



DEAS 1119-3: 2023

ICS 71.100.70 , 65.100

DRAFT EAST AFRICAN STANDARD

Skin applied mosquito repellents — Specification — Part 3: Wipes

EAST AFRICAN COMMUNITY

Copyright notice

This EAC document is copyright-protected by EAC. While the reproduction of this document by participants in the EAC standards development process is permitted without prior permission from EAC, neither this document nor any extract from it may be reproduced, stored or transmitted in any form for any other purpose without prior written permission from EAC.

Requests for permission to reproduce this document for the purpose of selling it should be addressed as shown below or to EAC's member body in the country of the requester:

© East African Community 2023 — All rights reserved
East African Community
P.O. Box 1096,
Arusha
Tanzania
Tel: + 255 27 2162100
Fax: + 255 27 2162190
E-mail: eac@eachq.org
Web: www.eac-quality.net

Reproduction for sales purposes may be subject to royalty payments or a licensing agreement. Violators may be prosecuted.

Contents

Page

Foreword	v
1 Scope	7
2 Normative references	7
3 Terms and definitions	8
4 Symbols and/or abbreviated terms	9
5 Active ingredients	9
5.1 Natural repellents	9
5.2 Synthetic repellents	10
6 Requirements.....	10
6.1 General requirements	10
6.2 Specific requirements	10
6.3 Microbiological requirements	11
6.4 Biological efficacy	12
7 Packaging.....	12
8 Labelling.....	1
8.1 Primary packaging	1
8.2 Secondary packaging	1
9 Sampling	1
Annex A (normative) Determination of DEET content.....	2
A.1 General	2
A.2 Apparatus	2
A.3 Preparation of calibration curve	2
A.4 Procedure.....	2
A.5 Calculation	3
Annex B (normative) Determination of ethyl butylacetamidopropionate (IR3535).....	4
B.1 Outline of method.....	4
B.2 Reagents	4
B.3 Apparatus.....	4
B.4 Procedure.....	4
B.4.1 Chromatographic conditions (typical)	4
B.5 Preparation of sample.....	5
B.6 System equilibration	5
B.7 Determination.	5
B.8 Calculation	6
Annex C (normative) Determination of icaridin.....	7
C.1 outline of method	7
C.2 Reagents	7
C.3 Apparatus.....	7
C.4 procedure	7
C.4.1 Chromatographic conditions (typical)	7
C.5 Preparation of sample.....	8
C.6 Equilibration of the system	8
C.7 Determination	8
C.8 Calculation	8
Annex D (normative) Determination of total pyrethrins	10
D.1 General	10

D.2	Reagents	10
D.3	Apparatus	10
D.4	Operating conditions	10
D.5	Preparation of the standard	10
D.6	Sample preparation	11
D.7	Procedure	11
D.8	Calculation of the % total pyrethrins (active ingredient)	11
Annex E (normative) Determination of absorbency rate		12
E.1	Apparatus	12
E.2	Preparation of test specimens	12
E.3	Procedure	12
D.4	Calculation	12
Annex F (normative) Determination of moisture content		13
F.1	Principle	13
F.2	Apparatus	13
F.3	Sample preparation	13
F.4	Procedure	13
F.5	Calculation	13
Annex G (normative) Determination of length and width		15
G.1	Apparatus	15
G.2	Procedure	15
G.2.1	Procedure for width	15
G.2.2	Procedure for length	15
Bibliography		16

Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS). The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the Principles and procedures for development of East African Standards. XXXXXX.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

The committee responsible for this document is Technical Committee EASC/TC 078, *Healthcare and medical devices*.

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

DEAS 1119 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: Bathing soaps*
- *Part 5: Bracelets, wristbands and patches*
- *Part 6: Jelly*

Skin applied mosquito repellents — Specification — Part 3: Wipes

1 Scope

This Draft East Africa Standard specifies requirements, sampling and test methods for skin applied mosquito repellent wipes

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.

DEAS 1120-1, *Mosquito repellent — Performance test guidelines — Part 1: Skin applied*

EAS 846, Glossary of terms relating to the cosmetic industry

ISO 139, Textiles — Standard atmospheres for conditioning and testing

ISO 1833-1, Textiles — Binary fibre mixtures — Quantitative chemical analysis

ISO 3071, Textiles — Determination of pH of aqueous extract

ISO 9073-1, Textiles — Test methods for nonwovens — Part 1: Determination of mass per unit area

ISO 9073-18, Textiles — Test methods for nonwovens — Part 18: Determination of breaking strength and elongation of nonwoven materials using the grab tensile test

ISO 18416, *Cosmetics — Microbiology — Detection of *Candida albicans**

ISO 20743, *Textiles — Determination of antibacterial activity of textile products*

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

ISO 21150, *Cosmetics — Microbiology — Detection of *Escherichia coli**

ISO 22717, *Cosmetics — Microbiology — Detection of *Pseudomonas aeruginosa**

ISO 22718, *Cosmetics — Microbiology — Detection of *Staphylococcus aureus**

ISO 2859-1, *Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <http://www.iso.org/obp>

3.1

mosquito

blood-sucking dipterous insect of the family Culicidae. Aedes, Anopheles, Culex, Mansonia, and Stegomyia are genera containing most species involved in the transmission of protozoan and other disease-causing parasites.

3.2

mosquito repellent

substance applied to deter mosquito from approaching or settling.

3.3

natural repellent

repellent that contain, plant-based compounds

3.4

synthetic repellent

conventional repellent containing chemical compounds manufactured to imitate the natural compounds

3.5

absorbency time

time taken for a liquid to be dispersed into the nonwoven disposable wet wipe

3.6

acceptable

recognized by the authority administering this standard, or to the parties concluding the purchase contract, as relevant

3.7

length of piece

distance between the beginning and the end of the sample in the lengthwise or machine direction

3.8

overall width of piece

distance between the outermost edges of the sample, measured perpendicular to the longitudinal edges

3.9

nonwoven

structure of textile material, such as fibres, continuous filaments, or chopped yarns of any nature or origin, that have been formed into webs by any means, and bonded together by any means, excluding the interlacing of yarns as in woven fabric, knitted fabric, laces, braided fabric or tufted fabric

3.10

mosquito repellent wipe

piece of soft, wet cloth or paper containing mosquito repellent

4 Symbols and/or abbreviated terms

DEET N, N-Diethyl-meta-toluamide or diethyltoluamide

IR3535 ethyl butylacetylaminopropionate

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

5 Active ingredients

5.1 Natural repellents

5.1.1 Active ingredients used in natural repellents shall be plant-based compounds which are able to deter mosquitoes from approaching or settling. Such shall be essential oils or any other plant extract approved by relevant authority as mosquito repellents.

5.1.2 The manufacturer shall provide adequate data on the repellence of such ingredients. Adequate data shall include laboratory studies showing estimation of effective dose (technical material) and estimation of complete protection time; and the outcomes of field trials showing efficacy and persistence of technical material; efficacy and persistence of formulated product.

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

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

- a) cedarwood;
- b) tea tree;
- c) geranium ;
- d) rosemary ;
- e) lemongrass ;
- f) citronella ;
- g) eucalyptus ;
- h) cinnamon ;
- i) neem ;
- j) peppermint;
- k) garlic;

- l) lavender
- m) cloves
- n) basils
- o) jasmine; and
- p) pyrethrum

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

5.1.6 Pyrethrum extracts such as pyrethrins shall be considered in natural repellents.

5.2 Synthetic repellents

5.2.1 Synthetic repellents shall contain synthetic chemical compounds which are able to deter mosquitoes from approaching or settling on the surface.

5.2.2 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.

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

6 Requirements

6.1 General requirements

6.1.1 The product shall constitute a mosquito repellent that is formulated as wipes and shall be essentially a product which has active ingredient(s) added to a certain level.

6.1.2 The product shall be of acceptable uniform make and finish.

6.1.3 The product shall be free from defects that might impair their appearance or serviceability (or both).

6.1.4 When applied to the skin, the product shall have the benefit of repelling mosquitoes and shall not have a harmful effect to the skin.

6.2 Specific requirements

6.2.1 Active ingredients and their content in mosquito repellent wipes shall meet the requirements given in Table 1 when tested in accordance with the test methods specified therein

Table 1 — Requirements for active ingredients in mosquito repellent wipes

S/N	Characteristic	Requirement	Test method
i	DEET ^a , % w/w.	4 – 30	Annex A
ii	IR3535 ^a , % w/w.	7.5 – 20.07	Annex B
iii	Picaridin ^a , % w/w.	5 – 20	Annex C

iv	Pyrethrins ^b , % w/w	0.3 - 0.5	Annex D
^a Applicable to synthetic mosquito repellent wipes			
^b Applicable to mosquito repellent containing extracts from pyrethrum.			

6.2.2 The product shall comply with the specific requirements given in Table 2 when tested in accordance with the test methods specified therein.

Table 2 — Specific requirements for skin applied mosquito repellents wipes

S/N	Parameter		Requirement	Test method	
i)	Fibre composition, % m/m	Cellulose, min.	20	ISO 1833-1	
		Others, max.	80		
ii)	Grammage, g/m ² , min.		36	ISO 9073-1	
iii)	pH		5.5 - 8.0	ISO 3071	
iv)	Absorbency time (for manufacturer), s, max.		5	Annex E	
v)	Moisture content, % m/m, min.		50	Annex F	
vi)	Size, mm	Length	As declared on the label	Annex G	
		Width			
vii)	Breaking strength, min. N,	Machine direction	Dry	ISO 9073-18	
			Wet		30
		Cross section	Dry		3.5
			Wet		2.5
viii)	Anti-bacterial activity (A), min.		2	ISO 20743	

6.3 Microbiological requirements

Skin applied mosquito repellent wipes shall comply with the microbiology limits specified in Table 3 when tested in accordance to the methods described therein

Table 3 — Microbiological limits for skin applied mosquito repellent wipes

S/N	Microorganism	Requirement	Test method
i	Total viable count, cfu/g, max.	1 000	ISO 21149
ii	<i>Staphylococcus aureus</i> , /g	Not detected	ISO 22718
iii	<i>Pseudomonas aeruginosa</i> , /g	Not detected	ISO 22717
iv	<i>Escherichia coli</i> , cfu/g	Not detectable	ISO 21150
v	<i>Candida albicans</i> , /g	Not detected	ISO 18416

6.4 Biological efficacy

When tested in accordance with DEAS 1120-1, the product shall repel 100 % of the mosquitoes from approaching or settling on the surface, within protection time indicated by the manufacturer.

7 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.

PUBLIC REVIEW DRAFT

8 Labelling

8.1 Primary packaging

The primary package shall be legibly and indelibly labelled in English and/or any other official language (French, Kiswahili, etc.) used in the importing East African Partner State with the following information

- a) name of product as “Mosquito repellent wipes”;
- b) name and physical address of the manufacturer;
- c) importer/distributors name, address (if applicable);
- d) number of wipes in a pack;
- e) intended use, as ‘for skin application’;
- f) size of wipes in the pack;
- g) fibre or material content and active ingredient used;
- h) instructions for use;
- i) storage conditions;
- j) instructions on disposal;
- k) country of origin;
- l) date of manufacture;
- m) expiry date
- n) batch number;
- o) safety caution; and
- p) protection time.

8.2 Secondary packaging

The exterior of each secondary package shall be legibly and indelibly labelled with the following information:

- a) manufacturer’s name and/or registered trademark;
- b) the words “Mosquito repellent wipes”; and
- c) number of packages.

9 Sampling

Sampling shall be done in accordance with ISO 2859-1.

Annex A (normative)

Determination of DEET content

A.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.

A.2 Apparatus

A.2.1 Double-beam infrared spectrophotometer

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

A.3 Preparation of calibration curve

A.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.

A.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.

A.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 (mg/L) of the meta-isomer.

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

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

Where ref. = determination with reference standard

blank = determination on CS₂ blank

A.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 clause 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.

A.5 Calculation

DEET content (g/kg)

$$= \frac{(C_1 \times P)}{C_2}$$

where,

C₁ concentration (g/L) of standard DEET found from calibration curve

C₂ concentration (g/L) of sample taken

P purity (g/kg) of the reference standard

Annex B (normative)

Determination of ethyl butylacetamidopropionate (IR3535).

B.1 Outline of method

B.1.1 Ethyl butylacetamidopropionate is determined by capillary gas chromatography using flame ionisation detection and internal standardisation.

B.1.2 The retention time of the ethyl butylacetamidopropionate peak of the sample solution should not deviate by more than 2 % from that of the calibration solution.

B.2 Reagents

B.2.1 Acetonitrile

B.2.2 Ethyl butylacetamidopropionate, standard of known purity

B.2.3 Methyl tridecanoate, internal standard

B.2.4 Calibration solution.

B.2.4.1 Weigh (to the nearest 0.1 mg in duplicate) into volumetric flasks (10 ml) about 100 mg ethyl butylacetamidopropionate standard (s mg) and about 100 mg methyl tridecanoate (r mg).

B.2.4.2 Dissolve in acetonitrile and fill to the mark with acetonitrile (solutions C1 and C2). The solutions are stable for one week at room temperature.

B.3 Apparatus

B.3.1 Gas chromatograph equipped with a split/splitless injection and a flame ionisation detector

B.3.2 Capillary column fused silica, 25 m × 0.32 (i.d.) mm, coated with CP-Sil 5 CB, film thickness: 1.2 µm

B.3.3 Electric integrator or data system

B.4 Procedure

B.4.1 Chromatographic conditions (typical)

B.4.1.1 Column fused silica, 25 m × 0.32mm (i.d.), film thickness: 1.2 µm, coated with CP-Sil 5 CB

B.4.1.2 Injection system

B.4.1.2.1 Injector: split injection

B.4.1.2.2 Split ratio:1:50

B.4.1.2 Detector: Flame ionisation

B.4.1.3 Temperatures

B.4.1.3.1 Injection port: 300 °C

B.4.1.3.2 Detector: 310 °C

B.4.1.3.3 Oven program

B.4.1.3.3.1 initial: 120 °C

B.4.1.3.3.2 Program rate: 10 °C/min

B.4.1.3.3.3 Final: 260 °C

B.4.1.4 Injection volume: 5 µl

B.4.1.5 Gas flow rates

B.4.1.5.1 Helium 1.1 ml/min

B.4.1.5.2 Helium (make up) 45 ml/min

B.4.1.5.3 Hydrogen 40 ml/min

B.4.1.6 Retention times

B.4.1.6.1 ethyl butylacetamidopropionate: about: 10.4 min

B.4.1.6.2 methyl tridecanoate: about: 10.9 min

B.5 Preparation of sample.

B.5.1 Weigh in duplicate (to the nearest 0.1 mg) into volumetric flasks (10 ml) sufficient sample to contain about 100 mg ethyl butylacetamidopropionate (w mg) and about 100 mg methyl tridecanoate (q mg).

B.5.2 Dissolve in acetonitrile and fill to the mark with acetonitrile (solutions S1 and S2). The solutions are stable for one week at room temperature.

B.6 System equilibration

Inject 0.1 µl portions of the calibration solution and repeat the injections until retention times and calibration factors vary by less than 0.1 % of the mean of three successive injections.

B.7 Determination.

B.7.1 Inject 0.1 µl portions of the calibration solution and sample solutions in the following sequence: C₁, S₁, C₂, S₂,...etc.

B.7.2 Determine the peak areas and calculate the mean response factor (*f*) of the calibration solution injections bracketing the injections of the sample solutions.

B.8 Calculation

B.8.1 Individual response factor f_i is calculated as follows:

$$f_i = \frac{I_r \times S \times P}{H_s \times r}$$

B.8.2 Ethyl butylacetamidopropionate shall be calculated as follows:

$$\frac{H_w \times f \times q}{I_s \times w}$$

where:

f_i individual response factor

f mean response factor

H_s peak area of ethyl butylacetamidopropionate in the calibration solution

H_w peak area of ethyl butylacetamidopropionate in the sample solution

I_r peak area of the internal standard in the calibration solution

I_q peak area of the internal standard in the sample solution

s mass of ethyl butylacetamidopropionate standard in the calibration solution (mg)

r mass of internal standard in the calibration solution (mg)

q mass of internal standard in the sample solution (mg)

w mass of sample taken (mg)

P purity of ethyl butylacetamidopropionate standard (g/kg)

Repeatability r 18 g/kg at 1002 g/kg active ingredient content

Reproducibility R 18 g/kg at 1002 g/kg active ingredient content

Annex C (normative)

Determination of icaridin

C.1 outline of method

Icaridin is determined by capillary gas chromatograph using internal standardisation and flame ionisation detection.

C.2 Reagents

C.2.1 Icaridin reference standard with known content

C.2.2 Dimethyl Phthalate internal standard

C.2.3 Propan-2-ol

C.2.4 Calibration solution.

Weigh (to the nearest 0.1 mg) about 100mg icaridin reference substance (*s* mg) and 100 mg dimethyl phthalate (*r* mg) into a volumetric flask (20 ml). Fill to the mark with propan-2-ol and homogenise.

C.3 Apparatus

C.3.1 Gas chromatograph capable of operating in the range 150 °C to 330 °C, fitted with a flame ionisation detector, a split injector and an autosampler

C.3.2 Column quartz, 30 m × 0.25 mm (i.d), coated with dimethyl polysiloxane/ diphenyl polysiloxane 95/5% (e.g. DB5), film thickness 0.25 µm

C.3.3 Electronic integrator or data system

C.4 procedure

C.4.1 Chromatographic conditions (typical)

C.4.1.1 Column: Quartz, 30 m × 0.25mm (i.d.), coated with dimethyl polysiloxane/ diphenyl polysiloxane 95/5% (e.g. DB5), film thickness 0.25 µm

C.4.1.2 Injection system

C.4.1.2.1 Injector: split injection

C.4.1.2.2 Split flow: 40ml/min

C.4.1.2 Detector: Flame ionisation

C.4.1.3 Temperatures

C.4.1.3.1 Injector: 240 °C

C.4.1.3.2 Detector: 330 °C

C.4.1.3.3 Oven program: 150°C hold for 2 min, gradient: 10°C/ min to 330 °C, hold for 3 min.

C.4.1.4 Injection volume: 1 µl

C.4.1.5 Gas flow rates

C.4.1.5.1 Helium (carrier) 1.5 ml/min (100kpa)

C.4.1.5.2 Hydrogen: about 30ml/min

C.4.1.5.3 Air: about 300ml/min

C.4.1.5.4 Nitrogen (makeup): about 25ml/min

C.4.1.6 Run time: about 25 min

C.4.1.7 Retention times

C.4.1.6.1 Dimethyl Phthalate: about 3 min

C.4.1.6.2 Icaridin: about 4.5 min

C.5 Preparation of sample

Weigh (to the nearest 0.1 mg) into a volumetric flask (20 ml) sufficient sample to contain about 100 mg icaridin (w mg) and about 100 mg dimethyl phthalate (q mg). Fill to the mark with propan-2-ol and homogenise.

C.6 Equilibration of the system

Inject 1 µl portion of calibration solution and repeat the injections until retention times and the icaridin to the internal standard peak area ratio vary by less than 0.5% of the mean for successive injections.

C.7 Determination

C.7.1 Inject in duplicate 1 µ portions of the calibration solution (C_1 and C_2) and of the sample solution (S_1 , S_2 , etc.) in the following sequence: C_1 , S_1 , S_2 , ... C_2 .

C.7.2 Determine the peak areas and calculate the response factor (f) from the calibration solutions bracketing the injections of the sample solutions. Calculate the content of the sample solutions.

C.8 Calculation

C.8.1 Response factor f is calculated as follows:

$$f = \frac{I_r \times S \times P}{H_s \times r}$$

C.8.2 Icaridin content shall be calculated in g/kg as follows:

$$\frac{H_w \times f \times q}{I_s \times w}$$

where:

f mean response factor

H_s peak area of icaridin in the calibration solution

H_w peak area of icaridin in the sample solution

I_r peak area of dimethyl phthalate in the calibration solution

I_q peak area of dimethyl phthalate in the sample solution

s mass of icaridin in the calibration solution (mg)

r mass of dimethyl phthalate in the calibration solution (mg)

q mass of dimethyl phthalate in the sample solution (mg)

w mass of sample taken (mg)

P purity of icaridin standard (g/kg)

Repeatability r 12 g/kg to 14 g/kg at 986 g/kg active ingredient content

Reproducibility R 14 g/kg at 986 g/kg active ingredient content

Annex D (normative)

Determination of total pyrethrins

D.1 General

The active ingredients in pyrethrum extract may be determined using a HPLC system first by injecting a solution of the analyte into the chromatograph, followed by the separation and comparison of peaks areas of the analytes in the sample with that of an external standard containing a known amount of the analytes. The peaks are eluted in the following order: Cinerin II, Pyrethrin II, Jasmolin II (total Pyrethrins II) and Cinerin I, Pyrethrin I, Jasmolin I (total Pyrethrins I).

D.2 Reagents

D.2.1 World pyrethrum standard, 50 %

D.2.2 Acetonitrile, HPLC grade

D.2.3 Water, HPLC grade

D.3 Apparatus

A liquid chromatography System equipped with an auto-sampler, a Variable Wavelength Detector (or equivalent) and a Column (Phenomenex, 250 x 4.6 mm Luna Phenyl-Hexyl 5 μ Reverse Phase (or equivalent)).

D.4 Operating conditions

D.4.1 Flow rate: 1.5 ml/min

D.4.2 Composition: 40:60 (% v/v water/acetonitrile)

D.4.3 Elution: Isocratic

D.4.4 Oven temperature: 40 °C

D.4.5 Wavelength: 240 nm

D.4.6 Injection Volume: 15 μ l

D.4.7 Stop time: 22 min

D.4.8 Post time: 1 min

D.5 Preparation of the standard

Weigh 20 mg of the pyrethrum standard to the nearest 0.0001 g in a 100 mL volumetric flask and dilute to volume with acetonitrile and label it. Transfer a small portion to a sample vial and label it accordingly.

D.6 Sample preparation

In a 100 ml volumetric flask, weigh 20 mg to the nearest 0.0001 g of the sample to be analyzed and dilute to volume with Acetonitrile. Sample this solution using a vial and label it accordingly.

D.7 Procedure

After the chromatograph is stable, make a minimum of three injections for the standard solution as well as for the analyte and average the area counts. The relative standard deviation between injections should be within 2 %.

D.8 Calculation of the % total pyrethrins (active ingredient)

The total pyrethrins, expressed as percent shall be calculated as follows;

$$\frac{\text{(Average sample area X weight of standard X Purity of the standard (in \%))}}{\text{(Average standard area X Weight of sample)}}$$

Annex E (normative)

Determination of absorbency rate

E.1 Apparatus

- E.1.1 Water tub, of a depth of at least 100 mm and maintained at room temperature
- E.1.2 Stopwatch, with an accuracy of 0.2 s
- E.1.3 Cylindrical basket, weighing $2.7 \text{ g} \pm 0.3 \text{ g}$ of height 80 mm, diameter 50 mm with square opening of 15 mm to 20 mm, made of copper wire of 0.4 mm diameter
- E.1.4 Analytical balance
- E.1.5 Forceps

E.2 Preparation of test specimens

Take three test specimens, each of mass at least 1 g and composed of a number of pinches of fibres taken from widely separated parts of the conditioned laboratory sample.

E.3 Procedure

- E.3.1 Compress the first test specimen to a volume of approximately 20 ml.
- E.3.2 By means of the forceps, place the test specimen lightly on the surface of the distilled water and simultaneously start the stopwatch.
- E.3.3 Using the stopwatch, measure the time it takes the basket and its contents to sink below the surface of water in seconds.
- E.3.4 Record the absorption period to the nearest 0.1 s.
- E.3.5 Repeat the test for at least three test specimens.

E.4 Calculation

Calculate, to the nearest second, the arithmetic mean of the three test results.

Annex F (normative) Determination of moisture content

F.1 Principle

A specimen of specified mass of filler material of the nonwoven disposable wet wipe is dried in an oven at a specified temperature and the moisture content is determined.

F.2 Apparatus

F.2.1 Balance, with an accuracy of 0.05 % of the weighed mass

F.2.2 Sample container, waterproof when sealed, will be used for transfer of analyzed material and during weighing

F.2.3 Oven, well ventilated with a temperature of 102 °C to 105 °C

F.3 Sample preparation

F.3.1 Take a sufficient number of dry sample containers, number them and take their masses after they are held open for a short period of time so that they will have the same air pressure as the surrounding atmosphere. Then leave them open until you take the test piece.

F.3.2 Take five random pieces of the wet wipes. The test pieces shall weigh 5 g.

F.3.3 If the surrounding atmosphere is hot and humid, prevent water condensation on the internal and external surfaces of the container.

F.3.4 Handle the test pieces gently to prevent dirt or changes in water content. Do not touch the test pieces with your bare hands. Put the test pieces in a container just after taking them and close the container immediately.

F.4 Procedure

F.4.1 Dry the test pieces in an oven with a temperature of 102 °C to 105 °C. Open the containers lid and dry the specimen inside the container. Open the container for a moment, to balance the air pressure inside the container with the surrounding pressure, weigh the container that holds the specimen again and calculate the weight of the specimen.

F.4.2 First cycle of drying will last at least 30 min. Return the container with the test pieces to the oven, for at least half the first cycle drying time. Take the container out and take the mass with the test pieces inside. Repeat the drying and weighing cycles. When the drying time on every cycle is at least half of the total previous drying cycle times. Continue the process until the difference between two consecutive masses does not exceed 0.1 % of the original mass of the specimen

F.5 Calculation

Calculate the moisture content using the following formula and round the results up to the nearest 0.1 %.

$$V = \frac{100(a - b)}{(a - c)}$$

where

- V is the moisture content, in percentage weight;
- a is weight, in grams, of the container with the specimen, before drying;
- b is weight, in grams, of the container with the specimen after drying; and
- c is weight, in grams, of the container

PUBLIC REVIEW DRAFT

Annex G (normative)

Determination of length and width

G.1 Apparatus

G.1.1 Steel scale, that is of a length exceeding the width of the fabric to be measured, and is graduated in centimeters and millimeters

G.1.2 Marking pen

G.2 Procedure

G.2.1 Procedure for width

G.2.1.1 Lay the test sample flat and full width (without subjecting it to tension) on a plane surface and condition it, in that state for at least 24 h in accordance with ISO 139.

G.2.1.2 Take, to the nearest 1 mm, five measurements across the overall width or between the innermost selvedge threads (as relevant) of the conditioned test sample at approximately equal intervals throughout its length.

G.2.1.3 Calculate the arithmetic mean of the five measurements and record it as the width of the sample.

G.2.2 Procedure for length

G.2.2.1 Take a laboratory sample as specified in the relevant standard. Where no standard exists, take the laboratory sample as agreed upon between the test laboratory and the manufacturer to ensure a reasonable and acceptable reliability at a reasonable and acceptable confidence level.

G.2.2.2 Lay the laboratory sample flat and full width (without subjecting it to tension) on a plane surface and condition it in that state for at least 24 h in accordance with ISO 139.

G.2.2.3 From the conditioned laboratory sample, cut a test specimen across the full width of the sample, along a datum line drawn at right angles to the selvages and as close as possible to the beginning and the end of the sample.

G.2.2.4 Take, to the nearest millimetre, five measurements (see G.2.1.2) of the length of the test specimen at approximately equal intervals across its width.

G.2.2.5 Calculate the arithmetic mean of the five measurements and record it as the length, in metres (accurate to the nearest centimetre), of the laboratory sample

Bibliography

- [1] RS 392-3: 2018, Skin applied mosquito repellents — Specification — Part 3: Wipes
- [2] US 2296-3: 2022, Skin applied mosquito repellents — Specification — Part 3: Wipes

PUBLIC REVIEW DRAFT

PUBLIC REVIEW DRAFT