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Cypermethrin 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.

RS 580-1 was prepared by Technical Committee RSB/TC 64, *Pesticides.*

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

ES 705-1: Pesticides — Cypermethrin — Part 1: Emulsifiable concentrates (EC) — Specification

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

RS 580 consists of the following parts, under the general title *Cypermethrin pesticides — Specification*:

— *Part 1: Technical material (TC)*

— *Part 2: Emulsifiable concentrates (EC)*

— *Part 3: Wettable powders (WP)*

— *Part 4: Ultra low volume liquids (ULV)*

— *Part 5: Technical concentrates (TK)*

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

P-TECHNIKS Ltd

Rwanda Inspectorate, Competition and Consumer Protection Authority

Rwanda Investigation Bureau

RAIDO

Rwanda Standards Board (RSB) – Secretariat

Introduction

A paragraph.

Cypermethrin Pesticides — Specification — Part 2: Emulsifiable concentrates (EC)

1 Scope

This Draft Rwanda Standard specifies the requirements for the emulsifiable concentrates pesticides based on cypermethrin 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 405, *Pesticides — Sampling*

RS 406, *Pesticides — Terminology*

DRS 580-1, *Cypermethrin pesticides — Specification — Part 1: Technical material (TC)*

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

DRS 591, *Pesticides — Determination of total cypermethrin content and diastero isomer ratio*

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

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

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

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

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

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

RS ASTM D 6450-12, *Standard Test Method for Flash Point by Continuously Closed Cup (CCCFP) Tester*

3 Terms and definitions

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

4 Requirement

4.1 General requirements

4.1.1 Description

The material shall consist of technical cypermethrin, complying with the requirements of DRS 580-1 dissolved in suitable solvents, with any other necessary formulants. It shall be in the form of a stable liquid, free from visible suspended matter and sediment.

4.1.2 Active ingredient

4.1.2.1 Identity tests

Where the identity of the active ingredient is in doubt it shall be determined in accordance with DRS 590 and then the isolated active ingredient shall comply with at least one additional test.

4.1.2.2 Cypermethrin

The cypermethrin content shall be declared in g/kg or g/l at 20ºC and, when determined in accordance with DRS 590, the content obtained shall not differ from that declared by more than the following amounts:

Declared content	**Permitted tolerance**
Above 25 up to 100 g/l or g/kg	±10% of the declared content
Above 100 up to 250 g/l or g/kg	±6% of the declared content
Above 250 up to 500 g/l or g/kg	±5% of the declared content

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

4.1.3 Cis-isomer

The *cis*-isomer content shall be declared and shall be between 40% minimum and 60% maximum of the declared cypermethrin content.

When determined in accordance with DRS 590, the permitted tolerance shall be ± 10% of the declared *cis*-isomer content.

4.2 Specific requirements

The EC Cypermethrin pesticides shall comply with the specific requirements given in Table 1 when tested in accordance with test methods specified therein

Table 1 — Specific requirements for the cypermethrin pesticides

S/N	Parameter		Requirement		Test methods
i.	Water content, g/kg		2		ASTM E1064-12
ii.	Acidity, g/kg, max.		Shall not exceed 1.5		Annex A
iii.	Flash point		Not less than the minimum declared flash point		ISO 2719
iv.	Emulsion stability and re-emulsion	Dilution at 0h	Limits of stability	Initial emulsification complete	Annex B
		Dilution at 0.5h		'Cream' maximum: 1 ml	
		Dilution at 2h		'Free oil': nil	
		Dilution at 24h		Re-emulsification complete	
		Dilution at 0.5h 24.5h		'Free oil' maximum 0.5 ml	
v.	Storage stability	at ±1ºC for 7 days	the volume of solid and/or liquid, which separates, shall not be more than 0.3ml		Annex C
		at 54 ± 2ºC for 14 days	average active ingredient content shall not be lower than 95%		Annex D

5 Packaging

The EC cypermethrin pesticides shall be packaged in accordance with RS 565-2.

6 Labelling and marking

The labelling and marking of EC cypermethrin pesticides shall be done in accordance with DRS 578.

7 Retail, distribution, storage and handling

The EC cypermethrin pesticides 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

Samples shall be taken 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 acidity or alkalinity

A.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.

A.2 Determination of acidity

A.2.1 Reagents

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

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

A.2.1.3 Standard sodium hydroxide solution – 0.05N

A.2.1.4 Standard hydrochloric acid – 0.05N

A.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.

A.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.

A.3 Determination of alkalinity

A.3.1 Reagents

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

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

A.3.1.3 Standard hydrochloric acid – 0.05N

A.3.1.4 Standard sodium hydroxide solution – 0.05N

A.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.

A.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 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 1 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

a) 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.

NOTE 2 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.

b) Emulsion stability on standing

Invert the cylinder 10 times (Note 2), 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 (Note 3), if any of free oil (Note 4), 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 3 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 4 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.

c) Emulsion stability on standing

At the end of the 24 h period invert the cylinder 10 times (Note 2). 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 (Note 3), to be uniform.

d) 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 C.2.1.1.1 together with shaking apparatus; the plate should not rotate at 30 rpm.

B.2.1.2.1 Procedure

a) **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.

b) **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.

c) **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 (Note 6)

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.2.2.1 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

a) 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 7 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.

b) 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).

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.

c) 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

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

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 stability of liquid formulations at 0 °C

C.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.

C.2 Emulsifiable concentrates and solutions

C.2.1 Apparatus

C.2.1.1 Refrigerator – Capable of maintaining a temperature at 0 ± 1 °C (Note 1)

C.2.1.2 Cone shaped centrifuge tubes, 100 ml.

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

C.2.1.4 Pipette, 100 ml.

NOTE 1 A domestic refrigerator is often unsuitable because the on/off cycle covers a range greater than 2 °C.

C.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}, Note 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.

NOTE 3 If the liquid phase is not homogenous, record the volume of each layer.

C.3 Aqueous solutions

C.3.1 Apparatus

C.3.1.1 Measuring cylinder, 100ml.

C.3.1.2 Refrigerator, at 0 ± 1 °C

C.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 D
(normative)

Determination of accelerated storage stability

D.1 Outline of the method

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

D.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.

D.3 Apparatus

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

D.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 1 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.

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

D.3.4 **Desiccator without desiccant**

D.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 2 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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