



DEAS 847-2: 2025

ICS 71.100.70

DRAFT EAST AFRICAN STANDARD

Cosmetics — Analytical methods — Part 2: Determination of moisture content and volatile matter

EAST AFRICAN COMMUNITY

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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 071, *Cosmetics and related products*

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.

This second edition cancels and replaces the first edition (EAS 847-2: 2017), which has been technically revised.

EAS 847 consists of the following parts, under the general title *Cosmetics — Analytical methods*:

- *Part 1: Glossary of terms*
- *Part 2 : Determination of moisture content and volatile matter content*
- *Part 3: Determination of insoluble impurities*
- *Part 4: Determination of acid value and free fatty acids*
- *Part 5: Determination of unsaponifiable matter*
- *Part 6: Determination of melting point*
- *Part 7: Determination of specific gravity*
- *Part 8: Titre test*
- *Part 9: Determination of colour*
- *Part 10: Determination of acetyl value and hydroxyl value*
- *Part 11: Determination of allyl isothiocyanate*
- *Part 12: Determination of flash point by Pensky – Martens Closed Cap Tester*
- *Part 13 : Determination of rancidity*

- *Part 14: Determination of Polenske value*
- *Part 15 :Determination of ash content*
- *Part 16: Determination of lead, mercury and arsenic content*
- *Part 17: Determination of pH*
- *Part 18: Determination of thermal stability*
- *Part 19: Determination of non-ionic, anionic and cationic surfactant content*
- *Part 20: Determination of lather volume (foaming power)*
- *Part 21: Determination of free acid in oils*
- *Part 22: Determination of sulphur and sulphides in oils*
- *Part 23:Test for absence of grit in powders*
- *Part 24:Determination of matter insoluble in boiling water*
- *Part 25: Determination of fineness*
- *Part 26: Determination of boric acid*
- *Part 27: Determination of total fatty substance by gravimetric method*
- *Part 28: Determination of free caustic alkali.*

Cosmetics — Analytical methods — Part 2: Determination of moisture content and volatile matter content

1 Scope

This Draft East African Standard prescribes the test methods for the determination of moisture content and volatile matter content in oils for cosmetic industry.

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

EAS 847-1, Cosmetics — Analytical methods — Part 1: Glossary of terms

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EAS 847-1 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/>

4 Principle

A known mass of the sample is evaporated to constant weight and the moisture and volatile matter content is determined as the difference in weights for the sample before and after evaporation.

5 Applicability

5.1 Three methods are employed, namely

- a) air-oven method;
- b) hot-plate method; and
- c) Dean and Stark distillation method.

5.2 The first two methods give the moisture and volatile matter content together while the distillation method gives only the water content. The hot-plate method is useful for a rapid preliminary screening. The air-oven method is applicable to all the ordinary oils and fats which have a relatively low moisture content (below one percent), but not to drying or semi-drying oils or oils of the coconut oil group. The hot-plate method is

applicable to all the ordinary oils and fats including coconut oil. Neither of these two methods is applicable to solvent extracted oils and fats, which may contain residues from solvents with fairly high boiling points. The Dean and Stark distillation method is applicable to all normal oils and fats, including emulsions, for the determination of moisture only as differentiated from moisture and volatile matter. This method is not applicable to samples of oils or fats containing volatile substances miscible with water.

6 Precaution

Since water tends to settle in samples of oils or fats which have softened or melted, care shall be taken to mix the samples thoroughly before analysis so as to distribute the water uniformly. Soften the sample with gentle heat (but do not melt), and mix thoroughly. This general precaution is applicable to all the three methods.

7 Test methods

7.1 Air-oven method

7.1.1 Apparatus

7.1.1.1 **Moisture dish**, provided with tight-fitting slip-over cover

7.1.1.2 **Desiccator**, containing an efficient desiccant, such as phosphorus pentoxide

7.1.1.3 **Air oven**, preferably electrically heated, with calibrated temperature control device

7.1.2 Procedure

Weigh approximately 10 g (W_1) of the oil or fat into a moisture dish which has been dried previously, cooled in the desiccator and then weighed. Place the dish in the air-oven for approximately one hour at $105\text{ }^\circ\text{C} \pm 1\text{ }^\circ\text{C}$. Remove the dish from the oven, cool in the desiccator to room temperature and weigh the dried material (W_2). Repeat this procedure but keep the dish in the oven only for half an hour each time until the difference between the two successive weighings does not exceed one milligram.

7.1.3 Calculation

The moisture and volatile matter content, expressed in percent by weight, shall be calculated using the formula below:

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

where

W_1 mass, in grams, of the material taken for the test; and

W_2 mass, in grams, of the dried material.

7.2 Hot-plate method

7.2.1 Apparatus

7.2.1.1 **Glass beaker**, 100-mL – 150-mL capacity

7.2.1.2 **Small glass rod**

7.2.1.3 Desiccator, containing an efficient desiccant, such as phosphorus pentoxide

7.2.1.4 Electric hot-plate, with variable heat control

7.2.2 Procedure

7.2.2.1 Weigh approximately 10 g (W_1) of the oil or fat into the glass beaker which has been previously dried along with the small glass rod, cooled in the desiccator, and weighed.

7.2.2.2 Heat the sample on the electric hot-plate, stirring continuously with the glass rod. Avoid spattering of the oil or fat which may result from too rapid an ebullition of moisture. The apparent end point is judged by the cessation of the rising bubbles of steam as well as by the absence of foam. Alternatively judge the end point by placing a clean, dry watch-glass on top of the beaker and observing when no further condensation takes place on the watch-glass.

7.2.2.3 When the apparent end point has been reached, heat momentarily to the point of incipient smoking taking care not to overheat. Cool to room temperature in the desiccator and weigh (W_2).

7.2.3 Calculation

The moisture and volatile matter content, expressed in percent by weight, shall be calculated using the formula below:

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

where

W_1 mass, in grams, of the material taken for the test; and

W_2 mass, in grams, of the material upon drying.

7.3 Dean and Stark Distillation method

7.3.1 Apparatus

7.3.1.1 The apparatus consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap and connected to the flask. The connections between the trap and the condenser and the flask should be interchangeable ground glass joints. The trap serves to collect and measure the condensed water, and to return the solvent to the flask.

7.3.1.2 The assembly of the apparatus is shown in Figure 1, and the various components are described below:

- a) **Flask**, a 500-mL – 1 000-mL flask of the shape shown in Figure 1, made of hard resistance glass, well annealed and as free as possible from striae and similar defects

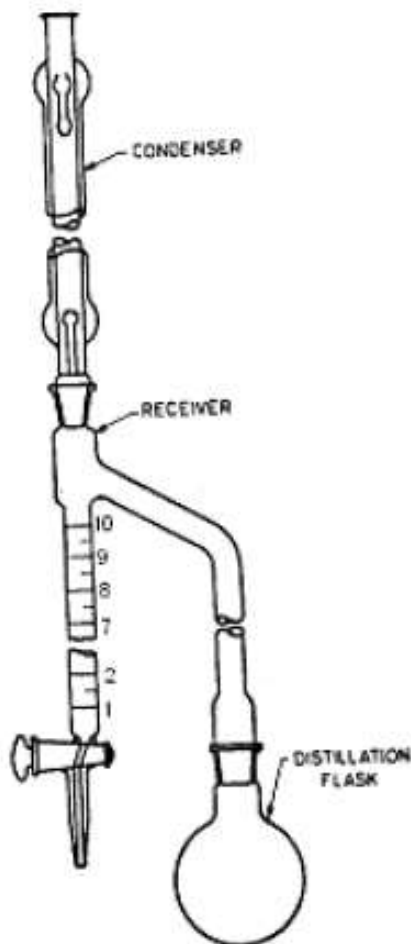


Figure 1 — Typical assembly of Dean and Stack apparatus

- b) **Condenser**, a glass water-cooled reflux type condenser, of the design and dimensions shown in Figure 2. The only mandatory dimensions for the condenser are the external diameters of the inner tube and of the jacket, which shall be 16 mm - 17 mm and 23 mm - 25 mm respectively. The joints A and B should be neatly finished as shown in Figure 2, particularly the bore at B shall have the minimum disturbance. The shoulder above the cone of Joint D shall be elongated as shown in Figure 2 to avoid a sharp re-entrant shape which may restrict the free flow of liquid down the inner wall. The cone shall be extended beyond the length appropriate to the Joint D, and the lower end ground at an angle of approximately 60° to the axis. The drainage tip shall be at the front of the condenser when the lower water connection is to the left, and the finish shall be either smooth or fire-polished. When inserted into the trap, the tip of the condenser shall be 6 mm - 7 mm above the surface of the liquid in the trap after distillation conditions have been established. The nominal dimensions of the Joint D are given in Table 1 below:

Table 1 — Nominal dimensions of Joint D

| Nominal diameter of large end of ground zone mm | Nominal diameter of small end of ground zone mm | Nominal length of ground zone measured axially mm |
|--|--|--|
| 18.8 | 16.2 | 26 |

All dimensions in millimetres

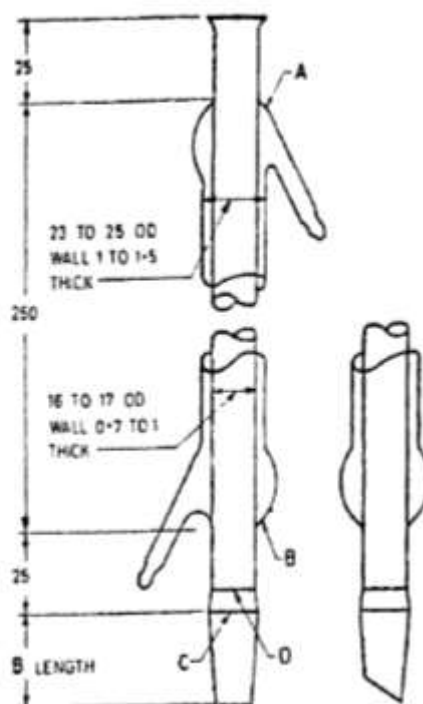


Figure 2 — Condenser

- c) **Receivers**, otherwise called the trap, made of hard resistance glass, well annealed and as free as possible from striae and similar defects, provided with ground glass joints, with the shape, dimensions and tolerances give in Figure 3 and Figure 4; consisting essentially of the upper chamber, together with the tube and ground joint leading to the flask, and the graduated tube. The receivers shall be of two sizes, namely, 2-mL capacity and 10-mL capacity (see Figure 3 and Figure 4). The requirements for the dimensions and tolerances for the receivers shall be as given in Table 2.

All dimensions in millimetres

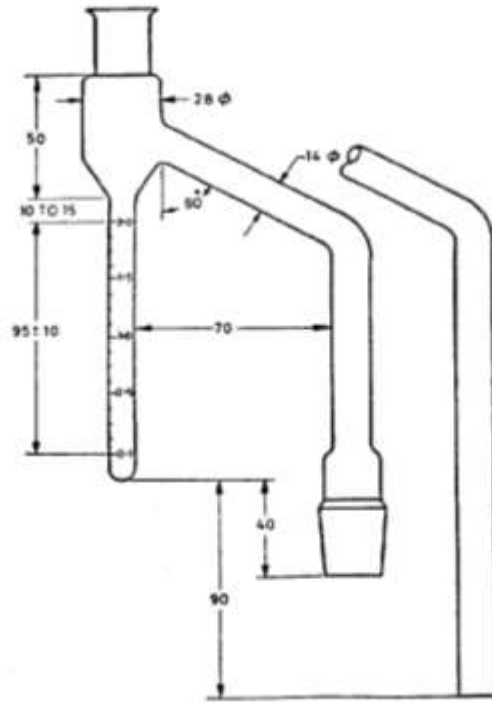


Figure 3 — 2-mL receiver

All dimensions in millimeters

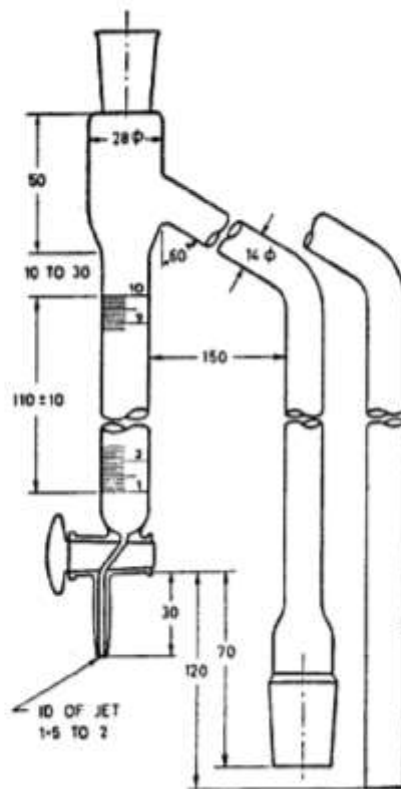


Figure 4 — 10-mL receiver

Table 2 — Requirements dimensions and tolerances for receiver

| S/N | Characteristic | Receiver | |
|------|--|----------|----------|
| | | 2-mL | 10-mL |
| i. | Volume, equivalent to smallest subdivision, mL | 0.05 | 0.1 |
| ii. | Scale length, mm | 95 ± 10 | 110 ± 10 |
| iii. | Length of cylindrical tube above upper graduation mark, mm | 10 – 15 | 10 – 30 |
| iv. | Maximum permissible leakage rate of stopcock, mL/min. | - | 0.004 |

The shoulder of the upper chamber of the receiver immediately below the conical joint shall be finished square, as shown in Figure 3 and Figure 4. The graduated portion of the receiver shall be cylindrical throughout its length. The bottom of the graduated tube of the 2-mL receiver shall be sealed, the end of the tube being approximately hemispherical in shape.

The graduated scales on the receivers shall be numbered and subdivided as shown in Figure 3 and Figure 4. The graduation marks shall be fine cleanly etched permanent lines of uniform thickness lying in planes at right angles to the axis of the tube. The graduation marks shall be confined to the cylindrical portion of the tube and there shall be no evident irregularity in their spacing. In these receivers the numbered graduation marks shall be carried completely round the tube, the shortest graduation mark shall be carried halfway round the tube, and the graduation marks of intermediate length shall be carried approximately two-thirds of the way round the tube and shall project equally at each end beyond the shortest graduation marks.

The capacity corresponding to any graduation mark is defined as the volume of water at 27 °C, expressed in millilitres, required to fill the graduated portion to that mark at 27 °C, the axis of the graduated portion being vertical and the lowest point of the water meniscus being set on the graduation mark. In the case of 10 ml receiver, the volume of the bore of the stopcock key and the volume of the jet below the stopcock key and the volume of the jet below the stopcock shall not be included as part of the measured volume.

The error at any point on the receiver scale, and also the difference between the errors at any two points on the scale, shall not exceed the figures given for the receivers in Table 2.

- d) **Heat source**, may be either an oil-bath or an electric heater provided with a sliding rheostat or other means of heat control. The temperature of the oil in the bath should not be very much higher than the boiling point of xylene or toluene, whichever solvent is used.
- e) **Copper wire**, long enough to extend through the condenser, with one end twisted into a spiral. The diameter of the spiral should be such that it fits snugly within the graduated portion of the receiver and yet may be moved up and down.

7.3.2 Reagents

7.3.2.1 Potassium dichromate-sulphuric acid cleaning solution

7.3.2.2 Xylene or toluene, saturate the xylene or toluene by shaking with a small quantity of water, and distil. Use the distillate for the determination of moisture.

7.3.3 Procedure

7.3.3.1 Clean the entire apparatus with potassium dichromate-sulphuric acid cleaning solution to minimize the adherence of water droplets to the sides of the condenser and the receiver. Rinse thoroughly with water and dry completely before using. The quantity of material taken for the test is determined by the amount of moisture present (w/w), as indicated below:

Table 3 — Amount of moisture present

| S/N | Moisture range | Quantity of material (approximately) |
|------|----------------|--------------------------------------|
| | % | g |
| i. | < 1 | 200 |
| ii. | 1 – 5 | 100 |
| iii. | > 5 | Proportionally smaller quantity |

7.3.3.2 Place the specified quantity of material, accurately weighed (W), in the distillation flask, add an equal volume of xylene or toluene, as desired, or at least 100 mL if less than 100 g of the material is used, and swirl to mix. Assemble the apparatus and fill the receiver with the solvent by pouring it through the condenser until it begins to overflow into the distillation flask. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture within the tube. In order that the refluxing may be under control, use tubes with ground quick fit ends. Heat the flask to between 80 °C - 90 °C. When the greater part of the water has distilled over, increase gradually to between 105 °C - 120 °C and continue until no more water is collected. Reflux until the water level in the receiver remains unchanged for 30 min, and then shut off the source of heat. Purge the reflux condenser after the distillation with 20-mL portions of xylene or toluene to wash down any moisture adhering to the walls of the condenser.

7.3.3.3 Immerse the receiver in water at about 27 °C for at least 15 min or until the xylene or toluene layer is clear, and then read the volume of water.

7.3.4 Calculation

The moisture content, expressed in percent by weight, shall be calculated using the formula below:

$$\frac{100 VD}{W}$$

where

V is the volume, in millilitres, of water;

D is the specific gravity of water at the temperature at which the volume of water is read; and

W is the weight, in grams, of the material taken for the test.

Bibliography

EAS 847-2: 2017, Cosmetics — Analytical methods — Part 2: Determination of moisture content and volatile matter content.

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