European Commission

JRC TECHNICAL REPORTS

EURL-FA Control Proficiency Test Report

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

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2017

Joint Research Centre

EUR 29038 EN

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

JRC 110506

EUR 29038 EN

PDF ISBN 978-92-79-77624-3 ISSN 1831-9424 doi:10.2760/9785

Luxembourg: Publications Office of the European Union, 2018

How to cite this report: P. Robouch, M. Chedin, H. Emteborg, Geert Van Britsom, Ch. von Holst and U. Vincent, "*EURL-FA Control Proficiency Test Report, Determination of the mass fraction of the total cobalt in compound feed for rabbits*", JRC Technical Reports, 2018, EUR 29038 EN, doi 10.2760/9785

European Union Reference Laboratory
Feed Additives

EURL-FA Control
Proficiency test report

Determination of the mass fraction of total cobalt 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 (EURL-FAC 2017-2) for the determination of the mass fraction of total cobalt in compound feed, to support the Commission Implementing Regulation (EU) No 131/2014 concerning the authorisation of various cobalt(II) compounds as feed additives. 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 the coated granulated cobalt (II) carbonate, 3b304[1]) which, after appropriate processing, was bottled, labelled and dispatched to participants on June 21, 2017. 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 two OCLs from 21 countries - representing EU Member States and Norway - registered to the exercise and reported results at the end of August 2017.

Laboratory results were rated using z' and zeta scores in accordance with ISO 13528:2015. A relative standard deviation for proficiency assessment (σ_{pt}) of 16 % of the assigned value was set according to the modified Horwitz equation.

Twenty (out of 23) laboratories reported satisfactory results (according to the z' score). This confirms the ability of most NRLs in monitoring maximum levels set by the Commission Implementing Regulation (EU) No 131/2014 in this type of animal feed.

The majority (74 %) of the participating laboratories provided realistic estimates of their measurement uncertainties.

[1] Coated granulated cobalt (II) carbonate is registered as a feed additive in Category 3, functional group b, in the sub-classification 'Compound of trace elements' with the code 3b304 (*https://ec.europa.eu/food/sites/food/files/safety/docs/animal-feed-eu-reg-comm_register_feed_additives_1831-03.pdf*)

1. Introduction

The European Union Reference Laboratory for Feed Additive Control (EURL-FA Control), hosted by the Joint Research Centre (JRC) in Geel, organised a proficiency test (PT) for the determination of the mass fraction of total cobalt 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 2017. 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] one of the core duties of EURLs is to organise interlaboratory comparisons for the benefit of NRLs.

The present PT aims to assess the performance of NRLs and OCLs in the determination of the mass fractions of total cobalt (Co) 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 Implementing Regulation (EU) No 131/2014 concerning the authorisation of various cobalt(II) compounds as feed additives [2].

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 [3].

This PT is identified as EURL-FA 2017-2.

3. Set up of the exercise

3.1 Time frame

The organisation of this PT was announced to the NRL network at the 5th EURL-FA Control Workshop held in Brussels on November 22-23, 2016. An invitation letter was sent (via e-mail) to the NRLs of the EURL-FA Authorisation, EURL FA Control and EURL for Heavy Metals (EURL-HM) networks on May 3, 2017 (Annex 1). The registration deadline was set to May 26, 2017. Samples were sent to participants on June 21, 2017. 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 August 29, 2017.

3.2 Confidentiality

The procedures used for the organisation of PTs are accredited according to ISO 17043:2010 [3] 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.

3.3 Distribution

Each participant received:

- One bottle of the test item (containing approx.. 15 g of material);
- The "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 "Accompanying letter" mentioned above. The measurand was defined as "the mass fraction of total Co in a compound feed for rabbits".

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

Results were to be reported relative to a feed with a moisture content of 12 % in line with Commission Implementing Regulation (EU) No 131/2014.

Upon specific request from DG SANTE, no instructions were provided by the EURL-FA Control to laboratories on how to perform the moisture corrections necessary for reporting, since official control laboratories are supposed to know the proper procedure.

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

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

The laboratory codes were given randomly and communicated to the participants by e-mail.

4. Test item

4.1 Preparation

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

Small pellets – compound feed for rabbits
Analytical Constituents: 16.7% crude proteins; 3.0% Fat; 7.8% crude ash; 13.0% cellulose; 1.06% Ca; 0.75% Total P; and 0.22% Na.
Nutritional additives: 10000 IU kg^{-1} Vitamin A; (3a672a) 800 IU kg^{-1} Vitamin D3 (E671); 70 mg kg^{-1} Vitamin E (3a700); 50 mg kg^{-1} $FeSO_4.H_2O$ (E1); 0.4 mg kg^{-1} $Ca(IO_3)_2/I_2$; **0.2 mg kg^{-1} $CoCO_3$/Co (3b304)**; 10 mg kg^{-1} $CuSO_4{\cdot}5H_2O$/Cu (E4); 20 mg kg^{-1} MnO/Mn (E5); 50 mg kg^{-1} ZnO/Zn (E6); 10 mg kg^{-1} $C_4H_8N_2O_4Zn$/Zn(E6); 0.30 mg kg^{-1} Na_2SeO_3/Se (E8); and 20.0 mg kg^{-1} salinomycin Na (E766).

Note: The label of the compound contains the added amount of the feed additive as specified by article 15 (f) and Annex VI, chapter 1 of Regulation (EC) No 767/2009 last amended Commission Regulation (EU) No 939/2010. 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 at a local market. 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, 9.4 kg of powder (with particle size below 250 µm) was homogenised using a Dynamix-CM200 mixer from WAB (Muttenz, CH). Mixing was performed for 1 h using a mixing program mimicking a Turbula mixer. 15-g portions were then filled into 80 units of 50-ml amber glass bottles equipped with a screwcap having a break-ring. Bottles were labelled from 1 to 80 according to filling order, and the name of the PT material was indicated. The final material had a water content of around 9 % m/m (determined by an oven method) 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 mass spectrometry (ICP-MS) was used after microwave digestion (0.3 - 0.5 g of sample in a mixture of HNO_3/H_2O_2) to determine the mass fraction of total Co.

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

Homogeneity was evaluated according to ISO 13528:2015 [4]. The test item proved to be adequately homogeneous for the investigated analytes.

The stability study confirmed that the material was stable at room temperature over the whole period of the PT (8 weeks, from dispatch of the samples to participants till the deadline for reporting of results). Hence, the uncertainty contribution due to stability was set to zero (u_{st} = 0).

The contribution from homogeneity (u_{hom}) to the standard uncertainty of the assigned value ($u(x_{pt})$) was calculated using SoftCRM [5]. The analytical results reported by the expert laboratories and the statistical evaluation of the homogeneity and stability studies are presented in Annex 6 and Table 1.

5. Assigned values and corresponding uncertainties

Table 1 summarises the assigned value for the mass fraction of cobalt, the associated uncertainty values and the standard deviation for proficiency assessment.

Table 1: Expert laboratory results and associated expanded measurement uncertainties; assigned value (x_{pt} and $u(x_{pt}, k=1)$); and standard uncertainties (u_{char} *and* u_{hom}). All values are expressed in mg kg^{-1}, relative to feed with a moisture content of 12 %.

	Exp.1	Exp.2	Exp.3	x_{pt}	u_{char}	u_{hom}	$u(x_{pt})$	σ_{pt}	$u(x_{pt})/\sigma_{pt/}$
Cobalt mass fraction	1.156 ± 0.061	1.052 ± 0.106	0.946 ± 0.019	1.051	0.061	0.011 (1.1 %)	0.062 (5.9 %)	0.168 (16 %)	0.37

5.1 Assigned values

The assigned value (x_{pt}) of the mass fraction of total Co in the compound feed for rabbits (relative to a 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 three expert laboratories reported results:

- ALS Scandinavia AB (Luleå, Sweden);
- SCK-CEN, Studiecentrum voor Kernenergie (Mol, Belgium); and
- 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 were to be reported in dry mass. The EURL-FA Control converted afterwards these results to a feed with a moisture content of 12 % as required by Commission Implementing Regulation (EU) No 131/2014.

- ALS Scandinavia used inductively coupled plasma mass spectrometry (ICP-MS) after closed microwave digestion using nitric acid (HNO_3), hydrogen peroxide (H_2O_2) and hydrofluoric acid (HF) in sealed Teflon containers.

- SCK-CEN applied instrumental neutron activation analysis (k_0-NAA). Three samples of (approx. 410 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 three reference materials (SMELLS II, SMELS III and BCR 278) used for validation.

- JRC used inductively coupled plasma optical emission spectrometry (ICP-OES) after closed microwave digestion using 5.0/0.5/0.5 ml HNO_3/HF/H_2O_2. Sample intake: ca. 0.5 g. An external calibration curve was prepared, and Co was measured at 228.61 nm.

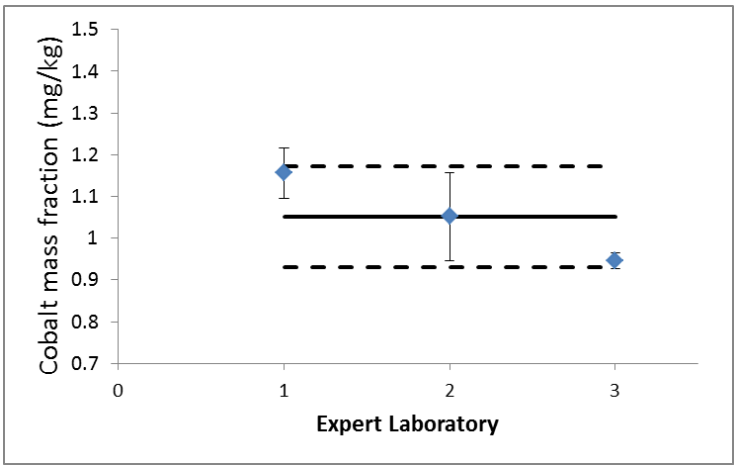


Figure 1:
Assigned value for cobalt in the compound feed for rabbit. Circles and error bars represent reported values by the expert laboratories ($x_i \pm 2u_i$). The solid line represents the assigned value (x_{pt}) while the dashed lines represent the assigned range ($x_{pt} \pm 2\, u(x_{pt})$)

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 characterization (u_{char}) with the standard uncertainty contributions from homogeneity (u_{hom}) and stability (u_{st}), in compliance with ISO 13528:2015 [4]:

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

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 ($\sigma_{pt,\%}$) of 16 % was calculated using the Horwitz equation [6].

6. Evaluation of results

6.1 Scores and evaluation criteria

The individual laboratory performance was expressed in terms of z and ζ scores according to ISO 13528:2015 [4]:

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

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

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 test assessment.

When $u(x_{pt}) > 0.3\ \sigma_{pt}$ (as in Table 1) ISO 13528:2015 advices 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 follows:

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

The interpretation of the z, z' and ζ scores is done as follows [4]:

|score| ≤ 2	satisfactory performance	(green in Annexes 6)
2 < |score| < 3	questionable performance	(yellow in Annexes 6)
|score| ≥ 3	unsatisfactory performance	(red in Annexes 6)

The z (or 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 estimation of the concentration, or of its measurement uncertainty, or both.

The standard measurement uncertainty of the laboratory $u(x_i)$ was obtained by dividing the reported expanded measurement uncertainty by the reported coverage factor, k. When no uncertainty was reported (see L18), it was set to zero ($u(x_i) = 0$). When k was not specified (see L02 and L11), the reported expanded measurement uncertainty was considered as the half-width of a rectangular distribution; $u(x_i)$ was then calculated by dividing this half-width by √3, as recommended by Eurachem [7].

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

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

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

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

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

6.2 Laboratory results and scorings

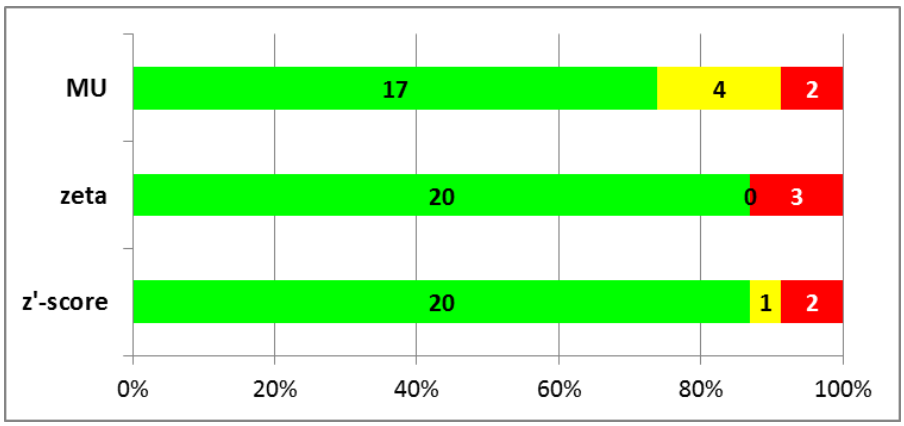
6.2.1 Performances

Annex 6 presents the results reported by the 23 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. **87% of the participants having reported results performed satisfactorily** according to both scores.

Most of the participants applied ICP-MS (52 %) and ICP-OES (26 %).. The experimental details are provided in Annex 7.

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



Note: Upon reception of the draft report, Lab 12 informed the EURL of having wrongly reported results obtained in a previous PT instead of the measurement results obtained for the current exercise. As the statement was coming after the deadline, the correct measurement results could not be taken into account in the evaluation in the frame and scope of the exercise. With the correct values, Lab 12 would have obtained satisfactory z' and zeta-scores.

6.2.2 Measurement uncertainties

Figure 2 shows that 74 % of the participants reported realistic measurement uncertainty estimates (case "a": $u(x_{pt}) \leq u(x_i) \leq \sigma_{pt}$).

The extremely high measurement uncertainties reported by L11 may be due to the wrong unit used (% instead of mg kg^{-1}).

L18 did not report a measurement uncertainty, while L04 and L23 reported a seemingly under-estimated relative expanded uncertainty of 4 %.

6.2.3 Compliance assessment

Commission Implementing Regulation (EU) No 131/2014 [2] (concerning the authorisation of several cobalt compounds as feed additives) sets a maximum content (also referred as maximum level, ML) of 1 mg kg^{-1} for (total) cobalt in complete feed with a moisture content of 12 %. This ML applies to the coated granulated cobalt(II) carbonate (feed additive 3b304) contained in the compound feed for rabbit distributed in the frame of this PT. The assigned value of 1.05 ± 0.12 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 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 relevant analyte;
    - to be compared to the relevant ML: $x_i - U_i$ > ML? (selecting the correct feed matrix (product intendent for animal feed));
- The laboratory justification (correct or incorrect).

Nineteen participants (out of 23 having made a compliance assessment) **correctly assessed the test item to be compliant** according to Commission Implementing Regulation (EU) No 131/2014. L09 assessed the test item to be non-compliant based on his over-estimated measurement result (2.55 mg kg^{-1}, z = 8.4). Two other laboratories assessed incorrectly the sample to be non-compliant while reporting satisfactory results but providing unclear/inappropriate justifications (L04: "*defined in EC no 1831/2003*"; L07: "*Cobalt deals with Regulation 1334/2003 (oligo-elements feed additives)*". Only L16 did not submit any compliance assessment

Since participants were not informed about the type of compound feed they received (the animal species (rabbit) was not disclosed in the accompanying letter), it is worth mentioning the wise comment made by L03: "*The use of Co in feedingstuffs is very limited. Only some animal species for which Co is allowed in feed with a maximal limit of 1 mg kg^{-1} (for ruminants, equidae, lagomorphs, rodents, herbivore reptiles and zoo mammals). For these species the test item would comply with the ML 1 mg kg^{-1} as set in Regulation 131/2014. For other animal species, it would not comply with the ML(Co) of 0 mg kg^{-1} of a non-authorised feed additive*".

Finally, there is a large discrepancy between the "added" content of $CoCO_3$ /Co (3b304) declared on the label (0.2 mg kg^{-1}, Table 2) according to the requirements set in Regulation (EC) No 767/2009 [9] and the assigned value (of 1.05 mg kg^{-1}). The permitted tolerances for the compositional labelling of feed material will be further investigated according to Commission Regulation (EU) No 939/2010 [10].

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 (12 laboratories); ICP-OES (6) and AAS (3). L04 and L16 did not report this information. Many analytical procedures (35 %) were single-laboratory validated; however, the following CEN standard methods were also mentioned: EN 15621 [11] (17 %), EN 15510 [12] (9 %) and EN 15763 [13], together with the VDLUFA monograph [14].

65 % of the results were not corrected for moisture content. Laboratories should have complied with the requirements set by the legislation "to report results referring to feed with 12 % moisture".

Several approaches were used to estimate measurement uncertainties (Table 2). Most of the laboratories derived their uncertainty estimates from their single-laboratory validation study. The majority of the NRLs (65 %) routinely report uncertainties for this type of analysis to their customers.

Most laboratories claimed to have determined a recovery factor by using a (certified) reference material (47 %) or by spiking (22 %) a known amount of the same analyte.

Half of the participants (12 out of 23) stated that they are accredited for the determination of cobalt in feed. 18 laboratories (78 %) acknowledged having participated to similar PTs in the past.

Most of the participants analyse ca. 50 "similar" samples per year (52 % from 1 to 49 samples, and 26 % from 50 to 250 samples). L18 (having reported a "questionable" result) stated to have limited experience with such type of analysis. L18 and L09 referred to a short digestion time (less than 2 min) at low temperatures (80 to 95 °C).

Annex 7 summarises the experimental details, the technique used and the limits of quantification (LOQ) for the determination of Co. Large discrepancies in reported LOQs are observed (from 0.0003 to 0.5 mg kg^{-1}) even among laboratories using the same technique.

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

Approach	N° of labs
According to ISO-GUM	4
From known uncertainty of a standard method	None
Derived from a single-laboratory validation study	15
Determined as standard deviation of replicate measurements	10
Estimation based on judgment	2
Derived from interlaboratory comparison data	3

7. Conclusions

The proficiency test EURL-FA 2017-2 was organised in 2017 to assess the analytical capabilities of the EU NRLs and OCLs on the determination of the mass fraction of total Co in a compound feed for rabbits.

The overall performance of the participants (20 out of 23, 87 %) was satisfactory. This confirms their analytical capabilities to enforce the Commission Implementing Regulation (EU) No 131/2014 setting maximum levels of cobalt in feedingstuffs for several animal species and categories.

Similarly, 19 participants (83 %) correctly assessed the test item to be compliant according to the Commission Implementing Regulation (EU) No 131/2014. Only two laboratories provided a contradictory conclusion with an unclear justification.

Seventeen participants (74 %) reported reasonable measurement uncertainty estimates.

Acknowledgements

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

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

Organisation	Country
AGES GmbH	Austria
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
Feedstuffs analysis laboratory in Thessaloniki	Greece
National Food Chain Safety Office (NEBIH)	Hungary
The State Laboratory	Ireland
Istituto Zooprofilattico Sperimentale, Torino	Italy
Istituto Zooprofilattico Sperimentale del Lazio e della Toscana - M. Aleandri	Italy
Institute of Food Safety, Animal Health and Environment	Latvia
RIKILT	Netherlands
National Institute of Nutrition and Seafood Research (NIFES)	Norway
National Research Institute of Animal Nutrition, National Laboratory for Feedingstuffs	Poland
The Central Control and Testing Institute in Agriculture (ÚKSÚP)	Slovakia
National Veterinary Institute	Slovenia
Laboratori Agroalimentari (GENCAT)	Spain
Laboratorio Arbitral Agroalimentario (MAPAMA)	Spain
National Food Agency (SLV)	Sweden
LGC Ltd	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] Commission Implementing Regulation (EU) No 131/2014 of 11 February 2014 amending Implementing Regulation (EU) No 601/2013 concerning the authorisation of cobalt(II) acetate tetrahydrate, cobalt(II) carbonate, cobalt(II) carbonate hydroxide (2:3) monohydrate, cobalt(II) sulphate heptahydrate and coated granulated cobalt(II) carbonate hydroxide (2:3) monohydrate as feed additives.

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

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

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

[6] M. Thompson, "Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing", Analyst. 125 (2000) 385–386. doi:10.1039/B000282H.

[7] S L R Ellison and A Williams (Eds). Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, Third edition, (2012) ISBN 978-0-948926-30-3. Available from www.eurachem.org

[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 of 13 July 2009 on the placing on the market and use of feed.

[10] Commission Regulation (EU) No 939/2010 of 20 October 2010 amending Annex IV to Regulation (EC) No 767/2009 on permitted tolerances for the compositional labelling of feed materials or compound feed as referred to in Article 11(5).

[11] EN 15621:2012, "Animal feeding stuffs - Determination of calcium, sodium, phosphorus, magnesium, potassium, sulphur, iron, zinc, copper, manganese and cobalt after pressure digestion by ICP-AES", European Committee for Standardisation (CEN)

[12] EN 15510: 2007, "Animal feeding stuffs - Determination of calcium, sodium, phosphorus, magnesium, potassium, iron, zinc, copper, manganese, cobalt, molybdenum, arsenic, lead and cadmium by ICP-AES", European Committee for Standardisation (CEN)

[13] EN 15763:2009, "Foodstuffs - Determination of trace elements - Determination of arsenic, cadmium, mercury and lead in foodstuffs by inductively coupled plasma mass spectrometry (ICP-MS) after pressure digestion", European Committee for Standardisation (CEN)

[14] VDLUFA-Methodenbuch III (2017), "Die chemische Untersuchung von Futtermitteln“, Methode 17.9.1 "As, Be, Bi, Cd, Co, Cr, Cu, Mo, Ni, Pb, Sb, Tl, U, V in Pflanzen sowie in Grund- und Mischfuttern".

List of abbreviations

DG SANTE	Directorate General for Health and Food Safety
FAAS	Flame Atomic Absorption Spectrometry
GF-AAS	Graphite Furnace – Atomic Absorption Spectrometry
GUM	Guide for the Expression of Uncertainty in Measurement
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
JRC	Joint Research Centre
EURL	European Union Reference Laboratory
EURL-FA	EURL for Feed Additives
EURL-FA Control	EURL for Feed Additives Control
EURL-FA Authorisation	EURL for Feed Additives Authorisation
EURL-HM	EURL for Heavy Metals in feed and food
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PT	Proficiency Test
σ_{pt}	Standard deviation for proficiency assessment

Annex 1: Invitation letter

Dr Ursula Vincent
Project Leader European Reference Laboratory for Feed Additives (Control)
(EURL-FA Control)
Joint Research Centre

03 Mai 2017

Call for participation in an inter-comparison study for the determination of the total mass fraction of cobalt in feed at authorised level

1. Introduction

Feed additives are authorised within the European Union according to Regulation (EC) No 1831/2003 requiring various criteria to be fulfilled including the need of providing suitable methods of analysis for official control in feedingstuffs. Coated granulated cobalt(II) carbonate (3b304) is an authorised feed additive containing cobalt II and is classified in the category 3 (nutritional additives) and functional group b (compounds of trace elements). In addition, authorisation of this substance includes a maximum content of Cobalt of 1 mg kg^{-1} in complete feedingstuffs as specified in Commission Implementing Regulation (EU) No 131/2014.

Enforcing the legislation and performing the compulsory monitoring requires the availability of reliable analytical methods. In a continuous effort to be updated on the reliability of analytical results delivered by laboratories in charge of ensuring the official control of additives in feed, the European Commission's Directorate-General for Health and Food Safety requested the EURL-FA Control to organise an inter-comparison study for the determination of cobalt in feed in 2017. This letter therefore constitutes the formal call for participation to this first proficiency testing exercise targeting the total mass fraction of cobalt in animal feed.

As National Reference Laboratory representative, you are kindly reminded that according to Regulation (EC) No 882/2004 it is your duty 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.

Your participation is free of charge.

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.

2. The organising team

The study will be conducted by the European Union Reference Laboratory for Feed Additives Control (EURL-FA Control), hosted at the European's Commission Joint Research Centre in Geel, Belgium.

3. Objective

The major objective of this study is to assess the proficiency of the participating laboratories to correctly determine cobalt in feed samples at levels authorised in the European legislation. This aim will be achieved by conducting an inter-comparison study in which the laboratories analyse feed samples applying the analytical method(s) referred to in the authorisation regulation and reporting the results to the organiser of the study. 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.

4. Test material

Samples are prepared containing typical compound feed fortified with Coated granulated cobalt(II) carbonate (3b304) (Regulation (EC) No 1831/2003, Community Register of Feed Additives pursuant to Regulation (EC) No 1831/2003) at authorised concentration level. Each laboratory will have to quantitatively analyse 1 sample and to submit the results in terms of the mass fraction of total Cobalt in the feed sample to the coordinator of the study.

Prior to sending out the samples to the participants the organising team will have demonstrated sufficient homogeneity of the test material by analysing randomly taken sub-samples.

5. General outline of the exercise

The participants are requested to report the results of the analyses together with the information about the analytical method applied. Each laboratory will be assigned a unique code and only the organiser of the study (EURL-FA Control) knows the key to this code. The EURL-FA Control will send out a report on the outcome of the study containing information about the score of the laboratories; the personal laboratory keys will be individually communicated to each participant.

Appropriate statistical tool for data evaluation will be used to investigate the proficiency of the laboratories. Statistical assessment of the proficiency of laboratories will be evaluated by calculating an individual dimensionless Z-score calculated according to ISO 13528.

$$z = \frac{(x - x_a)}{\sigma_p} \qquad \text{Eq. 1}$$

Where x = the value reported by the participant

x_a = the assigned value

σ_p = the target standard deviation

The proficiency test is scheduled for the period **mid June 2017 – 25 August 2017**.

The exercise will be completed by the organisation of a workshop opened to all participants to the study and to all NRLs for Feed additives. The target date for the workshop is 22-23 November 2017. 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.

6. Expression of interest

National Reference Laboratories for Feed Additives (control) defined according to Regulation (EC) No 882/2004 **shall** participate in the proficiency exercise organised by the EURL-FA Control or mandate a representative laboratory to participate on their behalf, depending on their expertise. Other official control laboratories may also participate in the proficiency exercise if places are still available. The total number of participating laboratories may not exceed 40.

All laboratories are kindly asked to register for participation **copying** the following URL in your navigator: https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=1744

The deadline for registration is **26 May 2017**. Please kindly fill in the form comprehensively. These details will be used for the dispatch of the materials and for any further correspondence.

Best regards,

Dr Ursula VINCENT
Study coordinator, EURL-FA Control
Functional mailbox: JRC-EURL-FEED-ADDITIVES-CONTROL@ec.europa.eu

Annex 2: Accompanying letter

21 June 2017

Proficiency Test exercise 2017

Determination of the total mass fraction of cobalt in feed at authorised level

INSTRUCTIONS

The materials you received should be analysed in routine conditions, i.e. utilizing your usual method for control. Your method for official control should be retrieved from the authorising regulation.
Please check the content of the package, fill in and send back the 'List of Contents of the Package' form as specified below.

The test materials have been grinded and homogenised; no further pre-treatment is necessary before the analysis.
Analyse the test material you received for the presence, the content and the correct labelling of the authorised cobalt. The content should be reported as the total mass fraction of the detected cobalt in **mg kg^{-1}** of feed given with 3 decimals. When cobalt is present, the mass fraction(s) are as defined in the Regulation (EC) No 1831/2003 for cobalt authorized as feed additive(s).

Results reporting
The reporting of results will be opened as of 26.06.2017. To report your results, please copy the URL below into your Internet navigator:
https://web.jrc.ec.europa.eu/ilcReportingWeb
Type in your participant key and login.
Do not forget to submit your results when your encoding is complete.

Method details
Reporting your results shall be accompanied by filling in the method questionnaire. Please fill in the method questionnaire when reporting your results via https://web.jrc.ec.europa.eu/ilcReportingWeb
Do not forget to submit your questionnaire when your encoding is complete.

Please note that the deadline for reporting the results is **29 August 2017**, the on-line system will be closed after this date.

Please remember that the major objective of this study is to assess the proficiency of the participating laboratories to correctly determine the total mass fraction of cobalt potentially present in feed samples at levels authorised in the European legislation and to report results in the specified units and accompanied with a compliance statement on the analysed samples within a defined time frame.

Any deviation as regards the non-respect of the deadline and/or the specified units for reporting will lead to the exclusion of your results from the statistical evaluation.

In Autumn 2017, a draft report including the statistical evaluation of all valid participant results will be issued and distributed to all reporting participants. The report will be confidential. The list of participating laboratories will be included but any result will be strictly linked only to the laboratory code. Each participant will be able to retrieve its results and calculated z-score using its individual confidential laboratory code communicated on the list of contents of the package form included in this letter.

Finally, a concluding workshop opened to all participants to the study will be organised to close the exercise in November 2017 (target date: 22-23.11.2017, 1 day). Participation to the PT and to the workshop is free of charge. However, travel and accommodation costs will only be reimbursed for one representative of each National reference Laboratory for Feed Additives defined according to Regulation (EC) No 882/2004 and Regulation (EC) No 1831/2003.

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

If you have any question, please contact the EURL-FA (Control), JRC-EURL-FEED-ADDITIVES-CONTROL@ec.europa.eu.

Kind regards,

Dr Ursula VINCENT (Proficiency Test Coordinator)
EURL for Feed Additives Control

Annex 3: Confirmation of receipt form

LIST OF CONTENTS OF THE PACKAGE[1]

ACKNOWLEDGEMENT OF RECEPTION

Dear «Title» «Name»,

please find below the list of contents for the proficiency test exercise related to the determination of the total mass fraction of cobalt in feed at authorised levels, organised by the EURL-Feed Additives (Control), on behalf of DG SANTE (European Commission).
Please check that the sample codes of the samples you received correspond to those declared on this list. The samples should **be stored at -18°C / -20°C** upon reception and until one hour before the analysis, when they are left at room temperature until analysis.

Your laboratory code and your participant key are given below. The laboratory code will be applied to your laboratory for the whole PT exercise.
Use the participant key to login to report your results.

Your laboratory code is: **«Lab_Code»**

Your participant key is: **«PartKey»**

Number of samples in the package: 1

Sample code	Present Y/N
«MAT»	

Samples received on (dd/mm/yyyy) :
Content checked on (dd/mm/yyyy) :
Comments (if applicable) :

Date: Signature:

[1] Form to fill in, sign and send back to the EURL-Feed additives (control) by electronic mail (JRC-EURL-FEED-ADDITIVES-CONTROL@ec.europa.eu) or by fax (+32 14 571 787)

3

Annex 4: Questionnaire

Milc questionnaire

Comparison for EURL-FAC 2017-02

This questionnaire is meant to collect additional information about your laboratory and experimental details about your analytical method(s). Your answers will be used in the evaluation of the proficiency test EURL-FAC 2017-02. Please enter the information related to the method used for the identification and quantification of the cobalt. Please do so comprehensively, in order to allow appropriate evaluation and relevant discussion of the results. Please use the "comments" text box (Q 15.) for any additional information you wish to provide and/or to specify an "other" answer when relevant. Please be accurate. In addition, please send to us the chromatograms from all analyses either by e-mail jrc-eurl-feed-additives-control@ec.europa.eu or by normal mail. The information collected will be kept confidential and only used for discussion and exchange during the final workshop.

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 Directive 2002/32/EC?**

- ○ a) Yes
- ○ b) No

2.1. If "Not compliant" specify why. *

3. **Did you correct your result for moisture content?**

- ○ a) Yes
- ○ b) No

4. **Did you correct your result for recovery?**

- ○ a) Yes
- ○ b) No

4.1. What was your recovery correction? *

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

- ○ a) Using a spike
- ○ b) Using a CRM
- ○ c) Other

5.1. If "Other" please specify: *

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

See table **Recovery and limit of quantification (LOQ)** at bottom

7. **Which standard methods did you apply for the determination of the analyte?**

8. **Which Reference Materials did you use for (instrumental) calibration and/or (method) validation**

See table **Calibration and Validation** at bottom

9. **Are you accredited for Cobalt determination in feed?**

- ○ a) Yes
- ○ b) No

10. **How many analyses of this type does your laboratory perform on a yearly basis?**

- ○ 1. Never
- ○ 2. 0-49
- ○ 3. 50-249
- ○ 4. 250-999
- ○ 5. 1000+

11. **Please provide the following information:**

See table **Experimental details** at bottom

12. **Do you ususally provide an uncertainty statement to your customers?**

- ○ a) Yes
- ○ b) No

13. **How did you estimate your measurement uncertainties? (multiple choice)**

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

14. **Did your laboratory participate in other PTs for this type of analysis?**

- ○ a) Yes
- ○ b) No

15. **Do you have any comments? Let us know, please.**

Calibration and Validation

Questions/Response table	*Cobalt (Co)*
a) Calibration	
b) Validation	

Experimental details

Questions/Response table	*Cobalt (Co)*
a) Digestion Type	
b) Digestion Mixture	
c) Digestion time (min)	
d) Digestion temperature (oC)	

Recovery and limit of quantification (LOQ)

Questions/Response table	*Cobalt (Co)*
Recovery (%)	

Questions/Response table	*Cobalt (Co)*
LOQ (mg/kg)	

Annex 5: Homogeneity results

Bottle #	Replicate1	Replicate 2
1	0.99	1.02
2	1.04	1.02
3	1.00	1.06
4	0.97	0.99
5	1.01	1.00
6	1.04	0.99
7	1.02	1.04
8	0.99	1.02
9	1.03	1.00
10	1.00	1.01

mean	1.012
σ_{pt}	0.167
0.3 * σ_{pt}	0.050
s_x	0.016
s_w	0.023
s_s	0
$s_s \leq 0.3 * \sigma_{pt}$	passed
u_{hom} (= u*)	**0.011** (1.1 %)

(all values in mg kg^{-1}, not corrected for moisture content)

Where: σ_{pt} is the standard deviation for the PT assessment,
s_x is the standard deviation of the sample averages,
s_w is the within-sample standard deviation,
s_s is the between-sample standard deviation

Annex 6: Results for Cobalt

Assigned values: x_{pt} = 1.05; $U(x_{pt})$ = 0.12 (k = 2); and σ_{pt} = 0.17 ; all values in mg kg^{-1}; relative to a feed with a moisture content of 12 %.

Lab Code	x_i	±	k	technique	u_i	z'-score (#)	Zeta (#)	unc.(@)
L01	1.065	0.266	2	ICP-OES	0.133	0.1	0.1	a
L02	0.93	0.11	1.73*	GF-AAS	0.064	-0.7	-1.4	a
L03	0.975	0.195	2	ICP-MS	0.098	-0.4	-0.7	a
L04	1.0466	0.0468	2	?	0.023	0.0	-0.1	b
L05	1.06	0.26	2	ICP-OES	0.130	0.0	0.1	a
L06	1.123	0.225	2	ICP-OES	0.113	0.4	0.6	a
L07	0.944	0.245	2	ICP-OES	0.123	-0.6	-0.8	a
L08	1.142	0.15	2	ICP-MS	0.075	0.5	0.9	a
L09	2.551	0.182	95 (%?)	F-AAS	0.091	8.4	13.6	a
L10	0.95	0.19	2	ICP-OES	0.095	-0.6	-0.9	a
L11	1.069	25 (%?)	1.73*	ICP-MS	14.451	0.1	0.0	c
L12	0.37	0.16	2	ICP-MS	0.080	-3.8	-6.7	a
L13	1.045	0.33	2	ICP-MS	0.165	0.0	0.0	a
L14	0.984	0.147	2	ICP-MS	0.074	-0.4	-0.7	a
L15	1.011	0.081	2	ICP-MS	0.041	-0.2	-0.5	b
L16	1.1	0.44	2	?	0.220	0.3	0.2	c
L17	1.05	0.29	2	ICP-MS	0.145	0.0	0.0	a
L18	0.671			GF-AAS	0.000	-2.1	-6.2	b
L19	1.01	0.13	2	ICP-MS	0.065	-0.2	-0.5	a
L20	1.08	0.183	2	ICP-MS	0.092	0.2	0.3	a
L21	0.958	0.308	2	ICP-OES	0.154	-0.5	-0.6	a
L22	0.958	0.316	2	ICP-MS	0.158	-0.5	-0.5	a
L23	1.05	0.05	2	ICP-MS	0.025	0.0	0.0	b

(*) √3 is set by the ILC coordinator when no coverage factor k is reported. The reported uncertainty was assumed to have a rectangular distribution with k = √3,

(#) performance: satisfactory, questionable, unsatisfactory,

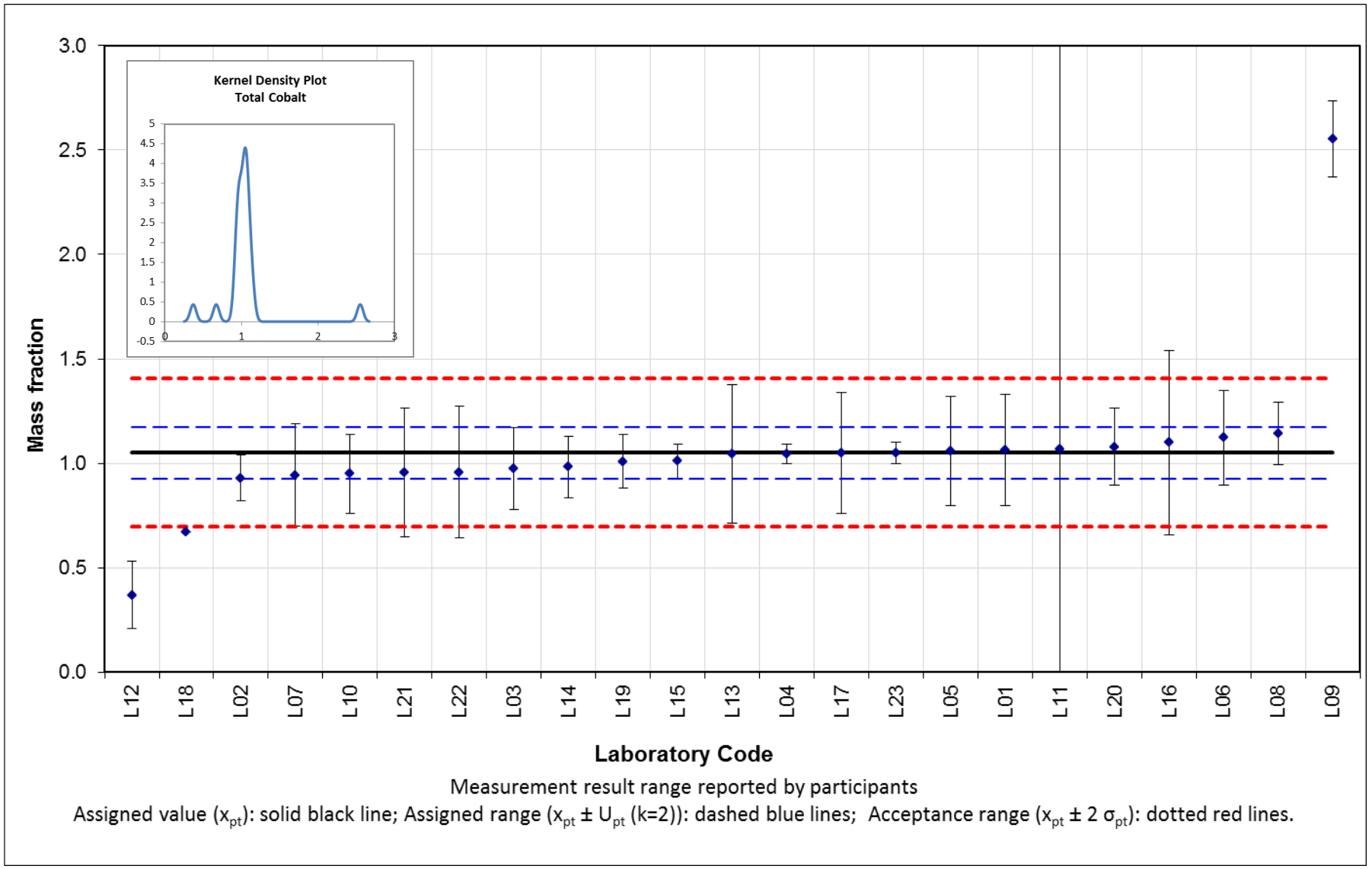
(@) a: $u(x_{pt}) \leq u_i \leq \sigma_{pt}$; b: $u_i < u(x_{pt})$; and c: $u_i > \sigma_{pt}$

Remarks:

- L04 and L16 did not indicate their measurement technique
- L02 and L11 did not indicate the coverage factor used (see *)
- L09 reported a confidence interval of 95 %, instead of a coverage factor of 2
- L18 did not report the measurement uncertainty
- L11 probably reported their expanded uncertainty in %, instead of mg kg^{-1}

Total cobalt in compound feed for rabbit

x_{pt} = 1.05; $U(x_{pt})$ = 0.12 (k=2); σ_{pt} = 0.17 (mg kg^{-1})



Measurement result range 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

Laboratories with "questionable" or "unsatisfactory" performances (according to z scores) are highlighted in yellow or red, respectively

LCode	Q1.Type	Q2: compliant ~~(2002/32/EC)~~ (131/2014)?	Q3. Moisture correction?	Q4. Recovery correction?	Q5. Recovery - How?	Q5.1: Recovery (%)	Q6. LOQ (mg kg^{-1})	Q7. Standard methods used?	Q8a. CRM used for Calibration	Q8b. CRM used for Validation
L01	NRL	Yes	Yes	No		100	0.2	EN 15621	None	NIST
L02	NRL	Yes	Yes	No	Other		0.1		Co std 1000ppm	
L03	NRL	Yes (*)	Yes	No	RM	95-105	0.05	prEN 17053:2017, EN 15621 (+ICP-MS)	Astasol,ANALYTIKA	PT ALVA (AUS),IRM UKZUZ
L04	NRL	No (*)	Yes	Yes	Spike	102	0.1	EN 15621	std.curve	TORT-3
L05	NRL	Yes	No	No			0.3	EN 15621	Perkin-Elmer multistandard	sheep feed CEN TC 327/WG1
L06	NRL	Yes	No	Yes	Spike	89.4	0.1			IAG Mixed feed 2013
L07	NRL	No (*)	Yes	No	n.a		0.05	EN 15510	multi-elementary standard	Premix BIPEA
L08	NRL	Yes (*)	No	No		102	0.013	VDLUFA III, 17.9.1		
L09	NRL	No (*)	No	Yes	Int.Std	95	0.5	in-house	Merck Cobalt standard sol	internal sample
L10	NRL	Yes	No	Yes	CRM	85	0.1	EN 15510	CPA Chem	
L11	NRL	Yes	No	No	n.a		0.0025	in-house		NIST 1573a
L12	NRL	Yes	Yes	Yes	CRM	99.2	0.01	in-house		Tomato Leaves 1573A
L13	OCL	Yes (*)	No	No	Spike	96-106	0.05	in-house	C. Erba cod.504352-1ppm	
L14	NRL	Yes	No	No	CRM	100	0.033		EURL-HM-25	EURL-HM-25
L15	NRL	Yes	No	No	CRM	101	0.006	in-house		BCR 482 lichen
L16	?	?	No	No			0.02			
L17	NRL	Yes	No	Yes	CRM	101	0.1	in-house	SCP33MS	LGC71162
L18	NRL	Yes	No	Yes	CRM			in-house	Co AA standard	
L19	NRL	Yes	Yes	Yes	CRM	86	0.008	n.a.	Inorg Ventures VAR-TS-MS	SRM 1570a
L20	NRL	Yes	No	No	Spike	99	0.1			NIST WAtER
L21	OCL	Yes	No	No	Spike	75-104	0.4	in-house based CEN/TS 15621	Standards ICP	Standards ICP
L22	NRL	Yes	Yes	No	RM	95	0.0061	EN 15763:2009	certified stock solution	CRMs
L23	NRL	Yes	No	No	CRM	94	0.0003			

LCode	Q9.Accredited for Cobalt in feed?	Q10.How many samples/year analysed (of this type)?	Q11a. Digestion Type	Q11b. Digestion Mixture	Q11c. Digestion time (min)	Q11d. Digestion temperature (°C)	Q12.MU provided regularly to customers?	Q13.MU estimation - How? (multiple choice)	Q14.PT participation for such analyses?
L01	Yes	50-249	MW	HNO_3+HCl	60	230	Yes	a),c) d) e)	Yes
L02	No	0-49	MW	HNO_3+H_2O_2	90	200	No	d)	No
L03	Yes	0-49	MW	HNO_3	20	210	Yes	a), c), d)	Yes
L04	No	Never	pressure digestion	HNO_3	45	240	Yes	c), d)	No
L05	Yes	50-249	MW	HNO_3+H_2O_2	40	230	Yes	g)	Yes
L06	Yes	50-249	MW closed	HNO_3+H_2O_2 (5ml+2ml)	32	100-200	Yes	c), e)	Yes
L07	Yes	0-49	Ashing	?	240	450	No	c)	Yes
L08	No	0-49	MW	HNO_3+H_2O_2 (6ml+1ml)	20	210	No	d)	Yes
L09	No	50-249	wet digestion	HCl (3N)	1	95	Yes	d)	Yes
L10	Yes	0-49	MW	HNO_3+H_2O_2	20	200	Yes	d)	Yes
L11	Yes	50-249	MW	HNO_3+H_2O_2+HCl	20	120	Yes	c)	Yes
L12	No	0-49	MW	HNO_3+H_2O_2	28	200	Yes	a) ,c) ,d)	Yes
L13	No	Never	MW	HNO_3+H_2O_2 (8ml+1ml)	20	190	No	g)	Yes
L14	No	0-49	MW closed	HNO_3+H_2O_2	30	180	Yes	a) ,f), g)	Yes
L15	No	Never	MW	HNO_3	90	210	No	c)	No
L16	No	250-999	wet digestion	HNO_3	30	260	No	c)	Yes
L17	Yes	0-49	MW	HNO_3+H_2O_2 (6ml+0.5ml)	30	200	Yes	c), f)	Yes
L18	No	Never	acid digestion	HNO_3	1.8	80-2300(?	No	g)	No
L19	No	0-49	MW	HNO_3+H_2O_2	20	200	Yes	c)	No
L20	Yes	50-249	?	HNO_3+H_2O_2	40	200	Yes	c)	Yes
L21	Yes	0-49	MW	HNO_3+H_2O_2 (3ml+0.5ml)	30' predigestion +90' MW	220	Yes	c), d)	Yes
L22	Yes	0-49	MW	HNO_3+HCl (6ml+1ml)	25	220	Yes	a), c), f)	Yes
L23	Yes	0-49	MW	HNO_3 (14%)	25	210	No	c), d)	Yes

MW: microwave;
Measurement uncertainty (MU) derived from: (a) ISO GUM; (b) reproducibility of a standard method; (c) in-house validation; (d) replicate analyses; (e) judgement; (f) interlaboratory comparison; (g) other

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doi:10.2760/9785

ISBN 978-92-79-77624-3