RWANDA STANDARD

DRS 465-1

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Pyrethrum-based insecticides — Specification —

Part 1:

Dusting powders (DP)

ICS 65.100.10

Reference number

DRS 465-1: 2021

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 465-1 was prepared by Technical Committee RSB/TC 007, *Agrochemicals.*

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

IS 6178: Specification for pyrethrum dusting powders

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

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

Pyrethrum owes its insecticidal properties to esters which are reportedly produced by a number of different cell types (oil glands, resin ducts and mesophyll cells). Pyrethrin I, jasmolin I and cinerin I are esters of chrysanthemic acid (chrysanthemum monocarboxylic acid), while pyrethrin II, jasmolin II and cinerin II are esters of pyrethric acid (monomethyl ester of chrysanthemum dicarboxylic acid). The biosynthesis of pyrethrin I in seedlings of C. cinerariifolium has been studied using [1-C]-d-glucose as a precursor; the acid portion of the molecule is derived from d-glucose and the alcohol moiety possibly from linoleic acid.

Pyrethrum Extract contains 20-50% of total pyrethrins; it may be prepared extemporaneously from the flower-heads and is used for the preparation of the dusting powder and spray. The dusting powder (pyrethrum extract, diatomite, talc) has a pyrethrin content of 0.10–0.50%, of which not less than half consists of pyrethrin I. It is assayed by titrimetry for both pyrethrin I and II. Extracts containing 50% more active material compared with commercial extracts can be obtained by extraction of the plant material with liquified carbon dioxide (100 bar).

Pyrethrum-based insecticides — Specification — Part 1: Dusting powders (DP)

1 Scope

This Draft Rwanda Standard prescribes the requirements, sampling and test methods for pyrethrum-based dusting powders used in the control of insect pests of agricultural importance, especially in grain storage.

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 extracts

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

3.3

pyrethrins

the six naturally occurring isomers that are esters of pyrethric acid and chrysenthemic 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

dusting powder (DP)

a free-flowing powder suitable for dusting

4 Requirements

4.1 General requirements

4.1.1 The product shall consist of a homogeneous mixture of pyrethrum extracts, complying with the requirements of RS 191, together with carriers and any other necessary formulant.

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 a fine, free-flowing powder, free from visible extraneous matter and hard lumps.

4.1.4 The product, when dusted through a hand rotary duster shall issue freely without clogging or bridging.

4.1.5 The product shall be whitish to brown in colour.

4.2 Specific requirements

4.2.1 The product shall comply with the requirements given in Table 1 when tested in accordance with the test methods prescribed therein.

Table 1 — Specific requirements for pyrethrum-based dusting powder insecticides

S/N	Parameters	Requirements	Test methods
i.	Total pyrethrins content, % by mass	0.10 — 5.0	Annex A
ii.	Material passing through 75 µm, % by mass, min.	90	Annex B
iii.	Bulk density after compacting	Not exceed the value obtained before compacting by more than 60%	Annex C
iv.	Acidity (as H_2SO_4), % by mass, max.	0.25	Annex D
v.	Alkalinity (as NaOH), % by mass, max.	0.05	
vi.	pH range	5 — 7	Annex E

4.2.2 **Storage stability** — After storage at 54 ± 2°C for 14 days, the product shall remain stable without any changes on quality and performance properties.

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.

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

Ww 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 5µ Reverse Phase (or equivalent)}.

A.4 Operating conditions

Flow rate:	1.5 ml/min
Composition:	40:60 (%, v/v water/acetonitrile
Elution:	isocratic
Column temperature:	40 °C
Wavelength:	240 nm
Injection volume:	15 µl
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:

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

Annex B
(normative)

Test sieves

B.1 Apparatus

Test sieve, 75 µm.

B.2 Procedure

Weigh accurately 10.0 g of the material and transfer it to the test sieve. Cover the sieve and screen the material in a Ro-Tap or a similar machine for 20 minutes. Two small square rubber cubes are introduced along with the material on the sieve to facilitate the breaking up of any soft lumps of the caked material.er 20 minutes stop the machine and brush the residue on the sieve into a tared weighing dish. Weigh the dish and determine the mass of the residue.

B.3 Calculation

Material passing through test sieve, 75 µm:

$$= 100\,(1 - \frac{m}{M})$$

Where,

m = mass in g of the material retained on the test sieve, and

M = mass in g of the material taken for the test

Annex C
(normative)

Determination of bulk density after compacting

C.1 Apparatus

C.1.1 **Graduated cylinder** – of 100 ml capacity with internal diameter of 27 t0 29 mm.

C.1.2 **Funnel** – wide and short-stemmed.

C.2 Procedure

C.2.1 **Bulk density before compacting** – Rest the funnel over the top edge of the tared graduated cylinder. Fill the cylinder to the 100-ml mark by pouring the material through the funnel, without tapping, and level the powder with the minimum of disturbance. Leave the cylinder untouched for 5 minutes and add more powder if necessary, to bring the contents to the 100-ml mark, level again with the minimum of disturbance. Weigh the filled cylinder and calculate the bulk density before compacting (mass of the material/volume occupied by the material).

C.2.2 **Bulk density after compacting** – Stopper the filled cylinder, and drop it 20 time through a height of 15 cm on to a felt pad resting on a hard surface. Note the volume of the material after compacting. Calculate the bulk density of the material after compacting (mass of the material/volume occupied by the material after compacting).

C.3 Test evaluation

The value obtained in C.2 shall not exceed the value obtained in C.1 by more than 60%.

Annex D
(normative)

Determination of acidity or alkalinity

D.1 Qualitative test

Procedure – Take about 0.5 g of the material in a test-tube and mix with about 1 ml of water. Test the mixture for acidity or alkalinity with a litmus paper. Determine the acidity or alkalinity, as the case may be.

D.2 Determination of acidity

D.2.1 Reagents

D.2.1.1 Methyl red indicator solution-aqueous – one percent (m/v)

D.2.1.2 Bromocresol purple indicator solution – one percent (m/v) in ethyl alcohol

D.2.1.3 Standard sodium hydroxide solution – 0.05N

D.2.1.4 Standard hydrochloric acid – 0.05N

D.2.2 Procedure

Weigh accurately 10.0 g of the material into a dry conical flask, add 25 ml of acetone and mix. Warm the flask gently to effect the solution of the active ingredient present. Add 75 ml of water and let it stand for an hour. Filter the supernatant aqueous extract and take 50 ml filtrate. Titrate immediately with the standard sodium hydroxide solution using methyl red or bromocresol purple as the indicator. Alternatively, the end point may be determined electrometrically.

Carry out a blank determination on an aliquot of 50 ml made from 25 ml acetone and 75 ml water.

D.2.3 Calculation

$$\text{Acidity (as } H_2SO_4\text{), \% m/m} = \frac{4.9\ x\ 2(V-v)N}{M}$$

Where;

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N = normality of the standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

In case the blank shows alkaline reaction, neutralize with the standard hydrochloric acid and calculate the acidity as follows:

$$\text{Acidity (as } H_2SO_4\text{), \% m/m} = \frac{4.9\ x\ 2(VN1 - vN2)}{M}$$

Where;

V = volume in ml of the standard sodium hydroxide solution required for the test with the material,

N_1 = normality of the standard sodium hydroxide solution,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N_2 = normality of the standard hydrochloric acid, and

M = mass in g of the material taken for the test.

D.3 Determination of alkalinity

D.3.1 Reagents

D.3.1.1 Methyl red indicator solution-aqueous – one percent (m/v)

D.3.1.2 Bromocresol purple indicator solution – one percent (m/v) in ethyl alcohol

D.3.1.3 Standard hydrochloric acid – 0.05N

D.3.1.4 Standard sodium hydroxide solution – 0.05N

D.3.2 Procedure

Weigh accurately 10.0 g of the material into a dry conical flask, add 25 ml of acetone and mix. Warm the flask gently to effect the solution of the active ingredient present. Add 75 ml of water and let it stand for an hour. Filter the supernatant aqueous extract and take 50 ml of folterate. Titrate immediately with the standard hydrochloric acid using methyl red or bromocresol indicator as the indicator. Alternatively, the end point may be determined electrometrically.

Carry out a blank determination on 50 ml aliquot made from 25 ml acetone and 75 ml water.

D.2.3 Calculation

$$\text{Alkalinity (as NaOH), \% m/m} = \frac{4.0\ x\ 2(V - v)N}{M}$$

Where;

V = volume in ml of the standard hydrochloric acid required for the test with the material,

v = volume in ml of the standard hydrochloric acid required for the blank determination,

N = normality of the standard hydrochloric acid, and

M = mass in g of the material taken for the test.

In case the blank shows acid reaction, neutralize with the standard sodium hydroxide solution and calculate the alkalinity as follows:

$$\text{Alkalinity (as NaOH), \% m/m} = \frac{4.0\ x\ 2(VN1 - vN2)}{M}$$

Where;

V = volume in ml of the standard hydrochloric acid required for the test with the material,

N_1 = normality of the standard hydrochloric acid,

v = volume in ml of the standard sodium hydroxide solution required for the blank determination,

N_2 = normality of the standard sodium hydroxide solution, and

M = mass in g of the material taken for the test.

Annex E
(normative)

Determination of pH value

E.1 Outline of method

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

E.2 Reagents

E.2.1 Potassium hydrogen phthalate (COOH-C_6H_4-COOK) 0.05 mol/l (0.05M) – Dissolve 10.21 g in freshly boiled distilled water and make up to 1000 ml. do no keep the solution for longer than one month.

E.2.2 Disodium tetraborate ($Na_2B_4O_7.10H_2O$ 0.05M – Dissolve 19.07 g in freshly boiled distilled water and make up to 1000 ml. do no keep the solution for longer than one month.

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

E.3 Apparatus

E.3.1 pH meter

E.3.2 Glass electrode and reference electrode

E.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 tetraborateTemperature, °C	10	15	20	25	30
pH	9.32	9.28	9.22	9.18	9.14

E.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.

Bibliography

[1] IS 6940, *Methods of test for pesticides and their formulations*, 2012

[2] ES 757, *Pesticides — Determination of pH values,* 2002

[3] ES 750, *Pesticides — Determination of free acidity or alkalinity,* 2002

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