

DRAFT TANZANIA STANDARD

Draft for comments only CDC3(5128)P3 Rev of TZS 318 (Part 1):1999

Petroleum jelly for cosmetic industry - Specification

TANZANIA BUREAU OF STANDARDS

0 Foreword

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

This draft Tanzania standard is the revision of TZS 318 (part 1):1999 Petroleum jelly for cosmetics industry – Specification.

Petroleum jelly consists essentially of microcrystalline waxes in association with substantial quantities of oil. Yellow petroleum jelly is a semi-solid mixture of hydrocarbons obtained from petroleum oils. The white petroleum jelly is the same except that, in addition to that it is bleached.

Petroleum jelly is used in the manufacture of various types of cosmetics. It is incorporated in the manufacture of creams, lipsticks, hair dressings, lubricating creams, rouges, foundation creams of greasy type etc. It is also employed as an emollient for chapped skin.

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.

In the preparation of this Tanzania Standard assistance was derived from IS 4887-2006 Specification for petroleum jelly for cosmetic industry published by Indian Standards Institution.

1 Scope

This draft Tanzania Standard prescribes the requirements and method of sampling and test for petroleum jelly for cosmetic industry.

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:

2.1 TZS 59- Water for analytical laboratory use – Specification and test method

2.2 TZS 76 General method for determination of arsenic silver diethyldithiocarmate photometric method

2.3 TZS 314 Cosmetics and toiletries products – Methods of sampling

3 Requirements

3.1 General requirements

3.1.1 Petroleum jelly shall be translucent, soft mass, unctuous to touch and shall retain these characters on storage.

3.1.2 Petroleum jelly shall be free from aromatic compounds and any extraneous colouring matter.

3.1.3 Petroleum jelly shall be tasteless and odourless. It shall also not be fluorescent in daylight.

3.1.4 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.

3.1.5 The colour of the material shall be either yellow or white.

3.1.6 The material shall be odourless at room temperatures when rubbed on the skin. Further, the material, when heated to between 95°C and 98°C on a boiling water bath for thirty minutes, shall give no acrid odour.

3.2 Specific requirements

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

Table 1 — Requirements for petroleum jelly for cosmetic industry

SN	Characteristic	Requirement	Method of test
1	Kinematic viscosity at 100°C Cst	6.8	Annex A
2	Melting point; °C	38 to 56	Annex B
3	Specific gravity at 60°C/60°C	0.815 to 0.880	Annex C
4	Acidity and alkalinity	Neutral	Annex D
5	Saponifiable matter	NIL	Annex E
6	Organic acids	To pass the test	Annex F
7	Sulphated ash, <i>per cent by mass, max</i>	0.10	Annex G
8	Sulphur and sulphides	To pass the test	Annex H
9	Arsenic (as As ₂ O ₃) <i>mg/kg max</i>	2	Annex I
10	Heavy metal (as Pb) <i>mg/kg, max</i>	20	Annex J
11	Iodine value (<i>Wijs</i>), <i>max</i>	1.5	Annex K
12	Light absorption, <i>max</i>	0.5	Annex L

4 Packaging, marking and storage

4.1 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 between the purchaser and the supplier.

4.2 Marking

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

- a) name of the material;
- b) mass of the material;
- c) manufacturers name, address and trade mark if any;
- d) batch number in code or otherwise, and
- e) date of manufacture.
- f) expiry date
- g) country of origin

4.3 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. 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 shall be employed in tests.

ANNEX A

DETERMINATION OF KINEMATIC VISCOSITY

A.1 Outline of the method

A.1.1 The kinematic viscosity is determined by using the viscometers. The specific details of operation vary for different types of viscometers.

A.1.2 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 seconds. The kinematic viscosity is calculated from the measured efflux time. The viscometer are 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 $\pm 0.01^\circ\text{C}$.

ANNEX B

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 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 the variation in the three determinations is more than one degree, make two additional determinations and take the average of the five.

ANNEX C

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 $60^{\circ}\text{C} \pm 1^{\circ}\text{C}$.

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 $60^{\circ}\text{C} \pm 1^{\circ}\text{C}$. Keep the entire bulb completely immersed in water and hold at that temperature for one hour. Carefully remove any water which has exuded from the capillary opening. Remove from the bath, wipe completely dry, cool to room temperature and weigh.

C.2.2 Melt approximately 40g 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 $60^{\circ}\text{C} \pm 1^{\circ}\text{C}$. Carefully remove any material which exudes from the capillary opening, wipe the bottle dry and cool at room temperature and weigh.

C.3 Calculation

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

where

m_1 - mass in grams of specific gravity bottle with the material

m_2 - mass in grams of the specific gravity bottle

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

ANNEX D

DETERMINATION OF ACIDITY AND ALKALINITY

D.1 Reagents

D.1.1 *Phenolphthalein indicator solution* - 1 per cent solution in 95% rectified spirit.

D.1.2 *Methylorange indicator*- Dissolve 0.01 g of methylorange 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 to five minutes. Draw off the separated water layer in the beaker. Wash the sample further with two 50 ml portion 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 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

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 in 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 end point 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

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 end point 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

DETERMINATION OF SULPHATED ASH

G.1 Reagents

Dilute sulphuric 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 1g of the sample in the dish, moisten with sulphuric acid ignite gently by means of a bunsen burner. Again moisten with sulphuric 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 0.5mg.

G.3 Calculation

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

where

m_1 = mass in grams of the residue

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

ANNEX H

DETERMINATION OF SULPHUR AND SULPHIDES

H.1 Reagents

Copper strips - 1 cm in width,

and freshly polished.

H.2 Procedure

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.

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.

ANNEX I

DETERMINATION OF ARSENIC

I.1 Reagents

I.1.1 Concentrated sulphuric acid -

analytical grade

I.1.2 Concentrated nitric acid -

analytical grade.

I.2 Preparation of sample

Weigh 2.00 g of the sample in Kjeldahl flask of 500 ml capacity. Add 15 ml of concentrated sulphuric 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.

Carry out test for arsenic with the solution prepared in I.2 as described in TZS 76. Compare the stain obtained with that produced with 0.004 g of arsenic trioxide.

ANNEX J

DETERMINATION OF HEAVY METALS

J.1 Reagents

J.1.1 *Ammonium acetate solution* - 10 %.

J.1.2 *Ammonium citrate solution* - Dissolve 8.75 g of citric acid in water, neutralize with ammonia and dilute with water to 100 ml.

J.1.3 *Ammonium hydroxide* - 10 % (m/m).

J.1.4 *Potassium cyanide solution* - 10 %.

Warning: This chemical is highly hazardous handle with care.

J.1.5 *Sodium sulphide solution* - 10 %.

J.1.6 *Standard lead solution* - Dissolve 1.6 g of lead nitrate in water, and 10 ml of concentrated HNO₃ and dilute to 1000 ml. Pipette out 10 ml of the solution and dilute again to 1000 ml with water. One millilitre of the final solution contains 0.01 mg of lead (Pb). The solution should be freshly prepared.

J.2 Procedure

J.2.1 *Preparation of sample;* Treat 2.00 g of the sample as prescribed in I.2.

J.2.2 Take the solution prepared in J.2.1 in a Nessler tube (with 50 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 sulphide 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.

J.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.

ANNEX K
DETERMINATION OF IODINE VALUE

K.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.

K.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, and length of about 370 mm and diameter of about 6 mm.

K.3 Reagents

K.3.1 Carbon tetrachloride or chloroform

K.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 K.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 12mm 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 seconds and consider the temperature at which the reading remains constant for at least two minutes as the true melting point.

b) Test for reducing impurities

Potassium permanganate test

Dilute 2 ml of acetic acid with 10ml of water and add 0.1ml 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 two hours.

K.3.3 Potassium dichromate - finely ground.

K.3.4 Starch solution - Mix 5g of starch and 0.01g of mercuric iodide with 30ml of cold water and slowly pour it while stirring into one litre of boiling water. Boil for three minutes. Allow the solution to cool and decant off the supernatant clear liquid.

K.3.5 Standard sodium thiosulphate

solution - 0.2mol/ l.

K.3.6 Chlorine gas - dry

K.3.7 Iodine trichloride

K.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 one litre 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 slightly 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 require 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 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 10ml of the mixture are equivalent to 20 ml standard sodium thiosulphate 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 minutes and cool. Prevent access of water vapour in preparing the solution.

K.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 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). Replace the glass stopper after wetting with potassium iodine solution, swirl for intimate mixing, and allow to stand in the dark for 45 minutes. 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.

K.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

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ANNEX L

DETERMINATION OF LIGHT ABSORPTION

L.1 Apparatus

UV spectrophotometer

L.2 Procedure

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

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