



DRAFT TANZANIA STANDARD

Petroleum jelly for cosmetic industry — Specification

Draft Standard for Comment Only

TANZANIA BUREAU OF STANDARDS

TBS/CDC 3(4243) CD2

Foreword

This Draft Tanzania Standard was prepared by the Cosmetics and Creameries Technical Committee, under the supervision of the Chemicals Divisional Standards Committee, and it is in accordance with the procedures of the Bureau.

This third edition cancels and replaces the second edition, TZS 318-1:2017, which has been technically revised.

In reporting the result of a test or analysis made in accordance with this Tanzania Standard, if the final value, calculated or observed, is to be rounded off, it shall be done in accordance with TZS 4.

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Petroleum jelly for cosmetic industry — Specification

1. Scope

This Draft Tanzania Standard specifies the requirements, sampling and test methods for petroleum jelly used in the cosmetic industry.

This Draft Tanzania Standard applies to petroleum jelly used in the manufacture of various types of cosmetics, such as creams, lipsticks, hair dressings, lubricating creams, rouges, foundation creams of a greasy type, and employed as an emollient for chapped skin.

2. Normative references

The following referenced documents are indispensable for the application of this Tanzania Standard. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies:

TZS 59, *Water for analytical laboratory use — Specification and test method*

TZS 76, *General method for determination of arsenic, silver diethyldithiocarmate photometric method*

TZS 314, *Cosmetics and toiletries products — Methods of sampling*

TZS 2126/EAS 846, *Glossary of terms relating to the cosmetic industry*

TZS 2127-16/EAS 847-16 *Cosmetics — Analytical methods — Part 16: Determination of lead, mercury and arsenic content*

3. Terms and definitions

For the purposes of this document, the terms and definitions given in TZS 2126 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>

— IEC Electropedia: available at <http://www.electropedia.org/>

4. Requirements

4.1 General requirements

- a) Petroleum jelly shall be a translucent, soft mass, unctuous to the touch, and shall retain these characteristics on storage.
- b) Petroleum jelly shall be free from aromatic compounds and any extraneous colouring matter.
- c) Petroleum jelly shall be tasteless and odourless. It shall also not be fluorescent in daylight.
- d) Petroleum jelly shall be practically insoluble in water and ethanol (96 %), soluble in ether and chloroform. In petroleum spirit (boiling range 40 °C – 60 °C), the solution sometimes shows a slight opalescence.

- e) The colour of the material shall be either yellow or white.
- f) The material shall be odourless at room temperatures when rubbed on the skin. When heated to between 95 °C and 98 °C on a boiling water bath for thirty minutes, it shall give no acrid odour.

4.2 Specific requirements

The material shall comply with the requirements given in Table 1 when tested according to the methods given in Table 1.

Table 1 – Requirements for petroleum jelly for cosmetic industry

S/No	Characteristic	Requirement	Test method
i.	Kinematic viscosity at 100 °C Cst	4.5-8.0	Annex A
ii.	Melting point; °C	38 - 56	Annex B
iii.	Specific gravity at 60 °C	0.815 - 0.880	Annex C
iv.	Acidity and alkalinity	Neutral	Annex D
v.	Saponifiable matter	NIL	Annex E
vi.	Organic acids	To pass the test	Annex F
vii.	Sulphated ash, % by mass, max.	0.10	Annex G
viii.	Sulphur and sulphides	To pass the test	Annex H
ix.	Arsenic (as As ₂ O ₃) mg/kg max.	2	TZS 2127-16/EAS 847-16
x.	Heavy metal (as Pb) mg/kg, max.	20	
xi.	Iodine value (Wijs), max.	1.5	Annex I
xii.	Light absorption, max.	0.5	Annex J

5. Packaging, marking, and storage

5.1 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. The size and design of the container shall be agreed upon between the purchaser and the supplier.

5.2 Marking

The packages shall be securely closed, legibly and indelibly marked in Kiswahili and/or English, and any other language as agreed between the manufacturer and supplier, with the following information:

- a) name of the material;
- b) quantity of the material;
- c) address and trademark, if any;
- d) manufacturer's name;

- e) batch number in code or otherwise;
- f) date of manufacture;
- g) expiry date; and
- h) country of origin.

5.3 Storage

All packages shall be stored under cover; ingress of water should be avoided. The products should not be stored above 60 °C, exposed to hot sun or freezing conditions.

5. Sampling

For the purpose of deciding whether petroleum jelly product conforms to the requirements of this Tanzania Standard, representative samples shall be collected for test primarily from the factory and also from anywhere else following the procedure of random selection in accordance with TZS 314 (see clause 2). The containers shall only be opened during testing.

6. Test method

Quality of reagents

Unless specified otherwise, analytical grade reagents and water of distilled quality as described in TZS 59 (see clause 2) shall be employed in tests.

Annex A
(normative)

Determination of kinematic viscosity

A.1 Outline of the method

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

A.1.1 Procedure

The time is measured for a fixed volume of sample, contained in a glass of a 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 must be maintained within ± 0.01 °C.

Annex B
(normative)

Determination of melting point

B.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 the 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 five minutes into a water bath having a temperature not higher than 16 °C.

B.2 Fix the thermometer prepared in B.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.

Annex C
(normative)

Determination of specific gravity

C.1 Apparatus

C.1.1 Specific gravity bottle, 25 ml capacity, with a well-fitting ground glass stopper with a capillary.

C.1.2 Water bath, maintained at 60 °C ± 1 °C.

C.2 Procedure

C.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 one hour. Carefully remove any water that has exuded from the capillary opening. Remove from the bath, wipe completely dry, cool to room temperature, and weigh.

C.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 one hour 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.

C.3 Calculation

$$\text{Specific gravity, } 60\text{ }^{\circ}\text{C} = \frac{m_1 - m_2}{m_3 - m_2}$$

Where

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

m_2 = mass in grams of the specific gravity bottle, and

m_3 = mass in grams of the specific gravity bottle with water.

Annex D
(normative)

Determination of acidity and alkalinity

D.1 Reagents

D.1.1 Phenolphthalein indicator solution, 1 % solution in 95 % rectified spirit.

D.1.2 Methylorange indicator: Dissolve 0.01 g of methyl orange in 100 ml of water.

D.2 Procedure

Take 35 g of the sample in a 250 ml separating funnel. Add to it 100 ml of boiling water and shake vigorously for five minutes. 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.

The sample shall be taken to have passed the test if neither a pink colour is produced with phenolphthalein nor a red or pink colour is produced with methyl- orange.

Annex E
(normative)

Determination of saponifiable matter

E.1 Reagents

E.1.1 Methyl ethyl ketone, analytical grade, stored in amber coloured bottle.

E.1.2 Standard alcoholic potassium hydroxide solution, 0.5 mol/L standardized before use.

E.1.3 Petroleum ether, boiling range 80 °C to 100 °C.

E.1.4 Standard hydrochloric acid, 0.5 mol/L accurately standardized.

E.1.5 Phenolphthalein indicator solution, same as in D.1.1.

E.2 Procedure

Accurately weigh into a flask about 5 g of the sample and add 25 ml \pm 1 ml of methyl ethyl ketone, followed by a 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 endpoint is reached, when the indicator colour is discarded and does not immediately reappear upon the addition of three more drops of indicator.

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.

Annex F
(normative)

Test for organic acids

F.1 Reagents

F.1.1 Dilute rectified spirit, prepared by diluting 1 volume of 95 % rectified spirit with 2 volumes of water, and neutralized to phenolphthalein indicator.

F.1.2 Phenolphthalein indicator, same as in D.1.1.

F.1.3 Standard sodium hydroxide solution, exactly 0.1 mol/L.

F.2 Procedure

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 endpoint in the alcohol water layer.

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.

Annex G
(normative)

Determination of sulphated ash

G.1 Reagents

Dilute sulfuric acid. Approximately 2.5mol/L.

G.2 Procedure

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 for 2 hours. Cool and weigh, again ignite for 15 min and repeat this procedure until two successive weightings do not differ by more than 0.5 mg.

G.3 Calculation

$$\text{Sulphated ash, \% by mass} = \frac{m_1 \times 100}{m_2}$$

where

m₁ = mass in grams of the residue, and

m₂ = mass in grams of the sample taken for the test.

Annex H
(normative)

Determination of sulphur and sulphides

H.1 Reagents

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

H.2 Procedure

Melt about 100 g of the sample in a beaker and keep it 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 it to remain for 10 min.

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.

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Annex I
(normative)

Determination of iodine value

I.1 Outline of the method

The material is treated with a known excess of iodine monochloride solution in glacial acetic acid. The excess of iodine monochloride is determined iodometrically.

I.2 Apparatus

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 stem, is recommended. The thermometer shall have an auxiliary reservoir at the upper end, a length of about 370 mm and a diameter of about 6 mm.

I.3 Reagents

I.3.1 Carbon tetrachloride or chloroform

I.3.2 Acetic acid

Glacial acetic acid, 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 follows:

a) 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 I.2 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.

b) 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 M 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.

I.3.3 Potassium dichromate, finely ground.

I.3.4 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 L of boiling water. Boil for 3 min. Allow the solution to cool and decant off the supernatant clear liquid.

I.3.5 Standard sodium thiosulphate solution, 0.2mol/L.

I.3.6 Chlorine gas, dry

I.3.7 Iodine trichloride

I.3.8 Wijs iodine monochloride solution

Prepare this solution by one of the following two methods, and store in a glass-stoppered bottle in a cool place, protected from light and sealed with paraffin until taken for use.

a) Dissolve 13 g of re-sublimed iodine in 1 L of acetic acid, using gentle heat if necessary, and determine the strength by titration with standard sodium thiosulphate 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 slight excess of iodine does not harm, but avoid an excess of chlorine.

Example: If the titration of 20 ml of original iodine acetic acid solution requires 22 ml of standard sodium thiosulphate solution, then 20 ml of the finished Wijs solution requires between 43 ml and 44 ml (and not more than 44 ml) of the same sodium thiosulphate.

b) As an alternative method of preparing Wijs solution, dissolve 8 g of iodine trichloride in approximately 450 ml of acetic acid. Dissolve separately 9 g of iodine in 450 ml of acetic acid using heat if necessary. Add the iodine solution to the iodine trichloride solution gradually 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 is equivalent to 20 ml standard sodium thiosulphate solution when the halogen content is estimated by titration in the presence of an excess of potassium, iodine and water. Heat the solution at 100 °C for 20 min and cool. Prevent access to water vapour in preparing the solution.

I.4 Procedure

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 has 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. 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 thiosulphate 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.

I.5 Calculation

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

where,

V_1 = volume in millilitres of standard sodium thiosulphate solution required for the blank,

V_2 = volume in millilitres of standard sodium thiosulphate solution required for the material,

M = Molarity of standard sodium thiosulphate solution, and

m = mass in grams of the material taken for the test

Annex J
(normative)

Determination of light absorption

J.1 Apparatus

UV spectrophotometer

J.2 Procedure

Make a solution of 0.05 % *m/v* of the petroleum jelly in 2,2,4-trimethylpentane, then determine the absorbance at 290 nm.