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Lambda-cyhalothrin — Specification — Part 2: Emulsifiable concentrates (EC)

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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 593-2 was prepared by Technical Committee RSB/TC 64, *Pesticides*.

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

ES 716-2: Pesticides — Lambda-cyhalothrin, Part 2: Emulsifiable concentrates (EC) — Specification

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

DRS 593 consists of the following parts, under the general title *Lambda-cyhalothrin pesticides — Specification*:

— *Part 1: Technical material*

— *Part 2: Emulsifiable concentrates (EC)*

— *Part 3: Water dispersible granules*

— *Part 4: Rapid-release capsule suspension*

Committee membership

The following organizations were represented on the Technical Committee on *Pesticides* (RSB/TC 64) in the preparation of this standard.

Rwanda Food and Drugs Authority

Rwanda Forensic Institute

University of Rwanda/College of Sciences and Technology

Standards of Sustainability

CYIRA Ltd

Rwanda Inspectorate, Competition and Consumer Protection Authority

Rwanda Investigation Bureau

Rwanda Agriculture and Inputs Organization (RAIDO)

Rwanda Standards Board (RSB) – Secretariat

Introduction

A paragraph.

Lambda-cyhalothrin pesticides — Specification — Part 2: Emulsifiable concentrates (EC)

1 Scope

This Draft Rwanda Standard specifies the requirements, sampling and test methods for lambda-cyhalothrin pesticides in form of emulsifiable concentrates (EC) meant for plant protection purpose.

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*

DRS 594, *Pesticides — Determination of lambda-cyhalothrin content*

DRS 593-1, *Lambda-cyhalothrin — Specification — Part 1: Technical material*

ASTM E1064-12, *Standard Test Method for water in organic liquids by Coulometric Karl Fischer Titration*

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

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

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

FDRS 589, *Pesticides — Guidelines for the disposal of bulk quantities of obsolete pesticides*

RS 405, *Pesticides — Sampling*

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 product shall consist of technical lambda-cyhalothrin, complying with the requirements of DRS 593-1 dissolved in suitable solvents together with any other necessary formulants.

The product shall be in the form of a clear to slightly hazy, stable homogeneous liquid, free from visible suspended matter and sediment, to be applied as an emulsion after dilution with water.

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 emulsifiable concentrate lambda-cyhalothrin pesticides

S/N	Parameters		Requirements	Test methods
i.	Lambda-cyhalothrin content, % by mass, min.		2.5	DRS 594
ii.	pH range (1% aqueous dispersion)		6 – 8	Annex A
iii.	Emulsion stability and re-emulsification		Pass the test	Annex B
iv.	Persistent foam (after 1 minute), ml, max.		15	Annex C
v.	Storage stability	At 0 ± 2°C for 7 days, ml, max.	0.3	Annex D
		At 54 ± 2°C for 14 days, % m/m, min.	95	Annex E

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 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.

Annex A
(normative)

Determination of pH

A.1 Outline of the method

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

A.2 Reagents

A.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.

A.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.

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

A.3 Apparatus

A.3.1 pH meter

A.3.2 Glass electrode and reference electrode

A.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

A.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 B
(normative)

Determination of characteristics of emulsion (EW) insecticides

B.1 Outline of the method

Five ml of the product are mixed with a standard water to give 100 ml of aqueous emulsion. The stability of this emulsion is then assessed in terms of the amounts of free 'oil' or 'cream' which separates while the emulsion is allow to stand undisturbed for 24 h. The ability of the system to re-emulsify at the end of 24 h period is also determined. If required, the test is repeated on a fresh sample of the emulsion.

B.2 Methods of determination

B.2.1 Five percent v/v oil phase

B.2.1.1 Hand shaking

B.2.1.1.1 Apparatus

Measuring cylinders – A 100-ml glass stoppered, the volume between the 100 ml graduated mark and the bottom of the stopper should be not more than 40 ml and not less than 35 ml. The apparatus must be clean and free from grease.

Constant temperature bath – Large enough to allow several 100 ml measuring cylinders to be immersed in an upright position in the water to the neck; maintained at 30 ± 1 °C (Note).

NOTE Any vibration can alter the properties of the dilute emulsion in the cylinder. The cylinder should, therefore, be supported or clamped in such a way that it is not in contact with the body of the water bath. The stirrer assembly should preferably be clamped independently of the water bath.

Adjustable lamp – Fitted with a 60-watt bulb.

Measuring cylinders, 5 ml.

B.2.1.1.2 Procedure

Initial emulsification-fill a 100 ml measuring cylinder to the 95 ml mark with standard water at 30 ± 1 °C unless otherwise specified. The sample (5 ml at the same temperature as the standard water) is gently poured on to the surface of the water; the stopper is replaced and the cylinder is inverted once (Note).

After 30 sec observe whether the mixture has emulsified spontaneously giving 100 ml of an emulsion which appears, on visual examination, to be uniform. Note any froth produced.

The expression 'invert the cylinder' implies that the stoppered cylinder is tipped by hand through 180 degrees, and is then brought back to its original position, the whole operation being completed in approximately 2 sec.

Emulsion stability on standing – Invert the cylinder 10 times and allow the cylinder and its contents of stand undisturbed in the constant temperature bath at 30 ± 1 °C for 24 h. Record the volume if any of free oil), froth and 'cream' formed either at the top or the bottom of the emulsion, after standing for 30 min, 2 h and 24 h.

NOTE 1 An adjustable lamp, fitted with a 60-watt pearl bulb, should be used to illuminate the cylinder. The position and angle of the light should be adjusted for optimum viewing of the phase boundary. It is often easier to see this by reflected, rather than by transmitted light.

NOTE 2 If, initially, difficulty is experienced in distinguishing between oil and cream, a dye soluble in the oil phase may be used, but the final tests should be carried out without the addition of dye. It has been found that dyes which give a deep blue solution in aromatic hydrocarbon solvents, e.g. oil Blue SWS, 1,4- is (isopropylamine) anthraquinone (CI 61551), are most suitable for this purpose. The dye (0.1g/100ml) should be added to the emulsion before carrying out the test. If oil is present then the dye will colour it deep blue; if extensive creaming has occurred, the dye will give a pale blue layer; if little or no creaming has occurred then no definite colour band will be produced.

At the end of the 24 h period invert the cylinder 10 times. Allow to stand for 30sec, then observe whether any free oil, froth, 'cream' or solid matter found after standing for 24 h is re-emulsified, giving 100 ml of an emulsion which appears, on visual examination, to be uniform.

Final emulsion stability – Allow the cylinder to remain undisturbed for a further period of 30 min.

Record the volume, if any, of free oil, froth, 'cream', or solid matter present at the end of the 30 min period.

B.2.1.2 Mechanical shaking

B.2.1.2.1 Apparatus

As for D.2.1.1.1 together with shaking apparatus; the plate should not rotate at 30 rpm.

B.2.1.2.2 Procedure

Initial emulsification – Fill a 100-ml measuring cylinder to the 95 ml mark with standard at 30 ± 1 °C. pour the emulsion, which should be at the same temperature as the standard water, gently (5 ml from a measuring cylinder) on the surface of water, replace the stopper and invert the cylinder once (Note 2).

After 30 sec observe whether the mixture has emulsified spontaneously giving 100 ml of an emulsion which appears, on visual examination, to be uniform. Note any froth produced.

Emulsion stability on standing - Shake the cylinder for 20 sec. in the shaking machine (i.e. 10 inversions) and allow the cylinder and its contents to stand undisturbed in the constant temperature bath at 30 ± 1 °C for 24 h. record the volume (Note 3), if any, of free oil, froth and the total volume of 'cream' formed either at the top or the bottom of the emulsion, after standing for 30 min, 2 h and 24 h.

Re-emulsification after standing for 24 h-At the end of the 24 h period shake the cylinder for 20 sec as before. Record whether any free oil, froth, 'cream' or solid matter, found after standing for 24 h is re-emulsified, giving 100 ml of an emulsion which appears, on visual examination (Note 3), to be uniform.

Final emulsion stability – Allow the cylinder to remain undisturbed for a further period of 30 min. Observe the volume, if any, of free oil, froth, 'cream', or solid matter present at the end of the 30 min period.

B.2.2 1% v/v oil phase

B.2.2.1 Preliminary examination

Prepare a 5% v/v dilution of the emulsion in water and allow to stand in a 100 ml measuring cylinder. Allow to remain undisturbed at room temperature to determine whether top or bottom creaming occurs.

In the sample does not separate, no further testing is required.

If it does separate after 24 h, continue the test by the appropriate section of clause C.2.1.2.1**.**

B.2.2.2 Dispersion stability

B.2.2.2.1 Apparatus

Measuring cylinder – A 250-ml fitted with stopper, and with dimensions of between 20 and 21.5 cm from the bottom, i.e. the 0-ml mark to the 250-ml mark.

Water bath – At 30 ± 1 °C unless otherwise specified.

Sampling tube – A piece of a small bore tubing 2 mm internal diameter, about 30 cm long, fitted at one end with a two-way stopcock. One of the arms of the stopcock is connected to a Drechsel bottle with nylon tubing; the outlet from the bottle is connected to a vacuum source via a second stopcock. The tube is fitted with a bung to act as a stop.

- Pipette, 2 ml

B.2.2.2.2 Procedure

Active ingredient in initial dispersion – Determine the content of active ingredient in 100 m of the initial dispersion (Note 7). Express the result in grams (x g) per 100 ml of the dilute emulsion.

NOTE The content of active ingredient in the initial dispersion must be determined by the same method as is used for determination of the active ingredient in the dilute emulsion after estimation of dispersion stability.

Top creaming – Removal of 100 ml of emulsion from the bottom of the cylinder, i.e. between 0 and 100 ml marks. Adjust the stop on the glass tube so that the tip of the tube is below the 10 ml graduation mark near the bottom of the cylinder. Remove the tube and fill the cylinder to the 100 ml mark with water. Fill the sampling tube to the top with water and close the two-way stopcock. Calibrate the cylinder by inserting the tube into the cylinder (i.e. near the bottom) and mark the new level of water on the side of the cylinder.

Pour the standard water (198 ml at 30 °C unless otherwise specified) into the cylinder and add the emulsion (2 ml). Insert the stopper, invert the cylinder 30 times (Note 2), and put the cylinder in the water bath at 30 ± 1 °C (note 1).

At the end of the specified time place the sample tube, filled with water, in position in the cylinder, open the stopcocks, and remove the dilute emulsion until the surface of the liquid reaches the calibration mark of the cylinder. Close the stopcock on the tube, remove the tube from the cylinder, and wash any material adhering to

the outside of the tube directly into the cylinder. Open the stopcock, cuck the remainder of the liquid into the bottle, and wash the tube and leads to the bottle by inserting the tube in distilled water, and applying suction.

Determine the insecticide content (y g) of the dilute emulsion remaining in the cylinder (Note 8).

The insecticide content of the emulsion drawn into the Drechsel bottle may also be determined (z g) to check the recoveries of the active ingredient, since y + z should equal 2x.

Bottom creaming - (Removal of 100 ml of emulsion from the top of the cylinder i.e. between the 100 and 200 ml graduations). Pour standard water (198 ml at 30 °C unless otherwise specified) into the cylinder and add the emulsion (2.0 ml). Insert the stopper, invert the cylinder 30 times, and place in the water bath at 30 ± 1 °C. At the end of the specified time insert the sampling tube into the cylinder and draw the dilute emulsion over into the Drechsel bottle, until the surface of the liquid in the cylinder reaches the 100 ml mark, maintaining the tip of the tube just below the sinking level of the liquid. Withdraw the sampling tube and wash the tube and leads to the bottle by inserting the tube into distilled water and applying suction.

Determine the insecticide content (y g) of the dilute emulsion remaining in the cylinder (Note 8).

B.2.2.2.3 Dispersion stability

$$Dispersion\ stability,\ \%\ m/m = \frac{100(2X-y)}{X}$$

NOTE 6 The method is not suitable for formulations intended for low volume spraying and/or aerial application; it may not be suitable for 'invert' formulations.

B.2.3 Results

After testing, the formulation shall comply with the following:

Time after dilution	Limits of stability
0 h	Initial emulsification: complete
0.5 h	"Cream", maximum: 0 ml
2.0 h	"Cream", maximum: 0 ml
	"Free oil", maximum: 0 ml
24 h	Re-emulsification: complete
24.5 h	"Cream", maximum: 0 ml
	"Free oil", maximum: 0 ml

Annex C
(normative)

Determination of persistent foaming

C.1 Apparatus

C.1.1 Stoppered measuring cylinder, 100 ml – if possible select one whose volume between 100 ml graduation mark and the bottom of the stopper is, not more than 40 ml and not less than 35 ml (Note 1).

NOTE 1 The cylinder should be clean and free from grease.

C.1.2 **Weighing bottle**.

C.1.3 **Graduated cylinder** – Glass stoppered, 250 ml capacity with 2 ml graduations, the distance between the 0 mark and the 250 ml mark being 20 – 21.5 cm, and between the 250 ml mark and the bottom of the stopper, 4 – 6 cm.

C.1.4 **Stopwatch**.

C.2 Procedure

C.2.1 Weigh out the specified amount of the material and add it to standard water (95 ml) in the measuring cylinder and make up to the mark. Stopper the cylinder and invert 30 times (Note 2). Stand the cylinder on the bench and leave undisturbed for the specified time. Note the volume of foam (Note 3).

NOTE 2 The expression 'invert the cylinder' means that the stoppered cylinder in held by two hands, one at each end of the cylinder, which are insulated from the cylinder by means of a cloth. The upright cylinder in turned through 180 degrees and back to its original position without any 'bounce' occurring, this operation taking approximately 2 sec. It is convenient to observe a stop-clock equipped with a second hand which sweeps once every 60 sec. while doing this.

NOTE 3 A few bubbles round the periphery are not significant. Any volumes above the 100 ml mark or the 250 ml mark should be marked on the outside and the volume of foam thus determined.

C.2.2 The mass of sample to be taken is that mass required to make 200 ml of a suspension with a concentration recommended in the directions for use supplied with the product. Where several concentrations are recommended, the maximum concentration shall be used.

C.2.3 Put about 180 ml of standard water into 250 ml measuring cylinder standing on a top pan balance and weigh in the required amount of the sample. Top up with standard water until the distance between the suspension surface and the bottom of the ground glass joint is 9 ± 0.1 cm. stopper the cylinder and invert 30 times. Place the stoppered cylinder upright on the bench and immediately start the stopwatch. Read the volume of foam produced and remaining after 10 ± 1 sec, 1, 3 and 12 min ± 10 sec.

Annex D
(normative)

Determination of stability of liquid formulations at 0 °C

D.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.

D.2 Emulsifiable concentrates and solutions

D.2.1 Apparatus

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

D.2.1.2 Cone shaped centrifuge tubes, 100 ml.

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

D.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.

D.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}).

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

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.

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

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

D.3 Aqueous solutions

D.3.1 Apparatus

D.3.1.1 Measuring cylinder, 100ml.

D.3.1.2 Refrigerator, at 0 ± 1 °C

D.3.2 Procedure

Put 100 m 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 E
(normative)

Determination of accelerated storage stability

E.1 Outline of the method

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

E.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.

E.3 Apparatus

E.3.1 **Beaker** – 250-ml, 6 to 6.5 internal diameter.

E.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.

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

E.3.4 **Desiccator without desiccant**

E.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 (Note 2). 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.

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