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DRAFT TANZANIA STANDARD

Milking jelly — Specification

TANZANIA BUREAU OF STANDARDS

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Foreword

This Draft Tanzania Standard has been 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 Standard is being prepared to ensure the safety and quality of milking jelly (sometimes named as milking salve) used to lubricate and moisturizing a cow's teats when milking. The use of milking jelly prevents the teats from cracking which can lead to diseases such as mastitis.

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.

Milking jelly — Specification

1. Scope

This Draft Tanzania Standard specifies the requirements, sampling and test methods for milking jelly.

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 768/ISO 3104 *Petroleum products – Transparent and opaque liquids – Determination of kinematics viscosity and calculation of dynamic viscosity*

ISO 21150 *Cosmetics – Microbiology – Detection of Escherichia coli*

ISO 21149 *Cosmetics – Microbiology – Enumeration and detection of aerobic mesophilic bacteria*

AOAC 999.10, *Test method for determination of Lead, Cadmium, Zinc, Copper and Iron in foods - Atomic absorption spectrophotometry after microwave digestion*

3. Terms and definition

Milking jelly
jelly applied on cow's teats when milking for lubrication and moisturizing purposes to prevents the teats from cracking and chapping.

NOTE: Other names such as milking salve, udder balm, is used interchangeably

4. Requirements

4.1 General requirements

4.1.1 Milking jelly shall be light-coloured, soft mass, unctuous to touch and shall retain these characters on storage.

4.1.2 Milking jelly shall be tasteless and odourless.

4.2 Specific requirements

4.2.1 Milking jelly shall comply with the requirements given in table 1.

Table 1 – Specific requirements for milking jelly

S/No	Characteristic	Requirement	Test method
i.	Kinematic viscosity at 100 °C cSt, *min*	6.8	TZS 768,
ii.	Melting point; °C	36 – 56	Annex B
iii.	Specific gravity at 60 °C/60 °C	0.815 to 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.	Sulfur and sulfides	To pass the test	Annex G
viii.	Iodine value (Wijs), max	1.5	Annex H
ix.	Cone Penetrometer 1/10, mm	160 -250	Annex I
x.	Heavy metal (as Pb) mg/kg, *max*.	0.02	AOAC 999.10

Table 2 Microbial Analysis

Total plate count	<150 cfu/g - ISO 21149
E.coli	Not to be detected - ISO 21150

5. Packaging and labelling

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.

5.2 Labelling

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

a) name of the product;
b) name and address of manufacturers;
c) trade mark if any;
d) net content;
e) list of ingredients
f) batch number;
g) date of manufacture and best before date;

h) country of origin; and
i) instructions for use, storage.

6. Sampling

For the purpose of deciding whether milking jelly conforms to the requirements of this Tanzania Standard, representative samples shall be collected in accordance with Annex K.

7. Test method

Quality of reagents

Unless specified otherwise, analytical grade reagents and distilled water as described in TZS 59 shall be employed in tests.

Annex A
(normative)

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 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 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 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 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

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

where

m_1 = mass in grams of 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 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
(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 in a flask about 5 g of the sample and add 25 mL ± 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
(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 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
(normative)

Determination of sulfur and sulphides

G.1 Reagents

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

G.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 H
(normative)

Determination of iodine value

H.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.

H.2 Apparatus

Thermometer

An engraved stem thermometer, calibrated between 10 °C and 65 °C in 0.1degree 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 a length of about 370 mm and a diameter of about 6 mm.

H.3 Reagents

H.3.1 *Carbon tetrachloride or chloroform*
H.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 H.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.

H.3.3 *Potassium dichromate,* finely ground.
H.3.4 *Starch solution* - Mix 5.0 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.
H.3.5 *Standard sodium thiosulphate solution*, 0.2mol/L.
H.3.6 *Chlorine gas*, dry
H.3.7 *Iodine trichloride or resublimed Iodine*
H.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 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 10 ml 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 min and cool. Prevent access of water vapour in preparing the solution.

H.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 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.

H.5 Calculation

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

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 I
(normative)

Cone Penetrometer 1/10 mm

I.1 Procedure

Fill the sample cup with sample (make sure there is no porosity). Keep sample in the cup for cooling up to 25°C for min 8 hrs. Place the cup on the Penetrometer table.
Level the machine with leveling screw. Set the mechanism on the 'Zero' position and adjust the cone tip on the surface of sample at centre of test specimen. Power on the timer panel box and press reset button (It will allow cone to dip for 5.0+/-0.1 seconds) then read the penetration from the indicator in mm.

I.2 Precautions

Thoroughly clean the penetrometer cone carefully before each test with a soft cloth or paper wiper; and use only cups that are clean and free from any residue from pervious runs. When the interior plating of cup shows the indication of impurity, clean it.

Annex J
(normative)

Total aerobic microbial count

J.1 Procedure:
Pre-treat the sample as described below:

J.1.1 *Water soluble products:* Dissolve 10 g or dilute 10 mL of the sample preparation being examined in sterile Phosphate buffer and adjust the volume to 100 ml with the same medium.

J.1.2 *Water insoluble products* (non – fatty): Suspend 10 g or 10 mL of the sample preparation being examined in sterile Phosphate buffer medium and dilute to 100 mL with the same. Homogenize the suspension. In case of poorly wet table substances a suitable surface active agent such as 0.1 % w/v Polysorbate 80 is added to assist formation of suspension e.g. In case of Talc, Magnesium Stearate, Carbopol.

J.1.3 Fatty Products: Homogenise 10 g or 10 mL of the sample preparation being examined with 5 gm of Polysorbate 20 or Polysorbate 80. Add 85 mL of Phosphate buffer and mix. If required heat to not more than 40°C and maintain this temperature for the shortest time necessary for Plate count

J.2 Procedure

J.2.1 *For bacteria:* Pipette out 1 mL of the pre-treated preparation in each of the two sterile petri plates and bulk seed with about 15 mL of liquefied sterile Soya bean casein digest agar at not more than 45 °C. Incubate the Petri plates at 37 °C ±1 for 24 to 48 hrs. A negative control should be run along with the test sample. After incubation, count the number of colonies formed. Calculate the results using plates.

J.2.2 *For fungi:* Proceed as described in test for bacteria but use sterile Sabouraud Dextrose Agar/ Potato Dextrose Agar in place of Soya bean casein digest agar. Incubate the plates at 20 °C– 25 °C for 5 days. A negative control should be run along with the test sample. formation of an emulsion and in any case for not more than 30 min.

Annex K
(Normative)

Sampling of petroleum jelly

K.1 General requirements of sampling

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

K.1.1 Samples shall not be taken in an exposed place.
K.1.2 The sampling instrument shall be clean and dry.
K.1.3 Precaution shall be taken to protect the samples, the materials being sampled, the sampling instrument and the containers or samples from adventitious contamination.
K.1.4 To draw representative sample, the content of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.
K.1.5 The samples shall be kept in clean dry airtight glass or other suitable containers.
K.1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.
K.1.7 Each sample container shall be sealed airtight 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.

K.2 Scale of sampling

K.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 or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.
K.2.2 Samples shall be tested from each lot for ascertaining conformity of the material to the requirement of this specification
K.2.3 The number of containers (*n*) to be chosen from the lot shall depend on the size of the lot (*N*) and shall be as given in Table 2.
K.2.4 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose, random number tables shall be used. In case such tables are not available, the following procedure may be adopted. Starting from any container count 1, 2, 3 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.

Table 2 — Number of containers to be selected for sampling (K.2.3)

Lot size ***N***	**Number of containers to be selected** ***n***
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
Greater than 650	7

K.3 Test samples and referee sample

K.3.1 Preparation of test samples

K.3.1.1 Draw with an appropriate sampling instrument a small portion of the material from different pans of each container selected (see Table 2). The total quantity of the material drawn from

each container shall be sufficient to conduct the tests for all the characteristics given in Clause 4 and shall not be less than 250 g.

K.3.1.2 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantities shall be taken from each selected container and shall be well mixed up together so as to form a composite sample weighing not less than 0.5 kg. This composite sample shall be divided into three equal parts, one for the purchaser and the supplier and shall be used in case of dispute between the two.

K.4 Number of tests

Tests for all the characteristics given in Table 1 shall be conducted on the composite sample.

K.5 Criteria for conformity'

A lot shall be declared as conforming to this specification if the composite sample satisfies the requirements for each of the characteristics listed in Table 1. If the requirements for any of the characteristics are not met; the lot shall be declared to have not satisfied the requirements of the specification.

Bibliography

EAS 126:1999 Pure petroleum jelly for cosmetics industry — Specification

TZS 318-1: 2017 Petroleum jelly for cosmetic industry – Part 1: Specification