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**Cosmetics — Analytical methods —  
Direct determination of traces of  
mercury in cosmetics by thermal  
decomposition and atomic absorption  
spectrometry (mercury analyser)**

*Cosmétiques — Méthodes d'analyse — Dosage direct des traces  
de mercure dans les cosmétiques par décomposition thermique et  
spectrométrie d'absorption atomique (analyseur de mercure)*

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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](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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](http://www.iso.org/iso/foreword.html).

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](http://www.iso.org/members.html).

## Introduction

This document has been developed in parallel with ISO 23821<sup>[1]</sup>. 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](#)).

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# Cosmetics — Analytical methods — Direct determination of traces of mercury in cosmetics by thermal decomposition and atomic absorption spectrometry (mercury analyser)

## 1 Scope

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

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

## 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 <https://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

Note 1 to entry: The validated range shall not be confused with the *validation range* (3.1); it can be smaller.

## 4 Principle

The described method uses integrated instruments allowing mercury traces 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 through a measuring cuvette system. The quantification occurs thanks to absorption at 253,7 nm. A wide dynamic range may be achieved by simultaneously passing mercury vapours through measurement cells of different lengths.

## 5 Reagents

**WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not address all the safety risks associated with its use. It is the responsibility of the analyst to take all appropriate measures for ensuring the safety and health of the personnel prior to application of the document.**

**5.1 Water**, conforming to Grade 1 of ISO 3696 (conductivity below 0,1  $\mu\text{S}\cdot\text{cm}^{-1}$  at 25 °C).

**5.2 Hydrochloric acid**, minimum mass fraction  $w = 30\%$ , density = 1,15 g/ml, suitable for elemental analysis.

**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**, prepared by diluting nitric acid of minimum mass fraction  $w=60\%$ , density = 1,15 g/ml, suitable for ICP-MS with pure water (5.1) at a ratio of 1+9 respectively.

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

**5.5 Analyte standard stock solutions (mercury)**, 1 000  $\mu\text{g}/\text{ml}$  (commercially available).

**5.6 Analyte standard stock solutions (mercury)**, 10  $\mu\text{g}/\text{ml}$  [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 1 000  $\mu\text{g}/\text{ml}$  such as 5.5].

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

## 6 Apparatus and equipment

All apparatus and equipment that come into direct contact with samples 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 laboratory materials made up of borosilicate glass. The use of diluted nitric acid (5.4) is also permitted. To check the efficiency of the cleaning step, a blank measurement can be performed prior to the analytical sequence.

Elemental mercury analyser with instrument control unit and sample containers made of suitable materials (e.g. nickel, quartz glass or ceramic) are used. The sample containers usually have a capacity of approximately 500 mg of solid sample or 500  $\mu\text{l}$ , 1 000  $\mu\text{l}$  and 1 500  $\mu\text{l}$  of liquids. Mercury analysers are equipped with an element-specific lamp for mercury. The mercury line of 253,7 nm is used.

Many instruments from several brands are available on the market and often marketed as “mercury analysers”. The list of instruments that have been used for the interlaboratory test validating this document is given in [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 quality control (QC) requirements are met (see 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 (see 7.2) or solid (see 7.3) calibration standards. However, moisture and organic contents may affect quantification. Such differences between calibration standard and cosmetic samples shall be neutralized. For differences in moisture content, optimize the drying times. For differences in quantity of organic material to be combusted, optimize the time and temperature of combustion. For differences in interfering element, optimize time for the catalytic conversion of elements needing catalytic conversion into 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 (see Table 1).
- Introduce increasing amounts of mercury using increasing volumes of one or more standard solutions with a given mercury concentration (see 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 in µg/g (ppm) in liquid calibration standards	Mercury amount in the boat (ng)
Calibration blank	—	10	Blank	0
Calibration solution 1	0,01	9,99	0,01	1
Calibration solution 2	0,02	9,98	0,02	2
Calibration solution 3	0,05	9,95	0,05	5
Calibration solution 4	0,1	9,9	0,1	10
Calibration solution 5	0,2	9,8	0,2	20
Calibration solution 6	0,5	9,5	0,5	50
Calibration solution 7	1	9	1	100
Calibration solution 8	2	8	2	200
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 in µg/g (ppm) in liquid calibration standards	Volume loaded in the boat (µl)	Mercury amount in the boat (ng)
Calibration blank	—	10	Blank	100	0
Calibration level 1	0,02	9,98	0,02	50	1
Calibration level 2	0,02	9,98	0,02	100	2
Calibration level 3	0,02	9,98	0,02	250	5
Calibration level 4	0,2	9,8	0,2	50	10
Calibration level 5	0,2	9,8	0,2	100	20
Calibration level 6	0,2	9,8	0,2	250	50
Calibration level 7	2	8	2	50	100
Calibration level 8	2	8	2	100	200
Calibration level 9	2	8	2	250	500

Recommendations from the supplier of standard solutions regarding stability (expiry date and storage conditions) shall be followed to avoid mercury loss.

### 7.3 Solid calibration standards

Solid certified reference materials (CRM), with known concentrations of mercury can be used in order to calibrate the instrument prior analysing the samples (see Table 3). Since the technique is relatively insensitive to the matrix type, these CRM can be chosen among a wide variety of nature that are not necessarily cosmetic matrices (sediments, rocks, sewage sludge, etc.).

To ensure similar analysis behaviour, CRMs with sufficient homogeneity shall be selected so that sample sizes similar to those of test samples may be used.

Table 3 — Example of calibration using solid CRMs

Calibration standards	Name	Mercury content in the solid sample (µg/g - ppm)	Approximate weigh of solid standard loaded in the boat (mg)	Approximate amount of mercury in the boat (ng)
Calibration blank	Blank	0	0	0
Calibration solid 1	SRM 2685c	0,1494	13,3	2
Calibration solid 2	SRM 2685c	0,1494	33,5	5
Calibration solid 3	SRM 2685c	0,1494	66,9	10
Calibration solid 4	SRM 2702	0,4474	44,7	20
Calibration solid 5	SRM 2702	0,4474	111,8	50
Calibration solid 6	ERM-EC680m	2,56	39,1	100
Calibration solid 7	ERM-EC680m	2,56	78,1	200
Calibration solid 8	ERM-EC680m	2,56	195,3	500

Recommendations from the supplier of certified reference materials regarding stability (expiry date and storage conditions) shall be followed to avoid mercury loss. It is advised to select CRM with low uncertainty on the certified content.

## 8 Procedure

### 8.1 General

During all the process steps it shall be ensured that there is no loss of mercury and that contamination is kept as low as possible.

### 8.2 Preparation of samples

#### 8.2.1 General recommendations

Homogenize the sample using a suitable device, thoroughly cleaned to avoid contamination. Ensure that the weighed sample portion is homogeneous.

#### 8.2.2 General of samples

Precisely weigh 0,1 g of sample (0,05 g to 0,200 g) into sample boats. Place each boat onto the instrument's carousel.

Some instrument suppliers recommend adding additives (e.g. activated alumina, sodium carbonate) to the samples to avoid potential scatter.

## 9 Instrument parameters

Operators shall follow recommendations on technical parameters and maintenance guidelines from instrument supplier. The procedure can be divided in four major steps; drying, decomposition, purge and amalgam step, for which parameters can be controlled.

Instrument parameters can be adjusted by the operator in order to maximize intensity and stability of the signal. To adjust instrument parameters, it is recommended to use samples of known concentrations of traces of mercury and similar physicochemical nature as the sample that will be further analysed. Drying and decomposition parameters, such as time and temperature, can be adapted according to the nature of the sample, as long as they are included in the range of experimental conditions validated by the laboratory.

Because instrument parameters are linked, according to the instrument, several conditions can be used to perform an accurate mercury quantification. Experimental conditions reported by laboratories participating to the interlaboratory test have proven to be suitable for mercury traces quantification in cosmetic products and are reported in [Table A.4](#).

## 10 Quality control of the analysis

### 10.1 General

Recovery and relative standard deviation (RSD) mentioned in [10.2](#) are acceptance criteria obtained by a single laboratory and enabling to assess the quality of the measurement. This intralaboratory variability shall be lower than the total error (see [Clause 12](#)) of the method ( $\pm 30\%$ ) that has been determined by the means of interlaboratory ring test and detailed in the [Annexes A](#) and [B](#).

### 10.2 Quality control procedure

#### 10.2.1 Analysis blanks

Analysis blanks shall be regularly analysed to verify that no carry-over exists. Mercury content in the blank should be low enough so that the measurement results are not significantly changed through subtraction of the blank value. Wheat flour sample with no detectable mercury content, or even an