RWANDA STANDARD

DRS 590

First edition

2024-mm-dd

Pesticides — Determination of total cypermethrin content

ICS 65.100.10

Reference number

DRS 590: 2024

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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 590 was prepared by Technical Committee RSB/TC 64, *Pesticides.*

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

ES 722: Pesticides — Determination of total cypermethrin content

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

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.

Pesticides — Determination of total cypermethrin content

1 Scope

This Draft Rwanda Standard gives the method for the determination of total cypermethrin content in the technical product by Gas chromatographic method. It is also applicable for formulated products.

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 591, *Pesticides — Determination of total cypermethrin contend and diastero isomer ration*

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for determination of repeatability and reproducibility of a standard measurement method.*

3 Terms and definitions

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

4 Outline of the method

The sample is dissolved in 4-methylpentan-2-one containing di-(2-ethylhexyl) phthalate as internal standard. Separation is carried out on a column of chromosorb W-HP coated with silicone OV 101. The cypermethrin, isolated as one peak, is determined by comparison with calibration solutions.

5 Reagents

Unless otherwise specified use the following reagents of recognized analytical grade.

5.1 4-methylpentan-2-one (methyl isobutyl ketone); MIBK

5.2 di-(2-2thylhexyl) phthalate (DEHP) internal standard

5.3 DEHP solution

Dissolve DEHP (10 g) in MIBK (500 ml). Ensure a sufficient quantity of this solution is prepared for all samples and calibration standards being analysed (solution L).

5.4 Cypermethrin working standard

Known cypermethrin content (minimum 900 g/kg) with a ratio of cis to trans isomer content similar to that of the sample being analysed. Store the standard in a cool dry place, preferably in a discator. The isomers may crystallize out of the mixture at ambient temperature and the analytical standard shall be homogeneous before use.

5.5 Calibration solution

Warm the sealed bottle of cypermethrin standard (purity p g/kg) at between 40ºC and 50 ºC until no crystals remain; and then shake the bottle. Weigh in duplicate (to the nearest 0.1 mg) approximately 0.2 g of standard M_A and M_B, g). Transfer to 50 ml volumetric flasks and dissolve in a few mls of MIBK. Add by pipette 10.0 ml of internal standard solution to each flask and dilute to 50 ml with MIBK (solutions C_A and C_B). Prepare a solution without internal standard by dissolving 0.1 of cypermethrin standard in 25 ml of MIBK (solution C_D).

6 Apparatus

6.1 Gas chromatography

Capable of operating over the range 100 to 300 ºC with a flame ionization detector.

6.2 Column

1 m X 4 mm i.d. glass column packed with 3% silicone OV 101 on chromosorb W-HP 100 to 120 mesh (80 to 100 mesh has also given satisfactory results). Before use, condition a freshly prepared column by purging with nitrogen overnight at 260ºC. during this operation the column shall nog be connected to the detector.

6.3 Electronic integrator

Compatible with the gas chromatograph

6.4 25 and 50 ml volumetric flasks.

7 Sampling

7.1 Sample shall be taken in accordance with RS 405.

7.2 Make sure that the bulk material shall be homogenized by heating to about (40 – 50) ºC before taking the sample.

7.3 Take at least 25 g and re-homogenize before taking a sub-sample for analysis of technical product.

7.4 Take at least 500 ml of 500 g for formulated products.

8 Procedure

8.1 Identity test

8.1.1 Principle

Suitable techniques for separating a quantity of the active ingredient for spectroscopic examination are HPLC, TLC and GLC. The chosen technique may be used either (i) preparatively, when the isolated material is examined subsequently, or (ii) directly, when the material is transferred immediately on elution, for example as in GLC linked to mass spectroscopy. The comparisons should reveal no significant difference between sample and standard. Use at least two of the following techniques, one of which should be spectroscopic.

8.1.2 Techniques

8.1.2.1 Chromatographic techniques: Compare retention times.

8.1.2.1.1 GLC method

8.1.2.1.2 HPLC, determine as for CD xxx (ref. ES 723)

8.1.2.2 Spectroscopic method: Compare whole spectra of separated material and of standard.

8.1.2.2.1 IR

Warm the sample to between 40 – 50 ºC until no crystals remain, then shake to ensure homogeneity. Smear a portion of the material between two KBr discs and scan between 700 and 3500 cm^{-1}.

8.1.2.2.2 MS

The sample should be introduced via the direct insertion probe, or via the direct insertion probe, or via a gas chromatography link, into a spectrometer operating by electron impact with an ionizing energy of about 70 eV. In typical circumstances the eight most significant peaks above m/z 100 in each compound are as follows;

m/z	*127*	167	165	181	208	209	*245*	*417*
Relative abundance	19	100	65	51	26	24	5	3

Particular attention should be paid to the molecular ion peaks (italic).

8.2 Determination of cypermethrin content

8.2.1 Preparation of the sample solutions

Homogenize the material by the method given for the standard. Prepare a solution without internal standard by dissolving sufficient sample to contain about 0.1 g ok cypermethrin in 25 ml of MIBK (solution S_0). Weigh in duplicate (to the nearest 0.1 mg) into 50 ml volumetric flasks sufficient sample (W_A and W_B, g) to contain

approximately 0.2 g of cypermethrin. Add by pipette 10.0 ml of DEHP internal standard solution to each flask and then dilute to volume with MIBK (solution S_A and S_B);

NOTE Cypermethrin content in wettable powders shall be determined in accordance with clause 8, except substitute the following for caluse 801: (Preparation of sample):

Weigh (to the nearest mg) in duplicate sufficient sample (wg) to contain about 0.2 g of cypermethrin in to 50 ml conical flasks. Add each, 10.0 ml of DEHP solution l from a pipette and shake the flasks thoroughly for 10 min. to dissolve the cypermethrin. Dilute to 50 ml with MIBK. Allow the insoluble material to settle (if external calibration is being used, the sample solution should not be filtered) and filter the supernatant liquid through a whatman No 54 or equivalent filter paper and use the filtrate for the analysis (solutions S_A and S_B). prepare a solution without internal standard by shaking a similar amount of sample with MIBK (50 ml; solution S_0).

8.2.2 Gas chromatographic conditions

8.2.2.1 Column

Material	**Glass**
Length X i.d.	1.0 m X 4 mm
Stationary phase/recommended solvent	OV 101/dichloromethane
Solid support	Chromosorb W-HP 100 to 120 mesh (125 to 150 µm)
Mass ratio: stationary phase/support	3/97
Typical packing density	0.29 g/ml
Typical column efficiency (number of theoretical plates determined for DEHP peak)	Using DEHP peak

8.2.2.2 Detector system

Type	**FID**
Sensitivity	No special requirements

8.2.2.3 Temperature

Column	**Use a set temperature of 235 ºC**
Injection port	250 ºC

Detector	250 ºC

8.2.2.4 Carrier gas	Nitrogen (oxygen-free, i.e. contains less than 10 ppm) 0.05 l/m
8.2.2.5 Calibration	Internal. Response factor, peak area measurement
8.2.2.5 Quantity to be injected	1.5 µl of a solution containing 4 mg cypermethrin/ml MIBK. "On-column" injection, i.e. on to or just into the support material, is essential to ensure satisfactory chromatography. Cypermethrin is thermally labile, and on-column decomposition can occur from active sites on silica, or glass wool, column glass or support material. If a glass-wool or silica plug is used, it is recommended that the syringe needle should penetrate through the plug into the column packing material.
8.2.2.5 Retention time (typical)	DEHP: 5.4 min Cypermehrin: 11.4 min

8.2.3 Equilibration of the system

Using the conditions described in clause 8.2, inject 1.5 µl portions of solutions I, C_0 and S_0 and check whether there are any interfering peaks from impurities. If there are, make any necessary corrections or alternatively, use external calibration. Inject standard solutions C_A and C_B to set the integrator parameters. Calculate response factors for these injections to check stability of the instrument. Response factors should not differ by more than ± 0.5% of the mean.

8.2.4 Calculation

8.2.4.1 Calculate the relative response factors (f_1, f_2, etc.) for the pair of calibration injections which bracket the sample injections, e.g. use C_{A1} and C_{B1} for sample injections SA_1 and SA_2, and obtain the mean response factor "f" using the following equation:

$$Relative\ sponsefactor\ fl\ (etc.) = \frac{Hs}{IrMP}$$

Where;

H_s is area of cypermethrin peak of the calibration solution;

I_r is area of DEHP peak of the calibration solution;

M is mass of cypermethrin analytical standard in calibration solution (g); and

P is the purity of the cypermethrin standard (g/kg)

The mass of internal standard is common to both calibrated and sample solutions and has therefore been omitted successive measurements of the response factors should agree to within ± 0.5% of their mean value, if not, repeat the analysis. For each sample injection, e.g. SA_1, calculate the cypermethrin content using the following equation:

$$Cypermethrin\ content = \frac{Hw}{fIqW}\ g/kg$$

Where;

f is mean relative response factor;

H_w is area for the cypermethrin peak in sample solution;

I_q is area of DEHP in sample solution; and

W is mass of sample (g).

Calculate the cypermethrin content of the sample as the mean of the four determination as follows:

Sample injection	Use relative response factor from	Cypermethrin	
SA_1	C_{A1} and C_{B1}	Q%	} X%
SA_2	C_{A1} and C_{B1}	R%	
SB_1	C_{A2} and C_{B2}	S%	} Y%
SB_2	C_{A2} and C_{B2}	T%	

Where;

X is mean of Q and R; and

Y is mean of S and T

Take the mean of the two values X and Y as the cypermethrin content.

8.2.5 Repeatability and reproducibility of methods

8.2.5.1 Technical product

Repeatability r = 2.5 at 90 – 98% active ingredient (ISO 5725-2)

1.7 at 72% active ingredients (ISO 5725-2)

Reproducibility R = 4.3 at 90 – 98% active ingredient (ISO 5725-2)

2.7 at 72% active ingredients (ISO 5725-2)

8.2.5.2 Other products

	ISO 5725-2	GLC	HPLC
Wettable powders	Repeatability “r”	0.8 at 13% active ingredient	0.5 at 13% active ingredient
	Reproducibility “R”	0.8 at 13% active ingredient	0.8 at 13% active ingredient
Emulsifiable concentrates	Repeatability “r”	0.5 at 20-40% active ingredient 0.2 at 5% active ingredient	1.1 at 20-40% active ingredient 0.3 at 5% active ingredient
	Reproducibility “R”	1.0 at 20-40% active ingredient 0.3 at 5% active ingredient	2.3 at 20-40% active ingredient 0.4 at 5% active ingredient
Ultra low volume liquids	Repeatability “r”	0.3 at 5% active ingredient	0.3 at 5% active ingredient
	Reproducibility “R”	0.3 at 5% active ingredient	0.6 at 5% active ingredient

9 Test report

The test report shall include the following information;

a) The result and method of expression used;

b) All information necessary for complete identification of sample;

c) A reference number of this standard;

d) Any operation included in this standard; and

e) The date of test

Annex A
(normative)

Annex title

A.1 General

Bibliography

[1] ISO/IEC Directives, Part 2, *Rules for the structure and drafting of International Standards*, 2016

[2] ISO/IEC TR 10000-1, *Information technology — Framework and taxonomy of International Standardized Profiles — Part 1: General principles and documentation framework*

DRS 590: 2024

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