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Herbal petroleum jelly — Specification

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Rwanda Standards Board

P.O Box 7099 Kigali-Rwanda

Tel. +250 252 586103/582945

Toll Free: 3250

E-mail: info@rsb.gov.rw

Website: www.rsb.gov.rw

Contents

Page

Foreword

Rwanda Standards are prepared by Technical Committees and approved by Rwanda Standards Board (RSB) Board of Directors in accordance with the procedures of RSB, in compliance with Annex 3 of the WTO/TBT agreement on the preparation, adoption and application of standards.

The main task of technical committees is to prepare national standards. Final Draft Rwanda Standards adopted by Technical committees are ratified by members of RSB Board of Directors for publication and gazettment as Rwanda Standards.

DRS 361 was prepared by Technical Committee RSB/TC 011, Cosmetics, toiletries and surface active agents.

In the preparation of this standard, reference was made to the following standards:

IS 15735: 2006 *Herbal cosmetics — General guidelines*

US 191: 2016 *Petroleum jelly — Specification (3rd Edition)*
The assistance derived from the above source is hereby acknowledged with thanks.

Committee membership

The following organizations were represented on the Technical Committee on *Cosmetics, toiletries and surface active agents* (RSB/TC 011) in the preparation of this standard.

ALYVO Rwanda Ltd

Better Home Ltd

EDEN BUSINESS CENTER Ltd

Halth Care Pharmacy

Kacyiru Hospital (KH)

Ministry of Health (MoH)

National Industrial Research and Development Agency (NIRDA)

Pharmacie Conseil

Pharmacie Nova

Private Sector Federation/Beauty Makers Association (PSF/BMA)

Rwanda Biomedical Center (RBC/MPPD)

Rwanda Dermatology Society (RDS)

Rwanda National Police (RNP/CID)

SULFO Rwanda Industries Ltd

The Ihangane Project (TIP)

University of Rwanda — College of Science and Technology (UR — CST)

ZIRUMUZE Cooperative

Rwanda Standards Board (RSB) — Secretariat

Herbal petroleum jelly— Specification

1 Scope

This Draft Rwanda Standard specifies the requirements and methods of testing for herbal petroleum jelly.

This document does not apply to products intended to be used for medicinal purpose.

2 Normative references

The following referenced documents are indispensable for the application 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.

DRS 333, *Herbal cosmetics products – General requirements*

ISO 18664, *Traditional Chinese Medicine — Determination of heavy metals in herbal medicines used in Traditional Chinese Medicine*

RS EAS 346, *Labelling of cosmetics — General requirements*

RS EAS 377*(all parts)*, *Cosmetic and cosmetic products*

RS 278, *Cosmetics – Methods of sampling*

RS EAS 123, *Distilled water - Specification*

ASTM D217 – 10*, Standard Test Methods for Cone Penetration of Lubricating Grease*

3 Terms and definitions

For the purposes of this standard, the following terms and definitions apply.

3.1
herbal petroleum jelly
hereinafter referred to as “petroleum jelly” is formulated, using various permissible ingredients and to form the base in which one or more herb(s)/herbal ingredient(s) are used to provide defined product benefits with a label declaration as ”herbal petroleum jelly”

NOTE Terms and definitions quoted in DRS 333 should be applied on this document.

4 Requirements

4.1 General requirements

4.1.1 To ensure the quality of the product and the well being of the consumer, herbal petroleum jelly shall conform to requirements prescribed in DRS 333.

4.1.2 All the ingredients used shall conform to the requirements in RS EAS 377 (all parts).

4.2 Physical requirements

4.2.1 Solubility

Herbal petroleum jelly shall be insoluble in water and ethanol (96 %), but soluble in ether and chloroform. In cosmetic spirit (boiling range 40 °C – 60 °C) the solution sometimes shows a slight opalescence.

4.2.2 Colour

The colour of herbal petroleum jelly shall be characteristic of plant used.

4.2.3 Odour

The material shall be of acceptable odour at room temperature when rubbed on the skin.

4.3 Specific requirements

4.3.1 Herbal petroleum jelly shall comply with the requirements given in Table 1 when tested according to the methods given in Annex A. Reference to the relevant clauses of Annex A is given in the third column of the table.

Table 1 — Requirements for herbal petroleum jelly

S/N	Characteristics	Requirements	Method of test (Clause number to be referred to)
1.	Herbal content % by mass, min	1.5	-
2.	Kinematic viscosity at 100°C Cst	4 to 6.8	A.2
3.	Melting point °C	40 to 60	A.3
4.	Specific gravity at 60°C, g/cc	0.815 to 0.880	A.4
5.	pH	4.5 – 7.0	A.5
6.	Saponifiable matter	Nil	A.6
7.	Organic acids	To pass the test	A.7
8.	Sulphated ash, % by mass, max	Not greater than 0.10	A.8
9.	Sulphur and sulphides	To pass test	A.9
10.	Iodine Value (Wijs) max	1.5	A.12
11.	Light absorption	Not greater than 0.8	A.13
12.	Cone penetration value at 25°C	100-275 1/10mm in checking for consistency and hardness of jellies	ASTM-D 217
13.	Volatile matter, % m/m, max.	5	A.14

5.5.2 The products shall comply with the requirements for contaminants in accordance with Table 2.

Table 2 — Requirements for contaminants

S/N	Characteristic	Requirement	Method of test
1.	Lead, mg/kg, max	20	A.11
2.	Arsenic, mg/kg, max	2	A.10
3.	Mercury, mg/kg, max	2	A.11
NOTE The total amount of heavy metals as lead, mercury and arsenic, biological contaminants and prohibited and restricted ingredients in combination, in the finished product should not exceed 20 mg/kg.			

5 Sampling

For the purpose of this Rwanda Standard, general precaution, scale of sampling and preparation of test samples shall be done as prescribed in RS 278.

6 Packaging, labelling and storage

6.1 Packaging

Herbal petroleum jelly shall be packaged in containers that shall protect the contents, effectively screen the content from UV light when stored and shall not cause any contamination or react with the product.

6.2 Labelling

In addition to the labelling requirements of RS EAS 346, the following information shall be indelibly and legibly marked on the container:

a) product name that is “Herbal petroleum jelly”;

b) percentage of herbal preparations used;

6.3 Storage

During storage all packages shall be covered and ingress of water shall be avoided. The products shall not be stored above 40°C, exposed to hot sun or freezing conditions.

Annex A
(normative)

Method of test for herbal petroleum jelly products

A.1 Quality of reagents

Unless specified otherwise, analytical grade reagents and distilled water (as described in EAS 123) shall be employed in test.

A.2 Determination of kinematic viscosity

A.2.1 Determination

The kinematic viscosity is determined by using the viscometers. The specific details of operation vary for different types of viscometers.

A.2.2 Procedure

The time is measured for a fixed volume of sample, contained in a glass of viscometer, to flow through a calibrated capillary under an accurately reproducible head of liquid and at 100 °C. This temperature must be controlled. The viscometer selected should give an efflux time greater than 200 s. The kinematic viscosity is calculated from the measured efflux time. The viscometer is calibrated by using standard oil having viscosities established with reference to water in master viscometers or by direct comparison with carefully calibrated viscometers. The temperatures of the bath used shall be maintained within ± 0.01 °C.

A.3 Determination of melting point

A.3.1 Melt a quantity of the sample slowly while stirring until it reaches a temperature of 90 °C to 92 °C. Remove the source of heat and allow the molten sample to cool to a temperature of 8 °C to 10 °C above the expected melting point. Chill the bulb of a thermometer (range: 1 °C to 100 °C) to 5 °C, wipe it dry and while it is still cold, dip it into molten sample so that approximately half of the bulb is “submerged" Withdraw it immediately and hold it vertically away from heat until the wax surface dulls, then dip it for 5 min into a water bath having a temperature not higher than 16 °C.

A.3.2 Fix the thermometer prepared in A.3.1 securely in a test tube so that its lowest point is about 15 mm above the bottom of the test tube. Suspend the test tube in a water bath adjusted to 16 °C, and raise the temperature of the bath at a rate of 1 degree/min and note the temperature at which the first drop of the melted sample leaves the thermometer. Repeat the determination twice on a freshly melted portion of the sample. If the variation in three determinations is less than one degree take the average of three as the melting point. If the variation in the three determinations is more than one degree, make two additional determinations and take the average of the five.

A.4 Determination of specific gravity

A.4.1 Apparatus

A.4.1.1 Specific gravity bottle, 25 mL capacity, with a well-fitting ground glass stopper with a capillary

A.4.1.2 Water bath, maintained at 60 °C ± 1 °C

A.4.2 Procedure

A.4.2.1 Clean and dry the specific gravity bottle, and weigh it. Then fill it with water, insert the stopper and immerse in the water bath at 60 °C ± 1 °C. Keep the entire bulb completely immersed in water and hold at that temperature for 1 h. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, cool to room temperature and weigh.

A.4.2.2 Melt approximately 40 g of the material in a porcelain dish and fill the dry specific gravity bottle with it. Keep the bottle for 1 h in a water bath at 60 °C ± 1 °C. Carefully remove any material which exudes from the capillary opening, wipe the bottle dry and cool at room temperature and weigh.

A.4.3 Calculation

$$\text{Specific gravity } 60°C/60°C = \frac{m_1 - m_2}{m_3 - m_2}$$

Where

m_1 is the mass, in grams, of specific gravity bottle with the material

m_2 is the mass, in grams, of the specific of the gravity bottle

m_3 is the mass, in grams, of the specific gravity bottle with water

A.5 Acidity and alkalinity

A.5.1 Reagents

A.5.1.1 Phenolphthalein indicator solution, 1 % solution in 95 % rectified spirit.

A.5.1.2 Methyl orange indicator, dissolve 0.01 g of methyl orange in 100 ml of water.

A.5.2 Procedure

A.5.2.1 Take 35 g, of the sample in a 250 mL separating funnel. Add to it 100 mL of boiling water and shake vigorously for 5 min. Draw off the separated water layer in the beaker. Wash the sample further with two 50 mL portions of boiling water and add the washings again to the beaker. To the collective washings add one drop of phenolphthalein indicator solution and boil. If no pink colour is produced, add 0.1 ml of methyl orange indicator and see if any red or pink colour is produced.

A.5.2.2 The sample shall be taken to have passed the test if neither a red colour is produced with phenolphthalein nor a pink colour produced.

A.6 Determination of saponifiable matter

A.6.1 Reagents

A.6.1.1 Methyl ethyl ketone, analytical grade, stored in amber coloured bottle.

A.6.1.2 Standard alcoholic potassium hydroxide solution, 0.5 mol/L standardized before use.

A.6.1.3 Herbal cosmetic ether, boiling range 80 °C to 100 °C.

A.6.1.4 Standard hydrochloric acid, 0.5 mol/L accurately standardized.

A.6.1.5 Phenolphthalein indicator solution, same as in A.5.1.1.

A.6.2 Procedure

A.6.2.1 Accurately weigh in flask about 5 g of the sample and add 25 mL ± 1 mL of methyl ethyl ketone, followed by 25mL standard alcoholic potassium hydroxide solution from a burette. Connect the flask to a condenser and heat for half an hour after refluxing begins. Disconnect the condenser, add 50 mL of petroleum ether and titrate the solution while hot (without heating) with standard hydrochloric acid, using three drops of phenolphthalein indicator. When the indicator colour is discharged add three drops more of the indicator. If this addition restores the colour, continue the titration. Proceed in this manner until the end point is reached when the indicator colour is discarded and does not immediately reappear upon the addition of three more drops of indicator.

A.6.2.2 The sample shall be taken to have passed the requirement prescribed in Table 1 if the blank reading does not differ from the sample reading by more than 0.1 mL.

A.7 Test for organic acids

A.7.1 Reagents

A.7.1.1 Dilute rectified spirit, prepared by diluting one volume of 95 % rectified spirit with two volumes of water, and neutralized to phenolphthalein indicator.

A.7.1.2 Phenolphthalein indicator, same as in A.5.1.1.

A.7.1.3 Standard sodium hydroxide solution, exactly 0.1 mol/L.

A.7.2 Procedure

A.7.2.1 Add 100 mL of dilute rectified spirit to 20 g of the sample, agitate thoroughly, and heat to boiling. Add 1 ml of phenolphthalein indicator and titrate rapidly with standard sodium hydroxide solution with vigorous agitation to a sharp pink end point in the alcohol water layer.

A.7.2.2 The material shall be taken to have passed the test if not more than 0.4 mL of standard sodium hydroxide solution is required for the titration.

A.8 Determination of sulphated ash

A.8.1 Reagents

A.8.1.1 Dilute sulfuric acid, approximately 2.5 mo1/L

A.8.1.2 Heat a platinum dish to redness for 10 min; allow to cool in a desiccator and weigh. Place 1 g of the sample in the dish, moisten with sulfuric acid, and ignite gently by means of a Bunsen burner. Again moisten with sulfuric acid and ignite at about 800 °C in a muffle furnace. Cool and weigh, again ignite for 15 min and repeat this procedure until two successive weighings do not differ by more than 0.5 mg.

A.8.2 Calculation

$$\text{Sulfated ash, percent by mass} = \frac{m_1 x 100}{m_2}$$

where

m_1 is the mass in grams of the residue;

m_2 is the mass in grams of the sample taken for the test.

A.9 Determination of sulfur and sulfides

A.9.1 Reagents

Copper strips, 1 cm in width, and freshly polished.

A.9.2 Procedure

A.9.2.1 Melt in a beaker about 100 g of the sample and keep on a water bath at a temperature of 95 °C. Then place a strip of copper in the melted sample so that it is partially immersed in it and allow to remain for 10 min.

A.9.2.2 The material shall be taken to have passed the test if the copper strip used in the test shows no tarnishing when compared with another freshly polished copper strip.

A.10 Determination of arsenic

A.10.1 Reagents

A.10.1.1 Concentrated sulfuric acid, reagent grade.

A.10.1.2 Concentrated nitric acid, reagent grade.

A.10.2 Preparation of sample

A.10.2.1 Weigh 2.00 g of the sample in a Kjeldahl flask of 500 ml capacity. Add 15 ml of concentrated sulfuric acid followed by 4 ml of concentrated nitric acid. Heat cautiously. Add drop-by-drop more nitric acid, if required, from a pipette to speed up the oxidation of the sample. The total amount of nitric acid shall be noted for use in control test. When oxidation is complete the solution is clear and faint yellow, at that stage, add 20 ml of water and again boil to fuming, Ensure removal of all nitric acid.

A.10.2.2 Carry out test for arsenic with the solution prepared in A.10.2.1 as per Clause 2 of EAS 101. Compare the stain obtained with that produced with 0.004 g of arsenic trioxide.

A.11 Determination of heavy metals

A.11.1 Reagents

A.11.1.1 Ammonium acetate solution, 10 %.

A.11.1.2 Ammonium citrate solution, dissolve 8.75 g of citric acid in water, neutralize with ammonia and dilute with water to 100 mL.

A.11.1.3 Ammonium hydroxide, 10 % (m/m).

A.11.1.4 Potassium cyanide solution, 10 %.

A.11.1.5 Sodium sulfide solution, 10 %.

A.11.1.6 Standard lead solution, dissolve 16 g of lead nitrate in water and 10 mL of concentrated HNO_3 and dilute to 1000 ml. Pipette out 10 mL of the solution and dilute again to 1000 mL with water. 1 mL of the final solution contains 0.01 mg of lead (as Pb). The solution should be freshly prepared.

A.11.2 Procedure

A.11.2.1 In the preparation of sample treat 2.0 g of the sample as prescribed in A.10.2.

A.11.2.2 Take the solution prepared in A.11.2.1 in a Nessler tube (with 50 mL capacity); add 10 mL of ammonium acetate solution, (5 ml capacity); add 10 mL of ammonium acetate solution, 5 mL of ammonium hydroxide and 1 mL of potassium cyanide solution and dilute to 50 mL with water, then add two drops of sodium sulfide solution and mix well. In another Nessler tube carry out a control test using 4 mL of standard lead solution and same quantities of other reagents as used in the test with the material.

A.11.2.3 The material shall be taken to have passed the test as given in Table 1 if the intensity of colour produced with material is not greater than that produced in the control test.

A.12 Determination of iodine value

A.12.1 Apparatus

The material is treated with a known excess of iodine monochloride solution in glacial acetic acid. The excess of iodine monochloride is determined iodometrlcally. Thermometer, an engraved stem thermometer, calibrated between 10 °C and 65 °C in 0.1 degree intervals and with the 0 °C point marked on the steam is recommended. The thermometer shall have an auxiliary reservoir at the upper end, and length of about 370 mm and diameter of about 6 mm.

A.12.2 Reagents

A.12.2.1 Carbon tetrachloride or chloroform.

A.12.2.2 Acetic acid, glacial. 99 %, having a melting point of 14.8 °C and free from reducing impurities. Determine the melting point of the acetic acid and test it for reducing impurities as it follows:

A.12.2.2.1 Melting point determination — Take a 15 cm long test tube and fill it to about two thirds with the acetic acid. Insert into the acid a thermometer satisfying the requirements specified in A.12.1 through a cork stopper fitting the test tube. The amount of acid should be at least double the quantity required to cover the bulb of the thermometer when the bottom of the latter is 12 mm from the bottom of the test tube. Suspend this tube within a larger test tube through a cork. Cool the acid by immersing the assembly in ice water until the temperature is 10 °C, then withdraw the assembly from the ice water and stir the acid rather vigorously for a few moments, thus causing the super-cooled liquid to crystallize partially and give: a mixture of liquid and solid

acid. Take thermometer readings every 15 s and consider the temperature at which the reading remains constant for at least 2 min as the true melting point.

A.12.2.2.2 Test for reducing impurities — Potassium permanganate test, dilute 2 mL of acetic acid with 10 mL of water and add 0.1 mL of 0.5 mL potassium permanganate solution and maintain at 27 °C ± 2 °C. The test shall be taken as having been satisfied if the pink colour is not discharged at the end of 2 h.

A.12.2.3 Potassium dichromate, finely ground.

A.12.2.4 Starch solution, mix 5 g of starch and 0.01 g of mercuric iodide with 30 mL of cold water and slowly pour it while stirring into 1 litre of boiling water. Boil for 3 min. Allow the solution to cool and decant off the supernatant clear liquid.

A.12.2.5 Standard sodium thiosulfate solution, 0.2 mol/L.

A.12.2.6 Chlorine gas, dry.

A.12.2.7 Iodine trichloride.

A.12.2.8 Wijs iodine monochloride solution, prepare this solution by one of the following methods, and store in a glass stoppered bottle in a cool place, protected from light and sealed with paraffin until taken for use.

A.12.2.8.1 Dissolve 3 g of re-sublimed iodine in 1 L of acetic acid, using gentle heat if necessary, and determine the length by titration with standard sodium thiosulfate solution. Set aside 50 mL to 100 mL of solution and introduce washed and dried chlorine gas into the remainder until the characteristic colour change occurs and the halogen content is nearly doubled as ascertained again by titration. If the halogen content has been more than doubled, reduce it by adding the requisite quantity of the iodine - acetic acid solution. A slightly excess of iodine does not harm, but avoid an excess of chlorine.

For example, if the titration of 20 mL of original iodine acetic acid solution requires 22 mL of standard sodium thiosulfate solution then 20 mL of the finished Wijs solution should require between 43 mL and 44 mL (and not more than 44 mL) of the same sodium thiosulfate.

A.12.2.8.2 As an alternative method of preparing Wijs solution dissolve 8 g of iodine trichloride inapproximately 450 ml of acetic acid. Dissolve separately 9 g of iodine in 450 ml, of acetic acid using heat if necessary. Add gradually the iodine solution to the iodine trichloride solution until the colour has changed to reddish brown. Add 50 ml more of iodine solution and dilute the mixture with acetic acid till 10 ml of the mixture are equivalent to 20ml standard sodium thiosulfate solution when the halogen content is estimated by titration in the presence of an excess to potassium iodine and water. Heat the solution at 100 °C for 20 min and cool.Prevent access of water vapour in preparing the solution.

A.12.3 Procedure

A.12.3.1 Melt the material and filter through the filter paper to remove any impurities and the last trace of moisture. Make sure that the glass apparatus used is absolutely clean and dry. Weigh accurately by difference, about 10 g of the sample, into a clean, dry 500 mL glass stoppered bottle to which 25 mL of carbon tetrachloride or chloroform have been added, and agitate to dissolve the contents. Add 25 ml of Wijs solution (the quantity of Wijs solution added is 50 % - 60% more than the quantity required).

A.12.3.2 Replace the glass stopper after wetting with potassium iodine solution, swirl for intimate mixing, and allow to stand in the dark for 45 min. Carry out a blank test simultaneously under similar experimental conditions. After standing add 15 mL of potassium iodide solution and 100 mL of water, and titrate the liberated iodine with standard sodium thiosulfate solution, swirling the contents of the bottle continuously to avoid any local excess, until the colour of the solution is straw yellow. Add 0.5 mL of starch solution and continue the titration until the blue colour disappears.

A.12.4 Calculation

$$\text{Iodine value} = \frac{12.69(V_1 - V_2)M}{m}$$

where

V_1 is the volume, in millilitre, of standard sodium thiosulfate required for the blank;

V_2 is the volume, in millilitre, of standard sodium thiosulfate solution required for the material;

M is the molarity of standard sodium thiosulphate; and

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

A.13 Determination of light absorption

A.13.1 Apparatus

A.13.1.1 UV spectrophotometer

A.13.1.2 Make of solution of 0.05 % m/v of the herbal petroleum jellyin 2,2,4 – trimethylpentane, then determine the absorbency at 290 nm

A.14 Determination of volatile matter

A.14.1 Outline of the method

The sample is weighed in petri/glass dish and kept in an oven maintained at 105 ± 1^0C. the loss in mass is calculated as percentage of the mass of the sample taken.

A.14.2 Apparatus

A.14.2.1 Petri/Glass dishes – Made of heat resistant glass, 90 to 100 mm in diameter and 7 to 10 mm in height.

A.14.2.2 Thermometer – Any suitable thermometer having a range from 0 to 110^0C.

A.14.2.3 Air-Oven – An electrically or gas heated air oven capable of maintaining temperature at 105 ± 1^0C.

A.14.3 Procedure

Weigh about 10g of the sample, accurately to the nearest 0.1g, in a tare petri/glass dish, distributing the sample in as uniform a layer as possible with the help of a spatula over the whole of bottom of the dish. Keep it in the oven, maintained at 105 ± 1^0C for 24 h. cool and weigh the petri/glass dish to a constant mass.

A.14.4 Calculation and report

Calculate and report the evaporation loss as follows:

$$\text{Volatile matter per cent} = 100\ \frac{M1-M2}{M1}$$

Where,

M1 = mass in g of the sample taken for the test

M2 = mass in g of the sample after the test

ICS 71.100.70