BELGIAN NATIONAL REFERENCE LABORATORY FOR TRACE ELEMENTS IN FOOD AND FEED

PT-2022-NRL-TE-FASFC "Determination of As, Asi, Cd, Pb and Hg in unrefined salts"

FINAL REPORT ON THE 2022 PROFICIENCY TEST ORGANISED BY THE NATIONAL REFERENCE LABORATORY FOR TRACE ELEMENTS IN FOOD AND FEED

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Dr Grégoire Rondelet and Dr Karlien Cheyns

EXECUTIVE SUMMARY

From the 1st of January 2008, the laboratory for Trace Elements at Sciensano (former CODA-CERVA), Tervuren, operates as National Reference Laboratory for Trace Elements in Food and Feed (NRL-TE). One of its core tasks is to organise proficiency tests (PTs) among laboratories appointed by the Federal Agency for the Safety of the Food Chain. This report presents the results of the proficiency test organised by the NRL-TE which focused on the determination of trace elements in unrefined salts. The results from the PT were treated in Sciensano, Tervuren.

The 2022 PT was obligatory for all laboratories approved for the analysis of heavy metals in foodstuff by the Federal Agency for the Safety of the Food Chain (FASFC). Eleven laboratories registered for and participated in the exercise.

The test material used in this test was unrefined salt. The material was separated from the package after purchase and was spiked with Arsenic (As), Cadmiun (Cd) and Mercury (Hg). Each participant received approximately 15 g of test material.

Participants were invited to report the mean value and measurement uncertainty on their results for As, inorganic arsenic (Asi), Cd, lead (Pb) and Hg.

The assigned values (x_a) and their uncertainty ($u(x_a)$) were determined as the consensus of participant's results. Standard deviations for proficiency assessment were calculated using the modified Horwitz equation.

Of the 11 laboratories that registered for participation, 11 submitted results for As, Cd, Pb and Hg. All but one of the z-scores that were calculated, were satisfactory. Estimation of measurement uncertainty of the results of elements give overall satisfactory ζ-scores with 6 exceptions.

Five laboratories submitted results for As_i concentrations, with no quantified results. No conclusions are drawn for this analyte.

The performance of the laboratories to analyse As, Cd, Pb and Hg in this matrix was very succesfull.

CONTENTS

INTRODUCTION

Trace elements occur in varying amounts as natural elements in soils, plants and animals, and consequently in food and feed. To ensure public health, maximum levels for trace elements in foodstuff (including salt) have been laid down in the Commission Regulation (EC) N° 1881/2006 [1].

Next to maximum levels of Lead (Pb) and Cadmium (Cd); recently, the Commission Regulation (EU) 2022/617 of 12 April 2022 amending Regulation (EC) No 1881/2006 set a maximum level of Mercury (Hg) in salt [2] (Figure 1).

There is currently no European legislation regarding Arsenic (As) in salt.

Pb

	Foodstuffs (1)	Maximum levels (mg/kg wet weight)
3.1.27	Salt, except the following unrefined salts: 'fleur de sel' and 'grey salt' which are manually harvested from salt marshes with a clay bottom	1,0
	The following unrefined salts: 'fleur de sel' and 'grey salt' which are manually harvested from salt marshes with a clay bottom	2,0

Cd

3.2.21	Salt	0,50

Hg

3.3.5	Salt	0,10

Figure 1 : Snapshots of maximum limits of Pb, Cd and Hg (mg/kg) in salt as published in [1].

TIME FRAME, TEST MATERIAL AND INSTRUCTIONS TO PARTICIPANTS

Invitation letters to this PT were sent to participants in April (Annex 1). The 2022 PT was obligatory for all laboratories approved for the analysis of heavy metals in foodstuff by the Federal Agency for the Safety of the Food Chain (FASFC). Eleven laboratories registered for and participated in the exercise. The samples were dispatched to the participants by the end of May 2022. Reporting deadline was the 24th of June.

This year the test material was unrefined salt “Fleur de sel”. This type of salt is manually harvested as the first top layer that forms on the surface of salt marshes with a clay bottom. The sample was purchased in a local supermarket. After purchase, the material was spiked with As species (Dimethyl arsenic acid (DMMA) and methylarsenic acid (MMA)), Cd and Hg by adding element standards after dissolution of salts in bidistilled water (close to saturation point). After evaporation and crystallization in Petri dishes, the salts were harvested, grinded, mixed manually and divided in small containers. The samples were stored at ambient temperature.

The homogeneity of the test materials was tested following the recommended procedure according to IUPAC [3]. The trace elements appeared to be homogeneously distributed in the samples (Annex 2). Each participant received the test material samples, an accompanying letter (Annex 3) with instructions on sample handling (Annex 4), a receipt form (Annex 5) and a reporting form (Annex 6).

Participants were instructed to store the materials at ambient temperature until analysis. Before starting the analyses, the samples had to be homogenized according to the laboratoriums procedure. The procedure followed for the exercise, had to be as close as possible to the method used by the participant in routine sample analysis. The laboratories were asked to make a compliance statement based on their results.

A questionnaire was attached to the reporting form. The questionnaire was intended to provide further information on the measurements and the laboratories. A copy of the questionnaire is presented in Annex 6.

Laboratory codes were given randomly and communicated confidentially to the corresponding participant.

ASSIGNED VALUES

The assigned values for the different trace elements in unrefined salt “Fleur de sel” were determined as the consensus of participant’s results. The major advantages of consensus values are the straightforward calculation and the fact that none of the participants is accorded higher status. The disadvantages are that the consensus values are not independent of the participant’s results and, especially in the current case with 11 participants, that the uncertainty on the consensus (identified as the standard error) may be high and the information content of the z-scores will be correspondingly reduced. However, the IUPAC guide of 2010 on the selection and use of proficiency testing schemes for a limited number of participants [4] states that if the standard uncertainty of the assigned value $u(x_a)$ is insignificant in comparison to the fit-for-intended-use target standard deviation σ_p ($u(x_a)^2 < 0.1 * \sigma_p^2$), then z-scores can be calculated in a small scheme in the same matter as for a large scheme. This was the case for As (excluding the outlier), Cd, Pb and Hg, but not for Asi. A minimum of eight quantified results is accepted to calculate z- and ζ-scores (eight is the minimum number to create a Kernel density distribution).

First, it was checked whether the distribution of the reported results (the result of a laboratory is the average of the laboratories replicates) was apparently unimodal and roughly symmetric, possible extreme outliers aside. A Kernel distribution with a bandwith of 0.75 σ_p was plotted. It was analysed if this resulted in a unimodal and roughly symmetric kernel density, and if the mode and median were nearly coincident. If this was the case, robust statistics were accepted.

The ISO 13528:2015 guide was followed for the robust statistical analysis. There are many different robust estimators of mean ($\hat{\mu}_{rob}$) and standard deviation ($\hat{\sigma}_{rob}$) [5], [6]. The median and nIQR (normalised InterQuartile Range) were chosen here as robust estimators.

$$\hat{\mu}_{rob} = \text{median}\,(x)$$

$$\hat{\sigma}_{rob} = nIQR(x) = 0.7413(Q_3(x) - Q_1(x))$$

The standard uncertainty of the assigned value $u(x_a)$ was estimated as:

$$u(x_a) = 1.25\frac{\hat{\sigma}_{rob}}{\sqrt{n}}$$

With n the number of quantified results.

The factor 1.25 is based on the standard deviation of the median, or the efficiency of the median as an estimate of the mean. This factor has been recommended because proficiency testing results typically are not strictly normally distributed, and contain unknown proportions of results from different distributions.

The modified Horwitz equation was used to establish the standard deviation for proficiency testing (σ_p) [3][7]. It is an exponential relationship between the variability of chemical measurements and concentration. The Horwitz value is widely recognized as a fitness-for-purpose criterion in proficiency testing in food analysis.

For Asi as no quantified results were available, no value was assigned for this element and no scores were calculated.

The consensus values, their standard uncertainty and some other statistical parameters are summarised in Table 1.

Table 1 : Summary of statistical parameters for the test material.

	As mg/kg	Cd mg/kg	Pb mg/kg	Hg mg/kg
n (number of participants with quantifiable result)	10 [(1)]	11	11	11
Mean	0.075	0.77	0.40	0.30
Standard deviation (SD)	0.011	0.07	0.05	0.05
Robust mean (median)	0.072	0.80	0.39	0.32
Robust SD (nIQR)	0.012	0.08	0.05	0.05
Assigned value x_a	**0.072**	**0.80**	**0.39**	**0.32**
Standard uncertainty of the assigned value u(x_a)	**0.005**	**0.03**	**0.02**	**0.02**
σ_p	0.016	0.13	0.07	0.06

Assigned value x_a: median of the reported results, excluding outliers; σ_p: standard deviation for proficiency assessment. [(1)] one outlier

SCORES AND EVALUATION CRITERIA

Individual laboratory performances are expressed in terms of z-scores and ζ-scores in accordance with ISO 13528:2015 and the International Harmonised Protocol [3], [6].

$$z = \frac{x_{lab} - x_a}{\sigma_p}$$

$$\zeta = \frac{x_{lab} - x_a}{\sqrt{u^2(x_a) + u^2(x_{lab})}}$$

where:

x_{lab} is the mean of the individual measurement results as reported by the participant

x_a is the assigned value

σ_p is the standard deviation for proficiency assessment

$u(x_a)$ is the standard uncertainty for the assigned value

$u(x_{lab})$ is the reported standard uncertainty on the reported value x_{lab}. When no uncertainty was reported by the laboratory, it was set to zero.

The z-score compares the participant's deviation from the reference value with the standard deviation accepted for the proficiency test, σ_p. Should participants feel that these σ values are not fit for their purpose they can recalculate their scorings with a standard deviation matching their requirements.

The z-score can be interpreted as:

$\|z\| \leq 2$	satisfactory result
$2 < \|z\| \leq 3$	questionable result
$\|z\| > 3$	unsatisfactory result

The ζ-score states if the laboratory result agrees with the assigned value within the uncertainty claimed by this laboratory (taking due account of the uncertainty on the reference value itself). The interpretation of the ζ-score is similar to the interpretation of the z-score.

$\|\zeta\| \leq 2$	satisfactory result
$2 < \|\zeta\| \leq 3$	questionable result
$\|\zeta\| > 3$	unsatisfactory result

RESULTS

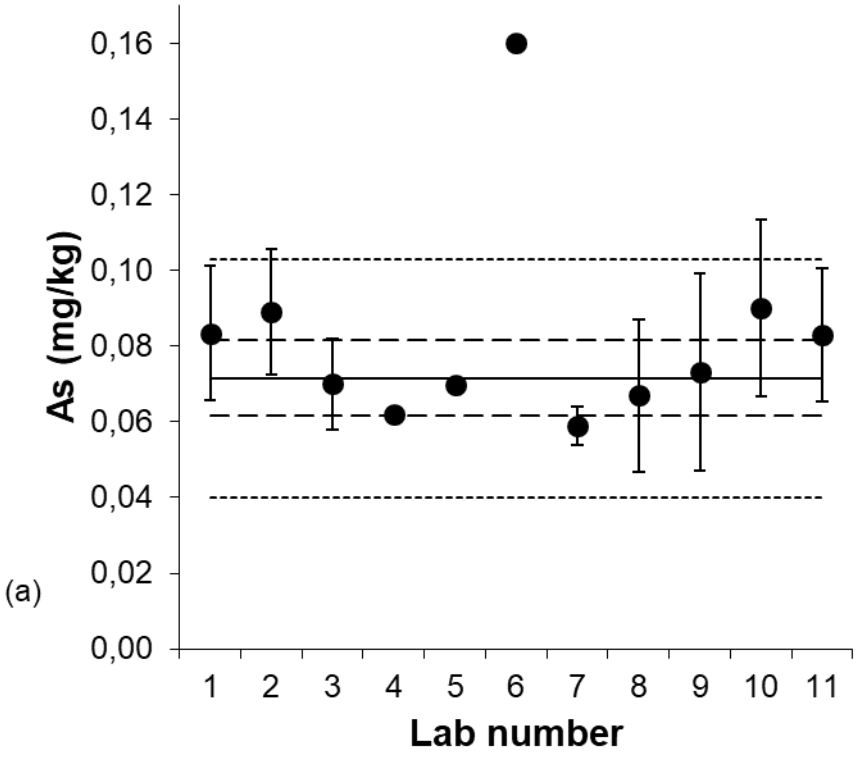
ARSENIC (As)

x_a = **0.072** ± 0.009 mg/kg (k = 2)

Eleven laboratories submitted results for total As concentrations. One value was identified as outlier (>50% higher than the median value), excluding this value resulted in an symmetric Kernel distribution. The median of remaining ten values was used as assigned value. Using this consensus value, ten laboratories obtained satisfactory z-scores for As against the standard deviation accepted for the proficiency test (Table 2, Figure 2). One laboratory (L06) obtained unsatisfactory z-score. Seven laboratories obtained satisfactory ζ-scores against their stated measurement uncertainty. Two laboratories (L04 and L07) obtained questionable ζ-scores. For L05 and L06, ζ-scores were not calculated as no measurement uncertainty was reported.

Table 2: Values reported for As (mg/kg) by the participants and scores calculated by the organizer

Lab code	Result 1 (mg kg^{-1})	Result 2 (mg kg^{-1})	Result 3 (mg kg^{-1})	Mean (mg kg^{-1})	Extended uncertainty (k = 2) (u_{lab}; mg kg^{-1})	z-scores	ζ-scores
1	0,079	0,088	0,083	0,083	0,018	0,8	1,2
2	0,089	/	/	0,089	0,017	1,1	1,8
3	0,070	/	/	0,070	0,012	-0,1	-0,2
4	0,059	0,057	0,069	0,062	0,001	-0,6	-2,1
5	0,071	0,068	/	0,070	/	-0,1	/
6	0,160	/	/	0,160	/	5,6	/
7	0,061	0,060	0,056	0,059	0,005	-0,8	-2,4
8	0,067	0,067	0,067	0,067	0,020	-0,3	-0,4
9	0,073	0,071	0,073	0,073	0,026	0,1	0,1
10	0,087	0,092	/	0,090	0,023	1,2	1,5
11	0,078	0,087	0,084	0,083	0,018	0,7	1,1



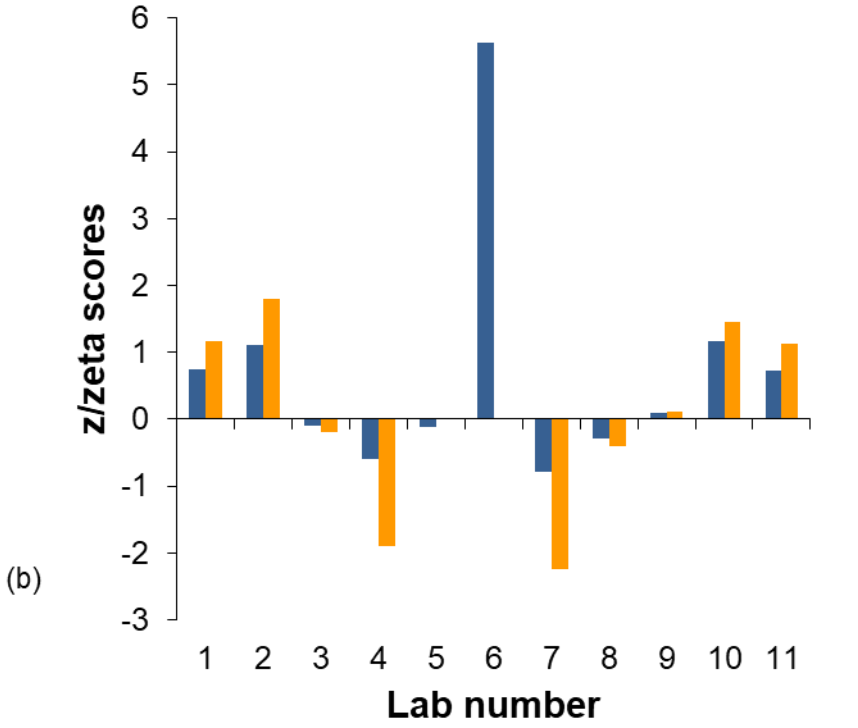


Figure 2: (a) Results with expanded uncertainty for As, as reported by the participants (dashed lines: x_a ± 2 $u(x_a)$, dotted lines: x_a ± 2 σ_p) and (b) z- (blue bars) and ζ-scores (orange bars)

INORGANIC ARSENIC (As_i)

Five laboratories submitted results for As_i concentrations. Due to the low concentration, no conclusions are drawn for this analyte and only LOQ values were reported (Table 3).

Table 3 : LOQ reported for Asi (mg/kg) by the participants.

Lab code	Result 1 (mg kg^{-1})	Result 2 (mg kg^{-1})	Result 3 (mg kg^{-1})	Mean (mg kg^{-1})	Extended uncertainty (k = 2) (u_{lab}; mg kg^{-1})
1	<0.004	<0.004	<0.004	**<0.004**	/
3	<0.027	/	/	**<0.027**	/
4	< 0.020	< 0.020	< 0.020	**< 0.020**	/
9	< 0.020	< 0.020	/	**< 0.020**	/
11	<0.004	<0.004	<0.004	**<0.004**	/

CADMIUM (Cd)

x_a = **0.80** ± 0.06 mg/kg (k = 2)

Eleven laboratories submitted results for Cd concentrations. The median of all results was used as assigned value. All eleven laboratories obtained satisfactory z-scores for Cd against the standard deviation accepted for the proficiency test (Table 4, Figure 3). Nine laboratories also obtained satisfactory ζ-scores against their stated measurement uncertainty. For L05 and L06, ζ-scores were not calculated as no measurement uncertainty was reported.

Table 4 : values reported for Cd (mg/kg) by the participants and scores calculated by the organizer

Lab code	Result 1 (mg kg^{-1})	Result 2 (mg kg^{-1})	Result 3 (mg kg^{-1})	Mean (mg kg^{-1})	Extended uncertainty (k = 2) (u_{lab}; mg kg^{-1})	z-scores	ζ-scores
1	0,85	0,83	0,76	0,81	0,16	0,1	0,2
2	0,70	/	/	0,70	0,13	-0,8	-1,5
3	0,75	/	/	0,75	0,11	-0,4	-0,8
4	0,78	0,84	0,88	0,83	0,16	0,2	0,4
5	0,72	0,72	0,74	0,73	/	-0,6	/
6	0,80	/	/	0,80	/	0,0	/
7	0,61	0,73	0,58	0,64	0,16	-1,2	-1,9
8	0,69	0,69	0,69	0,69	0,14	-0,8	-1,4
9	0,85	0,81	0,80	0,82	0,20	0,2	0,2
10	0,88	0,77	/	0,83	0,11	0,2	0,4
11	0,85	0,87	0,82	0,84	0,17	0,3	0,5

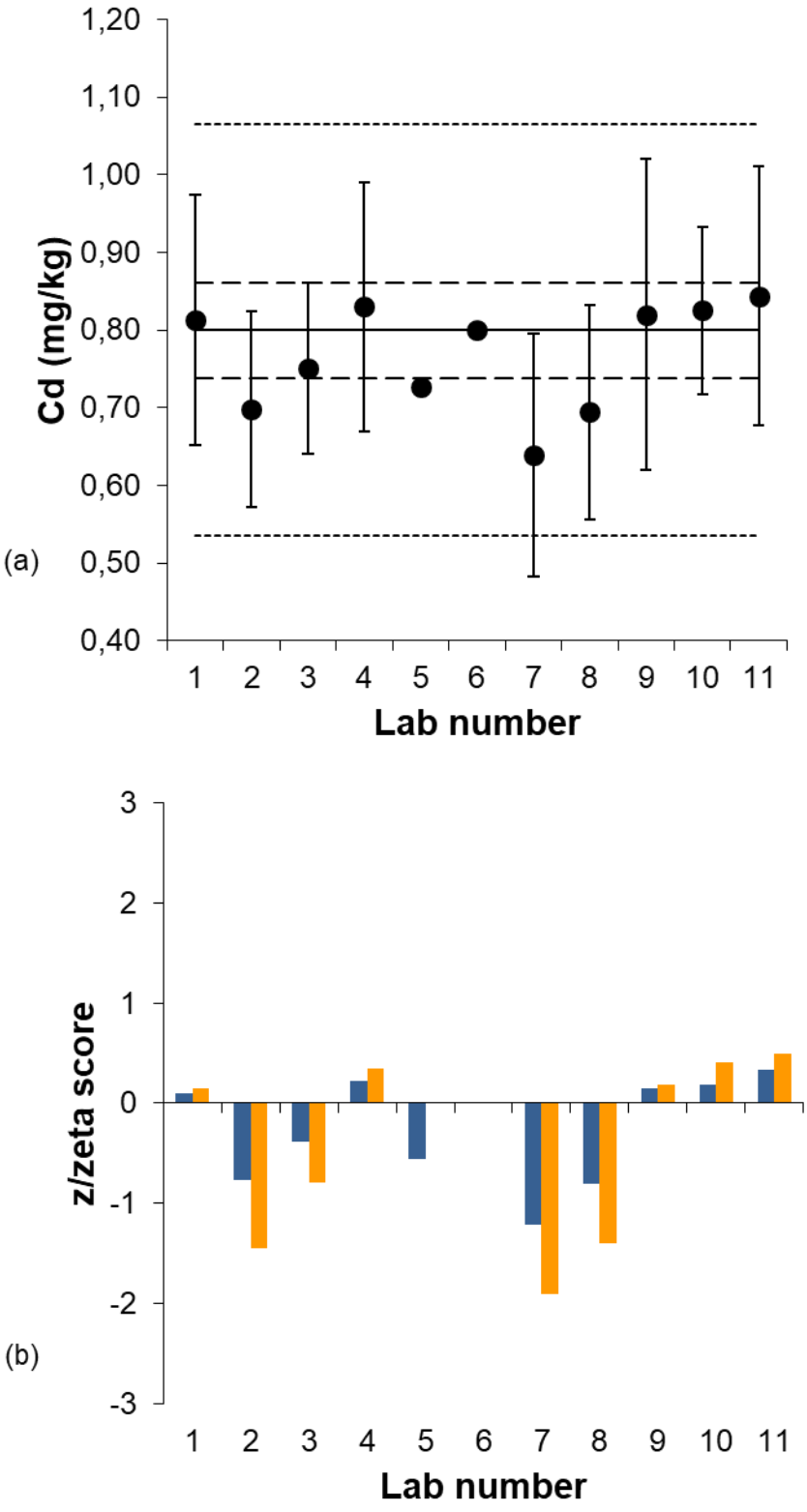


Figure 3: (a) Results with expanded uncertainty for Cd, as reported by the participants (dashed lines: $x_a \pm 2\ u(x_a)$, dotted lines: $x_a \pm 2\ \sigma_p$) and (b) z- (blue bars) and ζ-scores (orange bars)

LEAD (Pb)

x_a = **0.39** ± 0.04 mg/kg (k = 2)

Eleven laboratories submitted results for total Pb concentrations. The median of all results was used as assigned value. All eleven laboratories obtained satisfactory z-scores for Pb against the standard deviation accepted for the proficiency test (Table 5, Figure 4). Eight laboratories did obtain satisfactory ζ-scores against their stated measurement uncertainty. One laboratory (L10) obtained questionable ζ-score. For L05 and L06, ζ-scores were not calculated as no measurement uncertainty was reported.

Table 5 : values reported for Pb (mg/kg) in by the participants and scores calculated by the organizer

Lab code	Result 1 (mg kg^{-1})	Result 2 (mg kg-1)	Result 3 (mg kg-1)	Mean (mg kg-1)	Extended uncertainty (k = 2) (U_{lab}; mg kg-1)	z-scores	ζ-scores
1	0,42	0,42	0,37	0,40	0,09	0,2	0,3
2	0,38	/	/	0,38	0,08	-0,1	-0,1
3	0,37	/	/	0,37	0,07	-0,3	-0,4
4	0,37	0,38	0,37	0,37	0,13	-0,3	-0,3
5	0,39	0,39	0,39	0,39	/	0,0	/
6	0,48	/	/	0,48	/	1,3	/
7	0,32	0,36	0,29	0,32	0,07	-0,9	-1,6
8	0,36	0,36	0,36	0,36	0,09	-0,3	-0,5
9	0,46	0,44	0,42	0,44	0,16	0,7	0,6
10	0,43	0,53	/	0,50	0,08	1,5	2,4
11	0,40	0,43	0,40	0,41	0,09	0,3	0,4

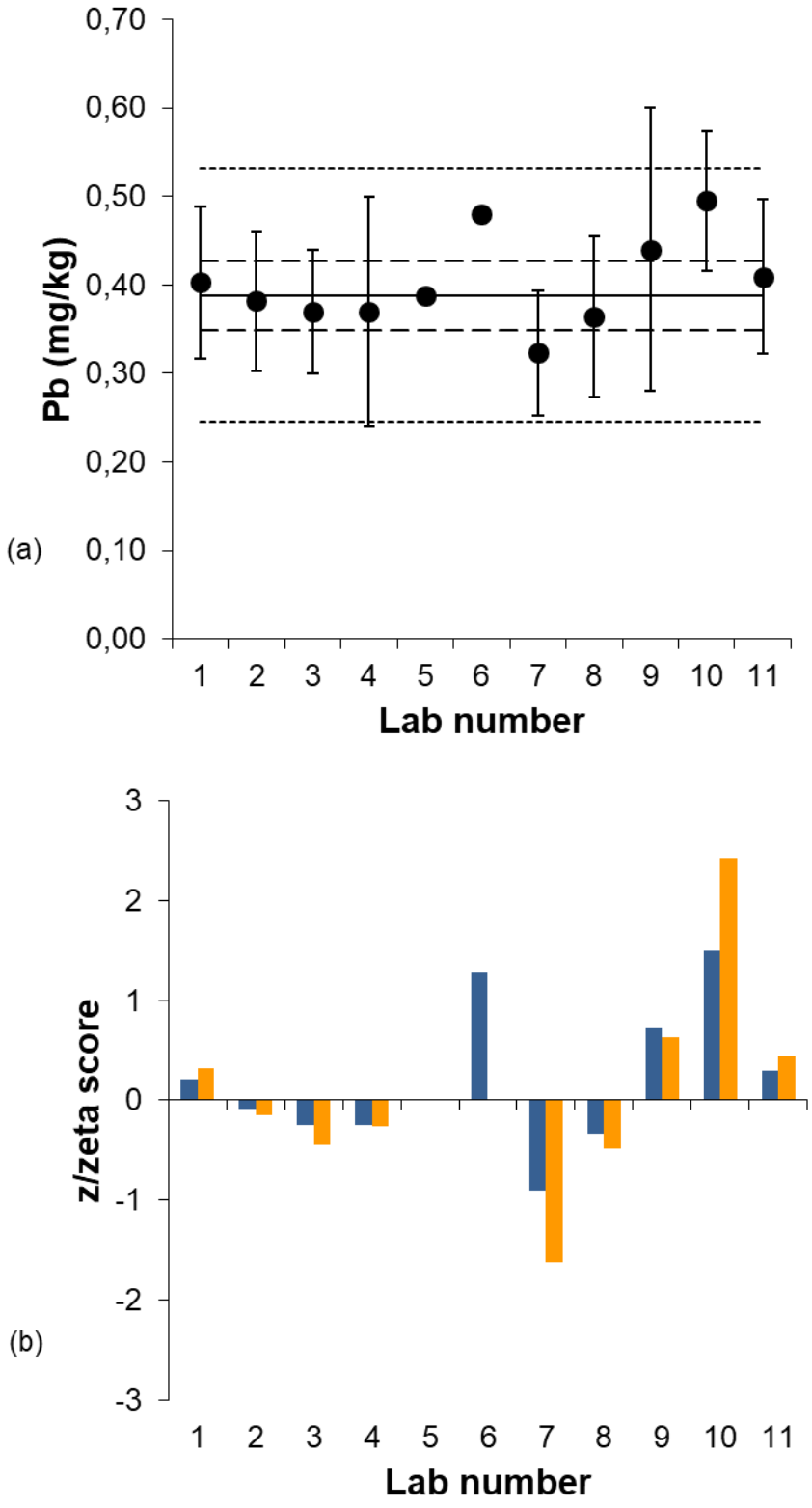


Figure 4 : (a) Results with expanded uncertainty for Pb, as reported by the participants (dashed lines: $x_a \pm 2\ u(x_a)$, dotted lines: $x_a \pm 2\ \sigma_p$) and (b) z- (blue bars) and ζ-scores (orange bars)

MERCURY (Hg)

x_a = **0.32** ± 0.04 mg/kg (k = 2)

Eleven laboratories submitted results for total Hg concentrations. The median of all results was used as assigned value. All eleven laboratories obtained satisfactory z-scores for Hg against the standard deviation accepted for the proficiency test (Table 6, Figure 5). Six laboratories did obtain satisfactory ζ-scores against their stated measurement uncertainty. Three laboratories (L04, L07, L11) obtained questionable ζ-scores. For L05 and L06, ζ-scores were not calculated as no measurement uncertainty was reported.

Table 6 : values reported for Hg (mg/kg) in by the participants and scores calculated by the organizer

Lab code	Result 1 (mg kg^{-1})	Result 2 (mg kg-1)	Result 3 (mg kg-1)	Mean (mg kg-1)	Extended uncertainty (k = 2) (U_{lab}; mg kg-1)	z-scores	ζ-scores
1	0,27	0,28	0,25	0,27	0,06	-0,9	-1,5
2	0,32	/	/	0,32	0,06	0,0	0,0
3	0,32	/	/	0,32	0,05	0,0	0,0
4	0,20	0,23	/	0,22	0,07	-1,7	-2,6
5	0,37	0,36	0,36	0,37	0,00	0,8	/
6	0,36	/	/	0,36	0,00	0,7	/
7	0,24	0,27	0,25	0,25	0,02	-1,1	-2,8
8	0,28	0,28	0,28	0,28	0,07	-0,6	-0,9
9	0,34	0,33	0,34	0,33	0,10	0,2	0,2
10	0,35	0,33	/	0,34	0,06	0,3	0,5
11	0,26	0,26	0,21	0,25	0,06	-1,2	-2,2

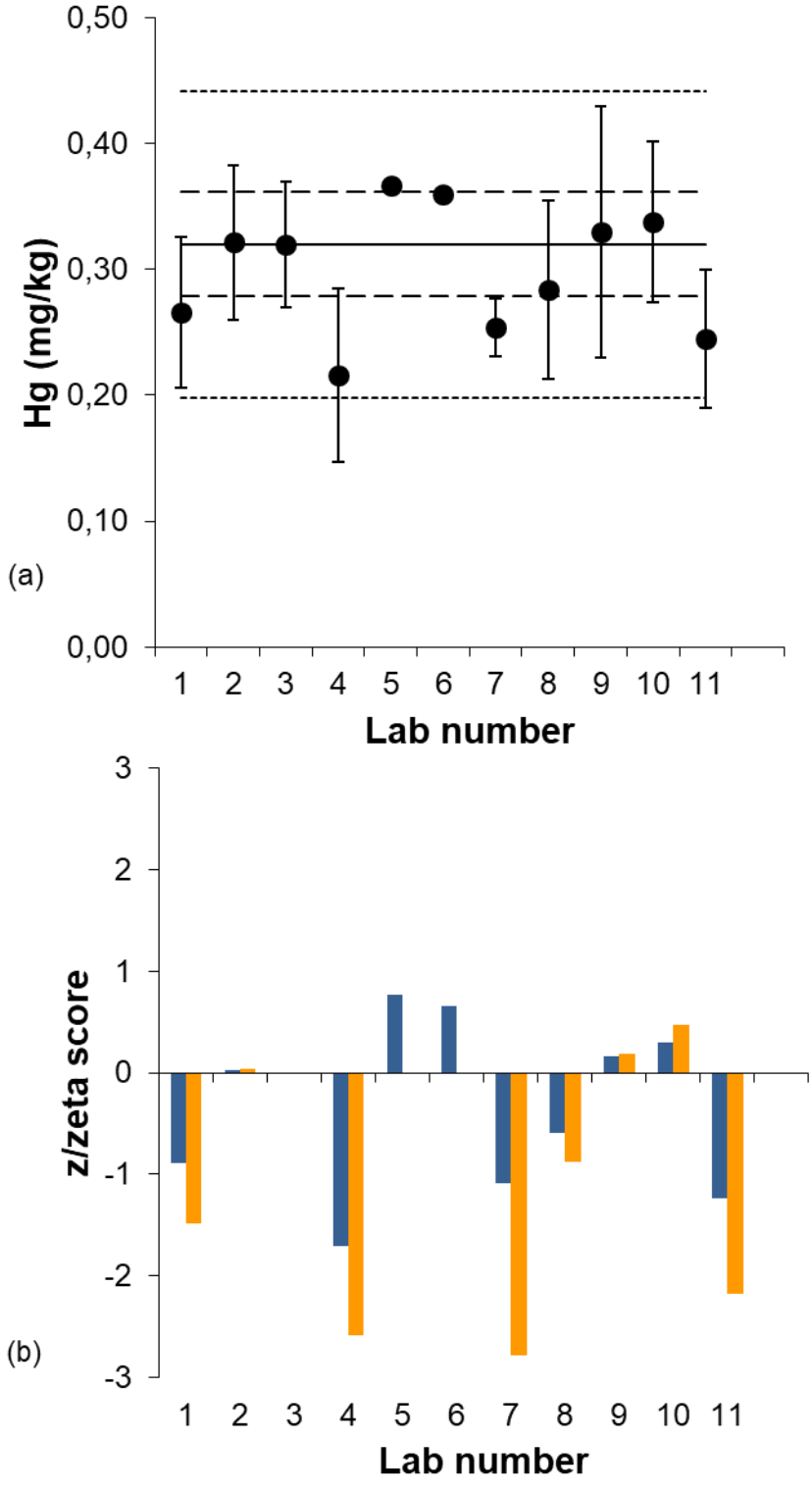


Figure 5 : (a) Results with expanded uncertainty for Hg, as reported by the participants (dashed lines: xa ± 2 u(xa), dotted lines: xa ± 2 σp) and (b) z- (blue bars) and ζ-scores (orange bars)

DISCUSSION AND CONCLUSION

The only used technique for the analysis of As, Cd, Pb was ICP-MS (Inductively Coupled Plasma-Mass Spectrometry). For Hg different techniques are used: CV-AAS (Cold Vapor-Atomic Absoprtion Spectroscopy), ICP-MS, AMA/DMA (Advanced/direct Mercury Analyser) and FIMS (Flow Injection Mercury System).

The laboratories were asked to state if the sample is compliant according to the current legislation. In Commission Regulation (EC) 333/2007 [8] it is described when a sample is accepted:

> *"The lot or sublot is accepted if the analytical result of the laboratory sample does **not exceed** the respective maximum level as laid down in Regulation (EC) No 1881/2006 **taking into account the expanded measurement uncertainty** and correction of the result for recovery if an extraction step has been applied in the analytical method used. The lot or sublot is rejected if the analytical result of the laboratory sample **exceeds beyond reasonable doubt** the respective maximum level as laid down in Regulation (EC) No 1881/2006 **taking into account the expanded measurement uncertainty** and correction of the result for recovery if an extraction step has been applied in the analytical method used."*

For the current matrix, maximum levels for Cadmium (ML of 0.5 mg/kg) and Mercury (ML of 0.1 mg/kg) were exceeded. Nine laboratories stated the sample correctly as non compliant. One laboratory stated the sample as compliant and one laboratory did not respond to this compliance matter.

Inorganic As concentration was too low to draw conclusions about the laboratories performance to analyse this matrix. Determination of As was interfered in one laboratory, all others performed good. In addition, the performance to determine Pb, Cd and Hg was excellent. This shows that laboratories use appropriate instrumentation and are capable to analyse this matrix for control purposes.

Estimation of measurement uncertainty of the results of elements give general satisfactory ζ-scores, though some measurement uncertainties can be optimized.

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ANNEXES

ANNEX 1: INVITATION LETTER TO LABORATORIES

Concern: PT-2022-NRL-TE-FASFC

Dear colleague,

It is our pleasure to invite you to participate in the proficiency test (PT) for the detection of trace elements in food, organized by the National Reference Laboratory (NRL) for trace elements in food and feed at Sciensano. The goal of the PT is to determine the performance of individual laboratories for specific tests. The PT is organised according to the ISO/IEC 17043 norm: 2010 Conformity assessment – General requirements for proficiency testing.

The following PT will be organized by the NRL for trace elements in food and feed in 2022 for the laboratories involved in the official control program of the Federal Agency for the Safety of the Food Chain (FASFC) and other interested laboratories:
PT-2022-NRL-TE-FASFC "Determination of As, Asi, Cd, Pb and Hg in unrefined salts"

• Closing date for the inscription:	29th of April 2022 (week 17)
• Shipment of the samples:	23th of May 2022 (week 21)
• Submission of the test results:	24th of June 2022 (week 25)
• Draft report:	29th of July 2022 (week 30)
• Final report:	30th of September 2022 (week 39)

If your laboratory is **approved by the FASFC** for trace elements in foodstuffs, participation to the PT-2022-NRL-TE-FASFC "Determination of As, Asi, Cd, Pb and Hg in unrefined salts" is **mandatory** for all accredited elements and the costs for this PT (€ 245.66) will be billed directly by the Federal Agency for the Safety of the Food Chain (FASFC). The individual results of the laboratories approved by the FASFC will be disclosed to the FASFC.

If your laboratory is not approved by the FASFC for trace elements in foodstuffs, participation to the PT-2022-NRL-TE-FASFC is voluntary and the costs for the PT, € 245.66 + shipment costs, will be billed by Sciensano. The results will not be disclosed to the FASFC.

You can receive more information about our PT programme by contacting karlien.cheyns@sciensano.be & gregoire.rondelet@sciensano.be

We hope you will find this a useful tool to support your laboratory's Quality Assurance system and look forward to receiving your **registration before the 29th of April 2022**. If you are not the correct contact person for this message or if you know other colleagues that might be interested, please feel free to forward this invitation to your own colleagues or colleagues from other institutes.

Kind regards,

Dr Karlien Cheyns and Dr Grégoire Rondelet

Belgian National Reference Laboratory for Trace Elements in Food and Feed
Service Trace elements and Nanomaterials
Sciensano

Sciensano · Rue Juliette Wytsmanstraat 14 · 1050 Brussels · Belgium
T + 32 2 642 51 11 · F + 32 2 642 50 01 · info@sciensano.be · www.sciensano.be

healthy all life long

ANNEX 2: RESULTS OF THE HOMOGENEITY STUDIES

	As	Cd	Pb	Hg
		Cochran test for variance outliers		
Cochran test statistic	0.243	0.282	0.268	0.305
Critical (95%)	0.602	0.602	0.602	0.602
Cochran < critical	use complete dataset	use complete dataset	use complete dataset	use complete dataset
		Test for sufficient homogeneity		
S_{an}^2	3.06	1175.19	253.56	268.71
S_{sam}^2	1.03	254.75	46.9	186.41
σ_{all}^2	1.14	210.2	71.82	268.73
F1	1.88	1.88	1.88	1.88
F2	1.01	1.01	1.01	1.01
Critical	5.23	1582.2	391.1	776.61
S_{sam}^2 < critical?	**accept**	**accept**	**accept**	**accept**

ANNEX 3: LETTER ACCOMPANYING THE SAMPLE

Concern: Shipment of sample PT-2022-NRL-TE-FASFC

Dear colleague,

Following your subscription for the proficiency test (PT-2022-NRL-TE-FASFC) for the detection of trace elements in food, we ship you the PT sample. You can find your unique lab code **on the sample**.

Enclosed you can find the instructions to the participants with a reporting form. In addition, a receipt form is added, please retour this by e-mail (gregoire.rondelet@sciensano.be). The time schedule of the PT is given below:
PT-2022-NRL-TE-FASFC "Determination of As, Asi, Cd, Pb and Hg in unrefined salts"

• Closing date for the inscription:	29th of April 2022 (week 17)
• Shipment of the samples:	23th of May 2022 (week 21)
• Submission of the test results:	24th of June 2022 (week 25)
• Draft report:	29th of July 2022 (week 30)
• Final report:	30th of September 2022 (week 39)

We expect the results of the analysis the latest by the end of week 25 (the **24th of June**).

We would like to remind you that if your laboratory is **approved by the FASFC** for trace elements in foodstuffs, participation to the PT-2022-NRL-TE-FASFC is **mandatory** for all accredited elements and the costs for this PT (€ 245.66) will be billed directly by the Federal Agency for the Safety of the Food Chain (FASFC). The individual results of the laboratories approved by the FASFC will be disclosed to the FASFC.

For any information about our PT programme you can contact gregoire.rondelet@sciensano.be

Kind regards,

Dr Grégoire Rondelet and Dr Karlien Cheyns

Belgian National Reference Laboratory for Trace Elements in Food and Feed
Service Trace elements and Nanomaterials
Sciensano

Sciensano · Rue Juliette Wytsmanstraat 14 · 1050 Brussels · Belgium
T + 32 2 642 51 11 · F + 32 2 642 50 01 · info@sciensano.be · www.sciensano.be
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ANNEX 4: INSTRUCTIONS TO PARTICIPANTS

FORM/25/06/DOC03/V04

Subject: **PT-2022-NRL-TE-FASFC: INSTRUCTIONS TO THE PARTICIPANTS**

Type of Proficiency test: **PT-2022-NRL-TE-FASFC "Determination of As, Asi, Cd, Pb and Hg in unrefined salts"**

Analytes :

1	As
2	Asi
3	Cd
4	Pb
5	Hg

Matrix : *Unrefined salts*

Number of materials sent : 1 package

Test material : *One container (~15 grams) : the sample should be treated as a sample for routine analysis including your laboratory's homogenization procedure*

Storage: *Store the test material in dark and dry conditions at ambient temperature*

Receipt form:

Please check if the received package is in agreement with what's specified on your registration and receipt form
Complete the receipt form, sign it and send it to gregoire.rondelet@sciensano.be

Data to be sent and to whom:

See 'results reporting form', to be transmitted to Grégoire Rondelet, preferably by e-mail: gregoire.rondelet@sciensano.be (an electronic version of the reporting form will be sent by e-mail). Address: Sciensano, Leuvensesteenweg 17, 3080 Tervuren

Deadline for submitting the results : 24/06/2022

Specific instructions:

- *Follow as close as possible the analysis method you use in routine sample analysis*
- *Report the extended uncertainty*

ANNEX 5: MATERIALS RECEIPT FORM

FORM 25/06/DOC04/V05:

PT-2022-NRL-TE-FASFC "Determination of As, Asi, Cd, Pb and Hg in unrefined salts"

PROFICIENCY TESTING MATERIAL RECEIPT FORM

LIST OF CONTENTS

Material : 1 pack (with one container (~15 grams))

Documents : Shipment letter
Receipt form
Instructions to the participants

NAME ORGANISATION (LAB) :

CONTACT PERSON

TEL :

E-MAIL : LAB NUMBER (see sample) :

DATE OF THE RECEIPT:

STATE OF MATERIALS RECEIVED

O GOOD

O OPEN

O NOT GOOD (specify) :

REMARKS :

DATE NAME & SIGNATURE

Please complete and sign this form and send it back to: gregoire.rondelet@sciensano.be

ANNEX 6: REPORTING FORM AND QUESTIONNAIRE

sciensano

NRL NATIONAL REFERENCE LABORATORIES

PT-2022-NRL-TE-FASFC
"Determination of As, Asi, Cd, Pb and Hg in unrefined salts"

RESULTS REPORTING FORM

Lab code: L .....

1. Does your laboratory carry out this type of analysis on a routine basis?
   As regards to:

   ☐ The matrix
   ☐ As
   ☐ Asi
   ☐ Cd
   ☐ Pb
   ☐ Hg

2. Which matrices/elements would be interesting for your laboratory for future PT's?

MATRICES:
- ☐ Terrestrial vegetable origin
- ☐ Aquatic vegetable origin
- ☐ Terrestrial animal origin
- ☐ Aquatic animal origin
- ☐ Drinks
- ☐ Processed food
- ☐ Feed
- ☐ Other: .....

ELEMENTS:
- ☐ As
- ☐ Asi
- ☐ Cd
- ☐ Pb
- ☐ Hg
- ☐ Cu
- ☐ Zn
- ☐ Ni
- ☐ Cr
- ☐ Other: .....

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Lab code: L .....

Element	Technique used	Units	Replicate 1	Replicate 2	Replicate 3	Mean value	Extended uncertainty (k=2)
As		mg/kg					
Asi		mg/kg					
Cd		mg/kg					
Pb		mg/kg					
Hg		mg/kg					

Is this sample compliant regarding current legislation? Y / N .....