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ICS NO:

PAKISTAN STANDARD

POWDER HAIR DYES

PAKISTAN STANDARDS AND QUALITY CONTROL AUTHORITY,
STANDARDS DEVELOPMENT CENTRE,
PSQCA Complex Plot # ST-7/A, Block-3, Scheme No: 36, Gulistan-e- Johar, Karachi.

PAKISTAN STANDARD SPECIFICATION
FOR
POWDER HAIR DYES
COSMETIC & TOILET GOODS SECTIONAL COMMITTEE

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PAKISTAN STANDARD SPECIFICATION

FOR

POWDER HAIR DYES

0. FOREWORD

0.1 This Pakistan Standard was adopted by Pakistan Standards Institution on 28th May, 1998 after the draft finalized by the Cosmetic & Toilet Goods Sectional Committee had been approved by the Chemical Divisional Council.

0.2 In general, hair dyes may be broadly clarified as powder hair dyes and liquid hair dyes, nevertheless this standard cover-only the requirements for powder hair dyes.

0.3 A powder hair dye usually contains an arylamine, that is, parapy-enylene diamine (PPD) as the active ingredient and a solid peroxide as the oxidizing agent. The brown variety contains other dye chemicals like ortho amino phenol, pare amino phenols, besides arylamine. The dyes may, in addition contain one or moro of the following ingredients:

a) Thickening agent,
b) Water softening agent,
c) Dispersing agent/ surface active agents,
d) Fillers,
e) Perfume,
f) Hair conditioner, and
g) Antioxidant.

0.4 In this standard a range for requirement of total active matter content for both the types is being prescribed. Fixing of lower limit for dye content was considered essential in order to safeguard consumer's interest and get him money's worth whereas upper limit is fixed to allow only a safe dye in the market as PPD is a known carcinogenic ingredient if used in concentration above 6 percent in dye ready for use in diluated from .

0.5 Further more a new requirement for active matter in the dye ready for use, prepared after recommended dilution with water as per manufacture's instructions in also being prescribed. The lower limit for this requirements shall take care of effective ness of the dye whereas the upper limit shall ensure the safe concentration of PPD. Another accelerated test to determine the probable reduction in active matter as arylamine in original pack on keeping has also been prescribed.

0.6 Marking clause has been elaborated, according to which it would be mandatory for the manufactures to declare PPD content in Powder dye, PPD content in dye ready to use, recommended dilution besides warning and declaration and other relevant instructions.

0.7 Expiry date is being prescribed as a regular requirement and declaration of list of critical ingredients on the carton/ package of dye has also been made compulsory in line with other cosmetic formulations.

0.8 For the purpose of deciding whether and 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 PS: 103-1991 (1st Rev.) Rules for rounding off numerical values. The number of significant places retained in the rounded off values shall be same as that the specified value in this standard.

0.1 **SCOPE**

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

2. **TYPES**

2.1 There shall be two types of material, namely;

Type 1 – Black

Type 2 – Brown

3. **REQUIREMENTS**

3.1 The powder hair dye shall be a fine, free flowing powder, usually coloured grey or grayish black.

3.2 **INGREDIENTS**

Unless specified otherwise, all raw materials used in the manufacture of powder hair shall conform requirements prescribed in the relevant Pakistan Standards where such standards does not exist. The Drugs and Cosmetics Act, as amended from time to time and updated shall apply.

3.3 The powder hair dye shall comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex B.

TABLE 1 REQUIREMENTS FOR POWDER HAIR DYES

S. NO	CHARACTERISTIC	REQUIREMENTS		METHOD OF TEST (REF TO CL NO. OF ANNEX B)
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	pH of percent (m/m) solution in water	7 to 10	7 to 10	B-2
ii)	Active matter as PPD content, percent by mass	10 to 30	5 to 20	B-3

3.4 The dye ready for use is prepared after mixing the powder dye with water as recommended by the manufacturer in the leaflet which is enclosed in the container, packed with the powder dye or may printed on the carton itself, as the case may be PPD content in dye ready for use may be calculated by the procedure given in Table 2. The lower limit of dye ready for use is being prescribed to check the effectiveness of the dye wheres the upper limit is being prescribed to check the concentration of PPD to ensure the same remains within the safe limits.

TABLE 2 FOR DYES READY FOR USE

S. NO	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Calculated active matter (as PPD) in the solution after recommended dilution with water, percent by mass	1.5-3.0	0.75-1.5	Procedure for calculation as given in note below.

NOTE: - The procedure for calculation of PPD content in solution after recommended with water is as follows:

If PPD content in powder hair dye is – d percent and manufacturer recommended that 1 part of dye may be mixed with y parts of water then ppD content in dye ready for use is:

$$= \frac{X}{Y + 1}$$

4. PACKING AND MARKING

4.1 PACKING

The material shall be packed in suitable air-tight containers.

NOTE: - The chemical gets oxidized in air and it is essential to keep it away from light and moisture, in air-tight containers.

4.2 MARKING

Each container (pouch glass bottles etc) and the package (carton / box) containing the same marked with the following information:

a) Name of material;
b) Name of manufacture;
c) Warning 'Shall not a used for dyeing eyelashes or eyebrows' (with proper illustration) and these hyper sensitive to PPD/Artificial colours.
d) Declaration 'Arylamine (P-phenylenediamine) not more then 4 percent after dilution as per manufacturer's instructions for use.
e) Net content;
f) Shade of dye;
g) The words, For best results use before…*
(Month and year to be given) and
h) Any other information required by statutory authorities. In addition to the above the following informations shall also be given in the attached leaflet;
a) Procedure for conducting preliminary test for sensitivity (patch test),
b) Instructions for use and
c) List of critical ingredients.

4.3 **CAUTION**

Para-Phenylenediamine may cause skin irritation in certain cases and so a preliminary test according to the accompanying direction should First be made (see 4.3.1). The material should not be used for dyeing the eyelashes or eyebrows, as it use may cause blindness.

4.3.1 Each package shall contain instructions in English and local languages on the following lines for carrying out the test. Para-phenylenediamine containing preparation may cause serious inflammation of the skin in some cases and see 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 and 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 hours, 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. This preparation should on no account be used for dyeing eyebrows or eyelashes as severe inflammation of the eye or even blindness may result.

5. **SAMPLING**

5.1 Representative samples of the material shall be drawn as prescribed in Annex. A.

5.2 Test for all characteristics shall be carried out on the composite sample.

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

A N N E X – A

SAMPLING

A-1 **LOT**

In a consignment all the containers containing hair dye representing the same batch of manufacturer shall constitute a lot.

A-2 **GENERAL REQUIREMENTS OF SAMPLING**

In drawing, preparing storing and handling samples, the following precautions shall be observed.

A-2.1 Samples shall be drawn in an environment not exposed to damp air, dust or soot.

A-2.2 Any sampling device may be used for drawing the materials from the containers. It shall be clean and dry when used.

A-2.3 The samples shall be placed in clean dry, glass or any other air-tight after filling and shall be marked with necessary details of sampling.

A-2.4 The material being sampled the samples, the sampling instrument and the sample containers shall be protected from adventious contamination.

A-2.5 Samples shall be stored so that conditions of storage do not affect the quality of the material.

A N N E X – B
METHOD OF TEST FOR POWDER HAIR DYE

B-1 **QUALITY OF REAGENTS**

B-1.1 Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

NOTE: *- 'Pure chemicals' shall mean chemicals that not contain impurities which affect the results of analysis.*

B-2 **DETERMINATION of pH**

B-2.1 **Apparatus**

A pH mater preferably equiped with glass electords

B-2.2 **Procedure**

Make 5 percent solution of powder hair dye and determine its pH at 270 $\pm$ 2^0C using the pH mater.

B-3 **DETERMINATION OF DYE CONTENT**

B-3.1 **Outline of the Method**

This method estimates the para phenylenodiamine as diacetyl derivative of para phenylenodiamine.

B-3.2 **Apparatus**

G 4e sintered glass crucible.

B-3.3 **Reagent**s

B-3.3.1 **Chlorofrom**

B-3.3.2 **Acetic Anhydride**

B-3.4 Weigh accurately 1 to 2 g of the sample into inner tube of continous extractor previously charged chloroform. Add about 60 ml of chloroform, and completely extract the dye, About 5 hours extraction is sufficient.

B-3.5 Remove the flask and transfer the filtrate to a 250 ml beaker. Rinse with few small portions of chloroform. Evaporate chloroform to about 25 ml and add 1 ml of acetic anhydride slowly, with stirring. Let it stand for one hour and filter on a weighed G 4e sintered glass crucible. Wash beaker and precipitate with three or more, 5 ml portions of chloroform. Carefully remove last traces, of precipitate from the beaker, Dry the crucible to constant mass at 120^0C and weigh the precipitate.

B-3.6 **Calculation:**

P=phenylenediamine, percent by mass $= \dfrac{M_2 \text{ X } 0.05626 \text{ X } 100}{M_1}$

M1 = mass in g of the precipitate and
M 2 = mass in g of the sample taken.

Note All dimensions in millimeters

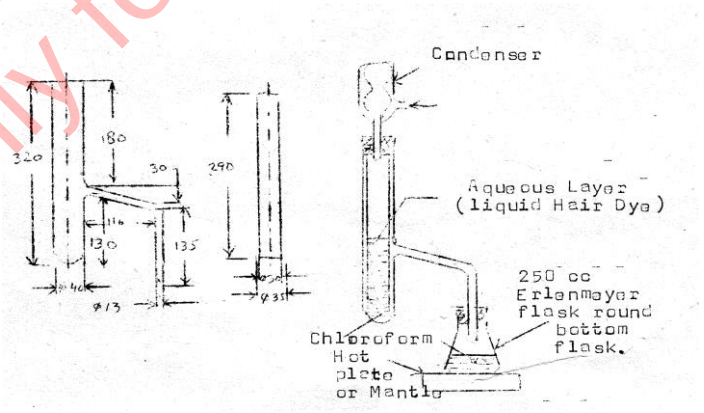

FIG. 1 CONTINOUS EXTRACTION APPARATUS