 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.

²Alternative 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 & 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)
1	Blank Solution	01
2	Standard Solution	03
3	Sample Solution – 1	01
4	Sample Solution – 2	01
5	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

Percent Content = $\frac{\text{Sample Area} \times \text{Standard Weight (g)} \times \text{Sample Dilution} \times \text{Percent Purity of standard}}{\text{Standard Area} \times \text{Standard Dilution} \times \text{Sample Weight (g)}}$

ANNEX E
[Clauses 4.5, Tables 1, SI No. (iii)]
DETERMINATION OF TOTAL FATTY SUBSTANCES

E-1 PRINCIPLE OF THE METHOD

The emulsion is broken with a dilute mineral acid and fatty matter is extracted with petroleum ether or ethyl ether. It is weighed after removal of the solvent.

E-2 REAGENTS

E-2.1 Dilute Hydrochloric Acid 1: 1 (v/v)

E-2.2 Ethyl Ether (40 to 60°C) or Petroleum Ether (60 to 80°C)

E-2.3 Methyl Orange Indicator Solution — Dissolve 0.1 g of methyl orange in 100 ml of water.

E-2.4 Sodium Sulphate Anhydrous

E-3 PROCEDURE

Weigh accurately about 4 to 5 g of the material into the conical flask, add 25 ml dilute hydrochloric acid, fit a reflux condenser into the flask, boil the contents until the solution is perfectly clear. Pour the contents of the flask into a 300 ml separating funnel and allow it to cool to 20°C. Rinse the conical flask with ethyl ether in portions of 10 ml. Pour the ether rinsings into the separating funnel. Shake the separating funnel well and leave until the layers separate. Separate out the aqueous phase and shake it out with 50 ml of ether twice. Combine all the ether extracts and wash them with water until free of acid (when tested with methyl orange indicator solution).

Filter the ether extracts through filter paper containing sodium sulphate into a conical flask, which has been previously dried at a temperature of $105 \pm 2^\circ\text{C}$ and then weigh. Wash the sodium sulphate on the filter with ether and combine the washings with filtrate. Distil off the ether and dry the material remaining in the flask at a temperature of $105 \pm 2^\circ\text{C}$ to constant mass.

E-4 CALCULATION

$$\text{Total fatty substance, percent by mass} = \frac{100 \times M_1}{M_2}$$

where

M_1 = mass in g, of the residue; and

M_2 = mass in g, of the sample taken for the test.

ANNEX F
[Clause 4.5, Table 1, SI No. (iv)]
TEST FOR HEAVY METALS

F-1 OUTLINE OF THE METHOD

The color 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 Dilute Ammonium Hydroxide — Approximately 5 N.

F-3.4 Hydrogen Sulphide Solution — Standard.

F-3.5 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.

ANNEX G

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

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.