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Pyrethrum-based insecticides — Specification —

Part 3:

Emulsion (Oil in water, EW)

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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 465-3 was prepared by Technical Committee RSB/TC 007, *Agrochemicals.*

DRS 465 consists of the following parts, under the general title *Pyrethrums-based insecticides — Specification*:

— *Part 1: Dusting powders (DP)*

— *Part 2: Grease (GS)*

— *Part 3: Emulsions (Oil in water, EW)*

Committee membership

The following organizations were represented on the Technical Committee on *Agrochemicals* (RSB/TC 007) in the preparation of this standard.

University of Rwanda/College of Sciences and Technology (UR/CST)

University of Rwanda/College of Education (UR/CE)

Rwanda Forensic Laboratory (RFL)

Ministry of Environment (MoE)

Standards for Sustainability (SfS)

AGROPY Ltd

Rwanda Inspectorate, Competition and Consumer Protection Authority (RICA)

Rwanda Agriculture and Animal Resources Development Board (RAB)

Rwanda Standards Board (RSB) – Secretariat

Introduction

Pyrethrum owes its insecticidal properties to esters which are reportedly produced by a number of different cell types (oil glands, resin ducts and mesophyll cells). Pyrethrin I, jasmolin I and cinerin I are esters of chrysanthemic acid (chrysanthemum monocarboxylic acid), while pyrethrin II, jasmolin II and cinerin II are esters of pyrethric acid (monomethyl ester of chrysanthemum dicarboxylic acid). The biosynthesis of pyrethrin I in seedlings of C. cinerariifolium has been studied using [1-C]-d-glucose as a precursor; the acid portion of the molecule is derived from d-glucose and the alcohol moiety possibly from linoleic acid.

Pyrethrum-based insecticides — Specification — Part 3: Emulsions (oil in water, EW)

1 Scope

This Draft Rwanda Standard prescribes the requirements, sampling and test methods for pyrethrum-based emulsion (oil in water, EW) insecticides for direct application or after dilution, used in animal and crop protection to control insect pests.

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*

RS 191, *Refined pyrethrum concentrate — Specification*

RS 405, *Pesticides — Sampling*

3 Terms and definitions

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

3.1

pyrethrum

genus of several Old World plants now classified as *Chrysanthemum* or *Tanacetum* (e.g., *C. coccineum*) which are cultivated as ornamentals for their showy flowers heads

3.2

pyrethrum concentrate

extract of the flowers of the plant, *Chrysanthemum cinerariaefolium*

3.3

pyrethrins

the six naturally occurring isomers that are esters of pyrethric acid and chrysenthemic acid viz: pyrethrin-I, pyrethrin-II, cinerin-I, cinerin-II, jasmolin-I, jasmolin-II; having insecticidal property and are extracted from the flower of Chrysanthemum cinerariaefolium.

4 Requirements

4.1 General requirements

4.1.1 The product shall consist of pyrethrum extracts, complying with the requirements of RS 191, in an aqueous phase together with suitable formulants.

4.1.2 **If** synthetic pyrethroids are added, the product shall be approved by the Competent Authority.

4.1.3 After agitation, the product shall be homogeneous liquid and suitable for dilution in water, free from sediment and/or suspended matter.

4.2 Specific requirements

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

Table 1 — Specific requirements for pyrethrum-based emulsion insecticides

S/N	Parameters		Requirements	Test methods
i.	Total pyrethrins content, % by mass	Ready to use	0.10 — 0.50	Annex A
		Dilutable	0.50 — 5.0	
ii.	Pourability % m/v, max.		0.5	Annex B
iii.	Emulsion stability and re-emulsification, at 30 ± 2°C.		Pass the test	Annex C
iv.	Persistent foam, after 1minute, volume, max.		Nil	Annex D
v.	Flash point, °C, min.		32	Annex E
vi.	Acidity (as H_2SO_4), g/kg, max.		3.0	Annex F
vii.	Alkalinity (as NaOH), g/kg, max.		0.10	
viii.	pH range		5 — 7	Annex G
ix.	Storage stability	At 0 ± 2°C for 7 days, ml, max.	Pass to iii)	Annex H
		At 54 ± 2°C for 14 days, percentage of declared total pyrethrins (% m/m), min.	98	Annex I

5 Packaging and labelling

5.1 Packaging

5.1.1 The product shall be packaged in a well closed container that will preserve its original characteristics.

5.1.2 The packaging material shall protect the contents from adventitious contamination under handling and storage conditions.

5.2 Labelling

The containers shall be closed and shall bear legibly and indelibly the following information in any of the three languages officially accepted in the Republic of Rwanda namely: Kinyarwanda, English and French.

a) Name of the product;

b) Name and address of the manufacturer;

c) Manufacture and expiry dates;

d) Batch number;

e) Active ingredient (s) contents;

f) Indicate: “ready to use” or “dilutable”

g) Storage conditions;

h) Instructions for use;

i) Precautions and warnings; and

j) Country of origin.

6 Sampling

Representative samples of the product shall be drawn as prescribed in RS 405.

Annex A
(normative)

Determination of total pyrethrins

A.1 General

The active ingredients in pyrethrum extract may be determined using a HPLC system first by injecting a solution of the analyte into the chromatograph, followed by the separation and comparison of peaks areas of the analytes in the sample with that of an external standard containing a known amount of the analytes. The peaks are eluted in the following order: Cinerin II, Pyrethrin II, Jasmolin II (total Pyrethrins II) and Cinerin I, Pyrethrin I, Jasmolin I (total Pyrethrins I).

A.2 Reagents

World pyrethrum standard, 50%

Acetonitrile, HPLC grade

Water, HPLC grade

A.3 Apparatus

A liquid chromatography System equipped with an auto-sampler, a Variable Wavelength Detector (or equivalent) and a Column {Phenomenex, 250 x 4.6 mm Luna Phenyl-Hexyl 5µ Reverse Phase (or equivalent)}.

A.4 Operating conditions

Flow rate:	1.5 ml/min
Composition:	40:60 (%, v/v water/acetonitrile
Elution:	isocratic
Column temperature:	40 °C
Wavelength:	240 nm
Injection volume:	15 µl
Stop time:	22 min
Post time:	1 min

A.5 Preparation of the standard

Weigh 20 mg of the pyrethrum standard to the nearest 0.0001 g in a 100 mL volumetric flask and dilute to volume with Acetonitrile and label it. Transfer a small portion to a sample vial and label it accordingly.

A.6 Sample preparation

In a 100 ml volumetric flask, weigh 20 mg to the nearest 0.0001 g of the sample to be analysed and dilute to volume with Acetonitrile. Sample this solution using a vial and label it accordingly.

A.7 Procedure

After the chromatograph is stable, make a minimum of three injections for the standard solution as well as for the analyte and average the area counts. The relative Standard Deviation between injections should be within 2 %.

A.8 Calculation

The total pyrethrins is calculated as follow:

$$\text{total pyrethrins}, \%\ \text{m/m} = \frac{Average\ sample\ area\ X\ weight\ of\ standard * Purity\ of\ the\ standard\ (in\ \%)}{Average\ standard\ area * Weight\ of\ sample}$$

Annex B
(normative)

Determination of pourability

B.1 Outline of the method

The product is allowed to stand for a definite time and the amount remaining in the container after a standardized pouring procedure is determined. The container is rinsed and the amount then remaining is determined.

B.2 Apparatus

Container – A 500-ml stoppered measuring cylinder.

Volume equivalent to 1 subdivision of the scale = 5 ml.

Capacity corresponding to lowest graduation mark = 50 ml

Capacity corresponding to highest graduation mark = 50 ml

Length of scale = 250 ml

Overall height = 39 cm

Diameter of base = 10 cm

Stopper = B34

NOTE High density polyethylene bottles, 100 ml volume and kilner jars, 700ml volume, can be used but this must be recorded with the result.

B.3 Procedure

Weigh the empty container and stopper (w_0 g) and add enough of the sample taken from a recently mixed bulk sample to leave approximately 20% of the volume of the container as ullage. Replace the stopper and reweigh the container (w_1 g). allow the container to stand undisturbed for 24 hours and then pour out the product (Note 1) for 60 s at an angle of 45 °C and then finally invert the container for 60 s (Note 2). Reweigh the container and stopper (w_2 g).

Add distilled water at 20 °C (a volume of 80% of that of the container) and replace the stopper. Invert the container 10 times (note 3) and empty the container as before and reweigh the container and stopper (w_3 g). Calculate the residue (R) and the rinsed residue (*R'*).

$$R\ (\%) = \frac{(w2 - w0)}{(w1 - w0)} X\ 100$$

$$R'\,(\%) = \frac{(w3 - w0)}{(w1 - w0)} X\ 100$$

NOTE 1 The length of the standing period and the temperature should be agreed previously.

NOTE 2 A square-sided container should be held so that a flat side is underneath.

NOTE 3 The term 'invert the container' means that the container's vertical axis is turned through 180 degrees and then brought back to its original position, the whole operation taking about 2 s.

Annex C
(normative)

Determination of characteristics of emulsion (EW) insecticides

C.1 Outline of 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.

C.2 Methods of determination

C.2.1 Five percent v/v oil phase

C.2.1.1 Hand shaking

C.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.

C.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.

C.2.1.2 Mechanical shaking

C.2.1.2.1 Apparatus

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

C.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.

C.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.

C.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**.**

C.2.2.2 Dispersion stability

C.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

C.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).

C.2.2.2.3 Dispersion stability

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

C.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 D
(normative)

Determination of persistent foaming

D.1 Apparatus

D.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.

D.1.2 **Weighing bottle**.

D.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.

D.1.4 **Stopwatch**.

D.2 Procedure

D.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.

D.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.

D.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 E
(normative)

Determination of flash point

E.1 General

This test method is similar to the Continuously Closed Cup Flash Point Test method. It utilizes a closed but unsealed cup with air injected into the test chamber.

E.2 Apparatus

The Continuously Closed Cup Flash Point tester shall essentially consist of:

E.2.1 **A lid of solid brass**: The temperature of which is controlled electrically. Two temperature sensors for the specimen and the lid temperatures, respectively, two electrically insulated pins for a high voltage arc, and a connecting tube for the pressure monitoring.

E.2.2 **Sample cup**: Made of nickel-plated aluminium with an overall volume of 4 mL and capable of containing 1 ± 0.1 mL of sample.

E.2.3 **Test Chamber**: Formed by sample cup and the temperature controlled lid and shall have an overall volume of 4 ± 0.2 mL

E.2.4 **Specimen Temperature Sensor**: A thermocouple (Ni-Cr-Ni or similar) in stainless steel of 1 mm diameter with a response time of t(90) = 3 sec. It shall be immersed to a depth of at least 2 mm into the specimen. It shall have a resolution of 0.1 °C and a minimum accuracy of ± 0.2 °C, with a digital readout.

E.2.5 **Magnetic Stirring**: A rotating magnet outside the sample cup that can drive a small stirring magnet, which is inserted into the sample cup after sample introduction. It should have a diameter of 3 ± 0.2 mm and a length of 12 ± 1 mm. The rotation speed of the driving magnet shall be between 250 and 270 revolution/minute.

E.2.6 **Air Introduction Provision**: For introduction of 1.5 ± 0.5 mL of air immediately after each test. The air shall be introduced by a short air pulse from a small membrane compressor by means of a T-inlet in the connecting tube to the pressure transducer.

E.2.7 **Arrangement Electrical heating and thermoelectric cooling of the lid**: used to regulate the temperature of the test chamber during the test. The temperature regulation shall have a minimum accuracy of ± 0.2 °C.

E.2.8 **A high voltage electric arc**: Used for the ignition of the flammable vapour. The energy of the arc should be 3± 0.5 mJ per arc and it shall be applied within 43 ± 3 ms.

E.2.9 **The pressure transducer** (for flash point detection): connected to the connecting tube in the lid; should have a minimum operational range from 80 to 177 kPa with a minimum resolution of 0.1 kPa and a

minimum accuracy of ± 0.5 kPa. It shall be capable of detecting an instantaneous pressure increase above barometric pressure of a minimum of 20 kPa within 100 ms.

E.3 Reagents

The reagents should be of analytical quality and of sufficient purity to guarantee the accuracy of the test results.

Anisole

Dodecane

Acetone (or toluene)

E.4 Procedure

E.4.1 Thoroughly clean and dry the lid together with the arc pins and the sample cup before starting the test. Remove any solvent used to clean the apparatus. If the expected flash point of a sample is more than 15 °C higher than the flash point of the previous sample, heat the lid together with an empty, dry sample cup to a temperature 30 °C higher than the expected flash point of the new sample.

E.4.2 Set the initial temperature to at least 18 °C below the expected flash point. Set the final temperature to a value beyond the expected flash point.

E.4.3 Set the pressure threshold for the flash detection to 20 kPa.

E.4.4 Initiate the test procedure to regulate the lid to the initial temperature. When the initial temperature is reached as indicated by the instrument, prepare to introduce 1± 0.1 mL specimen of the sample.

E.4.5 Ensure that the sealed sample and the sample cup are at least 18 °C below the expected flash point temperature, cool if necessary. Shake the sample thoroughly before opening the sample container. Extract 1 mL of sample with a pipette or syringe, and close the container. Transfer 1± 0.1 mL of the sample to be tested into the sample cup.

E.4.6 Insert a stirring magnet into the sample cup to ensure a consistent sample mixing. Put the sample cup onto the sample cup support of the tester, and start the procedure. Raise and press the sample cup onto the lid, which is at the initial temperature and thus at a higher temperature than the sample cup.

E.4.7 While the temperature of the sample cup and the lid are equalizing, apply a precautionary arc at 10 °C intervals. If a flash is detected at one of these precautionary arcs, discontinue the test, and discard the result. Repeat the test with a fresh specimen and with a lower initial temperature of at least 18 °C below the temperature at which the flash was detected.

E.4.8 After the temperature between the temperatures regulated lid and the specimen have equalized to within 1 °C, start the actual test for the flash point. Heat the lid with the programmed heating rate, and apply the arc ignition in equidistant temperature steps of 1 °C. Monitor the instantaneous pressure increase within 100 ms after the arc. Stop the test when a flash is detected or when the final temperature is reached in the case of no flash point. The flash point temperature is the specimen temperature at which the instantaneous pressure increase, due to the presence of the flash, exceeds 20 kPa.

E.4.9 When a flash is detected at a temperature that is higher than 26 °C above the initial temperature, or when a flash is detected at a temperature that is less than 10 °C above the initial temperature, consider the result approximate and repeat the test with a fresh test specimen. Adjust the expected flash point for this next test to the temperature of the approximate result. The initial temperature for this fresh test specimen shall be 18 °C below the temperature of the previous approximate result.

E.4.10 Record the specimen temperature reading at the detected flash point as the uncorrected flash point temperature. If no flash point was detected within the tested temperature range, record "*flash point is higher than the final temperature".*

E.4.11 At the conclusion of the test, cool the sample cup below 50 °C to withdraw it safely.

E.5 Results

E.5.1 Observe and record the ambient barometric pressure at the time of the test; when the pressure differs from 101.3 kPa, correct the flash point as follows:

Corrected flash point = C + 0.25 (101.3 – p)

Where,

C = observed flash point in °C, and

p = ambient barometric pressure in kPa.

Round up the corrected value to the nearest 0.5 °C.

E.5.2 The barometric pressure used in this calculation must be the absolute ambient pressure for the laboratory at the time of the test. Aneroid barometers, such as those used at weather stations and airports, are corrected to give sea level readings; these shall not be used.

Annex F
(normative)

Determination of acidity or alkalinity

F.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.

F.2 Determination of acidity

F.2.1 Reagents

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

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

F.2.1.3 Standard sodium hydroxide solution – 0.05N

F.2.1.4 Standard hydrochloric acid – 0.05N

F.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.

F.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.

F.3 Determination of alkalinity

F.3.1 Reagents

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

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

F.3.1.3 Standard hydrochloric acid – 0.05N

F.3.1.4 Standard sodium hydroxide solution – 0.05N

F.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.

F.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 G
(normative)

Determination of pH value

G.1 Outline of the method

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

G.2 Reagents

G.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.

G.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.

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

G.3 Apparatus

G.3.1 pH meter

G.3.2 Glass electrode and reference electrode

G.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 tetraborateTemperature, °C	10	15	20	25	30
pH	9.32	9.28	9.22	9.18	9.14

G.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 H
(normative)

Determination of stability of liquid formulations at 0 °C

H.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.

H.2 Emulsifiable concentrates and solutions

H.2.1 Apparatus

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

H.2.1.2 Cone shaped centrifuge tubes, 100 ml.

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

H.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.

H.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.

H.3 Aqueous solutions

H.3.1 Apparatus

H.3.1.1 Measuring cylinder, 100ml.

H.3.1.2 Refrigerator, at 0 ± 1 °C

H.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 I
(normative)

Determination of accelerated storage stability

I.1 Outline of the method

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

I.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.

I.3 Apparatus

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

I.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.

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

I.3.4 **Desiccator without desiccant**

I.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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