

व्यापकपरिचालनमसौदा

प्रलेखप्रेषणसंज्ञापन

संदर्भ

दिनांक

पीसीडी19(12411)C

19 02 2018

तकनीकसमीतिपीसीडी19

प्रेषिती

पैट्रोलियमतकनीकसमीति, पीसीडी19केसमस्तसदस्य।

पीसीडी19 कीसभीउपसमितियाँ

पैट्रोलियम, कोयलाएवंसंबंधितउत्पादविभागपरिषद) पीसीडीसीकेसदस्य।

रूचिरखनेवालेसभी निकाय।

महोदय /महोदया,

कृप्या आपके अवलोकन हेतु निम्नलिखित मसौदासंलग्न है :

क्रम संख्या	मसौदा संख्या	विषय
.1	पी सी डी 19(12411)C	हेना पाउडर-विशिष्ट

कृपया इस मसौदाका अवलोकन कर अपनी सम्मतियाँ यह बताते हुए भेजेकि यदि अंतत यह मानक मसौदा राष्ट्रीय मानक के रूप में प्रकाशित हो जाये तो इस पर अमल करने में आपके व्यवसाय अथवा कारोबार में क्या कठिनाईया आ सकती हैं।

सम्मतियाँ भेजने कि अंतिम तिथि :19 April 2018

सम्मतियाँ कृपया संलग्न प्रारूप में अधोहस्ताक्षरी को भेजे
धन्यवाद, भवदीय

ए.के .भटनागर

वैज्ञानिकएफएवंप्रमुख)पीसीडी)

प्रतिउपरिलिखित

मेल : pcd@bis.gov.in, pcd1@bis.gov.in

DRAFT IN WIDE
CIRCULATION

DOCUMENT DESPATCH ADVICE

REF:	DATE
Doc:PCD19 (12411)C	19 02 2018

Technical Committee: Cosmetics Sectional Committee, PCD 19

- i) All interested members of PCDC;
- ii) All members of Cosmetics Sectional Committee, PCD 19
- iii) All members of its subcommittees;
- iv) All others interested.

Dear Sir,

Please find enclosed the following documents:

Doc: No.	Title
PCD 19(12411)C	HENNA POWDER – SPECIFICATION

Kindly examine this draft and forward your views stating any difficulty which you are likely to experience in your business or profession, if this is finally adopted as a revised standard.

Last date for comments is : 19 April 2018

Comments if any, may please be made in the format as given overleaf and mailed to the undersigned at the above address. The document is also hosted on BIS website www.bis.org.in.

Thanking you,

Signature :

Name : A.K. Bhatnagar

Designation Sc.F& Head (PCD)
email pcd@bis.gov.in; pcd1@bis.gov.in

Encl: As above.

Feb 2018
BUREAU OF INDIAN STANDARDS

Draft Indian Standard

HEENA POWDER – SPECIFICATION

ICS 71.100.70

Last date for comments : 19 April 2018

FOREWORD

0.1 This Indian standard was adopted by the Indian Standards Institution on 12 December 1984, after the draft finalized by the Cosmetics Sectional committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Henna (called MEHENDI in Hindi) is the leaf of a small shrub. Botanically the plant is known as *LawsoniaInermis* Linn. syn. *L. alba*, fam. Lythraceae. The leaves of the plant, dried and powdered, are used to dye the hair and for colouring the finger nails, palms and soles of the feet. It is also exported in considerable quantities.

0.3 Henna powder quality is determined by its colour, purity, its dyeing property and fineness. The principal colouring matter is lawsone.

0.4 At times henna powder may be adulterated with sand, stems, fruit of henna plant, husk of paddy, leaves and twigs of other shrubs, etc. Certain requirements and tests, as in case of powdered spices have been included to restrict malpractices.

0.5 Microscopic examination of the powdered henna leaves show the following histological structures. Olive green or brownish green numerous fragments of cuticle and leaf parenchyma rosette aggregates and monoclinic prisms of calcium oxalate frequently upto 15 microns and occasionally upto 40 microns in diameter, globular mucilage cells, numerous fragments of intravascular tissues, long narrow and shorter fusi form schlerenchyma fibres with thick walls, some of the latter being wavy toothed, fragments of epidermis with stomata and striated cuticle, the stomata being surrounded by ordinary epidermal cells; occasional papillae or non-glandular hair fragments.

0.6 For the purpose of deciding whether a particular requirements 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*. The number of significant places retained in the rounded off value should be the same as that of specified value in this standards.

* Rules for rounding off numerical values (revised).

1. SCOPE

1.1 The standard prescribes the requirement, sampling procedure and methods of test Henna powder.

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 indicated below:

IS No.	Title
1070 : 1992	Reagent grade water (third revision)
3958 : 1984	Methods of sampling cosmetics (first revision)
4011 : 1997	Methods of test for safety evaluation of cosmetics (second revision)
4707	Classification of cosmetic raw materials and adjuncts:
(Part 1) : 2009	Dyes, colours and pigments (second revision)
(Part 2) : 2009	List of raw materials generally not recognized as safe for use in cosmetics (second revision)

3. REQUIRMENTS

3.1 Description – The material shall be in the form of fine powder obtained from dried fresh leaves of henna plant. A minimum of 95 percent should pass through 250 micron IS sieve [see IS : 4601) – 1978*]. It shall be free from extraneous adulterants (see 0.4).

3.2 Ingredients- Other herbal natural ingredients like Amla, Reetha, Bhringraj, jatamansietcAmla, Bhringraj, Ritha, Jatamansi etc. can also be mixed with Henna powder. for retail sale (to be directly used by consumer is small proportion).

3.3 It may contain small quantities of essential oils, preservative, citric acid and lemon juice etc. for colour enhancement and pH maintenance.

3.4 Unless otherwise specified, all the raw materials; used in the manufacture of henna paste shall conform to the requirements prescribed in the relevant Indian Standards where such Standard exists.

3.5 All ingredients of henna paste 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 framed there under.

3. 6 The materials shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR HENNA POWDER

SL No.	Characteristic	Requirement	Method of test ANNEXURE
i)	Moisture and volatile matter, percent by mass, Max	10	A
ii)	Cold water extract, percent by mass	25 to 32	B
iii)	Crude fibre, percent by mass	10 to 15	C
iv)	Mineral matter, percent by mass	8 to 12	D
v)	Acid insoluble ash, percent by mass	3 to 6	E
vi)	Extraneous sand, percent by mass, Max	5	F
vii)	Presence of extraneous dyes	To pass the test	G
viii)	Lawsone pigment, percent by mass, Min	1.0	H or I

Note- Total vegetable matter; percent by mass shall be approximately 95 percent.

Total vegetable matter= 100- (sand, percent by mass)

* Specification for test sieves: Part 1 Wire cloth test sieves (second revision)

4. PACKING AND MARKING

4.1 Packing

4.1.1 The material shall be packed in polythene lined hessian bags or in suitable containers as agreed to between the purchaser and the supplier in case of wholesale or industrial use.

4.1.2 In case henna powder is for retail sale it should not be packed in package exceeding 10 kg or more.

4.1.3 All containers in which the material is packed shall be dry, clean and tight so that extraneous impurities are not introduced. Flexible packaging or containers which can prevent absorption of moisture can also be used (package of less than 10 kg).

4.2 Marking

4.2.1 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser in case of wholesale or industrial use.

4.2.2 Each container shall be marked with the following information:

- a) Name of the material;
- b) Manufacturer's name or his trade-mark, if any;
- c) Gross weight and net weight when packed; and
- d) Date of packing.

4.2.3 For Henna powder packed in suitable package which is intended for retail purpose, following markings are required. The containers may also be marked with the Standard Mark.

Marking Requirement :Each container / package (pouch, carton, box, jar, bag etc.) should be marked with the following information.

- (a) Name of the material.
- (b) Indication of the source of manufacturer. Manufacturer's name or his trade-mark, if any;
- (c) Net contents. Net mass of the material.
- (d) B. no. Batch or lot number
- (e) Month & Year of manufacturing/ packing
- (f) Expiry date or “Best use before ...” (month & year to be declare by manufacturer)
- (g) List of key ingredients in case of additional ingredients are added to it.
- (h) Do not use in the area of the eyes, cut or abraded scalp.
- (i) Instructions for use (can be given on leaflet if attached).

4.2.4 BIS Certification Marking

4.2.3 The containers may also be marked with the Standard Mark.

4.2.4 The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufactures or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING OF HENNA POWDER

5.1 General Requirements

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

5.1.1 Samples shall not be taken in an exposed place.

5.1.2 The sampling instrument shall be clean and dry.

5.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument, the containers for samples, from adventitious contamination.

5.1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

5.1.5 The sample containers shall be placed in clean dry, air-tight glass or other suitable containers.

5.1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

5.1.7 Each sample container shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

5.2 Scale of Sampling

5.2.1 Lot – All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

5.2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

5.2.2 The number of containers (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be in accordance with col 1 and 2 of Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING	
Lot Size N (1)	Number of containers to be selected N (2)
1 and 2	Each container
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 to 1000	7

5.2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables (see IS : 4905-1968*) shall be used; in case such tables are not available, the following procedure shall be adopted:

Starting from any container, count them as 1, 2, 3, etc, upto r and so on in a systematic manner, where r is the integral part of N/n . Every r th container thus counted shall be withdrawn from the lot.

* Methods for random sampling.

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

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.

6. QUALITY OF REAGENTS

6.1 Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1977*) shall be employed in tests.

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

7. PREPARATION OF SAMPLE FOR ANALYSIS

7.1 Procedure – If the sample is in coarse condition, grind and / or crush it as rapidly as possible without loss or gain of moisture and with minimal exposure so that it passes through a 250-micron sieve. Mix the sample thoroughly, divide and keep at least 100 g portion in a non-corrodible, clean and dry air-tight container for analysis.

ANNEXURE A

[Clause 3.3 Table 1 S No. (i)]

DETERMINATION OF MOISTURE AND VOLATILE MATTER

Procedure – Weigh accurately about 5 g of the prepared sample material in a moisture dish, about 6 to 8 cm in diameter and about 2 to 4 cm in depth. Dry in an air oven at a temperature of $105 \pm 2^\circ\text{C}$ to constant mass (within ± 5 mg).

Calculation

Moisture and volatile matter, Percentage by mass = $\frac{100 \times M_1}{M}$

Where

M_1 = loss in mass in g on drying, and

M = mass in g. of the material taken for the test.

ANNEXURE B
[Clause 3.3 Table 1 S No. (ii)]

DETERMINATION OF COLD WATER EXTRACT

Procedure – Weigh to the nearest 0.001 g, about 2 g of the prepared sample. Transfer the material quantitatively with water to a 100 ml volumetric flask and fill to the mark with cold water. Stopper the flask and shake at approximately 30 minutes intervals for 8 hours and allow to settle for another 16 hours without shaking. Filter the extract through a dry filter paper. Reject first few milliliters, then evaporate a 25-ml aliquot to dryness in a tared dish on the water-bath and heat in the oven at $100 \pm 2^\circ\text{C}$ to constant mass. Record the final mass.

* Specification for water for general laboratory use (second revision)

Calculation

$$\begin{array}{l} \text{Cold water soluble extract,} \\ \text{Percent by mass} \end{array} = 4 \times \frac{M_2}{M_1}$$

Where

M_1 = mass in g of the test sample, and

M_2 = mass in g of the residue obtained.

ANNEXURE C
[Clause 3.3 Table 1 S No. (iii)]

DETERMINATION OF CRUDE FIBRE

Reagents:

Petroleum Ether - low boiling.

Dilute Sulphuric Acid- 1.25 percent (m/v), accurately prepared.

Sodium Hydroxide Solution- 1.25 percent (m/v), accurately prepared.

Ethyl Alcohol - 95 percent (v/v).

Procedure – Weigh accurately about 2.5 g of the prepared sample. Transfer the material into a one litre flask. Take 200 ml of the dilute sulphuric acid in a beaker and bring to boil. Transfer the whole of the boiling acid to the flask containing material and immediately connect the flask with a water-cooled reflux condenser and heat, so that the content of the flask begin to boil within 1 minute. Rotate the flask frequently, taking care to keep the material from remaining on the sides of the flask and out of contact with the acid. Continue boiling for exactly 30 minutes. Remove the flask and filter through fine linen (about 18 threads to the centimetre) or through a coarse acid - washed, hardened filter paper, held in a funnel, and wash with boiling water until the washings are no longer acid to litmus. Bring some quantity of sodium hydroxide

solution to boil under a reflux condenser. Wash the residue on the filter into the flask with 200 ml of boiling sodium hydroxid solution. Immediately connect the flask with the reflux condenser and boil for exactly 30 minutes. Remove the flask and immediately filter through the linen or through filter paper. Thoroughly wash the residue with boiling water and transfer to a Gooch crucible prepared with a thin but compact layer of ignited asbestos. Wash the residue thoroughly first with hot water and then with about 15 ml of ethyl alcohol and with three successive washings of 15 ml of petroleum ether each. Dry the Gooch crucible and contents at $105 \pm 1^\circ\text{C}$ in an air-oven for 3 hours, cool and weigh. Repeat the process of drying for 30 minutes, cooling and weighing until the difference between two consecutive weighing is less than 1 mg. Incinerate the contents of the Gooch crucible in the muffle furnace at $550 \pm 20^\circ\text{C}$ until all the carbonaceous matter is burnt. Cool the Gooch crucible containing the ash in a desiccators and weigh.

Calculation

$$\text{Crude fibre, percent by mass} = \frac{100 (M_1 - M_2)}{M}$$

Where

M_1 = mass in g of Gooch crucible and contents before ashing,

M_2 = mass in g of Gooch crucible and contents after ashing, and

M = mass in g of the material taken for the test.

ANNEXURE D

[Clause 3.3 Table 1 S No. (iv)]

DETERMINATION OF MINERAL MATTER

Procedure – Weigh accurately about 5 g of the prepared sample in a silica dish. Heat the dish at first on a low flame and then in a muffle furnace maintained at about 600°C . Cool in a desiccator and weigh. Repeat the process of heating, cooling and weighing until constant mass is obtained. Preserve the ash for test under 8.

Calculation

$$\text{Mineral matter, percent by mass} = \frac{M_2 \times 100}{M_1}$$

where

M_2 = mass in g of the ash, and

M_1 = mass in g of the material taken for the test.

ANNEXURE E
[Clause 3.3 Table 1 S No. (v)]

DETERMINATION OF ACID INSOLUBLE ASH

Reagent

Dilute Hydrochloric Acid – approximately 5 N.

Procedure – To the ash preserved in 7.1 add 25 ml of dilute hydrochloric acid (5 N), heat on water-bath for 10 minutes, allow to cool and filter the contents of the dish through Whatman filter paper No. 42, wash the filter with distilled water till the washing is free from acid. Return the filter and residues to the dish. Keep it in oven to dry and ignite to free from carbon (in muffle furnace to 600°C). Cool the dish in desiccators and weigh. Repeat the process of igniting, cooling and weighing, until the difference between two successive weighing is less than one milligram. Note the lowest mass.

Calculation

$$\text{Acid insoluble ash, percent by mass} = \frac{M_2}{M_1} \times 100$$

Where

M_2 = mass in g of the residue, and

M_1 = mass in g of the material taken for the test.

ANNEXURE F
[Clause 3.3 Table 1 S No. (vi)]

DETERMINATION OF EXTRANEIOUS SAND

Procedure – Weigh 5 g of prepared sample and transfer to a beaker. Add about 100 ml of carbon tetrachloride and mix the contents intimately and allow to settle. The supernatant liquid is decanted and the residue again stirred with another 100 ml portion of carbon tetrachloride. This process is repeated till it is freed of all vegetable matter. Transfer the residue to a tared silica dish, ignite and weigh to constant mass.

Note – That the residue has been freed from vegetable matter can be judged by the absence of formation of light leafy scum on inside of the beaker when carbon tetrachloride evaporates partially.

Calculation

$$\text{Extraneous sand, } M_2$$

$$\text{percent by mass} = \frac{\text{-----}}{M_1} \times 100$$

Where

M_2 = mass in g of sand, and

M_1 = mass in g of the material taken for the test.

ANNEXURE G
[Clause 3.3 Table 1 S No. (vii)]

THIN LAYER CHROMATOGRAPHIC (TLC)
TEST FOR DETECTION OF EXTRANEIOUS DYES

Preparation of Plates – Mix 10 g of the silica gel (of TLC grade, particle size 10 to 40 μm) with 20 ml of distilled water to make a slurry and spread over glass plates to a depth of 250 microns. Activate the plates for 30 minutes, by keeping in an oven maintained at 105°C.

Preparation of Sample – Extract about 0.1 g of the prepared sample with 5 ml of chloroform and apply one drop of the extract on the base line of the plate.

Development of Chromatogram – Keep the prepared plates in a jar containing a mixture of chloroform : methyl ethyl ketone : glacial acetic acid (5 : 4 : 1). Allow the solvent front to run up to 15 cm (the time taken is about 3 hours) at room temperature (25 to 30°C). Observe the spots obtained, under ultra violet light.

The principal ingredients of genuine henna powder, namely lawsone and chlorophyll) will give spots on the chromatogram as given below:

Lawsone : Reddish spot with Rf value approximately 0.4

Chlorophyll : Greenish spot coincident with the liquid front

Any other spots on the chromatogram indicate extraneous dyes.

For comparison, use a reference sample prepared from standard henna powder.

ANNEXURE H
[Clause 3.3 Table 1 S No. (viii)]

DETERMINATION OF LAWSONE PIGMENT CONTENT

Outline of Method – The pigment is extracted and the lawsone content is determined by comparing the observed optical density (measured colorimetrically) with a calibration curve, relating optical density to various concentrations of 2-hydroxy, 1-4-naphthoquinone.

Apparatus

Spectrophotometer or Photoelectric Colorimeter – with a filter of 490 nm.

Reagents

Sodium Bicarbonate Solution – 5 percent (m/v).

2-Hydroxy, 1-4-naphthoquinone

Procedure

Preparation of Standard Calibration Curve – Construct a calibration curve by dissolving known amounts of 2-hydroxy, 1-4-naphthoquinone ($C_{10}H_6O_3$) varying in concentration from 0 to 2 percent under the same condition as described below.

Weigh 2.0 g of the prepared sample. Transfer it to a 100 ml volumetric flask. Add 5 percent (m/v) solution of sodium bicarbonate and make up the volume to mark. Shake the contents of the flask every half an hour or so for about 8 hours. Allow to settle overnight, there after filter the solution through a filter paper and reject the first few millilitres. Take 10 ml of the filtrate in a 25 ml volumetric flask and dilute with distilled water up to the mark. Measure the optical density of this solution with a spectrophotometer at 490 nm.

Calculation

Refer to the calibration curve and determine the percent lawsone content of the sample from the curve.

ANNEXURE I
[Clauses 4.5, Table-1, SI No.(viii)]

DETERMINATION OF LAWSONE PIGMENT, BY HPLC

D-1 Chromatographic Conditions

Column :Microsorb - MV 100-5 C18 250 X 4.6mm
Mobile phase :Methanol:Water with 1% Ortho Phosphoric acid,(50:50)
Wavelength : 286 nm
Flow rate : 1 ml /min
Temperature : Ambient

D-2 Sample Preparation

Weigh 2.0 g of sample. Transfer it to a 100 ml volumetric flask. Add 5% (m/v) Solution of sodium bicarbonate and make up the volume to mark .Shake the contents of the flask every half an hour or so for about 8 hours. Allow to settle overnight, there-after filter the solution through a filter paper and reject the first few milliliter. Take 10 ml of the filtrate in a 25 ml volumetric flask and dilute with distilled water up to the mark. Use this solution for HPLC.

D-3 Standard Preparation

Take weight 0.0213g/100 ml of Standard 2-hydroxy-1 4-naphthoquinone and prepared same as described under sample preparation.

D-4 Stability and Storage Preparation

The standard and sample are stable when stored in glass volumetric flask and are refrigerated.

D-5 Calculations Area of sample x conc. of Std. x Purity (95%)

Area of Std. x conc. of sample.