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Pyrethrum-based insecticides— Specification —

Part 4: Sprays (Aerosol dispensers, AE)

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Reference number

DRS 465-4: 2023

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Foreword

Rwanda Standardsarepreparedby 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 465-4 was prepared by Technical Committee RSB/TC 064, *Pesticides.*

This first edition cancels and replaces respectively the second edition (RS 172:2019) and the first edition (RS 382: 2018) of which have been technically revised.

RS465consists of the following parts, under the general title *Pyrethrum-based insecticides— Specification*:

- *Part 1: Dusting powders (DP)*
- *Part 2: Grease (GS) for direct application*
- *Part 3: Emulsions (Oil in Water, EW)*
- *Part 4: Sprays (Aerosol dispensers, AE)*

Committee membership

The following organizations were represented on the Technical Committee on *Pesticides*(RSB/TC 064) in the preparation of this standard.

Rwanda Forensic Laboratory (RFL)

CYIRA Ltd

SOPYRWA

Rwanda Agriculture Board (RAB)

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

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 4: Sprays (aerosol dispensers, AE)

1 Scope

This DraftRwanda Standard prescribes the requirements, sampling and test methods for pyrethrum-based insecticide formulation to be used as spray treatment (aerosol dispensers, AE) with the aim of protecting crops, animals or public health from insects' pest or vectors.

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 editioncited 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*

RS ISO 1523, *Determination of flash point — Closed cup equilibrium method*

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

aerosol dispenser (AE)

device designed to produce a fine spray of liquid or solid particles that can be suspended in a gas such as the atmosphere. When a valve is released, the propellant forces the substance through an atomizer and out of the dispenser in the form of a fine spray

4 Requirements

4.1 General requirements

4.1.1 The product shall consist of a liquid formulation in a pressurized, non-refillable aerosol dispenser containing homogeneous mixture of pyrethrum extracts, complying with the requirements of RS 191, dissolved essentially in refined (quick-drying) solvents with the addition of deodorants, antioxidants and if required, a synergist may be used to impart only necessary efficiency to the formulation.

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

4.1.3 The product shall not contain the prohibited solvents given in Annex A as per the Montreal Protocol.

4.1.4 The formulation shall be clear and light in colour and shall have no unpleasant odour when sprayed; it should be non-irritant and non-toxic to humans, pets.

4.1.5 The product shall be non-reactive and non-staining to household structures, items and fabrics.

4.2 Specific requirements

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

Table 1 – Specific requirements for pyrethrum-based spray insecticides

S/N	Parameters	Requirements	Test methods
i.	Total pyrethrins content, % by mass	0.10 — 5.0	Annex B
ii.	Internal pressure at 30 ± 2˚C, MPa, max.	15	Annex C
iii.	Discharge rate, g formulation/second.	1	AnnexD
iv.	Clogging of aerosol dispenser valves	No clogging shall occur	Annex E
v.	pH	4 – 7	Annex F
vi.	Flash point, ºC, min.	32	RS ISO 1523

5 Packaging

The product shall be packaged in accordance with the requirements prescribed in CD xxx:2023.

6 Labelling

The product shall be labelled in accordance to the requirements prescribed in CD xxx:2023.

7 Sampling

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

Annex A
(normative)

Solvents not permitted for use in aerosols

a) Benzene

b) 2-butoxyethanol (ethylene glycol monobuthyl ether)

c) 2-butoxyethylacetate (ethylene glycol monobutyl ether acetate)

d) carbon tetrachloride

e) chlorobenzene

f) chloroform

g) 1,2-dichloroethane (ethylene dichloride)

h) 2-ethoxyethanol (ethylene glycol monoethyl ether)

i) 2-ethoxyethylacetate (ethylene glycol monoethyl ether acetate)

j) n-hexane

k) 2-hexanone (methyl n-butyl ketone)

l) 2-methoxyethanol (ethylene glycol monomethyl ether)

m) 2-methoxyethylacetate (ethylene glycol monomethyl ether acetate)

n) tetrachloroethylene

o) trichloroethylene.

NOTE The Montreal Protocol 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 total pyrethrins

B.1 General

The activeingredients 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).

B.2 Reagents

World pyrethrum standard, 50%

Acetonitrile, HPLC grade

Water, HPLC grade

B.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)}.

B.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

B.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.

B.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.

B.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 %.

B.8 Calculation

The total pyrethrins is calculated as follow:

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

Annex C

(normative)

Determination of pressure in finished aerosol pack

C.1 Introduction

The determination of the pressure existing in the finished aerosol packs is necessary to verify that the true pressure is compatible with the pressure limitations of the pack, and in accordance with the regulations in force.

True pressure is the relative pressure given by an accurate manometer, at a given temperature.

C.2 Objective

The determination of the true pressure in the finished aerosol pack:

a) In such a way that the measurement affects as little as possible the value of the real pressure;

b) In such a way that the manometer will not be polluted by the product under pressure present in the pack.

C.3 Scope

The method is recommended for determination of the true pressure of all filled aerosol packs.

C.4 Apparatus

C.4.1 The following are required:

a) a source of reference gas (nitrogen for instance) from which the pressure can be regulated by means of a control valve;

b) a manometer of high accuracy, if possible cushioned by an oil-bath and adapted to fit the aerosol container on which the measurement will be effected (Figure 1).

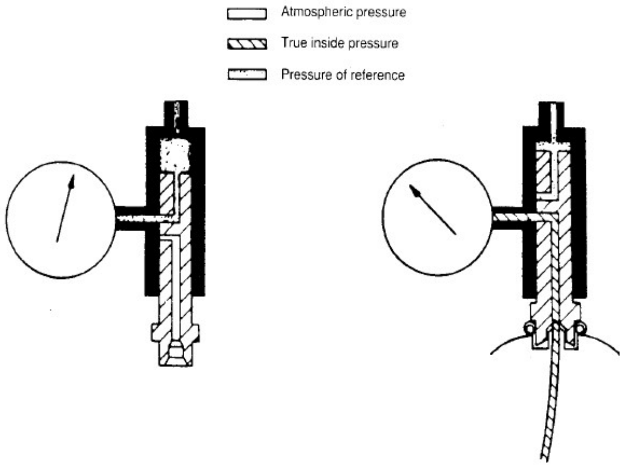

Figure 1 – Manometric measurement of internal pressure

C.4.2 The apparatus must be assembled in such a way that, in the state of rest, the manometer is connected to the reference gas (the pressure of this gas being slightly higher than the actual pressure in the pack) and, for taking the measurement, the manometer is connected to the interior of the pack to show the actual pressure.

C.5 Work operation

C.5.1 Make sure that the aerosol dispenser is thoroughly equilibrated to 30 ± 2ºC.

C.5.2 The measuring apparatus must be fitted with an appropriate adaptor for the valve employed.

C.5.3 The pressure of the reference gas must be regulated to a value slightly higher than the anticipated pressure of the dispenser.

C.5.4 Apply the measuring apparatus to the valve and press lightly in order to open the valve and the slide of the apparatus.

C.5.5 Read the true pressure on the manometer, when the needle has stabilized.

C.6 Accuracy and measurement

The measurement of true pressure will be the more accurate:

a) with larger aerosol dispenser sizes;

b) with only a small difference in pressure between the reference gas and the true pressure of the dispenser (if required, measurements can be made on additional dispensers after adjusting the reference gas pressure to a value very close to the true pressure);

c) if the dead volume of the manometer is small (less than 2 ml).

C.7 Test report

The test report must indicate, in addition to the results and test conditions, any relevant working details not specified in the method, especially if they are suspected of having influenced the results.

NOTE 1 It is necessary to recalibrate the manometer frequently, for example with the aid of a manometric balance.

NOTE 2 A non-return valve can be inserted in the apparatus to avoid the aerosol product penetrating the manometer if the pressure of the reference gas is inadvertently much lower than the true pressure in the dispenser.

Annex D
(normative)

Evaluation of discharged rate of filled aerosol dispensers

D.1 Scope

The method is applicable to the majority of aerosols marketed at present. It may be used, with discretion, for dispenser fitted with vapour-phase taps where there will be a continually changing composition as the contents are discharged. It is not suitable for use in the inverted position for dispensers with vapour phase taps, as there will be an erratic discharge dependent upon the liquid content of the dip tube. Where dispensers are intended to be used in an inverted position, it is vital that the test shall be done in that way. It is important that discharge tests follow the instructions for use given on the dispenser. The method is not intended for use with metering valves.

D.2 Principle

The discharge rate of an aerosol dispenser is determined by measuring the quantity of material expelled through the valve in the given time. The exact duration of discharge, normally 10 sec., and the temperature of the dispenser must be carefully controlled for good reproducibility. Normally the test is repeated three times to give three determinations but, in the case of products filled with vapour phase tap valves, it is preferable to reduce the discharge interval to 5 sec. and the number of determinations to two. This is to minimize the variation in composition that will occur as the contents are sprayed off. There will be some loss of accuracy using the shorter duration of discharge.

Alternatively, the discharge rate may be determined at different stages in the emptying of the dispenser (e.g. 90%, 70%, 50%, 30% and 10% fill) which then allows the values to be plotted graphically. Where the composition changes with the emptying of the packs, this offers a particularly convenient way of showing the effect of this change. In the case of storage test samples, a single test is normally performed at each examination to conserve the contents.

D.3 Apparatus

A water bath at 25°C ± 0.5°C, a stop watch accurate to 0.2 second, a balance weighing to 0.1 g and a pressure gauge accurate to 2 psig (0.1 $kg.cm_{-2}$) are required.

D.4 Procedure

a) The valve of the aerosol dispenser shall be handled according to label instructions and the valve shall be operated for five sec to remove material in the dip tube which may not be homogenous with the bulk of the filling.

b) b) The aerosol dispenser shall be immersed in a water bath maintained at 25°C ± 0.5°C for half an hour, or for sufficiently long for the contents to attain the temperature of the water bath.

c) c) The aerosol dispenser shall be removed from the water bath, wiped completely dry, the valve operated for one second to remove any water in the valve, the internal pressures of the dispenser shall be measured and the dispenser shall be weighed to within 0.1g.

d) d) The aerosol dispenser shall then be shaken for 3 sec by hand or other suitable means and the valve shall be operated fully open for 10 sec, timed by the stop watch. During the discharge, the dispenser shall be positioned as indicated in the instructions for use.

e) e) The aerosol dispenser shall be wiped clean of any liquid and re-weighed to within 0.1 g.

Procedures (b) to (d) shall be repeated twice or more and the internal pressure of the dispensers shall be measured again.

D.5 Calculation

The difference in weights derived from procedure (e) and procedure (c) shall be divided by 10 (sec). Results should not differ by more than 0.1 g from the mean of the three results. If a greater difference is found at least two more readings should be taken.

D.6 Reporting

Report valve discharge rates as g.sec^{-1} at the measured mean pressure of the dispenser. The method, e.g. 3 x 10 sec, 2 x 5 sec or 1 x 5 sec (90%....50%....10%) spraying, etc., shall be quoted.

NOTE 1 Where discharge rates are to be determined at various stages of pack emptying it is advisable to allow the pack to reach equilibrium at 25°C in the water bath, shake and spray for 5 sec, re-shake vigorously and re-spray for another 5 sec and replace in water bath. This procedure may then be repeated until the correct pack content is reached. This rather tedious procedure avoids errors arising from the fall in temperature of the contents of the pack during prolonged spraying.

NOTE 2 Results may show variations from sample to sample that are greater than might be expected from normal manufacturing tolerances. This may be due variations in the valve orifice diameter.

NOTE 3 The discharge rate of freshly prepared aerosol dispensers will not normally be the same as for samples allowed to ―mature‖, due to the effect of solvents on the gasket.

Annex E
(normative)

Testing of valves of filled aerosol dispensers for clogging

E.1 Apparatus

E.1.1 Fume hood

E.1.2 Protective clothing

E.2 Procedure

Shake the aerosol dispensers thoroughly and, keeping them in an upright position, disperse the contents of each into the fume hood. Actuate the valve in a series of cycles (30 sec. on, 30 sec. off) until the dispenser is emptied. Examine the valves for clogging.

Annex F
(normative)

Determination of pH value

F.1 Outline of the method

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

F.2 Reagents

F.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.

F.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.

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

F.3 Apparatus

F.3.1 pH meter

F.3.2 Glass electrode and reference electrode

F.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

F.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] Manual on development and use of FAO and WHO specifications for pesticides, November 2010, 2nd Edition.

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