SLOVENSKI STANDARD
oSIST prEN ISO 23674:2021

01-julij-2021

Kozmetika - Analizne metode - Neposredno določevanje živega srebra v sledovih v kozmetičnih izdelkih s termično razgradnjo - Atomska absorpcijska spektrometrija (analizator Hg) (ISO/DIS 23674:2021)

Cosmetics - Analytical methods - Direct determination of traces of mercury in cosmetics by thermal decomposition - Atomic absorption spectrometry (mercury analyzer) (ISO/DIS 23674:2021)

Kosmetische Mittel - Untersuchungsverfahren - Bestimmung von Quecksilberspuren in kosmetischen Mitteln mit integrierten Analysesystemen (ISO/DIS 23674:2021)

Cosmétiques - Méthodes d'analyse - Dosage direct des traces de mercure dans les cosmétiques par décomposition thermique - Spectrométrie d'absorption atomique (analyseur de mercure) (ISO/DIS 23674:2021)

Ta slovenski standard je istoveten z: **prEN ISO 23674**

ICS:

71.100.70	Kozmetika. Toaletni pripomočki	Cosmetics. Toiletries

oSIST prEN ISO 23674:2021 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 23674

ISO/TC **217**

Secretariat: **ISIRI**

Voting begins on:
2021-05-25

Voting terminates on:
2021-08-17

Cosmetics — Analytical methods – Direct determination of traces of mercury in cosmetics by thermal decomposition — Atomic absorption spectrometry (mercury analyzer)

ICS: 71.100.70

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 23674:2021(E)

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Published in Switzerland

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 217 *Cosmetics*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This standard specifies an analytical procedure for direct determination of traces of mercury in finished cosmetic products by thermal decomposition – atomic absorption spectrometry (mercury analyser).

Cosmetics — Analytical methods – Direct determination of traces of mercury in cosmetics by thermal decomposition — Atomic absorption spectrometry (mercury analyzer)

1 Scope

The aim of this document is to provide a procedure of quantification of mercury traces in cosmetic products that consumers might be exposed to in their usage. This method describes the determination of mercury traces in cosmetics by direct solid analysis with no need of external digestion. Total mercury (both inorganic and organic species) can be quantified either in solid or liquid samples.

This standard has been developed in parallel with another method of determination of traces of mercury in cosmetics: ISO 23821 Cosmetics – Analytical methods – Determination of traces of mercury in cosmetics by atomic absorption spectrometry (AAS) cold vapour technology after pressure digestion [1]. Knowing this, an interlaboratory test using either one or the other method was performed on same tailor-made cosmetic products in order to establish that both methods fulfilled the same requirements (see Annex B).

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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

Validation range: range of concentrations used for the method evaluation. It starts from the lowest concentration of the validation samples and ends at the highest concentration of the validation samples

Validated range: range of concentrations inside which the method performances are compliant with the method requirements, i.e. range of concentrations where the tolerance intervals are located inside the acceptance limits.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The described method uses integrated instruments allowing mercury traces determination and quantification. Samples are weighed with no need of any chemical sample preparation as they are thermally decomposed in the instrument (burned or ashed) in an oxygen flow at high temperature (between 650°C and 900°C). The combustion gases travel through a catalyst tube set at about 615°C. This step ensures conversion of interfering components to forms that do not interfere and that are subsequently flushed. The resulting mercury vapour is enriched on a downstream gold amalgamator and is then released as atomic vapour by rapid heating of the amalgamator at a temperature of 800°C to 900°C. The atomic vapour is passed into a measuring cuvette system and quantified by means of the

absorption at 253,7 nm. A wide dynamic range may be achieved by simultaneously passing mercury vapors through measurement cells of different lengths.

5 Reagents

WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to take appropriate measures for ensuring the safety and health of the operator prior to application of the standard and to fulfil local requirements for this purpose.

5.1 Ultrapure water, conductivity below 0,1µS/cm at 25°C according to Type 1 water specifications defined in ISO 3696 standard.

5.2 Hydrochloric acid, minimum w = 30 %, density = 1,15 g/mL

5.3 Diluted hydrochloric acid, produced by mixing hydrochloric acid (5.2) with ultrapure water (5.1) at a ratio of approximately 1+9 parts respectively.

5.4 Diluted nitric acid (0,1 mol/L).

NOTE If diluted nitric acid [2] is chosen to dilute the analyte stock solutions, it is recommended to add L-cysteine at 0,1 g/L [3].

5.5 Analyte standard stock solutions (Mercury), 1000 µg (Hg)/mL (commercially available)

5.6 Analyte standard stock solutions (Mercury), 10 µg(Hg)/mL (commercially available or freshly prepared by dilution in the same dilution medium as calibration solutions (5.3 or 5.4) of a more concentrated solution for example at 1000 µg/mL such as 5.5)

5.5 or 5.6 analyte standard solutions can be used for this standard according to their availability on the local market. Recommendations from the supplier of stock solutions regarding stability (expiry date and storage conditions) shall be carefully followed to avoid mercury loss.

6 Apparatus and equipment

WARNING — - All apparatus and equipment that come into direct contact with sample or solutions should be pre-cleaned with diluted hydrochloric acid (5.3) and rinsed with ultrapure water (5.1) to ensure the lowest analytical background. To prevent contamination and adsorption, do not use lab materials made with borosilicate glass.

Many instruments from several brands are available on the market and often marketed as “mercury analysers”. List of instruments that have been used for interlaboratory test in order to validate this standard is available on Annex A.

7 Calibration

7.1 General

The aim of this step is to build a calibration curve by introduction in the instrument of increasing mercury amounts. This calibration curve allows to get instrument response as a function of mercury amount (in ng). At least 5 calibration standards shall be used in a range including the expected amounts of mercury in the samples. Calibration of the instrument remains stable and is not mandatory prior to each series of analyses provided that QC requirements are met (10.2).

Due to the specificity of the technique towards mercury element, measurement of traces of mercury in samples is weakly affected by interferences and matrix effects. Since the technique is relatively insensitive to the matrix type, calibration can be performed either using in-house liquid (7.2) or solid (7.3) calibration standards. However, moisture and organic contents may affect quantification. Such differences between calibration standard and cosmetic samples shall be neutralized by optimizing drying times for moisture content, and combustion times and temperatures for quantity of organic material to be combusted or amount of interfering element needing catalytic conversion to a chemical form that does not interfere with mercury detection.

7.2 Liquid calibration standards

Calibration solutions should be prepared in either diluted hydrochloric acid (5.3) or diluted nitric acid containing L-Cysteine (5.4) to ensure stability of mercury. For laboratory convenience, calibration solutions may be prepared in other acid mixture solutions, provided the operator checks the stability of the mercury in that solution. Fresh calibration solutions should be prepared each time calibration is needed. Two solution calibration procedures are possible:

— Introduce increasing amounts of mercury in a constant volume using increasing concentrations of standard solution (Table 1).

— Introduce increasing amounts of mercury using increasing volumes of one or more standard solutions with a given mercury concentration (Table 2).

Below are examples for these two ways of performing calibration.

Table 1 — Example of calibration solutions using constant volumes of different standards

Liquid calibration standard solution	Part of 10 µg/g stock solution (mL) (5.6)	Part of dilution solution (mL) (5.3 or 5.4)	Mercury concentration (µg/g - ppm) in liquid calibration standards	Mercury amount in the boat (ng)
7.2.0 Calibration blank	-	10	Blank	0
7.2.1 Calibration solution 1	0,01	9,99	0,01	1
7.2.2 Calibration solution 2	0,02	9,98	0,02	2
7.2.3 Calibration solution 3	0,05	9,95	0,05	5
7.2.4 Calibration solution 4	0,1	9,9	0,1	10
7.2.5 Calibration solution 5	0,2	9,8	0,2	20
7.2.6 Calibration solution 6	0,5	9,5	0,5	50
7.2.7 Calibration solution 7	1	9	1	100
7.2.8 Calibration solution 8	2	8	2	200
7.2.9 Calibration solution 9	5	5	5	500

Table 2 — Example of calibration solutions using adjusted volumes of single standards

Liquid calibration standard solution	Part of 10 µg/g stock solution (mL) (5.6)	Part of dilution solution (mL) (5.3 or 5.4)	Mercury concentration (µg/g - ppm) in liquid calibration standards	Volume loaded in the boat (µL)	Mercury amount in the boat (ng)
7.2.10 Calibration blank	-	10	Blank	100	0
7.2.11 Calibration level 1	0,02	9,98	0,02	50	1
7.2.12 Calibration level 2	0,02	9,98	0,02	100	2
7.2.13 Calibration level 3	0,02	9,98	0,02	250	5