



CODA –CERVA

**Belgian National Reference Laboratory
for Trace Elements in Food and Feed**

**Final report
on the 2010 Interlaboratory Comparison
organised by the National Reference Laboratory
for Trace Elements in Food and Feed**

Trace elements in animal feed of plant origin

December 2010

**J.-C. Pizzolon and Dr. N. Waegeneers
CODA-CERVA, Tervuren, Belgium**

Table of Contents

1 Summary	2
2 Introduction.....	2
3 Test materials and instructions to participants.....	3
4 Assigned values	3
5 Scores and evaluation criteria	4
6 Results	6
6.1 Cadmium.....	6
6.2 Copper	8
6.3 Mercury	10
6.4 Zinc	11
6.5 Arsenic (optional)	13
6.6 Lead (optional)	14
6.7 Discussion.....	15
7 Conclusion.....	16
8 References	16
Annexes	17

1 Summary

From the 1st of January 2008, the laboratory for Trace Elements in the Veterinary and Agrochemical Research Centre, Tervuren, operates as a National Reference Laboratory for Trace Elements in Feed and Food (NRL-TE). One of its core tasks is to organise interlaboratory comparisons (ILCs) among laboratories appointed by the Federal Agency for the Safety of the Food Chain. This report presents the results of the interlaboratory comparison organised by the NRL-TE which focused on the determination of trace elements in a feed sample of plant origin.

The results from the ILC were treated in CODA-CERVA, Tervuren.

Seven laboratories associated with the Federal Agency for the Safety of the Food Chain registered for and participated in the exercise.

The test material used in this exercise was plant material, prepared from 10 kg of fodder wheat coming from Oost-Vlaanderen. The test sample was dried at 75°C, milled and then mixed during 48 hours prior to be bottled. Each participant received one bottle filled with approximately 40 g of wheat.

Participants were invited to report the mean value and measurement uncertainty on their results for cadmium (Cd), copper (Cu), mercury (Hg), zinc (Zn) and optionally for arsenic (As) and lead (Pb). The assigned values (x_a) and their uncertainty ($u(x_a)$) were determined as the consensus of participant's results. Given the low number of participants and the fact that the assigned values are determined as the consensus of participant's results, the laboratory results were rated with informal z- and zeta-scores (ISO 13528). Standard deviations for proficiency assessment were calculated using the modified Horwitz equation (Thompson, 2000).

All calculated z- and zeta-scores were satisfactory for Cd, Cu and Zn. The Hg concentration in the feed sample was below the limit of quantification for the majority of the laboratories and no assigned value or scores could be calculated. Due to their low concentration in the feed sample, the analysis of As and Pb were optional. The reported results for these elements were scattered and not consistent with each other.

2 Introduction

Trace elements occur in varying amounts as natural elements in soils, plants and animals, and consequentially in food and feed. Concerning food and feed of plant origin, the characteristics of the soil on which the plants are grown have a considerable influence on the content of trace elements in the plant. The concentration of trace elements in plants is often correlated to the corresponding concentrations in the soil on which they were grown, but also soil texture, soil pH and soil organic matter content influence the trace element content in the plants. To ensure public and animal health, maximum levels for trace elements in feed have been laid down in Directive 2002/32/EC.

The scope of this ILC was to test the competence of the participating laboratories to determine the total mass fraction of Cd, Cu, Hg, Zn and optionally As and Pb in feed of plant origin.

3 Test materials and instructions to participants

Seven laboratories associated with the Federal Agency for the Safety of the Food Chain registered for and participated in the exercise.

This year the test sample was fodder wheat. The sample was collected in Oost-Vlaanderen. The wheat was dried at 75°C in our laboratory, milled and then mixed during 48 hours. Approximately 40 g of wheat was placed in a plastic bottle prior to dispatch to each participant. Each participant received one bottle of test material, an accompanying letter with instructions on sample handling and reporting, and a form that had to be sent after receipt of the sample to confirm its arrival.

A special 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 5.

Reporting deadline was 30 September 2010.

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

The homogeneity of the test material was tested following the recommended procedure according to IUPAC (2006). All the trace elements appeared to be homogeneously distributed in the wheat samples (annex 2).

4 Assigned values

The assigned values for the different trace elements in the wheat sample are determined as the consensus of participant's results (IUPAC, 2006). 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 only 7 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. Therefore the z-scores will be presented as informal z-scores.

The robust statistic approach is a convenient modern method of handling results when they are expected to follow a near-normal distribution and it is suspected that they include a small proportion of outliers. There are many different robust estimators of mean and standard deviation (AMC, 2001). The median and MAD (median absolute difference) were chosen here as robust estimators.

The modified Horwitz equation was used to establish the standard deviation for proficiency testing (σ_p) (Thompson, 2000). 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.

The Kernel density estimate gives a good estimate of the population density function without making any assumptions that it is a normal distribution. However, the Kernel plot could not be presented because of the low number of participants (8 data points is the minimum requirement for the software provided by the Analytical Method Committee of the Royal Society of Chemistry).

The scheme that was followed to estimate the consensus and its uncertainty is outlined below:

- a) A visual presentation of the results was examined. It was checked whether the distribution was apparently unimodal and roughly symmetric, possible outliers aside.
- b) The robust mean $\hat{\mu}_{rob}$ and standard deviation $\hat{\sigma}_{rob}$ of the n results was calculated as $\hat{\mu}_{rob} = \text{median}$ and $\hat{\sigma}_{rob} = 1.4826 * \text{MAD}$. Since $\hat{\sigma}_{rob}$ was less than $1.2\sigma_p$, $\hat{\mu}_{rob}$ was used as the assigned value x_a and $\hat{\sigma}_{rob}/\sqrt{n}$ as its standard uncertainty $u(x_a)$.

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.

	Cd µg/kg	Cu mg/kg	Hg µg/kg	Zn mg/kg	As µg/kg	Pb µg/kg
n	7	6	1	5	3	2
Mean	75.2	3.33	-	32.6	231	75.6
SD	12.0	0.33	-	2.4	313	77.2
Robust mean (median)	74.1	3.27	-	33.2	_(¹)	_(¹)
Robust SD	16.5	0.38	-	3.4	-	-
Assigned value x_a	74.1	3.27	-	33.2	-	-
Standard uncertainty of the assigned value $u(x_a)$	6.2	0.15	-	1.5	-	-
σ_p	16.3	0.44	-	3.1	-	-

Assigned value (x_a): median of the reported results

σ_p : standard deviation for proficiency assessment

(¹) Inconsistent data

5 Scores and evaluation criteria

Individual laboratory performances are expressed in terms of z-scores and zeta-scores in accordance with ISO 135283 and the International Harmonised Protocol (IUPAC, 2006).

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

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

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

$u(x_{\text{lab}})$ is the reported standard uncertainty in 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 zeta-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 zeta-score is similar to the interpretation of the z-score.

$ \text{zeta} \leq 2$	satisfactory result
$2 < \text{zeta} \leq 3$	questionable result
$ \text{zeta} > 3$	unsatisfactory result

Given the low number of participants ($n = 7$) and the fact that the assigned values are determined as the consensus of participant's results, only informal z- and zeta-scores will be calculated.

Per trace element, a set of figures is provided. Each set includes (a) the individual mean values with their reported uncertainty, and (b) the z- and zeta-scores. The solid line represents the assigned value, the dashed lines delimit the reference interval ($x_a \pm 2u(x_a)$) and the dotted lines delimit the target interval ($x_a \pm 2\sigma_p$).

6 Results

6.1 Cadmium

All laboratories (n = 7) obtained good z-scores for cadmium against the standard deviation accepted for the proficiency test (Table 2; Fig 1a-b), as well as good zeta-scores against their stated measurement uncertainty.

Table 3: values reported for Cd by the participants and informal scores calculated by the organiser.

Lab code	Result 1 ($\mu\text{g kg}^{-1}$)	Result 2 ($\mu\text{g kg}^{-1}$)	Result 3 ($\mu\text{g kg}^{-1}$)	Mean ($\mu\text{g kg}^{-1}$)	Uncertainty (U_{lab}; $\mu\text{g kg}^{-1}$)	z-scores	zeta-scores
L01	93	93	94	93	20	1.2	1.6
L02	88.455	81.267	87.007	85.58	9.41	0.7	1.5
L03	81.8	73.0	67.5	74.1	19.3	0.0	0.0
L04	62	55	59	59	18	-0.9	-1.4
L05	73	76	68	72	5	-0.1	-0.3
L06	80	80	-	80	0	0.4	1.0
L07	68	59	61	63	8	-0.7	-1.5

Figure 1a: Results and expanded uncertainty for Cd, as reported by the participants

$$x_a = 74.1 \mu\text{g kg}^{-1}$$

$$u(x_a) = 6.2 \mu\text{g kg}^{-1}$$

$$\sigma_p = 16.3 \mu\text{g kg}^{-1}$$

(dashed lines: $x_a \pm 2 u(x_a)$,
dotted lines: $x_a \pm 2 \sigma_p$)

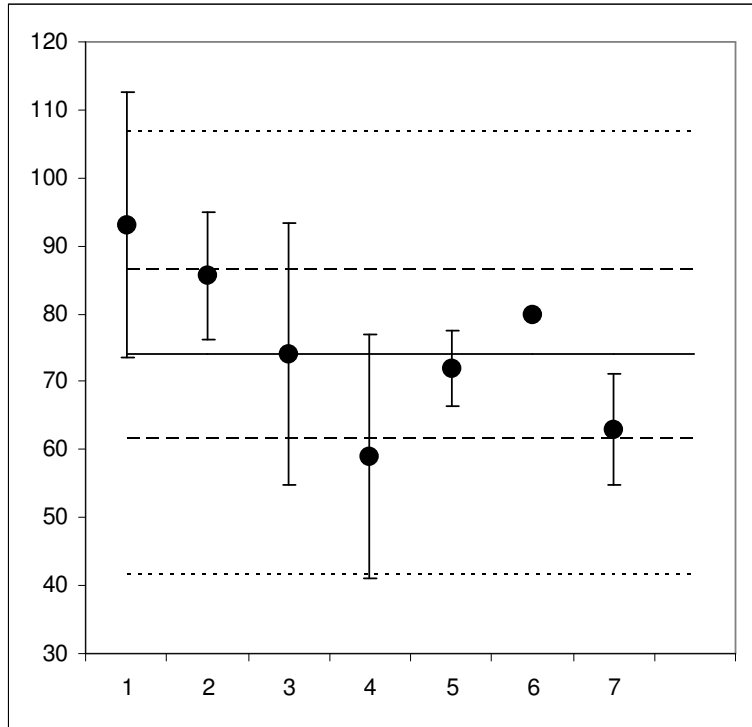
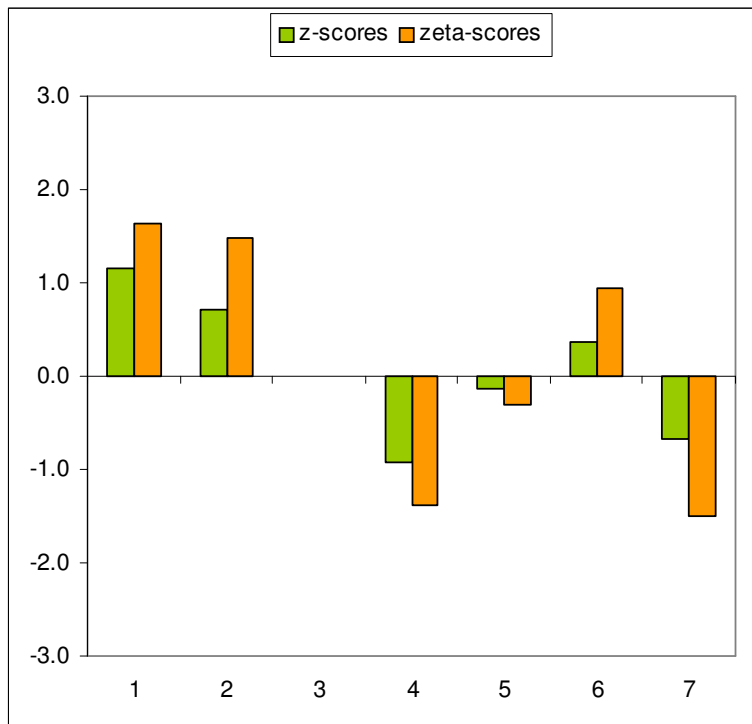


Figure 1b: z- and zeta-scores

$$z = (x_{\text{lab}} - x_a) / \sigma_p$$

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



6.2 Copper

All laboratories that submitted results (n = 6) obtained good z-scores for copper against the standard deviation accepted for the proficiency test, as well as good zeta-scores against their stated measurement uncertainty (Table 3; Fig 2a-b). One laboratory did not provide results.

Table 3: values reported for Cu by the participants and informal scores calculated by the organiser.

Lab code	Result 1 (mg kg ⁻¹)	Result 2 (mg kg ⁻¹)	Result 3 (mg kg ⁻¹)	Mean (mg kg ⁻¹)	Uncertainty (U _{lab} ; mg kg ⁻¹)	z-scores	zeta-scores
L01	3.43	3.37	3.51	3.44	0.3	0.4	0.8
L02	3.764	3.714	3.885	3.79	0.6	1.2	1.6
L03	4.00	3.41	3.45	3.62	0.9	0.8	0.7
L04	3.2	2.8	3.0	3.0	0.9	-0.6	-0.6
L05							
L06	3.1	3.1	3.1	3.1	0	-0.4	-1.1
L07	2.97	3.16	2.97	3.03	1.1	-0.5	-0.4

Figure 2a: Results and expanded uncertainty for Cu, as reported by the participants

$$x_a = 3.27 \text{ mg kg}^{-1}$$

$$u(x_a) = 0.15 \text{ mg kg}^{-1}$$

$$\sigma_p = 0.44 \text{ mg kg}^{-1}$$

(dashed lines: $x_a \pm 2 u(x_a)$,
dotted lines: $x_a \pm 2 \sigma_p$)

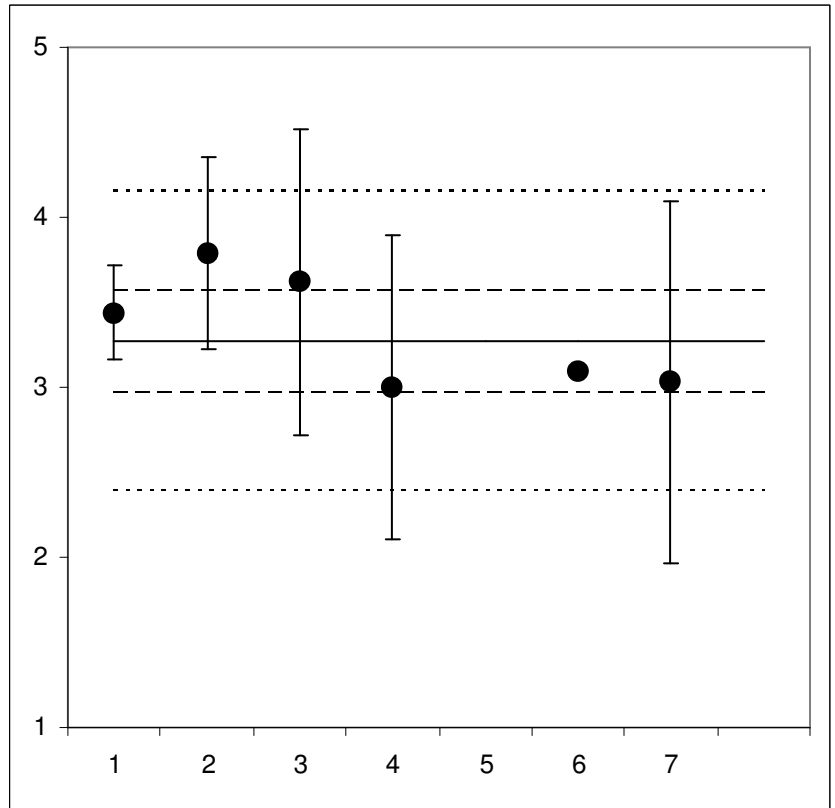
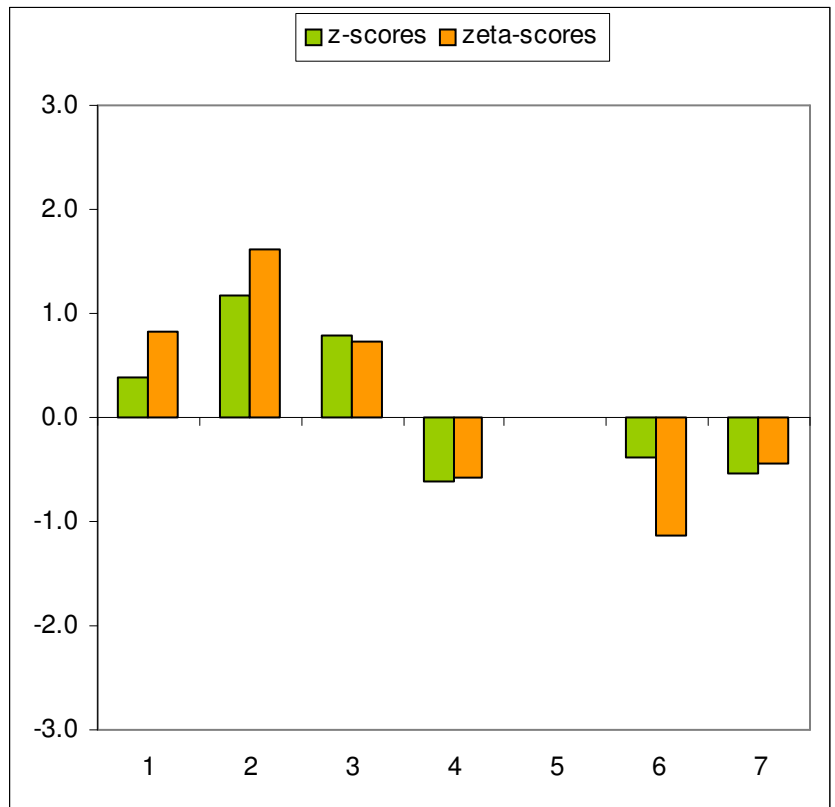


Figure 2b: z- and zeta-scores

$$z = (x_{lab} - x_a) / \sigma_p$$

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



6.3 Mercury

Only one of the participating laboratories submitted results other than “less than” for mercury (Table 4). Hence no assigned value could be calculated.

Table 4: values reported for Hg by the participants.

Lab code	Result 1 ($\mu\text{g kg}^{-1}$)	Result 2 ($\mu\text{g kg}^{-1}$)	Result 3 ($\mu\text{g kg}^{-1}$)	Mean ($\mu\text{g kg}^{-1}$)	Uncertainty (U_{lab}; $\mu\text{g kg}^{-1}$)
L01	<50	<50	<50	<50	
L02	5.427	4.672	5.461	5.19	3.11
L03	<6	<6	<6	-	
L04	<10	<10	<10	<10	
L05	-	-	-	<10	
L06	<10	<10	<10	<10	
L07	<20	<20	<50	<20	

6.4 Zinc

Five of the six laboratories that submitted results obtained good z-scores against the standard deviation accepted for the proficiency test, as well as good zeta-scores against their stated measurement uncertainty. One laboratory could not produce results above its limit of quantification (Table 5; Fig 3a-b). One laboratory did not submit results.

Table 5: values reported for Zn by the participants and informal scores calculated by the organiser.

Lab code	Result 1 (mg kg ⁻¹)	Result 2 (mg kg ⁻¹)	Result 3 (mg kg ⁻¹)	Mean (mg kg ⁻¹)	Uncertainty (U _{lab} ; mg kg ⁻¹)	z-scores	zeta-scores
L01	34.57	32.14	32.88	33.20	9.30	0.0	0.0
L02	35.116	35.646	37.022	35.93	6.47	0.9	0.8
L03	33.26	34.88	31.96	33.40	8.28	0.1	0.0
L04	32.5	28.3	27.8	29.6	8.9	-1.2	-0.8
L05							
L06	<0.50	<0.25	<0.25	<0.33			
L07	31.0	29.7	32.1	30.9	7.4	-0.7	-0.6

Figure 3a: Results and expanded uncertainty for Zn, as reported by the participants

$$x_a = 33.2 \text{ mg kg}^{-1}$$

$$u(x_a) = 1.5 \text{ mg kg}^{-1}$$

$$\sigma_p = 3.1 \text{ mg kg}^{-1}$$

(dashed lines: $x_a \pm 2 u(x_a)$,
dotted lines: $x_a \pm 2 \sigma_p$)

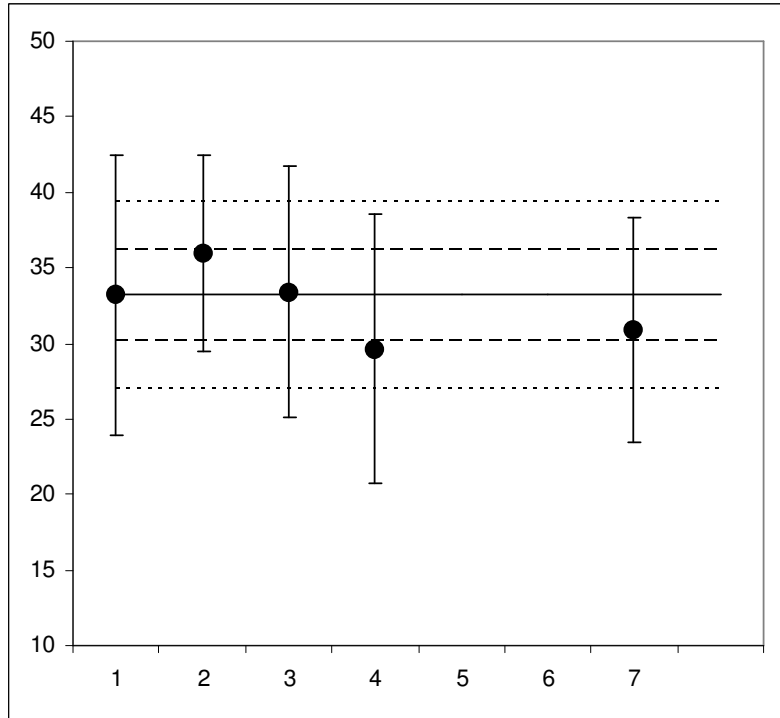
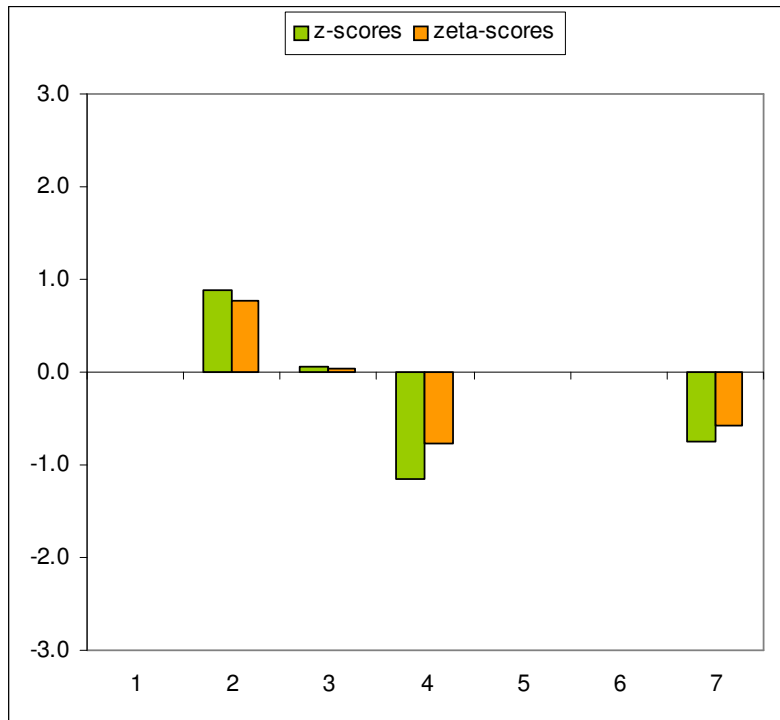


Figure 3b: z- and zeta-scores

$$z = (x_{\text{lab}} - x_a) / \sigma_p$$

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



6.5 Arsenic (optional)

Only three of the participating laboratories could submit results other than “less than” for arsenic (Table 6). The three values are, however, not consistent with each other and hence no assigned value could be calculated.

Table 6: values reported for As by the participants.

Lab code	Result 1 ($\mu\text{g kg}^{-1}$)	Result 2 ($\mu\text{g kg}^{-1}$)	Result 3 ($\mu\text{g kg}^{-1}$)	Mean ($\mu\text{g kg}^{-1}$)	Uncertainty (U_{lab} ; $\mu\text{g kg}^{-1}$)
L01	<50	<50	<50	<50	
L02	596.38	500.68	674.79	590.62	118.12
L03					
L04	85	74	106	88	26
L05	14.8	14.4	14.9	14.7	1.4
L06	<50	<50	-	<50	
L07	<500	<500	<1000	<500	

6.6 Lead (optional)

Only two of the participating laboratories could submit results other than “less than” for lead (Table 4). The two values are, however, not consistent with each other and hence no assigned value could be calculated.

Table 7: values reported for Pb by the participants.

Lab code	Result 1 ($\mu\text{g kg}^{-1}$)	Result 2 ($\mu\text{g kg}^{-1}$)	Result 3 ($\mu\text{g kg}^{-1}$)	Mean ($\mu\text{g kg}^{-1}$)	Uncertainty (U_{lab}; $\mu\text{g kg}^{-1}$)
L01	<50	<50	<50	<50	
L02	121.06	-	139.18	130.12	31.23
L03	<40	<40	<40	-	
L04	26	34	2	21	6.0
L05	-	-	-	<40	
L06	<130	<100	<100	<110	
L07	<200	<200	<500	<200	

6.7 Discussion

Of the seven laboratories that registered for participation, seven submitted results for Cd, six for Cu, seven for Hg, six for Zn, six for As and seven for Pb. From these results, values reported as “less than” were not included in the evaluation. This was the case for six laboratories for Hg, one laboratory for Zn, three laboratories for As and four laboratories for Pb. It should be noted that L06 reported for Zn a “less than” value which was lower than the corresponding $x_a - 2 u(x_a)$ value. This should be considered as an incorrect statement since they should have been able to detect the element.

All but one laboratory reported an uncertainty associated to their results. The laboratory that did not provide uncertainty information did not so because they are not accredited for those elements in this matrix.

Given the low number of participants and the fact that the assigned values are determined as the consensus of participant’s results, only informal z- and zeta-scores have been calculated. All these z- and zeta-scores were satisfactory, 15 out of 18 z-scores were even below 1.

For Hg, only one laboratory reported results other than “less than”. The six reported “less than” values were consistent with that result. Due to their low concentration in the feed sample, the analyses of As and Pb were optional. Three laboratories reported results other than “less than” for As. The reported values were, however, not consistent with each other, neither were they all consistent with the “less than” values. Two laboratories reported results other than “less than” for Pb. Again, the reported values were not consistent with each other. The lowest reported value was consistent with the four “less than” values, the highest reported value was only consistent with one of the “less than” values.

The most commonly used techniques for analyzing the trace element concentrations in the test samples were ICP-MS and ICP-AES or ICP-OES, AAS (flame and graphite furnace). For mercury, AAS (hydride generation and cold vapour) and FIMS (field ionization mass spectrometry) were used. No differences could be found between the different techniques that were used.

Additional information was gathered from the questionnaire that participants were asked to fill in. All the laboratories have a quality system in place (ISO 17025). Two out of seven participating laboratories did not carry out this type of analysis (as regards to parameters, matrix and methods) on a routine basis.

For uncertainty estimation, four participants used the uncertainty estimation calculated during the in-house validation of the method. Only one laboratory used the method prescribed by the Federal Agency for the Safety of the Food Chain (method based on CRM + duplicate samples and proficiency tests). One other laboratory based its uncertainty estimation on the results of proficiency tests.

7 Conclusion

Given the low number of participants and the fact that the assigned values are determined as the consensus of participant's results, only informal z- and zeta-scores have been calculated for Cd, Cu and Zn. All these z- and zeta-scores were satisfactory.

The Hg concentration in the feed sample was below the limit of quantification for the majority of the laboratories and no assigned value or scores could be calculated.

Due to their low concentration in the feed sample, the analysis of As and Pb were optional. The reported results for these elements were scattered and not consistent with each other.

8 References

AMC technical brief. 2001. Robust statistics: a method of coping with outliers.

Directive 2002/32/EC. 2002. Official Journal of the European Communities L140: 10-21.

IUPAC 2006. The International harmonized protocol for the proficiency testing of analytical chemistry laboratories. Pure Appl. Chem 78: 145-196.

Thompson, M. 2000. *Analyst*, 125: 385-386

Annexes

Annex 1: Invitation letter to laboratories.....	18
Annex 2: Results of the homogeneity studies.....	19
Annex 3: Letter accompanying the sample.....	20
Annex 4: Sample receipt confirmation form.....	22
Annex 5: Reporting form and questionnaire.....	23
Annex 6: Participating Laboratories:.....	25

Annex 1: Invitation letter to laboratories



V A R

VETERINARY AND AGROCHEMICAL RESEARCH CENTRE
NRL - VAR

your contact: Pizzolon Jean-Christophe
02/769 22 32
Leuvensesteenweg 17 - 3080 Tervuren

date
31 May 2010

SUBJECT: Intercomparison for Heavy Metals in Feed and Food

Dear colleague,

On behalf of the NRL Trace elements in Feed and Food, I would like to inform you about the ongoing organisation of a Proficiency Test for the determination of total As, Cd, Cu, Hg, Pb and Zn in a **feed sample**.

If your laboratory is accredited for trace elements determination in food and feed, you must provide results for Cd, Cu, Hg and Zn. Due to their low concentration, determination of As and Pb are optional.

In order to help us with a more effective organisation of this intercomparison, I kindly ask you to inform me **by email** if you are interested to participate.

The deadline for registration is **15 June 2010**.

The sample will be sent to participants by the end of June.

The deadline for submission of results is **30 September 2010**.

As I am the project leader for this inter-laboratory comparison please do not hesitate to contact me in case of questions/doubts.

Your participation is free of charge.

Yours sincerely:

Pizzolon Jean-Christophe

O.D. Chemical Safety of the Food Chain
NRL Trace Elements
Veterinary and Agrochemical Research Centre
VAR-CODA-CERVA
Leuvensesteenweg 17
B-3080 Tervuren

Tel: + 32 2 769 22 32

Fax: + 32 2 769 23 05

E-mail: jeanchristophe.pizzolon@var.fgov.be

Annex 2: Results of the homogeneity studies

	Cd	Cu	Hg	Zn	As	Pb
<i>Cochran test for variance outliers</i>						
Cochran test statistic	0.392	0.374	0.284	0.233	0.273	0.658
Critical (95%)	0.602	0.602	0.602	0.602	0.602	0.602
Cochran < critical?	accept	accept	accept	accept	accept	accept ⁽¹⁾
<i>Test for sufficient homogeneity</i>						
S_{an}²	6	7506	0.0063	414100	270	28
S_{sam}²	8	10544	0	1066399	244	6
σ_{all}²	25	20059	0.0006	670956	5	5
F1	1.88	1.88	1.88	1.88	1.88	1.88
F2	1.01	1.01	1.01	1.01	1.01	1.01
Critical	53	45293	0.00758	1679639	282	38
S_{sam}² < critical?	accept	accept	accept	accept	accept	accept

⁽¹⁾ The Cochran's test statistic value was still below the critical value at the 99% level (0.718), therefore there was no exclusion of one of the tested pairs.

Annex 3: Letter accompanying the sample



V A R

VETERINARY AND AGROCHEMICAL RESEARCH CENTRE
NRL - VAR

June 29th 2010

CODA-CERVA

National Reference Laboratory for Trace Elements in Food and Feed

Your Lab code : PT CODA 2010 – L...

Your contact : Jean-Christophe Pizzolon

JeanChristophe.Pizzolon@var.fgov.be

Phone: +32 2 769 22 32 Fax: +32 2 769 23 05

Subject : Proficiency Test for the determination of total Cd, Cu, Hg, Zn, As (optional) and Pb (optional) in a feed sample.

Dear colleague,

Thank you for participating to this Proficiency Test for trace elements in a feed sample. The organisation of this exercise is part of our responsibilities as NRL for trace elements in feed and food.

1° Receiving the sample:

- a) This parcel contains:
 - one plastic container with approximately 40 g of homogenised sample
 - a reporting form
 - a sample receipt confirmation form

- b) Please check whether the sample remains undamaged during the transport and send us as fast as possible the sample receipt confirmation form.

- c) It is advised to store the sample at room temperature, in a dark place until analysis.
- d) Before starting an analysis, re-homogenise the sample by shaking for ± 30 sec.

2° Reporting results:

The procedure you will follow for this exercise should be as close as possible to the method you use in routine sample analysis.

- a) Three independent measurements per parameter are needed.
- b) **Correct** the measurements results for **recovery** and **water content** (see procedure below) and report them in the reporting form.
- c) Report measurement uncertainty.
- d) The deadline for submission of results is **September 30th 2010**. Please use the joined reporting form to report your results and send it back to us by **fax at +32 2 769 23 05**.

3° Water content determination procedure:

- a) Weigh accurately 1.0 g of test material in a glass container, preferably with a lid.
- b) Place it in an oven for 24 ± 1 hour at 110 ± 5 °C.
- c) Place the glass container covered with its lid in a desiccator and allow to cool down (± 30 min) before weighing again.

Note1: perform the water content determination in triplicate.

Note2: do not use for trace elements determination the aliquots of test material that you have used for water content determination.

Your participation is greatly appreciated. If you have any remaining questions, please feel free to contact me.

Best regards,

Jean-Christophe Pizzolon

Annex 4: Sample receipt confirmation form



V A R

**VETERINARY AND AGROCHEMICAL RESEARCH CENTRE
NRL - VAR**

June 29th 2010

CODA-CERVA

National Reference Laboratory for Trace Elements in Food and Feed

Lab code : **PT CODA 2010 – L...**

Sample receipt confirmation form

Please return us as soon as possible this receipt confirmation form by fax at 02 769 23 05

Date of package arrival:

Remarks:

.....
.....
.....
.....

Signature:



V A R

**VETERINARY AND AGROCHEMICAL RESEARCH CENTRE
NRL - VAR**

June 29th 2010

CODA-CERVA

National Reference Laboratory for Trace Elements in Food and Feed

Lab code : **PT CODA 2010 – L...**

Results Reporting Form Trace elements in a feed sample June 2010

1) What is the basis of your measurement uncertainty estimate?

- Uncertainty calculation according to ISO-GUM
- Uncertainty of the method as determined during in-house validation
- FASFC method:
 - Based on CRM
 - Based on CRM + duplicate samples
 - Based on proficiency tests
- Uncertainty based on proficiency tests
- Other (please specify):

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

- Yes
- No

3) Does your laboratory carry out this type of analysis (as regards the parameters, matrix and methods) on a routine basis?

- Yes
- No

4) Does your laboratory have a quality system in place?

- Yes*
- No

*If yes please specify:

- ISO 17025
- ISO 9000 series
- Other (please specify):

Annex 6: Participating Laboratories:

Chemiphar N.V.
Eurofins Belgium
FAVV – FLVVG
Institut Scientifique de la Santé Publique
Laboratorium Ecce
Lareco S.A.
Lovap N.V.