RWANDA STANDARD

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Profenofos pesticides — Specification — Part 1: Technical material

ICS 65.100.10

Reference number

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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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Contents

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 618-1 was prepared by Technical Committee RSB/TC 007, *Agrochemicals*

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

ES 711-1: Pesticides — Profenofos — Part 1: Technical material (TC) — Specification

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

DRS 618 consists of the following parts, under the general title *Profenofos pesticides — Specification*:

— *Part 1: Technical material*

— *Part 2: Emulsifiable concentrate (EC)*

— *Part 3: Ultra low volume liquids (ULV)*

Committee membership

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

Rwanda Food and Drugs Authority

Rwanda Forensic Institute

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

University if Rwanda/College of Agriculture, Animal Sciences and Veterinary Medicine (UR/CAVM)

Standards of Sustainability

CYIRA Ltd

Rwanda Inspectorate, Competition and Consumer Protection Authority (RICA)

Rwanda Investigation Bureau (RIB)

Rwanda Agriculture and Inputs Organization (RAIDO)

VeR Alka Group Ltd

SAFE HARVEST SOLUTIONS Ltd

Rwanda Standards Board (RSB) – Secretariat

Introduction

Profenofos is an organophosphate insecticide. It is a liquid with a pale yellow to amber color and a garlic-like odor. Profenofos can be used on a variety of crops including cotton and vegetables such as maize, potato, soybean, and sugar beet. Mixed with phoxim, cypermethrin, beta-cypermethrin imidacloprid and deltamethrin, profenofos can be used against Cotton MealyBug, cabbage caterpillar, Plutella xylostella and asparagus caterpillars, as well as against wheat and cabbage aphids.

Like other organophosphates, the profenofos mechanism of action is via the inhibition of the acetylcholinesterase enzyme. Although it is used in the form of a racemate, the S(-) isomer is a more potent inhibitor. Profenofos can be synthesized by reacting phosphorus oxychloride with sodium ethoxide and sodium 1-propanethiolate, followed by treatment with 4-bromo-2-chlorophenol.

Profenofos pesticide — Specification — Part 1: Technical material

1 Scope

This Draft Rwanda Standard specifies the requirements, sampling and test methods for technical material of Profenofos pesticides.

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 405, *Pesticides — Sampling*

RS 406, *Pesticides — Terminology*

ASTM E 1064-12, *Standard Test Method for water in organic liquids by Coulometric Karl Fischer Titration*

RS 565-1, *Packaging of Pesticides — Requirements — Part 1: Solid pesticides*

RS 578, *Pesticides — Guidelines on good labelling practices*

RS 579, *Pesticides — Guidelines for retail, distribution, storage and handling*

RS 210, *Safety procedures for the disposal of surplus pesticides and associated toxic waste — Code of practice*

3 Terms and definitions

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

4 Requirements

4.1 General requirements

The technical material of profenofos shall be in the form of a yellowish to liquid brown clear to slight turbid liquid, free from extraneous impurities or added modifying agents.

4.2 Specific requirements

The technical material of profenofos shall comply with the requirements given in table 1.

Table 1 – Specific requirements for technical material of profenofos

S/N	Parameters	Requirements	Test methods
1	Profenofos content, % by mass, min.	89	Annex A
2	Moisture content, % by mass, max.	0.20	ASTM E 1064-12
3	Matter insoluble in acetone, % by mass, max.	0.15	Annex B
4	Acidity (as H_2SO_4), % by mass, max.	0.50	Annex C

5 Packaging

The product shall be packaged in accordance with RS 565-1.

6 Labelling and marking

The labelling and marking of the product shall be done in accordance with DRS 578.

7 Retail, distribution, storage and handling

The product shall be handled in accordance with DRS 579.

NOTE Attention is drawn to the appropriate national and/ or international regulations on the handling and transport of flammable materials.

8 Sampling

Sampling shall be done in accordance with RS 405.

9 Disposal

Disposal of bulk quantities of obsolete pesticides shall be in accordance with DRS 589.

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Annex A
(normative)

Determination of profenofos content

A.1 General

Profenofos is separated from other components by gas chromatography on a packed column with flame ionization detection and internal standardization.

A.2 Reagents

A.2.1 Acetone

A.2.2 Profenfos of known purity; better than 980 g/kg

A.2.3 Di-(2-ethylhexyl) adipate internal standard

A.2.4 Internal standard solution

Dissolve di-(2-ethylhexyl) adipate (980 to 1020 mg) in acetone (500 ml)

A.2.5 Calibration solution

Weight (to the nearest 0.1 mg) profenofos standard (90 to 100 mg) into a glass-stoppered conical flask (50 ml). add by pipette internal standard solution (25.0 ml), dilute to the mark with acetone and mix well. Prepare in duplicate.

A.3 Apparatus

A.3.1 Gas chromatograph – Fitted with a flame ionization detector

A.3.2 Column – Glass, 1.8 m X 2 mm (i.d.) packed with 3% OV-210 on supercoport, 80-100 mesh

A.3.3 Electronic integrator or data system

A.3.4 Microsyringe 10 µl

A.4 Procedure

A.4.1 Identity test

Use the GLC method below. The retention time of profenofos in the sample solution should not deviate by more than 0.2 min from that of the calibration solution.

A.4.2 Profenofos content

A.4.2.1 Operating conditions (typical):

Column	glass, 1.8 m x 2 mm (i.d.)
Packing	3% silicone OV-210 on Supelcoport 80 – 100 mesh
Column temperature	180 ˚C
Injector temperature	240 ˚C
Detector temperature	280 ˚C
Carrier gas	helium, 35 ml/min
Injection volume	1 µl
Retention time	profenofos: 6.5 min.
	Internal standard 11.4 min.

A.4.2.2 Linearity check

Check the linearity of the detector response by injecting 1 µl of solutions with profenofos with concentrations 0.5, 1 and 2 times that of the concentration of the calibration solution. Be sure that the concentrations of the solutions are in the linear range of the detector, otherwise alter the weighings or the dilutions accordingly. Inject each solution at least twice and determine the response factor (f). the single values should differ by less than 0.5 % from the mean value, otherwise repeat the calibration.

A.4.2.3 Preparation of sample

Weigh (to the nearest 0.1 mg) into a glass stoppered conical flask (50 ml) sufficient sample to contain 90 to 100 mg profenofos (w mg). add by pipette internal standard solution (25.0 ml), dilute to the mark with acetone and mix well. Prepare in duplicate.

A.4.2.4 Determination

Inject each sample solution in duplicate into the gas chromatograph and bracket a series of sample solutions by duplicate injections of the calibration solutions as follows: calibration solution 1 (double injection) calibration solution 2 (double injection), and so on. Measure the relevant peak areas.

Average the response factors of each double injection. Calculate the mean value of each pair of response factors bracketing the injections of two samples and use this value for calculating the profenofos contents of the bracketed sample injections.

A.4.2.4 Calculation

$$f = \frac{Ir\ X\ s\ X\ P}{Hs}$$

$$Content\ of\ profenofos = \frac{f\ X\ Hw}{I4\ X\ W}\ g/kg$$

Where,

f	is response factor;
Hs	is area of profenofos peak in the calibration solution;
Hw	is area of profenofos peak in the sample solution;
Ir	is area of internal standard peak in the calibration solution;
Iq	is area of internal standard peak in the sample solution;
s	is mass of profenofos in the calibration solution (mg);
W	is mass of sample taken (mg)
P	is purity of profenofos reference substance (g/kg)

Annex B
(normative)

Determination of material insoluble in acetone

B.1 Reagent

Acetone, $(CH_3)_2CO$ dried over anhydrous sodium sulphate, RE 34.

B.2 Apparatus

250 ml conical flask fitted with ground glass joint.

Reflux condenser to fit flask.

Sintered glass crucible-porosity P40 (pore size 16 – 40 µm)

Flame proof oven at 110 ˚C.

Water bath.

B.3 Procedure

Weigh about 10 g of sample (w g) into the conical flask, add acetone (150 ml) and reflux for a period of 10 min. filter the solution through the tared crucible (x g) and wash well with more acetone (3 X 20 ml). Dry at 110 ˚C for 30 min, cool and weigh (z g).

NOTE The crucible porosity is only given as a guide; it may be necessary to use a coarser or finer pore size in accordance with the particle size of the insoluble material.

B.4 Expression of results

Results shall be expressed in percent in accordance with the following equation:

$$\textit{Material insoluble in acetone, \% w/w} = \frac{100(z-x)}{w}$$

Where,

x is the weight of crucible;

z is the weight of crucible with dried residue; and

w is the weight of sample taken.

Annex C
(normative)

Determination of acidity or alkalinity

C.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.

C.2 Determination of acidity

C.2.1 Reagents

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

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

C.2.1.3 Standard sodium hydroxide solution – 0.05N

C.2.1.4 Standard hydrochloric acid – 0.05N

C.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.

C.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.

C.3 Determination of alkalinity

C.3.1 Reagents

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

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

C.3.1.3 Standard hydrochloric acid – 0.05N

C.3.1.4 Standard sodium hydroxide solution – 0.05N

C.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.

C.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.

Bibliography

[1] ES 765, *Pesticides — Determination of profenofos content*

[2] IS 15238, *Profenofos, Technical — Specification*

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