INTERNATIONAL STANDARD

ISO 21392

First edition
2021-08

Corrected version
2021-12

Cosmetics — Analytical methods — Measurement of traces of heavy metals in cosmetic finished products using ICP/MS technique

Cosmétiques — Méthodes d'analyse — Mesurage des éléments traces métalliques par ICP-MS dans les produits cosmétiques finis

Reference number
ISO 21392:2021(E)

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ISO 21392:2021
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Foreword

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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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This document was prepared by Technical Committee ISO/TC 217, *Cosmetics,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 392, *Cosmetics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

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.

This corrected version of ISO 21392:2021 incorporates the following corrections:

— the definition of Formula (1) has been corrected.

Introduction

This document specifies an analytical procedure for the determination of trace levels of heavy metals (e.g. chromium, cobalt, nickel, arsenic, cadmium, antimony and lead) in finished cosmetic products by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion of the sample. This type of analytical procedure is widely described in other areas such as environment [9][10][11], food[9][10][11] and pharmaceutical industry [12][13][14][15]. While it maximizes the detection of trace levels present in cosmetic products, it does not provide any methodology to directly evaluate systemic exposure of the consumers.

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Cosmetics — Analytical methods — Measurement of traces of heavy metals in cosmetic finished products using ICP/MS technique

1 Scope

This document provides a method for quantification of trace levels of heavy metals in cosmetic products.

This document refers only to chromium, cobalt, nickel, arsenic, cadmium, antimony and lead. The methodology can apply to other elements, however, it is the responsibility of the analyst to demonstrate that it fits that purpose.

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

For the purposes of this document, the following terms and definitions apply.

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 http://www.electropedia.org/

3.1
validation range
range from the upper to the lower concentration of samples used for the method evaluation

3.2
validated range
range of concentrations between the upper and lower levels that the method performance has been demonstrated to be compliant with the method requirements

4 Principle

Trace levels of heavy metals in cosmetic products are quantified by ICP-MS measurement of the solutions following digestion of the cosmetic products. Digestion takes place with mineral acids in sealed vessels heated to 200 °C by microwaves, producing high pressures.

In the sample preparation procedure, cosmetic ingredients are digested by using a nitric acid/hydrochloric acid mixture allowing the trace levels of heavy metal to be solubilized for measurement. It is possible that some cosmetic inorganic ingredients, such as silica or titanium dioxide, are not completely digested under the conditions of this document and that heavy metal confined in such ingredients are not fully extracted. However, the level of heavy metal trapped in these inorganic materials is not considered to significantly contribute to the exposure level of consumers to these heavy metals. The use of ICP-MS ensures reliable measurement of trace levels of heavy metals due to its proven high sensitivity and selectivity.

In order to obtain comparable results, it is absolutely mandatory to follow all the conditions linked to the digestion of the samples.

5   Reagents

Care shall be taken to assure that the analyte background in the reagents and the water used is negligible and will not interfere analysis. Unless specified otherwise, solutions are understood to be aqueous solutions.

5.1   **Water**, conforming to Grade 1 of ISO 3696 (conductivity below 0,1 µS.cm^{-1} at 25 °C [16]).

5.2   **Nitric acid**, minimum mass fraction $w = 60$ %, density = 1,38 g/ml, suitable for ICP-MS analysis.

5.3   **Hydrochloric acid**, minimum $w = 30$ %, density = 1,15 g/ml, suitable for ICP-MS analysis.

5.4   **Internal standard stock solutions**

For storage and stability conditions of the internal standard stock solutions, follow the specifications of the suppliers. The internal standard solution should contain a certified, or traceable to a certified reference material (CRM) content.

5.4.1   **Rhodium stock solution**, 1 000 mg/l.

5.4.2   **Lutetium stock solution**, 1 000 mg/l.

5.5   **Analytes stock solutions [chromium (Cr), cobalt (Co), nickel (Ni), arsenic (As), cadmium (Cd), antimony (Sb) and lead (Pb)]** 1 000 mg/l for each element.

Commercially available single element or mixed stock solutions with a known certified, or traceable to a CRM content can be used. The used stock solutions shall not contain other elements that could interfere with the analytes to be quantified.

5.6   **ICP-MS tune solution**, containing, for example, Ce, Co, Li, Mg, Tl and Y (1 µg/l) according to instrument manufacturer's recommendations.

6   Apparatus and equipment

All apparatus and equipment that come into direct contact with sample or solutions shall be pre-cleaned with diluted nitric acid (see 7.2) and rinsed with ultrapure water (5.1) to ensure the lowest analyte background. To prevent contamination and adsorption, do not use lab materials made with borosilicate glass.

6.1   Digestion vessels

6.1.1   General case

Use commercially available, safety-tested pressure vessels and inserts made of acid-resistant, 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.

It is recommended to keep digestion vessel used only for cosmetic analysis purposes to minimize cross contamination. In addition, the digestion vessels shall always be thoroughly washed after each use. After digesting highly loaded samples, it is recommended to perform a blank digestion with same conditions reported in this document to clean vessels before digesting subsequent samples.

6.1.2 Special care for quantification of antimony

Antimony has been reported to be prone to adsorb on the inner surface of walls of vessels and adsorption is empirically known to happen more frequently when using vessels with extensive period of usage.

Use only digestion vessels with minimal surface roughness to prevent antimony adhesion to the vessel surface[7],[16]. Thorough examination of the vessels shall be performed before their use to control for any scratch or damage or any deposit. If there is any doubt on the integrity of vessels inner surface, consider replacing with brand new digestion vessels.

This issue has been reported to more often happen when polytetrafluoroethylene digestion vessels are used because they can be easily damaged. Quartz containers are recommended because they are usually more resistant to attrition. Damage is also more easily visible because of their transparency. However, polytetrafluoroethylene vessels without any scratch or damage on its inner surface or any deposit are appropriate.

In case of doubt on the possible adsorption of antimony on vessels' walls (e.g. if significant deviations from the target value are observed), a control test is described in 8.4.2.

6.2 Microwave-assisted digestion instruments.

Microwave-heated systems shall be equipped with a temperature measurement unit, which simultaneously regulates the power control of the microwave. Calibrate the temperature sensor before use of at least once a year.

6.3 Membrane filter, 0,45 µm pore size.

The membrane filter used shall be suitable for inorganic traces analysis. It 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 [polytetrafluoroethylen (PTFE), polypropylene (PP), etc.] and their fit for purpose shall be verified by means of appropriate measurements [blanks, quality control (QC) samples, etc.].

6.4 ICP-MS

Mass spectrometer with inductively coupled argon plasma is composed of a sample introduction and an atomisation system, as well as an instrument control and evaluation unit. To prevent interferences with the masses of the heavy metals chromium, nickel, cobalt, arsenic and cadmium, use a mass spectrometer that is capable of compensating or minimizing such interferences (e.g. collision and/or reaction cell, resolution above 3 000, alternatively corrective formulae for higher concentrations).

7 Preparation of standards solutions

7.1 General

For all the solutions, the terminology "part" in the standard refers to either volume or weight. That means that standards and samples can be diluted by volume or weight. However, it should be consistent for both standards and samples.

7.2 Diluted nitric acid

Produced by mixing nitric acid (5.2) with pure water (5.1) at a ratio of approximately 1 + 9 parts respectively.

7.3 Diluting solution

The diluting solution shall have the same acid composition (total content and acid ratio) as the analytical solution (the diluted digest solution). This solution should contain:

— 2,5 parts of nitric acid (5.2);

— 0,5 part of hydrochloric acid (5.3);

— 97 parts of water (5.1).

7.4 Internal standard solutions

7.4.1 General

The internal standards (IS) selected should cover all the mass range of the considered analytes and have similar ionisation energy to the heavy metal for which it is used for correction purposes. It shall also be checked that the native concentration of the internal standards to be analysed is negligible and that they are not interfered by sample constituents.

Rhodium and lutetium have proven to be suitable as internal standards. Samples should be checked for negligible native concentrations and that the sample constituents do not interfere with it.

Rhodium is suitable for the determination of chromium, cobalt, nickel, arsenic, cadmium and antimony, whereas lutetium is suitable for the determination of lead. Alternatively, other elements may be used (for example indium or iridium). Scandium, however, is not suitable as IS due to calcium interferences. An IS with a mass (m/z) below 100 is not recommended because it may suffer from interferences from matrix components.

Internal standard solutions may be added in each sample and calibration solution at the same concentration or may be added through an online Y-fitting to a pump tube.

The concentration of the internal standard solutions shall be included in the range 1 mg/l to 2 mg/l. In 7.4.2 and 7.4.3, a concentration of 1 mg/l has been used for all the calculations.

7.4.2 Rhodium standard solution, 1 mg/l

Dilute the rhodium stock solution (5.4.1) 1 + 999 with diluting solution (see 7.3). This internal standard solution is stable at room temperature for 6 months.

Indium or iridium may also be used as internal standards.

7.4.3 Lutetium standard solution, 1 mg/l

Dilute the Lutetium stock solution (5.4.2) 1 + 999 with diluting solution (see 7.3). This internal standard solution is stable at room temperature for 6 months.

Indium or iridium may also be used as internal standards.

7.5 Standard solutions

7.5.1 General

The concentrations of these standard solutions are examples and should be adjusted to the specific conditions in the laboratories.

7.5.2   High concentration mixed standard solution, 10 mg/l

Dilute 100 times the analytes stock solution(s) (5.5) by adding:

— in the case of single analyte stock solutions, 1 part of each of these 7 solutions to 93 parts of the diluting solution (see 7.3);

— in the case of mixed stock solution, add 1 part of this solution to 99 parts of the diluting solution (see 7.3).

This high standard solution is stable at room temperature for 6 months.

7.5.3   Low concentration mixed standard solution, 0,1 mg/l

Dilute 100 times the high concentration standard solution (see 7.5.2) by adding 1 part of this solution to 99 parts of the diluting solution (see 7.3). This low standard solution is stable at room temperature for 3 months.

7.6   Calibration blank solution

The calibration blank solution corresponds to the matrix solution without any analyte of interest. Generally, it corresponds to the diluting solution with the suitable concentration of the appropriate internal standards if not added via a Y-fitting during the measurement.

7.7   Calibration solutions

Mixed calibration solutions are prepared by diluting the low concentration mixed standard solution (see 7.5.3) with the diluting solution (see 7.3) to levels in the linear range of the instrument and within the targeted concentration range. Include a suitable concentration of the appropriate internal standards, or add online the internal standards by means of pumping into the sample flow through a Y-fitting. At least 3 calibration solutions with various concentrations should be prepared. These calibration solutions shall be prepared daily.

Examples of preparation procedure of calibration solutions are detailed in Table 1 (with addition of the internal standards in all the calibration solutions) and Table 2 (with on line addition of the internal standards via a Y-fitting).

Table 1 — Example of calibration solutions of the ICP-MS — Addition of the internal standards in every calibration solution

Calibration solution	Part of low concentration mixed standard solution (7.5.3)	Part of rhodium standard solution (7.4.2)	Part of lutetium standard solution (7.4.3)	Part of the diluting solution (7.3)	Analyte concentration in the calibration solution (µg/l)
Calibration blank	0	2	2	496	0
Calibration solution 1	2,5	2	2	493,5	0,5
Calibration solution 2	5	2	2	491	1
Calibration solution 3	10	2	2	486	2
Calibration solution 4	25	2	2	471	5
Calibration solution 5	50	2	2	446	10