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

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

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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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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 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). This method was validated by means of an interlaboratory test according to ISO 5725-2[7] using 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.

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

1 Scope

This document specifies a method for determination of mercury in cosmetics by means of cold vapour atomic absorption spectrometry (AAS) with a prior pressure digestion.

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

No terms and definitions are listed in this document.

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

As a first step, the finished cosmetic product is digested in a closed vessel 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 conform with the conditions specified for this method.

The measurement solution is transferred to the reaction vessel 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 limits of quantification (LOQs).

5 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).

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

5.2 Nitric acid, minimum w = 65 %, density = 1,4 g/ml, suitable for elemental analysis.

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

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

Alternating operation with both reducing agents (5.4.1 and 5.4.2) 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 conformed with.

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

Weigh 50 g tin(ll) chloride, $SnCl_2 \cdot 2H_2O$ in a 500 ml volumetric flask, dissolve in approximately 100 ml hydrochloric acid (5.1), and fill up to 500 ml with water. The solution shall be freshly prepared prior to use.

5.4.2 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 fill up 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 hydrogen when combined with water and especially on reaction with acids, 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.

5.5 Stabilization

The standard, calibration and sample digestion solutions are stabilized with hydrochloric acid (5.1). 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 10).

5.6 Mercury stock solution, mercury ρ = 1 000 mg/l.

The stock solution is commercially available. It is recommended to use certified stock solutions.

5.7 Mercury standard solutions

Dilute the stock solution to the concentrations required for calibration and add the necessary amount of stabilisation reagent (5.5). In doing so, select concentrations that the linear range of the reference function is not exceeded. It is recommended to use at least three (3) standard solutions with different concentrations.

The acid concentration in the standard solutions 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.

5.8 Calibration blank solution

The calibration blank solution shall contain water, the same amount of stabilisation reactant as the mercury standard solutions (5.7) per litre and the quantities of nitric acid (5.2) and hydrochloric acid (5.1) that correspond to the acid concentrations in the measurement solution.

6 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 (5.3) over night or a prolonged period. Prior to use, rinse the apparatus with ultrapure water and dry. Steaming of chemically inert vessels (e.g. made of quartz glass) using nitric acid (5.2) is an effective cleaning method and is regularly used in element trace analysis. To prevent contamination and adsorption, only use lab materials made with borosilicate or quartz glass.

6.1 Digestion vessels.

Use commercially available, safety-tested pressure vessels and inserts made of acid resistant 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. The specific size of the vessels is not mandatory and depends on the used type of microwave.

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.

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. Reliable temperature measurement is obtained, for example, 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.

6.3 Membrane filter, 0,45 µm pore size.

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).

6.4 Atomic absorption spectrometer, optionally available with background correction and including accessories for cold vapour technology or amalgam technology.

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

6.5 Element-specific light for mercury

Measurement at 253,7 nm.

7 Procedure

7.1 General

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 user of this document to take appropriate measures for ensuring the safety and health of the personnel prior to application of the document.

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

7.2 Preparation of samples

Before the digestion of the sample, a suitable preparation shall 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 test portion quantity.

7.3 Pressure assisted digestion

7.3.1 General

WARNING 1 Depending on the type of reactivity of the sample, it can be required to weigh in lower quantities than specified in 7.3.2 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) can cause explosions. Alcohols or solvents in combination with concentrated nitric acid can cause delayed severe reactions already at room temperature. Therefore, it is highly recommended to gently evaporate all volatile components before adding the acid (7.3.3).

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 vessel. Prior to digestion, ensure that the entire sample is fully covered by the acid mixture.

Temperature and pressure inside the vessels shall be 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.

7.3.2 Preparation of sample by digestion — General case

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

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 (5.2) 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 min up to overnight.

Then add 1 ml of hydrochloric acid (5.1) 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.

7.3.3 Preparation of sample by digestion — Specific cases

— For highly water-based cosmetic products, such as lotion, milky lotion, cleanser or micellar water, a test portion could reach 400 mg. In this case no addition of water is required before addition of acids (7.3.2).

— For all the other specific cases, test portion can be adapted but the ratio between test portion and acid volumes (7.3.2) shall not be changed.

In case of products with a significant content of volatile components, for safety reasons, it is highly recommended to remove volatile components by carefully heating up the sample (e.g. in a water bath at 60 °C) after weighing the sample in the digestion vessel, but prior to the addition of the acid. The loss of volatile components should be determined at the end of the process. In this context, special care shall be taken to prevent losses of the specific elements.

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

7.3.4 Microwave digestion procedure

WARNING 1 During all steps of the digestion process, the manufacturer's safety information shall be accurately followed.

7.3.4.1 Process the samples using, for example, a three-step heating program:

a) ramp the heat up from room temperature to 200 °C in, for example, 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 2 Depending on reactivity of the sample, it can be necessary to select a lower heat-up rate than specified in order to prevent extreme reactions or explosions.

7.4.3.2 For reactive samples, a 7-step heating program with a slower heating ramp has been efficiently used:

a) ramp the heat up from room temperature to 160 °C in 25 min;

b) hold the temperature at 160 °C for 15 min;

c) ramp the heat up from 160 °C to 180 °C in 10 min;

d) hold the temperature at 180 °C for 10 min;

e) ramp the heat up from 180 °C to 200 °C in 35 min;

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

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

NOTE This information is given as an example of program that can be used in case of reactive samples. This alternative digestion program was not included in the validation studies. It is up to the user to show equivalency when used.

7.3.5 Preparation of measurement solutions

The digestion solution obtained after pressure digestion is filled up with water after cooling to obtain a defined volume, for example 20 ml, and is used for measurement. If required, further dilution steps can be performed using calibration blank solution (5.8).

Ensure that the measurement solution obtained in this way contains the same acid concentrations as the calibration solutions prepared according to 5.7.

Remove any residue by decanting or filtering the final solution by a membrane filter (6.3).

The stability of the mercury in the digestion solution depends on the type and concentration of the acids used for the digestion, the vessel materials used for storage and the mercury concentration. It is therefore recommended to stabilize the digestion solution, for example, with hydrochloric acid (5.1). If sufficient hydrochloric acid has already been added during the digestion, further stabilization may be dispensed with.

7.4 Atomic absorption spectrometry (cold vapour AAS)

7.4.1 Spectrometry settings

For development of a measurement program, 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 (5.4.1) or sodium borohydride (5.4.2).

7.4.2 Example for AAS determination using cold vapour technology

Perform zero setting of the instrument by means of the calibration blank solution as described in 5.8.

For the establishment of reference functions, use the respectively suitable standard solutions. If possible, calibrate the measured value display by means of the standard solutions, directly in the required concentration. Check the linear range of the reference function in regular intervals. Acid concentrations of the reference solutions shall be appropriately adjusted to the sample measurement solutions (7.3.5).

After establishment of the reference functions, the digestion solution can be used for measurement either directly or after appropriate dilution if its concentration is outside the linear range. For larger measurement series, it is recommended to check the zero point and the calibration in regular intervals.

Although background correction is rarely required with cold vapour technology, it shall still be verified for every matrix whether or not background correction is required.

7.5 Quality control of the analysis

For quality control, digest reference materials or control samples with reliably established contents of mercury and measure for every test series. In addition, prepare and measure blank solutions for every digestion series, also including all steps of the procedure. For this purpose, perform digestion as described in 7.3 without the sample and prepare the measurement solution (7.3.5). Blank digestion is intended to check blank values that can be caused by vessels and acids or other types of contaminations.

8 Evaluation

8.1 Calculation

Calculate the mercury content, w, by using Formula (1):

$$w = \frac{(a \cdot V \cdot F)}{m} \tag{1}$$

where

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

a is the mercury content in the sample measurement solution, in mg/l;

V is the volume of the filled-up digestion solution, in ml;

F is the dilution factor of the sample measurement solution;

m is the test portion for digestion in g or the sample volume for digestion, in ml.

It is not recommended to subtract blank values from the mercury content *a*, in the measurement solution. Operator has to check the possible input of the mercury blank value and has to check if subtraction from *a* is possible or repetition is needed.

8.2 Limit of quantification

The limit of quantification of the measurement solution depends on the following parameters:

— principle of mercury release (batch or flow system);

— enrichment (amalgam) or no enrichment;

— in case of flow systems:

  — continuous/discontinuous release of Hg;

  — quantity of the digestion solution used;

— structure of the instrument;

— matrix effects.

The limit of quantification generally is within a range between 0,05 µg/l and 5 µg/l, based on the measurement solution. With a test portion of 0,2 g and a final digestion volume of 20 ml, calculate the limit of quantification for the cosmetic product to be within the range between 0,005 mg/kg and 0,5 mg/kg.

8.3 Reliability of the method

The procedure was validated in 2015 by means of an interlaboratory test according to ISO 5725-2[7], including 8 laboratories participating. In total, six samples (lipstick, tattoo colourant, body lotion, toothpaste and eyeshadow) with varying mercury contents between 0,110 mg/kg and 5,84 mg/kg were analysed. A second interlaboratory test has been performed in 2019/2020 and was evaluated by the means of the accuracy profile methodology (as described in ISO/TS 22176[6]). For all test results, see Annex A and Annex B. For statistical characteristics of the second interlaboratory test see Annex B, Table B.1.

9 Test report

The test report should contain the data according to ISO/IEC 17025[5] 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 23821:2022;

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.

10 Alternative stabilizing reagents

10.1 Stabilization with sulfuric acid (2 %)

10.2 Stabilization with potassium bromide-potassium bromate reagent[3]

a) Potassium bromate solution, substrate concentration c = 0,033 mol/l.

   Dissolve 1,39 g of potassium bromate in 250 ml water. Possible contamination by mercury can be eliminated by drying in the muffle furnace overnight at 250 °C ± 20 °C. The solution is stable for approximately 1 month.

b) Potassium bromide solution, c = 0,2 mol/l.

   5,95 g of potassium bromide are dissolved in 250 ml of water. Possible contamination by mercury can be removed by drying in the muffle furnace overnight at 300 °C ± 20 °C. The solution is stable for approximately 1 month.

c) Potassium bromide-potassium bromate reagent, (c = 0,1 N).

   The potassium bromate (a) and potassium bromide solution (b) are mixed in equal parts. 100 samples can be treated with 200 ml of reagent. The reagent is stable for several days to weeks. This shall be checked. The solution should be colourless. Ampoules with ready-mixed potassium bromide-potassium bromate solution are commercially available and can also be used.

10.3 Stabilization with potassium dichromate solution, mass concentration ϱ = 5 g/l

Dissolve 5 g of potassium dichromate in 500 ml of diluted nitric acid (5.3) and fill up to 1 l with water.

11 Short-term stabilization when measuring with potassium permanganate solution

In order to prevent adsorption of mercury at the walls of the measuring containers and to increase the stability of the standard and measurement solutions for larger measurement series, it is possible to add a few drops of potassium permanganate solution to the measuring solution in the measuring containers until it remains pink.

Potassium permanganate solution, for example with a concentration of ϱ = 40 g/l.

0,4 g of potassium permanganate are dissolved in water and made up to 10 ml. The solution shall be prepared fresh every day.

Annex A
(informative)

Performance of the method determined via ISO 5725 statistical approach

The procedure has been verified in 2015 by means of interlaboratory test according to ISO 5725-2[7]. In total, six samples (lipstick, tattoo colorant, body lotion, tooth paste and eyeshadow) with varying contents of mercury were analysed. Two of these six samples were obtained from the same sample material (body lotion) and thus constitute blind duplicate samples. For each sample, the determination of mercury was carried out on the same day in duplicate, under repeatability conditions. The analysis was performed within 3 days to 7 days after digestion of the respective sample. The statistical analyses were performed using the statistical approaches according to ISO 5725-2[7]. The two body lotion samples 3 and 6 were also evaluated using the statistical approaches according to ISO 5725-3[8] for a nested interlaboratory test design.

The statistical evaluation according to ISO 5725-2[7] is based on the data after outlier elimination. In addition to the overall mean value, the results of this evaluation are the reproducibility standard deviation s_R and the repeatability standard deviation s_r.

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 s_r 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.

The combined evaluation of the body lotion samples 3 and 6 is carried out according to ISO 5725-3[8]. In addition to the repeatability and reproducibility standard deviation, the intermediate standard deviation s_I is determined, which characterizes the variability of the measured values between the two subsamples.

As for all sample-element combinations the relative reproducibility and repeatability standard deviation lay below 30 % the results are considered as acceptable. This is also shown by the fact that the HORRAT values are always below 2.

A comparison of the two blind duplicates of the body lotion samples shows that the obtained reproducibility standard deviations represent the actual standard deviations under repeatability conditions.

The interlaboratory test has been performed in the working range from 0,110 mg/kg up to 5,84 mg/kg. On the basis of all the data obtained, the procedure shows that it fits for purpose in various cosmetic matrices and for a range of concentration of interest.

The statistical characteristics of the inter laboratory tests are listed in Table A.1.

Table A.1 — Statistical characteristics (based on ISO 5725-2[7]) for determination of mercury in cosmetics by means of cold vapour AAS

Parameter	Lipstick	Tattoo colorant	Toothpaste	Eyeshadow	Body lotion – identical sampling material	
	Sample 1	Sample 2	Sample 4	Sample 5	Sample 3	Sample 6
Number of participating laboratories	8	8	8	8	8	8
Number of laboratories with quantitative values	8	8	8	8	8	8
Number of outliers (laboratories)	1	1	1	0	0	0
Number of laboratories for determination of characteristics	7	7	7	8	8	8
Mean value, mg/kg	0,259	0,110	0,166	5,84	1,42	1,40
± confidence interval, mg/kg	±0,036	±0,010	±0,010	±0,57	±0,18	±0,25
Reproducibility standard deviation s_R, mg/kg	0,048	0,013	0,014	0,82	0,26	0,35
Relative reproducibility standard deviation. $s_{R,rel}$, %	18,63	11,68	8,47	14,06	18,24	24,92
Reproducibility limit R, mg/kg	0,135	0,036	0,039	2,30	0,73	0,98
Relative reproducibility limit R_{rel}, %	52,16	32,71	23,71	39,37	51,07	69,79
Repeatability standard deviation s_r, mg/kg	0,010	0,003	0,005	0,22	0,05	0,04
Relative repeatability standard deviation. $s_{r,rel}$, %	3,78	2,84	2,95	3,69	3,75	3,04
Repeatability limit r, mg/kg	0,027	0,009	0,014	0,60	0,15	0,12
Relative repeatability limit r_{rel}, %	10,57	7,96	8,26	10,34	10,49	8,51
Relative Horwitz standard deviation, %	19,60	22,29	20,95	12,26	15,17	15,20
HORRAT	0,95	0,52	0,40	1,15	1,20	1,64

Annex B
(informative)

Common interlaboratory test results of ISO 23674[4] and this document

Determination of traces of mercury in cosmetics can be performed following ISO 23674[4] and this document.

To ensure that operators can choose either one or the other document 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 triplicates, using either ISO 23674[4] or this document. 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 ISO 23674[4] Then precise amounts of solid CRM have been dispersed in the lipstick base along with free from mercury colorants. Lipstick sample bulks were then homogenized in order to ensure the proper distribution of the CRM. 3 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 labs for analysis.

Table B.1 — Samples sent for second interlaboratory test

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

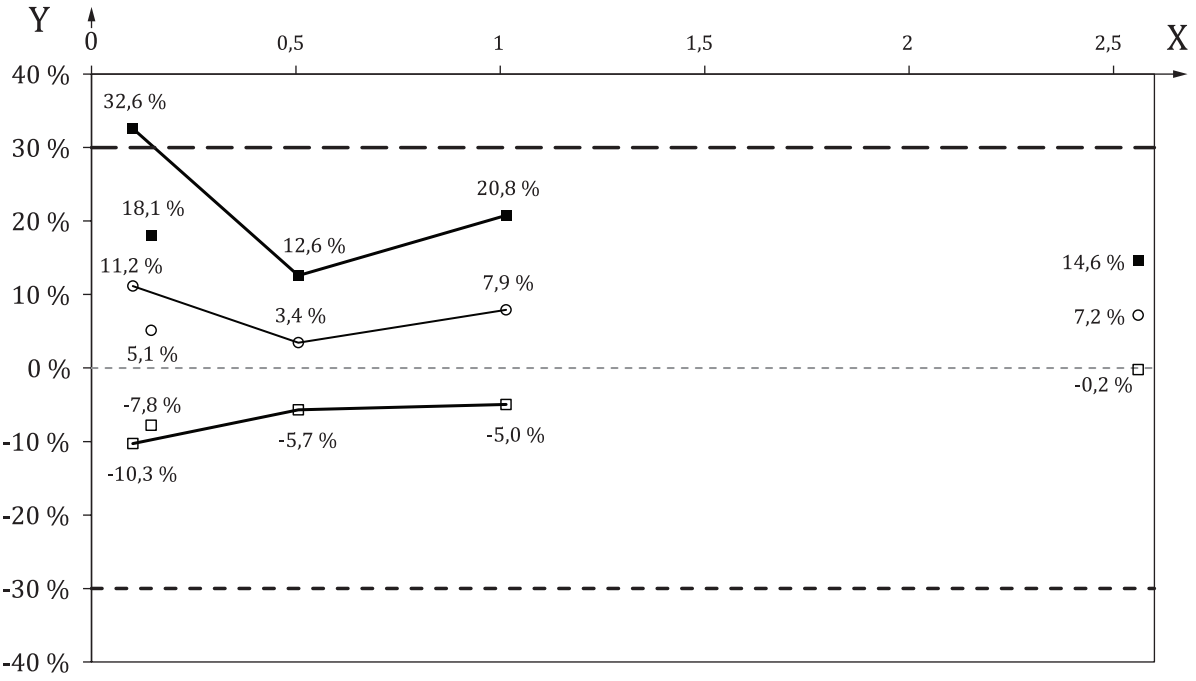
Nine laboratories provided results using ISO 23674[4] and among the six laboratories that provided results using this document, five performed an additional dilution step of a factor from 2 to 20 (see 7.3.5). One of the laboratories used both methods to analyse same samples.

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 follows:

a) from 0,15 mg/kg to 1 mg/kg for ISO 23674[4];

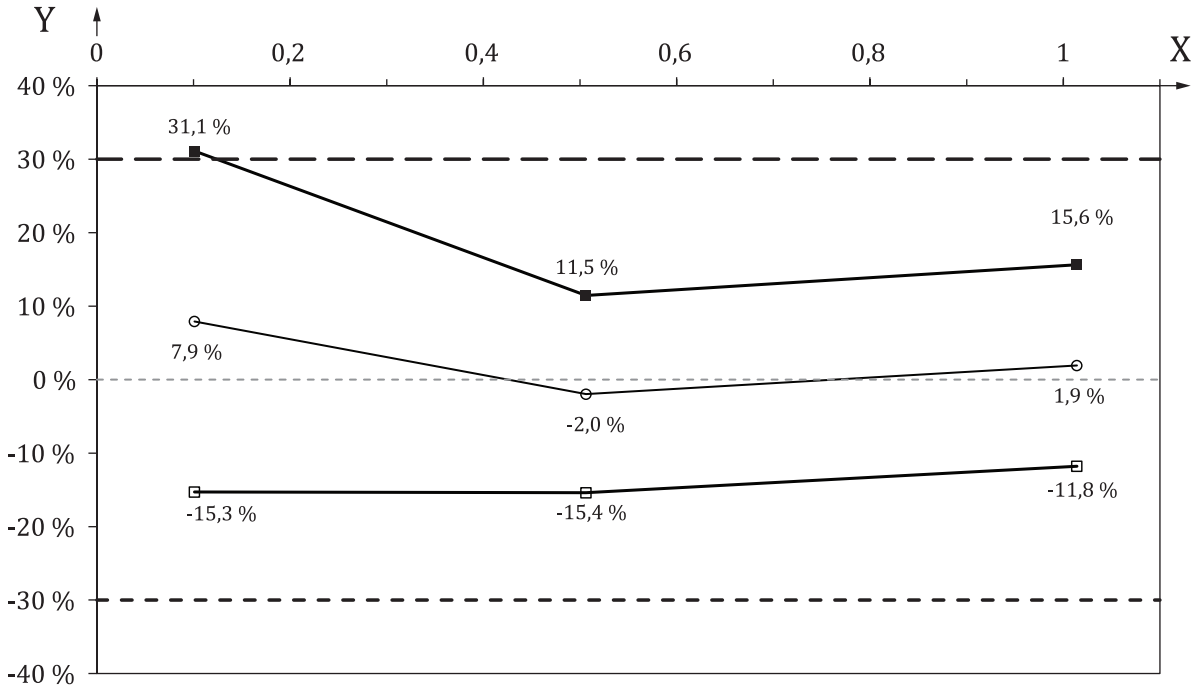
b) from 0,12 mg/kg to 1 mg/kg for this document.



Key

X	mercury concentration (mg/kg)		low acceptance limit
Y	accuracy (%)		high acceptance limit
	relative high tolerance limit (RHTL)	■	RHTL CRM
	mean relative bias (MRB)	○	MRB CRM
	relative low tolerance limit (RLTL)	□	RLTL CRM
	zero level		

Figure B.1 — Accuracy profile for mercury validating ISO 23674[4] (integrated system) in the range 0,15 mg/kg to 1 mg/kg



Key

X	mercury concentration (mg/kg)		relative low tolerance limit
Y	accuracy (%)		zero level
	relative high tolerance limit		low acceptance limit
	mean relative bias		high acceptance limit

Figure B.2 — Accuracy profile for mercury validating this document (CV-AAS) in the range 0,12 mg/kg to 1 mg/kg

Bibliography

[1] EN 13806, *Foodstuffs — Determination of trace elements — Determination of mercury by cold-vapour atomic absorption spectrometry (CVAAS) after pressure digestion*

[2] EN 14083, *Foodstuffs — Determination of trace elements — Determination of lead, cadmium, chromium and molybdenum by graphite furnace atomic absorption spectrometry (GFAAS) after pressure digestion*

[3] ISO 12846, *Water quality — Determination of mercury — Method using atomic absorption spectrometry (AAS) with and without enrichment*

[4] ISO 23674, *Cosmetics — Analytical methods — Direct determination of traces of mercury in cosmetics by thermal decomposition - Atomic absorption spectrometry (mercury analyzer)*

[5] ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

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