

## DRAFT TANZANIA STANDARD

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TBS/CDC-2(5415) P3- Baby toilet soap – Specification

TANZANIA BUREAU OF STANDARDS

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## Foreword

This Draft Tanzania Standard was developed by the Soap and Detergents Technical Committee under supervision of the Chemicals Divisional Standards Committee and it is in accordance with the procedures of the Bureau.

This Draft Tanzania Standard is the first edition.

In the preparation of this Draft Tanzania Standard assistance was drawn from IS 10523:1983, Specification for baby toilet soaps; published by the Indian Bureau of Standards.

In reporting the results of analysis of a test if the final value is to be rounded off, it shall be done in accordance with TZS 4 *Rounding off numerical values*

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## Introduction

Normally the general requirements for baby toilet soap are similar to those prescribed for toilet soap meant for adults. However, on account of natural difference between a baby's skin and an adult's skin due regard to toxicity and dermatological consideration is given during the manufacture of baby toilet soap. This is because the skin of babies, being much thinner and less cornified than that of an adult is highly susceptible to irritation and resistance to bacterial attack is not fully developed in the very young ones. If superficial layers of the epidermis are damaged by any means, for example, by the use of harsh toiletries, various skin disorders may develop. It is however not practicable to prescribe all such factors. It is, therefore, emphasized that manufacturers of baby toilet soap shall take the responsibility to establish and ensure dermatological safety of the soap.

In order to achieve dermatological safety of baby toilet soap the choice and selection of appropriate quality and quantity of different raw materials is very important.

In this specification, unlike normal toilet soap, the use of rosin has not been allowed because on account of its unsaturated nature, its soap may lead to irritation of the skin. On similar consideration a higher limit for total fatty matter (78%), low limits for free caustic alkali and free carbonated alkali and low limits for heavy metals, namely, nickel, copper and iron have been prescribed.

## Baby toilet soaps — Specification

### 1 Scope

This Draft Tanzania Standard specifies requirements and methods of sampling and test for baby toilet soap.

### 2 Normative references

The following referenced documents are indispensable for the application of this document. The latest edition of the referenced document (including any amendments) applies.

TZS 1396-4/ISO 456, *Surface active agents — Analysis of soaps — Determination of free caustic alkali*

TZS 1396-8.2/ISO 457, *Soaps — Determination of chloride content — Titrimetric method*

TZS 1396-2/ISO 672, *Soaps — Determination of moisture and volatile matter content — Oven method*

TZS 1396-3/ISO 673, *Soaps — Determination of content of ethanol-insoluble matter*

TZS 1396-12/ISO 684, *Analysis of soaps — Determination of total free alkali*

TZS 1396-6/ISO 685, *Analysis of soaps — Determination of total alkali content and total fatty matter content*

TZS 35, *Soaps – Sampling and test methods*

TZS 59/ISO 3696 *Water for analytical laboratory use – Specification and test method*

TZS 638/EAS 377(Part 1- 5) *Cosmetics and cosmetic products*

### 3 Terms and definitions

For the purpose of this standard the definitions given in TZS 35 shall apply.

### 4 Requirements

#### 4.1 General requirements

Baby toilet soap shall be a high grade, thoroughly saponified, milled soap or homogenized soap or both, white or coloured, mildly perfumed or unperfumed, and compressed in firm smooth cakes. It shall possess good cleaning and lathering properties. It shall be free from rancidity. It shall be free from toxic ingredients.

4.2 Baby soap shall have no objectionable odour, shall have good lathering and cleansing properties and shall be free from grit when tested in accordance with Annex A.

4.3 Baby soap may contain chelating agents, colouring matter, medicaments, mild perfume and antioxidants which shall be non-injurious in use with soap.

4.4 Baby soap shall not contain any rosin.

#### 4.5 Ingredients

Baby toilet soaps may contain the following: Colouring matter, perfume, preservatives and super fatting agents. All other ingredients except moisture shall be declared on the label. All these materials shall be non-injurious to skin and shall comply with TZS 638/EAS 377(Part 1- 5)

4.6 In addition to the above requirements, baby soap shall also comply with the requirements given in Table 1, when tested according to the corresponding test methods.

**Table 1 — Requirements for baby toilet soap**

S. No.	Characteristic	Requirements	Method of test
(1)	(2)	(3)	(4)
i)	Total fatty matter, percent by mass, min	78.0	TZS 35
ii)	Moisture and volatile matter (at 105 °C), percent by mass, max	15.0	TZS 35
iii)	Matter insoluble in alcohol, percent by mass, max	0.2	TZS 1396-3/ISO 673
iv)	Matter insoluble in water, percent by mass, max	0.5	TZS 35
v)	Free caustic alkali, as sodium hydroxide (NaOH), percent by mass, max	0.03	TZS 1396-4/ISO 456
vi)	Chlorides, as sodium chloride (NaCl), percent by mass, max	1.0	TZS 1396-8.2/ISO 457
vii)	Free carbonated alkali, percent by mass, max	0.5	TZS 1396-12/ISO 684
viii)	Free from grit	To pass the test	Annex A
ix)	Nickel content	Not detected	Annex B
x)	Iron content, mg/kg, Max	10	Annex C
xi)	Copper (as Cu) contents, mg/kg, max	3	Annex D
xii)	Free from rosin	To pass the test	Annexes E and F

**Note:**

**Calculation of results**

Baby toilet soap is liable to lose moisture on keeping. The results of analysis in respect of other characteristics shall be recalculated in relation to the minimum specified total fatty matter by means of the equations:

$$\text{Recalculated result} = \text{Actual result} \times \frac{\text{Minimum specified total fatty matter}}{\text{Actual total fatty matter}}$$

Where the minimum specified total fatty matter = 78.0 %.

## 5 Packing and labelling

### 5.1 Packing

The material shall be packed as agreed between the purchaser and the supplier.

### 5.2 Labelling

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

- a) name of the product;
- b) manufacturer's name and address and/or his recognized trade-mark, if any;
- c) net mass when packed;
- d) batch No. or Lot No. in code or otherwise;
- e) manufacture and expiry dates.
- f) The words 'baby soap'
- g) Country of origin

## 6 Sampling

**6.1** For this purpose, sampling and preparation of test samples shall be as prescribed in TZS 35

### 6.3 Criteria for conformity

For each of the characteristics which has been determined on the samples the mean ( $\bar{x}$ ) and the range ( $R$ ) of the test results shall be calculated as follows:

$$\text{Mean}(\bar{x}) = \frac{\text{The sum of the test results}}{\text{Number of test results}}$$

Range ( $R$ ) = The difference between the maximum and the minimum values of the test results

The lot/batch shall be deemed as conforming to the requirements given in Table 1 expressed as ( $\bar{x} - 0.6R$ ) is greater than or equal to minimum value given in Table 1, and ( $\bar{x} + 0.6R$ ) is less than or equal the maximum value given in Table 1.

## 7 Tests

**7.1** Tests shall be conducted in accordance with the standards listed in Clause 2 and Annexes A, B, C and D.

### 7.2 Quality of reagents

Unless specified otherwise, pure chemicals and distilled water (TZS 59) shall be employed in the tests.

NOTE 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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**Annex A**  
(normative)

**Test for “free from grit”**

**A.1 Outline of the method**

Soap is rubbed with water between the hands for a specified time and examined for its gritty or rough feel, if any.

**A.2 Procedure**

**A.2.1** Hold a bar of soap in running tap water and rub between both hands for about 3 min. No gritty or rough feel should be perceived while rubbing the soap surface. Examine the surface of the bar which shall not be rough and shall feel smooth when the fingers are moved across the surface.

**A.2.2** Set the bar on and let it dry for 24 h at room temperature and examine its surface. The soap shall be taken to have passed the test if there is no gritty or rough feel on the surface.



## Annex B (normative)

### Determination of nickel

#### B.0 Outline of the method

The method is based on the isolation of metal from the soap and reaction between nickel in the oxidized form with dimethyl glyoxime forming a red colour.

#### B.1 Reagents

**B.1.1 Sodium hydroxide** — AR grade.

**B.1.2 Concentrated hydrochloric acid** — Conforming to relevant standard.

**B.1.3 Dimethyl glyoxime solution** — 0.1 percent (v/v) in 95 percent ethyl alcohol.

**B.1.4 Saturated bromine water**

**B.1.5 Liquor ammonia** — Relative density 0.9.

#### B.2 Procedure

##### B.2.1 Isolation of metals from soap

Weigh 50 g of soap in a beaker and dissolve in hot water. Add to this soap solution 40 mL of concentrated hydrochloric acid, stir and keep on steam bath until fatty acid layer separates. Add 20 g of paraffin wax, stir at intervals and allow it to settle until phases are clear. Cool to room temperature.

**B.2.1.1** Remove wax cake with rod, rinse with water and add rinsing to aqueous phase. Evaporate, aqueous phase to about 60 mL by gentle boiling. Add 100 mL of water and filter through paper washed previously with hydrochloric acid. Evaporate filtrate and washings to about 60 mL. Cool and transfer to volumetric flask. Make up the volume to 100 mL. Take aliquot portions for metal estimation.

##### B.2.2 Determination of nickel content

Take 50 mL aliquot of the aqueous solution from the test solution in a 250 mL beaker. Evaporate the solution to about 15 mL by heating. Transfer the solution to a 50 mL glass stoppered volumetric flask using a small quantity of water for rinsing the solution from the beaker into the volumetric flask. Add to the flask 3 mL of saturated bromine water and allow to stand for one minute. Add liquor ammonia dropwise until excess bromine is destroyed as indicated by the disappearance of brown colour. Then add 5 mL of liquor ammonia in excess. If a precipitation occurs, filter the solution and wash the precipitate with distilled water; combine the filtrate and the washings and concentrate to a volume of a few millilitres and transfer to a 50 mL volumetric flask. Add 10 mL of dimethyl glyoxime solution, followed by 15-20 mL of 95 percent ethyl alcohol. Mix thoroughly and make up the volume and again mix thoroughly. Allow the solution to stand for 5 minutes to permit full development of colour. The sample shall be considered to have passed the test if no pink colour develops.

## Annex C (normative)

### Determination of iron

#### C.0 Outline of the method

The method is based on the isolation of metal from the soap by dissolving in hot water. The aqueous extract is treated with citric acid to sequester aluminium and then thioglycolic acid in ammoniacal solution is added and colour measured spectrophotometrically.

#### C.1 Apparatus

##### C.1.1 Spectrophotometer

#### C.2 Reagents

**C.2.1 Liquor Ammonia** — Relative density 0.9.

**C.2.2 Sulfuric acid** — 50 percent (v/v).

**C.2.3 Citric acid (aqueous solution)** — 50 percent (v/v).

**C.2.4 Thioglycolic acid (aqueous solution)**

**C.2.5 Standard iron solution** — Containing 10 µg of iron per mL prepared from ferric ammonium sulphate  $[\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 12\text{H}_2\text{O}]$  in acid solution.

**C.2.6 Methyl red indicator** — 0.1 percent aqueous solution.

**C.2.7** concentrated hydrochloric acid

**C.2.8** paraffin wax

#### C.3 Procedure

##### C.3.1 Isolation of metals from soap

Weigh 50 g of the soap sample in a beaker and dissolve it with hot water. To this soap solution add 40 mL of concentrated hydrochloric acid with constant stirring and keep the beaker on steam bath until fatty acid layer separates. Add 20 g of paraffin wax to this solution while hot. Stir the solution at intervals and allow it to settle until phases are clear. Cool the mass to room temperature. Remove the wax cake with rod, rinse with water and add the rinsing to aqueous phase. Evaporate the aqueous phase to above 60 mL by gentle boiling. Add 100 mL of water and filter through paper washed previously with hydrochloric acid. Evaporate the filtrate and washings to about 60 mL. Cool and transfer the solution to a volumetric flask and make up the volume to 100 mL. Take aliquot portion of the solution for metal estimation.

##### C.3.2 Determination of iron

Take 5 mL aliquot of the aqueous solution from the test solution in a 25 mL volumetric flask. To this add 4 mL citric acid solution and 0.02 mL methyl red indicator and liquor ammonia till the colour of the solution turns yellow. Then add 3 mL liquor ammonia in excess. Cool the solution and add 3 mL thioglycolic acid. Make up the volume to 25 mL and mix the solution thoroughly. Filter the solution through acid washed and dried filter paper. Measure absorbance of the clear solution at 540 nm in the spectrophotometer using water as reference. Prepare a calibration curve with standard iron solution and determine the iron content of the soap sample from it.

## Annex D (normative)

### Determination of copper

#### D.0 Outline of the method

The method is based on the isolation of metal from the soap and to make a copper complex using zinc dibenzyl dithiocarbamate in carbon tetrachloride solution and measure the colour of the solution spectrophotometrically.

#### D.1 Apparatus

##### D.1.1 Spectrophotometer

#### D.2 Reagents

**D.2.1 Zinc dibenzyl dithiocarbamate solution** — 0.05 percent in (v/v) in carbon tetrachloride.

**D.2.2 Standard copper solution** — Containing 1 µg of copper per mL (prepared from a stock solution of 100 times the concentration).  
concentrated hydrochloric acid  
paraffin wax  
sulphuric acid

#### D.3 Procedure

##### D.3.1 Isolation of metal from soap

Weigh 50 g of soap sample in a beaker and dissolve it with hot water. To this soap solution add 40 mL of concentrated hydrochloric acid with constant stirring and keep the beaker on steam bath until fatty acid layer separates. Add 20 g of paraffin wax to this solution while hot. Stir the solution at intervals and allow it to settle until phases are clear. Cool the mass to room temperature. Remove the wax cake with rod, rinse with water and add the rinsing to aqueous phase. Evaporate the aqueous phase to about 60 mL by gentle boiling. Add 100 mL of water and filter through filter paper washed previously with hydrochloric acid. Evaporate the filtrate and washings to about 60 mL. Cool and transfer the solution to a volumetric flask and made up the volume to 100 mL. Take aliquot portion of the solution for metal estimation.

##### D.3.2 Determination of copper

Take 20 mL aliquot of the aqueous solution and to it add 10 mL of zinc dibenzyl dithiocarbamate solution followed by 25 mL sulphuric acid in a separating funnel. Shake the solution for one minute and allow it to settle. Run the lower carbon tetrachloride layer in to a 25 mL volumetric flask. Wash the aqueous layer with carbon tetrachloride and transfer through glass wool to volumetric flask. Make up the volume and mix well. Measure absorption of the clear solution at 435 nm in the spectrophotometer. Prepare a calibration curve with standard copper solution and determine the copper content of the soap sample from the curve.

**NOTE** The standard solution shall also be extracted with carbon tetrachloride before estimation of colour.

## Annex E (normative)

### Identification of rosin content of fatty mater in soaps

#### E.1 Scope

This Annex specifies the Halphen-Grimaldi and Liebermann-Storch method for the identification of rosin in fatty matter.

#### E.2 Reagents

NOTE Use only analytical grade reagents and distilled water.

##### E.2.1 Acetic anhydride.

**E.2.2 Sulphuric acid, density 1.53 g/mL** (Cautiously add 97 mL of sulphuric acid “density 1.84 g/mL” to 100 mL of water).

**E.2.3 Solution A.** Dissolve 10 g of phenol in 27 mL of chloroform.

**E.2.4 Solution B.** Dissolve 10 mL of bromine in 40 mL of chloroform.

#### E.3 Procedure

##### E.3.1 Halphen-Grimaldi method

**E.3.1.1** Place one or two drops of the fatty matter, reserved from the determination of fatty matter (ISO 685), in a porcelain basin and add approximately 2 mL of the solution A to dissolve it. Wet the walls of the basin by tilting and turning it and let it stand for a few seconds so that the walls of the basin are covered with a very thin film of the solution.

**E.3.1.2** Hold the neck of the flask containing the solution B in such a position that the bromine vapour diffuses into the porcelain dish and comes into contact with the walls. In the presence of rosin a blueish-grey to purple colour develops immediately.

**E.3.1.3** Carry out a comparison test with a sample of fatty matter to which 2 % (*m/m*) of rosin has been added.

NOTE Should the colour reaction in A.3.1.2 be masked by other colours, conduct a second qualitative test, using the Liebermann-Storch method.

##### E.3.2 Liebermann-Storch method

**E.3.2.1** Place 1 g - 2 g of the fatty matter in a test tube, add 5 mL - 10 mL of the acetic anhydride, and heat the mixture in a boiling water bath for approximately 3 min. Cool to room temperature and pour 1 mL - 2 mL of the solution into a white porcelain basin.

**E.3.2.2** Allow one or two drops of the sulphuric acid to run down the side of the basin. If rosin is present, a violet colour immediately develops where the acid is in contact with the solution. This colour then turns brown on standing.

**E.3.2.3** Carry out a comparison test with a sample of fatty matter to which 2 % (*m/m*) of rosin has been added.

## Annex F (normative)

### Determination of rosin content of fatty mater in soaps

#### F.1 Scope

This Annex specifies a method for the determination of the rosin content of fatty matter in soaps.

NOTE 1 Before proceeding with the quantitative determination of rosin, first establish its presence by a qualitative test (see Annex E).

NOTE 2 The following method is not accurate for rosin concentrations below a mass fraction of 5 %.

#### F.2 Reagents

NOTE Use only analytical grade reagents and distilled water.

##### F.2.1 Diethyl ether (Free from peroxides)

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##### F.2.2 Sodium chloride

##### F.2.3 Anhydrous Sodium sulfate

##### F.2.4 Hydrochloric acid solution

Dilute one volume of concentrated hydrochloric acid (density 1.16 g/mL) with two volumes of distilled water.

##### F.2.5 Sodium hydroxide solution

10 % mass fraction aqueous solution.

##### F.2.6 Naphthalene-2-sulfonic acid solution

Dissolve 40 g of naphthalene-2-sulfonic acid in 1 L of absolute methanol.

##### F.2.7 Sodium chloride solution, saturated

Shake an excess of sodium chloride with water at ambient temperature until no more dissolves. Keep the solution over solid sodium chloride.

##### F.2.8 Sodium chloride solution, 10 %

Dissolve 10 g of sodium chloride in 100 mL of water.

##### F.2.9 Standard ethanolic potassium hydroxide solution, 0.2 N

###### F.2.9.1 Preparation

Purify 95 % (by volume fraction) ethanol by boiling 1.5 L of it over 20 g of potassium hydroxide for 1 h under reflux. Distil, discarding the first 50 mL of the distillate and stopping the distillation when approximately 1.3 L have been distilled. Dissolve 12 g of potassium hydroxide in 1 L of the purified ethanol, allow the solution to stand for approximately one week, and then decant the clear supernatant liquid from any potassium carbonate that has precipitated.

**F.2.9.2 Standardization**

Accurately weigh out approximately 1 g of potassium hydrogen phthalate (previously dried at  $110\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$  for 3 h) into a 250 mL Erlenmeyer flask. Add approximately 100 mL of carbon dioxide-free distilled water and three drops to five drops of the phenolphthalein indicator, and swirl gently until the solid has dissolved. Titrate the solution with the standard ethanolic potassium hydroxide solution, until a permanent pink colour is formed.

**F.2.9.3 Calculation**

Normality of the standard ethanolic potassium hydroxide solution ( $N$ ):

$$N = \frac{A \times 4.897}{B}$$

where

$A$  is the mass of the potassium hydrogen phthalate, in grams;

$B$  is the volume of the standard ethanolic potassium hydroxide solution used for the titration, in millilitres.

**F.2.10 Methyl orange indicator**

Dissolve 0.2 g of methyl orange in 100 mL of carbon dioxide-free water.

**F.2.11 Phenolphthalein indicator**

Dissolve 0.5 g of phenolphthalein in 100 mL of freshly boiled, 95 % (by volume fraction) ethanol.

**F.3 Procedure****F.3.1 Preparation of fatty matter****F.3.1.1 Carbolic soaps**

**F.3.1.1.1** Weigh out accurately into a 600 mL beaker such quantity of the test sample as contains approximately 40 g of fatty matter and dissolve it in approximately 400 mL of hot water, to which 40 mL of the sodium hydroxide solution has been added.

**F.3.1.1.2** Salt out the soap by adding sufficient sodium chloride to the hot solution to saturate it (at ambient temperature) with sodium chloride. Filter the soap quantitatively, and allow it to drain.

**F.3.1.1.3** Dissolve the drained soap in approximately 400 mL of hot water, and repeat the salting out and filtering procedure.

**F.3.1.1.4** Wash the soap thoroughly with the saturated sodium chloride solution, and proceed in accordance with F.3.1.2.2 to F.3.1.2.7.

**F.3.1.1.5** Reserve the combined filtrates and washings for the determination of carbolic acids.

**F.3.1.2 Other soaps**

**F.3.1.2.1** Weigh accurately into a 600 mL beaker such quantity of the test sample as contains approximately 40 g of fatty matter.

**F.3.1.2.2** Dissolve the soap in approximately 400 mL of hot distilled water, cool the solution, and slowly add an excess of the hydrochloric acid solution. Cover the beaker with a watch-glass and heat the contents until the fatty matter separates into a clear layer, but do not allow the temperature to exceed  $60\text{ }^{\circ}\text{C}$ .

**F.3.1.2.3** In the cases of liquid and gel soaps, acidify the soap as it is.

**F.3.1.2.4** Cool to approximately 25 °C and transfer the contents of the beaker to a separating funnel. Rinse the watch-glass and the beaker with portions of the diethyl ether totalling 100 mL and add them to the separating funnel. Shake the mixture in the separating funnel vigorously for 1 min, and let it stand until the two phases have separated.

**F.3.1.2.5** Draw off and discard the aqueous layer and wash the ether extract with 50 mL portions of the sodium chloride solution until the last washing is neutral to the methyl orange indicator.

**F.3.1.2.6** Filter the washed ether extract into a 250 mL beaker through a filter paper containing approximately 5 g of anhydrous sodium sulfate, and wash the separating funnel and the filter with small portions of the diethyl ether.

**F.3.1.2.7** Evaporate the ether extract plus washings on a warm water bath. When the residue is just dry, heat it rapidly to 130 °C and immediately place the beaker in a desiccator to cool.

**NOTE** F.3.1.2.2 to F.3.1.2.7 should be completed in the shortest possible period of time, so as to prevent oxidation of the fatty acids.

### **F.3.2 Determination of rosin content**

**F.3.2.1** Weigh accurately into a 150 mL flask with a ground-glass joint approximately 2 g of the prepared fatty matter (F.3.1.2.7) and reserve the rest in the case of soap powders for the fatty matter titre determination.

**F.3.2.2** Add exactly 25 mL of the naphthalene-2-sulfonic acid solution to the flask and two or three glass beads, and boil under reflux for 30 min.

**F.3.2.3** Carry out a blank test at the same time, using exactly 25 mL of the naphthalene-2-sulfonic acid solution only.

**F.3.2.4** Cool the contents of both flasks (sample and blank) to ambient temperature, add 0.5 mL of the phenolphthalein indicator to each flask, and immediately titrate to the end point with the standard ethanolic potassium hydroxide solution.

### **F.4 Calculation**

Rosin content of fatty matter, as a mass fraction percentage ( $R$ ):

$$R = \frac{(A - B) \times N \times 34.6}{C} - 1.0$$

where

$A$  is the volume of the standard ethanolic potassium hydroxide solution used for the titration of the sample solution, in millilitres;

$B$  is the volume of the standard ethanolic potassium hydroxide solution used for the titration of the blank solution, in millilitres;

$N$  is the normality of the standard ethanolic potassium hydroxide solution;

$C$  is the mass of the fatty matter taken, in grams.

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