



DRAFT EAST AFRICAN STANDARD

Pyrethrum extracts — Specification

EAST AFRICAN COMMUNITY

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Contents

Page

Foreword	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Requirements	2
4.1 General requirements	2
4.2 Specific requirements	2
5 Packaging	2
6 Labelling	2
7. Sampling	3
Annex B (informative) Identity test for pyrethrins	4
B.1 Principle	4
B.2 Apparatus	4
B.3 Reagents	4
B.4 HPLC conditions	4
B.5 Standard preparation	4
B.6 Sample preparation	5
B.7 HPLC injection procedure	5
Annex B (normative) Determination of volatile levels	6
B.1 Apparatus	6
B.2 Procedure	6
B.3 Calculation	6
Annex C (normative) Determination of total pyrethrin content	7
C.1 Principle	7
C.2 Reagents	7
C.3 Procedure	8
C.4 Determination of Pyrethrin I	8
C.5 Calculation	8
C.5 Determination of Pyrethrin II	9
C.6 Calculation	9
Annex D (normative) Determination of pH	10
D.1 Apparatus	10
D.2 Reagents	10
D.3 Procedure	10
Annex E (normative) Determination of optical density	11
E.1 General	11
E.2 Apparatus	11
E.3 Reagents	11
E.4 Procedure	11
Annex F (normative) Sampling of pyrethrum extracts	12
F.1 General precautions	12
F.2 Sampling tube	12
F.3 Scaling of sampling	12
F.4 Test samples and reference samples	13
A.5 Number of tests	13

Bibliography 14

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Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in the East African Community. It is envisaged that through harmonized standardization, trade barriers that are encountered when goods and services are exchanged within the Community will be removed.

The Community has established an East African Standards Committee (EASC) mandated to develop and issue East African Standards (EAS). The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the public and private sector organizations in the community.

East African Standards are developed through Technical Committees that are representative of key stakeholders including government, academia, consumer groups, private sector and other interested parties. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the Principles and procedures for development of East African Standards.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

The committee responsible for this document is Technical Committee EASC/TC 069, *Organic and Inorganic chemicals*.

Attention is drawn to the possibility that some of the elements of this document may be subject of patent rights. EAC shall not be held responsible for identifying any or all such patent rights.

Pyrethrum extracts — Specification

1 Scope

This Draft East African Standard specifies requirements, sampling and test methods for pyrethrum extracts.

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.

ISO 760, *Determination of water — Karl Fischer method (General method)*

ISO 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity*

ISO 12185, *Crude petroleum, petroleum products and related products - Determination of density - Laboratory density meter with an oscillating U-tube sensor*

ISO 13736, *Determination of flash point – Abel closed – cup method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <http://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

Pyrethrum

aromatic plant of the daisy family, typically having feathery foliage whose brightly coloured aromatic flower heads contain pyrethrins as the active compounds

3.2

Pyrethrum extracts

product in the form of liquid prepared by extracting pyrethrum (*Chrysanthemum cinerariifolium*) flowers

3.3

Pyrethrin

Six naturally occurring isomers found in the flower of *Chrysanthemum cinerariifolium* as esters of pyrethric acid (pyrethrin-II, cinerin-II, and jasmolin-II) and chrysanthemic acid (pyrethrin-I, cinerin-I and jasmolin-I) that have insecticidal property

4 Requirements

4.1 General requirements

4.1.1 The active ingredients in pyrethrum extract shall be composed of six esters, namely, pyrethrin I and II, cinerin I and II and jasmolin I and II when tested in accordance with Annex A.

4.1.2 Pyrethrum extract shall be an extract of pyrethrum flowers (*Chrysanthemum cinerariifolium*).

4.1.3 Pyrethrum extract shall be homogeneous viscous liquid.

4.1.4 Pyrethrum extract shall be free from sediments and suspended matter.

4.1.5 It shall be free from synthetic pyrethroids.

4.1.6 It shall be free from other synthetic pesticide residues.

4.1.7 Pyrethrum extract be greenish yellow to brown in colour and possess the characteristic odour of the commercial pyrethrum flowers.

4.2 Specific requirements

Pyrethrum extracts shall comply with the specific requirements given in Table 1 when tested in accordance with the test methods specified therein.

Table 1 — Specific requirements of pyrethrum extracts

S/N	Characteristics	Requirement	Test Method
i.	Flash Point, °C, min.	32	ISO 13736
ii.	Volatile level, %m/m, max	1.0	Annex B
iii.	Specific gravity at 15 °C.	0.91 – 0.98	ISO 12185
iv.	Total pyrethrin content, %, min.	25	Annex C
v.	pH, 1% solution.	5 – 7	Annex D
vi.	Moisture content, %m/m, max.	1.0	ISO 760
vii.	Viscosity, CPs, at 23 °C, min.	4 000	ISO 3104
viii.	Optical density, max.	0.9	Annex E

5 Packaging

Pyrethrum extract shall be packaged in suitable well-sealed containers that shall protect the contents and shall not cause any contamination or react with the product.

6 Labelling

Each package shall be legibly and indelibly labelled in English and/or any other official language (French, Kiswahili, etc) used in the importing East African Partner State with the following information:

- product name as "Pyrethrum extracts;
- name and physical address of manufacturer;
- registered trademark; if any,

- d) date of manufacture;
- e) batch number;
- f) net weight of the contents.
- g) country of origin;
- h) list of ingredients;
- i) date of expiry;
- j) instructions for use;
- k) storage and disposal instructions; and
- l) warnings/precautions

7. Sampling

Sampling shall be done in accordance with Annex F.

Annex A (informative)

Identity test for pyrethrins

A.1 Principle

HPLC method of analysis for pyrethrins.

A.2 Apparatus

A.2.1 High pressure liquid chromatograph, equipped with a C-8 reverse phase column (5 micron), 4.6 mm id. x 150 mm length or (equivalent column)

A.2.2 UV detector

A.3 Reagents

A.3.1 Acetone, HPLC Grade

A.3.2 Methanol, HPLC Grade

A.3.3 Water, HPLC Grade

A.4 HPLC conditions

A.4.1 Mobile phase, 85 % methanol/ 15 % water

A.4.2 Flow rate, 1.0 ml/min

A.4.3 Wavelength, 225 nm

A.5 Standard preparation

A.5.1 Weigh out the following standard amount into a 100-ml volumetric flask.

Active	MG. (actual)
Pyrethrum 20% standard	100 (20 mg PY's)

A.5.2 Add 5.0 ml acetone, mix and bring to volume with methanol.

A.5.3 A well-established standard quantitated using official AOAC Mercury reduction methodology should be used qualified standards solution may be supplied as needed by the manufacturers.

A.6 Sample preparation

A.6.1 Weigh out sample equivalent of 20 mgs of pyrethrins into a 100-ml volumetric flask.

A.6.2 Add 5.0 ml acetone, mix and dilute to 100 mL with methanol.

A.7 HPLC injection procedure

A.7.1 Inject 5 μ L of standard solution into HPLC instrument until acceptable reproducibility is obtained.

AA.7.2 Inject sample solution in the same manner. Use peak area or height for calculation. The pyrethrin II's are calculated using the sum of the two peaks at 2.9 min and 3.4 min. The pyrethrin I's are calculated using the sum of the two peaks at 4.8 min and 5.9 min.

Annex B (normative)

Determination of volatile levels

B.1 Apparatus

B.1.1 Petri dish

B.1.2 Analytical balance

B.1.3 Desiccator, containing an efficient desiccant, such as phosphorus pentoxide

B.1.4 Oven, preferably electrically heated, with calibrated temperature control device

B.2 Procedure

B.2.1 Weigh an empty petri dish and record the weight (W_1).

B.2.2 Weigh approximately 1g of sample into a petri dish which has been dried previously, cooled in the desiccator and record the weight of sample and petri dish (W_2). Place the dish in an oven for approximately two hours at $65^\circ\text{C} \pm 1^\circ\text{C}$.

B.2.3 Remove the dish from the oven, cool in the desiccator to room temperature and weigh the dried sample (W_3).

B.3 Calculation

The volatile level, expressed in percent by mass shall be calculated using the formula below:

$$\frac{W_2 - W_3}{W_2 - W_1} \times 100$$

where

W_2 mass, in grams, of the empty petri dish;

W_3 mass, in grams, of the petri dish and sample after cooling;

W_1 mass, in grams, of the petri dish and sample before drying

Annex C (normative)

Determination of total pyrethrin content

C.1 Principle

The pyrethrins after extraction are hydrolysed to chrysanthemum mono- and dicarboxylic acids. Monocarboxylic acid from 'Pyrethrin I, after extraction is reacted with Deniges reagent and mercurous sulphate formed is titrated with potassium iodate. Dicarboxylic acid from Pyrethrin II, remaining after the extraction of monocarboxylic acid is extracted and titrated with standard sodium hydroxide.

C.2 Reagents

C.2.1 Deniges reagents, mix 5 g yellow mercuric oxide with 40 ml water, and while stirring, slowly add 20 L sulphuric acid, then add additional 40 mL water and stir until all dissolves. Test for the absence of mercurous mercury by adding a few drops of iodine monochloride solution to 10 mL and titrating with potassium iodate standard solution as in B.3.2.

C.2.2 Iodine monochloride solution; Dissolve 10 g potassium iodide and .6.44 g potassium iodate in 75 mL water in glass stoppered bottle, add 75 mL hydrochloric acid and 5 mL chloroform and adjust to faint iodine colour (in chloroform) by adding dilute potassium iodide or potassium iodate solution. If much iodine is liberated, use stronger solution of potassium iodate than 0.01 M at first, making final adjustment with 0.01 M solution. Keep in dark and readjust when necessary. It should not be stored in refrigerator.

C.2.3 Potassium iodate standard solution, 0.01 M, Dissolve 2.14 g pure potassium iodate, previously dried at 105°C, in water and dilute to one litre. One ml of solution = 0.0057 g pyrethrin I.

C.2.4 Alcoholic sodium hydroxide solution, 1 N and 0.5 N

C.2.5 Petroleum ether, aromatic free, boiling range 40°C - 60°C or 60°C - 80°C

C.2.6 Ethyl ether, peroxide free

C.2.7 Sodium hydroxide standard solution, 0.02 N

C.2.8 Filter cell

C.2.9 Barium chloride solution, 10 % (m/v)

C.2.10 Dilute sulphuric acid, 1: 4 (v/v).

C.2.11 Ethyl alcohol, 95 % (v/v) and 99 % (v/v), anhydrous

C.2.12 Sodium chloride, solid as well as saturated solution

C.2.13 Chloroform

C.2.14 Dilute hydrochloric acid, 3: 2 (v/v)

C.2.15 Phenolphthalein indicator solution, 1 % (m/v) in ethyl alcohol

C.3 Procedure

C.3.1 Weigh sample containing 40 mg -150 mg total pyrethrins, add 50 ml petroleum ether and 1 g filter cell, and place in refrigerator at $0^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$ overnight. Filter through Gooch filter into 300-ml Erlenmeyer flask and wash with three 15 ml portions of cold petroleum ether, evaporate filtrate and washings on waterbath, using air current until the petroleum ether is almost entirely removed.

C.3.2 Add 20 ml 1 N alcoholic sodium hydroxide, or more, if necessary, to extract pyrethrins, connect to reflux condenser, and boil gently for 60 min to 90 min. Transfer to 600-ml beaker and add enough water to make aqueous layer 200 ml. If more than 20 ml alcoholic sodium hydroxide solution was used, add enough water so that all alcohol is removed when volume is reduced to 150 ml. Add few glass beads and boil aqueous layer down to 150 ml. Transfer to 500-ml separator and drain aqueous layer into 250-ml volumetric flask. Wash oil layer once with water and add washings to aqueous portion. If slight emulsion still persists after draining aqueous layer and washings, add two to three ml 10 percent barium chloride solution but do not shake vigorously after adding barium chloride, because reversed emulsion difficult to separate may form. To aqueous solution in 250- ml flask add 1 g filter cell and approximately 10 mL of the barium chloride solution, swirl gently and let stand for 30 min. Dilute to volume; mix thoroughly and filter off 200 ml. Test filtrate with barium chloride solution to see if enough has been added to obtain clear solution. Neutralize with sulphuric acid (1:4), using 1 drop phenolphthalein and add 1 ml excess. (If necessary to hold solution overnight at this point, leave in alkaline condition).

C.4 Determination of Pyrethrin I

C.4.1 Filter acid solution from C.3.2 through 7 cm paper, coated lightly with suspension of filter cell in water, on buchner, and wash with three 15 mL portions of water. Transfer to 500-mL glass stoppered separator and extract with two 50 mL portions petroleum ether. Shake each extract approximately for one minute, releasing pressure, if necessary, by inverting separator and carefully venting through stopcock. Let layers separate for approximately 5 min or until aqueous layer is clear before draining and re-extraction. Reserve aqueous layer for pyrethrin II determination. Do not combine petroleum ether extracts but wash each in sequence with same three 10 ml portions water, and filter petroleum ether extracts through small cotton plug, into a clean 250-mL separator. Wash separators and cotton in sequence with 5 ml petroleum ether.

C.4.2 Extract combined petroleum ether solutions with 5 ml 0.1 N sodium hydroxide, shaking vigorously for approximately one minute. Let layers separate for approximately 5 minutes before draining aqueous layer into 100-ml beaker. Wash petroleum ether with additional 5-ml portion 0.1 N sodium hydroxide and with 5 ml water, adding washings to beaker. Add 10 mL Deniges reagent and let stand in complete darkness for one hour at $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Add 20 ml alcohol and precipitate mercurous chloride with 3ml saturated sodium chloride solution. Warm to approximately 60°C and let stand several minutes until precipitate coagulates and settles. Filter through small paper, transferring all precipitate to paper, and wash with approximately 10 mL hot alcohol.

C.4.3 Wash with 2 or more 10 mL portions hot chloroform and place paper and contents in 250-ml glass stoppered conical flask. Add 50 ml cooled dilute hydrochloric acid (3: 2). Add 5 ml chloroform and 1 ml freshly adjusted iodine monochloride solution and titrate with standard potassium iodate solution, shaking vigorously approximately 30 s after each addition, until no iodine colour remains in chloroform layer. Take as end point when red colour disappears from solvent layer and does not return within 3 min. From standard potassium iodate solution used in titration and blank on Deniges reagent, calculate percent pyrethrin I.

C.5 Calculation

$$1\text{ml of } 0.01\text{M KIO}_3 = 0.0057 \text{ g pyrethrin I}$$

NOTE - *Chrysanthemum* monocarboxylic acid reacts with Deniges reagent to form series of colours beginning with phenolphthalein red, which gradually changes to purple, then to blue and finally to bluish green. Colour reaction is very distinct with 5 mg monocarboxylic acid and amounts as low as 1 mg can usually be detected. Therefore, no pyrethrin I should be reported if colour reaction is negative. With samples containing much perfume or other saponifiable ingredients, it may be necessary to use as much as 50 ml 1 N alcoholic sodium hydroxide. When lethanes are present, after washing

mercurous chloride precipitate with alcohol and chloroform, wash once more with alcohol and then several times with hot water

C.5 Determination of Pyrethrin II

C.5.1 If necessary, filter aqueous residue reserved in C.4 from petroleum ether extract through Gooch. Concentrate filtrate to approximately 50 ml and transfer to 500-ml glass stoppered separator. Wash beaker with three 15 ml portions water. Acidify with 10 ml hydrochloric acid and saturate with sodium chloride. (acidified aqueous layer shall contain visible sodium chloride crystals throughout following extractions.)

C.5.2 Extract with 50-ml ether, drain aqueous layer into second separator and extract again with 50 ml ether. Continue extracting and draining aqueous layer, using 35 ml for third and fourth extractions. Shake each extract for approximately one minute, releasing pressure, if necessary, by inverting separator and carefully venting through stopcock. Let layers separate for approximately 5 min or until aqueous layer is clear before subsequent draining and extraction.

C.5.3 Combine ether extracts, drain and wash with three 10 ml portions saturated sodium chloride solution. Filter ether extracts through cotton plug into 500-ml conical flask and wash separator and cotton with additional 10 ml ether. Evaporate ether on water bath and remove any fumes of hydrochloric acid with air current and continue heating for approximately 5 min. Dry for 10 min at 100 °C. Add 2 ml neutral alcohol and 20 ml water and heat to dissolve acid. Cool, filter through Gooch, if necessary, add two drops phenolphthalein and titrate with 0.02 N sodium hydroxide. Check normality of 0.02 N sodium hydroxide on the same day, as the sample is titrated.

C.6 Calculation

$$1 \text{ ml of } 0.02 \text{ N NaOH} = 0.00374 \text{ g pyrethrin II}$$

Total pyrethrin shall be obtained by adding pyrethrin I and pyrethrin II.

NOTE — Total pyrethrin should be in percentage (w/w)

Annex D **(normative)**

Determination of pH

D.1 Apparatus

A pH electrode system such as a single or dual glass electrode system conditioned and maintained according to the manufacturer's instructions.

D.2 Reagents

Distilled/deionized water

D.3 Procedure

D.3.1 Prepare 1 % solution of the extract by weighing 1.0 g of sample into a calibrated beaker. Add 50 ml distilled water or equivalent neutral organic solvent such as ethanol. Top up reagent water to 100 ml, mix vigorously using magnetic stirring rod until the formulation is completely mixed or dispersed.

D.3.2 Prior to pH measurement, calibrate the pH-meter using a standard buffer 4, 7 and 10 but ensure that the temperature of the solution does not differ from the reference solutions used for calibration. Rinse the cell several times with a small amount of the solution. Immerse the electrode into the solution and measure the pH without stirring. Record the pH value after a stable figure is obtained.

Annex E

(normative)

Determination of optical density

E.1 General

The natural colouring matter content of pyrethrum extract is extracted with mineral turpentine oil. The absorbance of the solution obtained is measured by using a UV-Visible Spectrometer at a wavelength of 435 nm.

E.2 Apparatus

E.2.1 UC-visible Spectrophotometer suitable for measuring at a wavelength of 435 nm and having a band width of 2 nm.

E.2.2 20-ml volumetric flask

E.2.3 Measuring cylinder

E.2.4 Pair of cuvettes

E.3 Reagents

Mineral turpentine oil of known purity.

E.4 Procedure

E.4.1 Measure and transfer exactly 1 ml of the pyrethrum extract in the 20-ml volumetric flask. Add to it mineral turpentine oil and make up the volume up to the mark. Shake the flask to mix the content thoroughly.

E.4.2 Take a pair of clean cuvettes. Switch on the Spectrophotometer and set the wavelength at 435 nm. In one cuvette take the solvent and place this cuvette in the reference cell of the spectrophotometer. Take the diluted sample from the volumetric flask in the other cuvette and place the cuvette in the sample cell of the spectrophotometer. Close the chamber and measure the absorbance at 435 nm.

Annex F **(normative)**

Sampling of pyrethrum extracts

F.1 General precautions

In drawing, preparing, storing and handling test samples, the following, precautions and directions shall be observed.

F.1.1 Samples shall not be taken in exposed place.

F.1.2 The sampling equipment shall be clean and dry when used.

F.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling equipment and the containers for samples from adventitious contamination.

F.1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed by any suitable means so as to bring all portions into uniform distribution (See F.3.1).

F.1.5 The samples shall be placed in suitable, clean, dry and air-tight amber coloured glass, containers, on which the material has no action.

F.1.6 The sample container shall be of such as size that they are almost, but not completely, filled by the sample.

F.1.7 Each container shall be closed tightly with a stopper after filling and marked with full details of sampling, the date of manufacture, name of the manufacturer and other particularities of the consignment.

F.1.8 Sample shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature. It should preferably be refrigerated at 0 °C or below.

F.2 Sampling tube

A pipette of convenient volume may be used for drawing the sample.

F.3 Scaling of sampling

F.3.1 All the containers in a single consignment of the material drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or is known to consist of different batches these shall be grouped together and each such, group shall constitute a separate lot.

F.3.2 Samples shall be tested for each lot for ascertaining the conformity of the material to the requirement of the specification.

F.3.3 The number (n) of containers to be chosen from the lot shall depend on the size of the lot and shall be in accordance with Table A.1.

F.3.4 These containers shall be chosen at random from the lot and in order to ensure that randomness of selection, some random number table, as agreed to between the purchaser and the vendor, shall be used. In case such a table is not available, the following procedure shall be adopted.

F.3.5 Starting from any container in the lot, count them as 1, 2, 3 etc, up to r in a systematic manner, where r is equal to the integral part of the value of N/n . N being the total number of containers in the lot and n the number of containers to be chosen (see Table A.1). Every r th container thus counted shall be separated until the requisite number of containers is obtained from the lot to give samples for test.

Table A.1 — Number of containers to be chosen for sampling

S.N	Lot size (N)	No. of containers to be selected (n)
i.	3 – 15	3
ii.	16 – 40	4
iii.	41 – 65	5
iv.	66 – 110	7
v.	Over 110	10

F.4 Test samples and reference samples

F.4.1 Before drawing the test sample, mix thoroughly the contents of each container selected (see Table A1) by shaking or stirring by suitable means or by rolling. Draw, by inserting the pipette through the bung hole or any other convenient opening, small portions of the material from different parts of each container selected. The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given in 4.1 and 4.2.

F.4.2 Mix thoroughly all portions of the material drawn from the same container. Out of these portions, a small but equal quantity shall be taken for each selected container and shall be well mixed together so as to form a composite sample of not less than 200 ml. This composite (sample shall be divided into three equal parts, one for the purchaser, another for the vendor and the third for the referee.

F.4.3 Referee samples - Referee samples shall consist of the composite sample (see F.4.2) marked for this purpose and shall bear the identification mark of the purchaser and the vendor. These shall be kept at a place agreed to between the two.

A.5 Number of tests

A.5.1 Tests for the determination of total pyrethrin content of the material shall be conducted on composite samples. (See F.4.2).

A.5.2 Test for the determination of the remaining characteristics namely, colour and odour and flash point shall be conducted on the composite sample as prepared under F.4.2

Bibliography

- [1] TZS 3163: 2023, *Pyrethrum extracts — Specification*
- [2] KS 3004:2024, *Pyrethrum refined extracts — Specification*
- [3] RS 191: 2019, *Refined pyrethrum concentrate — Specification*

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