



**RWANDA
STANDARD**

**DRS
465-2**

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**Pyrethrum-based insecticides —
Specification —**

Part 2:

Grease (GS) for direct application

ICS 65.100.10

Reference number

DRS 465-2: 2021

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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 465-2 was prepared by Technical Committee RSB/TC 007, *Agrochemicals*.

DRS 465 consists of the following parts, under the general title *Pyrethrums-based insecticides — Specification*:

- *Part 1: Dusting powders (DP)*
- *Part 2: Grease (GS)*
- *Part 3: Emulsions (Oil in water, EW)*

Committee membership

The following organizations were represented on the Technical Committee on *Agrochemicals* (RSB/TC 007) in the preparation of this standard.

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

University of Rwanda/College of Education (UR/CE)

Rwanda Forensic Laboratory (RFL)

Ministry of Environment (MoE)

Standards for Sustainability (SfS)

AGROPY Ltd

Rwanda Inspectorate, Competition and Consumer Protection Authority (RICA)

Rwanda Agriculture and Animal Resources Development Board (RAB)

Rwanda Standards Board (RSB) – Secretariat

Introduction

A paragraph.

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Pyrethrum-based Insecticides — Specification — Part 2: Grease (GS) for direct application

1 Scope

This Draft Rwanda Standard prescribes the requirements, sampling and test methods for pyrethrum-based grease insecticides for direct application used in animal protection to control insect pests, including ticks, lice and mites.

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 406, *Pesticides — Terminology*

RS 191, *Refined pyrethrum concentrate — Specification*

RS 405, *Pesticides — Sampling*

3 Terms and definitions

For the purposes of this standard, the terms and definitions given in RS 406 and the following apply.

3.1

pyrethrum

genus of several Old World plants now classified as *Chrysanthemum* or *Tanacetum* (e.g., *C. coccineum*) which are cultivated as ornamentals for their showy flowers heads

3.2

pyrethrum concentrate

extract of the flowers of the plant, *Chrysanthemum cinerariaefolium*

3.3

pyrethrins

the six naturally occurring isomers that are esters of pyrethric acid and chrysanthemic acid viz: pyrethrin-I, pyrethrin-II, cinerin-I, cinerin-II, jasmolin-I, jasmolin-II; having insecticidal property and are extracted from the flower of *Chrysanthemum cinerariaefolium*.

3.4

grease (GS)

very viscous formulation based on oil or fat

4 Requirements

4.1 General requirements

4.1.1 The product shall consist of a homogeneous mixture of pyrethrum extract, complying with RS 191, together with inert carriers such as petrolatum grease and any other necessary formulants.

4.1.2 The product may contain one or more synthetic pyrethroid, but the formulation shall be approved by the Competent Authority.

4.1.3 The product shall be in form of soft, homogenous unctuous mass.

4.2 Specific requirements

The product shall comply with specific requirements given in table 1 when tested according to the method prescribed therein.

Table 1 – Specific requirements for pyrethrum-based grease insecticides for direct application

S/N	Parameters	Requirements	Test methods
i.	Total pyrethrins content, % by mass	0.10 – 2.0	Annex A
ii.	pH range	5 – 7	Annex B
iii.	Melting point, °C	45 – 60	Annex C
iv.	Sulphated ash, % by mass, max.	0.10	Annex D
v.	Sulphur and sulphide	To pass test	Annex E
vi.	Stability	To pass test	Annex F

5 Packaging and labelling

5.1 Packaging

5.1.1 The product shall be packaged in a well closed container that will preserve its original characteristics.

5.1.2 The packaging material shall protect the contents from adventitious contamination under handling and storage conditions.

5.2 Labelling

The containers shall be closed and shall bear legibly and indelibly the following information in any of the three languages officially accepted in the Republic of Rwanda namely: Kinyarwanda, English and French.

a) Name of the product;

- b) Name and address of the manufacturer;
- c) Manufacture and expiry dates;
- d) Batch number;
- e) Active ingredient (s) contents;
- f) Storage conditions;
- g) Instructions for use;
- h) Precautions and warnings; and
- i) Country of origin.

6 Sampling

Representative samples of the product shall be drawn as prescribed in RS 405.

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Annex A (normative)

Determination of total pyrethrins

A.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).

A.2 Reagents

World pyrethrum standard, 50%

Acetonitrile, HPLC grade

Water, HPLC grade

A.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)}.

A.4 Operating conditions

Flow rate: 1.5 ml/min

Composition: 40:60 (% , v/v water/acetonitrile)

Elution: isocratic

Oven temperature: 40 °C

Wavelength: 240 nm

Injection volume: 15 μ l

Stop time: 22 min

Post time: 1 min

A.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.

A.6 Sample preparation

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

A.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 %.

A.8 Calculation

The total pyrethrins is calculated as follow:

$$\text{total pyrethrins, \% m/m} = \frac{\text{Average sample area} \times \text{weight of standard} \times \text{Purity of the standard (in \%)}}{\text{Average standard area} \times \text{Weight of sample}}$$

Annex B (normative)

Determination of pH value

B.1 Outline of the method

The pH value of a liquid is determined by means of pH meter and a glass electrode.

B.2 Reagents

B.2.1 Potassium hydrogen phthalate (COOH-C₆H₄-COOK) 0.05 mol/l (0.05M) – Dissolve 10.21 g in freshly boiled distilled water and make up to 1000 ml. do not keep the solution for longer than one month.

B.2.2 Disodium tetraborate (Na₂B₄O₇·10H₂O 0.05M – Dissolve 19.07 g in freshly boiled distilled water and make up to 1000 ml. do not keep the solution for longer than one month.

B.2.3 Water – Freshly boiled and cooled distilled water of pH 5.5 to 7.0

B.3 Apparatus

B.3.1 pH meter

B.3.2 Glass electrode and reference electrode

B.4 Procedure

Operate the pH meter and electrode system in accordance with the manufacturer's instructions. Standardize the meter and electrodes with the 0.05M phthalate (pH 4.00) when an acid solution is being measured or 0.05M borate when an alkaline solution is being measured (see Table B1). The reading should not differ by more than 0.02 pH units from the original value at which the apparatus was standardized. If the difference is greater than 0.05, then repeat the measurements.

Table B1 – pH values of 0.05M disodium tetraborate Temperature, °C	10	15	20	25	30
pH	9.32	9.28	9.22	9.18	9.14

B.5 pH of aqueous dispersion

weigh 1 g of sample, transfer to the measuring cylinder containing water (about 50 ml), make up to 100 ml with water, and shake vigorously for 1 min. allow any suspension to settle for 1 min and then measure the pH of the supernatant liquid.

Annex C (normative)

Determination of melting point

C.1 Heat a quantity of the sample on a water bath while stirring until it reaches a temperature of 90 °C to 92 °C. Cool the molten sample to a temperature of 8 °C to 10 °C above the expected melting point. Chill the bulb of a thermometer (range 1 °C to 100 °C) to 5 °C, wipe it dry and while it is still cold, dip it into the molten sample so that approximately half of the bulb is submerged. Withdraw it immediately and hold it vertically away from heat until the wax surface dulls, then dip it for 5 min into a water bath having a temperature not higher than 16 °C.

C.2 Fix the thermometer prepared in securely in a test tube so that its lowest point is about 15 mm above the bottom of the test tube. Suspend the test tube in a water bath adjusted to 16 °C and raise the temperature of the bath at a rate of 2 °C per minute up to 30 °C, then change the rate of rise to 1 °C per min and note the temperature at which the first drop of the melted sample leaves the thermometer. Repeat the determination twice on a freshly melted portion of the sample. If the variation in three determinations is less than 1 °C take the average of the three as the melting point. If the variation in three determinations is more than 1 °C, make two additional determinations and take the average of five.

Annex D (normative)

Determination of sulphated ash

D.1 Reagents

Dilute sulphuric acid, approximately 5 N

D.2 Procedure

Heat a porcelain or silica dish of 50 mL to 100 mL capacity to redness; cool in a desiccator and weigh. Place about 20 g of the sample, accurately weighed, in the dish. Heat the dish gently by means of a Bunsen burner until the oil can be ignited at the surface. Remove the burner and allow the oil to burn completely, taking care that all the free carbon on the sides of the dish is completely burnt. Heat the residue with a strong flame or in a muffle furnace until all the carbonaceous matter has disappeared. Cool the dish; add a few drops of dilute sulphuric acid; heat gently to drive off the acid and then heat strongly. Cool the dish again in the desiccator and weigh it. Repeat the heating, cooling and weighing until constant mass is obtained.

D.3 Calculation

The sulphated ash content shall be calculated as follows

$$\text{Sulphated ash, \% by mass} = \frac{M_2 - M_1}{M} \times 100$$

where

M_1 is the mass in g of the residue, and

M_2 is the mass in g of the sample taken for the test.

Annex E (normative)

Determination of sulphur and sulphides

E.1 Principle

The presence of sulphur and sulphides in oils is qualitatively determined using copper strips.

E.2 Apparatus and materials

E.2.1 Copper strips, 1 cm in width and freshly polished

E.2.2 Analytical balance, well calibrated and maintained

E.2.3 Thermostatically controlled water bath, capable of maintaining a temperature of 95 °C

E.2.4 Stop watch

E.3 Procedure

Weigh approximately 50 g of the sample in a beaker and place on a water-bath maintained at temperature of 95 °C. Place a strip of copper in the sample so that it is partially immersed in it and allow to remain for 10 min.

E.4 Results

The sample shall be taken to have passed the test if the copper strip used in the test shows no tarnishing when compared with another freshly polished copper strip.

Annex F (normative)

Determination of stability

F.1 Apparatus

Ultra violet lamp, with emission at 360 nm

F.2 Procedure

Place 50 mL of the material in a 100-ml glass beaker. Turn on the ultra violet lamp and expose the samples at a distance of 12 cm - 14 cm below the lamp for 6 h. After the specified time, remove the sample, cool to room temperature and compare for any change in odour or colour. The same volume of material shall be employed for all tests so that comparison is ensured on a reproducible basis.

NOTE — The output of the ultra violet lamp diminishes with time in service. A log of number of hours of the lamp in use should be maintained. The lamp is to be replaced after the specified hours of service, as recommended by the lamp manufacturer.

F.3 Evaluation

Evaluation is done by comparing the test material against an unexposed specimen from the same sample.

Bibliography

- [1] ES 757(First edition), *Pesticides — Determination of pH value*, 2002
- [2] RS EAS 847-22, *Cosmetics — Analytical methods — Part 22: Determination of sulphur and sulphides in oils*
- [3] RS EAS 342, *Pomades and solid brilliantines — Specification*

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