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Profenofos pesticides — Specification — Part 2: Emulsifiable concentrates (EC)

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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 618-2 was prepared by Technical Committee RSB/TC 64, *Pesticides*

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

1) ES 711-2: Pesticides — Profenofos — Part 2: Emulsifiable concentrates (EC) — Specification

2) IS 15236: Profenofos + Cypermethrin emulsifiable concentrate — Specification

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

DRS 918 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 pesticides — Specification — Part 2: Emulsifiable concentrates (EC)

1 Scope

This Draft Rwanda Standard specifies the requirements, sampling and test methods for profenofos pesticides in form of emulsifiable concentrates (EC) for plant protection purpose. It covers also profenofos + cypermethrin emulsifiable concentrates.

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*

RS 590, *Pesticides — Determination of total cypermethrin content*

RS 580-1, *Cypermethrin Pesticides — Specification — Part 1: Technical material*

DRS 618-1, *Profenofos Pesticides — Specification — Part 1: Technical material*

RS 565-2, *Packaging of Pesticides — Requirements — Part 2: Liquid 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*

RS ISO 2719, *Determination of flash point — Pensky-Materns closed cup method*

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

4.1.1 The product shall consist of technical profenofos, complying with the requirements of DRS 618-1, dissolved in suitable solvents together with any other necessary formulants.

4.1.2 The profenofos content shall be declared in (g/kg or g/l at 20 ± 0.5 ºC and, when determined in accordance with annex A, the content obtained shall not differ from that declared by more than the following amounts:

Table 1—Permitted tolerance on declared content

Declared content	Permitted tolerance
Above 100 up to 250 g/kg or g/l	± 6% of the declared content
Above 250 up to 500 g/kg or g/l	± 5% of the declared content
Above 500 g/kg or g/l	± 2.5% of the declared content

NOTE If the buyer requires both g/kg and g/l at 20 ºC, then in case of dispute the analytical result shall be calculated as g/kg.

4.1.3 Tha cypermethrin may be combined with profenofos, and the cypermethrin shall comply with the requirements of RS 580-1.

4.1.4 The product shall be in the form of clear, homogeneous and stable liquid, free from visible suspended matter and sediment.

4.1.5 The product shall readily form an emulsion on dilution with water.

4.2 Specific requirements

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

Table 2 — Specific requirements for profenofos emulsifiable concentrates pesticides

S/N	Parameters		Requirements		Test methods
			Profenofos	Profenofos + Cypermethrin	
i.	Profenofos content, % by mass		As per 4.1.2-	400 g/L	Annex A
ii.	Cypermethrin content, % by mass		-	40 g/L	RS 590
iii.	Cold test		No turbidity or separation of solid oily matter		Annex B
iv.	pH range		3.0 – 7.0		Annex C
v.	Flash point, °C, min.		24.5		RS ISO 2719
vi.	Acidity (as H_2SO_4), g/l, max.		-	0.6	Annex D
vii.	Storage stability	At 0 ± 2°C for 7 days, ml, max.	0.3		Annex E
		At 54 ± 2°C for 14 days, % m/m, min.	95		Annex F

5 Packaging

The product shall be packaged in accordance with RS 565-2.

6 Labelling and marking

The product shall be labelled and marked in accordance with RS 578.

7 Retail, distribution, storage and handling

The product shall be handled in accordance with RS 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 RS 210.

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
(informative)

Cold test

B.1 Procedure

Take 50 ml of the material in a 100-ml transparent glass container and close it with a cork or stopper fitted with the thermometer. Cool the material to 10 ºC (see note) by placing the container in ice cold water. When the temperature of the material in the container reaches 10 ºC, add to it a small seeding crystal of the pesticide with minimum opening of the stopper within the shortest possible duration of time. In case the formulation has been prepared from technical liquid pesticides, there is no need to seed with crystal. Gently stir the material in the container at short intervals for one hour maintaining the temperature of the material at 10 ºC. at the end of one hour, examine the material for any turbidity or separated solid or oily matter or both.NOTE In case the purchaser specifies that the test is required to be carried out at 0 ºC, the material in a container shall be cooled to 0 ºC by placing the container in a bath of crushed ice and the test carried out at 0 ºC instead of 10 ºC.

Annex C
(normative)

Determination of pH value

C.1 Outline of the method

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

C.2 Reagents

C.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.

C.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.

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

C.3 Apparatus

C.3.1 pH meter

C.3.2 Glass electrode and reference electrode

C.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

C.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 D
(normative)

Determination of acidity or alkalinity

D.1 Quality 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 = is the volume in ml of the standard sodium hydroxide solution required for the test with the material,

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

N = is the normality of the standard sodium hydroxide solution, and

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

N_1 = is the normality of the standard sodium hydroxide solution,

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

N_2 = is the normality of the standard hydrochloric acid, and

M =is the 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.3.3 Calculation

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

Where

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

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

N = is the normality of the standard hydrochloric acid, and

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

N_1 = is the normality of the standard hydrochloric acid,

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

N_2 = is the normality of the standard sodium hydroxide solution, and

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

Annex E
(normative)

Determination of stability of liquid formulations at 0 °C

E.1 Outline of the method

A sample is maintained at 0 °C for 1 h and the volume of any separated solid or oily matter is then recorded. Storage at 0 °C is continued for 7 days, any solid matter is settled by centrifuging and its volume recorded.

E.2 Emulsifiable concentrates and solutions

E.2.1 Apparatus

E.2.1.1 Refrigerator – Capable of maintaining a temperature at 0 ± 1 °C.

E.2.1.2 Cone shaped centrifuge tubes, 100-mL.

E.2.1.3 Centrifuge – equipped with buckets capable of holding the specified tubes.

E.2.1.4 Pipette, 100-mL.

NOTE A domestic refrigerator is often unsuitable because the on/off cycle covers a range greater than 2 °C.

E.2.2 Procedure

Transfer 100 ± 1.0 mL of a sample of the product to a centrifuge tube. Cool the tube and its content to (0 ± 1) °C in the refrigerator. Allow the tube and its contents to remain at (0 ± 1) °C for 1 h, and during this time stir the contents of the tube at intervals of approximately 15 min, each time for approximately 30 s. After this period examine the tube and record whether any solid or oily matter is present. Replace the tube in the refrigerator and allow it to remain at (0 ± 1) °C for a total period of 7 days.

At the end of 7 days, remove the tube from the refrigerator, and allow it to remain undisturbed at room temperature for 3h. invert the centrifuge tube once, and centrifuge for 15 min at such a speed that the relative centrifugal force (RCF) at the tips of the tubes is about 550 x G (the acceleration due to gravity = 981 cm/s^{-2}.

Record the volume of any separated material at the bottom of the tube to the nearest 0.005 mL.

NOTE 2 : RCF = $\frac{(rpm)^2 d}{179000}$ and rpm = $\sqrt{98.45\ X\ d^{-1}\ X\ 10^3}$

Where:

RCF is relative centrifuge force;

d is diameter of swing (in cm) measured from the tips of the opposite tubes when in the position occupied during the centrifuging.

If the liquid phase is not homogenous, record the volume of each layer.

E.3 Aqueous solutions

E.3.1 Apparatus

E.3.1.1 Measuring cylinder, 100-mL.

E.3.1.2 Refrigerator, at 0 ± 1 °C

E.3.2 Procedure

Put 100 ml of the product in the measuring cylinder and then put it in the refrigerator for 48 h at 0 ± 1 °C. At the end of this time, note the amount of separated material, if any, then allow the cylinder to reach room temperature and again note the amount of separated material.

Annex F
(normative)

Determination of accelerated storage stability

F.1 Outline of the method

Representative sample is stored in a screw-capped bottle in an oven at a specified temperature and time.

F.2 General method

As this is intended as a model procedure, temperature and times specified are examples only since the parameters will normally be given for individual pesticide formulations.

F.3 Apparatus

F.3.1 **Beaker** – 250-mL, 6 to 6.5 internal diameter.

F.3.2 **Metal disc** – Plastic coated; a loose fit in the beaker, and of such dimensions that an even pressure of 25 g/cm^2 can be produced on the surface of the sample in the beaker.

NOTE Alternatively, a close fitting cylinder with a flat bottom, containing lead shot, can be used, the lead shot may be sealed in with molten wax so as ti give the correct weight, and prevent the shot from being lost.

F.3.3 **Oven** – Thermostatically controlled to the specified temperature (± 2 °C)

F.3.4 **Desiccator without desiccant**

F.4 Procedure

Put the sample into the beaker and spread it, without using any pressure, in a smooth even layer of constant thickness. Place the disc on the surface of the solution in the beaker, and put in the oven. After the specified time remove the beaker, take out the disc, and allow the beaker to cool in the desiccator.

NOTE Use the specified temperature and time given in the specification of method of analysis. If no temperature of time is specified, store the sample at 54 ± 0.2 °C for 14 days.

Ensure that each sample taken is truly representative of that left in the beaker. Sampling of a hard cake may be carried out conveniently by removing several cores with a small diameter (6 mm) cork borer.

Bibliography

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

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