European Commission

JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

Determination of the mass fractions of total aluminium, nickel, antimony and zinc in food simulant B

FCM-18-02 Proficiency Test Report

F. Cordeiro, G. Beldi, J. Snell,
S. García-Ruiz, G. Van Britsom,
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Determination of the mass fractions of total aluminium, nickel, antimony and zinc in food simulant B

FCM-18-02 Proficiency test report

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268-PT Accredited by the
Belgian Accreditation Body (BELAC)

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Executive summary

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM) organised a proficiency test (FCM-18-02) for the determination of the mass fractions of total aluminium, nickel, antimony and zinc in food simulant B (acetic acid, 3 % w/v). This proficiency test was open to National Reference Laboratories (NRLs) and official control laboratories (OCLs).

The test item was a food simulant B solution spiked with Al, Ni, Sb and Zn at concentration levels close to their respective legislative specific migration limits (SML). The homogeneity and stability of the test item were evaluated and the assigned values were derived from formulation.

Twenty six NRLs and twenty six OCLs from 26 countries - representing the EU Member States except Latvia, Malta and Romania, and Switzerland - registered to the exercise; 51 of them reported results.

Laboratory results were rated using z and ζ (zeta) performance scores in accordance with ISO 13528:2015. Based on expert opinion, a relative standard deviation for proficiency assessment (σ_{pt}) was set to 15 % of the assigned value for Al, Ni and Sb and 12 % for Zn.

More than 85 % of the participants performed satisfactorily (according to the z performance score) for the four elements. These results confirm that most NRLs are able to monitor mass fractions of the investigated metals at specific migration levels set by Commission Regulations related to plastic materials and articles intended to come into contact with food.

List of abbreviations

DG SANTE	Directorate General for Health and Food Safety
EURL-FCM	European Union Reference Laboratory for Food Contact Materials
F-AAS	Flame - Atomic Absorption Spectrometry
FS-B	Food simulant B solution (acetic acid, 3 % w/v)
GF-AAS	Graphite furnace - Atomic Absorption Spectrometry
ICP-OES	Inductively Coupled Plasma - Optical Emission Spectrometry
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ISO	International Organization for Standardization
JRC	Joint Research Centre
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PT	Proficiency Test
SML	Specific migration limit

List of symbols and definitions

k	coverage factor
σ_{pt}	standard deviation for proficiency assessment
$u(x_i)$	calculated standard measurement uncertainty (of participant "i")
$u(x_{pt})$	standard uncertainty of the assigned value
u_{char}	(standard) uncertainty contribution due to characterisation
u_{hom}	(standard) uncertainty contribution due to inhomogeneity
u_{st}	(standard) uncertainty contribution due to instability
$U(x_i)$	reported expanded uncertainty by participant "i"
$U(x_{pt})$	expanded uncertainty of the assigned value
x_i	reported mean value by participant "i"
x_{pt}	assigned value
z (or z')	z (or z') score
ζ	zeta score

1. Introduction

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM), hosted by the European Commission, DG Joint Research Centre, organised a proficiency test (PT) for the determination of the mass fraction of total Al, Ni, Sb and Zn in food simulant B solution (acetic acid, 3 % w/v). The background of this PT is the change of the specific migration limit of Zn and the introduction of a specific migration limit of Al as per 14 September 2018, and the introduction of a specific migration limit of Ni as per 19 May 2019. Sb was added because some laboratories performed less in a previous PT.

This PT was agreed with the Directorate General for Health and Food Safety (DG SANTE) as part of the EURL-FCM annual work programme 2018. The PT was open to National Reference Laboratories (NRLs) and to Official Control Laboratories (OCLs) willing to participate.

This report summarises the outcome of the PT.

2. Scope

As stated in Regulations (EC) No 882/2004 [1] and (EU) 2017/625 [2] one of the core duties of EURLs is to organise interlaboratory comparisons for the benefit of NRLs.

The present PT aims to assess the performance of NRLs and OCLs in the determination of the mass fractions of total Al, Ni, Sb and Zn in a food simulant B (FS-B) solution. In addition, participants were asked to evaluate the conformity of the investigated food simulant solution according to the specific migration limit (SMLs) set in Commission Regulations 10/2011, 2016/1416 and 2017/752 [3-5] concerning plastic materials and articles intended to come into contact with food.

The reported results were assessed following the administrative and logistic procedures of the JRC Unit in charge of the EURL-FCM, which is accredited for the organisation of PTs according to ISO 17043:2010 [6].

This PT is identified as FCM-18-02.

3. Set up of the exercise

3.1 Time frame

The organisation of this PT was agreed at the EURL-NRL-FCM Plenary Meeting held in Ispra on October 24-26, 2017. An invitation letter was sent (via e-mail) to the NRLs of the EURL-FCM network on March 26, 2018 (Annex 1). The registration deadline was set to May 18, 2018. Test items were sent to participants on June 12-13, 2018. The dispatch was monitored by the PT coordinator using the messenger's parcel tracking system on the internet. The deadline for reporting of results was set to July 27, 2018.

3.2 Confidentiality

The procedures used for the organisation of PTs are accredited according to ISO 17043:2010 [6] and guarantee that the identity of the participants and the information provided by them is treated as confidential. However, the laboratory codes of NRLs appointed in line with Regulation (EC) 2017/625 [2] may be disclosed to DG SANTE upon request for the purpose of an assessment of their (long-term) performance. Similarly, laboratory codes of appointed OCLs may be disclosed to their respective NRL upon request.

3.3 Distribution

Each participant received:

- One bottle of the test item (containing approx. 50 mL of test item);
- The "Test item accompanying letter" (Annex 2); and
- A "Confirmation of receipt form" to be sent back to the JRC after receipt of the test item (Annex 3).

All test items were dispatched at room temperature.

3.4 Instructions to participants

Detailed instructions were given to participants in the "Test item accompanying letter" mentioned above.

The measurand was defined as "mass fraction of total Al, Ni, Sb and Zn in a food simulant B solution".

Participants were asked to perform two or three independent measurements applying the method used for official control, to report their calculated mean (x_i) and the associated expanded measurement uncertainty ($U(x_i)$) together with the coverage factor (k) and the analytical technique used for analysis.

A density of 1.0 g mL^{-1} was used as conversion factor for the test solution. Results had to be reported in the same format (e.g. number of significant figures) as normally reported to customers.

Since the homogeneity study was performed with 50 µL sample intakes, the recommended minimum sample intake was set to 50 µL.

Participants were informed that the procedure used for the analysis should resemble as closely as possible their routine procedures for this type of matrix/analytes and mass fraction levels.

Participants received an individual code to access the on-line reporting interface, to report their measurement results and to complete the related questionnaire. A dedicated questionnaire was used to gather additional information related to measurements and laboratories (Annex 4).

The laboratory codes were given randomly and communicated to the participants by e-mail. NRLs were identified as N-xx and OCL as O-xx.

4. Test item

4.1 Preparation

Five litres of food simulant B solution (FS-B, 3 % acetic acid w/v [3]) were prepared and spiked with Al, Ni, Sb and Zn by the EURL-FCM. For each element, the spiked concentration was close to the respective legislated SML listed in mg kg^{-1} of food. A density of 1 g cm^{-3} was assumed for the FS-B solution. Portions of approximately 50 mL were manually filled into 50 mL screw capped Schott vials, and then stored at 4 °C until shipment.

4.2 Homogeneity and stability

Measurements for the homogeneity and stability studies were performed at the JRC-Geel. Inductively coupled plasma mass spectrometry (ICP-MS) was used to determine the mass fractions of total Al, Ni, Sb and Zn.

The statistical treatment of data was performed by the EURL-FCM.

The assessment of homogeneity was performed after the test item was packed in its final form and before distribution to participants. Ten bottles were randomly selected and analysed in duplicates. Results were evaluated according to ISO 13528:2015 [7]; the contribution from homogeneity (u_{hom}) to the standard uncertainty of the assigned value ($u(x_{pt})$) was calculated using the SoftCRM software [8]. The test item proved to be adequately homogeneous for all investigated analytes (Annex 5 and Table 1).

The stability study confirmed that the test item is adequately stable (i) at room temperature (*ca.* 20 °C) over the whole period of the PT (8 weeks, from value assignment till the deadline for reporting of results); and (ii) for 1 week at 60 °C (thus simulating extreme conditions which may occur during transport). Hence, the uncertainty contribution due to stability was set to zero (u_{st} = 0, Annex 5 and Table 1).

5. Assigned values and corresponding uncertainties

5.1 Assigned values

The assigned values (x_{pt}) of the mass fraction of total Al, Ni, Sb and Zn in the spiked food simulant B solution were derived from formulation by the EURL-FCM (Table 1). These values were obtained by gravimetric measurements of dilutions, using substitution weighing with buoyancy correction, and from the certificates of the reference materials (element solutions) used to spike the FS-B solution. These values were further confirmed experimentally in the frame of the homogeneity study (Annex 5).

5.2 Associated uncertainties

The associated standard uncertainties of the assigned values ($u(x_{pt})$) were calculated following the law of uncertainty propagation, combining the standard measurement uncertainty of the value assignment (test item characterization, u_{char}) with the standard uncertainty contributions from homogeneity (u_{hom}) and stability (u_{st}), in compliance with ISO 13528:2015 [7]:

$$u(x_{pt}) = \sqrt{u_{char}^2 + u_{hom}^2 + u_{st}^2} \qquad \text{Eq. 1}$$

where u_{char} was estimated from formulation, according to ISO/IEC Guide 98-3 [9].

5.3 Standard deviation for proficiency assessment, σ_{pt}

The following relative standard deviations for proficiency assessment (σ_{pt}) were set based on expert opinion (Table 1):

(i) 15 % of the assigned value for Al, Ni and Sb; and

(ii) 12 % of the assigned value for Zn.

Table 1: Assigned values (x_{pt}, derived from formulation), standard measurement uncertainty, u_{char} , u_{hom} , u_{st}, and $u(x_{pt})$ and standard deviation for PT assessment, σ_{pt}. All values (except the last two rows) expressed in mg kg^{-1}.

	Al	Ni	Sb	Zn
x_{pt}	**0.801**	**0.0202**	**0.102**	**5.024**
u_{char}	0.0025	0.00005	0.0004	0.0125
u_{hom}	0.0106	0.00010	0.0010	0.0305
u_{st}	0	0	0	0
$u(x_{pt})$	**0.011**	**0.00011**	**0.0010**	**0.0330**
σ_{pt}	0.120	0.0030	0.015	0.603
σ_{pt} (%)	**15%**	**15%**	**15%**	**12%**
$u(x_{pt})/\sigma_{pt}$	0.09	0.04	0.07	0.05

6. Evaluation of results

6.1 Performance scores and evaluation criteria

The individual laboratory performance was expressed in terms of z and ζ performance scores according to ISO 13528:2015 [7]:

$$z = \frac{x_i - x_{pt}}{\sigma_{pt}} \qquad \text{Eq. 2}$$

$$\zeta = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}} \qquad \text{Eq. 3}$$

Where: x_i is the measurement result reported by a participant;

$u(x_i)$ is the standard measurement uncertainty reported by a participant;

x_{pt} is the assigned value;

$u(x_{pt})$ is the standard measurement uncertainty of the assigned value;

σ_{pt} is the standard deviation for proficiency assessment.

The interpretation of the z and ζ performance scores was done as follows [7]:

$\lvert score \rvert \le 2$	satisfactory performance	(green in Annexes 6 to 10)
$2 < \lvert score \rvert < 3$	questionable performance	(yellow in Annexes 6 to 10)
$\lvert score \rvert \ge 3$	unsatisfactory performance	(red in Annexes 6 to 10)

The z scores compare the participant's deviation from the assigned value with the standard deviation for proficiency test assessment (σ_{pt}) used as common quality criterion.

The ζ scores state whether the laboratory's result agrees with the assigned value within the respective uncertainty. The denominator is the combined uncertainty of the assigned value $u(x_{pt})$ and the measurement uncertainty as stated by the laboratory $u(x_i)$. The ζ score includes all parts of a measurement result, namely the expected value (assigned value), its measurement uncertainty in 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 measurement, or of its measurement uncertainty, or both.

The standard measurement uncertainty of the laboratory $u(x_i)$ was obtained by dividing the reported expanded measurement uncertainty by the reported coverage factor, *k*. When no uncertainty was reported, it was set to zero ($u(x_i)$ = 0) by the PT coordinator.

Uncertainty estimation is not trivial, therefore an additional assessment was provided to each laboratory reporting measurement uncertainty, indicating how reasonable their measurement uncertainty estimation was.

The standard measurement uncertainty from the laboratory $u(x_i)$ is most likely to fall in a range between a minimum and a maximum allowed uncertainty (case a": $u_{min} \leq u_i \leq u_{max}$). u_{min} is set to the standard uncertainty of the assigned value $u(x_{pt})$. It is unlikely that a laboratory carrying out the analysis on a routine basis would determine the measurand with a smaller measurement uncertainty than the expert laboratories chosen to establish the assigned value. u_{max} is set to the standard deviation accepted for the PT assessment (σ_{pt}). Consequently, case "a" becomes: $u(x_{pt}) \leq u(x_i) \leq \sigma_{pt}$.

If $u(x_i)$ is smaller than $u(x_{pt})$ (case "b") the laboratory may have underestimated its measurement uncertainty. Such a statement has to be taken with care as each laboratory reported only measurement uncertainty, whereas the measurement uncertainty associated with the assigned value also includes contributions for homogeneity and stability of the test item. If those are large, measurement uncertainties smaller than $u(x_{pt})$ are possible and plausible.

If $u(x_i)$ is larger than σ_{pt} (case "c") the laboratory may have overestimated its measurement uncertainty. An evaluation of this statement can be made when looking at the difference between the reported value and the assigned value: if the difference is smaller than the expanded uncertainty $U(x_{pt})$ then overestimation is likely. If the difference is larger but x_i agrees with x_{pt} within their respective expanded measurement uncertainties, then the measurement uncertainty is properly assessed resulting in a satisfactory performance expressed as a ζ score, though the corresponding performance, expressed as a *z* score, may be questionable or unsatisfactory.

It should be pointed out that "u_{max}" is a normative criterion when set by legislation.

6.2 General observations

Twenty six NRLs representing the EU Member States except Latvia, Malta and Romania, and Switzerland, and twenty six OCLs registered to the exercise. All of them except one reported at least a result for one of the measurands. Only 20 NRLs and 17 OCLs reported results for all the four elements investigated.

The following data sets were evaluated: 47, 48, 39 and 45 for Al, Ni, Sb and Zn, respectively. Laboratory O-45 only reported two truncated values ("less than") for Ni and Zn. After the reception of the preliminary report, N-29 acknowledged having erroneously inverted the results for Ni and Sb.

The experimental details are provided in Annex 10.

6.3 Laboratory results and scorings

6.3.1 Performances

Annexes 6 to 9 present the tables of results, the graphical representation, and the corresponding Kernel density plot obtained using the software available from the Statistical Subcommittee of the Analytical Methods Committee of the UK Royal Society of Chemistry [10].

The laboratory performance for the determination of Al, Ni, Sb and Zn in the FS-B solution was assessed using z and ζ scores (Figure 1). Most of the laboratories having reported results performed satisfactorily for the four elements: 85 % and above according to the z score and 70 % and above for Al, Ni and Zn according to the ζ score (60 % for Sb). Table 2 presents the laboratories having reported questionable and unsatisfactory results according to the z scores.

The following instrumental techniques were applied: ICP-MS (64 %), GF-AAS (16 %) and ICP-OES (12 %). No correlation could be identified between the technique used and the participant performance. The experimental details are provided in Annex 10.

Table 2: Questionable and unsatisfactory performance expressed as a z score

	Reporting		z scores	
	NRLs	OCLs	Questionable	Unsatisfactory
Al	24	23	N12, N31	
Ni	25	25	N31	N-29*, N-36, O-09, O-22
Sb	21	18	N-10, O-23	N-24, N-29*, O-09, O-22
Zn	24	23	O-06, O-23, O-26	O-43

*inverted results (see main text)

6.3.2 Measurement uncertainties

Figure 1c shows that 70 % and above of the participants reported realistic measurement uncertainty (MU) evaluations (case "a": $u(x_{pt}) \leq u(x_i) \leq \sigma_{pt}$) for Al, Ni, Sb and Zn. Three to seven laboratories reported unrealistically small MU (smaller than $u(x_{pt})$ derived from formulation) probably based on replicate measurements. As for the seemingly over-estimated relative MU, laboratories may have applied the Thomson modified Horwitz equation and reported relative MU of the order of 20 %, which was higher than the selected σ_{pt}. Only two laboratories (N-07 and O-42) may have erroneously reported MU in the wrong unit (in % instead of mg kg^{-1}).

Fig. 1a

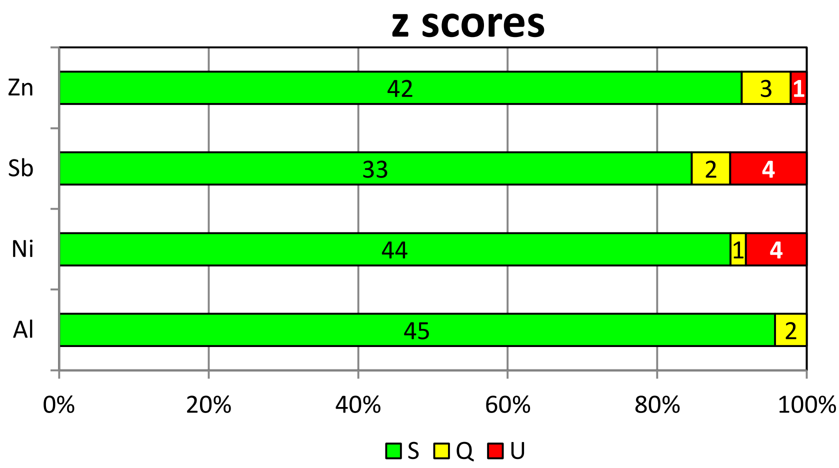


Fig. 1b

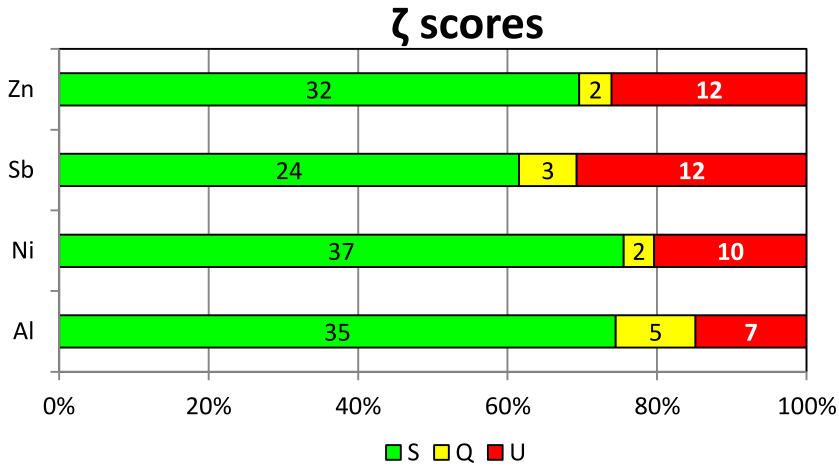


Fig. 1c

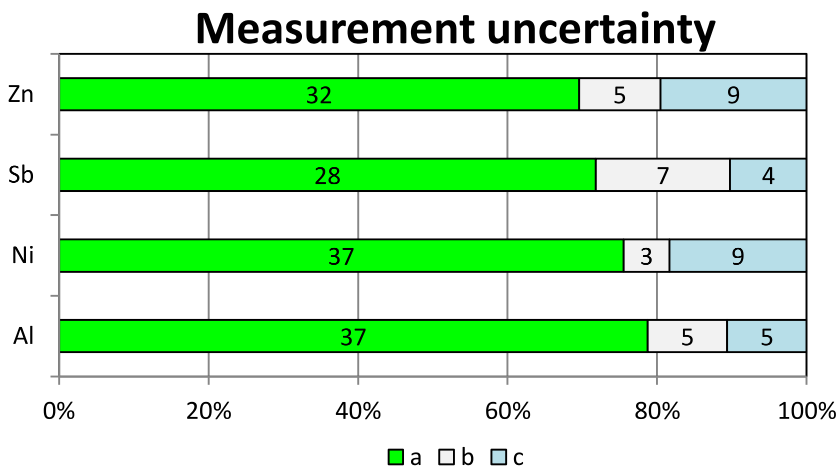


Figure 1: Overview of laboratory performance according to z and ζ scores, and measurement uncertainty evaluation for Al, Ni, Sb and Zn. Corresponding number of laboratories included in the graphs. Satisfactory (S), questionable (Q) and unsatisfactory (U) performances are indicated in green, yellow and red. Similarly cases "a"; "b"; "c" for MU are indicated in green, white and blue, respectively.

6.3.3 Compliance assessment

Commission Regulation 10/2011 [3] and Regulations (EU) 2016/1416 [4] and 2017/752 [5] concerning plastic materials and articles intended to come into contact with food set specific migration limits (SML) of 1.0 mg kg^{-1}, 0.02 mg kg^{-1}, 0.04 mg kg^{-1} and 5.0 mg kg^{-1} for Al, Ni, Sb and Zn, respectively (expressed as the mass fractions of the respective trace element per kg food or food simulant solution, assuming a density of 1 g cm^{-3} for this solution).

Participants were requested to assess the compliance of the test item, and to provide proper justification supporting their statement.

The assigned value for Sb (0.102 $\pm$ 0.002 mg kg^{-1} (k = 2)) clearly exceeds the set SML. The test item is therefore considered to be not compliant due to the Sb content only. Hence, participants that did not analyse Sb in the test item could not properly assess compliance.

In order to assess the consistency of the laboratory compliance statement, the following three components have to be considered:

i. The laboratory compliance statement (compliant or not compliant);
ii. The laboratory measurement results:
   - reported (or not) for the analyte of interest (particularly relevant for Sb);
   - to be compared to the relevant SML (x_i - $U(x_i)$ > SML ?)
     selecting the correct SML for the intended element;
iii. The laboratory justification for its compliance assessment (correct or incorrect).

The following conclusions were drawn from the information provided by the 51 participants:

- 12 laboratories (of which 2 NRLs) did not provide any compliance statement;
- 9 laboratories (5 NRLs) erroneously assessed the test item to be compliant without determining the Sb content, based on the satisfactory results for the other three elements;
- 5 laboratories incorrectly assessed the test item as compliant based either on their low (unsatisfactory) Sb results or on the grossly over-estimated measurement uncertainties;
- 18 laboratories (11 NRLs) analysed properly all the elements but drew the wrong conclusion;
- Only 7 laboratories (5 NRLs) correctly assessed the test item to be non-compliant.

6.3.4 Additional information extracted from the questionnaire

The questionnaire was answered by a majority of the participants giving valuable information on the laboratories, their way of working and their analytical methods. Annex 10 summarises the experimental details extracted from the questionnaire.

The majority of the participants stated to be accredited for the determination of trace elements in food simulant B solution and that they followed a standard method of analysis. They acknowledged having participated to similar PTs in the past.

The majority of the participant laboratories analyse ca. 50 "similar" samples per year, while 7 laboratories stated to have no experience with this type of analysis.

7. Conclusions

The proficiency test FCM-18-02 was organised to assess the analytical capabilities of the EU NRLs and OCLs on the determination of the mass fractions of total Al, Ni, Sb and Zn in a food simulant B solution. The test item was designed to be non-compliant based on the high antimony content spiked - well above the specific migration limit specified in the legislation.

The overall performance of the participants, ranging from 85 to 96 % (expressed as a z score), was satisfactory. The majority of the participants reported realistic measurement uncertainty evaluations. This confirms the analytical capabilities of EU NRLs and OCLs to enforce the Commission Regulations 10/2011, 2016/1416 and 2017/752 concerning plastic materials and articles intended to come into contact with food.

However, only seven laboratories (out of 51) correctly assessed the test item to be not compliant according to the relevant EU legislation.

Acknowledgements

The EURL-FCM wishes to thank colleagues from the JRC-Geel site for their valuable contributions during the processing of the proficiency test item.

The laboratories listed hereafter are kindly acknowledged for their participation in the PT.

Organisation	Country
AGES - Austrian Agency for Health & Food Safety	Austria
Sciensano	Belgium
National Center of Public Health and Analyses	Bulgaria
Croatian Institute of Publicc Health	Croatia
Ministry of Health	Cyprus
National Institute of Public Health (SZÚ)	Czech Republic
The Danish Veterinary and Food Administration	Denmark
Health Board	Estonia
Finnish Customs Laboratory	Finland
LNE	France
Service Commun des Laboratoires	France
German Federal Institute for Risk Assessment (BfR)	Germany
Thueringer Landesamt fuer Verbraucherschutz	Germany
Landesbetrieb Hessisches Landeslabor	Germany
Landeslabor Schleswig-Holstein	Germany
Chemisches und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe	Germany
CVUA-OWL (Chemisches u. Veterinäruntersuchungsamt OWL)	Germany
CVUA Stuttgart	Germany
General Chemical State Laboratory	Greece
National Food Chain Safety Office	Hungary
Public Analyst Laboratory Dublin	Ireland
Isttituto Superiore di Sanità	Italy
ARPA PIEMONTE	Italy
ARPA Umbria	Italy
ATS della Città Metropolitana di Milano	Italy
ARPA FVG	Italy
ARPAL	Italy
ARPALAZIO	Italy
APA PUGLIA	Italy
Istituto Zooprofilattico Sperimentale Del Piemonte, Liguria e Valle D'Aosta	Italy
Azienda USL Toscana centro	Italy
Settore Laboratorio -APPA	Italy
ATS Insubria - Varese Laboratorio Chimico	Italy
istituto zooprofilattico sperimentale lombardia emilia romagna	Italy
National Public Health Surveillance Laboratory	Lithuania
Laboratoire National de Santé	Luxembourg
NVWA	Netherlands
National Institute of Public Health – National Institute of Hygiene	Poland
WSSE Rzeszów	Poland
Escola Superior de Biotecnologia - Universidade Católica Portuguesa	Portugal
Regional Public Health Authority	Slovakia
National Laboratory of Health, Environment and Food	Slovenia
Centro Nacional Alimentacion. AECOSAN	Spain
Public Health Laboratory of Alicante	Spain
Laboratorio Regional La Grajera	Spain
Centro Analítico de Inspección y Control de Calidad de Comercio Exterior	Spain
Laboratorio de Salud Pública de Galicia	Spain
Junta de Castilla Y Leon	Spain
National Food Agency	Sweden
Amt für Verbraucherschutz	Switzerland
Fera Science Ltd	United Kingdom

References

[1] Commission Regulation, (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules.

[2] Regulation (EU) 2017/625 of the European Parliament and of the Council on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products.

[3] Commission Regulation (EU) 10/2011 on plastic materials and articles intended to come into contact with food.

[4] Commission Regulation (EU) 2016/1416 amending and correcting Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food.

[5] Commission Regulation (EU) 2017/752 amending and correcting Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food.

[6] ISO/IEC 17043:2010 "Conformity assessment – General requirements for proficiency testing", issued by ISO-Geneva (CH), International Organization for Standardization.

[7] ISO 13528:2015 "Statistical methods for use in proficiency testing by interlaboratory comparisons", issued by ISO-Geneva (CH), International Organization for Standardization, 2015.

[8] SoftCRM, (n.d.). http://www.eie.gr/iopc/softcrm/index.html.

[9] ISO/IEC Guide 98-3: "Uncertainty of measurement -- Part 3: Guide to the expression of uncertainty in measurement (GUM), issued by ISO-Geneva (CH), International Organization for Standardization.

[10] Analytical Methods Committee, "Representing data distributions with kernel density estimates", AMC Tech. Br. 4 (2006) 2. http://www.rsc.org/images/brief4_tcm18-25925.pdf.

Annex 1: Invitation letter

EUROPEAN COMMISSION
Joint Research Centre
Directorate F – Health, Consumers & Reference Materials
European Union Reference Laboratory for Food Contact Materials

Geel, 26 March 2018

(sent by e-mail)

Subject: Invitation to participate in FCM-18-02 PT round

Dear National Reference Laboratory representative,

On behalf of the EURL for Food Contact Materials, we would like to invite you to participate in the Proficiency Test round "FCM-18-02".

The measurand for the current PT are the mass fractions of the total Al, Ni, Sb and Zn in food simulant B (assuming the density of food simulant B is 1 g cm^{-3}).

The PT has been set-up under the EURL-FCM mandate under Regulation (EC) No 882/2004 and 2017/625. According to Regulation (EC) No 882/2004 it is your duty as NRL to participate in PTs organised by the EURL-FCM.

Your participation is free of charge.

Please register electronically by using the link below and following the instructions on screen.

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1961

Once you have submitted your registration electronically, you will have to:

- Print your registration form, as indicated on screen
- Sign it, date it and send it to us by e-mail (JRC-EURL-FCM@ec.europa.eu)

Please register by Friday the 18th of May 2018.

Please inform us how many OCLs would be interested in participating, together with their addresses, contact number and responsible person. They should register electronically by using the link above.

Samples will be dispatched at the end of May 2018.

The deadline for submission of results is the 15th of July 2018.

Do not hesitate to contact us if you have any further questions.

Kind regards,

/signed electronically in Ares/
Dr. F. Cordeiro
FCM-18-02 PT Coordinator

/signed electronically in Ares/
Dr. E. Hoekstra
Operating Manager EURL-FCM

Cc: Hendrik Emons (Head of Unit, Food & Feed Compliance, F.5)

Annex 2: Test item accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Directorate F – Health, Consumers and Reference Materials
European Union Reference Laboratory for Food Contact Materials

Geel, 04 June 2018
Ares(2018)2903297

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

Subject: **Participation in the Proficiency Test round "FCM-18-02"**

Dear «Title» «Surname»,

Thank you for participating in the FCM-18-02 for the **"Determination of the mass fractions of the total Al, Ni, Sb and Zn in food simulant B"** (assuming the density of food simulant B is 1 g cm^{-3}). The PT fulfils EURL-FCM mandate under Regulation (EC) No 882/2004 and 2017/625.

Please keep this letter. You will need it to report your results.

The parcel you received contains, in addition to this letter:

- One glass bottle labelled "PT code: FCM-18-02; Sample Nr: xxxx" (with approx. 50 mL of acetic acid 3% (w/v), food simulant B);
- The "Confirmation of receipt" form.

Upon arrival of this parcel, please check whether the test items are undamaged after transport, and send us by email the **"Confirmation of receipt"** form, to JRC-EURL-FCM@ec.europa.eu.

Sample should be kept in the fridge at +4°C.

The measurand is:

"The mass fraction of the total Al, Ni, Sb and Zn in food simulant B"

The procedure used for the analyses should resemble as closely as possible the one you use in routine analyses.

Perform the measurements and report:

- The **mean** of your results (in mg kg^{-1}) using 2 significant figures (e.g. 0.033 mg kg^{-1}), (assuming that the density of food simulant B is 1 g cm^{-3}),
- The associated expanded **uncertainty** (in mg kg^{-1}) using 2 significant figures,
- The **coverage factor for the calculation of expanded uncertainty.**

Retieseweg 111, 2440 Geel, Belgium
https://ec.europa.eu/jrc/en/eurl/food-contact-materials ; E-mail: JRC-EURL-FCM@ec.europa.eu

The test item homogeneity is demonstrated at a certain sample intake level: **please use a sample intake for analysis not lower than 1 ml.**

The reporting is done using the software MILC (the same that was used for registration): https://web.jrc.ec.europa.eu/ilcReportingWeb

To assess the webpage you need the following personal password key: «Part_key».

The system will guide you through the reporting procedure. You will also need to complete the corresponding questionnaire.

Do not forget to confirm your results after submission using MILC.

Please check carefully your report. If mistakes are detected, please contact the PT coordinator as soon as possible before the reporting deadline.

The deadline for submission of results is the **27th July 2018**.

The procedures used for the organisation of PTs are accredited according to ISO/IEC 17043:2010 and guarantee that the identity of the participants and the information provided by them is treated as confidential. However, lab codes of National Reference Laboratories (NRLs) appointed in line with Regulation (EC) No 882/2004 as amended by Regulation (EC) 2017/625 will be disclosed to DG SANTE upon request for (long-term) performance assessment. Lab codes of appointed Official Control Laboratories (OCLs) may be disclosed to their NRLs upon request.

Remember 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.

Do not hesitate to contact me for further information.

With kind regards,

/signed electronically in Ares/

Dr. Fernando Cordeiro
FCM-18-02 Coordinator

Cc: H. Emons (Head of Unit, Food & Feed Compliance unit)
E. Hoekstra (Operating Manager of EURL-FCM)

Retieseweg 111, 2440 Geel, Belgium
https://ec.europa.eu/jrc/en/eurl/food-contact-materials ; E-mail: JRC-EURL-FCM@ec.europa.eu

Annex 3: Confirmation of receipt form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Directorate F – Health, Consumers and Reference Materials
European Union Reference Laboratory for Food Contact Materials

EURL
European Union Reference Laboratory
for Food Contact Materials

Geel, 04 June 2018
Ares(2018)2903297

«Title» «Firstname» «Surname»
«Organisation»
«Department»
«Country»

Subject: "Confirmation receipt" form Proficiency Test FCM-18-02 "Metals from Plastics"

Please return this form at your earliest convenience, to confirm that the package arrived well. If samples are damaged, mention it under "Remarks" and contact us as soon as possible.

Date of package arrival ______________________________

Remarks ______________________________

____

Signature ______________________________

Thank you for returning this form by email to:

Dr. Fernando Cordeiro
FCM-18-02 Coordinator

E-mail : JRC-EURL-FCM@ec.europa.eu

Retieseweg 111, 2440 Geel, Belgium
https://ec.europa.eu/jrc/en/eurl/food-contact-materials ; E-mail: JRC-EURL-FCM@ec.europa.eu

Annex 4: Questionnaire

Milc questionnaire

Comparison for FCM-18-02

Submission Form

1. **Are you a National Reference Laboratory (NRL)?**

☐ a) Yes

☐ b) No

1.1. If "No" have you been nominated by your NRL?

☐ a) Yes

☐ b) No

1.1.1. If "Yes" please identify your NRL

[ ]

2. **Considering your reported values do you consider the test item in compliance with the European legislation?**

☐ a) Test item compliant

☐ b) Test item not compliant

3. **Analytical method ("x" where applicable)**

See table **Analytical method** at bottom

4. **Analytical method (LOD / Dilution factor)**

See table **Analytical method (details)** at bottom

5. **Does your laboratory carry this type of analysis on a regular basis?**

See table **Laboratory experience (samples per year, "x" where applicable)** at bottom

6. **What was the basis for your measurement uncertainty evaluation**

See table **Measurement uncertainty evaluation (use a to g)** at bottom

7. **Do you usually provide an uncertainty statement to your customers?**

☐ a) Yes

☐ b) No

8. **Does your laboratory have a quality management system?**

☐ a) Yes

☐ b) No

9. **If "Yes" based on which standard?**

☐ a) ISO 17025

☐ b) ISO 9001 series

☐ c) Other

10. **Does your laboratory participate in interlaboratory comparisons for this type of analysis?**

☐ a) Yes

☐ b) No

Analytical method

Questions/Response table	*Standard method?*	*Validated method?*	*Accredited method?*
Al			
Ni			
Sb			
Zn			

Analytical method (details)

Questions/Response table	*LOD (mg/kg)*	*Dilution factor used*	*Isotope used (ICP-MS) or wavelenght (nm, ICP-OES)*
Al			
Ni			
Sb			
Zn			

Laboratory experience (samples per year, "x" where applicable)

Questions/Response table	*1) 0-50*	*2) 50-250*	*3) 250-1000*	*4) > 1000*	*5) Never*
Al					
Ni					
Sb					
Zn					

Measurement uncertainty evaluation (use a to g)

Questions/Response table	*Al*	*Ni*	*Sb*	*Zn*
a) Uncertainty budget (ISO GUM)				
b) Known uncertainty of standard method (ISO 21748)				
c) From in-house method validation				
d) Measurement of replicates (precision)				
e) Evaluation based on judgment				
f) From interlaboratory comparison				
g) Other (please specify)				

Annex 5: Homogeneity and stability studies

(all values in mg kg-1 or %)

A5.1 Homogeneity study

	Al		Ni		Sb		Zn	
Bottle	R_1	R_2	R_1	R_2	R_1	R_2	R_1	R_2
1	0.794	0.815	0.0199	0.0203	0.096	0.102	5.056	5.100
2	0.789	0.784	0.0201	0.0204	0.099	0.102	5.056	5.086
3	0.797	0.791	0.0205	0.0203	0.100	0.101	5.083	5.086
4	0.798	0.784	0.0202	0.0202	0.100	0.102	5.09	5.041
5	0.781	0.778	0.0204	0.0204	0.100	0.102	5.095	5.022
6	0.833	0.811	0.0208	0.0205	0.104	0.102	5.239	5.126
7	0.813	0.807	0.0203	0.0204	0.099	0.102	5.184	5.148
8	0.81	0.806	0.0209	0.0202	0.103	0.101	5.113	5.086
9	0.797	0.804	0.0201	0.0203	0.098	0.101	5.102	5.129
10	0.788	0.808	0.0204	0.0205	0.101	0.101	4.993	5.116
Mean	0.7994		0.0204		0.101		5.098	
S_w	0.0090		0.0001		0.002		0.045	
S_{bb}	0.0106		0.0001		0.001		0.031	
u_{hom}	1.3%		0.5%		0.9%		0.6%	
σ_{pt}	15%		15%		15%		12%	
$S_{bb} < 0.3\sigma_{pt}$	**Pass**		**Pass**		**Pass**		**Pass**	
	Test item adequately homogeneous							

$\boldsymbol{S_w}$: within-bottle standard deviation, $\boldsymbol{S_{bb}}$: between-bottle standard deviation,
$\boldsymbol{u_{hom}}$: standard uncertainty contribution due to inhomogeneity

A5.2 Stability study (at 20 °C for 8 weeks)

	Al	Ni	Sb	Zn
Y_1	0.799	0.0200	0.098	5.098
Y_2	0.822	0.0201	0.101	5.135
$\lvert Y_1-Y_2 \rvert$	0.023	0.0001	0.003	0.037
x_{pt}	0.801	0.0202	0.102	5.02
σ_{pt}	0.12	0.0030	0.015	0.60
$0.3\sigma_{pt}$	0.036	0.001	0.005	0.181
$\lvert Y_1-Y_2 \rvert < 0.3\sigma_{pt}$	**Pass**	**Pass**	**Pass**	**Pass**

Y_1 and Y_2: mean of results at zero and 8 weeks, respectively.

Test item adequately stable, $\boldsymbol{u_{stb}}$ set to zero.

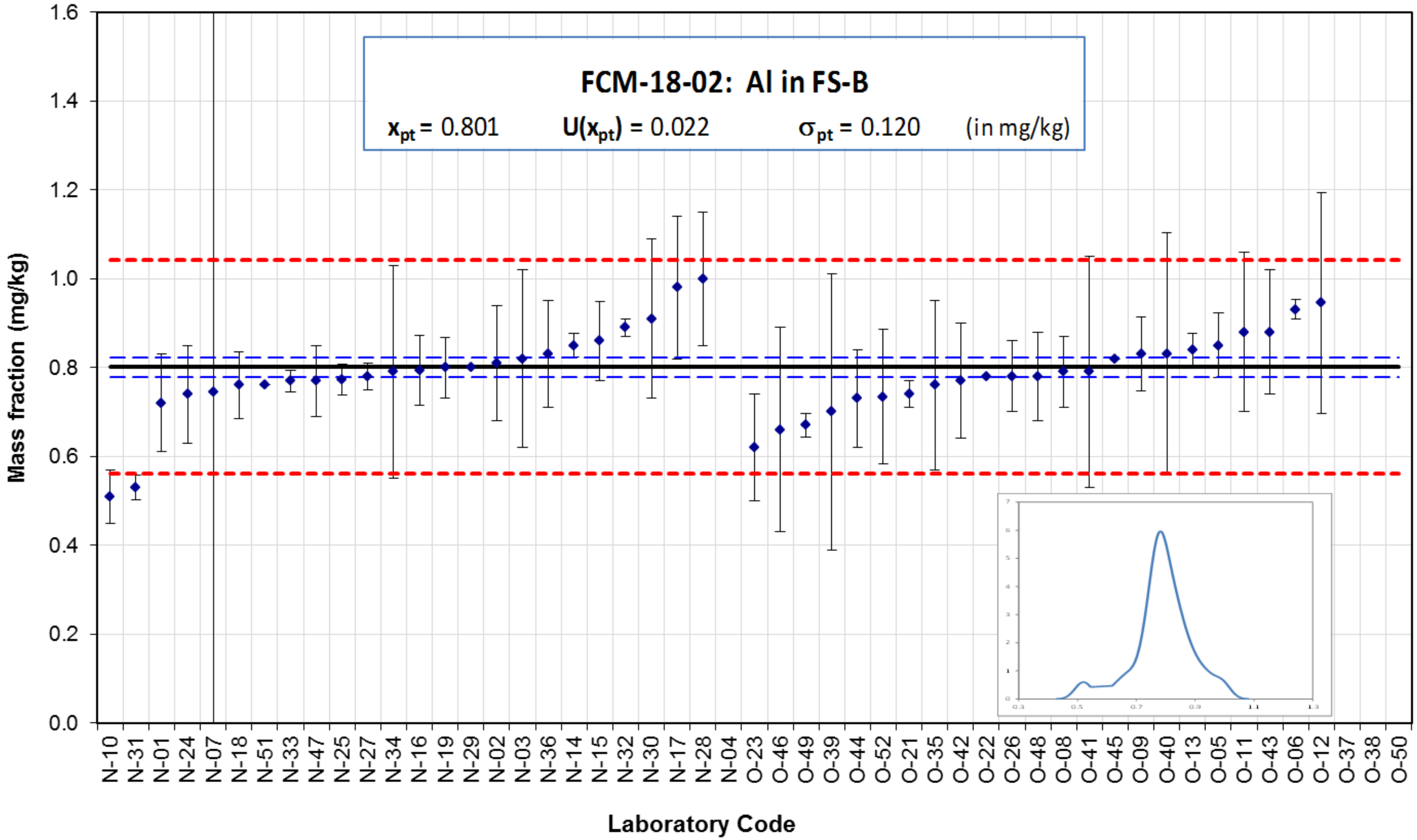
Annex 6: Results for mass fraction of total Al in FS-B

Assigned range ($x_{pt} \pm U(x_{pt})$, $k = 2$): 0.801 ± 0.022; and σ_{pt} = 0.120 (x_i, $U(x_i)$, $u(x_i)$ in mg kg^{-1}).

Lab Code	x_i	$U(x_i)$	k	Technique	$u(x_i)$	z score	ζ score	unc.
N-01	0.72	0.11	2	ICP-MS	0.055	-0.7	-1.4	a
N-02	0.81	0.13	2	ICP-MS	0.065	0.1	0.1	a
N-03	0.82	0.2	2	ICP-MS	0.100	0.2	0.2	a
N-04								
N-07	0.745	2.5	2	ICP-MS	1.250	-0.5	0.0	c
N-10	0.51	0.06	2	ICP-MS	0.030	-2.4	-9.1	a
N-14	0.85	0.027	2	ICP-MS	0.014	0.4	2.8	a
N-15	0.86	0.089	2	GF-AAS	0.045	0.5	1.3	a
N-16	0.793	0.079	2	ICP-MS	0.040	-0.1	-0.2	a
N-17	0.98	0.16	2	GF-AAS	0.080	1.5	2.2	a
N-18	0.76	0.075	2	ICP-MS	0.038	-0.3	-1.0	a
N-19	0.80	0.068	2	ICP-MS	0.034	0.0	0.0	a
N-24	0.74	0.11	2	ICP-OES	0.055	-0.5	-1.1	a
N-25	0.773	0.034	2	ICP-MS	0.017	-0.2	-1.4	a
N-27	0.78	0.03	2	GF-AAS	0.015	-0.2	-1.1	a
N-28	1.0	0.15	2	GF-AAS	0.075	1.7	2.6	a
N-29	0.80			ICP-MS	0.000	0.0	-0.1	b
N-30	0.91	0.18	2	ICP-MS	0.090	0.9	1.2	a
N-31	0.53	0.027	2	ICP-MS	0.014	-2.3	-15.6	a
N-32	0.89	0.02	2	ICP-OES	0.010	0.7	6.0	b
N-33	0.77	0.024	2	GF-AAS	0.012	-0.3	-1.9	a
N-34	0.79	0.24	2	ICP-MS	0.120	-0.1	-0.1	a
N-36	0.83	0.12	4.303	ICP-OES	0.028	0.2	1.0	a
N-47	0.77	0.08	2	ICP-OES	0.040	-0.3	-0.7	a
N-51	0.76			GF-AAS	0.000	-0.3	-3.8	b
O-05	0.85	0.073	2	ICP-MS	0.037	0.4	1.3	a
O-06	0.93	0.022	2	ICP-MS	0.011	1.1	8.3	a
O-08	0.79	0.08	2	ICP-MS	0.040	-0.1	-0.3	a
O-09	0.83	0.083	2	ICP-OES	0.042	0.2	0.7	a
O-11	0.88	0.18	2	ICP-MS	0.090	0.7	0.9	a
O-12	0.945	0.248	2	ICP-MS	0.124	1.2	1.2	c
O-13	0.84	0.036	2.3534	ICP-MS	0.015	0.3	2.1	a
O-21	0.74	0.03	2	ICP-OES	0.015	-0.5	-3.3	a
O-22	0.78			FAAS-HG	0.000	-0.2	-1.9	b
O-23	0.62	0.12	2	ICP-MS	0.060	-1.5	-3.0	a
O-26	0.78	0.08	2.13	ICP-MS	0.038	-0.2	-0.5	a
O-35	0.76	0.19	2	ICP-OES	0.095	-0.3	-0.4	a
O-37								
O-38								
O-39	0.70	0.31	2	ICP-MS	0.155	-0.8	-0.7	c
O-40	0.831	0.273	2	ICP-MS	0.137	0.2	0.2	c
O-41	0.79	0.26	2	ICP-OES	0.130	-0.1	-0.1	c
O-42	0.77	0.13	2	ICP-MS	0.065	-0.3	-0.5	a
O-43	0.88	0.14	2	ICP-MS	0.070	0.7	1.1	a
O-44	0.73	0.11	2	ICP-OES	0.055	-0.6	-1.3	a
O-45	0.82			ICP-MS	0.000	0.2	1.7	b
O-46	0.66	0.23	2	ICP-MS	0.115	-1.2	-1.2	a
O-48	0.78	0.1	2	ICP-MS	0.050	-0.2	-0.4	a
O-49	0.67	0.026	2	ICP-MS	0.013	-1.1	-7.7	a
O-50								
O-52	0.734	0.152	2	ICP-MS	0.076	-0.6	-0.9	a

Performance scores: satisfactory (green); questionable (yellow); unsatisfactory (red)

Measurement uncertainty assessment: a: $u(x_{pt}) \leq u_i \leq \sigma_{pt}$; b: $u_i < u(x_{pt})$; and c: $u_i > \sigma_{pt}$



Measurement result and associated expanded uncertainty as reported by participants

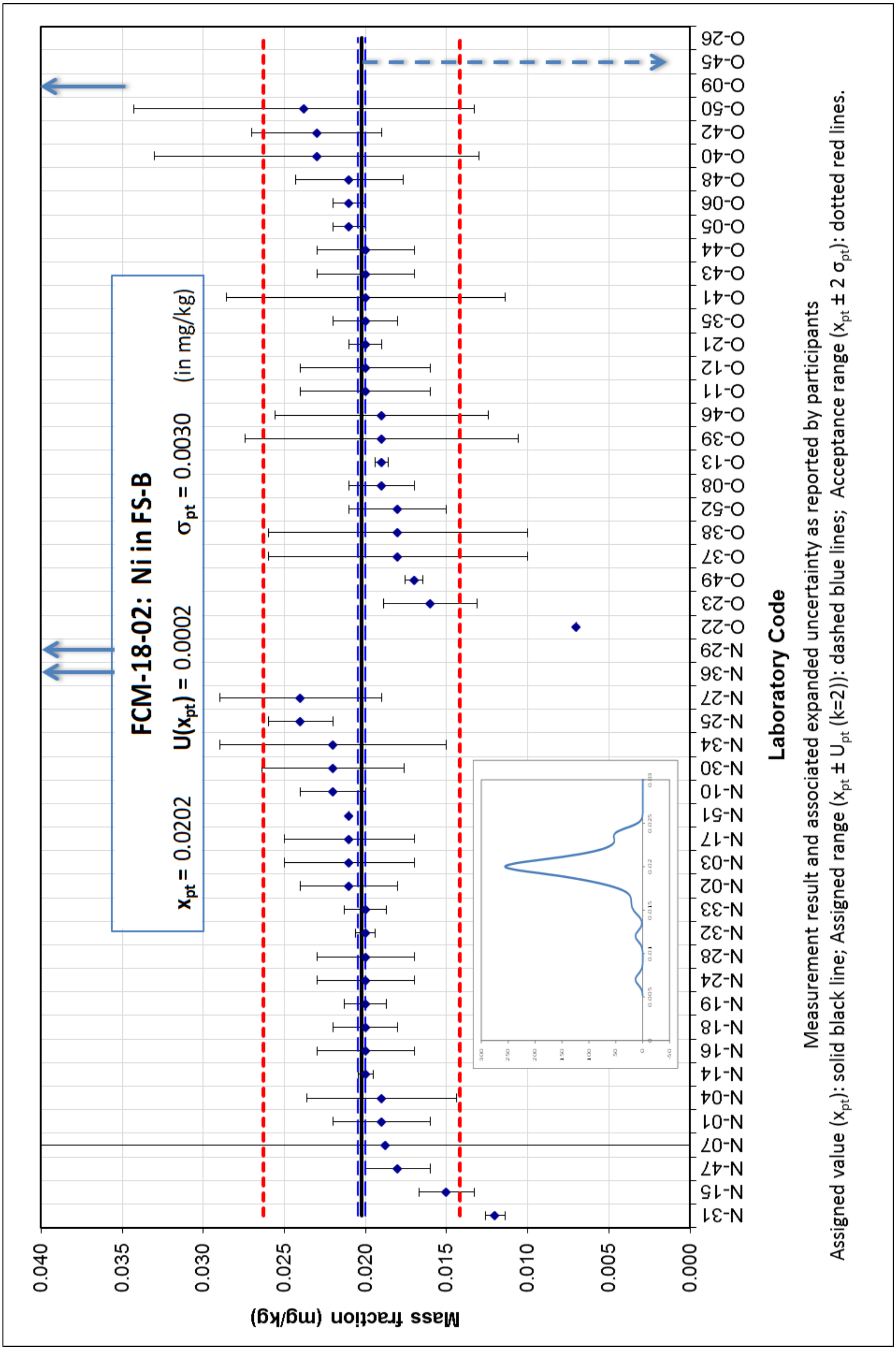
Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 7: Results for mass fraction of total Ni in FS-B

Assigned range ($x_{pt} \pm U(x_{pt})$, $k = 2$): 0.0202 ± 0.0002; and σ_{pt} = 0.0030 (x_i, $U(x_i)$, $u(x_i)$ in mg kg^{-1}).

Lab Code	x_i	$U(x_i)$	k	Technique	$u(x_i)$	z score	ζ score	unc.
N-01	0.019	0.003	2	ICP-MS	0.002	-0.4	-0.8	a
N-02	0.021	0.003	2	ICP-MS	0.002	0.3	0.5	a
N-03	0.021	0.004	2	ICP-MS	0.002	0.3	0.4	a
N-04	0.019	0.0046	2	GF-AAS	0.002	-0.4	-0.5	a
N-07	0.0188	3	2	ICP-MS	1.500	-0.5	0.0	c
N-10	0.022	0.002	2	ICP-MS	0.001	0.6	1.8	a
N-14	0.020	0.00046	2	ICP-MS	0.000	-0.1	-0.9	a
N-15	0.015	0.0017	2	GF-AAS	0.001	-1.7	-6.1	a
N-16	0.020	0.003	2	ICP-MS	0.002	-0.1	-0.2	a
N-17	0.021	0.004	2	GF-AAS	0.002	0.3	0.4	a
N-18	0.020	0.002	2	ICP-MS	0.001	-0.1	-0.2	a
N-19	0.020	0.0013	2	ICP-MS	0.001	-0.1	-0.3	a
N-24	0.020	0.003	2	ICP-OES	0.002	-0.1	-0.2	a
N-25	0.024	0.002	2	ICP-MS	0.001	1.2	3.7	a
N-27	0.024	0.005	2	GF-AAS	0.003	1.2	1.5	a
N-28	0.020	0.003	2	GF-AAS	0.002	-0.1	-0.2	a
N-29	0.085			ICP-MS	0.000	21.3	573.2	b
N-30	0.022	0.0044	2	ICP-MS	0.002	0.6	0.8	a
N-31	0.012	0.0006	2	ICP-MS	0.000	-2.7	-25.7	a
N-32	0.02	0.0006	2	ICP-OES	0.000	-0.1	-0.7	a
N-33	0.020	0.0013	2	GF-AAS	0.001	-0.1	-0.3	a
N-34	0.022	0.007	2	ICP-MS	0.004	0.6	0.5	c
N-36	0.062	0.0082	4.303	GF-AAS	0.002	13.8	21.9	a
N-47	0.018	0.002	2	ICP-MS	0.001	-0.7	-2.2	a
N-51	0.021			GF-AAS	0.000	0.3	6.8	b
O-05	0.021	0.001	2	ICP-MS	0.001	0.3	1.5	a
O-06	0.021	0.001	2	ICP-MS	0.001	0.3	1.5	a
O-08	0.019	0.002	2	ICP-MS	0.001	-0.4	-1.2	a
O-09	0.103	0.0041	2	ICP-OES	0.002	27.3	40.3	a
O-11	0.020	0.004	2	ICP-MS	0.002	-0.1	-0.1	a
O-12	0.020	0.004	2	ICP-MS	0.002	-0.1	-0.1	a
O-13	0.019	0.00042	2.3534	ICP-MS	0.000	-0.4	-5.8	a
O-21	0.02	0.001	2	ICP-MS	0.001	-0.1	-0.4	a
O-22	0.007			FAAS-HG	0.000	-4.4	-117.1	b
O-23	0.016	0.0029	2	ICP-MS	0.001	-1.4	-2.9	a
O-26								
O-35	0.020	0.002	2	ICP-MS	0.001	-0.1	-0.2	a
O-37	0.018	0.008	2	GF-AAS	0.004	-0.7	-0.6	c
O-38	0.018	0.008	2	GF-AAS	0.004	-0.7	-0.6	c
O-39	0.019	0.0084	2	ICP-MS	0.004	-0.4	-0.3	c
O-40	0.023	0.01	2	ICP-MS	0.005	0.9	0.6	c
O-41	0.020	0.0086	2	ICP-OES	0.004	-0.1	-0.1	c
O-42	0.023	0.004	2	ICP-MS	0.002	0.9	1.4	a
O-43	0.020	0.003	2	ICP-MS	0.002	-0.1	-0.2	a
O-44	0.020	0.003	2	ICP-OES	0.002	-0.1	-0.2	a
O-45	< 0.20			GF-AAS				
O-46	0.019	0.0066	2	ICP-MS	0.003	-0.4	-0.4	c
O-48	0.021	0.0033	2	ICP-MS	0.002	0.3	0.5	a
O-49	0.017	0.00056	2	ICP-MS	0.000	-1.1	-10.7	a
O-50	0.0238	0.0105	2	GF-AAS	0.005	1.2	0.7	c
O-52	0.018	0.003	2	ICP-MS	0.002	-0.7	-1.5	a

Performance scores: satisfactory (green); questionable (yellow); unsatisfactory (red)
Measurement uncertainty assessment: a: $u(x_{pt}) \le u_i \le \sigma_{pt}$; b: $u_i < u(x_{pt})$; and c: $u_i > \sigma_{pt}$



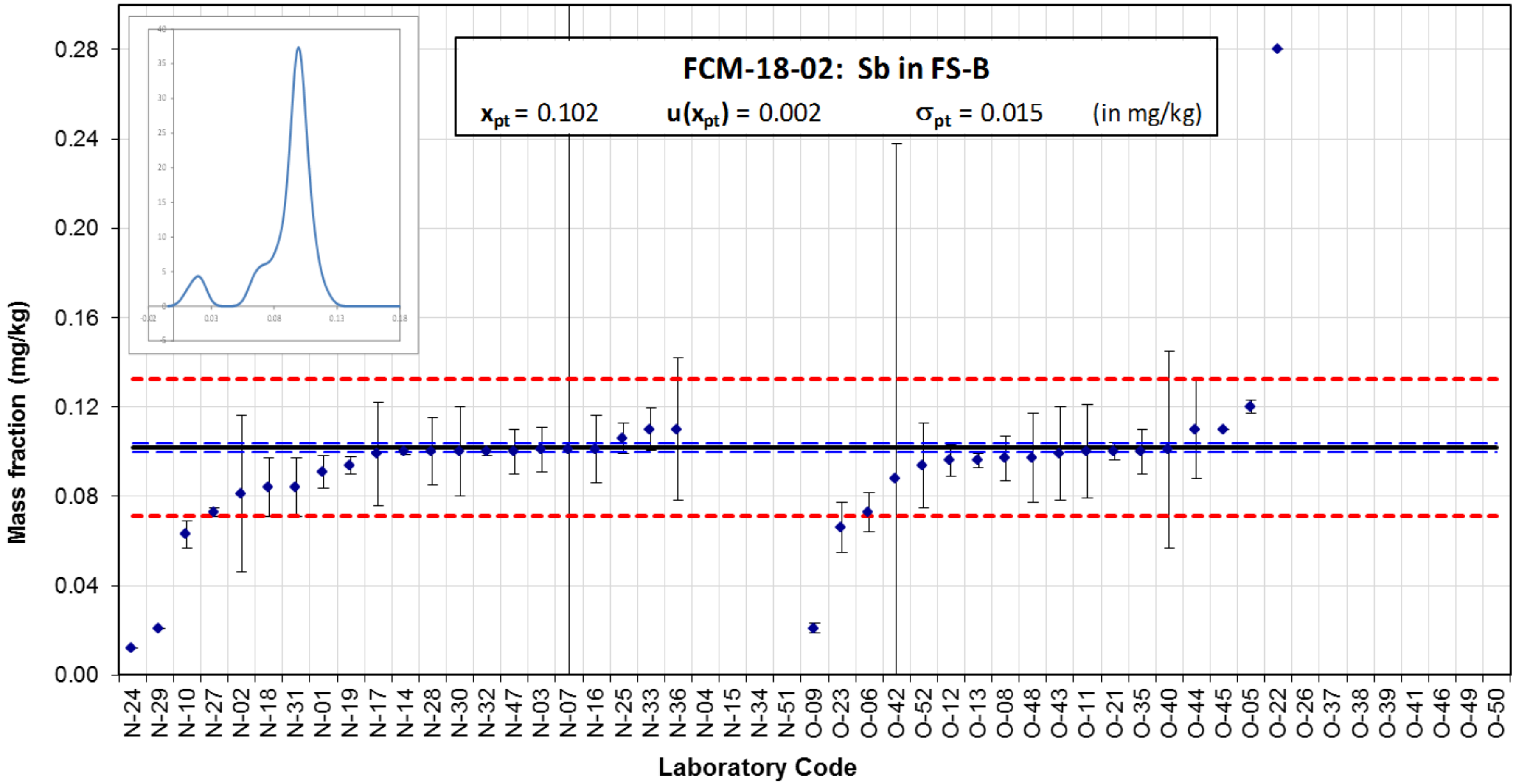
FCM-18-02: Ni in FS-B
x_{pt} = 0.0202
$U(x_{pt})$ = 0.0002
σ_{pt} = 0.0030
(in mg/kg)
Mass fraction (mg/kg)
Laboratory Code
Measurement result and associated expanded uncertainty as reported by participants
Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 8: Results for mass fraction of total Sb in FS-B

Assigned range ($x_{pt} \pm U(x_{pt})$, k = 2): 0.102 ± 0.002; and σ_{pt} = 0.015 (x_i, $U(x_i)$, $u(x_i)$ in mg kg^{-1}).

Lab Code	x_i	$U(x_i)$	k	Technique	$u(x_i)$	z score	ζ score	unc.
N-01	0.091	0.0073	2	ICP-MS	0.004	-0.7	-2.8	a
N-02	0.081	0.035	2	ICP-MS	0.018	-1.4	-1.2	c
N-03	0.101	0.01	2	ICP-MS	0.005	-0.1	-0.2	a
N-04								
N-07	0.101	2	2	ICP-MS	1.000	-0.1	0.0	c
N-10	0.063	0.006	2	ICP-MS	0.003	-2.5	-12.2	a
N-14	0.10	0.0014	2	ICP-MS	0.001	-0.1	-1.4	b
N-15								
N-16	0.101	0.015	2	ICP-MS	0.008	-0.1	-0.1	a
N-17	0.099	0.023	2	GF-AAS	0.012	-0.2	-0.2	a
N-18	0.084	0.013	2	ICP-MS	0.007	-1.2	-2.7	a
N-19	0.094	0.0039	2	ICP-MS	0.002	-0.5	-3.5	a
N-24	0.012			ICP-MS	0.000	-5.9	-86.7	b
N-25	0.106	0.007	2	ICP-MS	0.004	0.3	1.2	a
N-27	0.073	0.002	2	GF-AAS	0.001	-1.9	-20.0	b
N-28	0.10	0.015	2	GF-AAS	0.008	-0.1	-0.2	a
N-29	0.021			ICP-MS	0.000	-5.3	-78.0	b
N-30	0.10	0.02	2	ICP-MS	0.010	-0.1	-0.2	a
N-31	0.084	0.013	2	ICP-MS	0.007	-1.2	-2.7	a
N-32	0.1	0.002	2	ICP-OES	0.001	-0.1	-1.3	b
N-33	0.11	0.0096	2	FAAS-HG	0.005	0.5	1.7	a
N-34								
N-36	0.11	0.032	4.303	GF-AAS	0.007	0.5	1.1	a
N-47	0.10	0.01	2	ICP-MS	0.005	-0.1	-0.4	a
N-51								
O-05	0.12	0.003	2	ICP-MS	0.002	1.2	10.0	a
O-06	0.073	0.0087	2	ICP-MS	0.004	-1.9	-6.4	a
O-08	0.097	0.01	2	ICP-MS	0.005	-0.3	-0.9	a
O-09	0.021	0.0021	2	ICP-OES	0.001	-5.3	-54.8	a
O-11	0.10	0.021	2	ICP-MS	0.011	-0.1	-0.2	a
O-12	0.096	0.007	2	ICP-MS	0.004	-0.4	-1.6	a
O-13	0.096	0.003	2.3534	ICP-MS	0.001	-0.4	-3.5	a
O-21	0.1	0.004	2	ICP-MS	0.002	-0.1	-0.8	a
O-22	0.28			FAAS-HG	0.000	11.7	172.1	b
O-23	0.066	0.011	2	ICP-MS	0.006	-2.3	-6.4	a
O-26								
O-35	0.10	0.01	2	ICP-MS	0.005	-0.1	-0.4	a
O-37								
O-38								
O-39								
O-40	0.101	0.044	2	ICP-MS	0.022	-0.1	0.0	c
O-41								
O-42	0.088	0.15	2	ICP-MS	0.075	-0.9	-0.2	c
O-43	0.099	0.021	2	ICP-MS	0.011	-0.2	-0.3	a
O-44	0.11	0.022	2	ICP-OES	0.011	0.5	0.7	a
O-45	0.11			ICP-MS	0.000	0.5	7.9	b
O-46								
O-48	0.097	0.02	2	ICP-MS	0.010	-0.3	-0.5	a
O-49								
O-50								
O-52	0.094	0.019	2	ICP-MS	0.010	-0.5	-0.8	a

Performance scores: satisfactory (green); questionable (yellow); unsatisfactory (red)
Measurement uncertainty assessment: a: $u(x_{pt}) \le u_i \le \sigma_{pt}$; b: $u_i < u(x_{pt})$; and c: $u_i > \sigma_{pt}$



Measurement result and associated expanded uncertainty as reported by participants

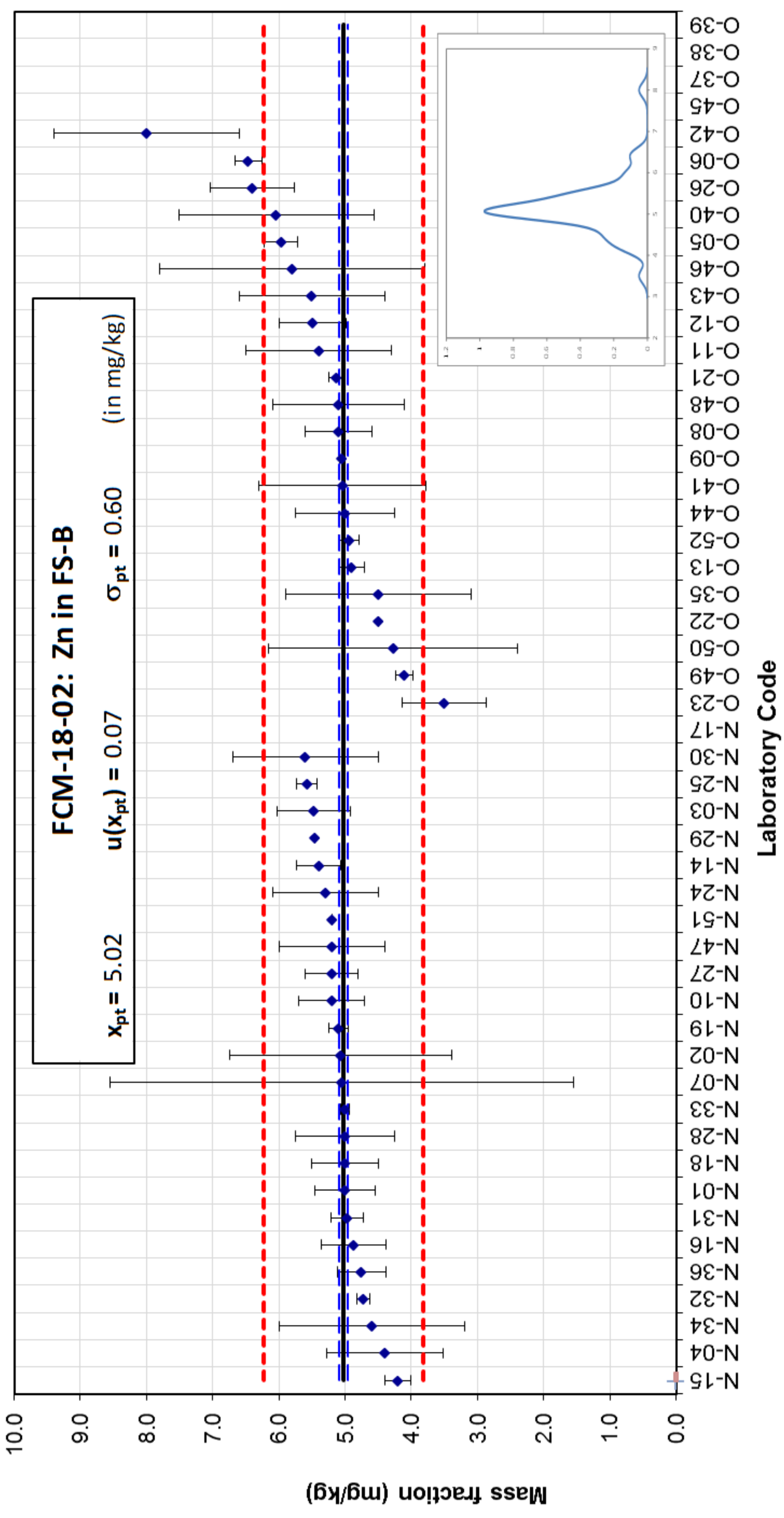
Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 9: Results for mass fraction of total Zn

Assigned range ($x_{pt} \pm U(x_{pt})$, k = 2): 5.02 ± 0.07; and σ_{pt} = 0.60 (x_i, $U(x_i)$, $u(x_i)$ in mg kg^{-1}).

Lab Code	x_i	$U(x_i)$	k	Technique	$u(x_i)$	z score	ζ score	unc.
N-01	5.0	0.45	2	ICP-MS	0.225	0.0	-0.1	a
N-02	5.07	1.68	2	ICP-MS	0.840	0.1	0.1	c
N-03	5.47	0.55	2	ICP-MS	0.275	0.7	1.6	a
N-04	4.4	0.88	2	FAAS-HG	0.440	-1.0	-1.4	a
N-07	5.054	3.5	2	ICP-MS	1.750	0.0	0.0	c
N-10	5.2	0.5	2	ICP-MS	0.250	0.3	0.7	a
N-14	5.4	0.33	2	ICP-MS	0.165	0.6	2.2	a
N-15	4.2	0.2	2	FAAS-HG	0.100	-1.4	-7.8	a
N-16	4.869	0.487	2	ICP-MS	0.244	-0.3	-0.6	a
N-17								
N-18	5.0	0.5	2	ICP-MS	0.250	0.0	-0.1	a
N-19	5.10	0.15	2	ICP-MS	0.075	0.1	0.9	a
N-24	5.3	0.8	2	ICP-OES	0.400	0.5	0.7	a
N-25	5.577	0.155	2	ICP-MS	0.078	0.9	6.6	a
N-27	5.2	0.4	2	FAAS-HG	0.200	0.3	0.9	a
N-28	5.0	0.75	2	FAAS-HG	0.375	0.0	-0.1	a
N-29	5.45			ICP-MS	0.000	0.7	12.9	b
N-30	5.6	1.1	2	ICP-MS	0.550	1.0	1.0	a
N-31	4.97	0.25	2	ICP-MS	0.125	-0.1	-0.4	a
N-32	4.73	0.097	2	ICP-OES	0.049	-0.5	-5.0	a
N-33	5.0	0.062	2	FAAS-HG	0.031	0.0	-0.5	b
N-34	4.6	1.4	2	ICP-MS	0.700	-0.7	-0.6	c
N-36	4.75	0.37	4.303	ICP-OES	0.086	-0.5	-3.0	a
N-47	5.2	0.8	2	ICP-MS	0.400	0.3	0.4	a
N-51	5.2			ICP-OES	0.000	0.3	5.3	b
O-05	5.97	0.25	2	ICP-MS	0.125	1.6	7.3	a
O-06	6.46	0.2	2	ICP-MS	0.100	2.4	13.6	a
O-08	5.1	0.5	2	ICP-MS	0.250	0.1	0.3	a
O-09	5.05	0.04	2	ICP-OES	0.020	0.0	0.7	b
O-11	5.4	1.1	2	ICP-MS	0.550	0.6	0.7	a
O-12	5.488	0.503	2	ICP-MS	0.252	0.8	1.8	a
O-13	4.9	0.19	2.3534	ICP-MS	0.081	-0.2	-1.4	a
O-21	5.13	0.12	2	ICP-OES	0.060	0.2	1.5	a
O-22	4.5			FAAS-HG	0.000	-0.9	-15.9	b
O-23	3.5	0.64	2	ICP-MS	0.320	-2.5	-4.7	a
O-26	6.4	0.64	2.13	ICP-MS	0.300	2.3	4.6	a
O-35	4.5	1.4	2	ICP-MS	0.700	-0.9	-0.7	c
O-37								
O-38								
O-39								
O-40	6.04	1.474	2	ICP-MS	0.737	1.7	1.4	c
O-41	5.04	1.26	2	ICP-OES	0.630	0.0	0.0	c
O-42	8.0	1.4	2	ICP-MS	0.700	4.9	4.2	c
O-43	5.5	1.1	2	ICP-MS	0.550	0.8	0.9	a
O-44	5.0	0.75	2	ICP-OES	0.375	0.0	-0.1	a
O-45	< 20			FAAS				
O-46	5.8	2	2	ICP-MS	1.000	1.3	0.8	c
O-48	5.1	1	2	ICP-MS	0.500	0.1	0.2	a
O-49	4.1	0.13	2	ICP-MS	0.065	-1.5	-12.7	a
O-50	4.271	1.879	2	FAAS-HG	0.940	-1.2	-0.8	c
O-52	4.939	0.152	2	ICP-MS	0.076	-0.1	-1.0	a

Performance scores: satisfactory (green); questionable (yellow); unsatisfactory (red)
Measurement uncertainty assessment: a: $u(x_{pt}) \leq u_i \leq \sigma_{pt}$; b: $u_i < u(x_{pt})$; and c: $u_i > \sigma_{pt}$



Measurement result and associated expanded uncertainty as reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 10: Experimental details and performance (expressed as z scores)

Lab		Standard method?	Validated method?	Accredited method?	LOD (mg/kg)	Dilution factor	Isotope used (ICP-MS) or wavelength	Samples /Year	Measurement uncertainty evaluation	Quality management system
N-01	Al			Yes	0.01			0-50	From interlaboratory comparison	Yes
	Ni			Yes	0.005			0-50		
	Sb			Yes	0.001					
	Zn			Yes	0.01					
N-02	Al			Yes	0.02	10	27	0-50	NORDTEST like	Yes
	Ni			Yes	0.0008	10	60	0-50		
	Sb			Yes	0.0002	10	121			
	Zn			Yes	0.003	10	66			
N-03	Al		Yes		0.006	10	27	Never	From in-house method validation, evaluation based on judgment	Yes
	Ni		Yes		0.001	10	60	Never		
	Sb		Yes		0.0003	10	121			
	Zn		Yes		0.006	10	66			
N-04	Ni	Yes	Yes	Yes	0,000958			0-50	From in-house method validation	
	Zn	Yes	Yes	Yes	0,023	1:20				
N-07	Al		Yes		0,0002	100, 50,10	27	Never	Measurement of replicates (precision)	Yes
	Ni		Yes		0,003	1, 2	60	Never		
	Sb		Yes		0,0001	2,10	121			
	Zn		Yes		0,0002	50, 100	66			
N-10	Al	Yes	Yes		0.003	5	27	0-50	From in-house method validation	Yes
	Ni	Yes	Yes		0.00003	5	60	51-250		
	Sb	Yes	Yes		0.00001	100	121			
	Zn	Yes	Yes		0.0002	100	66			
N-15	Al	No	No	No	0.0029	20		Never	Measurement of replicates (precision)	Yes
	Ni	No	No	No	0.0023			0-50		
	Zn	No	No	No	0.01	10				

Lab		Standard method?	Validated method?	Accredited method?	LOD (mg/kg)	Dilution factor	Isotope used (ICP-MS) or wavelength	Samples/ Year	Measurement uncertainty evaluation	Quality management system
N-16	**Al**		Yes		0.025	1	27	0-50	Evaluation based on judgment	Yes
	Ni		Yes		0.001	1	60			
	Sb		Yes		0.001	1	101			
	Zn		Yes		0.01	1	66			
N-17	**Al**	Yes			0.002	10		0-50		Yes
	Ni	Yes	Yes	Yes	0.0003	-		51-250		
	Sb	Yes			0.0005	10				
N-18	**Al**	Yes	Yes	Yes	0.008	10	27	51-250	From in-house method validation, measurement of replicates (precision)	Yes
	Ni	Yes	Yes	Yes	0.00024	10	60	51-250		
	Sb	Yes	Yes	Yes	0.00012	10	121	51-250		
	Zn	Yes	Yes	Yes	0.002	10	66	51-250		
N-19	**Al**		Yes		0,01	10	27	51-250	From in-house method validation	Yes
	Ni		Yes		0,005	5	60	51-250		
	Sb		Yes		0,005	5	121			
	Zn		Yes		0,01	50	66			
N-24	**Al**	No	No	No	0.19	1	308.2 nm	Never	Measurement of replicates (precision), evaluation based on judgment	Yes
	Ni	No	Yes	No	0.004	1	231.6 nm	0-50		
	Sb	No	No	No		5 and 10	121			
	Zn	No	Yes	No	0.013	1 and 10	206.2 nm			
N-25	**Al**		Yes		0.004	10	27	51-250	From in-house method validation	Yes
	Ni		Yes		0.001	neat	60	51-250		
	Sb		Yes		0.0001	10	121			
	Zn		Yes		0.002	100	64,66			
N-27	**Al**	Yes						0-50	Uncertainty budget (ISO GUM)	Yes
	Ni	Yes						0-50		
	Sb	Yes								
	Zn			Yes						

Lab		Standard method?	Validated method?	Accredited method?	LOD (mg/kg)	Dilution factor	Isotope used (ICP-MS) or wavelength	Samples/ Year	Measurement uncertainty evaluation	Quality management system
N-28	Al	Yes			0.003	50		0-50	Evaluation based on judgment	Yes
	Ni	Yes			0.003	20		0-50		
	Sb	Yes			0.006	100				
	Zn	Yes			0.015	10				
N-30	Al	Yes			0.003	10	27	0-50	From in-house method validation	Yes
	Ni	Yes			0.001	10	60	0-50		
	Sb	Yes			0.003	10	121			
	Zn	Yes			0.003	10	66			
N-31	Al	Yes	Yes		0.2	1	27	51-250	From in-house method validation	Yes
	Ni	Yes	Yes		0.009	1	58	51-250		
	Sb	Yes	Yes		0.02	1	121			
	Zn	Yes	Yes		0.2	1	64			
N-32	Al		Yes				167 nm	0-50	Measurement of replicates (precision)	Yes
	Ni		Yes				221 nm	0-50		
	Sb		Yes				217 nm			
	Zn		Yes				202 nm			
N-33	Al		Yes		0.11	50	309.3 nm	0-50	Uncertainty budget (ISO GUM)	Yes
	Ni		Yes		0.002	No	232 nm	0-50		
	Sb		Yes		0.0021	7	217.6 nm			
	Zn		Yes		0.1	10	213.9 nm			
N-34	Al				0.01	10	27	51-250	Evaluation based on judgment	Yes
	Ni				0.002	10	60	51-250		
	Zn				0.01	10	64			
N-36	Al	Yes			0.050	No	396.1 nm	Never	Measurement of replicates (precision)	Yes
	Ni	Yes			0.010	0.2% HNO_3		Never		
	Sb	Yes			0.005	0.2% HNO_3		Never		
	Zn	Yes			0.030	2% HNO_3	206,2 nm	0-50		

Lab		Standard method?	Validated method?	Accredited method?	LOD (mg/kg)	Dilution factor	Isotope used (ICP-MS) or wavelength	Samples/ Year	Measurement uncertainty evaluation	Quality management system
N-47	Al	Yes			0.028	1.5	394.4 nm	Never	From in-house method validation	Yes
	Ni			Yes	0.000013	1.5	60	0-50		
	Sb			Yes	0.000037	1.5	66			
	Zn	Yes			0.21	150	121			
N-51	Al		Yes		0.001	50	309.3 nm	0-50	From in-house method validation	Yes
	Ni		Yes		0.001	4	232 nm	0-50		
	Zn		Yes		0.05	2	213.9 nm			
O-05	Al	Yes	Yes	Yes	0.0002		27	251-1000	From in-house method validation	Yes
	Ni	Yes	Yes	Yes	0.00003		60	251-1000		
	Sb	Yes	Yes	Yes	0.000002		121	251-1000		
	Zn	Yes	Yes	Yes	0.0002		66	251-1000		
O-06	Al	Yes	Yes	Yes	0.01	10	27	0-50	Measurement of replicates (precision)	Yes
	Ni	Yes	Yes	Yes	0.001	10	60	0-50		
	Sb	Yes	Yes	Yes	0.001	10	121	Never		
	Zn	Yes	Yes	Yes	0.01	40	66	Never		
O-08	Al		Yes	Yes	0.01	1-4	27	Never	From in-house method validation	Yes
	Ni		Yes	Yes	0.0005	No	62	Never		
	Sb		Yes	Yes	0.0005	1-4	121			
	Zn		Yes	Yes	0.01	1-20	66			
O-09	Al	Yes	Yes	Yes	0.025	2	167 nm	51-250	NORDTEST	Yes
	Ni	Yes	Yes	Yes	0.002	2	221.647 nm	51-250		
	Sb	Yes	Yes	Yes	0.01	2	217.581 nm			
	Zn	Yes	Yes	Yes						
O-11	Al	Yes	Yes	Yes	0.001	6	27	0-50	Evaluation based on judgment	Yes
	Ni	Yes	Yes	Yes	0.0001	60	60	0-50		
	Sb	Yes	Yes	Yes	0.0001	60	121			
	Zn	Yes	Yes	Yes	0.01	6	66			

Lab		Standard method?	Validated method?	Accredited method?	LOD (mg/kg)	Dilution factor	Isotope used (ICP-MS) or wavelength	Samples/ Year	Measurement uncertainty evaluation	Quality management system
O-12	Al	Yes	Yes	Yes	0.050	25	27	51-250	From in-house method validation	Yes
	Ni	Yes	Yes	Yes	0.0001		60	51-250		
	Sb	Yes	Yes	Yes	0.0001		121	51-250		
	Zn	Yes	Yes	Yes	0.001	100	66	51-250		
O-13	Al	Yes					27	Never	Measurement of replicates (precision)	Yes
	Ni	Yes					60	Never		
	Sb	Yes					121			
	Zn	Yes					66			
O-21	Al	Yes	Yes	Yes	0.0005			51-250	From in-house method validation	Yes
	Ni	Yes	Yes	Yes	0,0004			51-250		
	Sb	Yes	Yes	Yes	0,0002					
	Zn	Yes	Yes	Yes	0.0002					
O-22	Al	Yes			1	1	309.3 nm	Never		Yes
	Ni	Yes			1	1	232 nm	Never		
	Sb	Yes			2	1	217.6 nm	Never		
	Zn	Yes			0.1	1	213.9 nm	Never		
O-23	Al		Yes		1.60E-03	1/10 and 1/5	27	0-50	From in-house method validation	Yes
	Ni		Yes		9.10E-05	1/5	60	0-50		
	Sb		Yes		7.00E-06	1/20	121			
	Zn		Yes		1.30E-03	1/20	66			
O-26	Al				0.005	1	27	0-50		Yes
	Zn				0.005	1	64			
O-35	Al			Yes				0-50		Yes
	Ni			Yes				0-50		
	Sb			Yes				0-50		
	Zn			Yes				0-50		
O-37	Ni			Yes	0.0073	1		51-250	Horwitz / Thompson	Yes
O-38	Ni				0.0050			0-50	Horwitz	Yes
O-39	Al				0,02	1:5	27		Horwitz	Yes
	Ni				0,002	1:5	60			

Lab		Standard method?	Validated method?	Accredited method?	LOD (mg/kg)	Dilution factor	Isotope used (ICP-MS) or wavelength	Samples/ Year	Measurement uncertainty evaluation	Quality management system
O-40	Al	Yes						Never	Horwitz	
	Ni	Yes						Never		
	Sb	Yes						Never		
	Zn	Yes						Never		
O-41	Al	Yes			0.01	5	396.2 nm	0-50	Horwitz / Thompson	Yes
	Ni		Yes	Yes	0.01	1	231.6 nm	51-250		
	Zn	Yes			0.01	50	206.2 nm	Never		
O-43	Al	Yes			0.0008	5	27	Never	Measurement of replicates (precision)	Yes
	Ni	Yes			0.0003	5	60	Never		
	Sb	Yes			0.00001	30	121	Never		
	Zn	Yes			0.001	30	66	Never		
O-44	Al		Yes		0.01	1 and 4	237.3 nm	0-50	From in-house method validation	Yes
	Ni		Yes		0.003	1 and 2	221.6 nm	0-50		
	Sb		Yes		0.01	1 and 10	206.8 nm	0-50		
	Zn		Yes		0.01	8 and 20	202.5 nm	0-50		
O-45	Al				0.003		27	51-250		Yes
	Ni		Yes	Yes	0.054		232 nm	51-250	From in-house method validation	
	Sb				0.003		121	51-250		
	Zn						213.9 nm	51-250		
O-46	Al		Yes		0.01	1	27	Never	From in-house method validation	Yes
	Ni			Yes	0.003	1	60	0-50		
	Zn		Yes		0.02	1	66	Never		
O-48	Al	Yes			0.0007	10	27	0-50	Measurement of replicates (precision), from interlaboratory comparison	Yes
	Ni	Yes			0.00004	10	60	0-50		
	Sb	Yes			0.000002	50	121	0-50		
	Zn	Yes			0.0006	50	66	0-50		
O-49	Al			Yes	0,001	1	27	0-50	From in-house method validation	Yes
	Ni			Yes	0,0001	1	60	0-50		
	Zn			Yes	0,003	3	66	0-50		
O-52	Al		Yes	Yes	nd	0.1	Ge	51-250	From in-house method validation	Yes
	Ni		Yes	Yes	nd	0.002	Ge	51-250		
	Sb	Yes			nd	0.005	In	0-50	Evaluation based on judgment	
	Zn		Yes	Yes	nd	0.5	Ge	51-250	From in-house method validation	

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