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Skin applied mosquito repellents —

Specification —

Part 2:

Sprays and roll-ons

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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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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 392-2 was prepared by Technical Committee RSB/TC 015, *Pharmaceutical Products*.

DRS 392 consists of the following parts, under the general title *Skin applied mosquito repellents — Specification*:

— *Part 1: Lotions, creams, gels and ointments*

— *Part 2: Sprays and roll-ons*

— *Part 3: Wipes*

— *Part 4: Soaps*

— *Part 5: Bracelets, wristbands and patches*

Committee membership

The following organizations were represented on the Technical Committee on Pharmaceutical Products (RSB/TC 015) in the preparation of this standard.

National Industrial Research and Development Agency (NIRDA)

National Pharmacy Council (NPC)

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

Pharmacie NOVA

Rwanda Development Board (RDB)

AGROPY LTD

IKIREZI NATURAL PRODUCTS

HORIZON/SOPYRWA

Rwanda Social Security Board (RSSB)

Pharmavie

University of Rwanda/College of Medicine and Health Sciences (UR/CMHS)

Rwanda Biomedical Center/ Malaria and Other Parasitic Diseases Division (RBC/MOPDD)

Society for Family Health (SFH) – Rwanda

Rwanda Biomedical Center/Medical Procurement and Production Division (RBC/MPPD)

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Introduction

Mosquito repellents are an effective complement to bed nets in the prevention of mosquito borne diseases, especially malaria.

Insecticides are used either for killing or controlling harmful insects. The insecticides which are applied for repelling insects are termed as “Repellents". Mosquito is one of the most harmful insects for mankind. To destroy them, many preparations are available on the market in various recipes like pest killer spray, soap, oil, powder, repellent etc. Out of these, mosquito repellent is the most popular as it has germicidal and disinfectant properties and is able to repel mosquitoes and is convenient to use.

The mosquito repellent is used for warding off mosquitoes which is the most harmful insect. Nowadays, mosquito repellents are used for controlling mosquitoes and are becoming most preferably than other mosquito destroyers gradually. With the rise in the standard of living, increasing urbanization and population, the demand of mosquito repellent mat is constantly increasing particularly in tropical places. It is a convenient method for protection against mosquitoes, so it has a tremendous market potential. Thus, there is a very good scope for development of such units in the country.

Skin applied mosquito repellents possess the potential to target residual transmission. Entomological evidence has shown that repellents provide personal protection against malaria. Product examples that are commonly used include lotions, gels, creams and ointments, among others. However, many more types of skin applied repellent products are now developed in various forms such as sprays and roll-ons, wipes, soaps and bracelets, wristbands and patches.

Skin applied mosquito repellents — Specification — Part 2: Sprays and roll-ons

1 Scope

This Draft Rwanda Standard prescribes the requirements, sampling and test methods for skin applied mosquito repellents formulated and/or prepared as sprays and roll-ons and meant to be applied directly to skin or to clothing and/or footwear.

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 846, *Glossary of terms relating to the cosmetic industry*

RS EAS 377 (all parts), *Cosmetics and cosmetic products*

RS 191, *Refined pyrethrum concentrate — Specification*

RS EAS 847-17, *Cosmetics — Analytical methods — Part 17: Determination of pH*

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

CIPAC 760, *Determination of picaridin*

CIPAC 667, *Determination of ethyl butylacetamidopropionate*

AOAC 986.03, *Determination of permethrin in pesticide formulations*

AOAC 997.07, *N-octyl Bicycloheptene dicarboximide (MGK 264), Pyrethrins and Piperonyl Butoxide (PB) in Technical materials, concentrates and Finished Products.*

DRS 394-1, *Mosquito repellents — Performance Test Guidelines — Part 1: Skin applied repellents*

RS 91, *Labelling and marking of pharmaceutical products — Specification*

RS ISO 24153, *Random sampling and randomization procedures*

3 Terms and definitions

For the purposes of this standard, the terms and definitions given in RS EAS 846 and the following apply.

3.1

mosquito

any of numerous arthropod animals of the class mosquito, having an adult stage characterized by three pairs of legs and a body segmented into head, thorax, and abdomen and usually having one or two pairs of wings. Mosquito include the flies, crickets, insects, beetles, butterflies and bees.

3.2

mosquito repellent

substance applied to skin, clothing, or other surfaces which discourages mosquito (and arthropods in general) from landing or climbing on that surface.

3.3

natural repellents/biopesticides

repellents that contain natural, plant-based compounds or other products approved by a competent authority

3.4

synthetic repellents

conventional repellents containing synthetic chemical compounds

3.5

roll-on

cosmetic preparation with the effect of deodorizing and providing antiperspirant properties to the body of the user. It is packed in a container fitted with a roll-ball.

3.6

roll-ball

spherically shaped object, with the capacity to roll in all directions. It is put at the opening of a roll-on container and serves the role of closing the container as well as dispensing the contents, when rolled on the skin

3.7

DEET

N,N-Diethyl-meta-toluamide or diethyltoluamide

3.8

IR3535

ethyl butylacetylaminopropionate

3.9

Picaridin

1-(1-methylpropoxycarbonyl)-2-(2-hydroxyethyl) piperidine or 2-(2-hydroxyethyl)-1-piperidinecarboxylic acid 1-methylpropyl ester

3.10

MGK 264

4-(2-Ethyl-hexyl)-4-aza-tricyclo[5.2.1.$0^{2,6}$]dec-8-ene-3,5-dione

3.7

PBO

Piperonyl butoxide

4 Requirements

4.1 General

4.1.1 The repellent shall contain required amounts of active ingredients and when applied to the skin shall have the effect of repelling insects. The repellent shall be in the form of:

a) *Sprays*: This shall consist of a liquid formulation in a pressurised, non-refillable aerosol dispenser, containing active ingredient(s), and/or synergist(s) and other formulants including propellants and solvents,
b) *Roll-ons*: The product may also be in form of cosmetic preparation meant to be applied as a roll-on, and which contains active ingredient(s), synergist(s) and/or other formulants

5.1.2 The formulation shall be of uniform colour and shall be free from visible impurities.

5.1.3 The aerosols shall not contain solvents and propellants listed in annex A.

5.1.4 All ingredients shall meet the requirements of RS EAS 377.

5.1.5 When applied to the skin, clothes or shoes, the product shall have the benefit of repelling mosquitoes and shall not have a harmful effect to the skin.

4.2 Active ingredients and synergists

4.2.1 Natural repellents

4.2.1.1 Active ingredients used in natural repellents shall be natural plant based active ingredients such as essential oils or any other plant extract approved as mosquito repellents.

4.2.1.2 The manufacturer shall provide adequate data on the repellence of such ingredients.

4.2.1.3 The manufacturer shall have adequate data justifying the proportion of ingredient(s) for which claims are made, used in the product.

4.2.1.4 The essential oils and other plant extracts used in natural repellents shall be, but not limited to:

a) Cedarwood oil;

b) Tea tree oil;

c) Geranium oil;

d) Rosemary oil;

e) Lemongrass oil;

f) Citronella oil;

g) Eucalyptus oil; and

h) Cinnamon oil

i) Neem oil

4.1.2.6 The proportion of single or blended active ingredient (s) in natural repellent shall be set by the manufacturer in accordance with specific standard and records shall be availed.

4.1.2.7 Pyrethrum extracts such as pyrethrins shall be considered in natural repellents. The limits of pyrethrins in natural repellents shall not be less than 0.5 % and the extract used shall meet the requirements of RS 191.

4.2.2 Synthetic repellents

4.2.2.1 Synthetic repellents shall contain synthetic chemical compounds which are able to discourage mosquitoes and send them flying or crawling away.

4.2.2.2 If a synthetic active ingredient is blended with other active ingredient (s), either natural or synthetic, the proportion shall be set by the manufacturer based on scientific research and records shall be availed.

4.2.2.3 Active ingredients and their content in synthetic repellents shall meet the requirements prescribed in table 1.

Table 1 – Active ingredients for synthetic repellents

S/N	Active ingredient	Limits (%, w/w)	Identification method
1	DEET	4 –50	Annex B
2	Picaridin	5 – 20	CIPAC 740
3	IR3535	7.5 – 20	CIPAC 667
4	Permethrin[a]	0.5	AOAC 986.03
a. Permerthrin shall be applied to clothing, footwear bed nets, and camping gear, but not directly to skin or to inner clothing (socks or underwear).			

4.2.2.4 Synthetic repellents and their active ingredients shall be approved and registered by competent authority before being released to the market.

4.2.3 Synergist content

4.2.3.1 The synergist content shall be declared and, when determined, the average content measured shall not differ from that declared.

4.2.3.2 The synergist shall be PBO, sesame seed oil (sesamin, sesamolin) and/or MGK 264. The synergist shall be tested in accordance with AOAC 997.07. The ratio of the active ingredient to the synergist shall be indicated and records availed.

4.2.3.3 The ratio of the active ingredient to the synergist shall be at the ratio range of 1 to 10 parts exponent's active ingredient to 1 part of an insecticide's active ingredient.

4.3 Specific requirements

The product shall comply with the quality requirements given in table 2.

Table 2 — Specific requirements

S/N	Parameters	Requirements	Test methods
i.	pH	3 – 7	RS EAS 847-17
ii.	Non-volatile matter, % m/m, min.	10	Annex D
iii.	Lead (Pb) , , mg/kg, max	20	RS EAS 847-16
iv.	Arsenic (As), mg/kg, max	2	
v.	Mercure (Hg), mg/kg, max	2	

4.4 Requirements for aerosol containers

The product packaged in aerosol containers shall meet the requirements given in table 3.

Table 3 — Specific requirements for aerosol containers

S/N	Parameters	Requirements	Test method
i.	CFCs	Absent	Annex E
ii.	Delivery rate g/s, min.	0.01	Annex F
iii.	Net weight delivery, % m/m, min	95	Annex G
iv.	General leakage	To pass test	Annex H

4.5 Biological efficacy

When tested in accordance with DRS 394-1, the product shall repel 100 % of the mosquitoes from landing or climbing on that surface, within protection time indicated by the manufacturer.

5 Packaging and labelling

The product shall be packaged as roll-ons or aerosols products and in any other suitable containers that shall protect the contents and shall not react with the product or cause any contamination during storage, handling or use.

5.1 Roll ball construction

5.1.1 If the container is fitted with a roll-on:

5.1.1.1 The roll ball shall be made of plastic material.

5.1.1.2 The roll ball shall be fitting on the container such that on holding the container inside down the contents shall not pour out.

5.1.1.3 The roll ball shall be free rolling, leaving a thin layer of the contents on the skin during dispensation.

5.2 Aerosol containers

Filled aerosol containers shall be appropriately classified in terms of flame propagation characteristics of their contents when tested in accordance with annex I.

Highly flammable — if the average length of the flame is greater than 0.45 m or if the flame burns back to the actuator, or continues to burn when the test flame is extinguished.

Flammable — if the average length of the flame is between 0.20 m and 0.45 m

Non-flammable — if the product does not burn in the manner described above (a) and (b)

5.3 Labelling

The containers shall be securely closed and in addition to the labelling requirements of RS 91, the following information shall be indelibly and legibly marked on the container:

a) name of the product;

b) name and full address of the manufacture;

c) batch number;

d) date of manufacture and expiry;

f) quantity in the container;

g) active ingredient content, percent (%,m/m);

h) list of other ingredients

i) net mass of content;

j) directions for use;

k) safety precaution;

l) special persons whose exposure is prohibited (out of reach of children and pregnant women); and

m) storage conditions.

6 Sampling

Random samples of the product shall be drawn for test in accordance with RS ISO 24153 from the market, factory or anywhere else.

Annex A
(normative)

Solvents not permitted for use in aerosols

1) Benzene
2) 2-butoxyethanol (ethylene glycol monobutyl ether)
3) 2-butoxyethylacetate (ethylene glycol monobutyl ether acetate)
4) carbon tetrachloride
5) chlorobenzene
6) chloroform
7) 1,2-dichloroethane (ethylene dichloride)
8) 2-ethoxyethanol (ethylene glycol monoethyl ether)
9) 2-ethoxyethylacetate (ethylene glycol monoethyl ether acetate)
10) n-hexane
11) 2-hexanone (methyl n-butyl ketone)
12) 2-methoxyethanol (ethylene glycol monomethyl ether)
13) 2-methoxyethylacetate (ethylene glycol monomethyl ether acetate)
14) tetrachloroethylene
15) trichloroethylene
16) Propellants

NOTE The Montreal Protocol and EU1 directive on the withdrawal of chlorofluorocarbons (CFCs) from aerosols were noted. Hydrocarbon propellants are recommended for insecticide aerosols, provided international safety standards are met by the aerosol producer. Industry should be encouraged to develop alternative and safer propellants and delivery systems.

Annex B
(normative)

Determination of DEET content

B.1 General

The sample is dissolved in carbon disulfide and the difference in absorbance at 14.18 µm and at 14.48 µm is determined. The quantity of meta-isomer is obtained from this value by means of a calibration curve prepared by the use of a reference standard.

B.2 Apparatus

B.2.1 Double-beam infrared spectrophotometer. Perkin-Elmer model 21 or equivalent.

B.2.2 Two equivalent infrared absorption cells, with sodium chloride windows and a path length of approximately 0.4 mm.

B.3 Preparation of calibration curve

B.3.1 Weigh (to the nearest 0.1 mg) into four volumetric flasks sufficient amounts of the reference DEET standard of known purity to give concentrations of approximately 20, 40, 60 and 80 g/L when dissolved in carbon disulfide.

B.3.2 Fill the reference cell with carbon disulfide and the sample cell with each of the standard solutions in turn, and record the spectra. The spectrum may be scanned rapidly, except for the region 12 – 15 µm, where a normal speed should be used. Carry out a blank measurement with carbon disulfide to correct for any inequality in the paired cells and to determine whether a cell correction is required.

B.3.3 Measure the absorbance at 14.18 µm and at 14.48 µm and calculate the difference between these values, ΔA, for each of the solutions. Plot the values of ΔA against the concentration (g/l) of the meta-isomer.

B.3.4 If a cell correction is required, the value of ΔA is determined from the formula:

$$\Delta A = [A_{14.18} - A_{14.48}]_{ref.} - [A_{14.48}]_{blank}$$

Where ref. = determination with reference standard
blank = determination on CS_2 blank

B.4 Procedure

Weigh (to the nearest 0.1 mg) about 0.5 g of the sample, transfer quantitatively to a 10 mL volumetric flask, and make up to the mark with carbon disulfide. Measure the infrared absorption at 14.18 µm and 14.48 µm using the same conditions as described in section A.3. Determine the concentration of meta-isomer by comparing this value with the calibration curve. A standard sample should be run each day to check the calibration of the instrument.

B.5 Calculation

DEET content (g/kg) = $\frac{C1\ x\ P}{C2}$

Where,

C_1 = concentration (g/L) of standard DEET found from calibration curve

C_2 = concentration (g/L) of sample taken

P = purity (g/kg) of the reference standard.

Annex C
(normative)

Stability of smell

C.1 Apparatus

C.1.1 Porcelain cup

C.1.2 Pincers

C.1.3 Ten pieces of bleached gauze of dimension 5 cm x 10 cm

C.1.4 Thermometer

C.1.5 Hygrometer

C.2 Procedure

Put some pieces of bleached gauze which have been pre-washed in hot water without soap and dried into a porcelain cup and pour 1.5 ml of the sample into this cup. After the gauze gets soaked, take it out with the help of pincers. Without squeezing it, dry it in a premise having temperature 37 °C ± 2 °C and humidity of 65 % ± 5 % for 12 h.

C.3 Result

The product shall be taken to have passed the test if, after 12 h, the smell of the sample can clearly be picked up.

Annex D
(normative)

Non-volatile matter

D.1 Apparatus

D.1.1 Moisture dish

D.1.2 Oven

D.1.3 Analytical balance

D.1.4 Desiccator

D.2 Procedure

Weigh accurately 1g ± 0.2 g of the sample in the dish and place it in an oven at 105 °C ± 2°C for 1 h. Cool to room temperature in a desiccator and weigh the dish. Repeat the process to bring it to constant mass.

D.3 Calculation

Non-volatile matter per cent by mass = $= \frac{M_2 - M_1}{M} \; x \; 100$

Where,

M = mass, in grams of the material taken;

M_1 = mass in grams of the dry and empty dish, and

M_2= mass in grams of the dish and dried material.

Annex E
(normative)

Determination of propellant composition

E.1 Procedure

E.1.1 The analysis of the propellant mixture in most aerosols is carried out conveniently by gas chromatography. For Sampling, a hypodermic needle is fitted to the valve of the aerosol can and approximately 0.5 g of the propellant is injected into the heavy duty centrifuge tube closed with serum cap, containing about 8 ml of benzene. After mixing, 5µl samples are taken out from this tube with a microlitre syringe and injected into the gas chromatograph.

E.1.2 Two 4572 mm × 6.35 mm OD columns operated at 40 °C are recommended for the analysis containing 20 percent weight hexadecane and diethylhexyl sebacate respectively on silanized chromosorb W60/S0 mesh.

E.1.3 The first column should be used mainly for initial screening and the second column for the confirmation and determination of the identified propellants.

E.1.4 Table C1 lists the relative retention data of the most widely used propellant together with some other fluorinate hydrocarbons and benzene used as the solvent IN the two columns.

Table C1 – Relative retention data of propellants

Chemical name	Stationary phase diethylhexyl sebacate	Stationary phase hexadecane
Octafluorocylobutane	0.214	0.122
1-chloro-1,2,2 trifluoroethylene	0.268	0.196
Propane	0.275	0.22
1,2-difluoroethane	0.289	0.141
Dichlorodifluoromethane	0.296	0.220
1,2-dichloro1,1.2,2-tetrafluoromethane	0.345	0.290
Isobutane	0.366	0.378
Monochlorodiflouromethane	0.368	0.152
1-Chloro-1,1-difluoroethane	0.402	0.236
n- butane	0.449	0.527
Vinylchloride	0.529	0.353
Trichlorofluoroethane	1.000	1.000
1,1,2-trichloro-1,2,2-tetrafluoroethane	1.254	1.342
Dichloromonofluoroethane	1.354	0.515
1,2-dibromo-1,1,2,2 tetrafluoroethane	1.634	1.363
Methylene Chloride	2.565	1.070
Benzene	6.786	5.661

E.2 Results

The sample shall be considered as having failed the test if it contains any of the above CFCs

Annex F
(normative)

Determination of delivery rate of the dispenser

F.1 Material and apparatus

The following material and apparatus shall be used in this test

F.1.1 Any suitable timing device

F.1.2 Balance

Having accuracy to 0.01 g and with a capacity greater than 500 g.

F.1.3 Pair of gloves

Made of cloth or fabric or towel for handling dispensers during test.

F.1.3 Pair of tongs

For removing dispensers from water bath.

F.1.3 Water bath

Set at 26 °C ± 0.3 °C, thermostatically controlled.

F.2 Procedure

F.2.1 Hold a dispenser upright, spray for two seconds to fill the eduction tube. Then weigh the dispenser

F.2.2 Submerge the dispenser into the water bath for 15 minutes using tongs, remove the dispenser from the bath and immediately dry the container with a towel Spray the dispenser in one continuous burst for 10 seconds. Re-weigh the dispenser.

F.2.3 Repeat the procedure and take an average of three tests. The difference between the maximum and minimum delivery rates shall not exceed 0.2 g per second.

F.3 Calculation

Calculate the delivery rate according to the following formula;

Delivery rate (in g per second) = $\frac{M1-M3}{N}$

where;

M_1 = initial weight of the dispenser in grams

M_2 = final weight of the dispenser in grams

N = time in seconds

Annex G
(normative)

Net weight delivery

G.1 Procedure

G.1.1 For the determination of the net weight delivery, a random sample of at least three packages is selected. After the removal of any dust cover or caps not required for dispensing the product, the gross weight of each package is determined and after shaking for 15 sec, the content of the lightest container is drained by holding the valve wide open. Now the exhausted container is weighed .The result is called wet –tare weight and is equal to the weight of the container plus any product remaining after draining.

G.1.2 Consequently, the regeneration allowance is determined and subtracted from the wet-tare weight to obtain the corrected wet-tare weight. The regeneration allowance is defined as the difference between the weight of the product which would be delivered through normal usage and the weight of the product delivered by the present accelerated procedure.It is calculated by multiplying the label weight of the container by 0.02 g and rounding the result to the next lowest gram.

G.1.3 By subtracting the corrected tare weight from the gross weight, the adjusted net weight of the package is obtained. If this is greater than 95 % of the label weight the lot is assumed to be satisfactory. However, if it is less than 95 % of the label weight, the lot is rejected.

Annex H
(normative)

Testing of filled aerosol containers

H.1 Procedure

H.1.1 All filled aerosol containers shall be tested by immersion in a water bath set at 55 °C.

H.1.2 The container shall be such that the pressure generated within the immersed container reaches not less than 90 percent of the pressure generated within the containers at equilibrium at 55 °C.

H.2 Interpretation of results

Any filled aerosol container that shall leak, get distorted or burst as a result of this test shall be considered to have failed the test and shall be discarded.

Annex I
(normative)

Flame propagation

I.1 Principle

The filled aerosol container is sprayed as a test flame under controlled conditions and length of the burning spry cone is measured.

I.2 Apparatus

I.2.1 In its simplest form, the apparatus consists of a base marked at 0.15 m intervals, an adjustable stand to carry the aerosol container which may be raised or lowered to accommodate differences in container height, a means of measuring the burning spray cone (usually a one metre fuel placed horizontally at the same level as the top third of the flame, the hottest part) a means of igniting the spray cone in the form of a test flame 0.05 m ± 0.005 m in height (usually a candle flame is used).

I.2.2 Water bath maintained at 20 °C .This equipment shall be used to bring the aerosol container and its contents to equilibrium at 20 °C (Heat the cans to 20 °C in the water bath).

I.3 Procedure

I.3.1 Place the aerosol container on the stand. Depress the actuator and adjust the height of the stand so that the spray cone will pass through the upper third test flame (hottest part).

I.3.2 Bring the aerosol container and its content to the equilibrium temperature of 20 °C. Place the container on the stand so that the point where the spray emerges is 0.15 m from the test flame. Then light the test flame and depress the actuator for 15 seconds to 20 seconds. Record the total length of the burning spray cone and specify whether or not it burns back to the actuator.

I.3.3 Extinguish the test flame and record whether the spray cone continues to burn while the actuator is depressed.

I.3.4 Repeat each test twice and record the flame length as the average of the three tests.

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