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DRAFT EAST AFRICAN STANDARD

Cosmetics — Analytical methods — Part 10: Determination of acetyl value and hydroxyl value

EAST AFRICAN COMMUNITY

Second Edition 2025

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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 847-10:2017), 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 10: Determination of acetyl value and hydroxyl value

1 Scope

This Draft East African prescribes the test methods for the determination of acetyl value and hydroxyl value in oils and fats 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 Test method

4.1 General

Two methods have been prescribed, namely Method A and Method B.

4.1.1 Method A

4.1.1.1 Principle of the method

The process consists of acetylating the oil or fat with a measured quantity of acetic anhydride in pyridine decomposing the excess anhydride by boiling with water and then, after the addition of sufficient butyl alcohol to give a homogeneous solution, titrating with alkali. A control test with the acetic anhydride and pyridine without the oil provides a measure of the acetic anhydride available for acetylation; a similar test with the oil or fat and the pyridine without the acetic anhydride provides a measure of the free fatty acid present. From the values obtained, the acetyl value or the hydroxyl value of the oil or fat is calculated.

4.1.1.2 Reagents

4.1.1.2.1 **Pyridine**, reflux with powdered barium oxide and distil. Use the fraction distilling above 114 °C

4.1.1.2.2 Acetic anhydride

4.1.1.2.3 Acetylating agent, mix one volume of acetic anhydride and seven volumes of pyridine

4.1.1.2.4 Standardized ethanolic potassium hydroxide solution, 0.30 N

4.1.1.2.5 Normal butyl alcohol

4.1.1.2.6 Phenolphthalein solution, dissolve 0.1 g in 100 mL of 60 % rectified spirit.

NOTE In the determination of substances giving dark-coloured soap solutions, observation of the end point of the titration may be facilitated either:

a) by substitution of thymolphthalein or alkali blue 6B for phenolphthalein or

b) by the addition of one millilitre of 0.1 % solution of methylene blue to each 100 mL of the phenolphthalein solution before titration.

4.1.1.3 Apparatus

4.1.1.3.1 Round bottomed flask, Class A glass with a capacity of 200 mL - 250 mL with a 100-cm ground-end condenser tube

4.1.1.3.2 Burette

4.1.1.4 Procedure

4.1.1.4.1 Weigh accurately 0.5 g - 3.0 g of the sample in the flask. (The following weights serve as a rough guide for different types of materials: fatty alcohols 0.5 g - 0.7 g, castor oil about 1 g and ordinary oils 2 g - 3 g). From a 10-mL burette add, 5 mL of the acetylating agent dropwise into the flask. Before attaching the condenser, moisten the neck of the flask with pyridine to act as a seal, and make sure that the seal is maintained during the acetylation.

NOTE Owing to the presence of vapours of pyridine and butyl alcohol, it is preferable to carry out the tests in a fuming chamber. The analyst should wear appropriate personal protective equipment.

4.1.1.4.2 Mix the sample and the acetylating agent by shaking well, add one or two small pieces of pumice, and boil the contents of the flask gently for 60 min, maintaining the boiling so that the vapour rises no higher than the bottom end of the condenser to maintain the pyridine seal.

4.1.1.4.3 Cool the flask to about 50 °C and with a rotary motion to assist in washing the condenser tube, add 5 mL of distilled water from the top of the condenser. Shake the mixture well, and then boil it gently for 5 min - 10 min.

4.1.1.4.4 After cooling the flask and the contents to room temperature and before detaching the condenser, wash the condenser with 30 mL of butyl alcohol. Detach the condenser and wash the neck and mouth of the flask and the tip of the condenser with a further 20 mL of butyl alcohol and then, if the contents of the flask are not homogeneous, add butyl alcohol until they become homogeneous. Titrate the free acetic acid with carbonate-free 0.35 N potassium hydroxide solution in the presence of a few drops of phenolphthalein indicator.

4.1.1.4.5 Carry out the same series of operations with 5 mL of acetylating agent alone, also with a corresponding weight of the sample and 5 mL of pyridine.

4.1.1.5 Calculation

The hydroxyl value, *H*, shall be expressed as follows:

$$H = \frac{56.1\,NV}{m}$$

The acetyl value, *A*, shall be expressed as follows:

$$A = \frac{H}{1 + 0.000\,75\,H}$$

where

N is the normality of potassium hydroxide solution,

V is the volume, in millilitres, of potassium hydroxide solution corresponding to the amount of acetylated fat formed,

$$V = a + b - c$$

where

a, b and c are respectively the volumes, in milllitres, of potassium hydroxide required by blank with acetylating agent, sample plus pyridine, and sample plus acetylating agent, and

m is the mass, in grams, of sample taken for test.

NOTE It is to be noted that a slight increase in the acetyl value has been found to occur with increasing free fatty acid content of the sample.

4.1.2 Method B

4.1.2.1 Principle of the method

A sample of oil or fat acetylated by refluxing with acetic anhydride and the excess anhydride is decomposed with water and sodium bicarbonate solution. The saponification value of the washed and dried acetylated oil or fat is determined.

4.1.2.2 Apparatus

4.1.2.2.1 Beaker, 800-mL capacity

4.1.2.2.2 Separating funnel, 500-mL capacity

4.1.2.2.3 Conical flask, 250-mL and 500-mL capacity

4.1.2.2.4 Reflux condenser, any efficient reflux condenser, at 65 cm long

4.1.2.2.5 Sand bath or electric hot-plate, with rheostat control

4.1.2.3 Reagents

4.1.2.3.1 Acetic anhydride, containing 95 % - 100 % (by weight) of the actual acetic anhydride. Determine the acetic anhydride content as follows:

4.1.2.3.1.1 Weigh approximately 2 g of the acetic anhydride into 200-mL glass-stoppered conical flask, cool in ice and add 5 mL of freshly distilled aniline. Insert the glass stopper immediately, shake vigorously and allow to stand at room temperature for 30 min. Wash down the sides of the flask with cold water. Mix well and titrate with previously standardized 1 N sodium hydroxide solution, using phenolphthalein as indicator, until the pink colour persists for 10 min.

$$A = \frac{Volume\ in\ millilitres of\ 1N\ sodium\ hydroxide\ solution\ required}{Mass\ in\ grams\ of\ acetic\ anhydride\ taken\ for\ the\ test}$$

4.1.2.3.1.2 Weigh approximately 2 g of the acetic anhydrides into another flask. Add 50 mL of water, allow to stand for 30 min and titrate with the standard sodium hydroxide solution to the same end point as above using phenolphthalein as indicator.

$$B = \frac{Volume\ in\ millilitres of\ 1N\ sodium\ hydroxide\ solution\ required}{Mass\ in\ grams\ of\ acetic\ anhydride\ taken\ for\ the\ test}$$

$$Percent\ by\ weight\ = 10.209\ (B\ -\ A)$$

4.1.2.3.2 **Sodium bicarbonate solution**, freshly prepared 0.5 % (m/v) and neutral to litmus

4.1.2.3.3 **Anhydrous sodium sulphate**

4.1.2.3.4 **Alcoholic potassium hydroxide solution**, 0.5 N

4.1.2.3.5 **Phenolphthalein indicator solution**, dissolve 0.1 g in 100 mL of 66 % rectified spirit

4.1.2.3.6 **Standard hydrochloric acid**, approximately 0.5 N

4.1.2.3.7 **Aniline**

4.1.2.4 Procedure

4.1.2.4.1 Weigh approximately 10 g of the sample in a conical flask, add 20 mL of acetic anhydride and boil the mixture under a reflux condenser for about 2 h. Pour the mixture into a beaker containing 500 mL of water and boil for 15 min. Add anti-bumping stones. Discontinue boiling, cool slightly, and remove the water by decanting. Add another 500 mL of water and boil again. Discontinue boiling, cool and transfer the contents of the beaker to separating funnel and discard the lower layer. Wash the acetylated sample successively:

a) three times with 50 mL of water;

b) twice with 50 mL of sodium bicarbonate solution; and

c) twice with 50 mL of hot water (60 °C - 70 °C09).

4.1.2.4.2 Drain and remove as much of the water as possible and then transfer the acetylated sample to a beaker and add approximately 5 g of anhydrous sodium sulphate. Allow to stand for about 1 h with occasional stirring. Filter through a dry filter paper. Remove the filter and keep the sample in the oven at 100 °C - 110 °C until it is thoroughly dry. Determine the saponification values of the original materials and the acetylated product.

4.1.2.5 Calculation

4.1.2.5.1 The hydroxyl value, *H*, shall be expressed using the formula below:

$$H = \frac{S' - S}{1.000 - 0.000\,75\,S'}$$

4.1.2.5.2 The acetyl value, *A*, shall be expressed using the formula below:

$$A = \frac{S' - S}{1.000 - 0.000\,75\,S}$$

where

S' is the saponification value after acetylation, and

S is the saponification value before acetylation

Bibliography

EAS 847-10: 2017, *Cosmetics — Analytical methods — Part 10: Determination of acetyl value and hydroxyl value*