European Commission

JRC TECHNICAL REPORTS

EURL-FA Control Proficiency Test Report

Determination of the mass fraction of the total selenium in compound feed for rabbits

F. Cordeiro, P. Robouch, J. Snell, S. García-Ruiz, G. Van Britsom, H. Emteborg, A. Cizek-Stroh, C. von Holst, U. Vincent

2018

Joint Research Centre

EUR 29408 EN

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JRC Science Hub
https://ec.europa.eu/jrc

JRC 113627

EUR 29408 EN

ISBN 978-92-79-96747-4 (online) ISSN 1831-9424 (online) DOI: 10.2760/562439 (online)

Geel, Belgium: European Commission, 2018

How to cite: F. Cordeiro, P. Robouch, J. Snell, S. García-Ruiz, G. Van Britsom, H. Emteborg, A. Cizek-Stroh, C. von Holst, U. Vincent, "*EURL-FA Control Proficiency Test Report, Determination of the mass fraction of the total selenium in compound feed for rabbits*", JRC Technical Reports, October 2018, EUR 29408 EN, doi 10.2760/562439

European Union Reference Laboratory
Feed Additives

EURL-FA Control
Proficiency test report

Determination of the mass fraction of total selenium in compound feed for rabbits

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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 Feed Additive Control (EURL-FA Control) organised a proficiency test (PT) for the determination of the mass fraction of total selenium in compound feed, to support the Commission Directive 86/403/EEC concerning additives in feeding stuffs. This proficiency test was open to National Reference Laboratories (NRLs) and official feed control laboratories (OCLs).

The material used as test item was a commercially available compound feed for rabbits (containing, among others, selenium as sodium selenite) which, after appropriate processing, was bottled, labelled and dispatched to participants on May 22, 2018. The homogeneity and stability of the test item were evaluated and the assigned values were derived from the results reported by the selected expert laboratories.

Twenty one NRLs and four OCLs from 20 countries - representing several EU Member States, Norway and Switzerland - registered to the exercise and reported results at the end of June 2018.

Laboratory results were rated using z' and ζ (zeta) performance scores in accordance with ISO 13528:2015. The relative standard deviation for proficiency assessment (σ_{pt}) of 15 % of the assigned value was derived from the reproducibility standard deviation reported in the CEN standard EN 16159:2012.

Twenty three (out of 25) laboratories reported satisfactory results (according to the z' score). This confirms the ability of most NRLs in monitoring maximum levels set by the Commission Directive 86/403/EEC concerning additives in feeding stuffs.

List of abbreviations

DG SANTE	Directorate General for Health and Food Safety
EURL-FA Control	European Union Reference Laboratory for Feed Additives Control
ET-AAS	Electro-Thermal – Atomic Absorption Spectrometry
HG-AAS	Hydride Generation - Atomic Absorption Spectrometry
ICP-OES	Inductively Coupled Plasma - Optical Emission Spectrometry
ICP-SFMS	Inductively Coupled Plasma – Sector Field Mass Spectrometry
JRC	Joint Research Centre
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PT	Proficiency Test

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 Feed Additives Control (EURL-FA Control), hosted by the Joint Research Centre of the European Commission, organised a proficiency test for the determination of the mass fraction of total selenium in a compound feed for rabbits.

This PT was agreed with the Directorate General for Health and Food Safety (DG SANTE) as part of the EURL-FA Control 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 Regulation (EC) No 882/2004 [1] and Regulation (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 fraction of total selenium (Se) in a compound feed for rabbits. Participants were also asked to evaluate the conformity of the investigated feed according to the maximum levels (MLs) set in Commission Directive 86/403/EEC concerning additives in feeding stuffs [3].

The reported results were assessed following the administrative and logistic procedures of the JRC Unit in charge of the EURL-FA Control, which is accredited for the organisation of PTs according to ISO 17043:2010 [4].

This PT is identified as FAC-18-01.

3. Set up of the exercise

3.1 Time frame

The organisation of this PT was announced to the NRL network at the 6th EURL-FA Control Workshop held in Brussels on November 22-23, 2017. An invitation letter was sent (via e-mail) to the NRLs' networks of the EURL-FA Authorisation and of the EURL-FA Control on March 26, 2018 (Annex 1). The registration deadline was set to April 15, 2018. Test items were sent to participants on May 22, 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 June 30, 2018.

3.2 Confidentiality

The procedures used for the organisation of this PT are accredited according to ISO 17043:2010 [4] and guarantee that the identity of the participants and the information provided by them are treated as confidential. However, the laboratory codes of those NRLs appointed in line with Regulation (EU) 2017/625 [2] may be disclosed to DG SANTE upon request for the purpose of an assessment of their (long-term) performance; while 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. 25 g 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).

3.4 Instructions to participants

Detailed instructions were given to participants in the test item "Accompanying letter" mentioned above. The measurand was defined as "the mass fraction of total Se in a compound feed for rabbits".

Participants were asked to perform measurements according to the method they use for official control, to report their result (x_i) and the associated expanded measurement uncertainty ($U(x_i)$) together with the coverage factor (k) and the analytical technique used for analysis.

Results had to be reported relative to a feed with a moisture content of 12 % in line with Council Directive 70/524/EEC concerning additives in feeding stuffs [5].

No instructions were provided by the EURL-FA Control to laboratories on how to perform the moisture corrections necessary for reporting, since official methods for moisture measurement exist.

Laboratory codes were given randomly and communicated to the participants by e-mail. The individual code had to be used to access the on-line reporting interface in order to report the measurement results and to complete the related questionnaire. This dedicated questionnaire was used to gather additional information related to measurements and laboratories (Annex 4).

4. Test item

4.1 Preparation

The commercially available compound feed for rabbits was purchased at a local market in Geel, Belgium. The producer reported the following composition on the label:

Small pellets – compound feed for rabbits
Analytical Constituents: 9.2 % crude ash, 17.0 % crude fibre, 15.0 % proteins, 3.3 % crude fat, 1.20 % calcium, 0.58 % phosphorus, 0.21 % sodium.
Nutritional additives: 10000 IU vitamin A (3a672a), 1000 IU vitamin D3 (3a671), 70 IU vitamin E (all-rac-alpha-tocopheryl acetate) (3a700), 8 mg Copper (II) sulphate pentahydrate (E4), 50 mg Iron (II) sulphate monohydrate (E1), 20 mg Manganese (II) oxide (3b502), 40 mg Zinc oxide (3b603), 0.1 mg Cobalt (coated granulated cobalt) (II) carbonate (3b604), 0.4 mg (3b201) Iodine (potassium iodide), 0.2 mg Selenium (sodium selenite) (E8) and (coccidiostats and histomonostats kg^{-1}) 66 mg robenidine hydrochloride (5 1 758).

Note: The mass fraction for the trace element containing feed additives is expressed in terms of the trace element.

One paper bag containing 10 kg of pelleted all-round feed for rabbits (starting material) was purchased. Pellets were pre-cooled over liquid nitrogen then fed into a cryogenic mill (Palla VM-KT, Humboldt-Wedag, Colone, DE). Milling was performed at -196 °C to -100 °C. All machine parts in contact with the animal feed were made of high-purity titanium. The resulting powder was sieved using a Russel Finex Industrial sieve equipped with a 250-µm stainless steel mesh (London, UK). After sieving, 9.6 kg of powder (with particle size below 250 µm) was homogenised using a Dynamix-CM200 mixer from WAB (Muttenz, CH). Mixing was performed during 1 h using a mixing program mimicking a Turbula mixer. 25-g portions were then filled into 80 units in 125-ml amber glass bottles and then closed with a screwcap with break-ring. Bottles were labelled from 1 to 80 according to filling order, and the name of the PT material was indicated on the label. The final material had a water content of about 7.7 % m/m (determined by volumetric Karl Fischer titration) and the top particle size was below 230 µm for the X90 fraction, which is consistent with sieving over a 250 µm mesh. The samples were kept at room temperature until shipment.

4.2 Homogeneity and stability

Measurements for the homogeneity and stability studies were performed by ALS Scandinavia AB (Luleå, Sweden). Inductively coupled plasma sector field mass spectrometry (ICP-SFMS) was used after microwave digestion (0.3 - 0.5 g of sample in a mixture of nitric acid (HNO_3), hydrogen peroxide (H_2O_2) and hydrofluoric acid (HF) in closed Teflon containers) to determine the mass fraction of total Se.

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

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 [6]; the contribution from homogeneity (u_{hom}) to the standard uncertainty of the assigned value ($u(x_{pt})$) was calculated using single-factor ANOVA. The test item proved to be adequately homogeneous for the investigated analyte (Annex 5).

The stability study confirmed that; i) the test item is adequately stable 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) the test item is adequately stable for 2 weeks 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).

The analytical results reported by the expert laboratories and the statistical evaluation of the homogeneity and stability studies are presented in Table 1.

5. Assigned values and corresponding uncertainties

5.1 Assigned values

The assigned value (x_{pt}) of the mass fraction of total Se in the compound feed for rabbit (relative to moisture content of 12 %) was calculated as the mean of the results reported by expert laboratories selected on the basis of their demonstrated measurement capabilities. The following expert laboratories reported results:

- ALS Scandinavia AB (Luleå, Sweden);
- SCK-CEN, Studiecentrum voor Kernenergie (Mol, Belgium);
- JRC. Directorate F – Health, Consumers and Reference Materials (Geel, Belgium).

The expert laboratories were asked to use the method of analysis of their choice and no further requirements were imposed regarding methodology. They were also requested to report their results together with the associated expanded measurement uncertainty and with a clear and detailed description on how their measurement uncertainty was calculated. Results had to be reported in dry mass. The EURL-FA Control converted afterwards these results to a feed with moisture content of 12 % as required by Council Directive 70/524/EEC [5] and Commission Directive 86/403/EEC [3].

- ALS Scandinavia used ICP-SFMS after closed microwave digestion using HNO_3 / H_2O_2 and HF in sealed Teflon containers. The ICP-SFMS analysis was carried out according to EN ISO 17294-1 and US EPA Method 200.8.

- SCK-CEN applied instrumental neutron activation analysis (k_0-NAA). Three samples of approx. 450 mg were transferred in standard high-density polyethylene vials and weighed. Samples were irradiated for seven hours in channel Y4 of the BR1 reactor together with several IRMM-530 (Al-0.1 % Au alloy) neutron flux monitors and two reference materials (SMELS III and ERM-BB422) used for validation.

- JRC-Geel followed the standard EN 16159 (hydride generation atomic absorption spectrometry (HG-AAS) after microwave digestion using HNO_3 (65 %) and H_2O_2 (30 %) as acid mixture [7].

Figure 1 presents the reported results and associated expanded uncertainties. Expert laboratories do not necessarily correspond to the order they were presented.

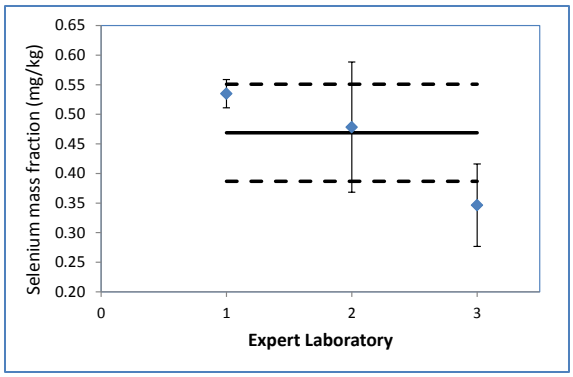

Figure 1:
Assigned value for Se in the compound feed for rabbit. Circles and error bars represent reported values by the expert laboratories, $x_i \pm 2u(x_i)$. The solid line refers to the assigned value (x_{pt}) while the dashed line refers to the assigned range ($x_{pt} \pm 2\,u(x_{pt})$).

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 [6]:

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

The uncertainty u_{char} is estimated according to the recommendations of ISO 13528:2015:

$$u_{char} = \frac{s}{\sqrt{p}} \qquad \text{Eq. 2}$$

where "s" refers to the standard deviation of the mean values obtained by the expert laboratories and "p" refers to the number of expert laboratories.

5.3 Standard deviation for proficiency assessment, σ_{pt}

A relative standard deviation for proficiency assessment (σ_{pt}) of 15 % of the assigned value was derived from the reproducibility standard deviation reported in the CEN standard EN 16159:2012 [7].

Table 1: Results reported by expert laboratories, their associated expanded uncertainties; the assigned value (x_{pt} and $u(x_{pt}, k=1)$); standard measurement uncertainties (u_{char}, u_{hom}). All values (except in the last column) are expressed in mg kg^{-1}, relative to feed with a moisture content of 12 %.

	Expert laboratories	x_{pt}	u_{char} [(b)]	u_{hom} [(b)]	$u(x_{pt})$ [(b)]	σ_{pt}	$u(x_{pt})/\sigma_{pt}$
Se	0.535 ± 0.024 [(a)] 0.478 ± 0.110 [(a)] 0.394 ± 0.079 [(a)]	0.469	0.041 (8.7 %)	0.020 (4.3 %)	0.046 (9.8 %)	0.070 (15 %)	0.65 *(unitless)*

(a) expanded uncertainty (k = 2); and (b) standard measurement uncertainty (k = 1).

6. Evaluation of results

6.1 Scores and evaluation criteria

The individual laboratory performance is generally expressed in terms of z and ζ performance scores according to ISO 13528:2015 [6]:

$$z = \frac{x_i - x_{pt}}{\sigma_{pt}} \qquad \text{Eq. 3}$$

$$\zeta = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}} \qquad \text{Eq. 4}$$

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.

As can be seen in Table 1 $u(x_{pt})$ *was* > 0.3 σ_{pt}; in such case ISO 13528:2015 advises to take into account the uncertainty of the assigned value ($u(x_{pt})$) by expanding the denominator of the z score and calculating the z' score as in Equation 5, together with the ζ score, to express the individual laboratory performance.

$$z'_i = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u^2(x_{pt})}} \qquad \text{Eq. 5}$$

The interpretation of the z' and ζ performance scores is done as follows [6]:

|score| ≤ 2	satisfactory performance	(green in Annex 6 and 7)
2 < |score| < 3	questionable performance	(yellow in Annex 6 and 7)
|score| ≥ 3	unsatisfactory performance	(orange in Annex 6 and 7)

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.

Uncertainty estimation is not trivial, therefore an additional assessment is 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 *or* 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 Laboratory results and scorings

6.2.1 Performances

Annex 6 presents the results reported by the 25 laboratories having registered to this PT. It includes the table 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 [8].

Figure 2 presents the laboratory performances assessed by the z' and ζ scores. 88 % of the participants having reported results performed satisfactorily according to both scores. Laboratories C-02 and O-06 reported results significantly higher than the assigned value (ca. 2 and 5 times higher) which resulted in z' scores largely above 3.

Most of the participants applied ICP-MS (64 %), HG-AAS (16 %) or ICP coupled with optical emission spectrometry (ICP-OES, 12 %). The experimental details are provided in Annex 7.

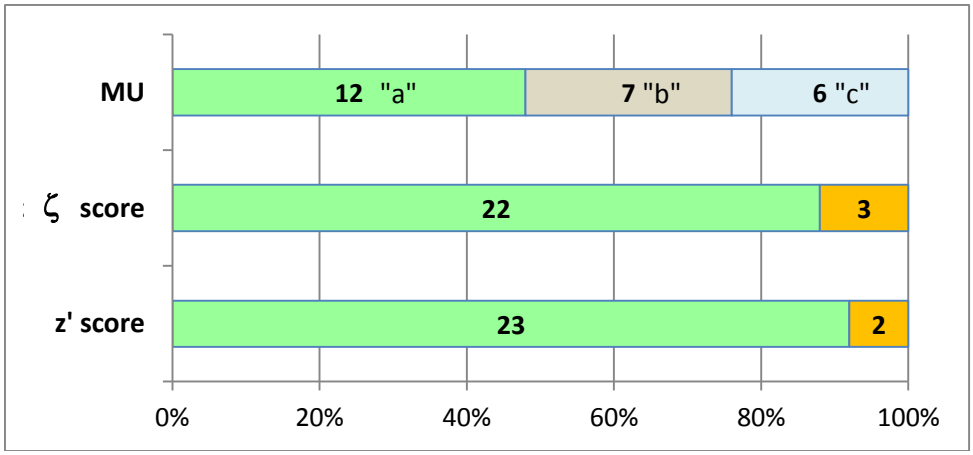

Figure 2: Overview of laboratory performance according to z' and ζ (zeta) scores, together with the measurement uncertainty (MU) evaluation. Corresponding number of laboratories included in the graph. Satisfactory and unsatisfactory performances indicated in green and orange; case "a"; "b"; "c" for MU indicated in green, grey and blue, respectively.

The robust mean (0.493 mg kg^{-1}) and its associated standard measurement uncertainty (expressed as a robust standard deviation, 0.083 mg kg^{-1}) from all the values reported by the participants were computed according to ISO 13528:2015 (Algorithm A). These values are in agreement with the assigned range, $x_{pt} \pm U(x_{pt})$ which demonstrates the absence of any significant bias of the robust mean of results from the participant laboratories.

6.2.2 Measurement uncertainties

Figure 2 shows that 48 % of the participants reported realistic measurement uncertainty evaluations (case "a": $u(x_{pt}) \leq u(x_i) \leq \sigma_{pt}$). Similarly, 36 % of them reported a potentially under-estimated (relative) measurement uncertainty around 5 % (case "b"), while the remaining laboratories reported seemingly over-estimated (relative) measurement uncertainties, of the order of 20 to 22 % (case "c"), probably derived from the Horwitz model. Laboratory A-21 may have reported its' measurement uncertainty in % instead of mg kg^{-1}.

6.2.3 Compliance assessment

Council Directive 70/524/EEC [5] and Commission Directive 86/403/EEC [3] concerning additives in feeding stuffs set a maximum content (also referred as maximum level, ML) of **0.5 mg kg^{-1}** (expressed as Se) for sodium selenite in complete feed with a moisture content of 12 %. This ML applies to the compound feed for rabbit distributed in the frame of this PT.

Technical tolerances have been established for feed additives by Regulation (EC) No 767/2009 [9], which needs to be used when comparing results of analysis against the labelled content of these feed additives in compound feed. Furthermore, this legislation specifies that these technical tolerances do not apply, when checking for compliance with a maximum content as established in the respective authorisation act. In such a case, exclusively the measurement uncertainty needs to be taken into account and corresponding provisions for undesirable substances established by Regulation (EC) No 152/2009 [10] have been adopted for the assessment of this study. In particular, this is relevant for laboratories that have obtained a result of analysis, which is above the maximum content. Thus, only analytical compliance do apply in the present study.

The assigned value of 0.469 ± 0.092 mg kg^{-1} (k = 2) clearly overlaps with the maximum content set by the recent legislation. **The test item is therefore considered to be compliant**.

Participants were requested to assess the analytical compliance of the test item, and to provide proper justification supporting their statement. In order to assess the consistency of the laboratory compliance statement, the following three components have to be considered:

- The laboratory compliance statement (compliant or non-compliant);
- The laboratory measurement results:
  - reported (or not) for the analyte of interest;
  - to be compared to the relevant ML (x_i - $U(x_i)$ > ML ?) selecting the correct ML for the intended feed matrix;
- The laboratory justification for its compliance assessment (correct or incorrect).

Eighteen participant laboratories (out of the 22 which have provided the analytical compliance assessment statement, 82 %) **correctly assessed the test item to be compliant** according to Commission Directive 86/403/EEC, and their decision is in line with their reported values and associated (expanded) measurement uncertainty.

Laboratory A-17 incorrectly considered the test item as non-compliant, while reporting a range well below the ML (0.390 ± 0.059 < 0.5 mg kg^{-1}, cf. Figure Annex 6).

Based on their reported results (with ranges clearly above 0.5 mg kg^{-1}), three laboratories should have assessed the sample as not compliant (C-02, O-06 and O-23). Only the later provided a coherent statement of non-compliance, while the other two incorrectly assessed the sample as compliant.

Three laboratories did not provide any analytical compliance assessment (A-25; C-10 and O-11).

6.2.4 Additional information extracted from the questionnaire

The questionnaire was answered by all participants giving valuable information on the laboratories, their way of working and their analytical methods (details provided in Annex 7).

The following instrumental techniques were used: ICP-MS (16 laboratories); ICP-OES (3); HG-AAS (4); ICP-OES (3) and electro-thermal-AAS (ET-AAS, 2). Ten participants (40 %) followed a standard method. Only 4 participants corrected their results for recovery.

Seventeen participants stated that they are accredited for the determination of selenium in feed, while 21 laboratories (88 %) acknowledged having participated to similar PTs in the past.

Most of the laboratories used microwave digestion with nitric acid and hydrogen peroxide (or with HCl) to ensure complete digestion of the feed test sample. The recovery factor was mainly determined using a (certified) reference material (71 %) or by spiking (24 %) a known amount of the same analyte.

Several approaches were used to estimate measurement uncertainties (Table 2). Most of the laboratories (56 %) derived their measurement uncertainty from their single-laboratory validation studies.

The majority of the participant laboratories are analysing ca. 50 "similar" samples per year (58 % from 1 to 50 samples, and 21 % from 51 to 250 samples). Only one laboratory stated to have no experience with such type of analysis (never analysed such type of sample).

Annex 7 summarises the experimental details, the technique used and the limits of quantification (LOQ) for the determination of Se. Large discrepancies in LOQs are observed (from 0.002 to 2.5 mg kg^{-1}) even among laboratories using the same instrumental technique (ICP-OES).

Table 2: Overview of the approaches used to estimate measurement uncertainties (multiple selections are possible).

Approach	N° of labs
According to ISO-GUM	4
From known uncertainty of a standard method	1
Derived from a single-laboratory validation study	14
Determined as standard deviation of replicate measurements	10
Estimation based on judgment	2
Derived from interlaboratory comparison data	4

7. Conclusions

The proficiency test FAC-18-01 was organised in 2018 to assess the analytical capabilities of the EU NRLs and OCLs on the determination of the mass fractions of total Se in a compound feed for rabbits.

The overall performance of the participants (23 out of 25, i.e. 92 %) was satisfactory. This confirms their analytical capabilities to enforce the Commission Directive 86/403/EEC amending the Annexes to Council Directive 70/524/EEC setting maximum levels of selenium in feeding stuffs.

Similarly, 18 participants (82 %) correctly assessed the test item to be compliant according to the Commission Directive 86/403/EEC. Three laboratories provided erroneous conclusions contradicting their reported result and associated measurement uncertainty; three other laboratories did not provide any assessment.

Most of the participants reported reasonable measurement uncertainty estimations.

Acknowledgements

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

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

Organisation	**Country**
Austrian Agency for Health and Food Safety (AGES GmbH)	Austria
Federal Agency for the Safety of the Food Chain (FAVV-AFSCA)	Belgium
Analytical Laboratories Section – Feeding Stuffs Quality Control Laboratory	Cyprus
Central Institute for Supervising and Testing in Agriculture (ÚKZÚZ)	Czech Republic
The Danish Veterinary and Food Administration (FVST)	Denmark
Agricultural Research Centre	Estonia
Finnish Food Safety Authority (Evira)	Finland
Service Commun des Laboratoires (DGDDI+DGCCRF)	France
Federal Institute for Risk Assessment (BfR)	Germany
Thüringer Landesanstalt für Landwirtschaft (TLL)	Germany
National food Chain Safety Office (NEBIH)	Hungary
The State Laboratory	Ireland
Institute of Food Safety, Animal Health and Environment	Latvia
National food and veterinary risk assessment institute	Lithuania
RIKILT Wageningen University and Research	Netherlands
Institute of Marine Research	Norway
National Research Institute of Animal Production	Poland
University of Ljubljana Veterinary Faculty, National Veterinary Institute	Slovenia
National Food Agency (SLV)	Sweden
Federal Department of Economic Affairs, Education and Research EAER, Agroscope, Institute for Livestock Sciences ILS, Animal Feed Analytics	Switzerland
LGC Limited	United Kingdom
Worcestershire Scientific Services	United Kingdom
Tayside Scientific Services	United Kingdom
Public Analyst Scientific Services Limited	United Kingdom
Lancashire County Scientific services	United Kingdom

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[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 Directive 86/403/EEC amending the Annexes to Council Directive 70/524/EEC concerning additives in feeding stuffs.

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

[5] Council Directive 70/524/EEC concerning additives in feeding-stuffs.

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

[7] EN 16959:2012, *"Animal feeding stuffs - Determination of selenium by hydride generation atomic absorption spectrometry (HG-AAS) after microwave digestion (digestion with 65 % nitric acid and 30 % hydrogen peroxide)"*, European Committee for Standardisation (CEN).

[8] 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.

[9] Regulation (EC) No 767/2009 of the European Parliament and of the Council on the placing on the market and use of feed, amending European Parliament and Council Regulation (EC) No 1831/2003 and repealing Council Directive 79/373/EEC, Commission Directive 80/511/EEC, Council Directives 82/471/EEC, 83/228/EEC, 93/74/EEC, 93/113/EC and 96/25/EC and Commission Decision 2004/217/EC.

[10] Commission Regulation (EC) No 152/2009 laying down the methods of sampling and analysis for the official control of feed.

[11] ISO Guide 35:2017 *"Reference materials – Guidance for the characterization and assessment of homogeneity and stability"*, ISO-Geneva (CH), International Organization for Standardization.

Annex 1: Invitation letter

EUROPEAN COMMISSION
Joint Research Centre
Directorate F – Health, Consumers & Reference Materials
European Union Reference Laboratory for Feed Additives Control

Geel, 26 March 2018

(sent by e-mail)

Subject: Invitation to participate in Proficiency Test round "FAC-18-01"

Dear National Reference Laboratory representative,

On behalf of the EURL for Feed Additives Control, we would like to invite you to participate in the Proficiency Test (PT) round FAC-18-01 on the **"Determination of the mass fraction of total selenium in compound feed".**

The PT fulfils the EURL-FA Control 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-FA Control if you hold a mandate for this type of matrix or to mandate a laboratory to represent you in the exercise.

In case you plan to pay for the participation of official feed control laboratories belonging to your national network, please inform them that their identity will be disclosed to you.

Your participation is free of charge.

The main objective of this study is to assess the proficiency of the participating laboratories to correctly determine total selenium in feed samples at levels authorised in the European legislation. The evaluation of the results will show which laboratories deliver acceptable results. The proficiency test also includes evaluating the capability of the laboratories to carry out the requested analysis within a defined time frame.

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=1982

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-feed-additives-control@ec.europa.eu)

Please register by the 15th of April 2018. Please fill in the form comprehensively. These details will be used for the dispatch of the materials and for any further correspondence.

Official control laboratories may also participate in the proficiency exercise if places are still available. The total number of participating laboratories may not exceed 30. 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 middle of May 2018.

The deadline for submission of results is the end of June 2018.

The exercise will be completed by the organisation of the annual workshop opened to all participants to the study and to all NRLs for Feed additives. The target period for the workshop is

18 October 2018 – 05 November 2018. The workshop is free of charge. Travel and accommodation costs will be reimbursed for one representative of each National reference Laboratory for Feed Additives defined according to Regulation (EC) No 882/2004 or Regulation (EC) No 1831/2003.

Do not hesitate to contact us if you have any further questions at JRC-EURL-FEED-ADDITIVES-CONTROL@ec.europa.eu.

Kind regards,

/signed electronically in Ares/
Dr. F. Cordeiro
FAC-18-01 PT Coordinator

/signed electronically in Ares/
Dr. U. Vincent
Team leader EURL-FA Control

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

Annex 2: Test item accompanying letter

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate F - Health, Consumers and Reference Materials
European Union Reference Laboratory for Feed Additives Control

EURL
European Union Reference Laboratory
Feed Additives

Geel, 04 June 2018

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

Subject: Participation in FAC-18-01

Dear «Title» «Surname»,

Thank you for participating in the FAC-18-01 proficiency test. This PT is organised in support to Commission Directive 86/403/EC concerning additives in feeding stuffs.

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

- one bottle of the test item (approx. 25 g); and
- "Confirmation of receipt" form.

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

Upon arrival of this parcel, please check whether the test item is undamaged after transport, and send us by fax or email the "Confirmation of receipt" form.

Store the samples until analysis in a dark place at +4°C (fridge).

The mandatory measurand is **"the mass fraction of total Se in compound feed".**

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

The test item homogeneity is demonstrated at a certain sample intake level: **please use a test portion for analysis not lower than 0.4 g.**

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

Determine the moisture content and correct the measurement results for moisture content as prescribed in DIR 2002/32/EC.

1

Perform two or three independent measurements and report:

- the result for the **moisture content** determination (in % w/w),
- the **mean** of your two or three measurements results (in mg kg^{-1}),
- the associated expanded **uncertainty** (in mg kg^{-1}),
- the **coverage factor**, and
- the **analytical technique** used.

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

The reporting website is https://web.jrc.ec.europa.eu/ilcReportingWeb

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

The system will guide you through the reporting procedure. Then complete the corresponding questionnaire. **Do not forget to submit and confirm when required.**

Directly after submitting your results and the questionnaire information online, you will be requested to print the completed report form.

Please check carefully your report. In the case mistakes are detected contact the PT coordinator as soon as possible before the reporting deadline.

The deadline for submission of results is **30/06/2018**.

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.

Your participation in this project is greatly appreciated.

Do not hesitate to contact me for further information.

With kind regards,

/signed electronically in Ares/

Dr. Fernando Cordeiro
FAC-18-01 PT Coordinator

Cc: H. Emons (Head of Unit, JRC F.5, Food & Feed Compliance unit)
U. Vincent (Team leader EURL-FA Control)

Annex 3: Confirmation of receipt form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Directorate F – Health, Consumers and Reference Materials
European Union Reference Laboratory for Feed Additives Control

Geel, 22 May 2018
Ares(2018)2984004

Attn.: «Title» «Firstname» «Surname»
«Organisation»
«Department»
«Country»

Subject: "Confirmation receipt" form
FAC-18-01 "Determination of the mass fraction of total Se in compound feed"

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. F. Cordeiro
FAC-18-01 PT Coordinator
e-mail : jrc-eurl-feed-additives-control@ec.europa.eu

Retieseweg 111, B-2440 Geel – Belgium Tel.: +32 14 57 12 11. Direct line: +32 14 57 16 87.
E-mail: jrc-eurl-feed-additives-control@ec.europa.eu

Annex 4: Questionnaire

Milc questionnaire

Comparison for FAC-18-01

This questionnaire is meant to collect additional information about your laboratory and experimental details about your analytical method. Your answers will be used in the evaluation of the proficiency test FAC-18-01. Please enter the information related to the method used for the determination of the mass fraction of total Se in feed. Please do so comprehensively, in order to allow appropriate evaluation and relevant discussion of the results.

Submission Form

1. **Please identify yourself - you are ...**

- ☐ a) National Reference Laboratory (NRL)
- ☐ b) Official Control Laboratory (OCL)
- ☐ c) Other

2. **Is the test item compliant according to the relevant European legislation?**

- ☐ a) Yes
- ☐ b) No

2.1. If "Not compliant" specify why

3. **Did you follow a standard method for the determination of the analyte?**

- ☐ a) Yes
- ☐ b) No

3.1. If "Yes" please identify it

4. **Did you correct your measurement for the analytical recovery?**

- ☐ a) Yes
- ☐ b) No

5. **How did you estimate your recovery?**

- ☐ a) Using a CRM
- ☐ b) Spiking
- ☐ c) Other

6. **Provide the recovery and the limit of quantification (LOQ)**

See table **Recovery (%) and LOQ (mg/kg) of the method:** at bottom

7. **Which calibrants did you use for (instrumental) calibration and method validation?**

See table **Instrument calibration and method validation** at bottom

8. **Are you accredited for Se determination in feed?**

- ☐ a) Yes
- ☐ b) No

9. **How many analysis of this type does your laboratory perform per year?**

- ☐ a) Never
- ☐ b) 0-50
- ☐ c) 51-250
- ☐ d) 251-1000
- ☐ e) > 1000

10. **Experimental details**

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

11. **Do you usually provide a measurement uncertainty statement to your customers?**

- ☐ a) Yes
- ☐ b) No

12. **How did you estimate your measurement uncertainty?**

- ☐ a) Uncertainty budget (ISO GUM)
- ☐ b) Known uncertainty from standard method (ISO 21748)
- ☐ c) From in-house (method) validation
- ☐ d) Measurement of replicates (precision)
- ☐ e) Estimation based on judgment
- ☐ f) From interlaboratory comparison
- ☐ g) Other

13. **Do you participate in other PTs for this type of analysis?**

- ☐ a) Yes
- ☐ b) No

14. **Do you have any comments? Let us know.**

Experimental details (method)

Please provide some details related with the method you used.

Questions/Response table	*Se (mass fraction)*
Digestion type (e.g. microwave, dry ashing, pressure bomb, etc)	
Digestion mixture (which acids were used)	
Digestion time (min)	
Digestion temperature (oC)	
Did you dilute the test solution after digestion?	

Instrument calibration and method validation

Questions/Response table	*Instrument calibration*	*Method validation*
Se		

Recovery (%) and LOQ (mg/kg) of the method:

Questions/Response table	*Recovery (%)*	*LOQ (mg/kg)*
Se		

Annex 5: Homogeneity and stability studies

A5.1 Homogeneity study

Bottle #	Replicate 1(*)	Replicate 2(*)
15	0.374	0.403
71	0.368	0.384
45	0.450	0.385
55	0.392	0.361
14	0.431	0.425
74	0.402	0.363
27	0.350	0.364
78	0.339	0.466
29	0.372	0.353
66	0.384	0.361

Mean(*)	0.3864	Parameter definition
s_s(*)	0	Between-sample standard deviation
s_w(*)	0.0231	Within-sample standard deviation
s_x(*)	0.0231	Standard deviation of the sample averages
u'_{bb}(*)	0.0167	Conservative value for the uncertainty associated with heterogeneity [11]
u_{hom} *(in %)*	**4.3%**	**Standard uncertainty contribution from homogeneity**
σ_{pt} (in %)	15%	Standard deviation for the PT assessment
$s_s < 0.3\ \sigma_{pt}$	**pass**	**Test item adequately homogeneous**

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

weeks:	1	1	8	8
Rep.1(*)	0.485	0.454	0.569	0.45
Rep.2(*)	0.506	0.454	0.508	0.461

Slope ± 2 $SE_{(slope)}$ = 0.0032 ± 0.0086 (mg kg^{-1} $week^{-1}$)
where $SE_{(slope)}$ is the standard error of the slope.

No significant slope detected; hence the **test item is adequately stable**, and u_{st} **= 0**

(*) Values expressed in mg kg^{-1}, not corrected for moisture content)

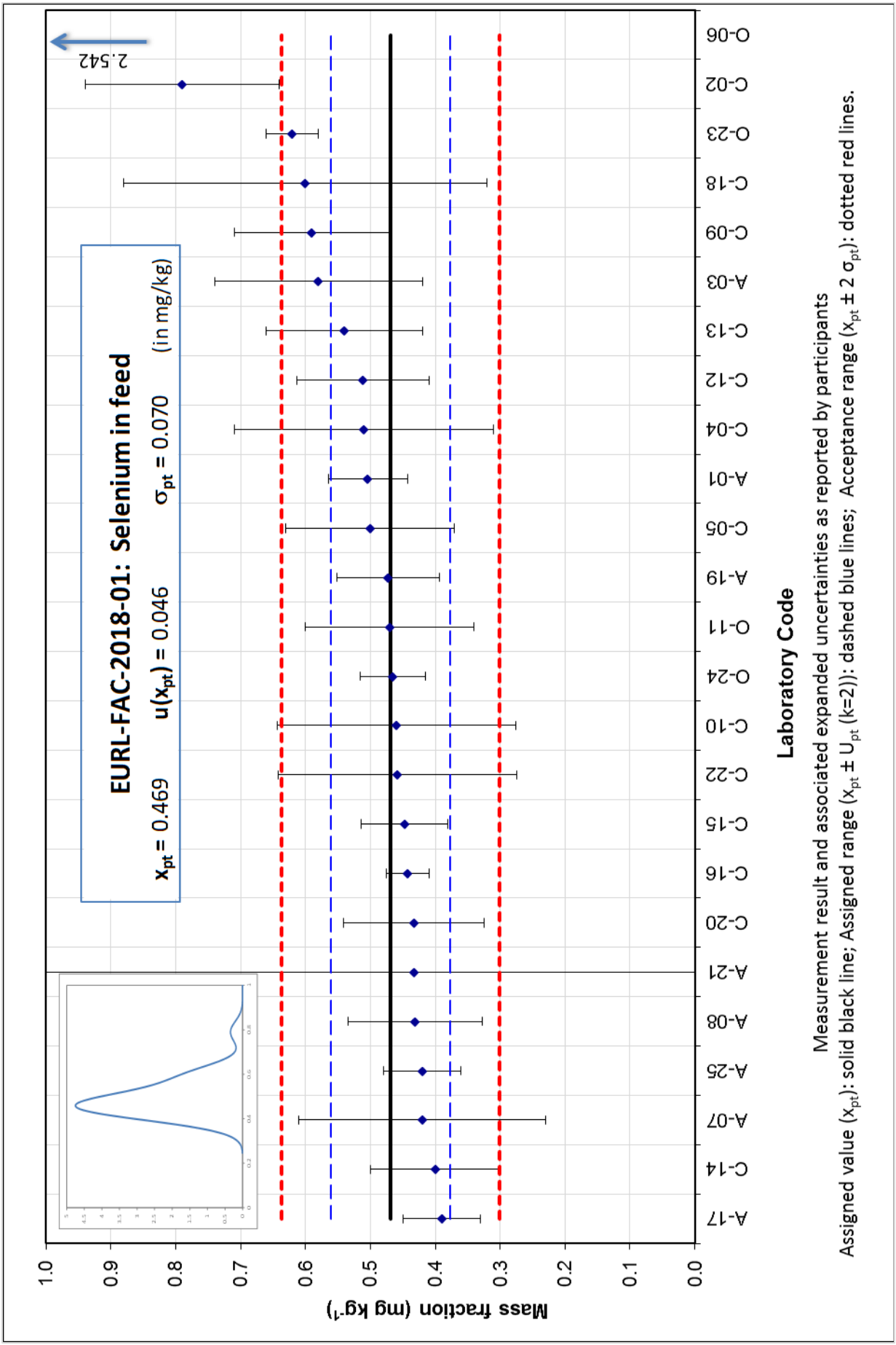
Annex 6: Results for mass fraction of total selenium

Assigned range: x_{pt} = 0.469 ± 0.092 $U(x_{pt}, k = 2)$; and σ_{pt} = 0.070
(x_i, $U(x_i)$ and $u(x_i)$ in mg kg^{-1}, relative to a feed with a moisture content of 12 %).

Lab Code	x_i	$U(x_i)$	k	Technique	$u(x_i)$	z' score	ζ score	unc.	Comply
A-01	0.504	0.061	2	ICP-MS	0.031	0.4	0.6	b	Yes
A-03	0.58	0.16	2	ICP-MS	0.080	1.3	1.2	a	Yes
A-07	0.42	0.19	2	ICP-MS	0.095	-0.6	-0.5	c	Yes
A-08	0.431	0.104	2	ICP-MS	0.052	-0.5	-0.5	a	Yes
A-17	0.390	0.059	2	ICP-OES	0.030	-0.9	-1.4	b	No
A-19	0.472	0.079	2	ICP-MS	0.040	0.0	0.1	a	Yes
A-21	0.432	10	2	HG-AAS	5.000	-0.4	0.0	c	Yes
A-25	0.42	0.06	2	ICP-MS	0.030	-0.6	-0.9	b	
C-02	0.79	0.15	2	HG-AAS	0.075	3.8	3.7	a	Yes
C-04	0.51	0.2	2	ICP-MS	0.100	0.5	0.4	c	Yes
C-05	0.50	0.13	2	ICP-MS	0.065	0.4	0.4	a	Yes
C-09	0.59	0.12	2	ET-AAS	0.060	1.4	1.6	a	Yes
C-10	0.46	0.184	2	ICP-MS	0.092	-0.1	-0.1	c	
C-12	0.512	0.102	2	HG-AAS	0.051	0.5	0.6	a	Yes
C-13	0.54	0.12	2	ICP-MS	0.060	0.8	0.9	a	Yes
C-14	0.4	0.1	2	ICP-MS	0.050	-0.8	-1.0	a	Yes
C-15	0.447	0.067	2	ICP-MS	0.034	-0.3	-0.4	b	Yes
C-16	0.442	0.033	2	ICP-MS	0.017	-0.3	-0.6	b	Yes
C-18	0.60	0.28	2	ICP-MS	0.140	1.6	0.9	c	Yes
C-20	0.433	0.108	2	ICP-MS	0.054	-0.4	-0.5	a	Yes
C-22	0.458	0.184	2	ET-AAS	0.092	-0.1	-0.1	c	Yes
O-06	2.542	0.112	2	ICP-OES	0.056	24.7	28.7	a	Yes
O-11	0.47	0.13	2	HG-AAS	0.065	0.0	0.0	a	
O-23	0.62	0.04	2	ICP-MS	0.020	1.8	3.0	b	No
O-24	0.465	0.05	2	ICP-OES	0.025	0.0	-0.1	b	Yes

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

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


EURL-FAC-2018-01: Selenium in feed
x_{pt} = 0.469 $u(x_{pt})$ = 0.046 σ_{pt} = 0.070 (in mg/kg)
2.542
Mass fraction (mg kg-1)
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
A-17
C-14
A-07
A-25
A-08
A-21
C-20
C-16
C-15
C-22
C-10
O-24
O-11
A-19
C-05
A-01
C-04
C-12
C-13
A-03
C-09
C-18
O-23
C-02
O-06
Laboratory Code
Measurement result and associated expanded uncertainties 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: Experimental details

LCode	Standard method	Recovery correction	Recovery estimation	Recovery (%)	LOQ (mg/kg)	Accredited	Samples /year	Uncertainty statement	Digestion type	Digestion mixture	Digestion time (min)	Digestion temp. (°C)	Dilute test solution	Instrument calibration	Method validation
A-01	No	No	CRM	100	0.065	No	0-50	Yes	CMW	HNO_3+H_2O_2	30	180	Yes	Se standard in HNO3	
A-03	Yes	No	CRM	100	0.34	Yes	0-50	Yes	MW	HNO_3+HCl	30	220	Yes	SpectraScan	Matrix CRM:s
A-07	No	No	CRM	75	0.2	Yes	0-50	Yes	MW	HNO_3+H_2O_2	160	180	Yes	external calibration	PT samples, routine sampl
A-08	Yes	No	CRM, other	96-110	0.1	No	0-50	Yes	MW	HNO_3+H_2O_2	55	210	Yes	0.5-25 µg/L	
A-17	No	Yes	CRM	90	0.05	Yes	0-50	Yes	MW	HNO_3+H_2O_2	30	200	Yes	CPAChem	
A-19	No	Yes	CRM	97	0.033	Yes	0-50	Yes	MW	HNO_3+H_2O_2	20	200	Yes	VAR TS-MS	Merck solution XVI
A-21	Yes	No	CRM	100	0.03	Yes	251-1000	Yes	MW	65 % HNO_3	30	250	Yes	extern matrixangepasst	DIN EN 17025
A-25	No	No	CRM	95.6	0.04	Yes	51-250	Yes	MW	HNO_3+H_2O_2	30	200	Yes	Nexton setup solution	SCP 33MS, SCP Science
C-02	Yes	No	Other		0.05	Yes	251-1000	Yes	DA	HNO_3	60	130-180	Yes	Std from sigma	Std from sigma
C-04	Yes	No	CRM	110	0.25	No	0-50	No	MW	HNO_3+H_2O_2	20	210	Yes	Perkin-Elmer multistd.	RM from PT
C-05	No	No	CRM	115	0.06	Yes	0-50	Yes	MW	HNO_3+H_2O_2	40	210	Yes	External linear	External linear
C-09	No	Yes	Other	85		No	Never	No	MW	HNO_3+H_2O_2	30	200	20ml		
C-10	Yes		CRM		0.01	Yes	> 1000	No	MW	HNO_3	45	260	Yes		
C-12	Yes	No		100	0.04	Yes	51-250	Yes	OD	HNO_3+$HClO_4$+HCl	540	250	Yes	Merck	NIST
C-13	Yes	No			0.1	Yes	51-250	Yes	CMW	HNO_3+H_2O_2	32	200	Yes	multielementary standard	NIES CRM No. 27
C-14	Yes	No			0.17	Yes	51-250	Yes	MW	HNO_3+HCl+H_2O_2	60	175	Yes		
C-15	No	No	CRM	95-110	0,05	Yes	51-250	Yes	OMW	HNO_3	10	190	Yes	CRM Astasol,Analytika CZ	PT ALVA 2018,EURL-HM-25
C-16	No	No	Spiking	98	0.01	Yes	251-1000	Yes	MW	HNO_3	30	200	No	Standard curve	standard curve
C-18	No	No	CRM, spiking	80	0.55	No	0-50	Yes	MW	HNO_3+H_2O_2	15	200	No	Se 5-200 ugl	Se 5-200 ugl
C-20	Yes	No			0.040	Yes	0-50	Yes	MW	HNO_3+H_2O_2	50	180	No		0.5-100 ppb
C-22	No	No	Spiking	101	0.2	No	0-50	No	MW	HNO_3+H_2O_2	35	200	No	Se standard	BIPEA Rabbit feed
O-06	No	No	Spiking	76.4	2.5	No	0-50	No	WD	65% HNO_3	25	100	No	commercially available se	selenium solutions in nit
O-11	No	No	CRM	101.6	0.05	Yes	0-50	No	DA	HNO_3	30		Yes	1000ug/ml (Guide 34)	
O-23	No	No	CRM	109	0.19	No	0-50	No	Digi prep	HNO_3+HCl	180	105	Yes	Multi element standard	Reference materials
O-24	No	Yes	Spiking	112	0.002	Yes	> 1000	No	MW	HNO_3+HCl	100, 160,180		Yes	ISO Guide 34	In-house QA/020

CMW: closed microwave; MW: microwave; DA: dry ashing; OD: open digestion; OMW: open microwave; WD: wet digestion

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KJ-NA-29408-EN-N

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doi:10.2760/562439

ISBN 978-92-79-96747-4