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Deltamethrin Pesticides — Specification — Part 1: Technical material

ICS 65.100.10

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Contents

Page

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 595-1 was prepared by Technical Committee RSB/TC 64, *Pesticides*

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

IS 12005, *Specification for Deltamethrin, Technical*

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

DRS 595 consists of the following parts, under the general title *Deltamethrin Pesticides — Specification*:

— *Part 1: Technical material*

— *Part 2: Wettable Powder*

— *Part 3: Dustable powder*

— *Part 4: Aqueous suspension concentration*

— *Part 5: Emulsifiable concentrate*

— *Part 6: Ultra low volume liquid*

— *Part 7: Water dispersible granules*

— *Part 8: Emulsion, oil-in-water*

— *Part 9: Emulsifiable granules*

— *Part 10: Long lasting treated storage bag*

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

Deltamethrin Pesticides — Specification — Part 1: Technical material

1 Scope

The Draft Rwanda Standard specifies the requirements for the technical material of deltamethrin pesticides for agricultural 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*

3 Terms and definitions

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

4 Requirements

4.1 Active ingredient

4.1.1 Identity tests

The active ingredient shall comply with an identity test and, where the identity remains in doubt, it shall be determined in accordance with annex A, and then it shall comply with at least one additional test.

4.1.2 Deltamethrin content

The deltamethrin content shall be declared (not less than 985 g/kg) and, when determined in accordance with annex A, the average measured content shall not differ from that declared by more than ±25g.

4.2 General requirements

4.2.1 The product shall consist of deltamethrin together with related manufacturing impurities.

4.2.2 It shall be a white to cream coloured crystalline powder, odourless and free from visible extraneous matter and added modifying agents

4.3 Specific requirements

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

Table 1 — Specific requirements for deltamethrin technical material

S/N	Characteristics	Requirements	Test method
i.	Deltamethrin content, % by mass, min.	98	Annex A
ii.	Melting point, ºC	98 – 101	
iii.	Optical rotation of 1% solution in benzene at 20 ºC	57±1.5 ºC	Annex B
iv.	Acid chloride corresponding to deltamethrin, % by mass, max.	0.2	Annex C
v.	Acid + anhydride corresponding to deltamethrin, % by mass, max.	1.0	Annex D

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.

Annex A
(normative)

Determination of deltamethrin content

A.1 General

Either of the two methods, namely, HPLC method (see A.2) or total bromine analysis method (see A.3) shall be used for estimating deltamethrin content. However, HPLC method will be the referee method in case of dispute.

A.2 HPLC METHOD

A.2.1 Principle

After dilution of the sample, deltamethrin content is determined by comparing the response of the sample with that of a deltamethrin standard of known purity by HPLC on a column packed with silica.

A.2.2 Apparatus

A.2.2.1 Liquid Chromatograph — A suitable instrument for use with stainless steel columns and capable of maintaining pressures of 150 bars, fitted with a 20 microlitre loop injector.

A.2.2.2 Detector — UV spectrophotometer capable to measure UV absorption at 254 nm. Check its linearity in the concentration zone used for the determination.

A.2.2.3 Liquid Chromatographic Column — Stainless steel, 15-18 cm long, 4’6 mm internal diameter packed with Lichrosorb silica 60-80 mesh.

A.2.2.4 Recorder — One mV full scale recorder

A.2.3 Reagents

A.2.3.1 Deltamethrin Standard — Of known purity.

A.2.3.2 Dioxan — UV spectroscopic grade, free of peroxides. Dioxan is mixed with 15 ml of water per litre

A.2.3.3 Iso-octane — UV spectroscopic grade

A.2.3.4 HPLC Mobile Phase — Mixture of dioxan and iso-octane in the ratio of 50 ml/950 ml.

A.2.4 Procedure

A.2.4.1 Preparation of the Standard 'Solution— Weigh, in duplicate, to the nearest mg approximately 50 mg of deltamethrin standard (r_1g and r_2g) into 50-ml volumetric flasks. Add a mixture of dioxan and iso-octane in the ratio of 200 ml/800 ml and shake the flasks -to ensure dissolution of the standards. Dilute to volume with the same mixture (Solutions R_l and R_2).

A.2.4.2 Sample Preparation— Weigh in duplicate to the nearest 0.1 mg samples containing approximately 50 mg of deltamethrin (M_1g and M_2g) into 50-ml volumetric flasks. Add a mixture of dioxan and iso-octane in the ratio of 200 ml/800 ml and shake the flasks to dissolve the samples. Dilute to volume with the same mixture (Solutions S_1 and S_2).

A.2.4.3 Liquid Chromatography Conditions — The conditions given below are typical. Conditions may have to be adjusted to obtain optimum results from a given apparatus provided standardization is done:

Column temperature: Ambient

Flow rate: 1 to 1.7 ml/m

Detector set at: 254 nm

Approximate retention time: 8 min

A.2.4.4 Procedure— With a 20-microlitre loop type injector, inject one of the standard solutions until peak height or area of two successive injections agree to within 2 percent. Inject the standard and sample solutions in succession according to the following sequences:

R_1S_1, R_2S_1, R_1S_2 and R_2S_2

A.2.5 Calculation

Deltamethrin content, percent by mass = $\frac{Ax \times r \times P}{Ar \times m}$

Where,

Ax = deltamethrin peak area of the sample solution (S_1 or S_2);

r = mass, in g, of the deltamethrin in the standard solution (R_1 or R_2);

P = purity of deltamethrin standard;

Ar = deltamethrin peak area of the standard solution (R_1 or R_2); and

m = mass, in g, of the sample in the sample solution (S_1 or S_2).

Note: Alternatively, the calculation can be based on the peak heights. The four results should agree to within ± 3 percent of their mean value. If not, repeat the analysis.

A.3 TOTAL BROMINE ANALYSIS METHOD

A.3.1 Reagents

A.3.1.1 Isopropanol— pure

A.3.1.2 Metallic sodium

A.3.1.3 Formaldehyde

A.3.1.4 Phenolphthalein Solution — one percent (m/m) in rectified spirit.

A.3.1.5 Nitric acid

A.3.1.6 Benzene

A.3.1.7 Silver Nitrate Solution— 0.1 N

A.3.1.8 Ammonium Thiocyanate Solution —0.1 N

A.3.1.9 Ferric Ammonium Sulphate Indicator Solution — Two percent (m/m)

A.3.1.10 Nitrobenzene

A.3.2 Procedure

A.3.2.1 Weigh accurately a quantity of the material containing about 0.15 g of deltamethrin and transfer it to a 250-ml conical flask with standard joint. Add 5 ml of benzene and then 30 ml of iso-propanol, shake to mix. Add 2.5 g of freshly cut sodium metal and reflux it on a very low flame cautiously, using a wire gauze for about 2 hours. Then destroy the excess sodium metal by carefully adding 50 percent aqueous iso-propanol solution through the condenser at the rate of one or two drops per second. Boil again for 10 minutes. Stop heating, co01 to room temperature. Add 20 ml water and shake. Then add 4 ml of formaldehyde, shake and keep for 10 minutes.

A.3.2.2 Add 1 nor 2 drops of phenolphthalein. Acidify the solution with 50 percent nitric acid till the reversal of phenolphthalein indicator. Shake well.

A.3.2.3 Add 25 ml of silver nitrate, 2 ml of nitrobenzene and 5 ml of ferric ammonium sulphate indicator. Shake well.

A.3.2.4 Back titrate the excess of silver nitrate with ammonium thiocyanate solution to brick red colour.

A.3.2.5 Carry out a blank titration by exactly proceeding according to A.3.2.1 to A.3.2.4.

A.3.3 Calculation

Deltamethrin content, percent by mass = $\frac{V \text{ x } 0.025 \text{ x } 25 \text{ xN x } 100}{M \text{ x } 0.1}$

Where

V= volume, in ml, of 0.1 N ammonium thiocyanate consumed by deltamethrin;

N= normality of ammonium thiocyanate solution; and

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

Annex B
(normative)

Determination of optical rotation

B.1 APPARATUS

B.1.1 A polarimeter on which angular rotation accurate to 0.05º can be read.

B.1.2 D-Line — Appropriate arrangement to obtain D-line of sodium or 546-l nm line of mercury spectrum.

B.1.3 Polarimeter tubes— Suitable polarimeter tube of 1 decimetre length

B.2 PROCEDURE

B.2.1 Sample Preparation— Weigh accurately 1 g of deltamethrin and dissolve in benzene solvent. Make to 100 ml with benzene. Maintain the temperature of this solution at 20°C.

B.2.2 Pour benzene solvent maintained at 20°C into the dry polarimeter tube and close the end caps tightly so as to ensure that there are no air bubbles. Measure the optical rotation. This is the blank reading.

B.2.3 Pour the sample obtained in B.2.1 into a dry polarimeter tube and close the end caps-tightly, so as to ensure that there are no air bubbles. Measure the optical rotation. This is the sample reading.

B.3 CALCULATION

Specific rotation= $\frac{a \times 100}{l \times c}$

Where

a = corrected observed rotation in degrees;

l = length of polarimeter tube in decimetre; and

c= concentration of solution expressed in g of the substance in 100 ml solution.

Annex C
(normative)

Determination of acid chloride corresponding to deltamethrin

C.1 Reagents

C.1.1 Methanolic potassium hydroxide solution — 0.02N

C.1.2 Neutralized Methanol — To 100 ml of methanol, add 3 drops of a 1 percent solution of bromophenol blue in methanol. If necessary, add 0.1 N solution of hydrochloric acid in methanol (obtained by diluting 8 ml of 12 N hydrochloric acid to 100 ml with methanol) until yellow colour develops. Add 0.02 methanolic potassium hydroxide until a purple blue colour develops.

C.2 Procedure

Transfer a sample of about 2g of product weighed to the nearest 0.1 mg into a 200-ml Erlenmeyer flask. Add 100 ml of neutralized methanol. Warm to dissolve and let cool to room temperature. Allow to stand for 5 min and titrate with the standard 0.02N methanolic potassium hydroxide solution to purple blue end-point.

C.3 Calculation

Milliequivalent for 1 g corresponding to the acid chloride **B** = $\frac{t \times N}{m}$

Where

t= volume, in ml, of standard methanolic potassium hydroxide used;

N= normality of the methanolic potassium hydroxide; and

m= mass, in g, of the sample taken for the test.

Acid chloride content, percent by mass = $\frac{B \times 316.45 \times 100}{1000}$ = B x 31.645

Annex D
(normative)

Determination of content of acid + anhydride corresponding to deltamethrin

D.1 Determination of acid

D.1.1 Reagents

D.1.1.1 Ethanolic Sodium Hydroxide Solution— 0.02

D.1.1.2 Neutralized Ethanol— To 100 ml of ethanol, add 10 drops of a 1 percent solution of alphanaphtholbenzene in ethanol. Add 0.02 N ethanolic sodium hydroxide until a true green colour develops.

D.1.2 Procedure

Transfer a sample of about 2g of product weighed to the nearest 0.1 mg into a 200-ml Erlenmeyer flask. Add 100 ml of neutralized ethanol. Warm to dissolve, cool in ice and titrate immediately the ice-cooled solution with the standard 0.02 N ethanolic sodium hydroxide solution to a true green end-point.

D.1.3 Calculation

Milliequivalent for 1g corresponding to acid + acid chloride C= $\frac{t1 \times N}{m1}$

Where

t_1= volume, in ml, of standard ethanolic sodium hydroxide used;

N= normality of the ethanolic sodium hydroxide; and

m_1= mass, in g, of the sample taken for the test.

Acid content, percent by mass = $\frac{(C-B) \times 297.95 \times 100}{1000}$ = (C – B) x 29.795

D.2 Determination of anhydride

D.2.1 Reagents

D.2.1.1 Aniline in Cyclohexane— 0.1 N

D.2.1.2 Perchloric Acid Solution in Glacial Acetic Acid — 0.1 N.

Mix 70 to 72 percent perchloric acid carefully with glacial anhydrous acetic acid (about 500 ml) and pure acetic anhydride (50 ml) and make up to one litre with acetic acid. The flask shall be well cooled while the reagents are being mixed.

D.2.1.3 Standardization— Weigh accurately about 0.02 g of anhydrous sodium carbonate and dissolve it in acetic acid (30 ml). Add 1-naphtholbenzene indicator 1 percent solution in benzene (2 drops) and titrate with perchloric acid until the colour of the solution changes from yellow orange to dark green.

Normality (N)= $\frac{1000\ m}{52.994\ t} = \frac{18.87\ m}{t}$

Where

m= mass of anhydrous sodium carbonate; and

t= volume of perchloric acid used

NOTE: When titrating with perchloric acid in glacial acetic acid, the following precautions shall be observed:

a) There should be complete absence of water. If water is present, no satisfactory end point will be obtained.

b) Titrations and standardizations should be carried out at the same temperature because acetic acid has a large coefficient of thermal expansion.

D.2.2 Procedure

Transfer a sample of about 1 g of product weighed to the nearest 0.1 mg into a 200-ml Erlenmeyer flask. Add 10 ml accurately measured 0.01 N aniline solution in cyclohexane and 10 ml of glacial acetic acid. Stopper the flask, mix and allow to stand for 1 hour at 20-25°C. Titrate with 0.01 N perchloric acid in the presence of 1 percent solution of crystal violet in acetic acid until the initially purple colour turns emerald green.

D.2.2.1 Run a blank test in the same conditions but omitting the sample.

D.2.3 Calculation

Milliequivalent for 1 g corresponding to anhydride and two times acid chloride D= $\frac{(T-t2)\ \mathrm{x\ N}}{m2}$

Where

T= volume, in ml, of 0.01 N perchloric acid used in the titration for the sample;

t_2= volume, in ml, of 0.01 N perchloric acid used for the blank;

N= normality of the perchloric acid used; and

m_2= mass, in g, of the sample taken for the test.

Anhydride content percent by mass= $\frac{(D-2B)\ x\ 577.9x\ 100)}{1000}$ = (D – 2B) x 57.79

x

Bibliography

[1] FAO Specifications and evaluations for agricultural pesticides *for Deltamethrin*

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