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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

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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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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.

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.

Introduction

This document has been developed in parallel with ISO 23821[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).

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 µS.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[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), 1 000 µg/ml (commercially available).

5.6 Analyte standard stock solutions (mercury), 10 µg/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 µg/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 µl, 1 000 µl and 1 500 µl 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)	Approximative weigh of solid standard loaded in the boat (mg)	Approximative 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 (±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 empty boat can be used as a blank. Blanks can also be added after the highest calibration standard or after a sample suspected to get high mercury content.

10.2.2 Quality control samples

Quality control (QC) sample is a sample with a known content of mercury similar to the sample to be tested. It is used to evaluate the whole analytical procedure for effectiveness by comparing the determined value with the reference value. This QC sample can be a well characterized material or a certified reference material (CRM).

Before starting a new series of analyses, the calibration shall be checked by analysing one or several quality controls (e.g. CRM). If discrepancies appear during the measurement, if maintenance work has been carried out, if the location of the instrument has been changed or whenever acceptance criteria of quality control in the laboratory are not met, a new calibration should be performed.

QC samples shall be analysed periodically throughout the series of sample measurements (e.g. every 10 samples). Acceptance criteria of quality control sample determined value are defined by the operators[4], according to their experience and can be notably designed by the operating laboratories thanks to control charts[5]. Recovery acceptance criterion commonly used for a QC in a single laboratory is ±20 %. If the value is not in the pre-defined range, it is advised to recalibrate the instrument and re-analyse the samples measured since the last acceptable QC determined value. If after these steps, recovery acceptance criteria on QC are still not met, it is recommended to perform instrument maintenance.

10.2.3 Replicates

Mercury content in cosmetics shall be quantified by the analysis of a minimum of 2 replicates per sample, to evaluate the sample homogeneity and precision. If the relative standard deviation between the replicates is above 20 %, repeat the analysis.

11 Calculation

Calculate the mercury content w in cosmetic samples using Formula (1):

$$w = \frac{a}{m} \tag{1}$$

where

w is the mercury content in the sample, mg/kg;

a is the mercury quantity in the sample boat, ng, determined from the calibration curve;

m is the test portion in the boat, mg.

12 Method performance

Method performance has been determined by the mean of the accuracy profile methodology (as described in ISO/TS 22176[6]) that enables to evaluate the global precision and the validated range for mercury quantification. Taking into account all the results obtained during the method validation interlaboratory tests, the acceptance limit has been set to ±30 %. All the accuracy profiles are presented in Annex A and Annex B. As a consequence, the analytical procedure in this standard has been validated in the range between 0,15 mg/kg (ppm) and 2,5 mg/kg (ppm) of mercury in cosmetics.

13 Test report

The test report should contain the data according to ISO/IEC 17025[4] and at least the following information:

a) all information necessary for the identification of the sample (kind of sample, origin of sample, designation);

b) a reference to this document, i.e. ISO 23674:2022;

c) the date and type of sampling procedure (if known);

d) the date of receipt;

e) the date of test;

f) the test results and the units in which they have been expressed;

g) any particular points observed in the course of the test;

h) any operations not specified in the method or regarded as optional, which might have affected the results.

Annex A
(informative)

Performance of the method determined by the accuracy profile methodology

A.1 General

To assess performance of the method described in this document, two independent interlaboratory tests have been set up.

A.2 Interlaboratory tests set up

In a first ring test, a set of 4 samples have been first analysed by several laboratories in triplicate. Those samples consisted in several cosmetic matrices containing increasing known amounts of mercury only originating from a solid certified reference material (marine sediment, ERM-CC580, 132 ± 3 mg(Hg)/kg (ppm)). Cosmetic products bases, without any source of mercury, have been prepared and controlled by using this document. Then, precise amounts of solid certified reference material (CRM) have been dispersed in the bases along with colorants with no detectable level of mercury. Cosmetic bulks were then thoroughly homogenized to ensure the proper distribution of the CRM. Each of the 4 bulks (see Table A.1) has then been aliquoted in several containers, every aliquot controlled prior to sending to 14 participating laboratories for analysis using various integrated mercury analysers (see A.4). Stability/homogeneity of the samples was tested over a month before shipment to participating laboratories, either control laboratories or private companies.

Table A.1 — Samples sent for first interlaboratory testing

Cosmetic product	Mercury content in mg/kg (ppm)
Face cream	0,50
Foundation	1,00
Lipstick	1,51
Mascara	2,50

In a second ring test, 3 cosmetic samples have been analysed by several laboratories in triplicate. Those samples consisted of tailor-made lipsticks with known contents of mercury that were only originating from a solid certified reference material [river sediment, ERM-CC020, 27,4 mg (Hg)/kg ± 0,6 mg (Hg)/kg (ppm)].

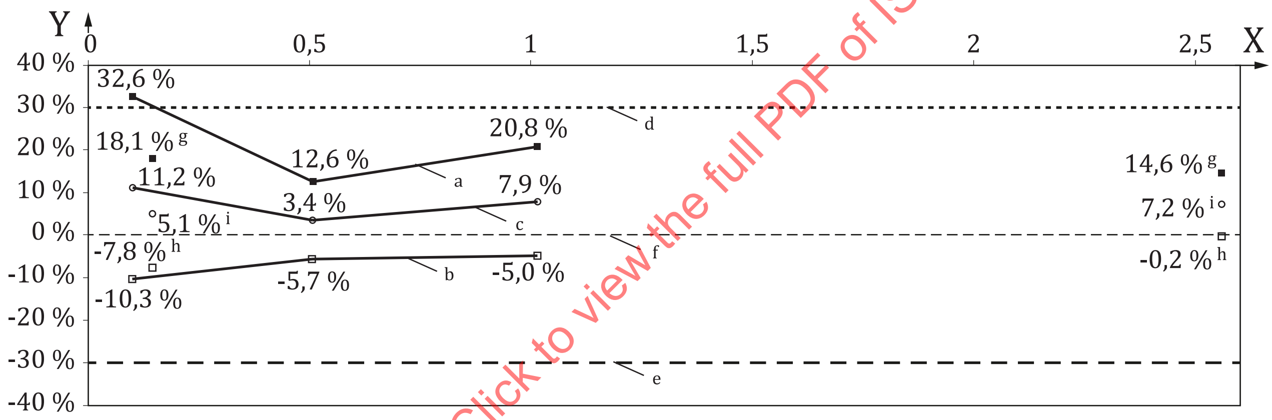
A lipstick base without any source of mercury has first been prepared and controlled by using this document. Then precise amounts of the solid CRM have been dispersed in the lipstick base along with colorants with no detectable level of mercury. Lipstick sample bulks were then thoroughly homogenized to ensure the proper distribution of the CRM. Three lipstick bulks with different amounts of dispersed CRM (corresponding to 3 different Hg levels) have been created and their mercury content controlled. Every of the 3 lipstick bulks has then been aliquoted in several containers, every aliquot controlled prior to sending to 9 participating laboratories for analysis.

Table A.2 — Samples sent for second interlaboratory testing

Cosmetic product	Mercury content mg/kg (ppm)
Lipstick sample 1	0,10
Lipstick sample 2	0,51
Lipstick sample 3	1,01

A.3 Results and statistical parameters from the interlaboratory tests:

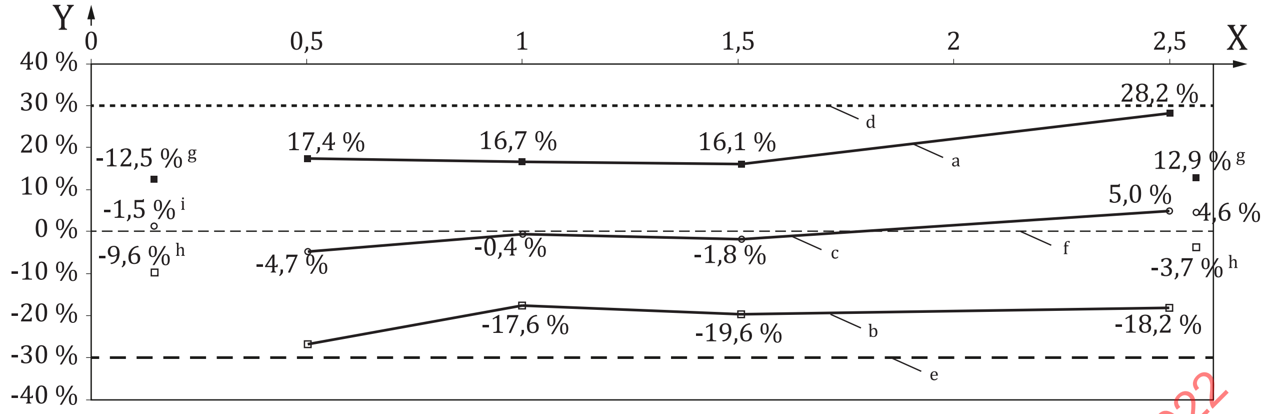
The evaluation of the method performance by the mean of the accuracy profile methodology (as described in ISO/TS 22176[6]) has been performed from data generated by the participating laboratories on the ring test samples. The complementary accuracy profiles obtained for the first (see Figure A.1) and second (see Figure A.2) interlaboratory ring tests are presented below. They enabled to validate the analytical methodology described in this standard for the ranges 0,15 mg/kg (ppm) to 1 mg/kg (ppm) (second ring test, Figure A.1) and 0,5 mg/kg (ppm) to 2,5 mg/kg (ppm) (first ring test, Figure A.2) with an acceptance limit of ±30 %.



Key

X mercury concentration (mg/kg)
Y accuracy (%)
a relative high tolerance limit
b relative low tolerance limit
c mean relative bias
d high acceptance limit
e low acceptance limit
f zero level
g RHTL_CRM
h RLTL_CRM
i MRB_CRM

Figure A.1 — accuracy profile for mercury validating this document in the range 0,15 mg/kg to 1 mg/kg (ppm)



Key

X	mercury concentration (mg/kg)
Y	accuracy (%)
a	relative high tolerance limit
b	relative low tolerance limit
c	mean relative bias
d	high acceptance limit
e	low acceptance limit
f	zero level
g	RHTL_CRM
H	RLTL_CRM
i	MRB_CRM

Figure A.2 — Accuracy profile for mercury validating this document in the range 0,5 mg/kg to 2,5 mg/kg (ppm)

As an information, to complement the evaluation of the method performance through accuracy profile methodology, the statistical evaluation according to ISO 5725-2[7] has been performed.

In addition to the overall mean value, the results of this evaluation are the reproducibility standard deviation *s*R and the repeatability standard deviation *sr*.

The reproducibility standard deviation *s*R characterizes the total variability of the measurement values, taking into account the variability between different laboratories. The repeatability standard deviation *sr* describes the variability within one laboratory and under constant measuring conditions (repeatability conditions).

From the reproducibility and repeatability standard deviation, the reproducibility limit *R* and the repeatability limit r are calculated. The reproducibility limit R describes the maximum expected deviation between two measured values from different laboratories for the same sample. For larger deviations, it can be assumed that either different samples were used or an error occurred during the measurement. The repeatability limit r describes the maximum expected deviation between two measured values under repeatability conditions, which were thus realized in the same laboratory shortly after each other.

The Horwitz function is often used to calculate the theoretically expected reproducibility standard deviation. Together with the resulting HORRAT value they are often used as a general criterion for the efficiency of a method as a function of the measured concentration. Here, a HORRAT value significantly larger (or smaller) than 1 means that the reproducibility standard deviation achieved in the interlaboratory test is significantly greater (or smaller) than the expected theoretical standard deviation (Horwitz standard deviation). In inter laboratory tests, HORRAT values up to 2 are generally considered inconspicuous.

Table A.3 — Statistical characteristics (based on ISO 5725-2[7]) for direct determination of mercury in cosmetics by means of mercury analyser (first ring test)

Parameter	Face cream Level 2	Foundation Level 3	Lipstick Level 4	Mascara Level 5
Number of participating laboratories	14	14	14	14
Number of laboratories with quantitative values	14	14	14	14
Number of outliers (laboratories)	0	0	0	0
Number of laboratories for determination of characteristics	14	14	14	14
Mean Value, mg/kg	0,48	1,00	1,48	2,62
± confidence interval, mg/kg	0,04	0,06	0,10	0,22
Reproducibility standard deviation s_R, mg/kg	0,08	0,13	0,20	0,42
Relative reproducibility standard deviation $s_{R,rel}$, %	17 %	13 %	13 %	16 %
Reproducibility limit R, mg/kg	0,23	0,36	0,55	1,17
Relative reproducibility limit R_{rel}, %	48 %	36 %	37 %	45 %
Repeatability standard deviation s_r, mg/kg	0,05	0,09	0,11	0,22
Relative repeatability standard deviation $s_{r,rel}$, %	11 %	9 %	8 %	8 %
Repeatability limit r, mg/kg	0,15	0,26	0,31	0,60
Relative repeatability limit r_{rel}, %	32 %	26 %	21 %	23 %
Relative Horwitz standard deviation, %	18 %	16 %	15 %	14 %
HORRAT	0,96	0,80	0,89	1,17

Table A.4 — Statistical characteristics (based on ISO 5725-2[7]) for direct determination of mercury in cosmetics by means of mercury analyser (second ring test)

Parameters	Lipstick Level 1	Lipstick Level 2	Lipstick Level 3
Number of participating laboratories	10	10	10
Number of laboratories with quantitative values	9	9	9
Number of outliers (laboratories)	0	0	0
Number of laboratories for determination of characteristics	9	9	9
Mean Value, mg/kg	0,11	0,52	1,09
± confidence interval, mg/kg	0,01	0,02	0,07
Reproducibility standard deviation s_R, mg/kg	0,02	0,03	0,09
Relative reproducibility standard deviation $s_{R,rel}$, %	14 %	6 %	8 %
Reproducibility limit R, mg/kg	0,04	0,09	0,25
Relative reproducibility limit R_{rel}, %	39 %	18 %	23 %
Repeatability standard deviation s_r, mg/kg	0,01	0,03	0,03
Relative repeatability standard deviation $s_{r,rel}$, %	12 %	5 %	3 %
Repeatability limit r, mg/kg	0,04	0,07	0,09
Relative repeatability limit r_{rel}, %	33 %	13 %	9 %
Relative Horwitz standard deviation, %	22 %	18 %	16 %
HORRAT	0,64	0,36	0,52

A.4 Mercury analysers used during the interlaboratory tests

The following instruments and parameters (Table A.4) have been used by the laboratories participating to the 2 interlaboratory ring tests:

- DMA 80 (Milestone)
- AMA 254 (Altec)
- MA3000 (Nippon Instrument Corporation, NIC)

Table A.5 — Examples of operating conditions for integrated mercury analysers used by the participating laboratories

		Condition 1	Condition 2	Condition 3	Condition 4	Condition 5
Instrument	Name	DMA 80	AMA 254	DMA 80	DMA 80	AMA 254
	Supplier	Milestone	Altec	Milestone	Milestone	LECO
Drying step	Time (s)	60	60	30	60	140
	Temperature (°C)	150	120	200	200	100
Decomposition step	Time (s)	270	150	300	240	150
	Temperature (°C)	150 °C to 800 °C in 2,5 min then 2 min at 800 °C	600 °C	2min at 250 °C, 250 to 650 °C in 1,5 min then 1,5 min at 650 °C	1 min at 200 °C, 200 to 650 °C in 2 min then 1 min at 650 °C	700 °C
Purge step	Time (s)	60	45	60	60	45
Amalgam step	Time (s)	12	45	12	12	57
	Temperature (°C)	900	900	850–950	850	850–950

		Condition 6	Condition 7	Condition 8	Condition 9	Condition 10
Instrument	Name	MA 3000	DMA 80	DMA 80	DMA 80	DMA 80
	Supplier	NIC	Milestone	Milestone	Milestone	Milestone
Drying step	Time (s)	60	70	60	60	60
	Temperature (°C)	150	200	150	250	150
Decomposition step	Time (s)	720	180	270	720	270
	Temperature (°C)	10 min at 250 °C then 2 min at 850 °C	200 to 650 °C in 1,5 min then 1,5 min at 650 °C	150 to 750 °C in 2,5 min then 2 min at 750 °C	650 °C	150 °C to 800 °C in 2,5 min then 2 min at 800 °C
Purge step	Time (s)	[a]	60	60	[a]	60
Amalgam step	Time (s)	[a]	12	12	12	12
	Temperature (°C)	850–950	850–950	850	850–950	900

[a] According to manufacturer default parameter

The catalyst tube is generally set at about 615 °C.

Annex B
(informative)

ISO 23674 and ISO 23821 common ring test results

Determination of traces of mercury in cosmetics can be performed following this document and ISO 23821 [1].

To ensure that operator can choose either one or the other standard to determine traces of mercury in cosmetics with methods fulfilling the same requirements, a common interlaboratory test has been launched using the same tailor-made cosmetic products.

A set of 3 samples (see Table B.1) have been analysed by several laboratories in triplicate, using either this document or ISO 23821[1]. Those samples consist in tailor made lipsticks with known contents of mercury that was only originating from a solid certified reference material (CRM).

A lipstick base without any source of mercury has first been prepared and controlled by using this document. Then precise amounts of solid CRM have been dispersed in the lipstick base along with free from mercury colorants. Lipstick sample bulks were then thoroughly homogenized in order to ensure the proper distribution of the CRM. Three lipstick bulks with different amounts of dispersed CRM (corresponding to 3 different mercury concentrations) have been created and their mercury content controlled. Every of the 3 lipstick bulks has then been aliquoted in several containers, every aliquot was controlled prior to sending to 17 participating laboratories for analysis.

Table B.1 — Samples sent for second interlaboratory test

Cosmetic product	Mercury content (mg/kg)
Lipstick sample 1	0,101
Lipstick sample 2	0,506
Lipstick sample 3	1,014

Nine participating laboratories used this document and among the six participating laboratories using ISO 23821,[1] five performed an additional dilution step of a factor from 2 to 20. One of the laboratories used both methods to analyse same samples.

Results obtained for mercury content are presented in Figure B.1 and Figure B.2 by the mean of the accuracy profile methodology (as described in ISO/TS 22176[6]). Results show that despite their different physicochemical principles, both methods present performances compliant with the level of performances required for their intended use.

As a consequence, the validated ranges with acceptance limit set at ±30 % are as follow:

a) from 0,15 to 1 mg/kg (ppm)for this document

b) from 0,12 to 1 mg/kg (ppm) for ISO 23821 [1]