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DRAFT EAST AFRICAN STANDARD

Henna powder — Specification

DEAS 337_2023

EAST AFRICAN COMMUNITY

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Third Edition 2020

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Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS). The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the Principles and procedures for development of East African Standards.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

The committee responsible for this document is Technical Committee EASC/TC 071, *Cosmetics and related products*.

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

This third edition cancels and replaces the second edition (EAS 337:2013), which has been technically revised.

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Henna powder — Specification

1 Scope

This Draft East African Standard specifies the requirements, sampling and test methods for pure henna powder.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EAS 346, Labelling of cosmetics — General requirements

EAS 377(all parts), *Cosmetics and cosmetic products*

EAS 846, Glossary of terms relating to cosmetic industry

EAS 847-16, Cosmetics — Analytical methods — Part 16: Determination of lead, mercury and arsenic content

ISO 16212, Cosmetics - Microbiology - Enumeration of yeast and mould

US ISO 18416, Cosmetics — Microbiology — Detection of Candida albicans

ISO 21149, Cosmetics — Microbiology — Enumeration and detection of aerobic mesophilic bacteria

ISO 22717, Cosmetics — Microbiology —Detection of Pseudomonas aeruginosa

ISO 22718, Cosmetics — Microbiology — Detection of Staphylococcus aureus

ISO 24153*, Random sampling and randomization procedures*

3 Terms and definitions

For the purposes of this standard, the terms and definitions given in EAS 846 shall apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Requirements

4.1 General requirements

4.1.1 The material shall be in the form of fine, dried powder obtained from fresh leaves of henna plant.

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4.1.2 The henna shall be free from extraneous adulterants. These include stems, small fruits, husk of paddy, leaves and twigs of other shrubs.

4.2 Specific requirements

Henna powder shall also comply with the specific requirements given in Table 1 when tested in accordance with the test methods specified therein.

Table 1 — Specific requirements for henna powder

S/N	Characteristic	Requirement	Test method
i.	Moisture and volatile matter, % by mass, max.	10	Annex A
ii.	Cold water extract, % by mass	25 - 32	Annex B
iii.	Crude fibre, %t by mass	10 - 15	Annex C
iv.	Mineral matter, % by mass	8 - 12	Annex D
v.	Acid insoluble ash, % by mass	3 - 6	Annex E
vi.	Extraneous sand, % by mass, max.	5	Annex F
vii.	Presence of extraneous dyes	To pass the test	Annex G
viii.	Lawsone pigment, % by mass, min.	1.0	Annex H
ix.	Residue on 250 micron sieve, % by mass, max.	5.0	Annex I

5 Heavy metal contaminants

Henna powder shall comply with the heavy metal contaminant limits given in Table 2 when tested in accordance with the test methods specified therein.

Table 2 — Heavy metal contaminant limits for henna powder

Heavy metal	Maximum limit [a],mg/kg	Test method
Lead (as Pb)	10	EAS 847-16
Arsenic (as As)	2	
Mercury (as Hg)	2	
[a] The total amount of heavy metals as lead, mercury and arsenic, in combination, in the finished product shall not exceed 10 mg/kg.		

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6 Microbiological limits

Henna powder shall comply with the microbiological limits given in Table 3 when tested in accordance with the test methods specified therein.

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Table 3 — Microbiological limits for henna powder

S/N	Microorganism	Limit	Test method
i.	Total viable count for aerobic mesophilic micro-organisms CFU/g, max.	100	ISO 21149
ii.	*Pseudomonas aeruginosa*	Not detectable in 0.5 g of cosmetic product	ISO 22717
iii.	*Staphylococcus aureus*		ISO 22718
iv.	*Candida albicans*		ISO 18416
v.	Yeast and moulds, CFU/g, max.	100	ISO 16212

5 Packaging

The product shall be packaged in suitable well-sealed containers that shall protect the contents and shall not cause any contamination or react with the product.

6 Labelling

6.1 In addition to the labelling requirements given in EAS 346, each package shall be legibly and indelibly labelled with product name as “Henna powder”.

6.2 In addition, the labelling shall include the instructions for use with a clear statement that only water shall be used as a solvent.

7 Sampling

Sampling shall be done in accordance with ISO 24153

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Annex A
(normative)

Determination of moisture and volatile matter

A.1 Procedure

Weigh accurately about 5 g of the prepared sample material in a moisture dish, about 6 cm to 8 cm in diameter and about 2 cm to 4cm in depth. Dry in an air oven at a temperature of 105 °C ± 2 °C to constant mass (within ± 5 mg).

A.2 Calculation

The moisture and volatile matter content, expressed as percent, shall be calculated as follows;

$$100 \times \frac{M_1}{M_2}$$

where

M_1 is the loss in mass, in grams, on drying, and

M_2 is the mass, in grams, of the material taken for test.

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Annex B
(normative)

Determination of cold water extract

B.1 Procedure

B.1.1 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 min intervals for 8 h and allow to settle for another 16 h without shaking.

B.1.2 Filter the extract through a dry filter paper. Reject first few millilitres, then evaporate a 25- mL aliquot to dryness in a tared dish on the water-bath and heat in the oven at 100 °C ± 2 °C to constant mass. Record the final mass.

B.2 Calculation

The cold water soluble extract content, expressed as percent, shall be calculated as follows;

$$4 \times \frac{M_2}{M_1} \times 100$$

where

M_1 is the mass, in grams, of the text sample, and

M_2 is the mass, in grams, of the residue obtained.

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Annex C
(normative)

Determination of crude fibre

C.1 Reagents

C.1.1 **Petroleum ether**, low boiling range of 40 °C - 60 °C

C.1.2 **Dilute sulphuric acid**, 1.25 % (v/v), accurately prepared

C.1.3 **Sodium hydroxide solution**, 1.25 % (m/v), accurately prepared

C.1.4 **Ethyl alcohol**, 95 % (v/v)

C.2 Procedure

C.2.1 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.

C.2.2 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 contents of the flask begin to boil within 1 min.

C.2.3 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 min.

C.2.4 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.

C.2.5 Wash the residue on the filter into the flask with 200 mL of boiling sodium hydroxide solution. Immediately connect the flask with the reflux condenser. Immediately connect the flask with the reflux condenser and boil for exactly 30 min. Remove the flask and immediately filter through the linen or through filter paper.

C.2.6 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 15ml of ethyl alcohol and with three successive washings of 15 mL of petroleum ether each.

C.2.7 Dry the Gooch crucible and contents at 105 °C ± 1 °C in an air- oven for 3 h, cool and weigh. Repeat the process of drying for 30 min, cooling and weighing until the difference between two consecutive weighings is less than 1 mg.

C.2.8 Incinerate the contents of the Gooch crucible in the muffle furnace at 550 °C ± 20 °C until all the carbonaceous matter is burnt. Cool the Gooch crucible containing the ash in a desiccator and weigh.

C.3 Calculation

The crude fibre content, expressed as percent, shall be calculated as follows;

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$$100 \times \frac{M_1 - M_2}{M}$$

where

M_1 is the mass, in grams, of Gooch crucible and contents before ashing;

M_2 is the mass, in grams, of Gooch crucible and contents after ashing; and

M is the mass of the material taken for the test.

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Annex D
(normative)

Determination of mineral matter

D.1 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 Annex E.

D.2 Calculation

The mineral matter content, expressed as percent, shall be calculated as follows;

$$100 \times \frac{M_2}{M_1}$$

where

M_2 is the mass, in grams, of the ash, and

M_1 is the mass in grams, of the material taken for the test.

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Annex E
(normative)

Determination of acid insoluble ash

E.1 Reagent

Dilute hydrochloric acid, approximately 5 N.

E.2 Procedure

E.2.1 To the ash preserved in D.1 add 25 mL of dilute hydrochloric acid (5 N), heat on water-bath for 10 min, 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.

E.2.2 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 desiccator and weigh. Repeat the process of igniting, cooling and weighing, until the difference between two successive weighings is less than one milligram. Note the lowest mass.

E.3 Calculation

The acid insoluble ash content, expressed as percent, shall be calculated as follows;

$$100 \times \frac{M_2}{M_1}$$

where

M_2 is the mass, in grams, of the residue, and

M_1 is the mass, in grams, of the material taken for test in D.1.

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Annex F
(normative)

Determination of extraneous sand

F.1 Procedure

F.1.1 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.

F1.2 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.

F.1.3 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.

F.2 Calculation

The extraneous sand content, expressed as percent, shall be calculated as follows;

$$100 \times \frac{M_2}{M_1}$$

where

M_2 is the mass, in grams, of the sand, and

M_1 is the mass, in grams, of the material taken for test

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Annex G
(normative)

Thin layer chromatographic (TLC) test for detection of extraneous dyes

G.1 Preparation of plates

Mix 10 g of the silica gel (of TLC grade, particle size 10 µm 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 min, by keeping in an oven maintained at 105 °C.

G.2 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.

G.3 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 h) at room temperature (25 °C to 30 °C). Observe the spots obtained under ultra violet light at 254 nm or 366 nm.

G.4 Results

G.4.1 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.

G.4.2 Any other spots on the chromatogram indicate extraneous dyes.

G.4.3 For comparison, use a reference sample prepared from standard henna powder.

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Annex H
(normative)

Determination of Lawsone pigment content

H.1 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.

H.2 Apparatus

Spectrophotometer or photoelectric colorimeter, with a filter of 490 nm

H.3 Reagents

H.3.1 Sodium bicarbonate solution, 5 % (m/v)

H.3.2 2-Hydroxy-1,4-naphthoquinone

H.4 Procedure for preparation of Standard Calibration Curve

H.4.1 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 % under the same condition as described in H.4.2 below.

H.4.2 Weigh 2.0 g of the prepared 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 h. Allow to settle overnight, there-after filter the solution through a filter paper and reject the first few millilitre. 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.

H.5 Calculation

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

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Annex I
(normative)

Determination of fineness

I.1 Reagent

Denatured spirit, filtered

I.2 Procedure

Place about 10 g of the sample, accurately weighed, in the specified sieve and wash by means of a low stream of running tap water and finally with fine stream from a wash bottle until, as much material as would pass through the sieve has passed. In case the material is not easily wetted by water, the washing could be started with a slow stream of filtered denatured spirit. Let the water drain from the sieve and then dry the sieve containing the residue on a steam bath. Transfer the residue onto a tared watch glass carefully and dry it to constant mass at 105 °C ± 2 °C.

I.3 Calculation

The material retained on the specified sieves content, expressed as percent, shall be calculated as follows;

$$100 \times \frac{M_1}{M_2}$$

where

M_1 is the mass, in grams, of the residue retained on the specified sieve and

M_2 is the mass, in grams, of the sample taken for test.

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