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Carbolic toilet soap — Specification

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In order to match with technological development and to keep continuous progress in industries, standards are subject to periodic review. Users shall ascertain that they are in possession of the latest edition

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Requests for permission to reproduce this document should be addressed to:

Rwanda Standards Board

P.O Box 7099 Kigali-Rwanda

KK 15 Rd, 49

Tel. +250 788303492

Toll Free: 3250

E-mail: info@rsb.gov.rw

Website: www.rsb.gov.rw

ePortal: www.portal.rsb.gov.rw

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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 148 was prepared by Technical Committee RSB/TC 042, *Surface Active Agents*.

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

CD-ARS 1459:2019, Carbohc toilet soap — Specification

The assistance derived from the above source is hereby acknowledged with thanks.

This edition cancels and replaces the second edition (RS 148:2014), which has been technically revised.

Committee membership

The following organizations were represented on the Technical Committee on *Surface Active Agents* (RSB/TC 042) in the preparation of this standard.

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

ALYVO Rwanda Ltd

HORIZON/SOPYRWA

University of Kibungo (UNIK)

Standards for Sustainability (SfS)

Catchup Industries Ltd

SULFO Rwanda Industries Ltd

MACPELA Investment Ltd

Rwanda Standards Board (RSB) – Secretariat

Introduction

A paragraph.

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Carbolic toilet soap — Specification

1 Scope

This Draft Rwanda Standard specifies the requirements, sampling and test methods for hard toilet soap containing additions of phenolic substances such as cresylic acid.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

RS EAS 377 (all parts), *Cosmetics and Cosmetic Products*

RS ISO 457, *Soaps — Determination of chloride content — Titrimetric method*

RS ISO 673, *Soaps — Determination of content of ethanol-insoluble matter*

RS ISO 684, *Analysis of soaps — Determination of total free alkali*

RS ISO 456, *Surface Active Agents — Determination of free caustic alkali*

ISO 4314, *Surface active agents — Determination of free alkalinity or free acidity — Titrimetric method*

RS ISO 1067, *Analysis of soaps — Determination of unsaponifiable, unsaponified and unsaponified saponifiable matter*

3 Terms and definitions

For the purposes of this standard, the terms and definitions given in RS ISO 862 and the following apply.

3.1

carbolic soap

a mildly antiseptic soap containing carbolic acid and /or cresylic acid, both of which are phenols derived from either coal tar or petroleum sources

3.2

batch

soap manufactured from one boil or, in the case of continuous production processes, the material from a single day's production

3.3

boil

soap from pan or vat

3.4

defective

soap or its container that fails in one or more respects to comply with the appropriate requirements of the specification

3.5

lot

that quantity of soap powder or chips bearing the same batch identification and from one manufacturer, submitted at any one time for inspection and testing

3.5

unit

bar or tablet of soap with no covering

4 Requirements

4.1 General requirements

4.1.1 The carbolic toilet soap shall be in form of bar or tablet

4.1.2 The carbolic toilet soap shall be kneaded, uniformly mixed and compressed in firm and uniform texture

4.1.3 The carbolic toilet soap may be perfumed. The odour of the soap and the odour when dissolved in water shall not be objectionable.

4.1.4 The carbolic toilet soap shall have a good lathering and cleaning properties and shall not irritate the skin.

4.1.5 All the substances used in carbolic toilet soap shall comply with the requirements of all parts of RS EAS 377 (all parts).

4.1.5 When tested as described in Annex C, the carbolic toilet soap shall not crumble and its surfaces shall remain smooth

4.1.6 The carbolic toilet soap shall remain stable, without any change on its quality performance, when stored under normal storage conditions for a period of six months.

4.2 Specific requirements

The carbolic toilet soap shall comply with the requirements given in Table 1, when tested in accordance with the methods described therein.

Table 1 — Specific requirements of carbolic toilet soap

S/N	Characteristics	Requirement	Test method
i.	Total fatty matter, min.	60.0	ISO 685
ii.	Rosin acids, % of total fatty matter max.	15.0	Annexes A&B
iv.	Matter insoluble in ethanol, max.	2.0	RS ISO 673
v.	Carbolic acid content, % by mass, min.	1.0 – 3.0	Annex D
vi.	Free caustic alkali as NaOH, % by mass, max.	0.1	RS ISO 684, RS ISO 456
vii.	Total free alkali as NaOH, % by mass, max.	0,2	RS ISO 684 RS ISO 4314
viii.	Free acidity content (as oleic acid), % by mass, max.	0.5	
ix.	Unsaponified plus unsaponifiable matter content, % by mass, max.	1.0	RS ISO 1067
x.	Chlorides as NaCl, max.	0.8 0.5	RS ISO 457
xi.	Matter insoluble in water, % by mass max.	0.5	Annex E

4.3 Basis of Test requirements

4.3.1 The soap covered by this standard is liable to lose moisture on keeping. The actual results obtained by the stated methods of analysis shall therefore be recalculated in relation to the specified minimum total fatty matter by means of the equation:

$$\text{Recalculated Results} = \text{Actual Result} \times \text{Min.} \frac{\text{TFM specified}}{\text{Actual TFM found}}$$

4.3.2 In each case, the requirements indicated in Table 1 shall be satisfied if the recalculated result obtained s above is within the specified range.

5 Packaging and labelling

5.1 Packaging

Each cake or number of cakes shall be packed in a suitable protective container. A number of cakes shall then be packed in suitable boxes or outer packaging to form a tight package,

5.2 Labelling

On the package and on each box or carton shall be labelled the following particulars:

- a) the name of the product 'carbolic toilet soap' and any other qualifying words where applicable;
- b) the name and address of the manufacturer or trade mark;
- c) nominal mass of each cake at the time of packing;
- d) on the box or the outer package, the number of cakes contained therein and the date of manufacture; and
- e) commercial brand names may be added as well as details of composition provided that this further information shall not be misleading
- f) information shall not be misleading

6 Sampling

Sampling shall be done in accordance with [ISO 15528].

Commented [F1]: to appear in normative reference

Annex A (normative)

Identification of rosin content of fatty matter in soaps

A.1 Scope

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

A.2 Reagents

NOTE Use only analytical reagents and distilled water

A.2.1 Acetic anhydride

A.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.

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

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

A.3 Procedure

A.3.1 Halphen-Grimaldi method

A.3.1.1 Place one or two drops of the fatty matter, reserved from the determination of fatty matter (ISO 865), 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.

A.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.

A.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.

A.3.2 Liebermann-Storch method

A.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.

A.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.

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

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Annex B (normative)

Determination of rosin content of fatty matter in soaps

B.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 of rosin, first establish its presence by a qualitative test (see Annex A).

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

B.2 Reagents

NOTE Use only analytical reagents and distilled water

B.2.1 Diethyl ether

Free from peroxides

B.2.2 Sodium chloride

B.2.3 Sodium sulfate, anhydrous

B.2.4 Hydrochloric acid solution

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

B.2.5 Sodium hydroxide solution

10% mass fraction aqueous solution

B.2.6 Naphthalene-2-sulfonic acid solution

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

B.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.

B.2.8 Sodium chloride solution, 10 %

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

B.2.9 Standard ethanolic potassium hydroxide solution, 0.2 N

B.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.

B.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.

B.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.

B.2.10 Methyl orange indicator

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

B.2.11 Phenolphthalein indicator

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

B.3 Procedure

B.3.1 Preparation of fatty matter

B.3.1.1 Carbolic soaps

B.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.

B.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.

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

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

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

B.3.1.2 Other soaps

B.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.

B.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 °C.

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

B.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.

B.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.

B.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.

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

B.3.2 Determination of rosin content

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

B.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.

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

B.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.

B.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.

Dissolve 10 g of sodium chloride in 100 mL of water

Annex C
(normative)

Texture and stability test

A whole cake of soap is immersed in distilled water at a temperature of 25°C — 30 °C contained in a vessel of one litre capacity. After one hour the cake is removed from water, allowed to dry at room temperature for 24h and then examined for crumbling and smoothness on its surfaces. It shall not crumble crack or break.

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Annex D (normative)

Determination of carbolic acid content

D.1 Scope

This standard specifies a method for the determination of the carbolic acid content of carbolic soap.

D.2 Reagents

NOTE Use only analytical grade reagents and distilled water

D.2.1 Diethyl ether, free from peroxide

D.2.2 Sodium sulfate, anhydrous

D.2.3 Hydrochloric acid solution

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

D.2.4 Methyl orange indicator

Dissolve 0.2 g of methyl orange in 100 mL of water.

D.3 Procedure

D.3.1 Transfer the combined filtrates and washing retained from the determination of rosin content (see Annex B) quantitatively to a separating funnel, add a few drops of the methyl orange indicator, and then slowly add the hydrochloric acid solution until there is an excess of about 5 mL.

D.3.2 Extract the acidified solution with 100 mL and then three times with 30 mL portions of the diethyl ether. Combine the ether extracts in a second separating funnel and wash four times with 20 mL portions of water.

D.3.3 Combine the washings and extract them with approximately 30 mL of the diethyl ether. Wash this extract with water until the washings are neutral to the methyl orange indicator and add this washed extract to the ether extract in the second separating funnel.

D.3.4 Filter the combined extracts into a 500 mL beaker through a filter paper containing approximately 5 g of the sodium sulfate. Wash the separating funnel and the filter thoroughly with small portions of the diethyl ether.

D.3.5 Evaporate off approximately two-thirds of the ether on a hot water bath, transfer the concentrated ethereal solution quantitatively to a tared crystallizing dish, using small portions of the diethyl ether to rinse the beaker, and evaporate to dryness on a hot water bath.

D.3.6 When just dry, leave the dish for a further 3 min on the water bath, and remove the last traces of ether by means of a current of dry air. Let the dish and its contents cool in a desiccator and weigh.

D.4 Calculation

Carbolic acid content, as a mass fraction percentage (M):

$$M = \frac{A \times M \times 100}{B \times N}$$

where

A is the mass of the residue, in grams;

B is the mass of the test sample used, in grams (see Annex B);

M is the required minimum fatty matter content of the sample, as a percentage;

N is the actual fatty matter content of the sample, as a percentage.

Annex E (normative)

Determination of matter insoluble in water

E.1 Scope

This annex specifies a method for the determination of the water insoluble matter content of soap products.

E.2 Apparatus

E.2.1 **Oven.** A drying oven capable of being maintained at 105 ± 5 °C

E.3 Procedure

E.3.1 Soap powders, gel soaps

Thoroughly wash the residue on the glass fibre filter, retained from the determination of free alkali content and free acid content, with hot distilled water. Dry the residue and the filter in the oven maintained at 105 ± 5 °C, cool in a desiccator, and weigh.

E.3.2 Scouring compounds

Dry the residue and the filter, retained from the determination of alkaline salts, in the oven maintained at 105 ± 5 °C, cool in a desiccator, and weigh. Repeat the drying and weighing procedure until no further loss in mass occurs.

E.4 Calculation

E.4.1 Soap powders, gel soaps

$$\text{Water insoluble matter content, \% (m/m)} = \frac{A \times M \times 100}{B \times N}$$

where

A = mass of the residue, g

B = mass of the test sample used, g

M = required minimum fatty matter content of the sample, %

N = actual fatty matter content of the sample, %.

C.4.2 Scouring compounds

$$\text{Water insoluble matter content, \% (m/m)} = \frac{A \times 100}{B}$$

where

A = mass of the residue, g

B = mass of the test sample used, g.

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