SLOVENSKI STANDARD
oSIST prEN ISO 23821:2021

01-marec-2021

Kozmetika - Analizne metode - Določevanje živega srebra v kozmetičnih izdelkih z atomsko absorpcijsko spektrometrijo (AAS) s tehniko hladnih par po razklopu pod tlakom (ISO/DIS 23821:2021)

Cosmetics - Analytical methods - Determination of traces of mercury in cosmetics by atomic absorbtion spectrometry (AAS) cold vapour technology after pressure digestion (ISO/DIS 23821:2021)

Kosmetische Mittel - Untersuchungsverfahren - Bestimmung von Quecksilberspuren in kosmetischen Mitteln durch Atomabsorptionsspektrometrie (AAS) Kaltdampftechnologie nach Druckaufschluss (ISO/DIS 23821:2021)

Cosmétiques - Méthodes d'analyse - Dosage des traces de mercure dans les cosmétiques par la technique de spectrométrie d'absorption atomique (SAA) de vapeur froide après digestion sous pression (ISO/DIS 23821:2021)

Ta slovenski standard je istoveten z: **prEN ISO 23821**

ICS:

71.040.50	Fizikalnokemijske analitske metode	Physicochemical methods of analysis
71.100.70	Kozmetika. Toaletni pripomočki	Cosmetics. Toiletries

oSIST prEN ISO 23821:2021 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 23821

ISO/TC **217**

Secretariat: **ISIRI**

Voting begins on:
2021-02-04

Voting terminates on:
2021-04-29

Cosmetics — Analytical methods — Determination of traces of mercury in cosmetics by atomic absorbtion spectrometry (AAS) cold vapour technology after pressure digestion

ICS: 71.100.70

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 23821:2021(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

European foreword

This document (prEN 23821:2020) has been prepared by Technical Committee CEN/TC 392 “Cosmetics”, the secretariat of which is held by AFNOR.

This document is a working document.

Cosmetics — Analytical methods — Determination of traces of mercury in cosmetics by atomic absorbtion spectrometry (AAS) cold vapour technology after pressure digestion

1 Scope

This European standard specifies a process for determination of mercury in cosmetics by means of cold vapour atomic absorption (AAS) with a prior pressure digestion. This process was validated by means of an interlaboratory test according to ISO 5725-2 [6] using e.g. lipstick, body lotion, toothpaste and eyeshadow, with a mercury concentration in the range of 0,110 mg/kg to 5,84 mg/kg. Statistical characteristics regarding this interlaboratory test are provided in Annex A, Table A.1.

This standard has been developed in parallel with ISO 23674. 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*

ISO 23674, *Cosmetics — Analytical Methods — Determination of mercury in cosmetics by integrated mercury analytical systems*

3 Principle

As a first step, the finished cosmetic product is digested in a closed container at high temperatures and pressure using mineral acids. Pressure digestion is carried out at a temperature of 200 °C obtained by means of microwave-assisted heating.

After digestion of the cosmetics the concentration of mercury is determined by quantification using the AAS cold vapour technology.

During mineralisation of the sample it is not possible to dissolve all cosmetics without residues, depending on their type and composition. In order to obtain comparable results, it is absolutely mandatory to comply with the conditions specified for this process.

The measurement solution is transferred to the reaction container of the mercury analysis unit. From there, mercury is rinsed out into the cuvette of the AAS instrument with the help of a carrier gas flow after reduction with divalent tin or sodium borohydride. Absorption at the mercury line of 253,7 nm is used as a measure for mercury concentration in the cuvette. By using a gold/platinum mesh (amalgam technology) for concentration of the rinsed-off mercury prior to measurement in the cuvette it is possible to achieve lower LOQs.

4 Reagents

The reagents and the water used shall be free of mercury to such an extent that the analysis is not impaired. Unless specified otherwise, pure-analysis chemicals shall be used and solutions are understood to be aqueous solutions. Use water conforming to Grade 1 of ISO 3696 (conductivity below 0,1 µS/cm at 25 °C).

4.1 Hydrochloric acid, minimum w = 30 %, density = 1,15 g/ml.

4.2 Nitric acid, minimum w = 65 %, density = 1,4 g/ml.

4.3 Diluted nitric acid, produced by mixing nitric acid (4.2) with water at a ratio of approximately 1 + 9 parts respectively.

4.4 Reducing agents, for example tin(II) chloride or sodium borohydride.

Alternating operation with both reducing agents (4.4.1 and 4.4.2) is not recommended. For this purpose, the appropriate information from the manufacturer of the instrument shall be followed.

The mass concentrations of the reducing agent solutions can vary, depending on the system. The corresponding data of the manufacturer of the instrument shall be complied with.

4.4.1 Tin(ll) chloride solution, for example mass concentration ρ = 100 g/l.

Weigh 50 g tin(ll) chloride, $SnCI_2{\cdot}2H_20$ in a 500 ml volumetric flask and dissolve in approximately 100 ml hydrochloric acid (4.1) and dilute to the mark with water. The solution shall be freshly prepared prior to use.

4.4.2 Sodium borohydride solution, for example ρ = 30 g/l.

Dissolve 3 g of sodium borohydride and 1 g of sodium hydroxide pellets in water and dilute with 100 ml water. The solution shall be freshly prepared every day and filtered prior to use.

WARNING — Compliance with the safety instructions is mandatory when working with sodium borohydride. Sodium borohydride forms acids when combined with hydrogen, which can result in an explosive air/hydrogen mixture. A fixed exhaust system shall be installed/present in the area where measurements are carried out.

4.5 Stabilization

The standard, calibration and sample digestion solutions are stabilized with hydrochloric acid (4.1). It is recommended to set a hydrochloric acid concentration of around ω = 1% in the solutions. Alternative stabilizing reagents can also be used (see Clause 9).

4.6 Mercury stock solution, mercury mass concentration of ρ = 1 000 mg/l.

The stock solution is commercially available. It is recommended to use certified stock solutions. As an alternative, the stock solution prepared from mercury(II) oxide can be stabilised using potassium dichromate[1] or solution prepared from mercury(II) chloride can be stabilised using nitric acid (4.2)[3].

4.7 Mercury standard solutions

Dilute the stock solutions to the concentrations required for calibration and add the necessary amount of stabilisation reagent (4.6), In doing so, select concentrations that the linear range of the reference

function is not exceeded. It is recommended to use at least 3 standard solutions with different concentrations.

The acid concentration in the standard solution shall correspond to the acid concentration of the measurement solution. Mercury standard solutions have a rather short shelf life, even at higher concentrations; therefore, they shall be freshly prepared every day.

4.8 calibration blank solution

The calibration blank solution shall contain water, the same amount of stabilisation reactant as the mercury standard solutions (4.7) per litre and the quantities of nitric acid (4.2) and hydrochloric acid (4.1) that correspond to the acid concentrations in the measurement solution.

5 Apparatus and equipment

For the determination of mercury all apparatus and equipment that come into direct contact with the sample and the solutions used shall be thoroughly pre-treated to ensure minimisation of contamination. The following steps are recommended for cleaning: Rinse with drinking water, treat with a scouring agent solution, repeat rinsing with drinking water and soak in diluted nitric acid (4.3) over night or a prolonged period. Prior to use, rinse the apparatus with ultrapure water and dry. Steaming of chemically inert vessels and containers (e.g. made of quartz glass) using nitric acid (4.2) is an effective cleaning method and is regularly used in element trace analysis. To prevent contamination and adsorption, do not use lab materials made with borosilicate glass.

5.1 Digestion vessels.

Use commercially available, safety-tested pressure vessels and inserts made of acid resist and, low-contamination materials. The assembled vessels shall be able to safely withstand temperatures up to at least 200 °C and pressures up to at least 40 bar.

NOTE Dedicated digestion vessels are recommended for the digestion of cosmetic samples, which may have high levels of elements to be determined. To avoid memory effects, perform a blank digestion to clean vessels after digesting highly loaded samples, before digesting sequent samples.

5.2 Microwave assisted digestion instruments.

Microwave-heated systems shall be equipped with a temperature measurement unit, which simultaneously regulates the power control of the microwave. Reliable temperature measurement is obtained e.g. through measurement sensors inserted into the pressure vessel. Only use microwave-assisted digestion instruments equipped with temperature sensors and calibrate the temperature sensor before use.

5.3 **Membrane filter,** pore size: 0,45 µm.

The membrane filter used shall be inert with regard to the acid concentration of the measurement solution and shall not bring any contamination into the measurement solution or adsorption of the analytes. Several types of membrane material are commercially available (e.g. PTFE, PP) and their fit for purpose shall be verified by means of appropriate measurements (e.g. blanks, QC samples).

5.4 Atomic absorption spectrometer, optionally available with background compensation function and including accessories for cold vapour technology or amalgam technology.

Flow injection systems can be used as an alternative of manual processes.

5.5 Element-specific light for mercury

Measurement at 253,7 nm.

6 Procedure

6.1 General

WARNING — The use of this standard can involve hazardous materials, operations and equipment. This standard does not 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 personnel prior to application of the standard and to fulfil statutory requirements for this purpose.

During all process steps it shall be ensured that there are no losses of analyte and that contamination is kept as low as possible.

6.2 Preparation of samples

Before the digestion of the sample a suitable preparation needs to be carried out (e.g. homogenizing, mixing, crushing[13]). After homogenization thoroughly clean the devices in order to rule out contamination of the subsequent sample. The sample preparation step shall ensure a homogeneous starting material for a weighed sample quantity.

6.3 Pressure assisted digestion

WARNING 1 Depending on the degree or reactivity of the sample, it may be required to weigh in lower quantities than specified in Section 6.3.1 in order to prevent extreme reactions or explosions. It shall be taken into account that digestion of samples with high carbon contents (e.g. carbohydrates, fats, oils, waxes) may cause explosions. Alcohols or solvents in combination with concentrated nitric acid may cause delayed severe reactions already at room temperature. Therefore it is highly recommended to gently evaporate all volatile components before adding the acid (Section 6.3.2).

WARNING 2 Samples that are not covered by acid can cause local overheating of the digestion vessel and thus lead to local melting and subsequent bursting of the digestion container. Prior to digestion, ensure that the entire sample is fully covered by the acid mixture.

Temperature and pressure inside the vessels shall be carefully controlled to ensure a proper digestion. To avoid differences in temperature and pressure among vessels, one should only digest samples with similar composition in the same microwave-assisted digestion batch.

6.3.1 Preparation of sample by digestion – General case

Precisely weigh about 200 mg of sample into a digestion container.

Add 1 ml of water and thoroughly mix with a shaking device until the sample is completely suspended in the water.

Add 5 ml nitric acid (4.2) to the mixture and mix again. The sample should be completely covered with the solution. Allow the mixture to rest in a closed digestion vessel to ensure that the preliminary reaction takes place. Depending on the reactive behaviour of the sample the duration of the preliminary reaction can require resting periods of 30 minutes up to overnight.

Then add 1 ml of hydrochloric acid (4.1) and briefly mix. After addition of the hydrochloric acid, the pressure vessel shall be closed and sealed immediately to make sure that the formed chlorine gas is available for the reaction and does not evaporate.

6.3.2 Preparation of sample by digestion – Specific cases

— For highly water-based cosmetic products, such as lotion, milky lotion, cleanser or micellar water, a sample mass could reach 400 mg. In this case no addition of water is required before addition of acids (6.3.1).

— For all the other specific cases, sample mass can be adapted but the ratio between sample mass and acid volumes (6.3.1) shall not be changed.

In case of high volatile content products, it is highly recommended for safety reason to completely remove volatile portions through a careful heat-up following the loss of sample weight (e.g. in a water bath at 60 °C) after weighing them into the digestion container but prior to adding the acid (6.3.1). In this context, special care shall be taken to prevent losses of the specific elements.

Due to sample heterogeneity concern, a weight below 100 mg is not recommended.

6.3.3 Microwave digestion procedure

WARNING — During all steps of the digestion process the manufacturer's safety information shall be accurately followed.

Process the samples using a 3 step heating program:

a) ramp the heat up from room temperature to 200 °C in 30 min;

b) hold the temperature at 200 °C for 30 min;

c) cool down to 50 °C, before removing the vessels from the microwave.

It is mandatory to maintain a temperature of 200 °C for 30 min to obtain comparable results, since complete digestion is not possible for all types of samples.

WARNING — Depending on reactivity of the sample, it may be necessary to select a lower heat-up rate than specified in order to prevent extreme reactions or explosions.

6.3.4 Preparation of measurement solutions

Cool the vessels to room temperature before opening. Follow any manufacturer's provisions. Quantitatively transfer the digestion solution to vessel and dilute to 20 ml with water. Further dilute the solution 1+9 with water.

NOTE A different intermediate dilution volume can be used when the digest solution is quantitatively transferred from the digestion vessel as long as the final dilution remains unchanged. For example, transfer the digestion solution and dilute to 50 ml with water then further dilute this solution 1 + 3 with water.

Further dilution of the samples can be performed if required to bring analyte concentration to within the linear calibration range. Ensure that the measurement solution obtained in this way contains the same acid concentrations as the calibration solutions prepared according to 4.10.

Remove any residue by decanting or filtering the final solution by a membrane filter (5.3).

The stability of the mercury in the digestion solution depends on the type and concentration of the acid used for the digestion, the vessel materials used for storage and the mercury concentration. It is therefore recommended to stabilize the digestion solution, e.g. B. with hydrochloric acid (4.1).

6.4 Atomic absorption spectrometry (cold vapour AAS)

6.4.1 Spectrometry settings

For development of a measurement program, at first adjust the instrument according to manufacturer's operating instructions. Subsequently, optimize the settings with particular focus on the gas flow and the fed quantities of tin(II) chloride (4.4.1) or sodium borohydride (4.4.2).