JRC SCIENTIFIC AND POLICY REPORTS

IMEP-35: Determination of Total Lead in Lipsticks

Interlaboratory Comparison Report

F. Cordeiro, P. Robouch, H. Emteborg,
J. Snell, M-F. Tumba-Tshilumba,
B. Kortsen, B. de la Calle

February 2013

Joint
Research
Centre

Legal Notice

Neither the European Commission nor any person acting on behalf of the Commission are responsible for the use which might be made of this publication.

Europe Direct is a service to help you find answers to your questions about the European Union

Freephone number (*): 00 800 6 7 8 9 10 11

(*) Certain mobile telephone operators do not allow access to 00 800 numbers or these calls may be billed.

A great deal of additional information on the European Union is available on the Internet.

It can be accessed through the Europa server http://europa.eu/.

Printed in Belgium

IMEP-35: Determination of Total Lead in Lipsticks

Interlaboratory Comparison Report

February 2013

F. Cordeiro (a)
P. Robouch (c)
H. Emteborg (c)
J. Snell (c)
M-F. Tumba-Tshilumba (c)
B. Kortsen (d)
Beatriz de la Calle (b, c)

(a) ILC coordinator, (b) IMEP programme coordinator,
(c) Technical / scientific support, (d) Administrative support,

IMEP®
★★★★★★★★★★★★★

Contents

1   Executive summary

The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre (JRC), a Directorate-General of the European Commission, operates the International Measurement Evaluation Programme (IMEP). It organises interlaboratory comparisons (ILC's) in support to EU policies. This report presents the results of a proficiency test exercise (PT) focussed on the determination of total lead in lipsticks which was organised in support of the European Council Directive 76/768/EEC (1976).

Eighteen participants from thirteen countries registered to the exercise, of which 17 reported results.

The test item used was a blend of commercially available lipsticks. The assigned value was obtained as the average of results reported by two expert laboratories having demonstrated experience in the analysis of trace elements in different matrices. The associated uncertainties of the assigned values were computed according to the ISO/IEC Guide 98:2008 (GUM).

Participants were invited to report their measurement uncertainties. This was done by all laboratories having submitted results in this exercise.

Laboratory results were rated with z- and zeta (ζ-) scores in accordance with ISO 13528. The standard deviation for the proficiency assessment was based on the expert judgment of the advisory board of this ILC exercise and it was fixed as 20 % of the assigned value.

The percentage of satisfactory z-scores was 75 %. Therefore, the outcome of the exercise shows an overall good performance for European control laboratories assuring compliance towards the European legislation related to cosmetic products.

2   Introduction

According to the European legislation for cosmetics [1], lead should not be present in cosmetics. A maximum level of 20 mg kg^{-1} for total lead in cosmetics is given in the German legislation for cosmetics [2]. Lead is an unintended contaminant or impurity that can be present at very low levels in some colour additives used by the cosmetic industries, particularly in the production of lipsticks. Recently, the U.S. Food and Drug Administration (FDA) conducted a survey on the content of lead in commercially available lipsticks [3]. For the purpose of that survey the FDA developed and single-laboratory validated a method for the determination of total lead in lipstick. The method is based on the use of microwave-assisted digestion with nitric acid and hydrofluoric acid with further determination of the total content of lead by inductively coupled plasma-mass spectrometry (ICP-MS). The FDA indicated that *"consistent results could usually be obtained only by including HF in the digestion procedure"*.

In 2011 the Joint Research Centre, a Directorate General of the European Commission, carried out a monitoring study on a reasonably large number of commercially available lipsticks [4], using the FDA method mentioned above. The concentration of total lead in the lipsticks analysed in the survey run by the JRC varied from below 1 mg kg^{-1} up to 3.75 mg kg^{-1}. The levels of lead found in some of the lipsticks analysed raise some concerns from the point of view of human health.

To have a good overview of the measurement capabilities of the European laboratories to determine total lead in lipstick, a proficiency test, IMEP-35, was organised by the JRC. This report summarises and evaluates the outcome of IMEP-35.

3   IMEP support to EU policy

The International Measurement Evaluation Programme (IMEP$^{®}$) is hold by the Joint Research Centre - Institute for Reference Materials and Measurements. IMEP provides support to the European measurement infrastructure in the following ways:

IMEP disseminates metrology from the highest level down to the field laboratories. These laboratories can benchmark their measurement result against the IMEP certified reference value. This value is established according to metrological best practice.

IMEP helps laboratories to assess their estimate of measurement uncertainty. The participants are invited to report the uncertainty on their measurement results. IMEP integrates the estimate into the scoring, and provides assistance for the interpretation.

IMEP supports EU policies by organising interlaboratory comparisons in the frame of specific EU Directives or on request of a specific EC Directorate-General. In the case of IMEP-35 it was organised as a request from the Directorate General for Health and Consumers (DG SANCO) to support the implementation of the Council Directive 76/768/EEC [1]. Furthermore, IMEP-35 provided support to the following stakeholders:

- The European Cooperation for Accreditation (EA) in the frame of a Memorandum of Understanding on a number of metrological issues, including the organisation of interlaboratory comparisons. National accreditation bodies were invited to nominate a limited number of laboratories for free participation in IMEP-35. The Danish Accreditation and Metrology Fund (DANAK) liaised between EA and IMEP for this ILC. This report does not discern the EA nominees from the other participants. Their results are however summarised in a separate report to EA.

- The Asia Pacific Laboratory Accreditation Cooperation (APLAC), in the frame of the collaboration with APLAC. Mr. Aparna Dhawan (APLAC PT Committee) liaised between APLAC and IMEP, announcing the exercise to the accreditation bodies in the APLAC network.

- The InterAmerican Accreditation Cooperation (IAAC). Mrs. Barbara Belzer liaised between IAAC and IMEP. She was invited to announce the exercise to the accreditation bodies in the IAAC network.

4   Scope and aim

As stated in *Council Directive 76/768/EEC on the approximation of the laws of Member States relating to cosmetics products*, Pb should not be present in cosmetics. The scope of this interlaboratory comparison exercise was to monitor the performance of European official control laboratories in the determination of total lead in lipsticks.

The administrative and logistic procedures of IMEP were applied. IMEP is accredited according to ISO 17043:2010 [5]. The name of this proficiency test is IMEP-35.

5   Set-up of the exercise

5.1   Time frame

The exercise was announced via the IMEP web page on the 6th of June 2012 (Annex 1). Additionally, the exercise was announced to the European Cooperation for Accreditation (EA), to the Asian Pacific Laboratory Accreditation Cooperation (APLAC) and to the

InterAmerican Accreditation Cooperation (IAAC). These announcements were made on the 28th March (Annex 2), on the 7th June 2012 (Annex 3) and on the 13th June 2012 (Annex 4), respectively.

Registration was opened till the 30th June 2012. The deadline for reporting results was the 24th August 2012. Dispatch was followed by the messenger's parcel tracking system on the internet.

5.2   Confidentiality

EA was invited to nominate laboratories for participation. The following confidentiality statement was made to EA: *"Confidentiality of the participants and their results towards third parties is guaranteed. However, IMEP will disclose details of the participants that have been nominated by EA to the EA working group for ILCs in Testing. The EA accreditation bodies may wish to inform the nominees of this disclosure.*"

5.3   Distribution

Test items were dispatched on the 3rd of July 2012. Each participant received one package containing:

- One bottle containing approximately 1.1 g of the test material,
- The "*Sample accompanying letter*" (Annex 5),
- A *"Confirmation of Receipt"* form (Annex 6).

5.4   Instructions to participants

Participants received an individual code to access the online reporting interface, to report their measurement results and to complete the related questionnaire. The questionnaire was used to extract all relevant information related to measurements and laboratories (Annex 7).

Participants were informed that the procedure used for the analysis should resemble as closely as possible their respective routine procedures for this particular matrix, analyte and concentration level. However, recommendation was provided not to use less than 0.2 g of test portion for analysis.

6   Test material

6.1   Preparation

The test item used for this ILC exercise was a blend of 38 units of commercially available lipsticks (purchased in different local cosmetic shops). These lipsticks had been pre-

screened for lead content by Solid Sampling Electrothermal Atomic Absorption Spectrometry (SS-ETAAS). Consequently the items with the highest concentrations were selected for the production. The lipsticks were molten at about 100 °C in an acid-washed beaker placed on a hot plate and mixed with an acid-washed magnetic stirring bar for 2 hours. About 150 g of liquid lipstick was available for filling. A total of 86 units were produced with approximately 1.1 g per unit filled into acid-washed transparent 5 ml vials. The liquid lipstick was constantly mixed during filling.

Prior to the production of the test items, a blank lipstick non contaminated with lead was subjected to the same melting, mixing and filling process to assess any potential procedural blank that could affect homogeneity of the batch produced. During pre-screening the blank lipstick was proven to be lead-free. No major difference was detected between the unprocessed lipstick and the 6 units of treated/processed blank. ICP-MS was used to perform measurements of total Pb in the lipstick used as blank due to the low limit of detection achieved with that technique (0.07 mg kg^{-1}).

6.2   Homogeneity and stability study

The homogeneity and stability studies were performed by ALS Scandinavia AB using inductively coupled plasma sector field mass spectrometry (ICP-SFMS) after sample digestion with a mixture of HNO_3/HF. Homogeneity was evaluated according to ISO 13528:2005 [6]. The material proved to be adequately homogeneous.

The stability study was conducted following an isochronous experimental design [7-8].

The material proved to be adequately stable for the eight weeks that elapsed between the dispatch of the samples and the deadline for submission of results.

The contribution due to the homogeneity (u_{bb}) and to the stability (u_{st}) to the uncertainty of the assigned value (u_{ref}) were calculated using SoftCRM [9].

The analytical results and the statistical evaluation of the homogeneity and stability studies are provided in Annex 8.

7   Reference values and their uncertainties

The total Pb mass fraction was determined by two expert laboratories:

- Flemish Institute for Technological Research (VITO), Belgium
- ALS Scandinavia AB, Sweden.

Experts were asked to use the method of their choice and no further requirements were imposed regarding methodology. The experts were also asked to report their results together with the measurement uncertainty and with a clear and detailed description on how uncertainty was estimated.

VITO used isotope dilution mass spectrometry (ID-MS) after sample digestion with HNO_3/HF, while ALS Scandinavia used inductively coupled plasma sector field mass spectrometry (ICP-SFMS) after sample digestion with a mixture of HNO_3/HF.

The mean of the independent means provided by the two expert laboratories was used to derive the assigned value (X_{ref}) for this PT. The standard uncertainty (u_{ref}) of the assigned value was calculated in compliance with ISO/IEC Guide 98 (GUM) [10] - taking into account the uncertainty contributions of homogeneity (u_{bb}) and stability (u_{st}):

$$u_{ref} = \sqrt{u_{char}^2 + u_{bb}^2 + u_{st}^2}$$ Eq. 1

where the standard uncertainty of characterisation (u_{char}) is calculated combining the standard uncertainties reported by the two expert laboratories (u_{exp1}, u_{exp2}):

$$u_{char} = \frac{1}{2} \sqrt{u_{exp1}^2 + u_{exp2}^2}$$ Eq. 2

Table 1 presents the results reported by the two expert laboratories, standard uncertainty contributions and the reference values (X_{ref}, u_{ref} and U_{ref}).

Table 1 – Reported values by the expert laboratories, uncertainty contributions, assigned value and corresponding combined and expanded uncertainties (in mg kg^{-1})

	Value	U_{exp} (k=2)	u_{char}	u_{bb}	u_{st}	X_{ref}	u_{ref}	U_{ref} (k=2)
Exp 1	1.10	0.05	0.013	0.031	0.051	1.10	0.06	0.12
Exp 2	1.10	0.02						

Note: Experts 1 and 2 do not necessarily correspond to the order they were presented.

8   Target standard deviation $\hat{\sigma}$

On the basis of previous experience for this type of analysis the standard deviation for proficiency assessment ($\hat{\sigma}$) was set as 20 % of the assigned value ($\hat{\sigma}$ = 0.20 X_{ref}).

9 Results and evaluation

Results were received from 17 of the 18 registered laboratories. One participant reported a "lower than" value ("less than 2 mg kg^{-1}"). Although this value is consistent with the assigned value of 1.1 mg kg^{-1}, it could not be scored (Fig. 1).

Participants were asked to perform two or three independent measurements, correct their measurements for recovery and report their calculated mean and its associated measurement uncertainty (u_{lab}).

Annex 9 presents the reported results as a tabular and as a graph. Furthermore, it includes the corresponding Kernel density plot, obtained using software available from the Statistical Subcommittee of the Analytical Methods Committee of the UK Royal Society of Chemistry [11].

9.1 Scores and evaluation criteria

Individual laboratory performance was expressed in terms of *z-* and *ζ-scores* in accordance with ISO 13528 [6], based on the measurement result *(x$_{lab}$)* and the corresponding standard uncertainty *(u$_{lab}$)* reported by a participant:

$$z = \frac{x_{lab} - X_{ref}}{\hat{\sigma}}$$
<div style="text-align:right">Eq. 3</div>

$$\zeta = \frac{x_{lab} - X_{ref}}{\sqrt{u_{ref}^2 + u_{lab}^2}}$$
<div style="text-align:right">Eq. 4</div>

The interpretation of the *z-* and *ζ-score* is done as follows (according to ISO/IEC 17043:2010, [5]):

Satisfactory performance	$|score| \leq 2$
Questionable performance	$2 < |score| \leq 3$
Unsatisfactory performance	$|score| > 3$

The z-score compares the participant's deviation from the reference value with the standard deviation for proficiency assessment ($\hat{\sigma}$) used as common quality criterion. $\hat{\sigma}$ is defined by the PT organiser as the maximum acceptable standard uncertainty. Value for $\hat{\sigma}$ in IMEP-35 was set to 20 % of the assigned value.

The ζ-score states if the laboratory result agrees with the assigned value within the respective uncertainty. The denominator is the combined uncertainty of the assigned value and the measurement uncertainty as stated by the laboratory. The ζ-score is

therefore the most relevant evaluation parameter, as it includes all parts of a measurement result, namely the expected value (assigned value), its uncertainty and the unit of the result as well as the uncertainty of the reported values. An unsatisfactory ζ-score can either be caused by an inappropriate estimation of the concentration or of its uncertainty or both.

The standard uncertainty of the laboratory (u_{lab}) was estimated by dividing the reported expanded uncertainty by the reported coverage factor, k. When no uncertainty was reported, it was set to zero ($u_{lab} = 0$). When k was not specified, the reported expanded uncertainty was considered as the half-width of a rectangular distribution; u_{lab} was then calculated by dividing this half-width by $\sqrt{3}$, as recommended by EURACHEM and CITAC [12].

Uncertainty estimation is not trivial; therefore an additional assessment was provided to each laboratory reporting uncertainty, indicating how reasonable their uncertainty estimate is. The standard uncertainty from the laboratory (u_{lab}) is most likely to fall in a range between a minimum uncertainty (u_{min}), and a maximum allowed (u_{max}, case "a"). u_{min} is set to the standard uncertainty of the reference value (u_{ref}). It is unlikely that a laboratory carrying out the analysis on a routine basis would measure the measurand with a smaller uncertainty than the expert laboratories chosen to establish the assigned value. u_{max} is set to the standard deviation ($\hat{\sigma}$) accepted for the PT assessment.

If u_{lab} is smaller than u_{min} (case "b") the laboratory may have underestimated its uncertainty. However, such a statement has to be taken with care as each laboratory reported only measurement uncertainty, whereas the uncertainty of the reference value also includes contributions of homogeneity and stability. If those are large, measurement uncertainties smaller than u_{min} (u_{ref}) are possible and plausible.

If u_{lab} is larger than u_{max}, (case "c") the laboratory may have overestimated the uncertainty. An evaluation of this statement can be made when looking at the difference of the reported value and the assigned value: if the difference is small and the uncertainty is large, then overestimation is likely. If, however, the deviation is large but is covered by the uncertainty, then the uncertainty is properly assessed, but large. It should be pointed out that u_{max} is only a normative criterion if set down by legislation.

9.2   Laboratory results and scorings

Figure 1 presents an overview of the z- and ζ-scores. The laboratories' performances appear to be good with up to 75 % of the participants reporting satisfactory z-scores.

Concerning the ζ-scores a lower percentage of the population obtained a satisfactory score (50 %). Thus, laboratories should enhance their effort in the estimation of the uncertainty associated to their measurements.

Fig. 1: Overview of scores (in %)
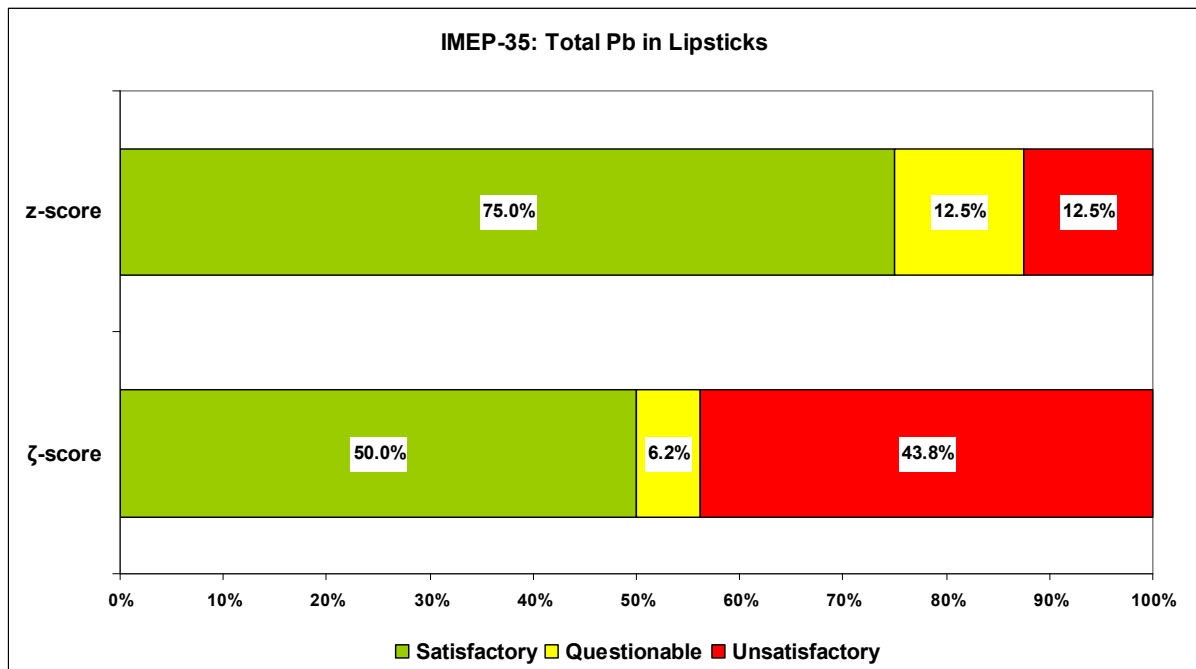
Annex 9 presents the results for this assessment where:

  i) 33 % of the participants reported reasonable uncertainties ($u_{ref} \leq u_{lab} \leq \hat{\sigma}$, case "*a*")
  ii) 47 % underestimated their uncertainties ($u_{lab} < u_{ref}$, case "b")
  iii) 20 % overestimated their uncertainties ($u_{lab} > \hat{\sigma}$, case "*c*").

One may notice that most of the laboratories having reported underestimated uncertainties obtained unsatisfactory ζ-scores.

9.3   Further information extracted from the questionnaire

In addition to the submission of results, participants were asked to answer a number of questions related to:

   i) The analytical method used

   ii) How the participants assure the quality of their reported results

In order to allow the identification of all major potential sources of variability among the reported results we investigated the relation between the reported value for total lead content and the set of responses provided in the questionnaire. A multivariate approach was favoured. The statistical data treatment was performed using The Unscrambler X 10.1 (CAMO Software AS, Norway). Answers were first transformed into numerical variables, before applying partial least square regression modelling (PLS-R). The model succeeds to "explain" 97 % of the total covariance relating the reported results and the set of answers. Good laboratory performance seems to be correlated to the following three parameters:

i) Appropriate amount of the test sample taken for analysis (test portion). Very low test portions were, generally, leading to poorer performance;

ii) Appropriate acid mixture (HNO_3/HF) for sample digestion; and

iii) Use of microwave digestion.

These observations confirm the finding of the FDA [3] stating that *"variable amounts of Pb can be extracted depending upon experimental conditions such as analytical portion, acids used, temperature, decomposition procedure, etc"*. Laboratories having used only HNO_3 (excluding HF) reported lower results, which may be attributed to an incomplete sample digestion.

Other potential influencing factors such as the technique used (ICP-MS, ETAAS or Atomic Absorption Spectrometry (AAS)), having a quality system in place, being accredited for this type of analysis or taking part in interlaboratory comparison exercises, were not identified as having a significant influence on the results.

Some experimental details, extracted from the questionnaire, are presented in Annex 10.

Table 2 presents the feedback received from some participants. As can be seen most of the comments were related to the amount of test item available for measurement.

Table 2 – Feedback from participants (as taken from the questionnaire)

Lab. Code	Do you have any comments? Please, let us know!
L04	We would like a larger quantity of the sample
L10	We concluded that this lipstick does not contain colorants with lead compounds, and we do not perform further examinations because the supplied amount of material would not be enough for this. Please, if the mean value of the lead content for this round examination is above the reported limit of determination, do not conclude that our result is "Unsatisfactory" without calculating a "z" value!
L15	We suggest that the amount of sample is to small (2g of samle at least)
L16	Small sample
L18	Part of the work that we perform on a regular basic is heavy metal trace analysis by ICP-MS on drugs, herbal products, vitamins, minerals and natural health products. We never had to analyse a cosmetic or lipstick. Therefore we never use HF to digest samples. We know that we have a possible part of Pb present in refractory minerals that will not digest by HNO3. Our goal was to challenge the method.

10   Conclusion

No particular problems could be identified on the determination of total lead in lipstick. Three major influencing factors, related to the experimental protocol used by the participants, were identified: - proper sample intake, - appropriate acid mixture (HNO_3/HF) for sample digestion; and - the use of microwave digestion.
The analytical detection systems were not identified as influencing the determination of total lead in lipstick.

11   Acknowledgements

Organisation	Country
Regional Health Inspectorate	BULGARIA
Health Canada	CANADA
Health Canada	CANADA
Institute of Public Health	CROATIA
Danish Technological Institute	DENMARK
Eurofins Miljø A/S	DENMARK
Nemzeti Fogyasztóvédelmi Hatóság	HUNGARY
Health Service Executive	IRELAND
CHELAB SRL	ITALY
Institute of Public Health	SERBIA
Institute of public health	SERBIA
Institut za zaštitu na radu a.d. Novi Sad	SERBIA
Coop Central Laboratory	SWITZERLAND
Service de la consommation et des affaires vétérinaires	SWITZERLAND
Labor Veritas AG	SWITZERLAND
Kantonales Laboratorium Bern	SWITZERLAND
Centre Technique de la Chimie	TUNISIA

Abbreviations

IRMM	Institute for Reference Materials and Measurements
JRC	Joint Research Centre
ILC	Interlaboratory Comparison
PT	Proficiency testing
IMEP	International Measurement Evaluation Programme
EU	European Union
ISO GUM	International Organisation for Standardisation – Guide to the expression of Uncertainty in Measurement
EA	European Cooperationn for Accreditation
APLAC	Asian Pacific Laboratory Accreditation Cooperation
IAAC	InterAmerican Accreditation Cooperation
HR-ICP-MS	High-Resolution Inductively-Coupled Plasma Mass Spectrometry
ICP-SFMS	Inductively-Coupled Plasma Sector Field Mass Spectrometry
ICP-MS	Inductively-Coupled Plasma Mass Spectrometry
ID-MS	Isotope Dilution Mass Spectrometry
SS-ETAAS	Solid Sampling ElectroThermal Atomic Absorption Spectrometry
PLS-R	Partial Least Squares Regression
AAS	Atomic Absorption Spectroscopy
ETAAS	Electrothermal Atomic Absorption Spectrometry

References

[1]   Council Directive 76/768/EEC on the approximation of the laws of Member States relating to cosmetics products

[2]   Kosmetische Mittel: BfR empfiehlt Schwermetallgehalte über Reinheitsanforderungen der Ausgangsstoffe zu regeln, Stellungnahme Nr. 025/2006 des BfR vom 05. April 2006, http://www.bfr.bund.de/cm/343/kosmetische_mittel_bfr_empfiehlt_schwermetallgehalte_ueber.pdf

[3]   N. M. Hepp, W. R. Mindak and J. Cheng, J. Cosmet. Sci., 60, 405-414 (2009)

[4]   P. Piccinini, M. Piecha and S. F. Torrent, "*Results of European Survey on Lead in Lipsticks*" JRC Technical Note EUR 24886 (2011), ISBN 978-92-79-18552-6

[5]   ISO/IEC 17043:2010 "*Conformity assessment - General requirements for proficiency testing*", issued by International Organisation for Standardisation, Geneva

[6]   ISO 13528:2005, "*Statistical methods for use in proficiency testing for interlaboratory comparisons*", issued by International Organisation for Standardisation, Geneva

[7]   Lamberty A., Schimmel H., Pauwels J. (1998) "*The study of the stability of reference materials by isochronous measurements*", Fresenius' Journal of Analytical Chemistry 360(3-4): 359-361

[8]   Linsinger T. P. J., Pauwels J., Lamberty A., Schimmel H. G., Van Der Veen A. M. H., Siekmann L. (2001) "*Estimating the uncertainty of stability for matrix CRMs*", Analytical and Bioanalytical Chemistry 370(2-3): 183-188

[9]   http://www.eie.gr/iopc/softcrm/index.html

[10]   ISO/IEC Guide 98:2008, "*Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement*" (GUM 1995), issued by International Organisation for Standardisation, Geneva

[11]   "*Representing data distributions with kernel density estimates*" (2006). AMC Technical Brief N° 4, issued by the Statistical Subcommittee of the Analytical Methods Committee (AMC) of the Royal Society of Chemistry, UK

[12]   "*Quantifying Uncertainty in Analytical Measurement*" (2000). EURACHEM/CITAC, http://www.eurachem.org

Annexes

Annex 1: Publication on IRMM website

JOINT RESEARCH CENTRE
Institute for Reference Materials and Measurements (IRMM)

EUROPA > European Commission > JRC > IRMM > Interlaboratory comparisons | Search IRMM Internet

Font Size: A A A A | News | Links | Press

Home   IMEP   NUSIMEP   REIMEP

Main Menu

- About IRMM
- Activities
- Reference materials
- EU Reference Laboratories
- Interlaboratory comparisons
- Job opportunities
- Events
- Training
- Calls
- Publications

IMEP-35: Total lead in lipstick

The IMEP-35 exercise focuses on the analysis of total lead in lipstick. This interlaboratory comparison (ILC) exercise is organised in support to the Council Directive 76/768/EEC of 27 July 1976 on the approximation of the laws of the Member States relating to cosmetic products.

IMEP-35 exercise is open to all laboratories.

The cost of this interlaboratory comparison is **EUR 400** per registration.

Please register using the following link:

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=820

Test materials and analytes

The test material to be analysed is lipstick contained in a glass bottle. Each participants will receive one bottle. The measurands are total Pb.

General Outline of the exercise

Participants are requested to perform 1-3 independent analyses using the method of their choice, and to report the mean, its expanded uncertainty and coverage facto k. Detailed instructions will be sent together with the sample.

Schedule

Registration	Sample dispatch	Reporting of results	Report to participants
Deadline 30/06/2012	First half of July 2012	Deadline 07/09/2012	First quarter of 2012

Annex 2: Announcement letter to European Accreditation

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements

28th March 2012

DANAK
Kirsten Andersen
Dyregaardsvej 5 B, DK-2740 Skovlunde,
DENMARK

Dear Kirsten,

Interlaboratory comparison for the determination of total lead in lipsticks
The Institute for Reference Materials and Measurements (IRMM) organises IMEP-35, an interlaboratory comparison for the **"Determination of the total lead in lipsticks"** in support to the *Council Directive 76/768/EEC of 27 July 1976 on the approximation of the laws of the Member States relating to cosmetic products.*

In the frame of the EA-IRMM collaboration agreement, IRMM kindly invites EA to nominate laboratories for free participation. They should hold (or be in the process of obtaining) an accreditation for this type of measurement.
I suggest that you forward this invitation to the national EA accreditation bodies for their consideration. There is a limited number of samples at your disposal and the number of nominees should not exceed 2-3 laboratories per country.
Confidentiality of the participants and their results towards third parties is guaranteed. However, IMEP will disclose details of the participants that have been nominated by EA to the EA working group for ILCs. The EA accreditation bodies may wish to inform the nominees of this disclosure.
The registration page for laboratories appointed by EA is open until **4 May 2012**. Distribution of the samples is foreseen for the second half of May 2012. The deadline for submission of results is **29 June 2012**.

In order to register, laboratories must:
1. **Enter** their details online:
https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=820

2. **Print** the completed form when the system asks to do so and clearly indicate on the printed form that you have been appointed by the European Cooperation for Accreditation to take part in this exercise **otherwise your laboratory will be invoiced for participation** with the tariff that will be charged to non-appointed laboratories.

3. **Send** the printout to both the IMEP-35 and the EA-IMEP-35 coordinators:

IMEP-35 coordinator	EA-IMEP-35 coordinator
Dr. Fernando Cordeiro	Mrs. Kirsten Andersen
Fax +32 14 571865	Fax +45 77 339501
E-mail jrc-irmm-imep@ec.europa.eu	E-mail kja@danak.dk

Please contact me if you have any questions or comments. We are looking forward to our cooperation!

With kind regards

Fernando Cordeiro
IMEP-35 Coordinator

Annex 3: Announcement letter to APLAC

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements

IMEP®

Geel, 07 June 2012

To: Aparna Dhawan
APLAC PT Committee

Intercomparison for Total lead in lipstick

Dear Aparna,

The Institute for Reference Materials and Measurements (IRMM) organises an interlaboratory comparison for the **"Determination of the total lead in lipstick ", (IMEP-35)**.

IRMM kindly invites APLAC to nominate 10 laboratories for free participation. However, they should hold (or be in the process of obtaining) an accreditation for this type of measurement. I suggest that you forward this invitation to a selection of specialised laboratories in this area.

In addition to the 10 laboratories above mentioned, other laboratories may take part in IMEP-35 paying a registration fee of 400 €

Confidentiality of the participants and their results towards third parties is guaranteed.

The registration page is open until **30 June 2012**. Distribution of the samples is foreseen for first half of July 2012. The deadline for submission of results is **7 September 2012**.

In order to register, laboratories must:

1   Enter their details online:
https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=820

2   Print the completed form when the system asks to do so and clearly indicate on the printed form that they have been appointed by APLAC to take part in this exercise **otherwise your laboratory will be invoiced 400 €for participation** normally applied for non-appointed laboratories.

3   Send the printout to both the IMEP-35 and the APLAC coordinators:

IMEP-35 coordinator	**APLAC coordinator**
Fernando Cordeiro	Aparna Dhawan
Fax +32 14 571 865	
E-mail: jrc-irmm-imep@ec.europa.eu	E.Mail: aparna@nabl-india.org

Please contact me if you have any questions or comments. We are looking forward to our cooperation!

With kind regards,

Fernando Cordeiro
IMEP-35 Coordinator

Annex 4: Announcement letter to IAAC

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements

IMEP®

Geel, 13 June 2012

To: Barbara Belzer
IAAC Lab Committee

Intercomparison for Total lead in lipstick

Dear Barbara,

The Institute for Reference Materials and Measurements (IRMM) organises an interlaboratory comparison for the **"Determination of the total lead in lipstick ", (IMEP-35)**.

IRMM kindly invites IAAC to nominate 10 laboratories for free participation. However, they should hold (or be in the process of obtaining) an accreditation for this type of measurement. I suggest that you forward this invitation to a selection of specialised laboratories in this area.

In addition to the 10 laboratories above mentioned, other laboratories may take part in IMEP-35 paying a registration fee of 400 €

Confidentiality of the participants and their results towards third parties is guaranteed.

The registration page is open until **30 June 2012**. Distribution of the samples is foreseen for first half of July 2012. The deadline for submission of results is **7 September 2012**.

In order to register, laboratories must:

4. **Enter** their details online:
    https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=820

5. **Print** the completed form when the system asks to do so and clearly indicate on the printed form that they have been appointed by IAAC to take part in this exercise **otherwise your laboratory will be invoiced 400 €for participation** normally applied for non-appointed laboratories.

6. **Send** the printout to both the IMEP-35 and the IAAC coordinators:

IMEP-35 coordinator Fernando Cordeiro Fax +32 14 571 865 E-mail: jrc-irmm-imep@ec.europa.eu	IAAC coordinator Barbara Belzer E.Mail: secretariat@iaac.org.mx

Please contact me if you have any questions or comments. We are looking forward to our cooperation!

With kind regards

Fernando Cordeiro
IMEP-35 Coordinator

Annex 5: Sample accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Institute for reference materials and measurements
Food Safety & Quality

IMEP

Geel, 03 July 2012
JRC.D5/FCR/bk/ARES(2012)/

«Firstname» «Surname»
«Organisation»
«Department»
«Address»
«Address2»
«Zip» «Town»
«Country»

Participation in IMEP-35, a proficiency test exercise for the determination of total lead in lipsticks

Dear «Title» «Surname»,

Thank you for participating in the IMEP-35 proficiency test for the determination of the total lead in lipsticks This proficiency test (PT) exercise is organised in support to the Council Directive 76/768/EEC of 27 July 1976 on the approximation of the laws of the Member States relating to cosmetic products.

Please keep this letter, you need it to report your results.

This parcel contains:
a) One bottle containing approximately > 1 g of the test material
b) A "Confirmation of Receipt" form
c) A summary of the questionnaire you will be prompted to answer on-line after reporting your results
d) This accompanying letter.

Please check whether the bottles containing the test material remained undamaged during transport. Then, please send the "Confirmation of receipt" form back (fax: +32-14-571865, e-mail: jrc-irmm-imep@ec.europa.eu). You should store the samples in a dark place at ≤18 °C until analysis.

The measurand is total lead in lipstick. The sample matrix is a mixture of commercially available lipsticks.

The procedure used for the analyses should resemble as closely as possible the one that you use in routine analyses. A minimum sample intake of 0.2 g is recommended.

The results are to be reported without correction for moisture since the moisture content in the test item is very low.

Retieseweg 111, B-2440 Geel - Belgium. Telephone: (32-14) 571 211. http://irmm.jrc.ec.europa.eu
Telephone: direct line (32-14) 571 682. Fax: (32-14) 571 865.

E-mail: jrc-irmm-imep@ec.europa.eu

Reporting of results
Please perform two or three independent measurements, correct the measurements results for recovery and report on the reporting website:

- the **corrected mean** on your two or three measurement results (mg kg^{-1})
- the associated expanded **uncertainty** (mg kg^{-1}),
- the **coverage factor** and
- the **technique** you used.

The results should be reported in the same form (e.g. number of significant figures) as those normally reported to the customer.

The reporting website is https://irmm.jrc.ec.europa.eu/ilc/ilcReporting.do

To access the webpage you need a personal password key, which is: **«Part_key»**. The system will guide you through the reporting procedure. After entering your results, please complete also the relating questionnaire.

Do not forget to submit and confirm always when required.

Directly after submitting your results and the questionnaire information online, you will be prompted to print the completed report form. Please do so, **sign the paper version and return it to IRMM by fax (at +32-14-571-865) or by e-mail.** Check your results carefully for any errors before submission, since this is your last definitive confirmation.

The **deadline** for submission of results is **24/08/2012**.

Please keep in mind that collusion is contrary to professional scientific conduct and serves only to nullify the benefits of proficiency tests to customers, accreditation bodies and analysts alike.

Your participation in this project is greatly appreciated. If you have any remaining questions, please contact me by e-mail: jrc-irmm-imep@ec.europa.eu

With kind regards

Dr. Fernando Cordeiro Raposo
IMEP-35 Co-ordinator

Enclosures: 1) One bottle containing the test material; 2) Confirmation of receipt form; 3) A summary of the questionnaire you will be prompted to answer on-line after reporting your results and 4) Accompanying letter.

Cc: F. Ulberth

Annex 6: "Confirmation of receipt" of samples

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for reference materials and measurements
Food Safety & Quality

Annex to
JRC.D5/FCR/bk/ARES(2012)/804506

"Title" "First name" "Surname"
"Organisation"
"Department"
"Address"
"Address 1"
"Address 2"
"Zip" "Town"
"Country"

IMEP-35

Total Lead in Lipstick

Confirmation of receipt of the samples

Please return this form at your earliest convenience.
This confirms that the sample package arrived.
In case the package is damaged,
please state this on the form and contact us immediately.

ANY REMARKS   …………………………….
                        …………………………….

Date of package arrival   …………………………….

Signature   …………………………….

Please return this form to:

Fernando Codeiro Raposo

IMEP-35 Coordinator
EC-JRC-IRMM
Retieseweg 111
B-2440 GEEL, Belgium

Fax   : +32-14-571865

e-mail : JRC-IRMM-CRL-HEAVY-METALS@ec.europa.eu

IMEP®

Annex 7: Questionnaire

Milc questionnaire

Comparison for IMEP-35

Please complete the questionnaire.

Submission Form

1. Have you analysed the sample accordingly to an official method?

☐ No

☐ Yes

1.1. If yes which one?

1.2. If no, please describe it in max. 150 characters.

1.2.1. Sample treatment

1.2.2. Digestion step

1.2.3. Extraction / separation step

1.2.4. Instrument calibration

2. Method related questions

2.1. Have you used a microwave digestion?

☐ No

☐ Yes

2.2. Which maximum temperature was used?

2.3. Which was the hold time at maximum temperature?

2.4. At what pressure?

2.5. What was the test portion used (in g)?

2.6. How did you perform the digestion?

☐ a) With HNO_3 alone?

☐ b) With HNO_3 + HF?

☐ c) Did you add H_3PO_4?

☐ d) Other

2.6.1. Which volumes were used (in ml)?
See table **Volumes used for the digestion (in ml)** at bottom

2.7. Did you noticed any undigested material?

☐ No

☐ Yes

3. What is your limit of detection (LOD in mg/kg)

4. Which was your recovery factor ((in %)

4.1. How did you estimate the recovery factor?

☐ a) Using a CRM?

☐ b) Adding a known amount of the analyte (spiking)?

☐ c) Other?

5. Do you usually provide an uncertainty statement to your customers for this type of analysis ?

☐ No

☐ Yes

6. What is the basis of your uncertainty estimate ? (multiple answers possible)

☐ a) uncertainty budget according to ISO-GUM

☐ b) known uncertainty of the standard method

☐ c) uncertainty of the method as determined during in-house validation

☐ d) measurement of replicates (i.e. precision)

☐ e) estimation based on judgement

☐ f) use of intercomparison data

☐ g) other

6.1. If other, please specify :

[   ]

7. What is the level of confidence reflected by coverage factor k reported with your results ? (in %)

[   ]

8. Does your laboratory have a quality system in place ?

☐ No

☐ Yes

8.1. If yes, which one ?

☐ ISO 17025

☐ ISO 9000 series

☐ Other

8.1.1. If other, please specify :

[   ]

8.2. Are you accredited ?

☐ No

☐ Yes

8.2.1. If yes, by which accreditation body ?

[   ]

9. Does your laboratory carry out this type of analysis on a regular basis ?

☐ No

☐ Yes

9.1. If yes, please estimate the number of samples :

☐ a) 0-50 samples per year

☐ b) 50-250 samples per year

☐ c) 250-1000 samples per year

☐ d) more than 1000 samples per year

10. Does your laboratory take part in similar interlaboratory comparisons on a regular basis ?

☐ No

☐ Yes

10.1. Which ILC scheme(s) ?

[   ]

11. Does your laboratory use a reference material for this type of analysis ?

☐ No

☐ Yes

11.1. If yes, which one ?

[   ]

11.2. Is the material used for the validation of procedures ?

☐ No

☐ Yes

11.3. Is the material used for the calibration of instruments ?

☐ No

☐ Yes

12. How have you heard about this exercise ?

[   ]

13. Do you have any comments ? Please, let us know ...

[   ]

Annex 8: Homogeneity and stability studies

Homogeneity study:

Bottle	Total lead (in mg kg^{-1})	
74	0.948	0.874
83	0.963	0.980
40	0.912	0.959
33	0.863	0.917
14	0.882	0.997
21	0.899	1.040
68	0.896	1.020
47	0.926	1.040
01	0.887	0.919
58	0.883	0.998
Homogeneity according to ISO 13528:2005	**(in mg kg^{-1})**	
Overall mean	0.940	
$\hat{\sigma}$	0.190	
Allowable criterion ($0.3\hat{\sigma}$)	0.060	
Standard deviation of sample averages (S_x)	0.031	
Within-sample Standard deviation (S_w)	0.066	
Between-sample standard deviation (S_S)	0 ($MS_b < MS_w$)	
Adequately homogeneous	Since $S_S < 0.3\hat{\sigma}$	
Uncertainty contribution, u_{bb} (in mg kg^{-1})	**0.031**	

MS_b and MS_w refers to the mean square between and within samples

Stability study:

Bottle	Total lead (in mg kg^{-1} – samples kept at 18 °C)			
	Time (in weeks)			
	0	**3**	**5**	**8**
1	0.900	1.050	0.918	0.920
2	0.978	0.964	0.899	0.906
Slope of the linear regression curve				- 0.006
Standard error of the slope				0.006
Slope of the linear regression NOT significantly different from zero				
Uncertainty contribution, u_{st} (in mg kg^{-1})				**0.051**

Annex 9: Results for total lead in lipsticks
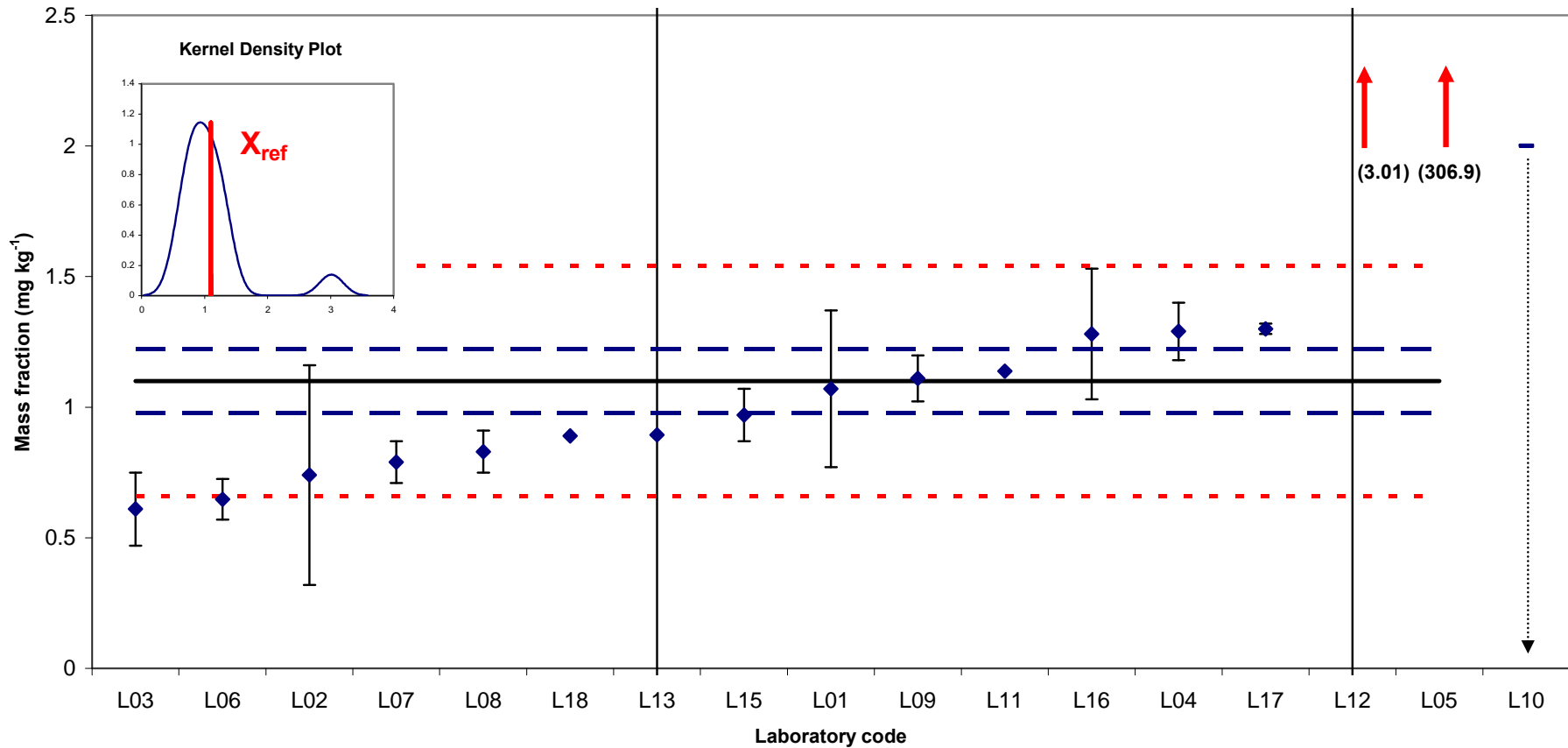
X_{ref} = 1.10, U_{ref} = 0.12 (k = 2), $\hat{\sigma}$ = 0.22 (all values in mg kg^{-1})

Lab. Code	X_{lab}	U_{lab}	k	u_{lab}	Technique	z-score [a]	ζ-score [a]	U [b]
Exp 1	1.10	0.05	2	0.025	ID-MS			
Exp 2	1.10	0.02	2	0.01	ICP-SFMS			
L01	1.07	0.3	2	0.15	ICP-MS	-0.1	-0.2	a
L02	0.74	0.42	2	0.21	ICP-MS	-1.6	-1.6	a
L03	0.61	0.14	2	0.07	ICP-MS	-2.2	-5.3	a
L04	1.29	0.11	2	0.055	AAS	0.9	2.3	b
L05	306.9	15.3	2	7.65	ETAAS	1390.0	40.0	c
L06	0.647	0.078	1	0.078	AAS	-2.1	-4.6	a
L07	0.79	0.08	2	0.04	AAS	-1.4	-4.3	b
L08	0.83	0.08	95%	0.04	AAS	-1.2	-3.7	b
L09	1.11	0.088	2	0.044	ICP-MS	0.0	0.1	b
L10	< 2.0				AAS			
L11	1.138	0	0		ICP-MS	0.2	0.6	b
L12	3.013	9.431	2	4.716	AAS	8.7	0.4	c
L13	0.894	11.3	2	5.65	ETAAS	-0.9	0.0	c
L15	0.97	0.1	2	0.05	ICP-MS	-0.6	-1.7	b
L16	1.28	0.25	2	0.13	ETAAS	0.8	1.3	a
L17	1.3	0.02	2	0.01	ICP-MS	0.9	3.3	b
L18	0.89				ICP-MS	-1.0	-3.5	

[a]   Satisfactory, Questionable, Unsatisfactory

[b]   "a": $u_{ref} \leq u_{lab} \leq \hat{\sigma}$; "b": $u_{lab} < u_{ref}$; "c": $u_{lab} > \hat{\sigma}$

IMEP-35: Total Pb in Lipsticks
Assigned value: X_{ref} = 1.10 ; U_{ref} = 0.12 (k = 2), σ = 0.22 (all in mg kg^{-1})

This graph displays the averaged value of the three replicates with their associated uncertainties. The uncertainties are shown as reported. The solid blak line corresponds to X_{ref}, the dashed blue lines to the boundaries of X_{ref} ($X_{ref} \pm 2u_{ref}$) the dotted red lines to the acceptance interval ($X_{ref} \pm 2\sigma$).

Annex 10: Experimental details

Lab.	Off. Method?	Which one?	Sample treatment	Digestion step	Extraction / separation step	Instrument calibration
L01	No		none	Digestion in MW oven, powercontrolled program, with HNO3, HCl and HF	sedimentation	external calibration with internal standard correction
L03	No		NONE	MICRO WAVE	NONE	EXTERNAL TARATURE
L04	Yes	Method validated of the National Centre of Hygiene, Medical Ecology and Feeding. Methods for determination of lead in cosmetic products by AAS flame,Collection of Hygienic Research Sofia, 2002,p.43-46	dissolved in 10 ml conc.HNO3 for 72 h	boiled in conc.HNO3 till stopped elimination of nitric oxides	deluted to 10 ml with 0.2 M HNO3	AAS - flame - linear calibrated in 0.0; 0.5; 1.0; 2.0; 4.0; 6.0 mg/l
L05	No		at 0.3 g of sample add 3 ml H2SO4 and heat on a hot plate at 120 °C. Continue heating the sample until carbonization occurs. At this time, add 0.5 ml HNO3	Place this in electric furnace, increase heat at 100 °C per 10 min, and at about 500 °C perform ashing over of 3 hours	Add 4 ml water to the ash, and after drynig, add 5 ml HCl to dissolve the salts and bring up to 25 ml using water	Place 20 μL each of pretreated sample into four (04) 20 ml volumetric flasks, and add standard solution (200 ppb) to three of them respectevly 0.5 ml; 1 ml and 2 ml so that one flask contains no standard solution and the other three have increasing concentrations of standard solution added to the sample. bring up to volume using water.
L06	No		none	digestion in HNO3, microwave assisted at high pressure and high temperature	dilution of sample with ultrapure water	external
L07	No		no	Microwave: 0.3 g and 0.4 g were digested with HNO3+H2O2+H2O	Centrifugation	Standard addition
L08	Yes	ISO 15586		acid hydrolysis with HNO3 and H2O2		yes
L09	No		-	High pressure microwave digestion	-	external calibration
L10	No		microwave digestion	four steps from 25 to 220 degree Celsius	centrifuge 5000 rpm 10 min	10-50 microgram pro L calibration standards
L11	No		Nitric acid microwave digestion	Microwave, 200degC	None	5 x Calibration Standards, QC
L13	Yes	Council Directive 76/768/EEC				
L15	No		Samples were prepared by microwave digestion.	Mixture of 5ml of HNO3 and 2ml of H2O2	Solution was filtrated and diluted to 50ml with deionised water.	Five point calibration curve, with blank sample analysed
L16	No		about 0.2g sample digested with 3ml HNO3+1ml H2O2 +1mlHCl	microwave digestion	digested sample filtered and transfered in 25ml flask	0-50μg/l
L17	No	NA	Sample was treated as is. Sample was digested by acid mixture in a microwave.	Microwave digestion was performed using HNO3:HF=4:1	No further extraction/separation was performed after digestion.	Daily optimization was performed prior to the analysis.
L18	No		Sample microwave digestion nitric acid, and H2O2 in closed vessel is performed to put the Pb in solution.	1- 800W , Ramp 15min, 120PSI, 180c Hold 20min 2- 800W, Ramp 5min, 120PSI, 200c Hold 20min	Let settle and take the supernatant	Periodically the instrument is calibrate

Author(s): F. Cordeiro, P. Robouch, H. Emteborg, J. Snell, M-F. Tumba-Tshilumba, B. Kortsen, B. de la Calle

Abstract
The Institute for Reference Materials and Measurements (IRMM) of the Joint Research Centre (JRC), a Directorate-General of the European Commission, operates the International Measurement Evaluation Programme (IMEP). It organises interlaboratory comparisons (ILC's) in support to EU policies. This report presents the results of a proficiency test exercise (PT) focussed on the determination of total lead in lipsticks which was organised in support of the European Council Directive 76/768/EEC (1976).
Eighteen participants from thirteen countries registered to the exercise, of which 17 reported results.

The percentage of satisfactory z-scores was 75 %. Therefore, the outcome of the exercise shows an overall good performance
for European control laboratories assuring compliance towards the European legislation related to cosmetic products.

As the Commission's in-house science service, the Joint Research Centre's mission is to provide EU policies with independent, evidence-based scientific and technical support throughout the whole policy cycle.

Working in close cooperation with policy Directorates-General, the JRC addresses key societal challenges while stimulating innovation through developing new standards, methods and tools, and sharing and transferring its know-how to the Member States and international community.

Key policy areas include: environment and climate change; energy and transport; agriculture and food security; health and consumer protection; information society and digital agenda; safety and security including nuclear; all supported through a cross-cutting and multi-disciplinary approach.