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DRAFT EAST AFRICAN STANDARD

Cosmetics — Analytical methods — Part 16: Determination of lead, mercury and arsenic content

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 847-16:2017), 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 16: Determination of lead, mercury and arsenic content

1 Scope

This Draft East African Standard prescribes the test method for the determination of lead, mercury and arsenic content in cosmetics and 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 methods

4.1 Determination of lead content in cosmetics and related products using Graphite Furnace Atomic Absorption Spectrophotometer (GFAAS)

4.1.1 General

This test method specifies an electrothermal atomization technique using graphite furnace AAS method for the determination of lead content in cosmetics and oils or fats for cosmetic industry.

WARNING AND SAFETY — The acids used in the test are highly corrosive and shall be handled with maximum care and where appropriate, use a fume hood during preparation of standards. Lead is very toxic/carcinogenic and shall be handled with maximum care avoiding physical contact. If spillage occurs, use adequate amounts of water and soap to wash off the spill.

4.1.2 Principle

This method involves injecting of the prepared solution into a graphite furnace and carrying out spectrometric measurements of the atomic absorption of the 228.8 spectral line emitted by lead Hollow Cathode Lamp (HCL).

4.1.3 Reagents and standards

4.1.3.1 Concentrated nitric acid

4.1.3.2 Nitric acid, (1:1) v/v, mix 1 volume of concentrated nitric acid (HNO_3) with 1 volume of distilled water.

4.1.3.3 Nitric acid, 0.1 N

4.1.3.4 Nitric acid, 2 N

4.1.3.5 Nitric acid, 0.5 N

4.1.3.6 Lead standard solution, 1 000 mg/kg, in 1-L volumetric flask, dissolve 1.598 g of $Pb(NO_3)_2$ in a minimum volume of 1 % v/v HNO_3 and finally top the mark using 1 % HNO_3.

NOTE Commercial grade standards can also be used when available.

4.1.3.7 Lead standard solution, 100 µg/L, prepared freshly by serial dilution of the lead solution

4.1.3.8 Purge gas, argon

4.1.4 Apparatus and equipment

4.1.4.1 Atomic absorption spectrometer, fitted with graphite furnace. The atomic absorption spectrometer used shall be satisfactory if after optimization according to the manufacturer's instructions, and when in reasonable agreement with the values given by the manufacturer, it meets the performance criteria as set out in the manual.

4.1.4.2 Lead hollow cathode lamp

4.1.4.3 Ordinary laboratory apparatus. All glassware shall first be washed in hydrochloric acid (density about 1.19 g/mL, diluted.)

4.1.4.4 Muffle furnace

4.1.4.5 Analytical balance

4.1.5 Performance

4.1.5.1 Sample preparation

Ignite 1 g of sample at 500 ºC ± 2 ºC to ash. Extract the lead from the ash with 20 mL of 2 N HNO_3, and repeat with 10 mL of 2 N HNO_3. Combine the extracts and dilute to 50 mL with 0.5 N HNO_3.

4.1.5.2 Calibration: preparation of calibration curve

4.1.5.2.1 Dilute the stock 100 µg/L solution with 0.1 N HNO_3 to obtain solutions with 10 µg/L, 20 µg/L, 40 µg/L, 60 µg/L, 80 µg/L and 90 µg/L of lead.

4.1.5.2.2 Inject 20 µL each of the six solutions in turns at the same rate starting from the lowest concentrated solution to the highest concentrated solution.

4.1.5.2.3 Record the corresponding absorbance values and plot calibration curve.

4.1.5.3 Quality control checks

4.1.5.3.1 Duplicates

In a set of ten or less number of samples, one sample shall be analysed in duplicate to comply with accepted criteria: The absolute difference between two independent test results obtained using the same procedure shall be not greater than 10 % of the arithmetic mean of the two results.

4.1.5.3.2 Spiked blank

Spiked sample or distilled water with the concentration 10.0 µg/L of lead shall be analysed alongside samples and a recovery of 100 % ± 5 % used as criteria of acceptance of results.

4.1.6 Procedure

4.1.6.1 Test portion

Use sample as prepared in 4.1.5.1.

4.1.6.2 Blank test

4.1.6.2.1 Run a parallel reagent blank determination replacing the test solution with distilled water.

4.1.6.2.2 Reagent blank shall be ≤ 0.000 1 µg/L of lead.

4.1.6.3 Instrumentation

4.1.6.3.1 Follow the manufacturer's instructions for preparing the instrument for use.

4.1.6.3.2 Install the appropriate lamp and adjust the current to the recommended value.

4.1.6.3.3 Ensure that the auto-sampler pipette is correctly aligned and that the drain is available.

4.1.6.3.4 Select the sample tray type installed.

4.1.6.3.5 Ensure that the graphite tube is in good condition and correctly aligned.

4.1.6.3.6 Switch on the cooling system, turn on the purge gas and finally start the instrument software.

4.1.6.3.7 Select the relevant method and then condition tube.

4.1.6.3.8 The following instrument conditions shall be used for the furnace during analysis of lead:

a) wavelength: 283.3

b) slit: 0.7

c) atomization site: pyro/platform

d) furnace condition for lead.

Refer to the instructions of the manufacturer.

4.1.6.4 Spectrometric measurements

4.1.6.4.1 Inject into the furnace the calibration standards, the blank solution and the test solution.

4.1.6.4.2 Record the absorbance reading.

4.1.6.4.3 If the absorbance of the sample is greater than the highest calibration standard, dilute the test solution appropriately using 0.1 N HNO_3 for lead, then measure the absorbance.

4.1.6.4.4 Inject the calibration solutions in ascending order. The calibration curve shall only be acceptable for analysis when the correlation coefficient (r_2) ≥ 0.99.

4.1.7 Expression of results

4.1.7.1 Method of calculation

The lead content, expressed in milligrams per litre, shall be calculated using the formula below:

$$\frac{C_1 - C_2}{M_0} \times V$$

where

C_1 is the lead content of test solution, in milligrams per litre, read from calibration curve;

C_2 is the lead content of blank solution, in milligrams per litre, read from calibration curve;

M_0 is the mass, in grams, of sample taken for analysis; and

V is the volume, in millilitres, of sample dilution

If the test solution was diluted, then the dilution factor shall be taken into consideration in calculation.

4.1.7.2 Expression of results

Report results of lead content in milligrams per litre as Pb in the sample into two significant figures.

4.1.8 Method validation

4.1.8.1 Precision repeatability

The absolute difference between two independent tests results, obtained using the same method, on identical test material, in the same laboratory by the same operator, using the same equipment, within a short interval of time shall be not greater than 10 % of the arithmetic mean of the two results.

4.1.8.2 Working range

Pb 10 µg/L – 100 µg/L

4 2 Determination of arsenic using Graphite Furnace Atomic Absorption Spectrophotometer (GFAAS)

4 2.1 General

This test method prescribes the determination of arsenic in cosmetics and oils for cosmetic industry.

WARNING AND SAFETY — The acids used in the test are highly corrosive and shall be handled with maximum care and where appropriate, use a fume hood during preparation of standards. Arsenic is very toxic/carcinogenic and shall be handled with maximum care avoiding physical contact. If spillage occurs, use adequate amounts of water and soap to wash off the spill.

4 2.2 Reagents

4 2.2.1 Hydrochloric acid (HCl), 1.5 % v/v, carefully add 15 mL concentrated HCl to distilled water and make up to 1 L.

4 2.2.2 Sodium hydroxide (NaOH) solution, 1 % w/v, carefully dissolve 10 g of NaOH in distilled water and make up to 1 L.

4 2.2.3 Sodium tetrahydroborate ($NaBH_4$) solution, 3 % w/v, dissolve 3 g of $NaBH_4$ in 1 % NaOH solution and make up to 100 mL with 1 % NaOH solution.

4 2.2.4 Stock solution, prepare the stock solution containing 1 000 mg/L As using commercially available concentrated calibration solutions.

4 2.2.5 Reductant solution, 3 % $NaBH_4$ in 1 % NaOH solution. Pre-reduction can be performed as follows either with potassium iodide (KI) in semi-concentrated (5 mol/L) HCl solution or, preferably, with L-cysteine:

a) **Potassium iodide (KI) solution**, dissolve 3 g KI and 5 g L (+)- ascorbic acid in 100 mL water. Prepare fresh daily. Add 1 mL of the KI solution per 10 mL of the sample solution in 15 % HCl and allow to stand for 30 min, or

b) **L-cysteine solution**, dissolve 5 g L-cysteine in 100 mL 1.5% HCl. This solution is stable for at least a month. Add 2 mL of the L-cysteine solution per 10 mL of the sample solution and allow to stand for 30 min.

4 2.3 Preparation of calibration curve

4 2.3.1 Dilute the stock 100 µg/L solution with 1.5 % HCl to obtain solutions with 10 µg/L, 20 µg/L, 40 µg/L, 60 µg/L, 80 µg/L and 90 µg/L of arsenic.

4 2.3.2 Inject 20 µL each of the six solutions in turns at the same rate starting from the lowest concentrated solution to the highest concentrated solution.

4 2.3.3 Record the corresponding absorbance values and plot calibration curve.

4 2.4 Quality control checks

4 2.4.1 Duplicates

In a set of ten or less number of samples, one sample shall be analysed in duplicate to comply with accepted criteria: The absolute difference between two independent test results obtained using the same procedure shall be not greater than 10 % of the arithmetic mean of the two results.

4 2.4.2 Spiked blank

Spiked sample or distilled water with the concentration 10.0 µg/L of arsenic shall be analysed alongside samples and a recovery of 100 % ± 5 % used as criteria of acceptance of results.

4 2.5 Instrument conditions

The following conditions shall be used for the furnace during analysis of arsenic:

a) wavelength: 193.7nm

b) slit: 0.7 nm

c) atomization site: pyro/platform

d) furnace condition for arsenic.

Refer to the instructions of the manufacturer.

4 2.6 Spectrometric measurements

4 2.6.1 Inject into the furnace the calibration standards, the blank solution and the test solution.

4 2.6.2 Record the absorbance reading.

4 2.6.3 If the absorbance of the sample is greater than the highest calibration standard, dilute the test solution appropriately using 1.5 % HCl for arsenic, then measure the absorbance.

4 2.6.4 Inject the calibration solutions in ascending order.

4 2.6.5 The calibration curve shall only be acceptable for analysis when the correlation coefficient (r^2) ≥ 0.99

4.2.7 Expression of results

4.2.7.1 Method of calculation

The arsenic content, expressed in milligrams per litre, shall be calculated using the formula below:

$$\frac{C_1 - C_2}{M_0} \times V$$

where

C_1 is the lead content of test solution, in milligrams per litre, read from calibration curve;

C_2 is the lead content of blank solution, in milligrams per litre, read from calibration curve;

M_0 is the mass, in grams, of sample taken for analysis; and

V is the volume, in millilitres, of sample dilution

If the test solution was diluted, then the dilution factor shall be taken into consideration in calculation.

4.2.7.2 Expression of results

Report results of arsenic content in milligrams per litre as As in the sample into two significant figures.

4.2.8 Method validation

4.2.8.1 Precision repeatability

The absolute difference between two independent tests results, obtained using the same method, on identical test material, in the same laboratory by the same operator, using the same equipment, within a short interval of time shall be not greater than 10 % of the arithmetic mean of the two results.

4.2.8.2 Working range

As 10 µg/L – 100 µg/L

4.3 Determination of mercury using Graphite Furnace Atomic Absorption Spectrophotometer (GFAAS)

4.3.1 General

This test method prescribes the procedure for determination of mercury in cosmetics and oils for cosmetic industry using sodium tetrahydroborate ($NaBH_4$) or tin (II) chloride $SnCl_2$ as a reductant.

WARNING AND SAFETY — The acids used in the test are highly corrosive and shall be handled with maximum care and where appropriate, use a fume hood during preparation of standards. Mercury is very toxic/carcinogenic and shall be handled with maximum care avoiding physical contact. If spillage occurs, use adequate amounts of water and soap to wash off the spill.

4.3.2 Principle

A solution of the sample is digested in a closed vessel and then it is reduced to a hydride for spectrometric measurement of the atomic absorption of the 253.6 nm spectral line emitted by the electrodeless discharge lamp or hollow cathode lamp for mercury.

4.3.3 Reagents

4.3.3.1 Using sodium tetrahydroborate ($NaBH_4$) as reductant

4.3.3.1.1 Hydrochloric acid (HCl), 1.5 % v/v, carefully add 15 mL concentrated HCl to deionized water and make up to 1 L.

4.3.3.1.2 Nitric acid (HNO_3), 1.5 % v/v, carefully add 15 mL concentrated HNO_3 to deionized water and make up to 1 L.

4.3.3.1.3 Potassium permanganate ($KMnO_4$) solution, 5 % w/v, dissolve 5 g of $KMnO_4$ in deionized water and make up to 100 mL.

4.3.3.1.4 Sodium hydroxide (NaOH) solution, 1 % w/v, carefully dissolve 10 g NaOH in deionized water and make up to 1 L.

4.3.1.5 Sodium tetrahydroborate ($NaBH_4$) solution, 3 % w/v, dissolve 3 g of $NaBH_4$ in 1 % NaOH solution and make up to 100 mL with 1 % NaOH solution.

4.3.1.6 Stock solution, contains 1 000 mg/L Hg

4.3.1.7 Reductant solution, 3 % $NaBH_4$ in 1 % NaOH solution

4.3.3.2 Using tin (II) chloride ($SnCl_2$) as reductant

4.3.3.2.1 Hydrochloric acid (HCl), 1.5 % v/v

4.3.3.2.2 Hydrochloric acid (HCl), 10 % v/v, carefully add 100 mL concentrated HCl to deionized water and make up to 1 L.

4.3.3.2.3 Nitric acid (HNO_3), 1.5 % v/v, carefully add 15 mL concentrated HNO_3 to deionized water and make up to1 L.

4.3.3.2.4 Potassium permanganate ($KMnO_4$) solution, 5 % w/v, dissolve 5 g of $KMnO_4$ in deionized water and make up to 100 mL.

4.3.3.2.5 Tin (II) chloride dihydrate ($SnCl_2.2H_2O$), 5 % w/v, dissolve 50 g of $SnCl_2.2H_2O$ in 10 % HCl solution and make up to 1 L with 10 % HCl solution.

4.3.3.2.6 Stock solution, prepare the stock solution containing 1 000 mg/L Hg using commercially available concentrated calibration solutions.

4.3.3.2.7 Reductant solution, 5 % $SnCl_2.2H_2O$ in 10 % HCl solution.

4.3.4 Preparation of the calibration curve

4.3.4.1 Using sodium tetrahydroborate as reductant

- Calibration solution: 1 mg Hg/L (in 1.5 % HNO_3 stabilized by the addition of a few drops of 5 % $KMnO_4$ solution)
- Aliquots for calibration: 100 µL, 200 µL, 500 µL.
- Corresponding to: 100 ng Hg, 200 ng Hg, 500 ng Hg.
- Diluent: 1.5 % (v/v) nitric acid or 1.5 % (v/v) hydrochloric acid.
- Calibration volume: 10 mL.

NOTE The use of commercially available concentrated calibration solutions for AAS is recommended.

4.3.4.2 Using tin (II) chloride ($SnCl_2$) as reductant

- Calibration solution, 1 mg Hg/L (in 1.5 % HNO_3 stabilized by the addition of a few drops of 5 % $KMnO_4$ solution).
- Aliquots for calibration: 100 µL, 200 µL, 500 µL.
- Corresponding to: 100 ng Hg, 200 ng Hg, 500 ng Hg.
- Diluent: 1.5 % (v/v) nitric acid or 1.5 % (v/v) hydrochloric acid.
- Calibration volume: 10 mL.

4.3.5 Instrument conditions

4.3.5.1 Instrument conditions using sodium tetrahydroborate ($NaBH_4$) as reductant

The following conditions shall be used for the furnace during analysis of mercury:

a) wavelength: 253.6 nm

b) slit: 0.7 nm low

c) atomization site: pyro/platform

d) furnace condition for mercury.

Refer to the instructions of the manufacturer.

NOTE1 Stabilize stock and calibration solutions by adding $KMnO_4$ or KI solution.

NOTE 2 Stabilize all solutions in the reaction flask by adding 1 drop of 5 % (w/v) $KMnO_4$ solution before starting the determination.

4.3.5.2 Instrument conditions tin (II) chloride ($SnCl_2$) as reductant

The following conditions shall be used for the furnace during analysis of mercury:

a) analytical wavelength: 253.6 nm

b) Slit width and height: 0.7 nm low

c) radiation source: Electrodeless discharge lamp or hollow cathode lamp for Hg

d) QTA heating: No flame required. If condensation in the QTA is a problem, heat the QTA gently by mounting an infrared lamp above it.

e) prepared measurement volume: 10 mL minimum to 50 mL maximum

f) Pre-reaction purge time: approximately 5 s

g) Post-reaction purge time: approximately 5 s

h) characteristic mass 4.68 ng Hg for 1 % absorption (A = 0.0044)

i) characteristic concentration: 0.468 µg/L / 1 % absorption for 10 mL calibration volume

j) characteristic concentration check 250 µL of the 1 000 mg/L Hg stock solution (250 ng) give an absorbance of approx. A = 0.2

NOTE 1 Stabilize stock and calibration solutions by adding $KMnO_4$ solution. Do not use KI solution since iodide interferes in the release of mercury.

NOTE 2 Stabilize all solutions in the reaction flask by adding 1 drop of 5 % (w/v) $KMnO_4$ solution before starting the determination.

All reagents used shall be analytical grade.

4.3.6 Spectrometric measurements

4.3.6.1 Inject into the furnace the calibration standards, the blank solution and the test solution.

4.3.6.2 Record the absorbance reading.

4.3.6.3 If the absorbance of the sample is greater than the highest calibration standard, dilute the test solution appropriately using 1.5 % HNO_3 for mercury, then measure the absorbance.

4.3.6.4 Inject the calibration solutions in ascending order.

4.3.6.5 The calibration curve shall only be acceptable for analysis when the correlation coefficient (r^2) ≥ 0.99.

4.3.7 Expression of results

4.3.7.1 Method of calculation

The mercury content, expressed in milligrams per litre, shall be calculated using the formula below:

$$\frac{C_1 - C_2}{M_0} \times V$$

where

C_1 is the lead content of test solution, in milligrams per litre, read from calibration curve;

C_2 is the lead content of blank solution, in milligrams per litre, read from calibration curve;

M_0 is the mass, in grams, of sample taken for analysis; and

V is the volume, in millilitres, of sample dilution

If the test solution was diluted, then the dilution factor shall be taken into consideration in calculation.

4.3.7.2 Expression of results

Report results of arsenic content in milligrams per litre as As in the sample into two significant figures.

4.3.8 Method validation

4.3.8.1 Precision repeatability

The absolute difference between two independent tests results, obtained using the same method, on identical test material, in the same laboratory by the same operator, using the same equipment, within a short interval of time shall be not greater than 10 % of the arithmetic mean of the two results.

4.3.8.2 Working range

As 10 µg/L – 100 µg/L

Bibliography

EAS 847-16: 2017, *Cosmetics — Analytical methods — Part 16: Determination of lead, mercury and arsenic content*