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NORMCONSTRUCT: Results of the collaborative assessment experiment for the validation of the precision of CEN/TS 17216

Paepen, J., Van Ammel, R., Hult, M., Marissens, G., Stroh, H., Emteborg, H., Seghers, J.

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Joint Research Centre

Contact information
Jan Paepen
Joint Research Centre
Retieseweg 111
2440 Geel
Belgium
jan.paepen@ec.europa.eu

EU Science Hub
https://joint-research-centre.ec.europa.eu

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Contents

Abstract

Among the non-energy extractive industries in the EU, the construction minerals sector is the largest one. These minerals may contain enhanced levels of natural radioactivity. CEN/TS 17216 aims at providing a reference method for the determination of the activity of the radionuclides Ra-226, Th-232 and K-40 in construction products using semiconductor gamma-ray spectrometry. The method is developed by CEN/TC 351/WG 3 "Construction products: Assessment of release of dangerous substances – Radiation from construction products" under Commission mandate M/366 which provides for the preparation of horizontal measurement/test methods in support of performance assessment of construction products (CE marking in the frame of the Construction Products Regulation, CPR).

The precision of the method was evaluated with six construction products, analysed by 15 laboratories in a collaborative assessment. The reproducibility relative standard deviation for Ra-226 is 12.6 (5.4) %, for Th-232 it is 7.7 (3.2) % and for K-40 it is 8.3 (5.0) %.

The bias of the method was re-assessed. The relative bias for Ra-226 is -7.5 (5.8) %, for Th-232 it is -1.8 (3.8) %, for K-40 it is 3.2 (2.4) %. All biases are insignificant at a 95 % confidence level. The bias for Ra-226 is the highest, but still insignificant caused by the higher reproducibility for Ra-226.

The values for precision and bias are shown with their standard uncertainty in parentheses and apply within a limited validity range, specified in the report.

Box 1. Disclaimer

The aim of the study is to validate the precision of the method in EN 17216 and not to evaluate the performance of the laboratories. Therefore, the results presented in this report shall not be used to demonstrate the competence of the individual participants.

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Special acknowledgement goes to the participating laboratories who invested a considerable effort in studying, applying and commenting on the draft standard which is the subject of this validation.

Authors

— Paepen, J.

— Van Ammel, R.

— Hult, M.

— Marissens, G.

— Stroh, H.

— Emteborg, H.

— Seghers, J.

1 Introduction

Box 2. Construction minerals

Among the non-energy extractive industries in the EU, the construction minerals sector is the largest one. It has the highest tonnage of extracted minerals, the greatest number of companies and employees, and the largest turnover. Typical construction minerals are aggregates (sand, gravel, and crushed natural stone), various brick clays, gypsum, and natural ornamental or dimension stone [1].

By means of Administrative Arrangement N° SI2.834008 - JRC.35292 [2], the European Commission's Joint Research Centre (JRC) was tasked by the Directorate-General for Internal Market, Industry, Entrepreneurship and SMEs (DG GROW) to organise a collaborative assessment experiment for the validation of the precision of CEN/TS 17216 "Construction products: Assessment of release of dangerous substances – Determination of activity concentrations of radium-226, thorium-232 and potassium-40 in construction products using semiconductor gamma-ray spectrometry". The technical specification CEN/TS 17216 is developed by technical group 31 of working group 3 of technical committee 351 of the European Committee for Standardization in the frame of the European Commission's Mandate M/366 from DG GROW to CEN. The CEN working group CEN/TC 351/WG 3 deals with the assessment of release of dangerous substances from construction products and, in particular, radiation from construction products. The Dutch normalisation institute NEN holds the secretariat of CEN/TC 351 "Construction products – Assessment of release of dangerous substances".

Construction products may contain minerals with enhanced levels of natural radioactivity. In support of performance assessment of construction products (CE marking), CEN/TS 17216 aims at providing a reference method for the determination of the activity of Ra-226, Th-232 and K-40 in construction products. The described method shall be validated for robustness and precision before it can advance to a European standard (EN). The robustness of the method was demonstrated already by Główny Instytut Górnictwa (GIG), Poland [3]. The precision is to be validated by a collaborative assessment in which several laboratories take part and follow the method rigorously. Clause 3.2 of ISO 5725-1 defines a collaborative assessment experiment as an interlaboratory experiment in which the performance of each laboratory is assessed using the same standard measurement method on identical material. The purpose of the study discussed in this report is to validate the precision of draft standard WD EN 17216 by means of such a collaborative assessment. The purpose is not to evaluate the performance of each laboratory.

A further developed version of CEN/TS 17216 is working draft WD EN 17216 [4], which is the subject of the validation discussed in this report. The participants of the collaborative exercise received WD EN 17216 and a set of six materials (construction products, or derived therefrom). The materials were screened by the JRC and deemed fit for purpose [5]. The participants of the collaborative exercise were asked to adhere to the method as much as possible for the analysis of the materials.

The Administrative Arrangement [2] specifies the tests that JRC will carry out in support of the validation:

- Task 1: Study and provide comments to TC 351 on the method described in CEN/TS 17216.
- Task 2: Make a minimum selection of (at least five) materials required to validate the method and covering as wide as possible the scope of CEN/TS 17216.
- Task 3: Perform a screening of the materials and assess their chemical composition, density and activity concentration levels for Ra-226, Th-232 and K-40. Verify if the homogeneity of the materials is sufficient after processing as specified in CEN/TS 17216.
- Task 4: Testing the radon-tightness of containers used for the in-house tests required to ensure that samples can be considered as being identical.
- Task 5: JRC will determine the reference values for the selected materials because there may be issues with the trueness of the method when applied to heterogeneous materials. This task requires the development of calibration standards and the assessment of the additional uncertainty component in the reproducibility assessment caused by the inhomogeneity.
- Task 6: Design and circulate a questionnaire for the participants, with the input from the TG, to verify that they have the necessary equipment and competence that is fit-for-purpose. Process the answers.
- Task 7: Draft instructions for the participants of the collaborative assessment exercise.
- Task 8: Prepare and distribute the samples to the laboratories.

— Task 9: Collect and analyse the data following the principles of relevant ISO/CEN standards and guidelines.

— Task 10: Draft the report(s) about the results of the validation study.

This report is the second of two deliverables agreed upon in the Administrative Arrangement [2]. It discusses the collaborative assessment and presents the determination of the repeatability and reproducibility of the method. In addition, also the bias of the method was determined. Refer to the first deliverable [5] for the outcome of tasks 2, 3, 4 and 5.

2 Design of the validation study

2.1 Invitation and selection of participants

15 laboratories were selected

On 05/07/2021, the secretariat of CEN/TC 351 invited the members of WG 3 "Radiation from construction products" to participate in the collaborative trial on the draft EN 17216 [6]. The invitation was shared in the plenary of CEN/TC 351 and its WG 3, in the EC subgroup on dangerous substances (SGDS) and in CEN/TC 430 "Nuclear energy, nuclear technologies and radiological protection". The invitees were encouraged to further distribute the call in their network.

21 laboratories expressed their interest and answered a short survey (See Annex 1) which records their competence and equipment, necessary for applying the method. Participants should not exclusively consist of reference laboratories (Clause 6.3.1 of ISO 5725-1 [7]). Therefore, a selection of 15 laboratories was made, including some that claim not to have experience in measurements of naturally occurring radionuclides in construction products using semiconductor gamma-ray spectrometric detectors and including some that claim not to have the necessary equipment for processing the samples. Annex 2 lists the participants in random order.

2.2 Selection, screening and preparation of materials

6 materials from 5 construction products

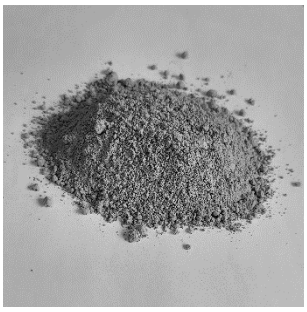
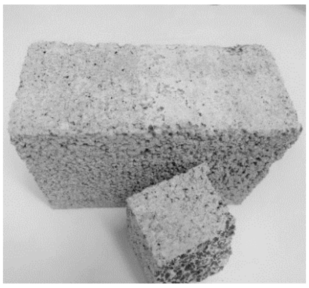
4 did not require further processing

1 required milling to < 2 mm

1 required breaking in pieces and milling to < 2 mm

Five construction products were selected and from these, six materials (named NORM1 to NORM6) were produced for the validation study. The selection, screening and processing of the materials for the study is discussed in a separate report [5]. Their origin and processing is summarised in Table 1. Their chemical composition, density and activity concentration levels for Ra-226, Th-232 and K-40 have been determined. The report demonstrates that the selected materials are sufficiently homogeneous and stable for their use in the validation study [5].

Table 1. Materials selected for the validations study.

Name	Base material	Description, material origin and processing by the JRC
NORM1		Cement — The base material was provided by a member of WG 3 and originates from a single production batch. — The base material is a fine powder with particles less than 2 mm. The material did not require milling. — The material was mixed and homogenised and then distributed over sample containers.
NORM2 (pieces) NORM3 (powder)		Expanded clay blocks — The blocks were provided by a member of WG 3 and originate from a single production batch. — A series of blocks was cleaved in pieces and sent to the participating laboratories as NORM2. — Another series of blocks was milled and sieved to a particle size below 250 µm, mixed, homogenised and distributed over sample containers. The pulverised material is NORM3.
NORM4		Plaster — Bags of plaster were purchased by JRC-Geel from a local shop. The bags have the same production batch number. — The base material is a fine powder with particles less than 2 mm. The material did not require milling. — The content of the bags was mixed and homogenised and distributed over sample containers.
NORM5		Aggregates — Milled aggregates were provided by a member of WG 3. — The material contains grains with a size larger than 2 mm. — The material was not further milled. — The material was mixed and homogenised and distributed over sample containers.
NORM6		Milled concrete — Cubes of $(15\ \text{cm})^3$ were provided by a WG 3 member. — A selection of cubes were milled and sieved to a particle size below 250 µm. — The material was mixed and homogenised and distributed over sample containers.

2.3 Distribution of materials

All laboratories received two containers (replicates) of each of the materials NORM1, NORM3, NORM4, NORM5 and NORM6, and two pieces of material NORM2. The sample containers and pieces were sent to the participants on 10/03/2023. The tracking numbers disclose that the courier delivered the samples on 14/03/2023 the latest. The laboratories confirm that they received the samples in good condition. One laboratory requested double the amount. The additional materials were sent in a separate shipment.

Together with the samples, an accompanying letter was sent to the participants (See Annex 3). The laboratories were asked to analyse the two samples of identical materials in repeatability conditions thereby following the draft standard WD EN 17216 [4] as close as possible and to report deviations from the method using a survey.

2.4 Reporting of results

The participants reported their results via the JRC's ILC reporting web application. Each participant received a personalised password key that provides access to the application.

The results of the determination of K-40, Ra-226 and Th-232 in each of the two replicates of each of the materials had to be reported, yielding a total of maximum 36 measurands or test properties to be reported by each participant analysing all samples.

results are expressed as massic activity in dry mass [Bg/kg], on the reference date 01/03/2023

For each test property, the participants had to report:

— The result of the analysis: the massic activity in dry mass, expressed in Bq/kg on the reference date of 01/03/2023;

— The uncertainty on the result and coverage factor k.

2.5 Reporting questionnaire

In principle, a method can only be validated when its normative sections are meticulously followed by the laboratories participating in the validation study. In practice, however, several reasons may exits that prevents the strict application of the draft. For that reason, the laboratories were asked to follow the draft standard as close as possible and to specify deviations from the method in the questionnaire (See Annex 4) included in the JRC's ILC reporting web application.

In addition to general questions about their organisation, the questionnaire asked the participants for technical details related to the application of the method and to the quality of the analysis. This includes the date of measurement, operator, uncertainty budget, test specimen mass and results of the dry matter determination (mass of the test portion and correction factor for dry mass).

The answers to the questionnaire are used to verify where the participants deviated from the method and to assess the impact on the reported results.

2.6 Data treatment

The link between the identity of any laboratory and the reported data is confidential. Throughout the validation study, the participants were identified with a code (L01 to L15). This code is not related with the order in which the participants are listed in Annex 2.

3 Evaluation of results

3.1 Methodology and structure of the report

For the processing and evaluation of the results, the following methodology was applied:

- The reported data were carefully studied. The participants were asked to clarify or to confirm doubtful data and missing information (See Section 3.2);
- The results of participants that did not use the correct type of gamma-ray spectrometer as specified in the method were rejected (See Section 3.3);
- The reported uncertainties were recalculated to standard uncertainties and the precision of the method was estimated applying ISO 5715-2 [8] (See Section 4);
- ISO 5715-4 [9] was followed to estimate of the bias of the method (See Section 5);
- The impact on the precision and bias of not following normative requirements of the method was assessed and is discussed in Section 6;
- The impact of other aspects was assessed and is discussed in Section 7;
- Section 8 includes comments on the draft WD EN 17216;
- Finally, Section 9 formulates conclusions.

Annex 1 contains the call for expression of interest, Annex 2 lists the participants of the collaborative assessment, Annex 3 provides instructions for the participants and Annex 4 is the reporting questionnaire. In this report, the questions in the reporting questionnaire are referred to with a Q, for example: Q 1.4 refers to question 1.4 of the questionnaire.

3.2 Scrutiny of reported values and uncertainties

The reported results and answers to the questionnaire were scrutinised. Laboratories who reported exactly the same results for the two replicates of a material and nuclide were asked to confirm these results. Note that it can be expected that both the value and uncertainty of the two results are equal, since the two samples are from the same material.

Laboratories were also asked to clarify unclear answers to the questionnaire and to complete unanswered questions. This communication was started 31/07/2023 by e-mail. A reminder was sent on 30/08/2023, asking the laboratories to at least confirm the equal results. On 25/09/2023, all information needed to evaluate the results was available.

3.3 Type of gamma-ray spectrometer

14 participants took part in the validation study

Clause 7.2.1.5 of WD EN 17216 requires the use of a semiconductor gamma-ray spectrometer. This requirement is considered as essential and is also included in the title of the standard.

Participant L07 did not use the required detector type (Q 4.1). Therefore, the results reported by L07 are not used in the statistical analysis and, consequently, L07 is excluded from further evaluation.

4 Statistical analysis of the precision experiment

4.1 Introduction and notation

Except where explicitly mentioned, the data is analysed following ISO 5725-2 [8]. The report follows the notation of that standard:

- The test properties are the massic activity of Ra-226, Th-232 and K-40 in dry mass of the materials NORM1 to NORM6. Hence, the data analysis is performed for each radionuclide and each material, yielding a total of 18 test properties or data sets.
- Each laboratory is testing one level ($q = 1$) of the test properties. The index $j = 1$ in the notation of ISO 5725-2 and can be omitted to simplify the notation.
- For each test property, each laboratory analysed two replicates, meaning $n_i = 2$ where index i identifies the laboratory ($i = 1, 2, ..., p$). The index k identifying the replicate (and not be confused with the coverage factor of expanded uncertainties) only attains two values ($k = 1, 2$).
- The number of laboratories who participated in the study: $p = 14$ for NORM1, NORM3, NORM4, NORM5 and NORM6; $p = 13$ for NORM2.

The relation between the index i and the laboratory is established in Table 2 and is independent of the order of participants listed in Annex 2.

Table 2. Relation between the index i and the participating laboratory.

i	Participant	i	Participant
1	L01	8	L09
2	L02	9	L10
3	L03	10	L11
4	L04	11	L12
5	L05	12	L13
6	L06	13	L14
7	L08	14	L15 ([1])

1 Participant L15 did not report results for material NORM2, hence $i = 14$ is not possible for NORM2.

In the following sections, the different steps in analysing the data are explained:

- The results reported by the participants are presented and the reported uncertainties recalculated to standard uncertainties;
- For each test property, the mean result and intracell spread reported by each laboratory is calculated;
- Applying Mandel's h and k statistics, the data is scrutinised to check between- and within-laboratory consistency;
- Cochran's test is applied to detect stragglers and outliers in the standard deviations for each test property;
- Grubbs' test is applied to detect stragglers and outliers in the mean for each test property;
- Based on Mandel's statistics, Cochran's and Grubbs' test, decisions are made to reject certain data;
- The repeatability and reproducibility is calculated for each of the test properties;
- It is verified if a relation exists between the precision and the level of massic activity, and the average repeatability and reproducibility over the six materials is calculated for the three radionuclides.

4.2 Collation of results

For the materials NORM1 to NORM6, respectively Table 3 to Table 8 present the results reported by the laboratories for each of the two replicates. The values are shown as reported by the participants, with the same number of decimals. The uncertainties are recalculated to a standard uncertainty for those participants who reported the uncertainty with a coverage factor other than $k = 1$. These are laboratories L03, L05, L10, L12 and L14, who reported with $k = 2$, laboratory L04 who reported with $k = 1.65$ and laboratory L06, who reported with $k = 1.645$.

ISO 5725-2 [8] notes the test results reported by laboratory i as y_{ik} with ($i = 1, 2, \ldots, p$) and ($k = 1, 2$). The standard does not consider the uncertainties on the reported values, but they are included in the data analysis as further explained. The (reported or recalculated) standard uncertainties are noted as u_{ik} and shown in parentheses in the tables.

Table 3. Individual results reported for material NORM1. Each cell contains the results y_{ik} and standard uncertainty u_{ik} for each of the two replicates ($k = 1, 2$).

NORM1 Cement	Massic activity ([1]) $\boldsymbol{y_{ik}}$ $(\boldsymbol{u_{ik}})$ [Bq/kg]		
Participant ([2])	Ra-226	Th-232	K-40
L01	88 (5) 89 (5)	57 (3) 53 (3)	193 (10) 190 (10)
L02	83 (2) 81 (2)	48 (1) 46 (1)	178 (5) 168 (4)
L03♦	79.4 (6.0) 79.1 (6.0)	47 (4) 48.1 (3.6)	211 (16) 203.7 (15.3)
L04♦	86.1 (7.0) 83.9 (6.8)	50.0 (4.1) 49.3 (4.1)	192.9 (12.4) 184.1 (11.8)
L05♦	76.82 (4.33) 73.5 (4.1)	47.07 (2.68) 46.68 (3.06)	180.9 (10.6) 179.0 (10.4)
L06♦	87.6 (0.9) 87.4 (1.7)	53 (1) 52.8 (1.0)	182 (4) 186 (4)
L08	87 (4) 86 (4)	52 (3) 54 (3)	194 (11) 195 (11)
L09	84.3 (1.5) 84.7 (1.6)	54.8 (1.9) 25.7 (1.4)	180 (40) 180 (40)
L10♦	86.3 (1.0) 85.7 (1.0)	53.8 (2.2) 50.6 (1.8)	178 (5) 187 (5)
L11	87.37 (0.65) 87.14 (0.62)	52.20 (0.87) 52.80 (0.87)	188.9 (3.1) 193.6 (3.0)
L12♦	66 (8) 65 (8)	48 (6) 47 (6)	155 (19) 161 (19)
L13	79.2 (2.9) 82.2 (3.0)	50.1 (2.2) 52.2 (2.2)	176.7 (8.9) 175.8 (9.9)
L14♦	77.1 (1.6) 78.6 (1.7)	47.0 (2.0) 50.5 (1.3)	196 (6) 205 (6)
L15	80.11 (3.59) 81.71 (3.64)	45.73 (2.88) 48.6 (2.54)	212.06 (14.67) 197.54 (13.44)

[1] The massic activity in dry mass at the reference date of 01/03/2023. The standard uncertainties ($k = 1$) are shown as absolute values (in Bq/kg) in parentheses. The indexes range as ($i = 1, 2, \ldots, p = 14$) and ($k = 1, 2$).

[2] Laboratories who reported the uncertainty with a coverage factor $k \neq 1$ are indicated with a diamond♦. Their reported uncertainties have been recalculated to a standard uncertainty ($k = 1$) and are shown with the same number of decimals as on the reported value.

Table 4. Individual results reported for material NORM2. Each cell contains the results y_{ik} and standard uncertainty u_{ik} for each of the two replicates ($k = 1, 2$).

NORM2 Expanded clay blocks (piece)	Massic activity (1) $\boldsymbol{y_{ik}}$ $(\boldsymbol{u_{ik}})$ [Bq/kg]		
Participant (2)	Ra-226	Th-232	K-40
L01	33 (2) 34 (2)	27 (2) 28 (2)	348 (10) 350 (10)
L02	32 (1) 32 (1)	22 (1) 22 (1)	313 (6) 335 (6)
L03♦	27.5 (2.1) 27.9 (2.1)	22.7 (1.7) 21.8 (1.7)	343.5 (25.8) 346.2 (26.0)
L04♦	35.6 (2.9) 37.1 (3.0)	24.7 (2.9) 26.4 (3.1)	344.8 (21.3) 357.6 (22.1)
L05♦	32.4 (1.9) 32.73 (1.87)	23.6 (1.6) 23.90 (1.64)	341.6 (19.6) 351.3 (20.2)
L06♦	34.5 (0.6) 34.3 (0.8)	23.4 (0.4) 24.5 (0.5)	307 (6) 323 (7)
L08	31 (1) 32 (2)	25 (2) 25 (2)	345 (16) 347 (17)
L09	26.3 (0.9) 26.2 (0.8)	19.1 (1.1) 18.5 (1.1)	254 (52) 258 (53)
L10♦	35.1 (0.7) 34.8 (0.7)	23.8 (0.9) 23.0 (1.3)	300 (7) 320 (7)
L11	34.95 (0.36) 34.35 (0.26)	24.64 (0.44) 24.69 (0.41)	331.0 (4.7) 335.1 (4.6)
L12♦	25.3 (2.9) 23.6 (2.7)	23.8 (2.9) 22.6 (2.8)	337 (38) 317 (36)
L13	34.1 (1.3) 31.9 (1.2)	24.9 (1.4) 23.7 (1.3)	307.1 (15.6) 298.8 (14.9)
L14♦	28.3 (1.1) 25.9 (0.8)	26.2 (2.0) 21.3 (1.4)	350 (11) 342 (8)
L15	No results reported		

1 The massic activity in dry mass at the reference date of 01/03/2023. The standard uncertainties ($k = 1$) are shown as absolute values (in Bq/kg) in parentheses The indexes range as ($i = 1, 2, \ldots, p = 13$) and ($k = 1, 2$).

2 Laboratories who reported the uncertainty with a coverage factor $k \neq 1$ are indicated with a diamond♦. Their reported uncertainties have been recalculated to a standard uncertainty ($k = 1$) and are shown with the same number of decimals as on the reported value.

Table 5. Individual results reported for material NORM3. Each cell contains the results y_{ik} and standard uncertainty u_{ik} for each of the two replicates ($k = 1, 2$).

NORM3 Expanded clay blocks (powder)	Massic activity ([1]) $\boldsymbol{y_{ik}}$ $(\boldsymbol{u_{ik}})$ [Bq/kg]		
Participant ([2])	Ra-226	Th-232	K-40
L01	32 (2) 30 (2)	25.5 (1.7) 26.2 (1.8)	330 (16) 332 (16)
L02	35 (1) 34 (1)	23 (1) 23 (1)	328 (6) 318 (6)
L03♦	26.6 (2.0) 28.1 (2.1)	22.1 (1.7) 21.4 (1.6)	335.1 (25.2) 320.9 (24.1)
L04♦	32.9 (2.7) 34.1 (2.8)	22.3 (1.9) 24.1 (2.1)	307.3 (19.0) 307.4 (19.0)
L05♦	28.35 (1.67) 29.77 (1.75)	22.02 (1.36) 21.54 (1.57)	287.2 (16.6) 291.4 (16.8)
L06♦	29.9 (0.6) 30.4 (0.6)	21.9 (0.5) 21.1 (0.4)	285 (6) 285 (6)
L08	34 (2) 35 (2)	23 (2) 24 (2)	327 (15) 339 (18)
L09	30.7 (1.1) 31.6 (1.2)	28.0 (1.5) 33.5 (1.8)	295 (60) 290 (60)
L10♦	35.2 (0.7) 34.0 (0.7)	23.8 (1.0) 23.2 (1.3)	307 (7) 304 (7)
L11	34.21 (0.30) 34.53 (0.29)	23.45 (0.43) 24.03 (0.43)	323 (4.7) 322 (4.6)
L12♦	23.7 (2.8) 25.3 (3.0)	20.8 (2.6) 22.3 (2.9)	282 (32) 290 (33)
L13	30.8 (1.2) 30.9 (1.2)	22.6 (1.0) 22.5 (1.0)	297.9 (14.8) 300.7 (15.0)
L14♦	28.7 (0.8) 27.1 (0.8)	21.0 (0.8) 19.8 (1.2)	324 (7) 290 (6)
L15	33.70 (1.44) 34.57 (1.78)	20.17 (1.04) 20.84 (1.94)	337.85 (15.21) 331.27 (18.82)

1 The massic activity in dry mass at the reference date of 01/03/2023. The standard uncertainties ($k = 1$) are shown as absolute values (in Bq/kg) in parentheses. The indexes range as ($i = 1, 2, \ldots, p = 14$) and ($k = 1, 2$).

2 Laboratories who reported the uncertainty with a coverage factor $k \neq 1$ are indicated with a diamond♦. Their reported uncertainties have been recalculated to a standard uncertainty ($k = 1$) and are shown with the same number of decimals as on the reported value.

Table 6. Individual results reported for material NORM4. Each cell contains the results y_{ik} and standard uncertainty u_{ik} for each of the two replicates ($k = 1, 2$).

NORM4 Plaster	Massic activity ([1]) $\boldsymbol{y_{ik}}$ $(\boldsymbol{u_{ik}})$ [Bq/kg]		
Participant ([2])	Ra-226	Th-232	K-40
L01	36 (3) 35 (3)	39 (3) 41 (3)	69 (6) 67 (6)
L02	21 (1) 20 (1)	34 (1) 32 (1)	61 (4) 54 (3)
L03♦	19.5 (1.5) 18.9 (1.4)	36.6 (2.8) 34.6 (2.6)	78.2 (5.9) 61.4 (4.6)
L04♦	23.4 (2.0) 23.3 (1.9)	40.2 (3.4) 40.7 (3.5)	59.2 (4.8) 69.1 (5.4)
L05♦	19.56 (1.20) 19.90 (1.23)	34.40 (2.34) 35.47 (2.11)	50.55 (3.38) 54.89 (3.73)
L06♦	21.2 (0.5) 21.7 (0.6)	34.7 (0.7) 35.3 (0.5)	52.3 (1.8) 50 (1.8)
L08	32 (1) 32 (1)	39 (3) 38 (3)	70 (6) 69 (6)
L09	22.2 (1.2) 21.5 (1.1)	42.2 (2.2) 38.1 (2.0)	51 (13) 52 (13)
L10♦	22.0 (0.7) 22.5 (0.5)	36.8 (1.7) 37.5 (1.7)	50.1 (5.2) 58.1 (5.4)
L11	22.18 (0.24) 21.86 (0.24)	39.65 (0.77) 39.94 (0.67)	59.5 (1.4) 61.4 (1.5)
L12♦	15.8 (1.9) 17.0 (2.1)	35.6 (4.3) 34.3 (4.1)	32 (6) 36 (7)
L13	20.2 (0.8) 21.0 (0.8)	37.0 (1.6) 37.2 (2.1)	54.3 (3.1) 52.5 (3.1)
L14♦	19.9 (0.7) 21.0 (0.7)	37.3 (1.7) 39.4 (1.8)	67.6 (4.4) 66.8 (4.2)
L15	21.50 (1.24) 22.86 (1.03)	35.32 (2.44) 33.26 (1.49)	64.79 (8.24) 54.55 (4.19)

1 The massic activity in dry mass at the reference date of 01/03/2023. The standard uncertainties ($k = 1$) are shown as absolute values (in Bq/kg) in parentheses The indexes range as ($i = 1, 2, \ldots, p = 14$) and ($k = 1, 2$).

2 Laboratories who reported the uncertainty with a coverage factor $k \neq 1$ are indicated with a diamond♦. Their reported uncertainties have been recalculated to a standard uncertainty ($k = 1$) and are shown with the same number of decimals as on the reported value.

Table 7. Individual results reported for material NORM5. Each cell contains the results y_{ik} and standard uncertainty u_{ik} for each of the two replicates ($k = 1, 2$).

NORM5 Aggregates	Massic activity ([1]) $\boldsymbol{y_{ik}}$ $(\boldsymbol{u_{ik}})$ [Bq/kg]		
Participant ([2])	Ra-226	Th-232	K-40
L01	126 (4) 126 (5)	71 (4) 75 (4)	1480 (60) 1475 (60)
L02	141 (3) 144 (3)	71 (1) 69 (1)	1490 (21) 1381 (19)
L03♦	96.4 (7.3) 102.4 (7.7)	59.1 (4.5) 64.6 (4.9)	1424 (107) 1524 (115)
L04♦	133.1 (10.7) 133.1 (10.7)	66.4 (5.5) 67.9 (7.5)	1444.0 (87.3) 1451.0 (87.9)
L05♦	107.3 (6.0) 121.1 (6.8)	57.6 (3.3) 61.06 (3.52)	1340 (76) 1389 (79)
L06♦	94 (4) 101.3 (4.9)	51.6 (1.1) 52.8 (1.1)	1189 (22) 1167 (22)
L08	121 (5) 126 (6)	63 (3) 62 (3)	1466 (59) 1470 (62)
L09	157 (6) 157 (8)	22.6 (1.2) 11.8 (0.7)	1370 (270) 1330 (270)
L10♦	123 (3) 114 (2)	67.3 (1.5) 67.4 (2.5)	1452 (29) 1471 (29)
L11	87.69 (0.54) 90.33 (0.56)	59.6 (1.0) 59.9 (1.1)	2165 (27) 2147 (27)
L12♦	88 (13) 81 (12)	62 (9) 60 (9)	1419 (199) 1446 (202)
L13	121.6 (4.4) 124.6 (4.6)	69.5 (3.8) 64.7 (2.9)	1324.9 (66.4) 1345.0 (67.4)
L14♦	107 (2) 112 (2)	59.1 (2.2) 60.0 (2.3)	1416 (19) 1437 (20)
L15	136.42 (5.76) 141.49 (5.77)	59.42 (3.18) 59.50 (2.25)	1453.59 (63.24) 1424.69 (59.53)

1 The massic activity in dry mass at the reference date of 01/03/2023. The standard uncertainties ($k = 1$) are shown as absolute values (in Bq/kg) in parentheses. The indexes range as ($i = 1, 2, \ldots, p = 14$) and ($k = 1, 2$).

2 Laboratories who reported the uncertainty with a coverage factor $k \neq 1$ are indicated with a diamond♦. Their reported uncertainties have been recalculated to a standard uncertainty ($k = 1$) and are shown with the same number of decimals as on the reported value.

Table 8. Individual results reported for material NORM6. Each cell contains the results y_{ik} and standard uncertainty u_{ik} for each of the two replicates ($k = 1, 2$).

NORM6 Milled concrete	Massic activity ([1]) $\boldsymbol{y_{ik}}$ ($\boldsymbol{u_{ik}}$) [Bq/kg]		
Participant ([2])	Ra-226	Th-232	K-40
L01	65 (4) 67 (4)	60 (3) 62 (4)	1275 (52) 1269 (54)
L02	47 (1) 47 (1)	55 (1) 55 (1)	1216 (18) 1235 (18)
L03♦	38.3 (2.9) 41.2 (3.1)	50.5 (3.8) 54.8 (4.1)	1296 (97) 1366 (103)
L04♦	54.6 (4.4) 57.2 (4.7)	58.5 (4.8) 60.7 (6.8)	1223 (74) 1265 (77)
L05♦	49.58 (2.81) 50.33 (2.86)	55.28 (3.14) 53.61 (3.50)	1198 (68) 1187 (67)
L06♦	42 (1) 41.7 (0.7)	49 (1) 49.3 (0.7)	1046 (19) 1057 (19)
L08	58 (3) 61 (4)	61 (4) 62 (4)	1281 (53) 1293 (51)
L09	52.3 (1.1) 41.9 (1.4)	59.9 (1.7) 57.99 (2.2)	1170 (240) 1190 (240)
L10♦	57.0 (1.0) 56.7 (1.1)	57.7 (2.1) 59.3 (2.4)	1209 (21) 1193 (22)
L11	54.79 (0.41) 57.43 (0.47)	60.72 (0.92) 60.05 (0.99)	1260 (17) 1241 (17)
L12♦	33.5 (3.9) 33.2 (3.8)	53 (6) 54 (7)	1191 (134) 1164 (131)
L13	52.0 (1.9) 50.7 (1.9)	58.9 (2.5) 58.3 (2.4)	1156.9 (57.1) 1150.8 (56.8)
L14♦	42.7 (1.8) 38.7 (1.0)	57.0 (3.9) 54.2 (2.2)	1282 (28) 1208 (18)
L15	50.06 (2.37) 50.63 (2.45)	55.10 (3.26) 47.06 (2.96)	1295.62 (58.30) 1277.68 (57.42)

[1] The massic activity in dry mass at the reference date of 01/03/2023. The standard uncertainties ($k = 1$) are shown as absolute values (in Bq/kg) in parentheses. The indexes range as ($i = 1, 2, ..., p = 14$) and ($k = 1, 2$).

[2] Laboratories who reported the uncertainty with a coverage factor $k \neq 1$ are indicated with a diamond♦. Their reported uncertainties have been recalculated to a standard uncertainty ($k = 1$) and are shown with the same number of decimals as on the reported value.

4.3 Mean results and spread

For each of the 18 test properties and participants, the mean result (or cell mean) is calculated as in Equation (2) of ISO 5725-2 [8], which reduces to:

$$\bar{y}_i = (y_{i1} + y_{i2})/2 \tag{1}$$

Equation (5) of ISO 5725-2 [8] calculates the spread between the values of the two replicates (the intracell spread) as:

$$s_{i;\mathrm{ext}} = |y_{i1} - y_{i2}|/\sqrt{2} \tag{2}$$

Note that ISO 5725-2 does not consider the reported uncertainties on the results of the measurements of the replicates. The method in Clause 8.4.2 of WD EN 17216 for calculating the uncertainty for multiple test specimens is adopted here. The intracell spread from ISO 5725-2, as calculated in Equation (2), corresponds with the external uncertainty $u_{i;\mathrm{ext}}$ from Equation (9) of WD EN 17216, apart from the different meaning of the index i. Since in this report, the notation of ISO 5725-2 prevails, the subscript "ext" was added to Equation (2), yielding the symbol $s_{i;\mathrm{ext}}$ for the "external" intracell spread.

Applying Equation (8) from WD EN 17216 results in the "internal" intracell spread:

$$s_{i;\mathrm{int}} = \frac{1}{2}\sqrt{u_{i1}^2 + u_{i2}^2} \tag{3}$$

The maximum of the two is further considered as the intracell spread, similarly as in Equation (11) of WD EN 17216, but adopting the notation of ISO 5725-2:

$$s_i = \max\{s_{i;\mathrm{int}}, s_{i;\mathrm{ext}}\} \tag{4}$$

Table 9 to Table 14 show the results of the calculation of the mean result $\bar{y}_i$ and intracell spread s_i. The results are displayed with one more significant digit than the reported test results in Section 4.2, but all calculations are performed using the maximum number of digits that the spreadsheet software supports. Each table is followed by a plot of the data. Note that the uncertainties in the tables are shown with a coverage factor of $k = 1$ while on the plots, for reasons of facilitating comparison, the error bars are expanded uncertainties with a coverage factor of $k = 2$. Note also that the mean massic activities $\bar{y}_i$ and intracell spreads s_i are specific for each material, each radionuclide and each laboratory i. The mean and intracell spread do not depend on the results reported by other laboratories (and evidently also not on the results of other radionuclides or other materials).

Table 9. Cell mean $\bar{y}_i$ and intracell spread s_i for material NORM1.

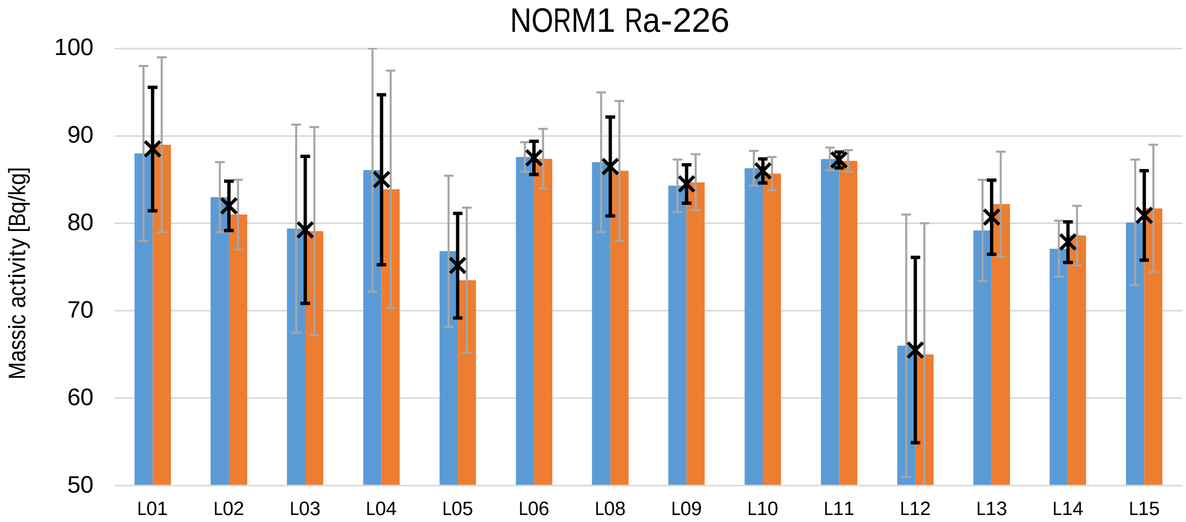
NORM1 Cement	Mean massic activity and intracell spread ([1]) $\bar{\boldsymbol{y}}_{\boldsymbol{i}}$ $(\boldsymbol{s}_{\boldsymbol{i}})$ [Bq/kg]		
Participant	Ra-226	Th-232	K-40
L01	88.5 (3.5)	55.0 (2.8)	191.5 (7.1)
L02	82.0 (1.4)	47.0 (1.4)	173.0 (7.1)
L03	79.25 (4.21)	47.55 (2.53)	207.35 (11.00)
L04	85.00 (4.86)	49.65 (2.89)	188.50 (8.55)
L05	75.160* (2.996)	46.875 (2.034)	179.95 (7.44)
L06	87.50 (0.95)	52.90 (0.63)	184.0 (3.0)
L08	86.5 (2.8)	53.0 (2.1)	194.5 (7.8)
L09	84.50 (1.10)	40.25 (20.58**)	180.0 (28.3)
L10	86.00 (0.69)	52.20 (2.26)	182.5 (6.4)
L11	87.255 (0.449)	52.500 (0.615)	191.250 (3.323)
L12	65.5* (5.3)	47.5 (3.9)	158.0 (13.3)
L13	80.70 (2.12)	51.15 (1.56)	176.25 (6.66)
L14	77.85 (1.17)	48.75 (2.47)	200.5 (6.4)
L15	80.910 (2.556)	47.165 (2.029)	204.800 (10.267)

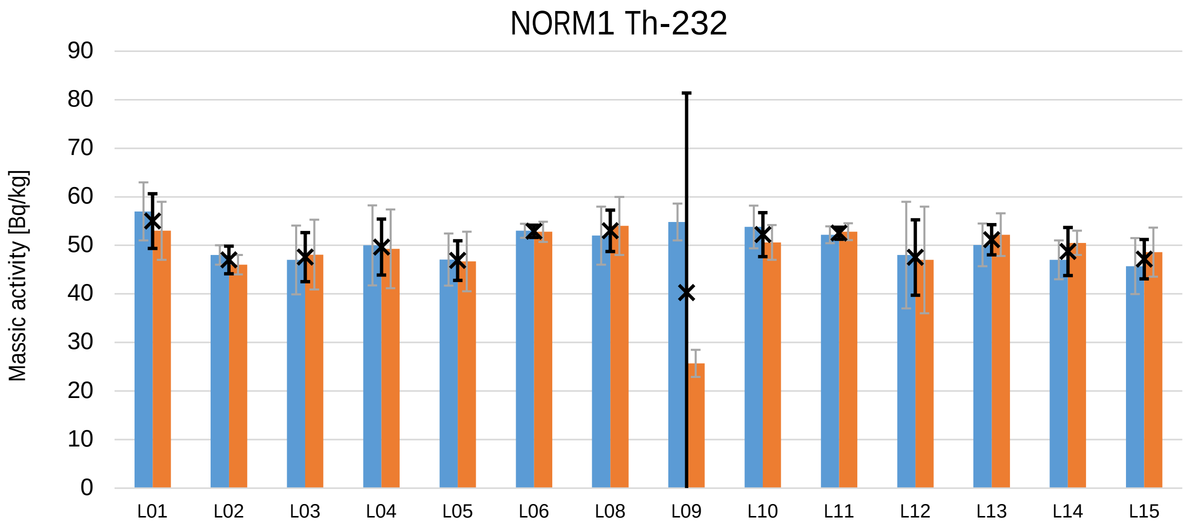
1 The mean massic activity in dry mass and intracell spread at the reference date of 01/03/2023. The spread is reported as a standard uncertainty. The indexes range as ($i = 1, 2, ..., p = 14$).

* A single asterisk identifies stragglers, either for the cell means (Grubbs' test) or the intracell spread (Cochran's test).

** A double asterisk identifies outliers, either for the cell means (Grubbs' test) or the intracell spread (Cochran's test).

Figure 1. Reported results (columns) of the measurement of the two replicates of material NORM1. The mean massic activities (in dry mass) $\bar{y}_i$ are shown using crosses, their error bars are the intracell spread s_i. Error bars are expanded uncertainties, plotted with a coverage factor of $k = 2$.





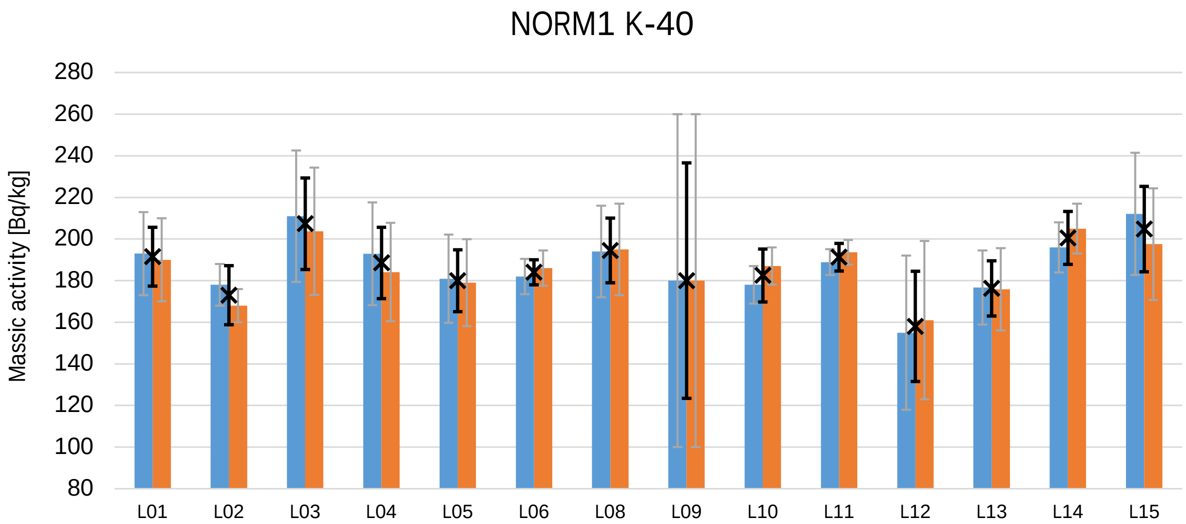


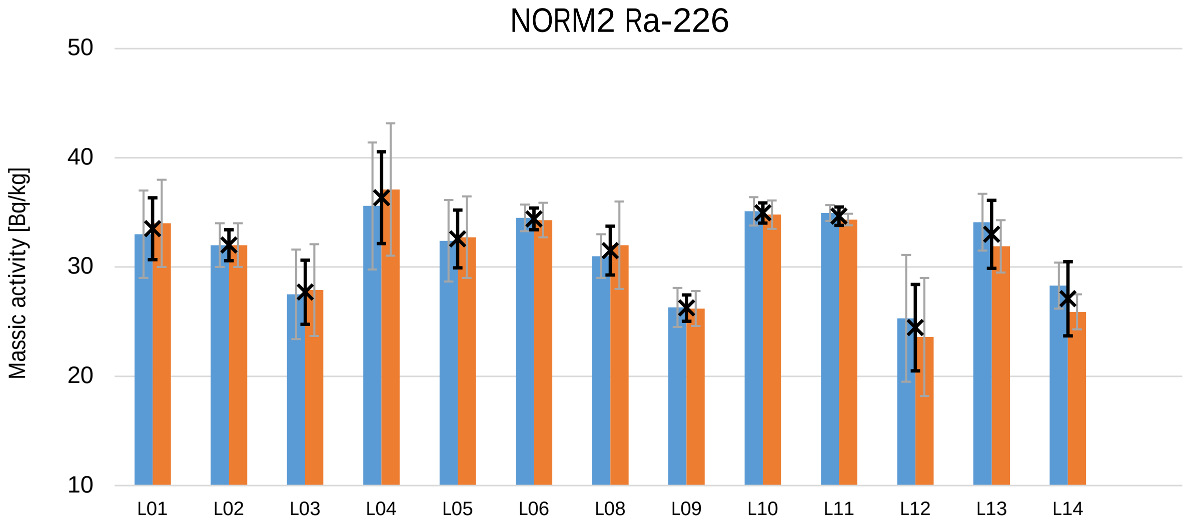
Table 10. Cell mean $\bar{y}_i$ and intracell spread s_i for material NORM2.

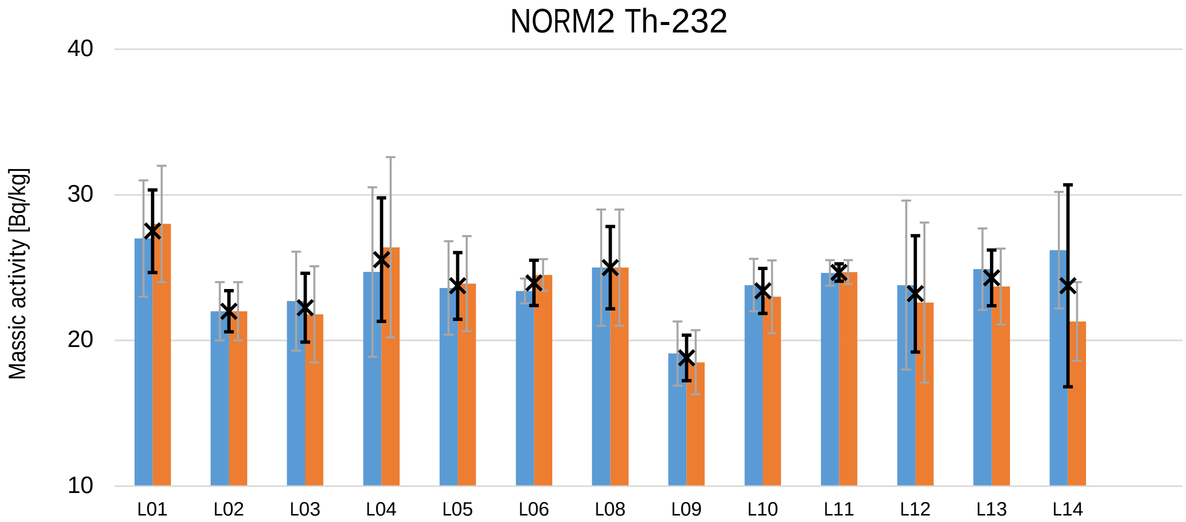
NORM2 Expanded clay blocks (piece)	Mean massic activity and intracell spread ([1]) $\bar{\boldsymbol{y}}_{\boldsymbol{i}}$ ($\boldsymbol{s_i}$) [Bq/kg]		
Participant	Ra-226	Th-232	K-40
L01	33.5 (1.4)	27.5 (1.4)	349.0 (7.1)
L02	32.0 (0.7)	22.0 (0.7)	324.0 (15.6)
L03	27.70 (1.47)	22.25 (1.18)	344.85 (18.28)
L04	36.35 (2.10)	25.55 (2.12)	351.20 (15.37)
L05	32.565 (1.320)	23.750 (1.146)	346.45 (14.06)
L06	34.40 (0.50)	23.95 (0.78)	315.0 (11.3)
L08	31.5 (1.1)	25.0 (1.4)	346.0 (11.7)
L09	26.25 (0.60)	18.80 (0.78)	256.0* (37.1)
L10	34.95 (0.46)	23.40 (0.77)	310.0 (14.1)
L11	34.650 (0.424)	24.665 (0.301)	333.05 (3.29)
L12	24.45 (1.98)	23.20 (2.00)	327.0 (26.2)
L13	33.00 (1.56)	24.30 (0.96)	302.95* (10.79)
L14	27.10 (1.70)	23.75 (3.46)	346.0 (6.8)
L15	No results reported		

[1] The mean massic activity in dry mass and intracell spread at the reference date of 01/03/2023. The spread is reported as a standard uncertainty. The indexes range as ($i = 1, 2, \ldots, p = 13$).

* A single asterisk identifies stragglers, either for the cell means (Grubbs' test) or the intracell spread (Cochran's test).

Figure 2. Reported results (columns) of the measurement of the two replicates of material NORM2. The mean massic activities (in dry mass) $\bar{y}_i$ are shown using crosses, their error bars are the intracell spread s_i. Error bars are expanded uncertainties, plotted with a coverage factor of $k = 2$.





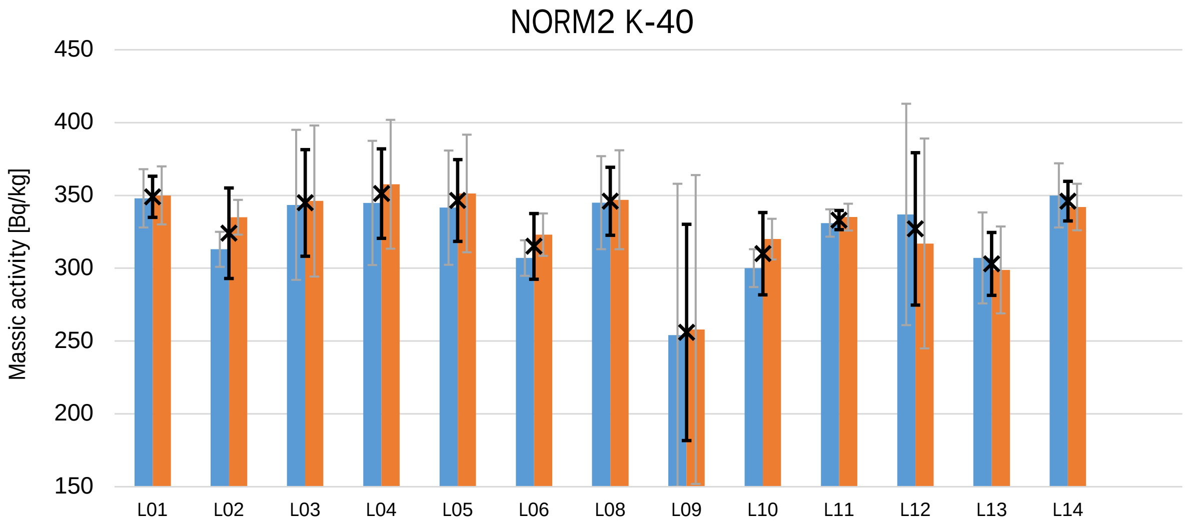


Table 11. Cell mean $\bar{y}_i$ and intracell spread s_i for material NORM3.

NORM3 Expanded clay blocks (powder)	Mean massic activity and intracell spread ([1]) $\bar{\boldsymbol{y}}_{\boldsymbol{i}}$ $(\boldsymbol{s}_{\boldsymbol{i}})$ [Bq/kg]		
Participant	Ra-226	Th-232	K-40
L01	31.0 (1.4)	25.85 (1.24)	331.0 (11.3)
L02	34.5 (0.7)	23.0 (0.7)	323.0 (7.1)
L03	27.35 (1.45)	21.75 (1.15)	328.00 (17.40)
L04	33.50 (1.93)	23.20 (1.44)	307.35 (13.44)
L05	29.060 (1.211)	21.780 (1.038)	289.300 (11.809)
L06	30.15 (0.43)	21.50 (0.57)	285.0 (4.3)
L08	34.5 (1.4)	23.5 (1.4)	333.0 (11.7)
L09	31.15 (0.81)	30.75** (3.89)	292.5 (42.4)
L10	34.60 (0.85)	23.50 (0.81)	305.5 (5.0)
L11	34.370 (0.226)	23.740 (0.410)	322.500 (3.288)
L12	24.50 (2.03)	21.55 (1.91)	286.0 (23.0)
L13	30.85 (0.85)	22.55 (0.71)	299.30 (10.54)
L14	27.90 (1.13)	20.40 (0.85)	307.0 (24.0)
L15	34.135 (1.145)	20.505 (1.101)	334.560 (12.099)

1 The mean massic activity in dry mass and intracell spread at the reference date of 01/03/2023. The spread is reported as a standard uncertainty. The indexes range as ($i = 1, 2, \ldots, p = 14$).

** A double asterisk identifies outliers, either for the cell means (Grubbs' test) or the intracell spread (Cochran's test).

Figure 3. Reported results (columns) of the measurement of the two replicates of material NORM3. The mean massic activities (in dry mass) $\bar{y}_i$ are shown using crosses, their error bars are the intracell spread s_i. Error bars are expanded uncertainties, plotted with a coverage factor of $k = 2$.

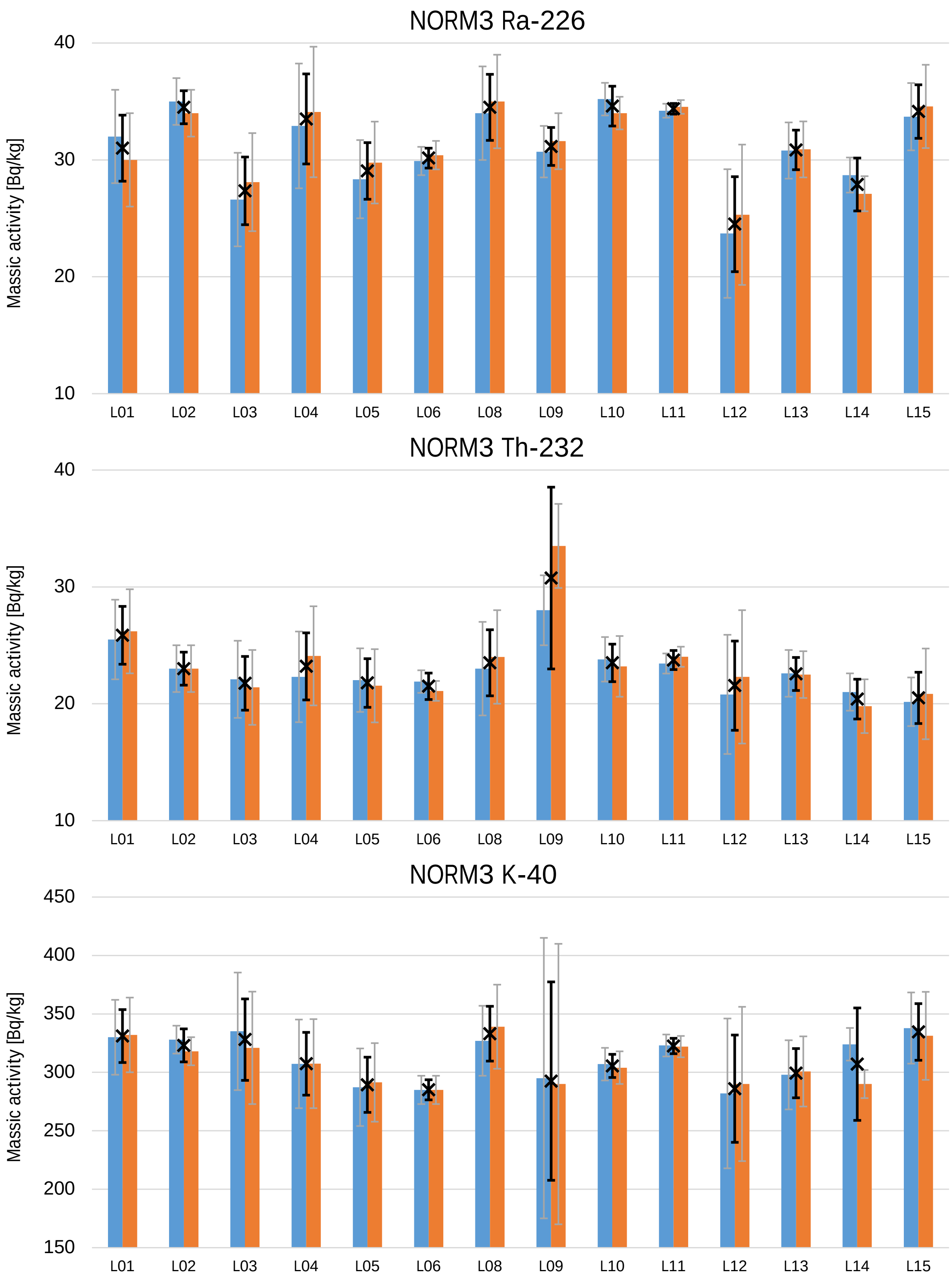


Table 12. Cell mean $\bar{y}_i$ and intracell spread s_i for material NORM4.

NORM4 Plaster	Mean massic activity and intracell spread ([1]) $\bar{\boldsymbol{y}}_{\boldsymbol{i}}$ $(\boldsymbol{s}_{\boldsymbol{i}})$ [Bq/kg]		
Participant	Ra-226	Th-232	K-40
L01	35.5* **(2.1)	40.0 (2.1)	68.0 (4.2)
L02	20.5 (0.7)	33.0 (1.4)	57.5 (4.9)
L03	19.20 (1.01)	35.60 (1.89)	69.80 (11.88)
L04	23.35 (1.39)	40.45 (2.42)	64.15 (7.00)
L05	19.730 (0.858)	34.935 (1.575)	52.720 (3.069)
L06	21.45 (0.41)	35.00 (0.46)	51.15 (1.63)
L08	32.0** (0.7)	38.5 (2.1)	69.5 (4.2)
L09	21.85 (0.81)	40.15 (2.90)	51.5 (9.2)
L10	22.25 (0.41)	37.15 (1.17)	54.10 (5.66)
L11	22.020 (0.226)	39.795 (0.510)	60.45 (1.34)
L12	16.40 (1.42)	34.95 (2.95)	34.0 (4.4)
L13	20.60 (0.57)	37.10 (1.32)	53.40 (2.19)
L14	20.45 (0.78)	38.35 (1.48)	67.20 (3.02)
L15	22.180 (0.962)	34.290 (1.457)	59.670 (7.241)

[1] The mean massic activity in dry mass and intracell spread at the reference date of 01/03/2023. The spread is reported as a standard uncertainty. The indexes range as $(i = 1, 2, ..., p = 14)$.

** A double asterisk identifies outliers, either for the cell means (Grubbs' test) or the intracell spread (Cochran's test).

* ** The cell mean tested as a straggler for the singles Grubbs' test and as an outlier for the doubles Grubbs' test.

Figure 4. Reported results (columns) of the measurement of the two replicates of material NORM4. The mean massic activities (in dry mass) $\bar{y}_i$ are shown using crosses, their error bars are the intracell spread s_i. Error bars are expanded uncertainties, plotted with a coverage factor of $k = 2$.

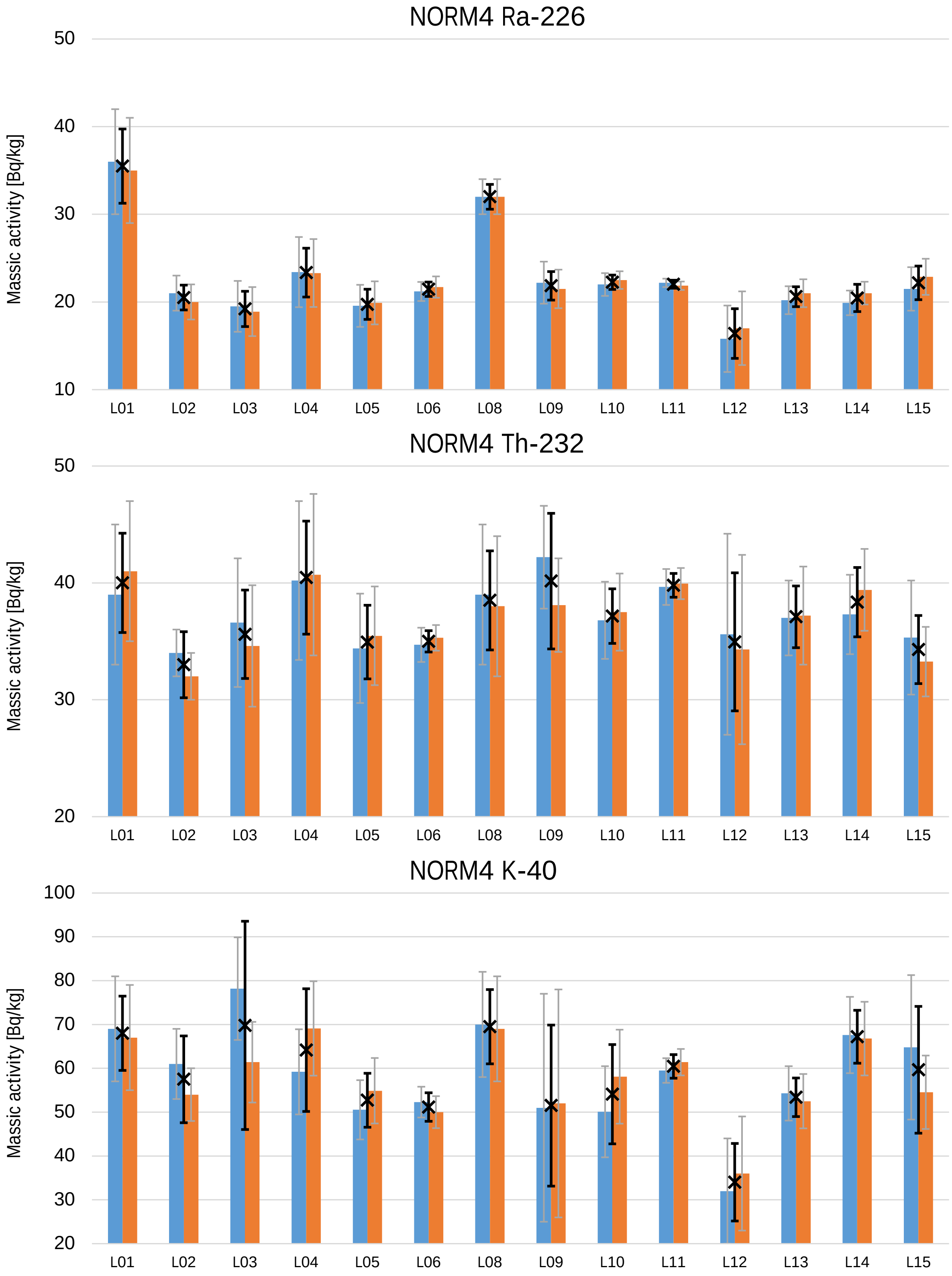


Table 13. Cell mean $\bar{y}_i$ and intracell spread s_i for material NORM5.

NORM5 Aggregates	Mean massic activity and intracell spread ([1]) $\bar{\boldsymbol{y}}_{\boldsymbol{i}}$ $(\boldsymbol{s}_{\boldsymbol{i}})$ [Bq/kg]		
Participant	Ra-226	Th-232	K-40
L01	126.0 (3.2)	73.0 (2.8)	1477.5 (42.4)
L02	142.5 (2.1)	70.0 (1.4)	1435.5 (77.1)
L03	99.40 (5.29)	61.85 (3.89)	1474.0 (78.4)
L04	133.10 (7.59)	67.15 (4.64)	1447.50 (61.93)
L05	114.20 (9.76)	59.330 (2.447)	1364.5 (54.7)
L06	97.65 (5.16)	52.20 (0.85)	1178.0** (15.6)
L08	123.5 (3.9)	62.5 (2.1)	1468.0 (42.8)
L09	157.0 (5.0)	17.20** (7.64)	1350.0 (190.9)
L10	118.5 (6.4)	67.35 (1.45)	1461.5 (20.3)
L11	89.010 (1.867)	59.75 (0.74)	2156.0** (19.1)
L12	84.5 (8.5)	61.0 (6.2)	1432.5 (141.6)
L13	123.10 (3.18)	67.10 (3.39)	1334.95 (47.31)
L14	109.5 (3.5)	59.55 (1.59)	1426.5 (14.9)
L15	138.955 (4.076)	59.460 (1.948)	1439.140 (43.426)

1 The mean massic activity in dry mass and intracell spread at the reference date of 01/03/2023. The spread is reported as a standard uncertainty. The indexes range as ($i = 1, 2, \ldots, p = 14$).

** A double asterisk identifies outliers, either for the cell means (Grubbs' test) or the intracell spread (Cochran's test).

Figure 5. Reported results (columns) of the measurement of the two replicates of material NORM5. The mean massic activities (in dry mass) $\bar{y}_i$ are shown using crosses, their error bars are the intracell spread s_i. Error bars are expanded uncertainties, plotted with a coverage factor of $k = 2$.

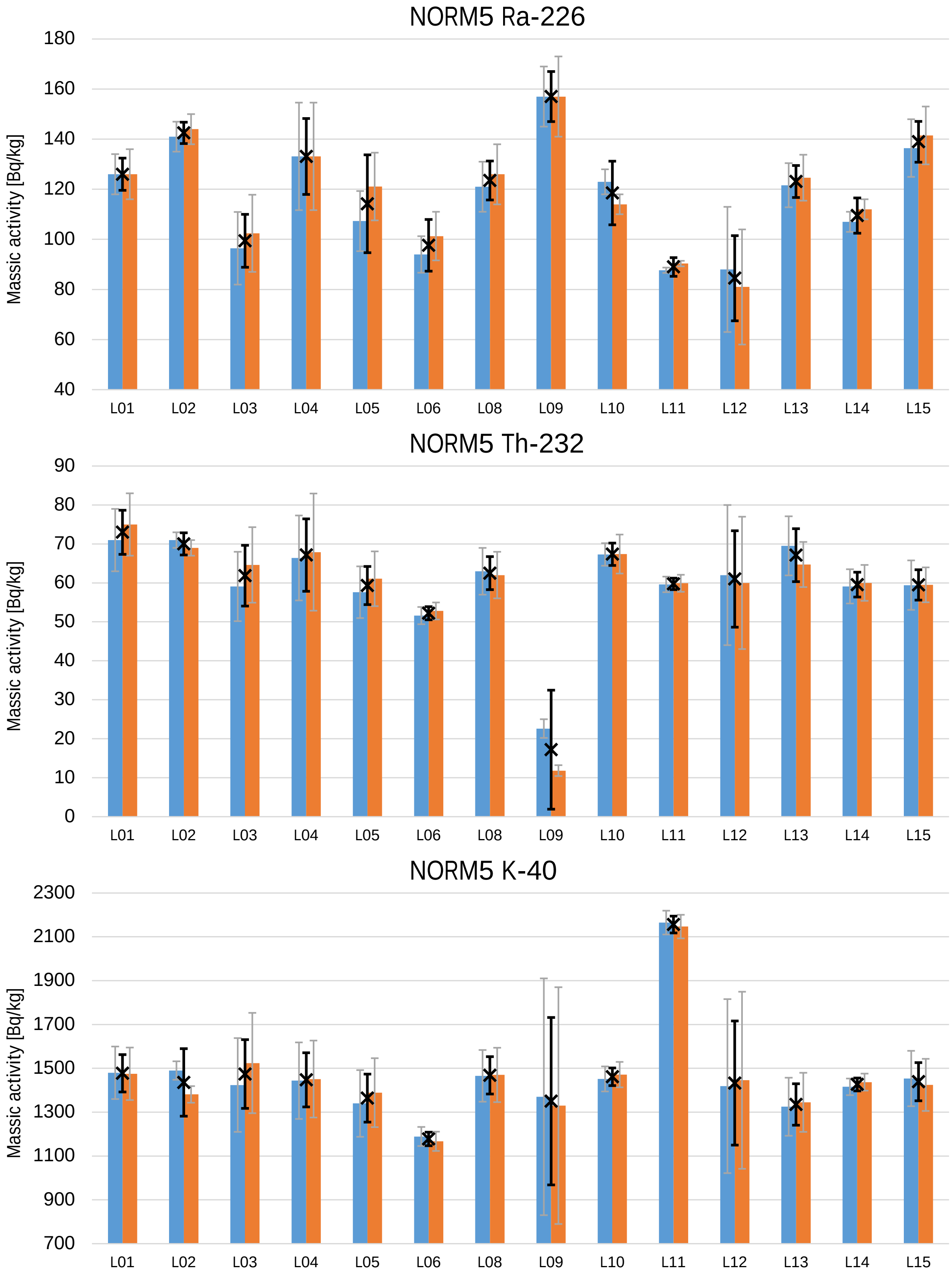


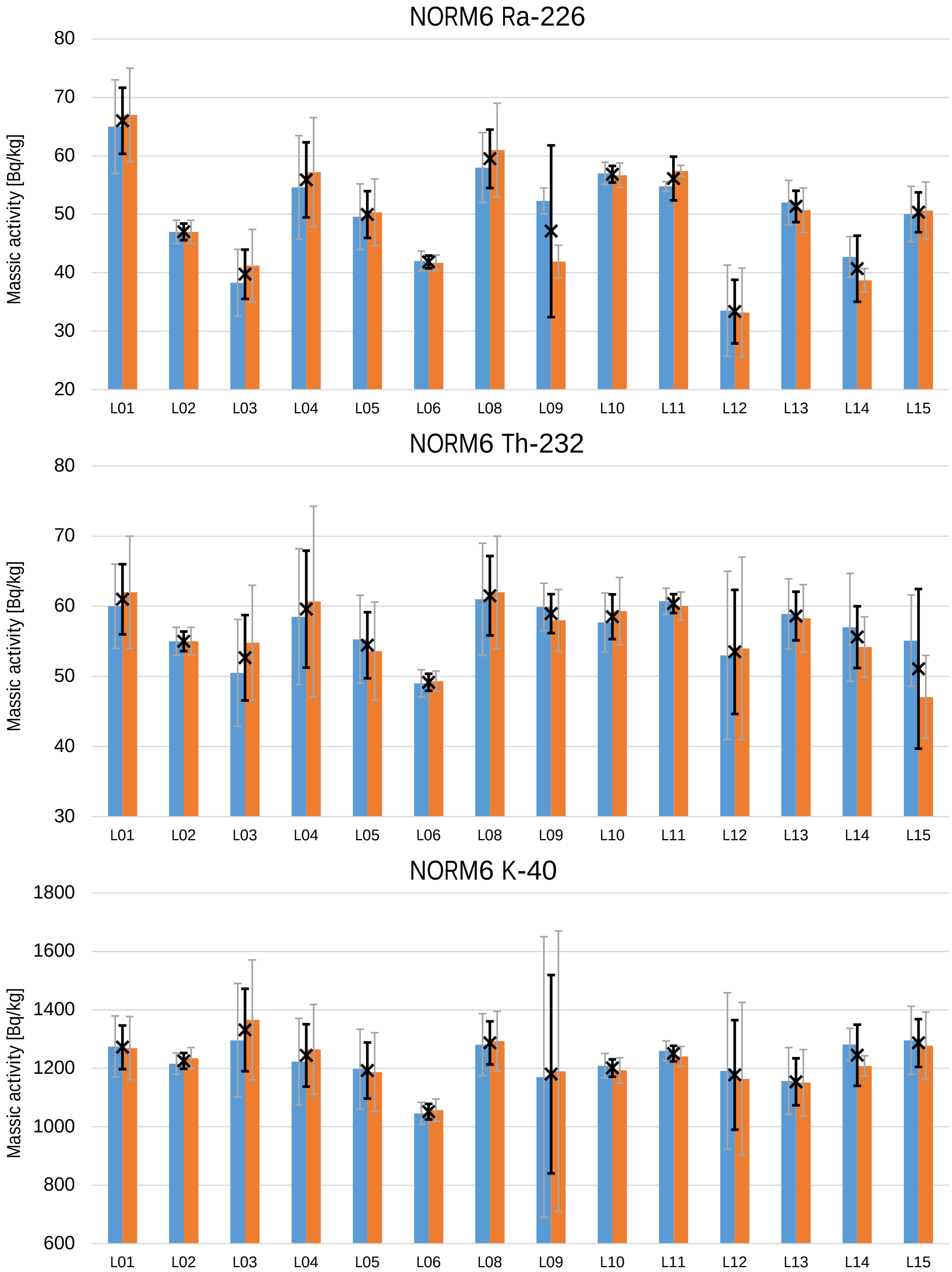
Table 14. Cell mean $\bar{y}_i$ and intracell spread s_i for material NORM6.

NORM6 Milled concrete	Mean massic activity and intracell spread ([1]) $\bar{\boldsymbol{y}}_{\boldsymbol{i}}$ $(\boldsymbol{s}_{\boldsymbol{i}})$ [Bq/kg]		
Participant	Ra-226	Th-232	K-40
L01	66.0 (2.8)	61.0 (2.5)	1272.0 (37.5)
L02	47.0 (0.7)	55.0 (0.7)	1225.5 (13.4)
L03	39.75 (2.11)	52.65 (3.04)	1331.0 (70.6)
L04	55.90 (3.22)	59.60 (4.17)	1244.0 (53.4)
L05	49.955 (2.005)	54.445 (2.352)	1192.5 (47.9)
L06	41.85 (0.54)	49.15 (0.61)	1051.5 (13.3)
L08	59.5 (2.5)	61.5 (2.8)	1287.0 (36.8)
L09	47.10 (7.35)	58.95 (1.39)	1180.0 (169.7*)
L10	56.85 (0.71)	58.50 (1.59)	1201.0 (15.0)
L11	56.110 (1.867)	60.385 (0.676)	1250.5 (13.4)
L12	33.35 (2.72)	53.50 (4.42)	1177.5 (93.7)
L13	51.35 (1.34)	58.60 (1.73)	1153.85 (40.27)
L14	40.70 (2.83)	55.60 (2.20)	1245.0 (52.3)
L15	50.345 (1.704)	51.080 (5.685)	1286.650 (40.914)

1 The mean massic activity in dry mass and intracell spread at the reference date of 01/03/2023. The spread is reported as a standard uncertainty. The indexes range as ($i = 1, 2, \ldots, p = 14$).

* A single asterisk identifies stragglers, either for the cell means (Grubbs' test) or the intracell spread (Cochran's test).

Figure 6. Reported results (columns) of the measurement of the two replicates of material NORM6. The mean massic activities (in dry mass) $\bar{y}_i$ are shown using crosses, their error bars are the intracell spread s_i. Error bars are expanded uncertainties, plotted with a coverage factor of $k = 2$.



4.4 Scrutiny of results for consistency

Mandel's h and k statistics are used to verify the consistency between the laboratories and the consistency within each laboratory.

For each of the 18 test properties, the general mean is calculated following Equations (23) and (24) of ISO 5725-2 [8], which reduces in this case to:

$$\hat{m} = \bar{\bar{y}} = \frac{\sum_{i=0}^{p}(y_{i1}+y_{i2})}{2p} \tag{5}$$

For each laboratory i, the Mandel's h statistic is calculated according to Equation (6) of ISO 5725-2 [8], leading to:

$$h_i = \frac{\bar{y}_i - \bar{\bar{y}}}{\sqrt{\frac{1}{p-1}\sum_{i=0}^{p}(\bar{y}_i - \bar{\bar{y}})^2}} \tag{6}$$

in which the nominator is the cell mean minus the general mean and the denominator is the standard deviation among cell means.

The within-laboratory consistency (Mandel's k statistic) is calculated for each laboratory i, following Equations (7) and (8) of ISO 5725-2 [8]:

$$k_i = \frac{s_i\sqrt{p}}{\sqrt{\sum_{i=1}^{p} s_i^2}} \tag{7}$$

Figure 7 and Figure 8 show a plot of the between- and within-laboratory consistency values h_i and k_i, respectively. The figures include the Mandel's indicator lines at a significance level $\alpha = 1$ % and 5 % (See Annex D of ISO 5725-2 [8] for their calculation). Values outside these indicators are expected to occur by chance with a frequency of α. Comparing the between- and within-laboratory consistency values h_i and k_i with the indicator values results in the following observations (See Table 15 for a count of the number of test properties which score outside the 5 % and 1 % indicators):

1. Laboratory L09 scores high k-values, witnessing poor within-laboratory consistency. For 7 out of 18 test properties, the k-value exceeds the 1 % indicator, while for another 2 test properties, the k-value exceeds the 5 % indicator but stays below the 1 % indicator. The poor within- laboratory consistency of laboratory L09 has two distinct causes: first, reporting of two inconsistent values for the two replicates with relatively low uncertainties, as is the case for Th-232 in NORM1, NORM3 and NORM5, and for Ra-226 in NORM6. Second, the reporting of two very close (or identical) values for the two replicates, but with relatively high uncertainty, for example for K-40 in NORM1, NORM2, NORM3, NORM5 and NORM6. In addition to the poor within- laboratory consistency, laboratory L09 also scores poor on between-laboratory consistency. The absolute h-values for L09 exceed the 1 % indicator for 5 out of 18 test properties (Th-232 in NORM1, NORM2, NORM5 and K-40 in NORM2 are too low while Th-232 in NORM3 is too high).
2. Laboratory L12 scores a h-value exceeding the 1 % indicator for Ra-226 in NORM1 and K-40 in NORM4, of which the reported massic activities are too low. The h-value exceeds the 5 % indicator but is lower than the 1 % indicator for the reported and low values by laboratory L12 for K-40 in NORM1 and Ra-226 in NORM3.
3. Laboratory L01 scores a h-value exceeding the 1 % indicator for reporting a too high Ra-226 value in NORM4. The reported high value for Th-232 in NORM2 results in a k-value that exceeds the 5 % indicator but stays below the 1 % indicator.
4. Laboratory L08 reports a high value for Ra-226 in NORM4 which scores a k-value that exceeds the 5 % indicator but stays below the 1 % indicator.
5. Laboratory L11 scores a h-value exceeding the 1 % indicator for reporting a too high K-40 value in NORM5.
6. Laboratory L06 scores a h-value exceeding the 1 % indicator for reporting a too low K-40 value in NORM6. The reported low value for Th-232 in NORM6 scores a k-value that exceeds the 5 % indicator but stays below the 1 % indicator.

7. For one out of 18 test properties, laboratories L01, L03 and L14 each score a k-value higher than the 5 % indicator but lower than the 1 % indicator. Laboratory L15 scores a k-value in that range for one out of 15 test properties. (L15 did not report results for NORM2).

The decisions taken based on these observations are discussed further in Section 4.6.

Table 15. Number of test properties on which the participants' results score between the 5 % and 1 % indicator and larger than the 1 % indicator, for both Mandel's within- and between-laboratory consistencies.

Participant	Number of test properties	Within-laboratory consistency, $\boldsymbol{k_i}$		Between-laboratory consistency, $\boldsymbol{h_i}$	
		Between the 5 % and 1 % indicator ([1])	Larger than the 1 % indicator ([2])	Between the 5 % and 1 % indicator ([1])	Larger than the 1 % indicator ([2])
L01	18	1	0	1	1
L02	18	0	0	0	0
L03	18	1	0	0	0
L04	18	0	0	0	0
L05	18	0	0	0	0
L06	18	0	0	1	1
L08	18	0	0	1	0
L09	18	2	7	0	4
L10	18	0	0	0	0
L11	18	0	0	0	1
L12	18	0	0	2	2
L13	18	0	0	0	0
L14	18	1	0	0	0
L15	15	1	0	0	0

1 Number of test properties where the participant scores a k- or h-value larger than the 5 % but lower than the 1 % indicator.
2 Number of test properties where the participant scores a k- or h-value larger than the 1 % indicator.

Figure 7. Between-laboratory consistency (Mandel's h statistics). The continuous and dashed lines show the indicators at the 1 % and 5 % significance level, respectively.

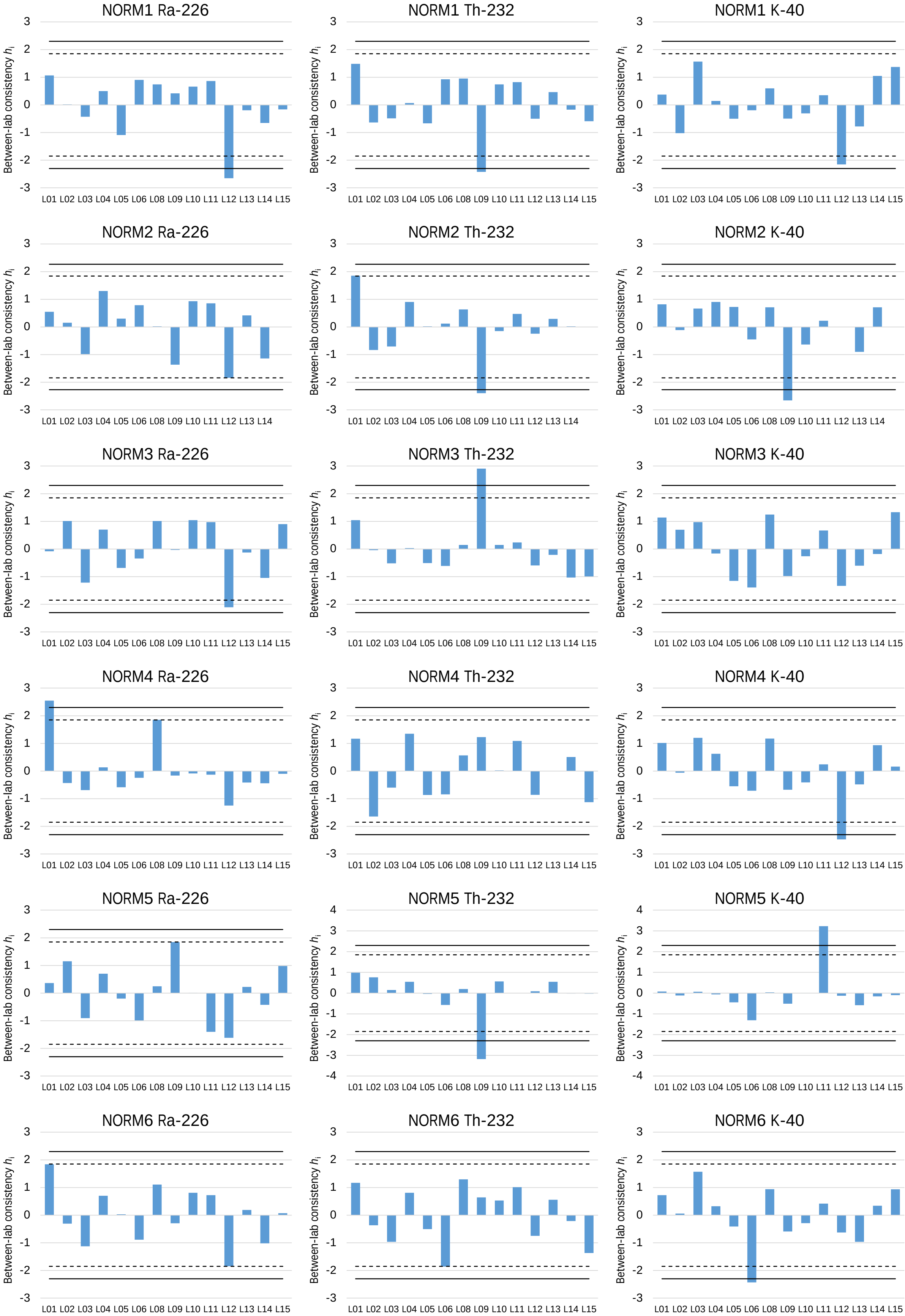
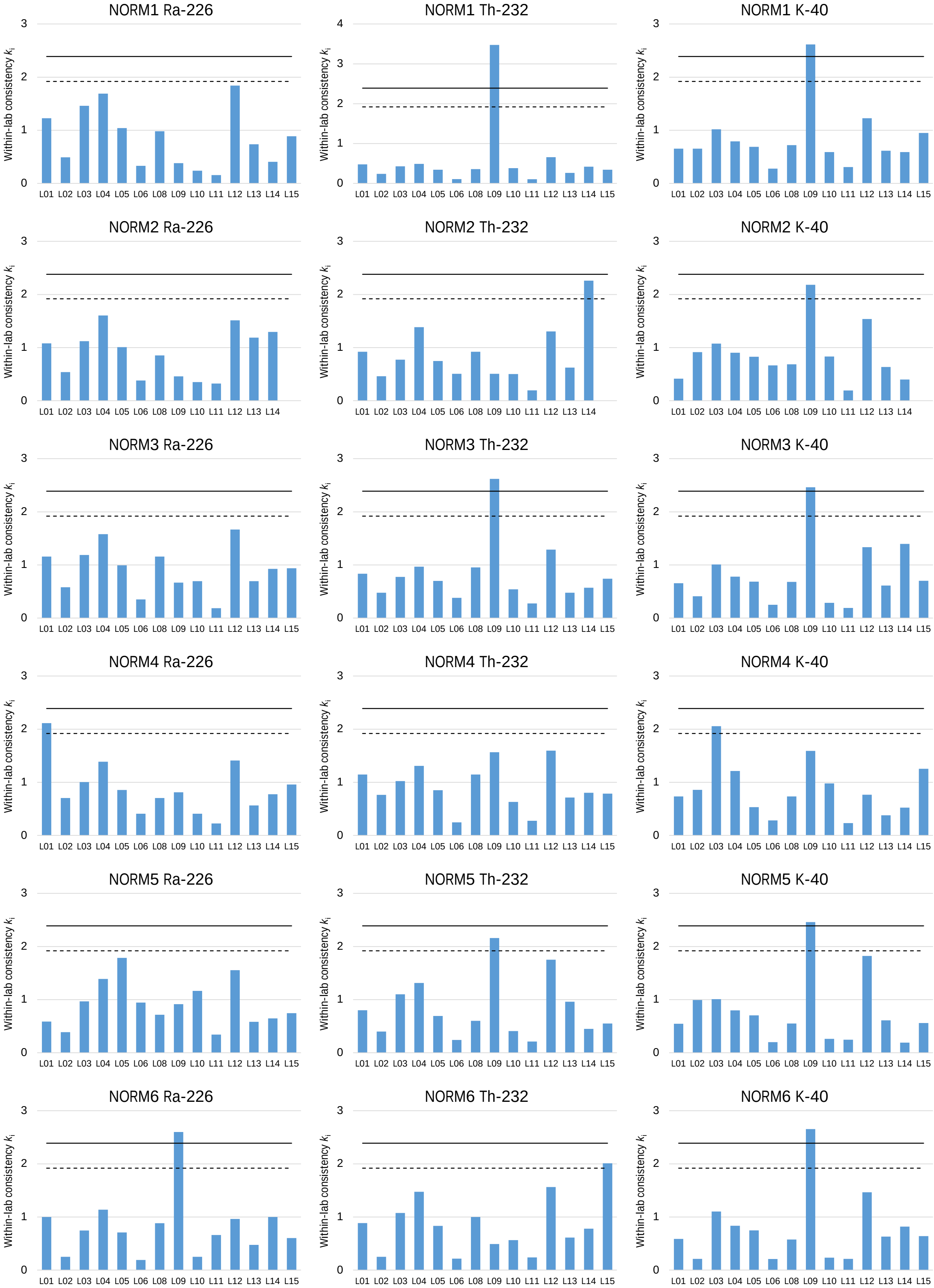


Figure 8. Within-laboratory consistency (Mandel's k statistics). The continuous and dashed lines show the indicators at the 1 % and 5 % significance level, respectively.



4.5 Scrutiny of results for stragglers and outliers

4.5.1 Cochran's test

Cochran's test is applied to detect stragglers and outliers in the standard deviations for each test property (the three radionuclides in the six materials). The test is performed on the intracell spread s_i, calculated in Section 4.3. The item tested is the highest standard deviation of the test properties reported by the laboratories.

$$s_{\max} = \max\{s_i\} \tag{8}$$

For the laboratory with the highest standard deviation, the Cochran's test statistic C is calculated following Equation (9) of ISO 5725-2 [8]:

$$C = \frac{s_{\max}^2}{\sum_{i=1}^{p} s_i^2} \tag{9}$$

The test statistic is then compared with the 5 % and 1 % critical value for n replicates and p laboratories. Depending on the test result, the item:

— Is accepted if the test statistic is less than or equal to the 5 % critical value;

— Is called a straggler if the test statistic is larger than the 5 % critical value but less than or equal to the 1 % critical value. The item tested is indicated by a single asterisk;

— Is called an outlier if the test statistic is larger than the 1 % critical value. The item tested is indicated by a double asterisk.

The critical values for Cochran's test were obtained from Table 5 of ISO 5725-2. The following stragglers and outliers are detected applying Cochran's test:

— The spread between the two results reported by laboratory L09 for K-40 in NORM6 is a straggler. One asterisk is added to the corresponding intracell spread in Section 4.3.

— The spread between the two results reported by laboratory L09 for Th-232 in NORM1 is an outlier. Two asterisks are added to the corresponding intracell spread in Section 4.3.

4.5.2 Grubbs' test

Grubbs' test is applied as in Clause 8.3.5 of ISO 5725-2 [8] to detect outliers in the cell means $\bar{y}_i$ calculated in Section 4.3. First, the test is applied for one outlying observation (Clause 8.3.5.1 of ISO 5725-2), by calculating:

$$G_{\max} = \frac{\bar{y}_{i;\max} - \bar{\bar{y}}}{s} \tag{10}$$

where $\bar{y}_{i;\max}$ is the largest cell mean amongst the laboratories for that test property, and $\bar{\bar{y}}$ the mean of the cell means over all laboratories:

$$\bar{\bar{y}} = \frac{1}{p} \sum_{i=1}^{p} \bar{y}_i \tag{11}$$

and

$$s = \sqrt{\frac{1}{p-1} \sum_{i=1}^{p} (\bar{y}_i - \bar{\bar{y}})^2} \tag{12}$$

Similarly, calculate:

$$G_{\min} = \frac{\bar{\bar{y}} - \bar{y}_{i;\min}}{s} \tag{13}$$

where $\bar{y}_{i;\min}$ is the smallest cell mean amongst the laboratories for that test property. (Note that the notation has been adjusted to reflect Section 4.3.)

If then either $G_{\min}$ or $G_{\max}$ is identified as an outlier by comparing with the 1 % critical value for p, exclude that cell mean and repeat the test at the other extreme.

If the Grubbs' test does not detect single outliers for the test property, then the test for two outliers is applied (Clause 8.3.5.2 of ISO 5725-2). Similarly as with single outliers, the test is to be done for the largest and the smallest observations, but now with the two most extreme observations on either side.

The means $\bar{y}_1, \bar{y}_2, \dots, \bar{y}_p$ are first sorted in ascending order, resulting in the data set $\bar{y}_{(1)}, \bar{y}_{(2)}, \dots, \bar{y}_{(p)}$.

The test statistic for the two largest observations is calculated according to:

$$G_{\max1,2} = \frac{s^2_{\max1,2}}{s^2_0} \tag{14}$$

where

$$s^2_0 = \sum_{i=1}^{p} (\bar{y}_i - \bar{\bar{y}})^2 \tag{15}$$

and

$$s^2_{\max1,2} = \sum_{i=1}^{p-2} (\bar{y}_{(i)} - \bar{y}_{\max1,2})^2 \tag{16}$$

and

$$\bar{y}_{\max1,2} = \frac{1}{p-2} \sum_{i=1}^{p-2} \bar{y}_{(i)} \tag{17}$$

The test statistic for the two smallest observations is calculated according to:

$$G_{\min1,2} = \frac{s^2_{\min1,2}}{s^2_0} \tag{18}$$

In which s^2_0 is calculated as in Equation (15) and

$$s^2_{\min1,2} = \sum_{i=3}^{p} (\bar{y}_{(i)} - \bar{y}_{\min1,2})^2 \tag{19}$$

and

$$\bar{y}_{\min1,2} = \frac{1}{p-2} \sum_{i=3}^{p} \bar{y}_{(i)} \tag{20}$$

(Note that the index in Equation (19) of ISO 5725-2 should run from $i = 3$ to p. Equation 19 here is the correct form.)

The critical values for Grubbs' test for one and two outlying observations were obtained from Table 6 of ISO 5725-2.

Applying the single outlier test to the data in Section 4.3 yields the following outliers and stragglers:

- L09 is the laboratory with the highest cell mean for Th-232 in NORM3, which is an outlier;
- L09 is the laboratory with the lowest cell mean for Th-232 in NORM5 which is an outlier;
- L11 is the laboratory with the highest cell mean for K-40 in NORM5 which is an outlier;
- L01 has the highest mean for Ra-226 in NORM4 which is a straggler;
- L09 has the lowest mean for K-40 in NORM2 which is a straggler;
- L12 has the lowest mean for Ra-226 in NORM1 which is a straggler.

The stragglers and outliers are identified by one and two asterisks, added to the cell means in the tables of Section 4.3.

After removing the three outliers from the data of the respective test property, the opposite single outlier test is done on the remaining data (e.g. the outlier test for the lowest observation is performed when the highest was identified as an outlier). This leads to the following:

- The lowest cell mean for K-40 in NORM5 is provided by laboratory L06 and is identified as an outlier;
- The lowest cell mean for Th-232 in NORM3 is provided by laboratory L14, but passes the test as being normal;
- The highest cell mean for Th-232 in NORM5 is provided by laboratory L01, but passes the test as being normal.

Similarly, the additional outlier is identified with two asterisks in Section 4.3.

When the outlying data provided by provided by laboratory L06 for K-40 in NORM5 is removed, the highest value in the remaining data passes the test as being normal.

Grubbs' test for double outliers is now applied to the test properties where no single outliers are detected, which are all data sets except Th-232 in NORM3, Th-232 in NORM5 and K-40 in NORM5. This leads to the following (double) outliers and stragglers:

- The two highest cell means for Ra-226 in NORM4 were reported by laboratory L01 and L08. They are identified as outliers;
- The two lowest cell means for Ra-226 in NORM1 were reported by laboratory L12 and L05 and are identified as stragglers;
- The two lowest cell means for K-40 in NORM2 were reported by laboratory L09 and L13 and are identified as stragglers.

After removing the results from laboratory L01 and L08, the Grubbs' test for double outliers is repeated for Ra-226 in NORM4 to check if the two lowest cell means are outliers. The lowest two cell means for Ra-226 in NORM4 pass the test as being normal. Again, the outliers and stragglers are marked in the tables of Section 4.3. Note that the cell mean of laboratory L01 for Ra-226 in NORM4 was already marked as a straggler but now becomes an outlier. The mean result from laboratory L12 for Ra-226 in NORM1 was also marked as straggler, and stays a straggler. The latter also applies to the cell mean for K-40 in NORM2 reported by laboratory L09.

4.6 Decisions on rejecting data

The decision to reject data is based on the criteria in ISO 5725-2. Table 16 summarises the outcome of the Cochran's test to identify stragglers and outliers in intracell spread and the Grubbs' test to detect stragglers and outliers in the cell means (Section 4.5). The table is sorted per laboratory and material.

Table 16. Results of straggler and outlier detection following Cochran's and Grubbs' test for in intracell spread and cell means, respectively.

Participant	Material	Radionuclide	Test	Result	Outcome
L01	NORM4	Ra-226	Grubbs' single	Straggler	Reject
			Grubbs' double	Outlier	
L05	NORM1	Ra-226	Grubbs' double	Straggler	
L06	NORM5	K-40	Grubbs' single	Outlier	Reject
L08	NORM4	Ra-226	Grubbs' double	Outlier	Reject
L09	NORM1	Th-232	Cochran's	Outlier	Reject
	NORM2	K-40	Grubbs' single	Straggler	
			Grubbs' double	Straggler	
	NORM3	Th-232	Grubbs' single	Outlier	Reject
	NORM5	Th-232	Grubbs' single	Outlier	Reject
	NORM6	K-40	Cochran's	Straggler	
L11	NORM5	K-40	Grubbs' single	Outlier	Reject
L12	NORM1	Ra-226	Grubbs' single	Straggler	
			Grubbs' double	Straggler	
L13	NORM2	K-40	Grubbs' double	Straggler	

Seven results are rejected as outliers identified by Cochran's and Grubbs' test. The stragglers are retained.

The key findings on the application of Mandel's statistics (Section 4.4) to assess the within- and between-laboratory consistency are:

1. A poor within- and between-laboratory consistency on the values reported by laboratory L09 (observation 1):
   - For 7 out of 18 test properties, the k-value exceeds the 1 % indicator, while for another 2 test properties, the k-value exceeds the 5 % indicator but stays below the 1 % indicator.
   - The absolute h-values for L09 exceed the 1 % indicator for 5 out of 18 test properties.
2. The results reported by laboratory L12 score normal on the within-laboratory consistency but a bit higher than what can statistically be expected on the between-laboratory consistency (observation 2).
3. On statistical ground, there are no reasons to doubt the between- and within-laboratory consistency for the results reported by the other laboratories (observations 3 to 7).

Considering that it is very unlikely that the poor within- and between-laboratory consistencies of the results reported by laboratory L09 are of random cause and considering that three out of seven detected outliers by Cochran's and Grubbs' test are reported by that laboratory, it is decided that all results reported by laboratory L09 are rejected from the study following Clause 8.6 of ISO 5725-2.

Repeating the Cochran's test without the results of laboratory L09 leads to the finding that now laboratory L12 has the highest intracell spread in 10 out of 18 test properties. This is also visible in the somewhat poorer performance of laboratory L12 on the between-laboratory consistency. However, because the Cochran's test statistic is lower than the 5 % critical value, the intracell spread is considered normal and the data from L12 is retained.

4.7 Repeatability and reproducibility standard deviation

In total, 15 laboratory reported results, but laboratory L07 did not use the required detector type and therefore was rejected from the analysis. There are six materials to be analysed, but laboratory L15 did not provide data for material NORM2. For each material, the labs received two sample replicates and had to determine the massic activity of three radionuclides. The mean of the two replicates and spread was calculated as explained in Section 4.3, yielding to a total of 249 results.

The means and spread were tested for consistency (Section 4.4) and for stragglers and outliers (Section 4.5). Based on the outcome of the tests, as discussed in Section 4.6, all results from laboratory L09 and four additional results from other laboratories were rejected on statistical grounds. The total number of results retained is 230. These remaining results are used to determine the repeatability and reproducibility of the method. For each test property, the general mean, repeatability variance, between-laboratory variance and the reproducibility variance is calculated as follows:

The general mean is calculated according to Equation (23) of ISO 5725-2, which reduces to (and noting that p depends on the test property):

$$\hat{m} = \bar{\bar{y}} = \frac{\sum_{i=1}^{p} \bar{y}_i}{p} \tag{21}$$

The repeatability variance is given by Equation (25) of ISO 5725-2, which becomes:

$$s_r^2 = \frac{\sum_{i=1}^{p} s_i^2}{p} \tag{22}$$

The between-laboratory variance is calculated following Equation (26) or Equation (30) of ISO 5725-2:

$$s_L^2 = \frac{1}{p-1} \sum_{i=1}^{p} (\bar{y}_i - \bar{\bar{y}})^2 - \frac{s_r^2}{2} \tag{23}$$

If s_L^2 is negative due to random effects, it is set to zero.

The reproducibility variance is given by Equation (31) of ISO 5725-2:

$$s_R^2 = s_r^2 + s_L^2 \tag{24}$$

The square root of the three variances yields the three corresponding standard deviations, which can also be expressed relatively, divided by the general mean.

4.8 Results - the precision of WD EN 17216

4.8.1 Precision for each test property (material and radionuclide)

Table 17 presents the results of the determination of the precision of WD EN 17216 [4], for each test property. For this evaluation, the massic activities (in dry mass) of three radionuclides in six construction products had to be determined. 15 laboratories participated, numbered from L01 to L15. The data provided by participant L07 were not considered since the participant did not use a semiconductor gamma-ray spectrometer. The results reported by laboratory L09 were rejected from further calculations due to the poor within- and between-laboratory consistencies of the results reported, demonstrated by three detected outliers. Four other outliers were detected and rejected: the results reported by L01 and L08 for Ra-226 in NORM4, and the results reported by L06 and L11 for K-40 in NORM5.

Table 17. Calculated mean $\hat{m}$, between-laboratory standard deviation s_L, repeatability standard deviation s_r and the reproducibility standard deviation s_R. The mean and (absolute) standard deviations are massic activities of the specified radionuclide in the material, expressed in becquerel per kilogram dry mass, at the reference date of 01/03/2023.

Material	Radio-nuclide	p (1)	$\hat{m}$ [Bq/kg]	s_L [Bq/kg]	s_r [Bq/kg]	s_R [Bq/kg]	RSD_L (2) [%]	RSD_r (2) [%]	RSD_R (2) [%]
NORM1	Ra-226	13	81.7	6.0	3.0	6.7	7.4	3.6	8.2
	Th-232	13	50.1	2.3	2.3	3.2	4.6	4.5	6.5
	K-40	13	187	12	8	15	6.6	4.3	7.9
NORM2	Ra-226	12	31.8	3.5	1.4	3.7	11	4.2	12
	Th-232	12	24.1	1.0	1.6	1.9	4.1	6.5	7.7
	K-40	12	333	14	14	20	4.1	4.2	5.9
NORM3	Ra-226	13	31.3	3.2	1.2	3.5	10	4.0	11
	Th-232	13	22.5	1.3	1.1	1.7	5.7	4.9	7.5
	K-40	13	312	15	13	21	5.0	4.3	6.6
NORM4	Ra-226	11	20.7	1.8	0.9	2.0	8.6	4.2	9.6
	Th-232	13	36.9	2.1	1.7	2.7	5.7	4.7	7.4
	K-40	13	58.6	9.2	5.4	10.7	16	9.3	18
NORM5	Ra-226	13	115	18	5	19	16	4.8	16
	Th-232	13	63.1	5.2	3.0	6.0	8.2	4.7	9.4
	K-40	11	1433	0	66	66	0	4.6	4.6
NORM6	Ra-226	13	49.9	9.1	2.1	9.3	18	4.2	19
	Th-232	13	56.2	3.4	2.9	4.5	6.1	5.2	8.0
	K-40	13	1224	64	47	79	5.2	3.8	6.4

1 p denotes the number of observations (the number of participants whose reported values were retained).

2 RSD is the relative standard deviation and equals the standard deviation s divided by the mean $\hat{m}$.

4.8.2 Relationship between the precision and the mean, effect of sample heterogeneity

To further investigate a possible relationship between the precision and the mean $\hat{m}$, the test properties are grouped per radionuclide, thereby making abstraction of the material. (Indeed, the method is intended to be applicable to any construction product.) Grouped per radionuclide, the plots in Figure 9 show the relative standard deviations from Table 17 as a function of the mean massic activity reported by the participants. The dashed line on each plot shows (as relative standard deviation) the maximum between-sample variability $u_{bb;\max}$ that was observed over the six materials during the homogeneity study [5].

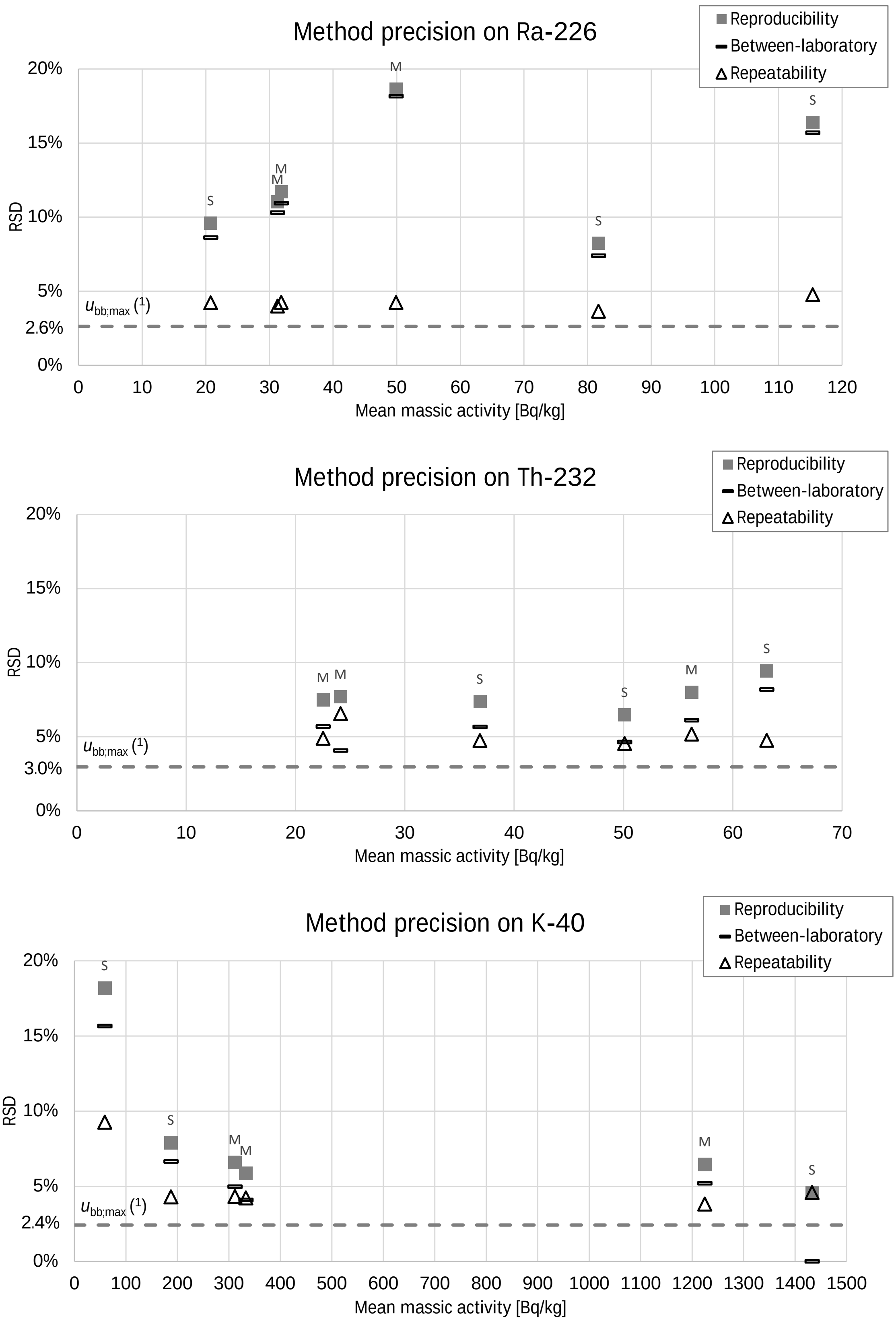
For each of the three radionuclides, two hypothesis tests were conducted to test if there is a correlation between the relative repeatability and the mean massic activity, and between the relative reproducibility and the mean massic activity. The null hypothesis is H_0: There is no correlation between relative repeatability/ reproducibility and mean. For both the repeatability and reproducibility all for three radionuclides, the relationship is not statistically significant at a significance level of α = 0.05. Hence, the null hypothesis cannot be rejected.

To assess if material heterogeneity forms an inseparable part of the variability of the test results, labels S and M are added to Figure 9. Data points from materials with a single component are indicated with an S (NORM1, NORM4 and NORM5), while an M indicates multiple component materials (NORM2, NORM3 and NORM6).

A number of observations can be made from these graphs:

- For any of the three radionuclides, the maximum between-sample variation $u_{bb;\max}$ obtained from the homogeneity study is lower than the repeatability. This is expected since the repeatability is calculated from the spread between two results, which is averaged over all the 11, 12 or 13 participants (See Equation (22)). On the other hand, the between-sample variability is determined from 10 samples and (normally) 3 sub-samples, measured by one laboratory [5]. It also means that the impact of between-sample variation on the repeatability is small.
- For Ra-226, the repeatability ranges from 3.6 % to 4.8 % (triangles in Figure 9, see also Table 17) over all the levels of massic activity tested. The repeatability is fair and consistent over the levels. The between-laboratory variability on the other hand, ranges from 7.4 % to 18 % and is not consistent over the levels. This could indicate issues with the sealing of the test specimen container or could be caused by not completely filling the test specimen container.
- For Th-232, the repeatability ranges from 4.5 % to 6.5 % (triangles in Figure 9, see also Table 17) over all the levels of massic activity tested. Also here, the repeatability is fair, albeit about 1 % larger than for Ra226, and consistent over the levels. The between-laboratory variability ranges from 4.1 % to 8.2 %, which is fair.
- For K-40, the repeatability ranges from 3.8 % to 9.3 % (triangles in Figure 9, see also Table 17) over the levels of massic activity tested. As observed from the tables with the mean results in Section 4.3 and the reference values provided further in Table 19, the materials NORM4 (about 60 Bq/kg) and NORM1 (about 180 Bq/kg) contain considerably less K-40 than the other materials (about 315 Bq/kg for NORM2 and NORM3, 1200 Bq/kg for NORM6 and 1350 Bq/kg for NORM5). In particular for NORM4, the higher repeatability may be explained by poorer counting statistics. In addition, the ratio of the Th-232 to K-40 massic activity is around 66 % for NORM4 and 28 % for NORM1 while for NORM2 and NORM3 the ratio is 7.3 % and for NORM5 and NORM6 the ratios are 5.0 % and 4.8 % respectively. Considering that Th-232 is determined via Ac-228, the impact of ignoring the interference of the 1459 keV peak of Ac-228 with the 1461 keV peak of K-40 is much larger for NORM4 and NORM1 than for the other four materials. Assuming that each participant analysed the two replicates in the same way, the impact of ignoring the interference will manifest itself in an increased between-laboratory variability for K-40 in NORM4 and NORM1, but not as an increased within-laboratory variability. For the other levels than the lowest one (NORM4), the repeatability is fair and consistent. The between-laboratory variability ranges from 0 % to 16 % (or 6.6 % when the lowest level is not considered). Not considering the lowest level, the between-laboratory variability is also fair for K-40.
- No different precision is noted for any of the radionuclides in multiple-component materials (M) compared to single-component materials (S).

Figure 9. Relation between the precision of the method and the mean massic activity in dry mass at the reference date of 01/03/2023. The precision is shown as a relative standard deviation of the repeatability, the between-laboratory variability and the reproducibility. The dashed line represents the maximum between-sample variability obtained from the homogeneity study. Single-component are indicated with S, multiple-component materials with M.



[1] $u_{bb;max}$ is the maximum uncertainty related to possible between-sample variation. It is the maximum u_{bb} obtained from the homogeneity study [5] over all six materials, for the nuclide shown in the plot.

4.8.3 Final results of the precision evaluation

Adding the between-laboratory variance and the repeatability variance yields the reproducibility variance (Equation (24)). Since it cannot be statistically rejected that there is no correlation between the variability and the levels of massic activity, an average repeatability and reproducibility is calculated and presented in Table 18. The uncertainty on the average is the maximum of the internal and external standard deviation, calculated following Equation (8) from WD EN 17216 (See also Section 4.3).

Table 18. Results of the determination the precision of WD EN 17216 following the method specified in ISO 5725-2. Standard uncertainties are shown in parentheses.

Radionuclide	Validity range (1) [Bq/kg]	Repeatability $\mathbf{RSD}_r$ [%]	Reproducibility $\mathbf{RSD}_R$ [%]
Ra-226	20 – 120	4.2 (1.7)	12.6 (5.4) (2)
Th-232	20 – 65	5.1 (2.1)	7.7 (3.2)
K-40	50 – 1450	5.1 (2.2)	8.3 (5.0)

1 The precision data presented in this report are only valid within the levels covered by the collaborative assessment.

2 The reproducibility for Ra-226 is dominated by the high between-laboratory variability, which could indicate issues with sealing of the test specimen container.

5 Determination of trueness

5.1 Methodology

The robustness of the method has been verified already [3]. However, an additional evaluation of the trueness, taking advantage of the results of the collaborative assessment and the answers that the participants provided in the reporting survey, may help to verify the impact of *not* following certain normative requirements in the method. The latter is further discussed in Section 6.

In addition, construction products may have more than one component. When the components have a different attenuation (elemental composition or density) and when they are not homogeneously mixed in the test specimen container, there is a risk that the detection efficiency for gamma-ray spectrometry is biased. Both have been further investigated and are discussed in this section, after the calculation of the bias.

The trueness is determined by calculating the bias of the method, following ISO 5725-4 [9]. The bias is given by Equation (16) in the standard:

$$\hat{\delta} = \bar{\bar{y}} - \mu \tag{25}$$

Where $\bar{\bar{y}}$ is the general mean of the test property over the laboratories and calculated following Equation (5). Note that the bias is calculated *after* rejecting outliers on the precision using Cochran's and Grubbs' test statistics. μ is the reference value of the test property. The variation of the estimation of the bias is estimated as in Equation (18) of the standard, which reduces to the following for two replicates ($n = 2$):

$$s_{\hat{\delta}} = \sqrt{\frac{s_R^2 - \frac{1}{2}s_r^2}{p} + u^2(\mu)} \tag{26}$$

A 95 % confidence interval for the bias of the measurement method is given by Equation (19) of the standard, which becomes:

$$[\hat{\delta} - \frac{s_R^2}{s_r}, \hat{\delta} + \frac{s_R^2}{s_r}] \tag{27}$$

The standard further states that, when this interval includes zero, the bias is insignificant at $\alpha = 0.05$. Otherwise, the bias is significant.

To facilitate comparison between radionuclides and materials, it is convenient to express the bias, the variation of the bias and the confidence interval in a relative way, by dividing by the reference value.

5.2 Reference values

The bias cannot be calculated without the reference values. Table 19 shows the reference values μ of the massic activities of the six materials selected for the validation study (from [5]). The combined standard uncertainties $u(\mu)$ are shown as absolute values (in Bq/kg) in parentheses. Since all laboratories were asked to report the activity per dry mass, is was not required to determine a reference value for the water content.

Table 19. Reference values of the massic activities of the six materials selected for the validation study.

Material	Reference values for massic activity (1) $\boldsymbol{\mu}\ (\boldsymbol{u}(\boldsymbol{\mu}))$ [Bq/kg]		
	Ra-226	Th-232	K-40
NORM1 – Cement	85.8 (2.4)	50.3 (1.0)	182 (4)
NORM2 – Expanded clay blocks (piece)	32.4 (1.0)	23.1 (0.6)	315 (10)
NORM3 – Expanded clay blocks (powder)	32.7 (1.1)	22.7 (0.5)	313 (5)
NORM4 – Plaster	22.0 (0.7)	38.6 (1.8)	57.0 (2.1)
NORM5 – Aggregates	140 (8)	67.4 (3.0)	1352 (50)
NORM6 – Milled concrete	55.9 (4.0)	58.1 (2.5)	1194 (40)

1 The massic activity in dry mass at the reference date of 01/03/2023. The combined standard uncertainties ($k = 1$) are shown as absolute values (in Bq/kg) in parentheses.

Source: Paepen, J., Van Ammel, R., Hult, M., Marissens, G., Stroh, H., Emteborg, H., Seghers, J., NORMCONSTRUCT: Results of material screening and radon leakage rate testing – Materials used in the collaborative assessment experiment for the validation of the precision of CEN/TS 17216, European Commission, Geel, 2023, JRC135452

5.3 Estimation of the bias

5.3.1 Bias for each test property

The result of the estimation of bias for all the test properties individually is presented in Table 20. (The general mean can be found in Table 17 and the reference values in Table 19.) In Figure 10, the bias for each test property is shown sorted from low (negative) to high (positive). The colour and shape of the data points represent the radionuclide.

Following observations are made:

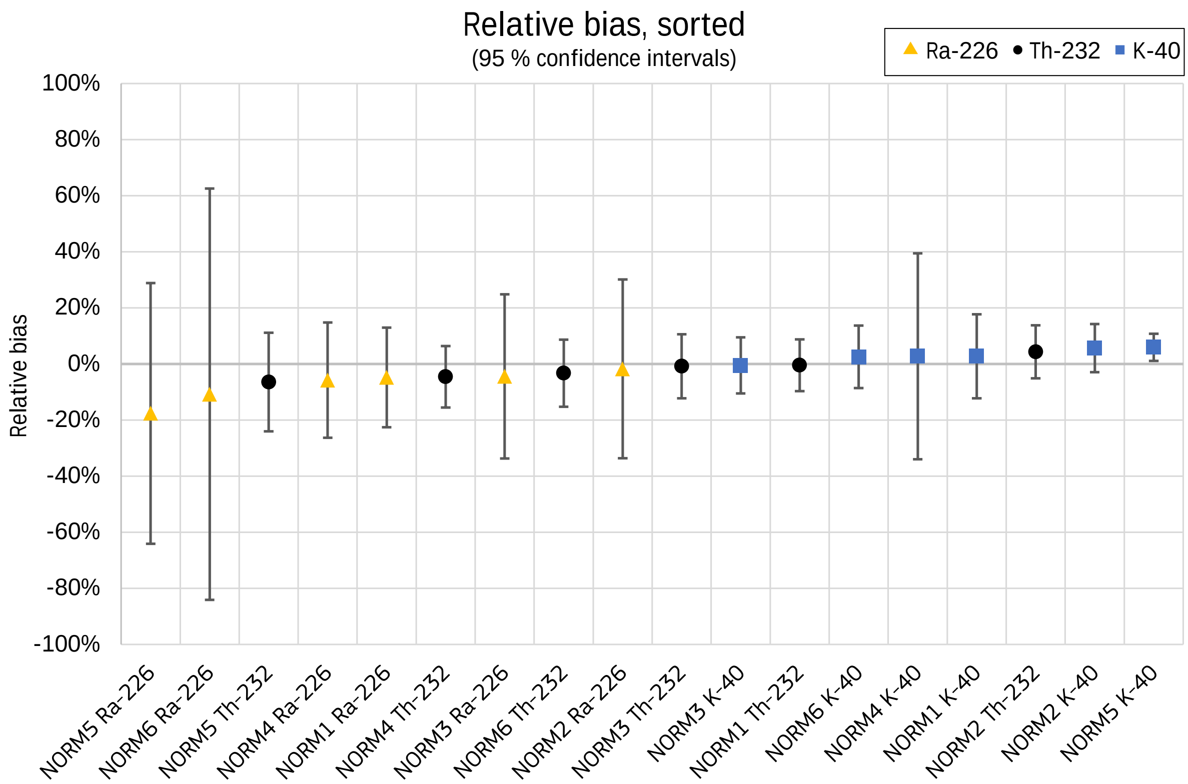
- The measurement of Ra-226 results in the largest bias and this bias is negative, but insignificant at a significance level of $\alpha = 0.05$, for all the materials. For Ra-226, the estimate of the variation of the bias $s_{\hat{\delta}}$ is dominated by the reproducibility standard deviation s_R, which is higher than for the other radionuclides due to the high between-laboratory standard deviation s_L compared to the repeatability s_r. The poorer between-laboratory consistency for Ra-226 results in a larger estimate of the variation of the bias, which renders the bias insignificant;
- The bias of the determination of K-40 in NORM5 is statistically significant at a significance level of $\alpha = 0.05$, because the 95 % confidence interval does not contain the zero, although the difference is small. As can be seen from Equation (23), random effects resulted in negative between-laboratory variance and therefore s_L was set to zero, which minimises the estimate of the variation of the bias $s_{\hat{\delta}}$, calculated from Equation (26). Would $s_{\hat{\delta}}$ be larger than 52.8 Bq/kg instead of the current value of 51.9 Bq/kg (rounded to 52 Bq/kg in Table 20), then the 95 % confidence interval would include zero and the bias would no longer be significant;
- All other biases than the bias for K-40 in NORM5 are insignificant at a significance level of $\alpha = 0.05$;
- Over all materials, the largest bias is -18 % for Ra-226, -6.4 % for Th-232 and 6.0 % for K-40. For all three radionuclides, the largest bias is found with material NORM5.

Table 20. Results of the estimation of the bias.

Material	Radio-nuclide	Absolute bias [Bq/kg]			Relative bias [%]		Significance of bias ($\alpha = 0.05$)
		Bias $\hat{\delta}$	Variation of bias (1) $s_{\hat{\delta}}$	95 % confidence interval	Relative bias $\hat{\delta}/\mu$	95 % confidence interval (2)	
NORM1	Ra-226	-4.1	3.0	[-19.3, 11.1]	-4.8	[-23, 13]	Insignificant
	Th-232	-0.2	1.3	[-4.8, 4.4]	-0.41	[-9.6, 8.8]	Insignificant
	K-40	5	6	[-22, 32]	2.8	[-12, 18]	Insignificant
NORM2	Ra-226	-0.6	1.4	[-10.9, 9.8]	-1.7	[-34, 30]	Insignificant
	Th-232	1.0	0.7	[-1.2, 3.2]	4.4	[-5.1, 14]	Insignificant
	K-40	18	11	[-9, 45]	5.7	[-2.9, 14]	Insignificant
NORM3	Ra-226	-1.4	1.4	[-11.0, 8.1]	-4.4	[-34, 25]	Insignificant
	Th-232	-0.2	0.6	[-2.8, 2.4]	-0.77	[-12, 11]	Insignificant
	K-40	-1	7	[-33, 30]	-0.43	[-10, 9.6]	Insignificant
NORM4	Ra-226	-1.3	0.9	[-5.8, 3.3]	-5.7	[-26, 15]	Insignificant
	Th-232	-1.7	1.9	[-6.0, 2.5]	-4.5	[-15, 6]	Insignificant
	K-40	1.6	3.5	[-19.3, 22.5]	2.8	[-34, 40]	Insignificant
NORM5	Ra-226	-25	10	[-90, 40]	-18	[-64, 29]	Insignificant
	Th-232	-4	3	[-16, 8]	-6.4	[-24, 11]	Insignificant
	K-40	81	52	[15, 146]	6.0	[1.1, 11]	Significant
NORM6	Ra-226	-6.0	4.7	[-47.0, 35.0]	-11	[-84, 63]	Insignificant
	Th-232	-1.9	2.7	[-8.8, 5.1]	-3.2	[-15, 8.7]	Insignificant
	K-40	30	45	[-103, 163]	2.6	[-8.6, 14]	Insignificant

1 The variation of the bias is a standard deviation.
2 The limits of the confidence intervals are relative values. For example, [-23, 13] shall be read as [-23 %, 13 %].

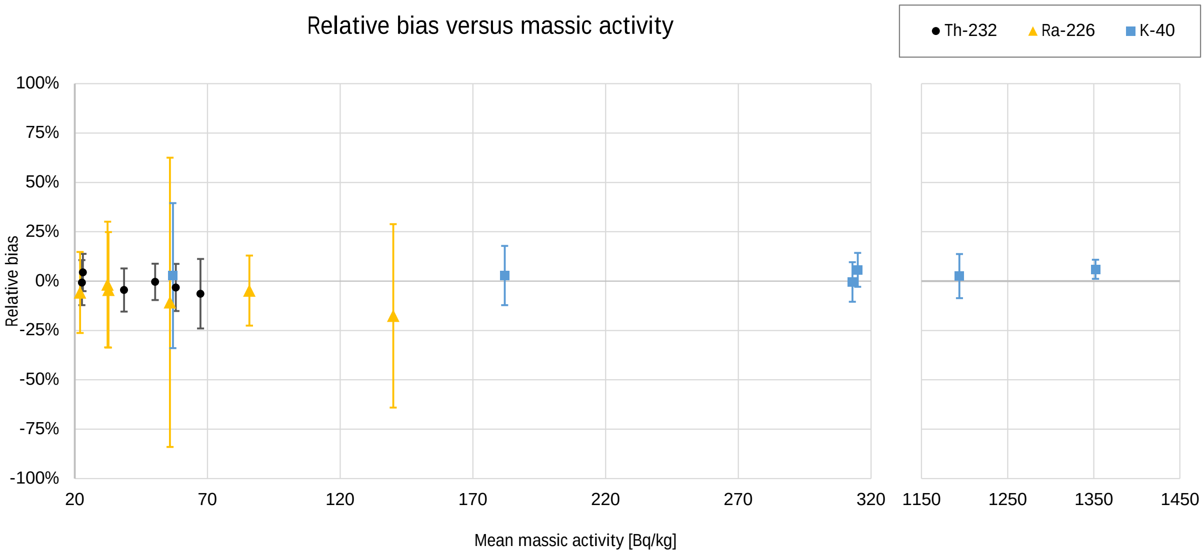
Figure 10. Plot of the relative bias, sorted. The colours and shapes of the data points reflect the radionuclide.



5.3.2 Relationship between the relative bias and the mean

Making abstraction of the material, Figure 11 plots the relative bias (the bias divided by the reference values) as a function of the mean massic activity. For each of the three radionuclides, a hypothesis test was conducted to test if there is a correlation between the relative bias and the mean massic activity. The null hypothesis is H_0: There is no correlation between relative bias and mean. For all three radionuclides, the relationship is not statistically significant at a significance level of $\alpha = 0.05$. Hence, the null hypothesis cannot be rejected.

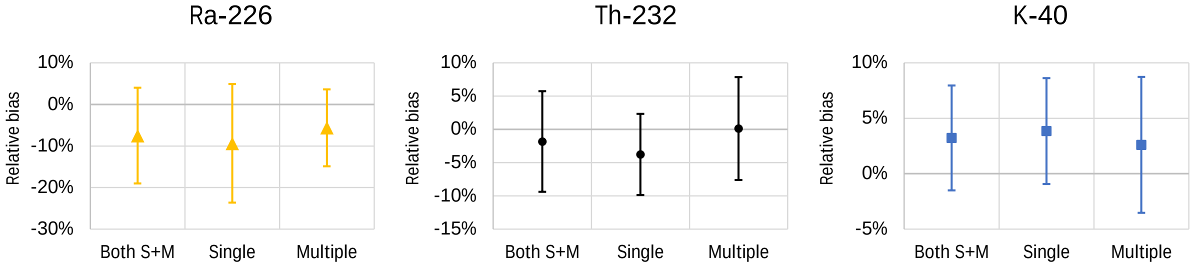
Figure 11. Plot of the relative bias (the bias divided by the reference values) as a function of the mean massic activity in dry mass. The error bars are 95 % confidence intervals.



5.3.3 Bias for heterogeneous materials

The materials NORM1, NORM4 and NORM5 could be considered as single-component materials while the other materials NORM2, NORM3 and NORM6 have multiple-components. For each radionuclide, the average bias was calculated for the three single-component materials and for the three multiple-component materials separately. Both averages were then compared with each other and with the average over all the six materials (See Figure 12). For none of the radionuclides, there is a significant difference between the average relative bias of single- and multiple-component materials.

Figure 12. The relative bias averaged for single-component materials (NORM1, NORM4 and NORM5), for multiple-component materials (NORM2, NORM3 and NORM6) and the pooled relative bias for both single and multiple-component materials. The error bars are expanded uncertainties with a coverage factor of $k = 2$.



5.3.4 Average bias

Since no correlation between the bias and the levels of massic activity can be demonstrated (See Section 5.3.2) and no difference is found between homogeneous and heterogeneous materials (See Section 5.3.3), the average bias is calculated for the tree radionuclides and shown in Table 21. The uncertainty on the average is the maximum of the internal and external standard deviation, calculated following Equation (8) from WD EN 17216 (See also Section 4.3).

Table 21. Results of the determination the bias of WD EN 17216 following the method specified in ISO 5725-2. Standard uncertainties are shown in parentheses.

Radionuclide	Validity range ([1]) [Bq/kg]	Relative bias $\hat{\delta}/\mu$ [%]
Ra-226	20 – 120	-7.5 (5.8)
Th-232	20 – 65	-1.8 (3.8)
K-40	50 – 1450	3.2 (2.4)

[1] The bias presented in this report is only valid within the levels covered by the collaborative assessment.

6 Impact of not following normative requirements of the method

6.1 Methodology

Contrary to the assessment of the precision in Section 4 and the trueness in Section 5, the data in this section includes the data from all fourteen participants. No outliers were rejected. The data reported by participant L09 is included. L07 remains rejected for the same reason as mentioned earlier in Section 3.3.

In order to compare between participants that did or did not follow a requirement specified in the method, two means are calculated: the mean result over all participants who adhered to the requirement, and the mean result over all participants who did not adhere to the requirement. The uncertainties on these means are the maximum of the internal and external standard deviations, calculated as described in Section 4.3.

The two means are then compared with each other and with the overall mean for that test property, calculated over all participants. The data is visually presented on a series of plots including also the reference value of the test property. The plots at the left are sorted in increasing massic activity. The outliers identified earlier by Cochran's and Grubbs' test in Section 4 are included in the calculation of the mean. On the plots, outliers are indicated with a double asterisk. The plots at the right allow visual comparison between the two means and with the overall mean. The data is also presented in tables.

> the data in this section includes participant L09 and the outliers identified earlier

Note that the error bars on the plots are shown with a coverage factor of $k = 2$ while the numbers between parentheses in the tables are standard uncertainties ($k = 1$).

Note also that, since the data in this section includes the outliers which were rejected for calculating the precision and trueness, the mean and spread is different in this section for the test properties that contain an outlier.

6.2 Maximum particle size: crushing, sieving and homogenisation

Clause 6.3.2.1 of WD EN 17216 requires that the test samples have a maximum particle size of 2 mm, unless the samples are flexible (mineral wool, etc.). Particle size shall be reduced by breaking (if necessary) and crushing, until all material passes through a 2 mm sieve. Then, the sieved material shall be homogenised using a mixer or manually with a spoon.

In the first survey, three participants (L02, L12 and L14) answered that they do not have the necessary equipment to process samples of construction products (e.g. by crushing, pulverising, sieving). From the answers to Q 2.1 and Q 2.2 of the reporting questionnaire, it appears that the three laboratories are lacking equipment to crush or break bigger samples: the laboratories had to manually break the pieces of expanded clay blocks using a hammer or a press. However, this did not prevent them from processing the materials NORM2 (pieces of expanded clay blocks) and NORM5 (aggregates) until a particle size of maximum 2 mm. The other materials did not require further milling.

Participant L15 did not report results for material NORM2 (pieces of expanded clay blocks). All other participants listed the method and/or equipment used for crushing. One of the 13 participants who analysed NORM2 indicated that they relied on an external service to crush the material. Equipment and methods used for crushing are a hammer (3x reported), crushing with a press (1x), drilling (1x), jaw crusher (7x), ring crusher (1x), ball mill (4x), disk mill (1x), widia granulator (1x) and agate mortar (1x).

Q 2.3 polled if the participants used sieves. Five laboratories replied that they did not sieve the material. Nine participants sieved to the required particle size. Four participants milled and sieved further to a particle size below 0.5 mm, while this is not required.

In reply to Q 2.4, the participants had to provide information on how the sieved material was homogenised. Participant L13 answered that the material was not homogenised. Homogenisation techniques used by the other participants are manual stirring (5x reported), shaking (3x), mixing (2x), rotation on a roller table (1x) and mechanical homogenisation (1x). One participant indicated to have "followed the protocol". It was not further clarified what that means.

As can be seen in Table 22, participants who crushed and sieved to the required particle size also homogenised the material. Green cells indicate that the participant followed the normative requirement.

Table 22. Sieving and homogenisation of the sample materials by the participants (Clause 6.3.2.1). Cells are coloured green where the participants followed the normative requirements from the draft standard.

Participant	Q 2.1, 2.2, 2.3: Crushed and sieved to < 2 mm?	Q 2.1, 2.2, 2.3: Crushed and sieved to < 0.5 mm? (Not required)	Q 2.4: Homogenised?
L01	NO	NO	YES
L02	YES	YES	YES
L03	YES	NO	YES
L04	NO	NO	YES
L05	YES	YES	YES
L06	NO	NO	YES
L08	NO	NO	YES
L09	YES	NO	YES
L10	YES	YES	YES
L11	YES	YES	YES
L12	YES	NO	YES
L13	NO	NO	NO
L14	YES	NO	YES
L15	YES	NO	YES

Figure 13 to Figure 18 show separately the massic activities reported by the participants who applied and who did not apply the requirement of milling and sieving the materials to a particle size less than 2 mm. Table 23 shows the data in numerical form.

Only materials NORM2 and NORM5 required crushing and sieving. Therefore, an impact of crushing and sieving could be expected for NORM2 and NORM5. Nevertheless, observing the data obtained from the collaborative assessment, the impact of crushing and sieving to particle sizes less than 2 mm cannot be proven.

In the same way, the impact of homogenising the materials has been assessed. Only participant L13 indicated that the test specimen was not homogenised. The means are shown in Table 24 and in Figure 19 to Figure 24. Also here, the impact of homogenising the material cannot be demonstrated.

based on the available data, the impact of crushing and sieving particles to sizes less than 2 mm and of homogenising the test specimen cannot be proven

Table 23. Mean results from participants who indicated that they yes/no crushed and sieved the material to particle sizes less than 2 mm.

Material ([1])	Radionuclide	Mean massic activity [Bq/kg] ([2])		
		Mean	Crushed and sieved to < 2 mm?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	85.6 (3.0)	79.8 (6.6)
	Th-232	49.4 (3.8)	52.3 (2.0)	47.8 (3.6)
	K-40	187 (13)	187 (7)	186 (16)
NORM2*	Ra-226	31.4 (3.8)	33.8 (1.8)	30.0 (4.1)
	Th-232	23.7 (2.0)	25.3 (1.4)	22.7 (1.8)
	K-40	327 (27)	333 (22)	323 (30)
NORM3	Ra-226	31.3 (3.2)	32.0 (1.9)	30.8 (3.8)
	Th-232	23.1 (2.6)	23.3 (1.6)	23.0 (3.1)
	K-40	310 (18)	311 (21)	310 (18)
NORM4	Ra-226	22.7 (5.0)	26.6 (6.7)	20.5 (1.9)
	Th-232	37.1 (2.5)	38.2 (2.2)	36.5 (2.5)
	K-40	58 (10)	61 (8)	56 (10)
NORM5*	Ra-226	118 (21)	121 (13)	117 (25)
	Th-232	60 (13)	64 (8)	57 (16)
	K-40	1460 (216)	1381 (127)	1504 (248)
NORM6	Ra-226	49.7 (8.9)	54.9 (9.1)	46.8 (7.7)
	Th-232	56.4 (3.9)	58.0 (5.1)	55.6 (3.1)
	K-40	1221 (70)	1202 (99)	1232 (52)

1 The materials that require crushing and sieving to a particle size of less than 2 mm are indicated with an asterisk.
2 Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 13. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants milled and sieved the material to a particle size less than 2 mm, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

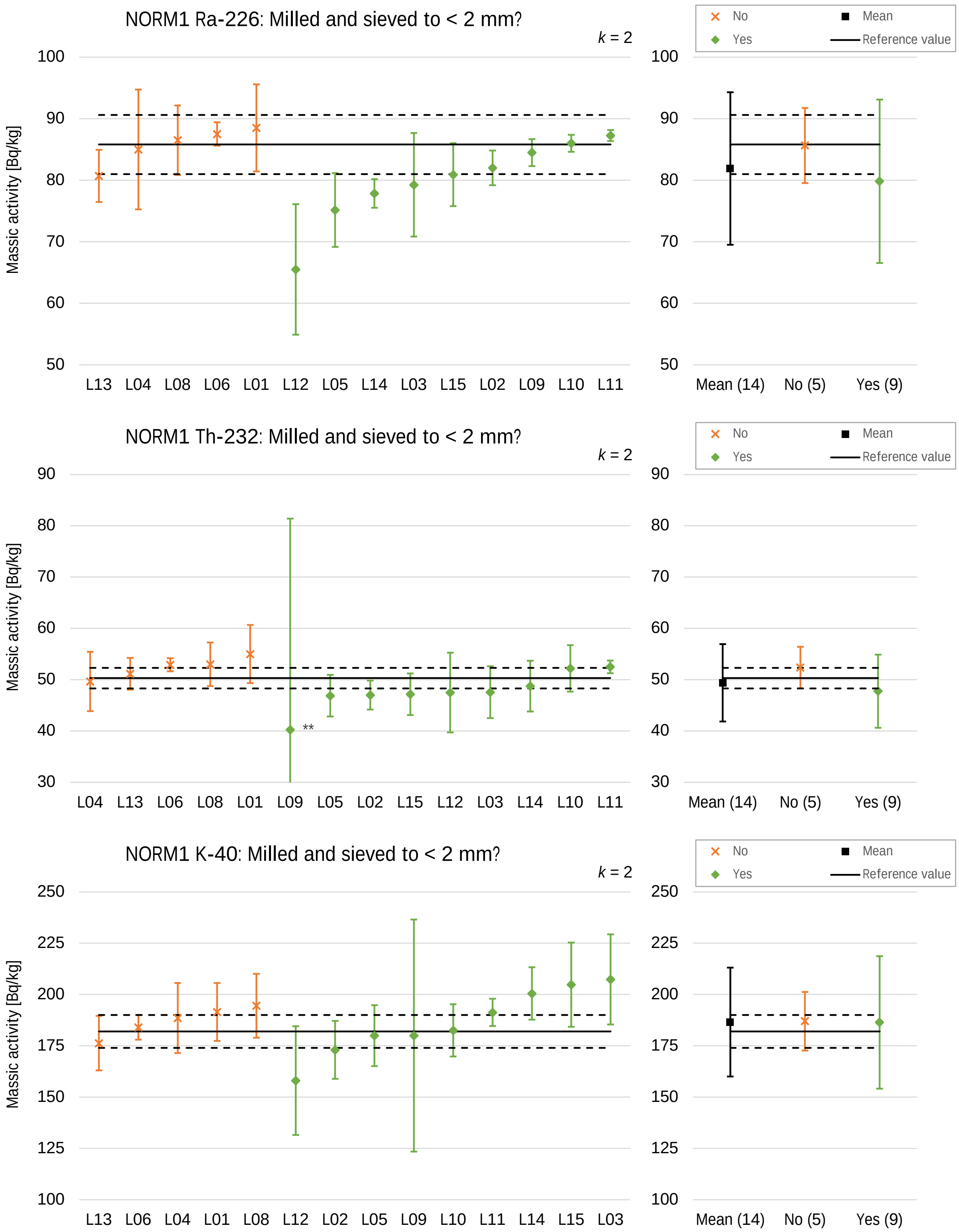


Figure 14. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants milled and sieved the material to a particle size less than 2 mm, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

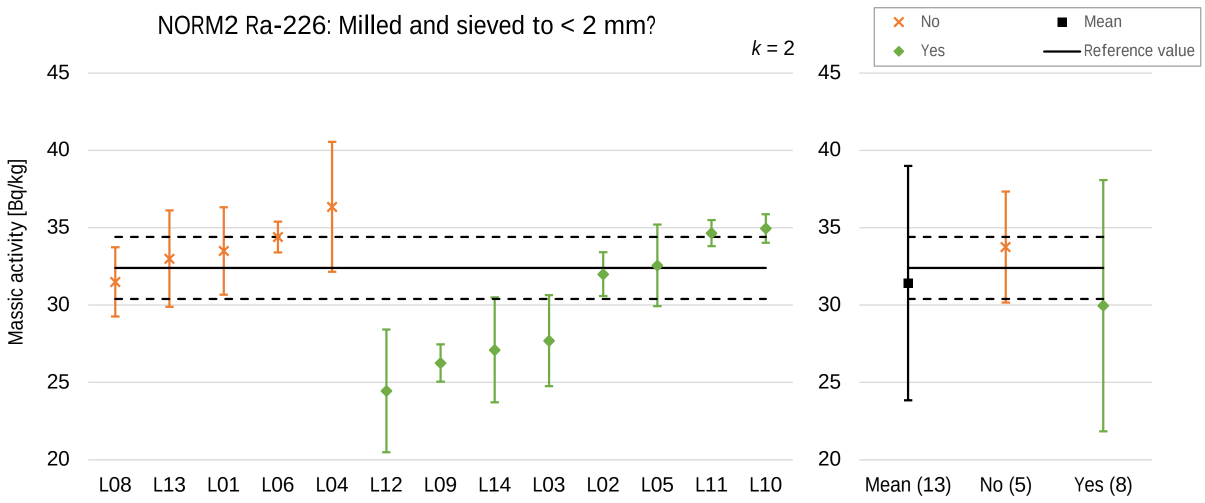


Figure 15. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants milled and sieved the material to a particle size less than 2 mm, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

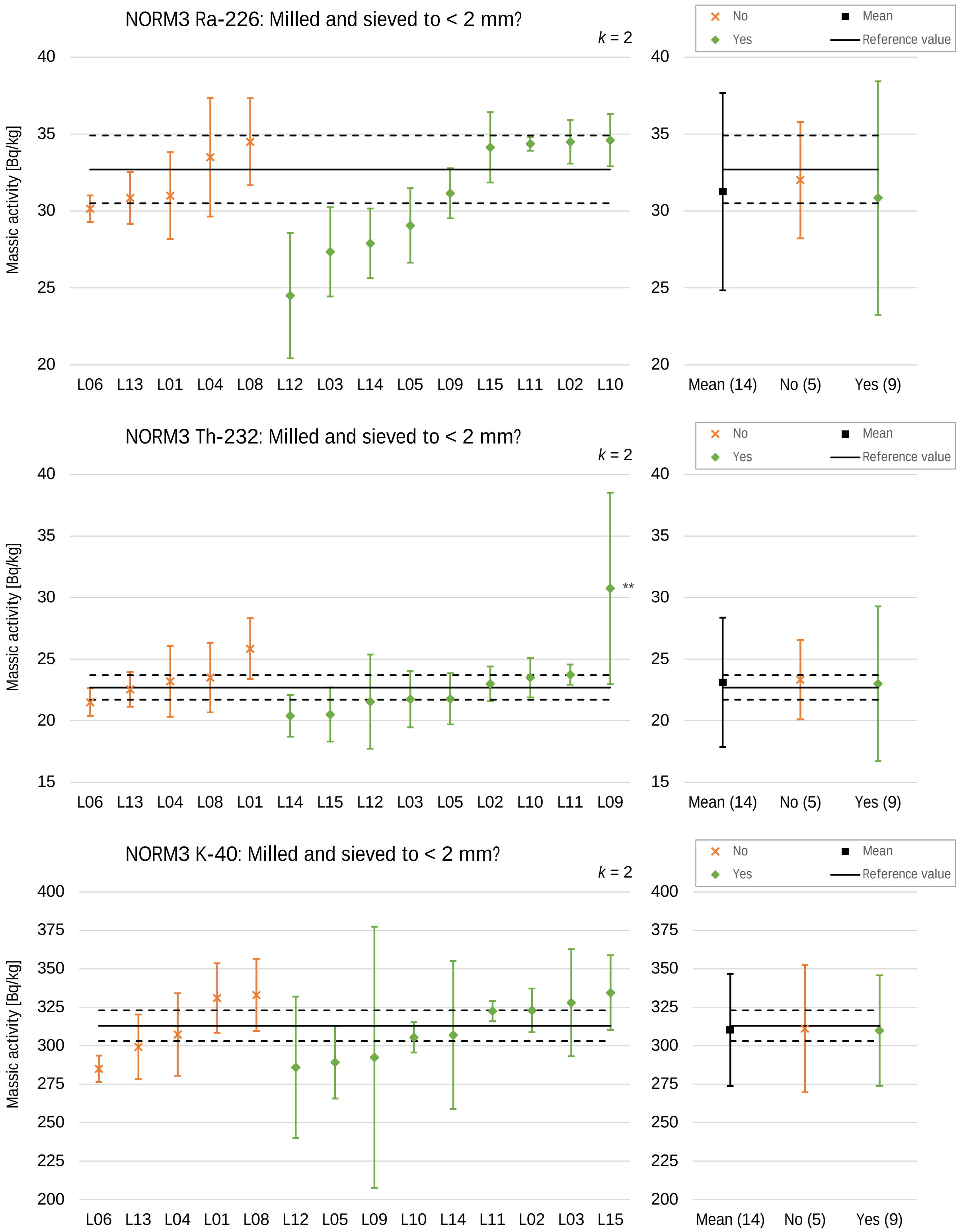


Figure 16. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants milled and sieved the material to a particle size less than 2 mm, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

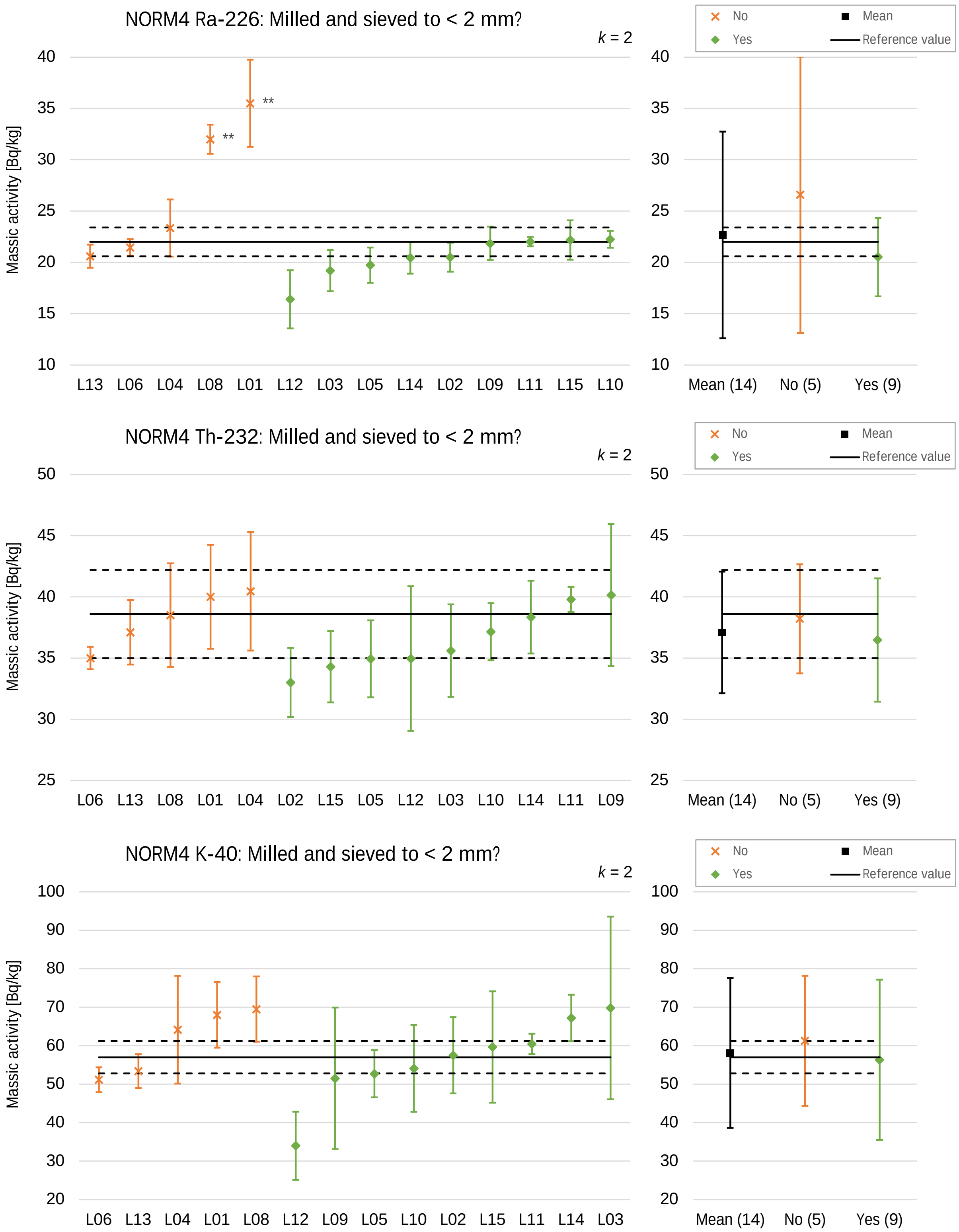


Figure 17. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants milled and sieved the material to a particle size less than 2 mm, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

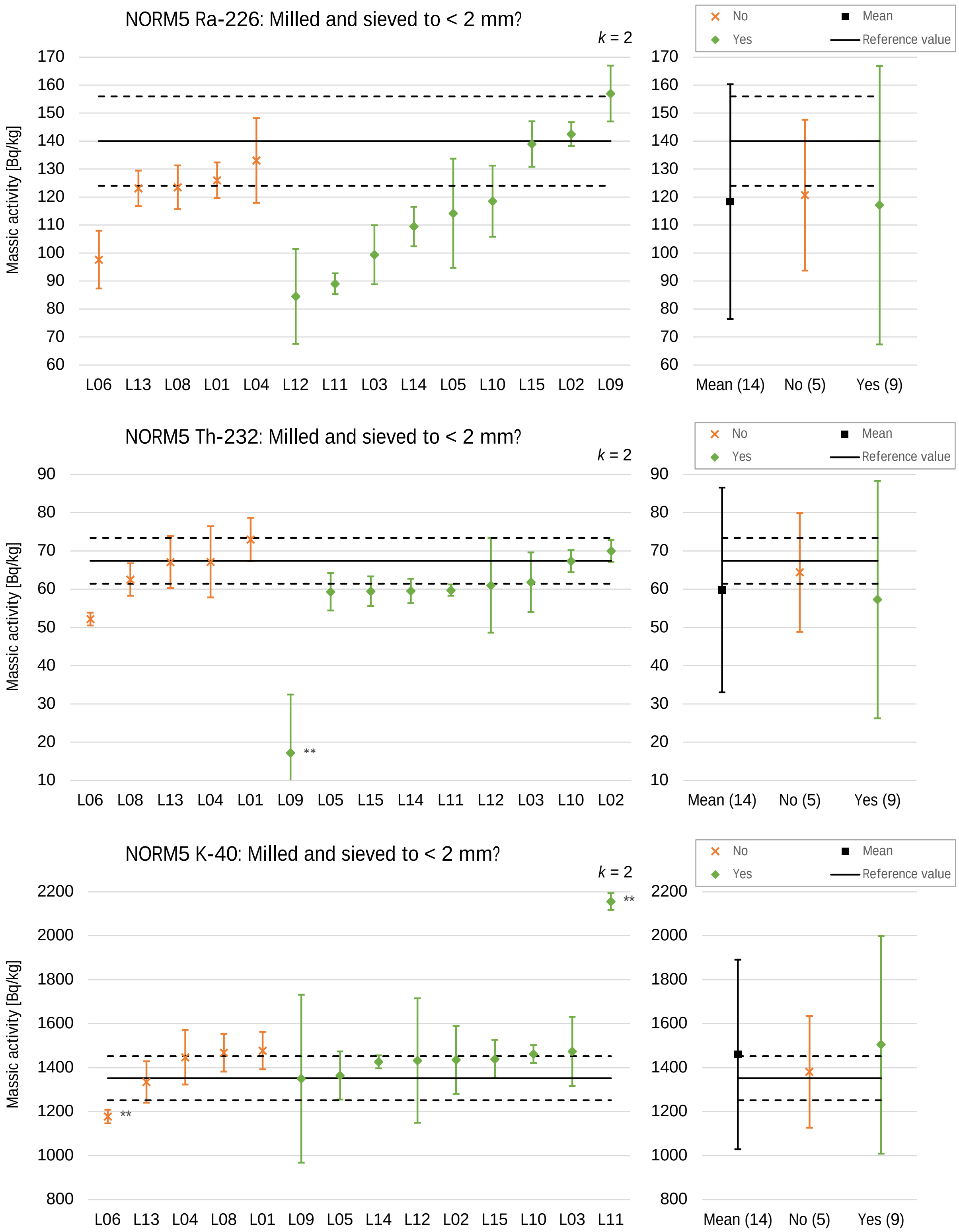
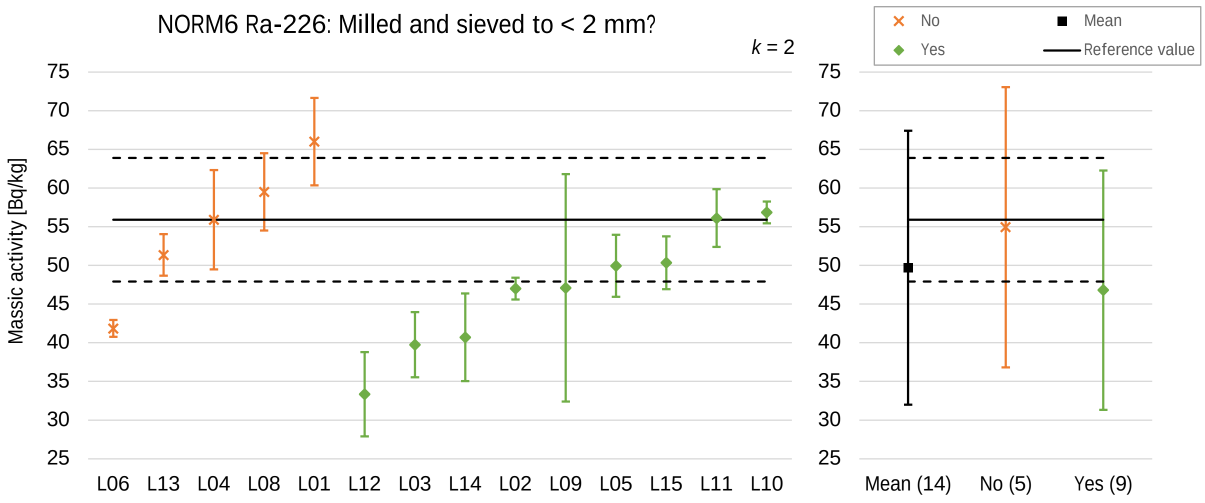


Figure 18. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants milled and sieved the material to a particle size less than 2 mm, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.



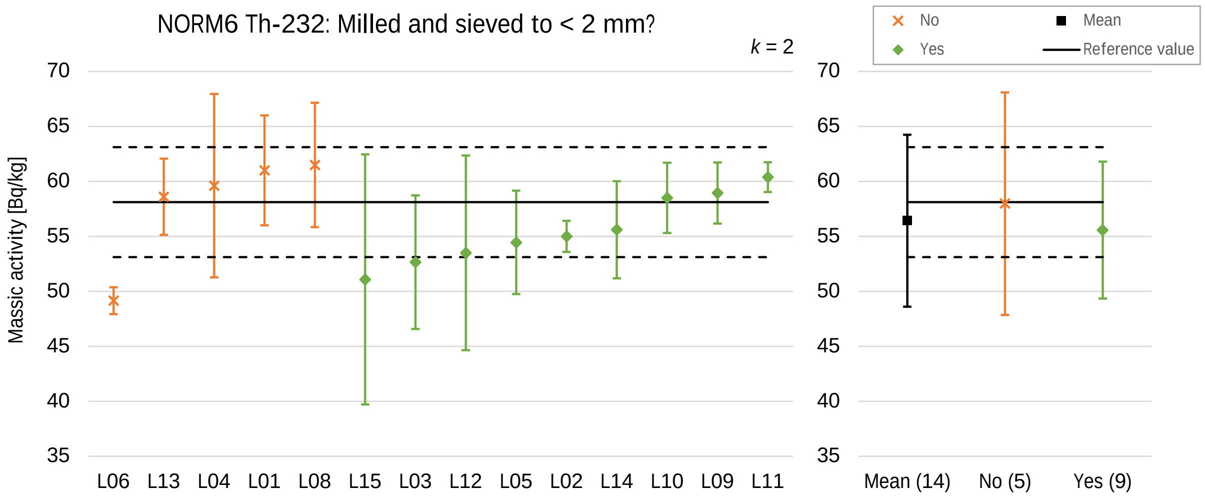


Table 24. Mean results from participants who indicated that they yes/no homogenised the material.

Material (1)	Radionuclide	Mean massic activity [Bq/kg] (2)		
		Mean	Homogenised?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	80.7 (2.1)	82.0 (6.4)
	Th-232	49.4 (3.8)	51.2 (1.6)	49.3 (3.9)
	K-40	187 (13)	176 (7)	187 (13)
NORM2*	Ra-226	31.4 (3.8)	33.0 (1.6)	31.3 (3.9)
	Th-232	23.7 (2.0)	24.3 (1.0)	23.7 (2.1)
	K-40	327 (27)	303 (11)	329 (27)
NORM3*	Ra-226	31.3 (3.2)	30.9 (0.8)	31.3 (3.3)
	Th-232	23.1 (2.6)	22.6 (0.7)	23.2 (2.7)
	K-40	310 (18)	299 (11)	311 (19)
NORM4	Ra-226	22.7 (5.0)	20.6 (0.6)	22.8 (5.2)
	Th-232	37.1 (2.5)	37.1 (1.3)	37.1 (2.6)
	K-40	58 (10)	53 (2)	58 (10)
NORM5	Ra-226	118 (21)	123 (3)	118 (22)
	Th-232	60 (13)	67 (3)	59 (14)
	K-40	1460 (216)	1335 (47)	1470 (221)
NORM6*	Ra-226	49.7 (8.9)	51.4 (1.3)	49.6 (9.2)
	Th-232	56.4 (3.9)	58.6 (1.7)	56.3 (4.0)
	K-40	1221 (70)	1154 (40)	1226 (70)

1 The heterogeneous materials are indicated with an asterisk.
2 Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 19. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants homogenised the test specimen. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

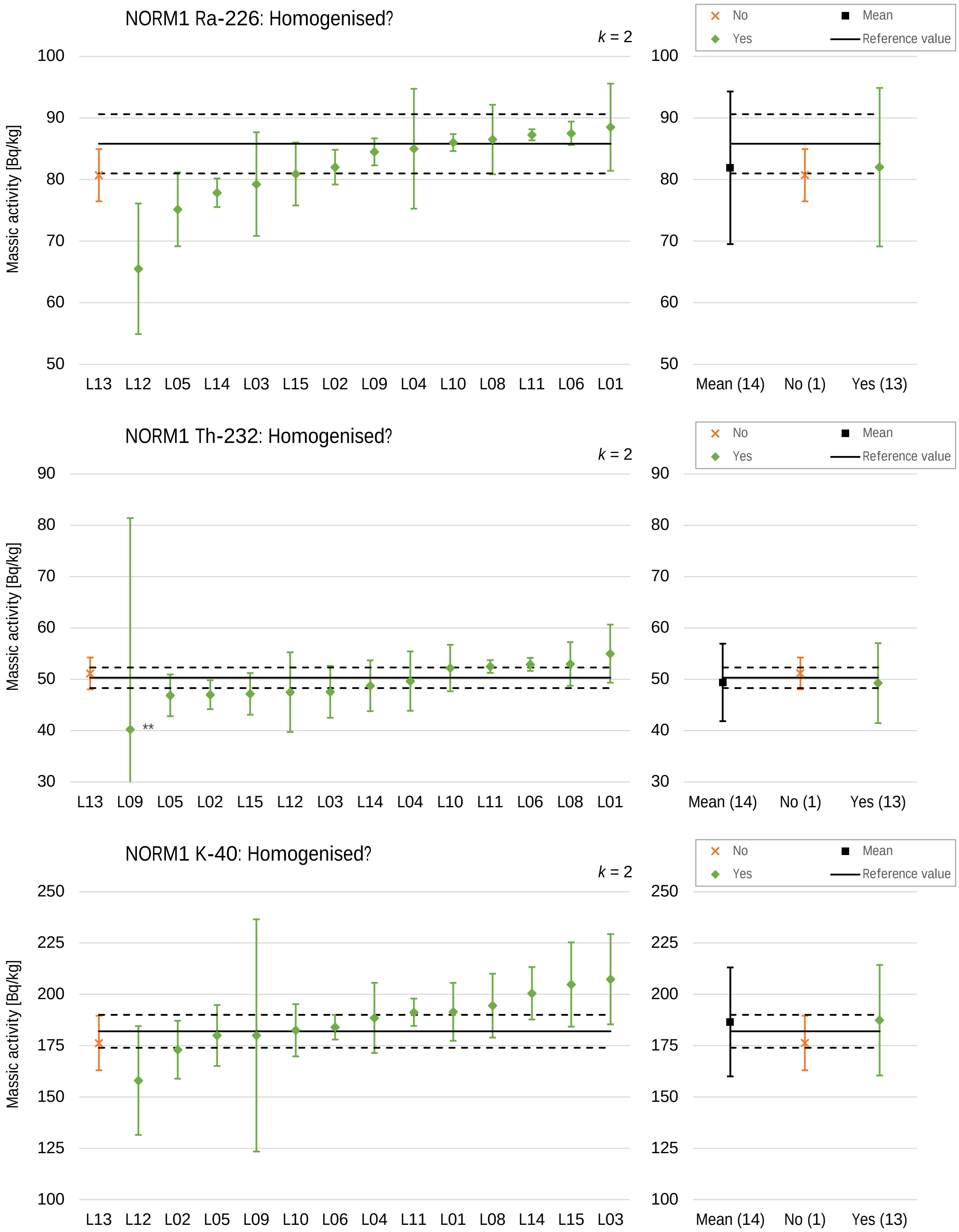


Figure 20. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants homogenised the test specimen. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

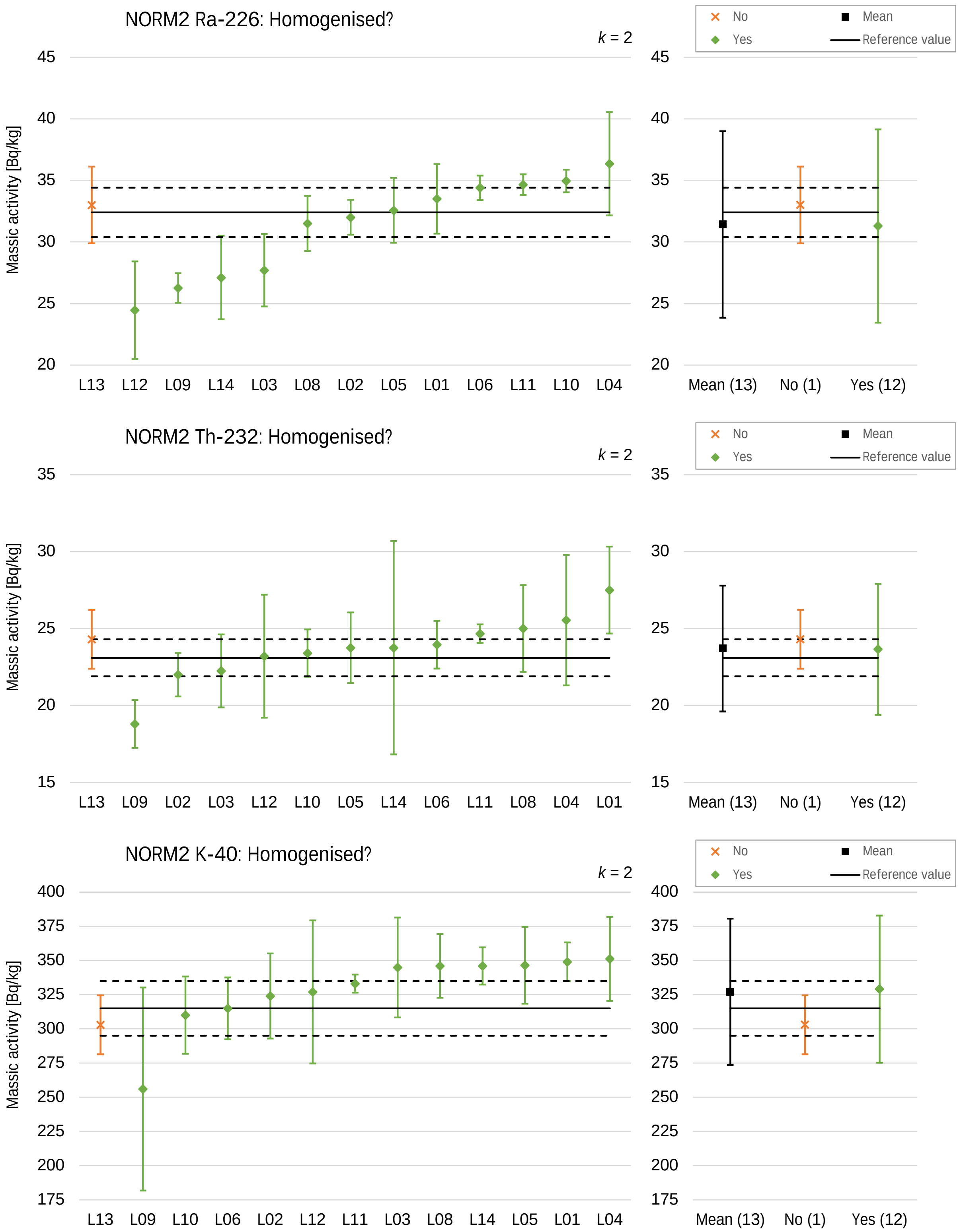


Figure 21. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants homogenised the test specimen. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

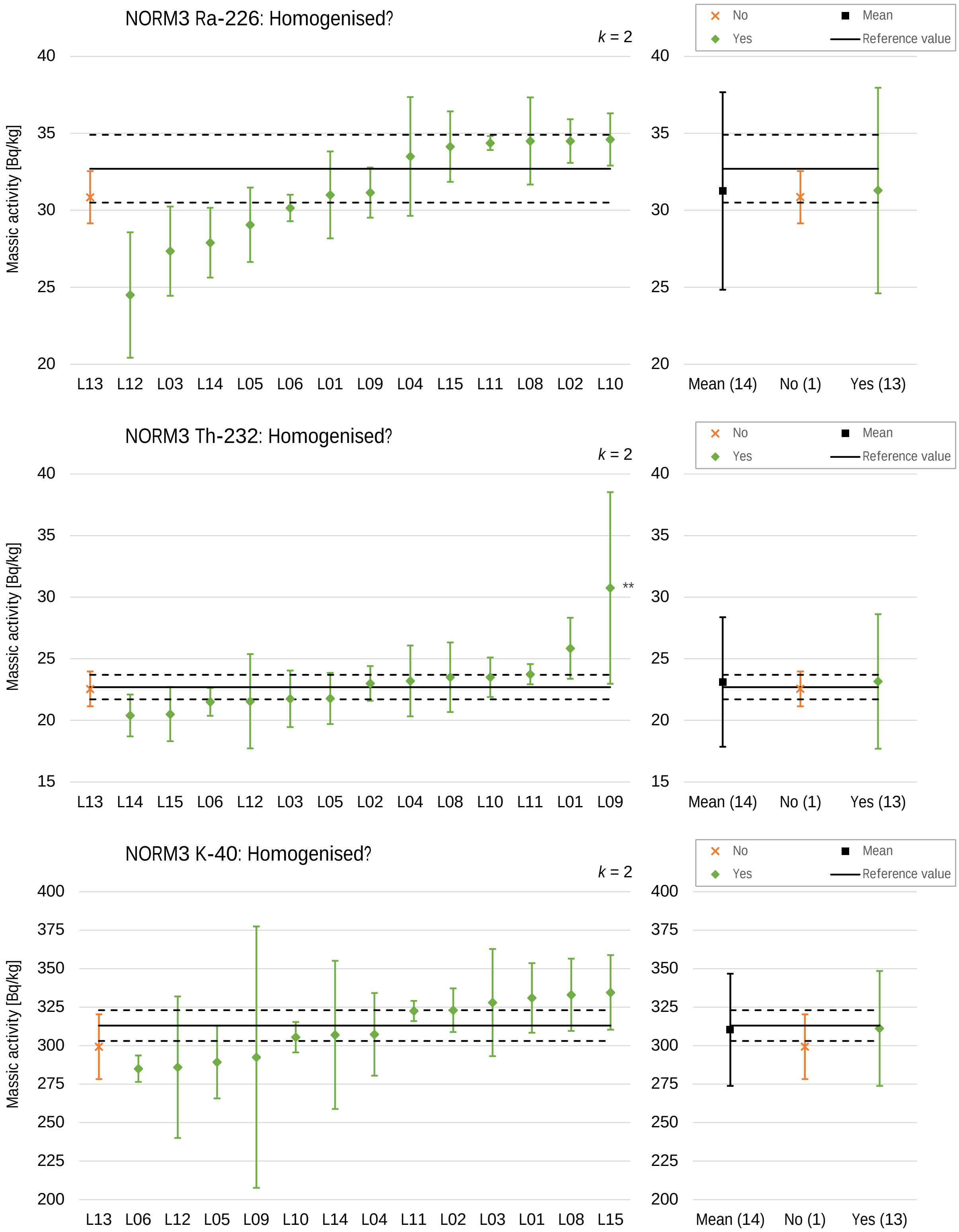


Figure 22. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants homogenised the test specimen. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

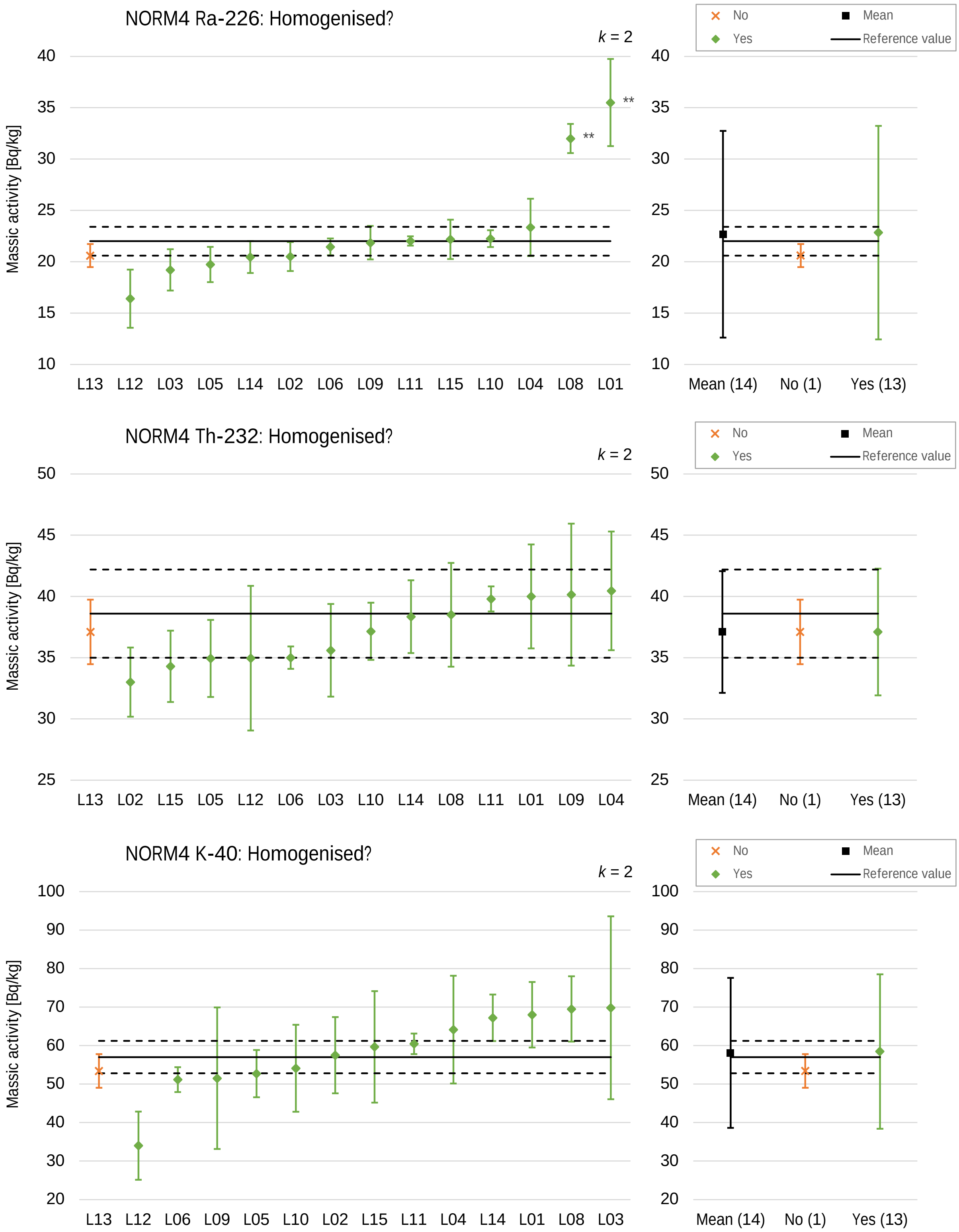


Figure 23. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants homogenised the test specimen. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

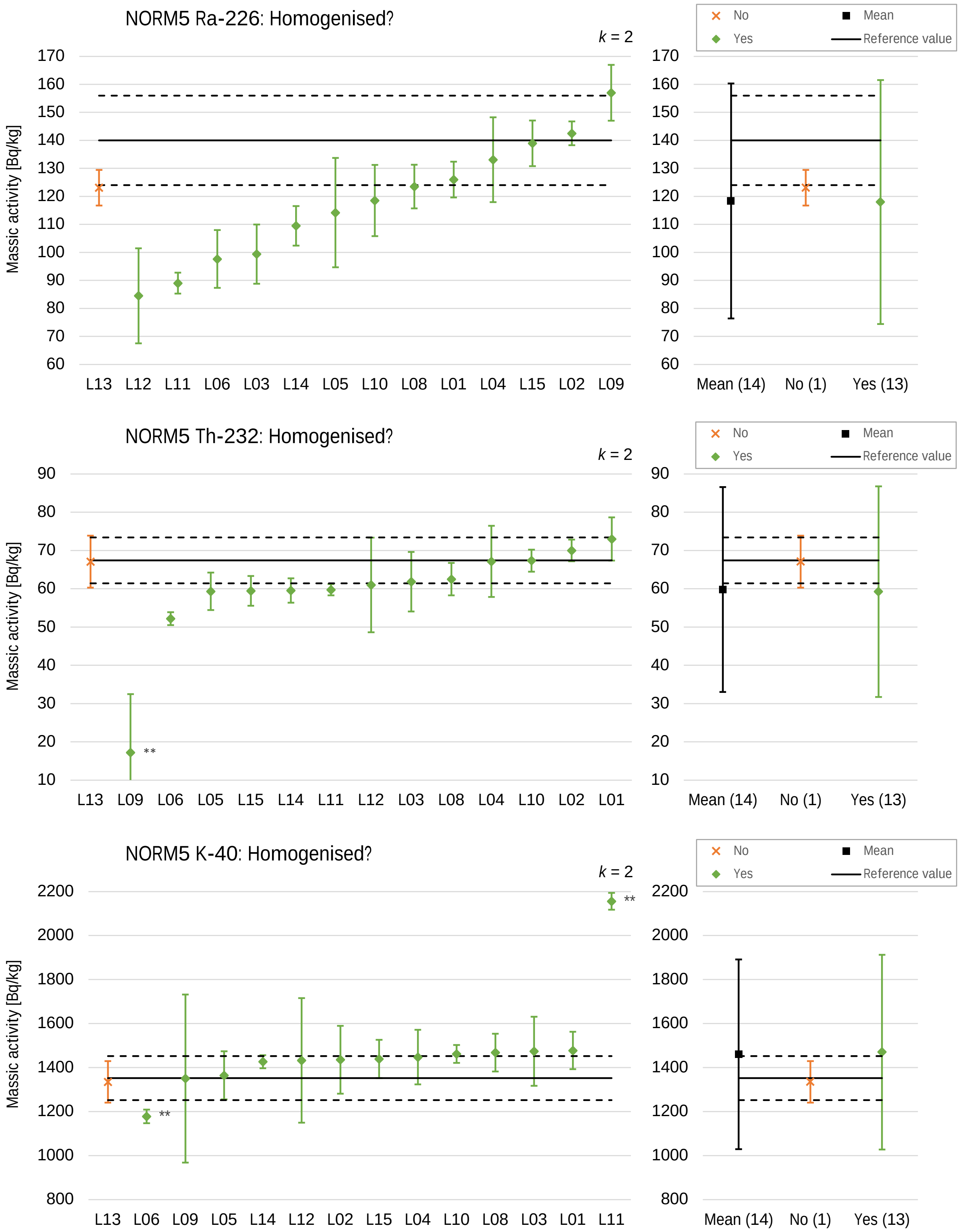
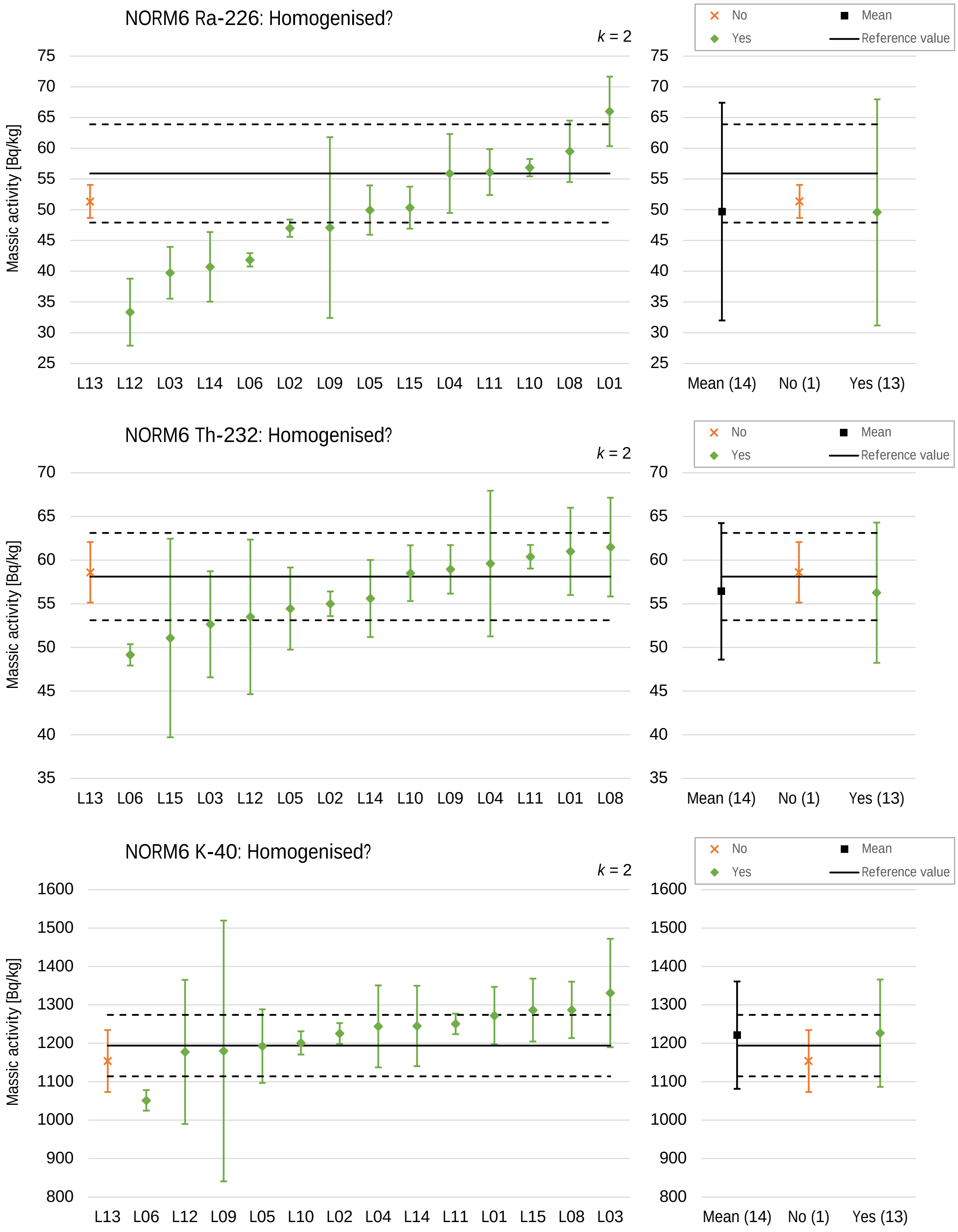


Figure 24. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants homogenised the test specimen. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.



6.3 Filling, compacting and sealing of the test specimen container

Clause 6.3.2.2 of WD EN 17216 specifies how the test specimen container shall be filled. During the filling process, the test specimen container shall be vibrated or tapped to achieve a constant compacted sample. It is further required to place the lid on (or in) the test specimen container such that it is resting on the material. In other words, the test specimen container shall be completely filled. Then, radon-tight sealing shall be applied, followed by a waiting time of at least three weeks.

In the reporting questionnaire, Q 2.7 polled if the test specimen containers were completely filled. Q 2.8 asked if the test specimen was vibrated to achieve a constant compacted sample. The participants who answered that they did not vibrate but compacted the test specimen (for example, manually) also received a "yes" in the evaluation. Q 2.11 asked how the test specimen container was made radon tight. Participants who answered that they only screwed on the lid, or those who wrote that they did not apply any method received a "no" score (See Table 25). Interpreting the replies to Q 2.13, all participants waited at least three weeks before starting the measurements.

The participants mentioned the following sealing techniques: sealing and storing in a vacuum bag (3x), adhesive tape (2x), parafilm tape (1x), paraffin (1x) and silicone (1x).

Table 25. Adherence to the requirements on filling, compacting and sealing of the test specimen container (Clause 6.3.2.2). Cells are coloured green where the participants followed the normative requirements from the draft standard.

Participant	Q 2.7: Test specimen containers completely filled?	Q 2.8: Test specimen vibrated (or compacted)?	Q 2.11: Was the test specimen container made radon tight?
L01	YES	YES	NO
L02	NO	NO	NO
L03	NO	NO	YES
L04	YES	YES	YES
L05	YES	YES	YES
L06	NO	NO	NO
L08	YES	YES	NO
L09	YES	YES	YES
L10	NO	YES	YES
L11	NO	YES	YES
L12	NO	NO	YES
L13	YES	NO	YES
L14	YES	YES	YES
L15	NO	NO	YES

Observing the data from Table 26 and Figure 25 to Figure 30, a difference between participants who filled the test specimen completely and those who did not cannot be proven.

based on the available data, the difference of filling the test specimen container completely versus not filling completley cannot be proven

Similarly, the data from Table 27 and Figure 31 to Figure 36 does not provide evidence of the effect of vibrating or compacting the test specimen.

based on the available data, the impact of vibrating or compacting the test specimen cannot be proven

Shaking may segregate smaller particles from bigger ones and this could result in a bias when is causes a heterogeneous distribution of the activity in the test specimen. Shaking was applied by participants L04, L09 and L15. The data provided by participants L04 and L15 does not demonstrate a potential effect of shaking. Participant L09 however, reports diverting results for Th-232 in NORM1, NORM3 and NORM5. Th-232 is too low in NORM1 and NORM5 but too high in NORM3. As discussed in Section 4.6, the reported values of participant are inconsistent and therefore care should be taken in drawing conclusions.

based on the available data, the impact of shaking the test specimen cannot be proven

The data in Table 28 and Figure 37 to Figure 42 reflects the results from participants who yes or no indicated that they made the test specimen container radon tight. An effect could be expected for the measurement of Ra-226. Earlier in Section 5 it was demonstrated that the relative bias for Ra-226 is -7.5 (5.8) %, but the data provides no evidence of a difference between the participants who claimed to have made the test specimen container radon tight and those who did not.

based on the available data, the impact of making the test specimen container radon tight cannot be proven

Table 26. Mean results from participants who indicated that they yes/no filled the test specimen container completely.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Test specimen container completely filled?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	81.2 (7.6)	82.6 (4.8)
	Th-232	49.4 (3.8)	49.5 (2.8)	49.2 (4.8)
	K-40	187 (13)	186 (17)	187 (9)
NORM2	Ra-226	31.4 (3.8)	31.4 (4.3)	31.5 (3.6)
	Th-232	23.7 (2.0)	23.2 (1.0)	24.1 (2.7)
	K-40	327 (27)	326 (13)	328 (36)
NORM3	Ra-226	31.3 (3.2)	31.4 (4.1)	31.1 (2.3)
	Th-232	23.1 (2.6)	22.2 (1.2)	24.0 (3.4)
	K-40	310 (18)	312 (20)	308 (17)
NORM4	Ra-226	22.7 (5.0)	20.6 (2.1)	24.8 (6.3)
	Th-232	37.1 (2.5)	35.7 (2.2)	38.5 (2.0)
	K-40	58 (10)	55 (11)	61 (8)
NORM5	Ra-226	118 (21)	110 (24)	127 (15)
	Th-232	60 (13)	62 (6)	58 (19)
	K-40	1460 (216)	1511 (302)	1410 (59)
NORM6	Ra-226	49.7 (8.9)	46.5 (8.7)	52.9 (8.3)
	Th-232	56.4 (3.9)	54.3 (4.0)	58.5 (2.6)
	K-40	1221 (70)	1218 (90)	1225 (50)

[1] Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 25. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants filled the test specimen container completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

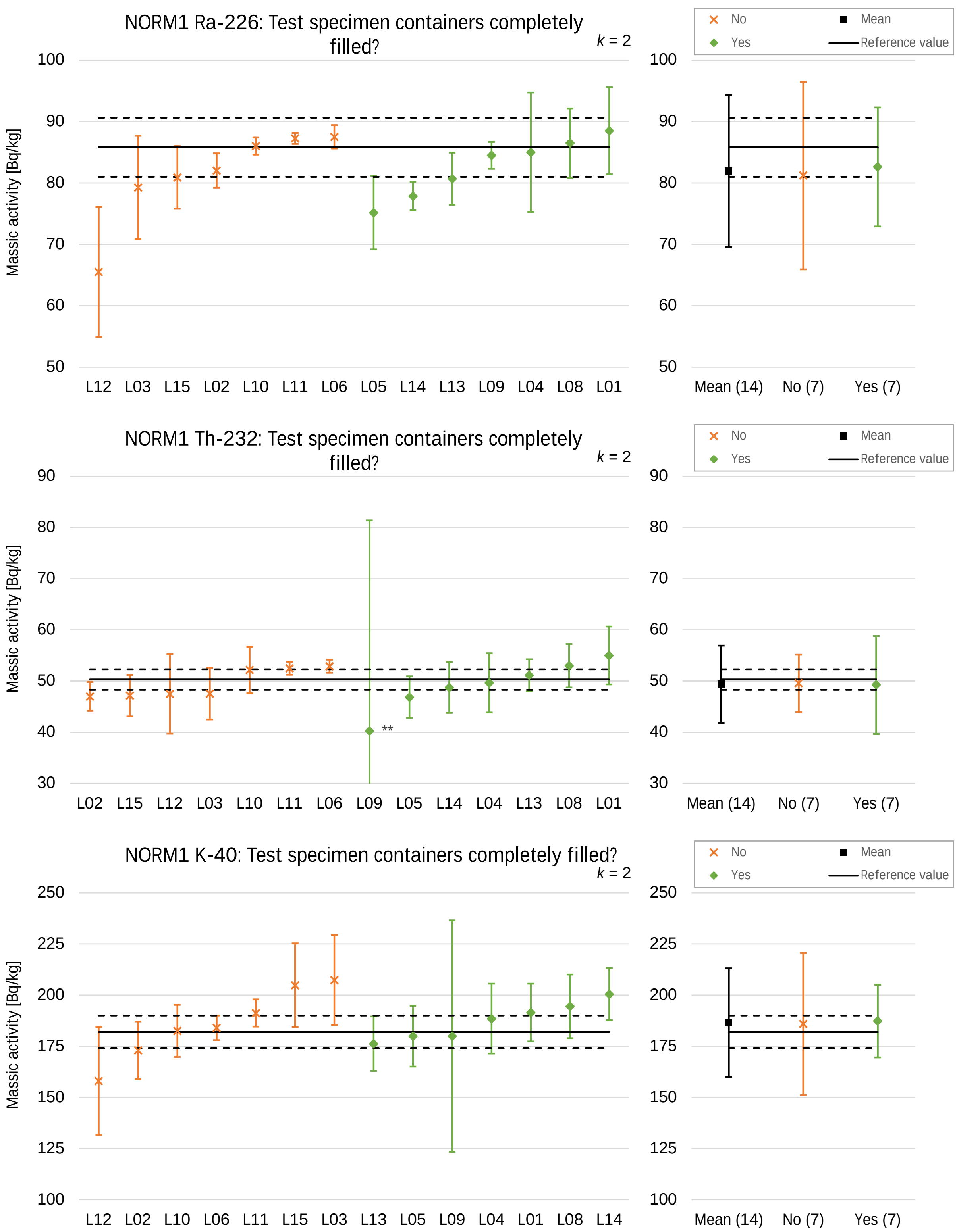


Figure 26. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants filled the test specimen container completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

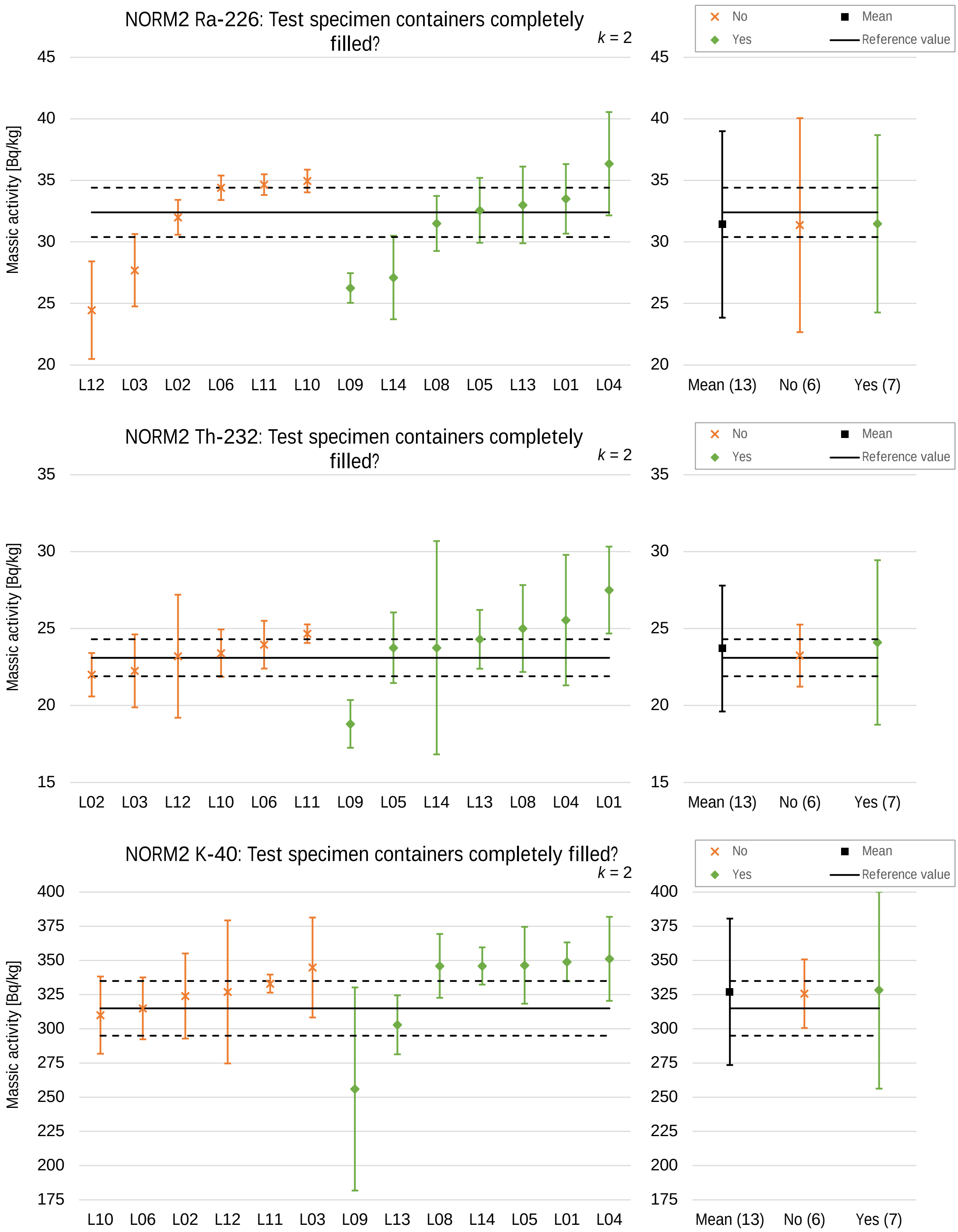


Figure 27. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants filled the test specimen container completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

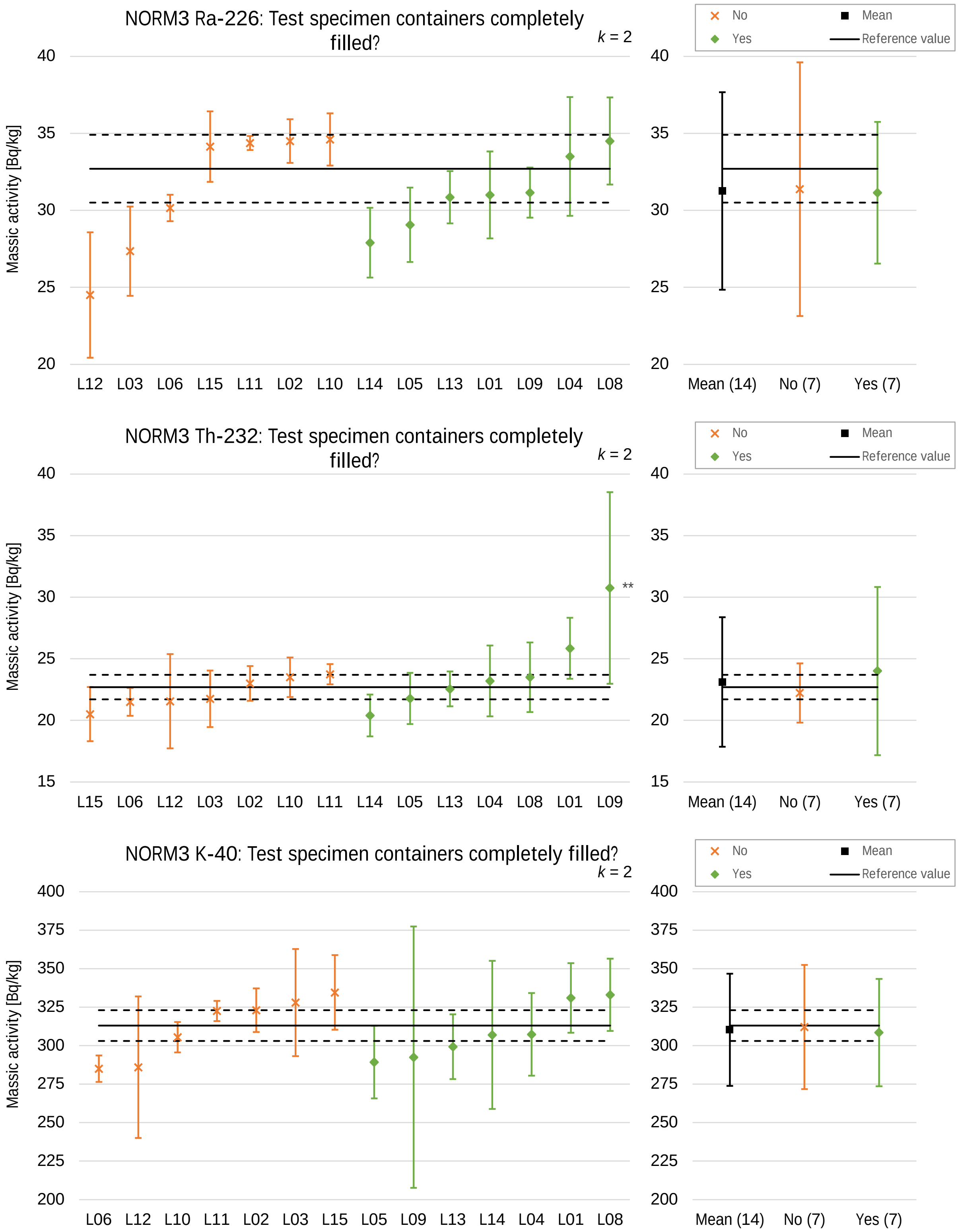


Figure 28. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants filled the test specimen container completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

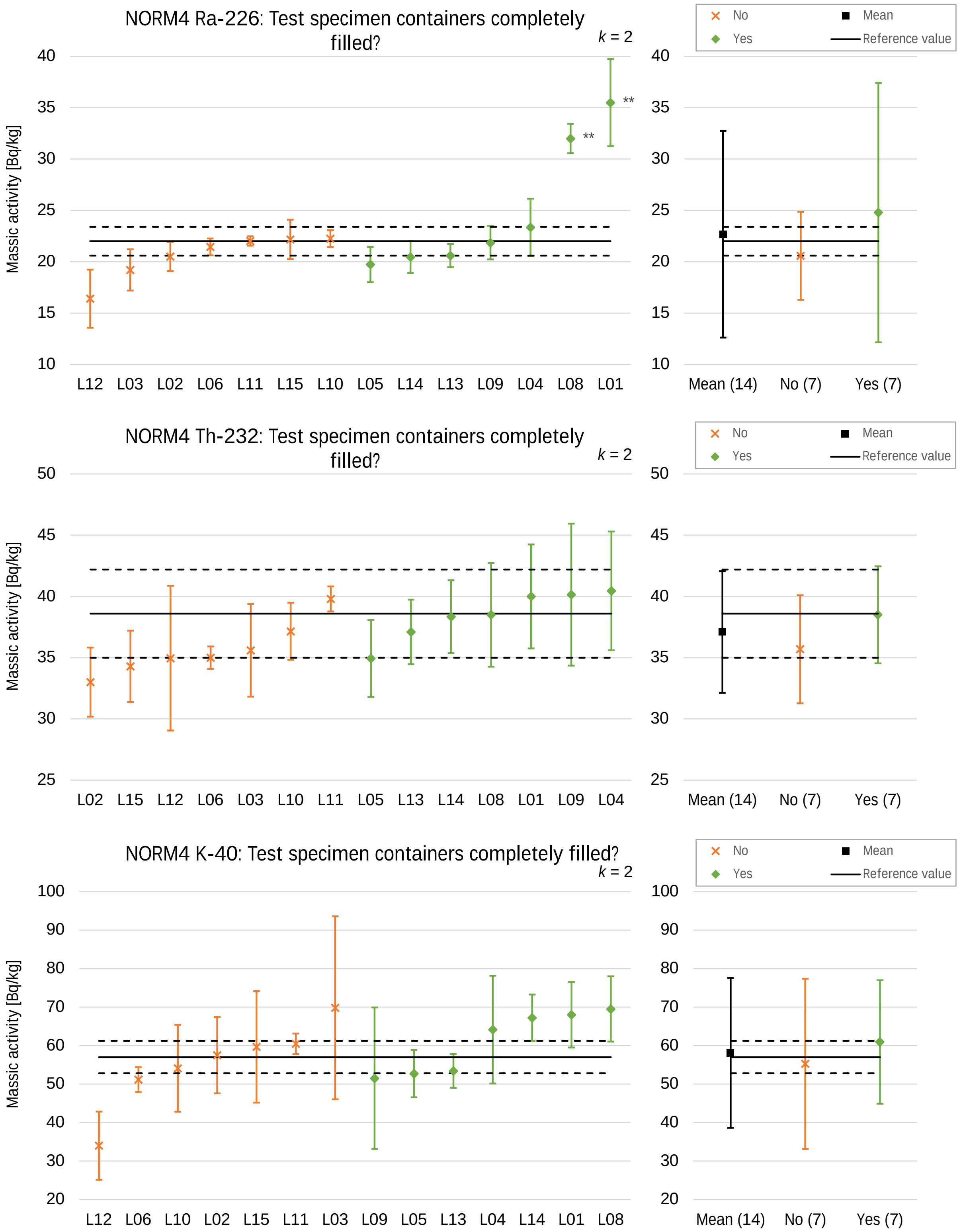


Figure 29. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants filled the test specimen container completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

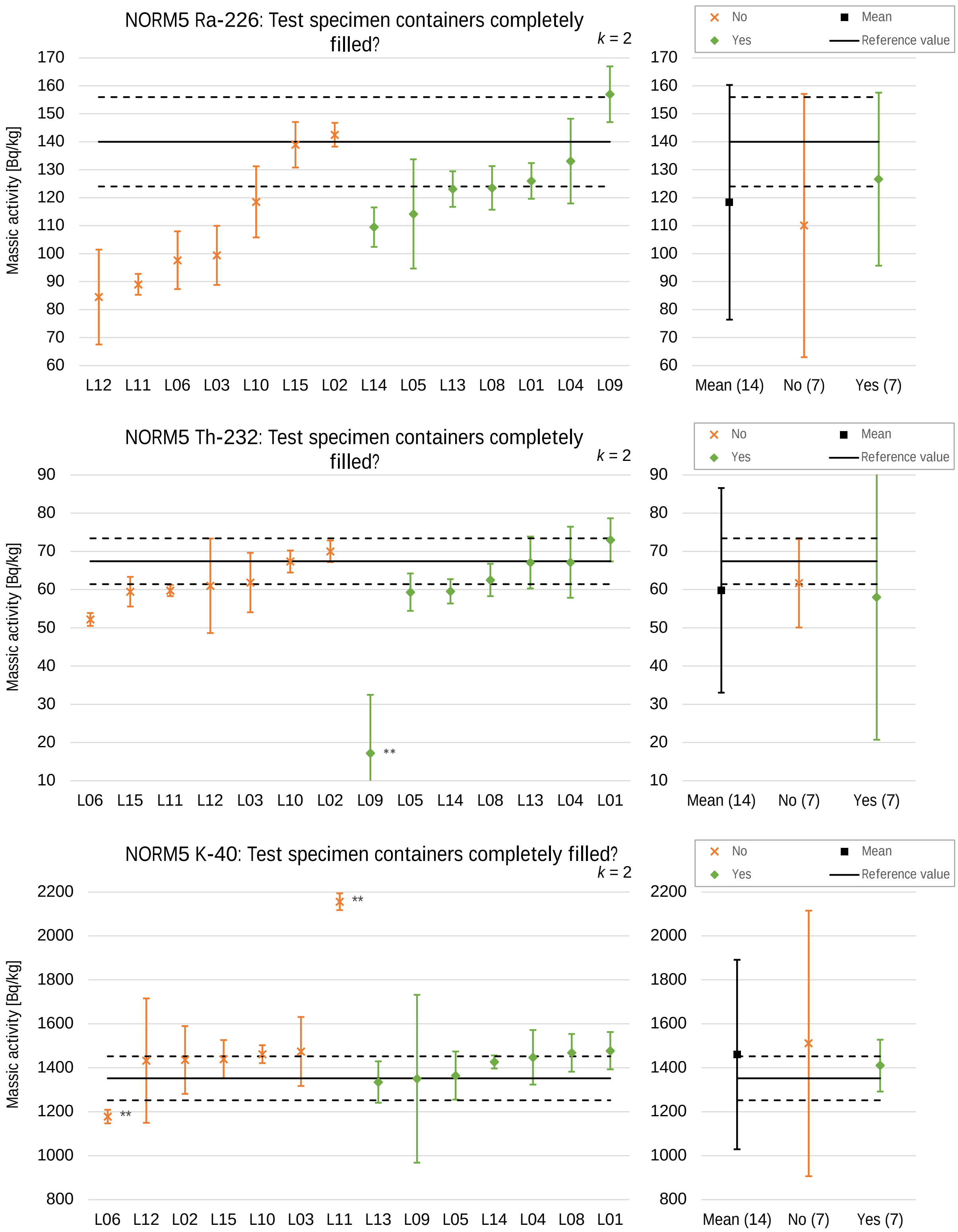


Figure 30. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants filled the test specimen container completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

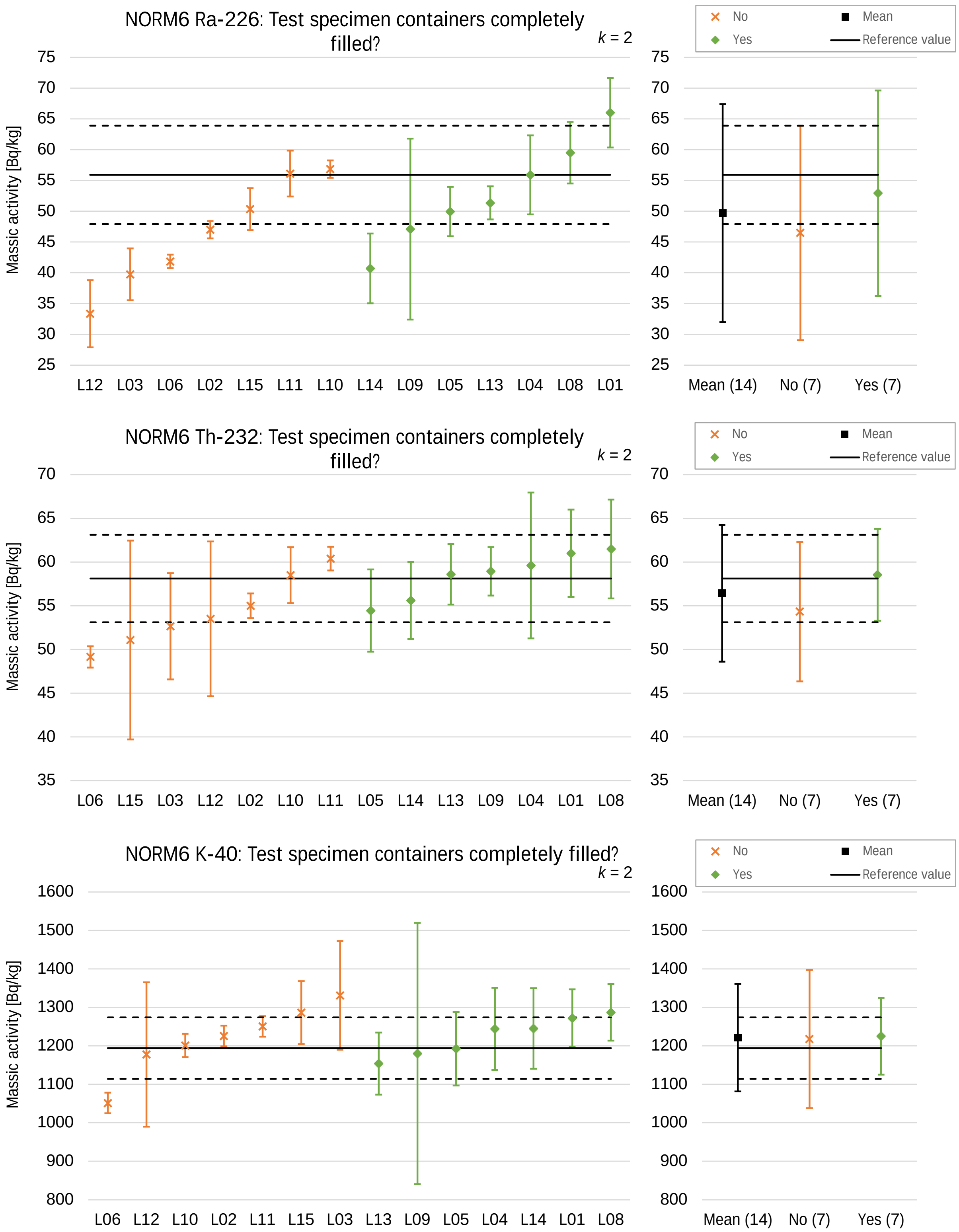


Table 27. Mean results from participants who indicated that they yes/no vibrated or compacted the test specimen.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Test specimen vibrated or compacted?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	79.3 (7.3)	83.8 (4.8)
	Th-232	49.4 (3.8)	48.9 (2.5)	49.8 (4.6)
	K-40	187 (13)	184 (19)	189 (7)
NORM2	Ra-226	31.4 (3.8)	30.3 (4.1)	32.1 (3.7)
	Th-232	23.7 (2.0)	23.1 (1.0)	24.1 (2.5)
	K-40	327 (27)	323 (15)	330 (33)
NORM3	Ra-226	31.3 (3.2)	30.2 (3.9)	32.0 (2.6)
	Th-232	23.1 (2.6)	21.8 (0.9)	24.1 (3.1)
	K-40	310 (18)	309 (22)	311 (16)
NORM4	Ra-226	22.7 (5.0)	20.1 (2.1)	24.6 (5.8)
	Th-232	37.1 (2.5)	35.0 (1.4)	38.7 (1.9)
	K-40	58 (10)	54 (12)	61 (7)
NORM5	Ra-226	118 (21)	114 (24)	121 (20)
	Th-232	60 (13)	62 (6)	58 (17)
	K-40	1460 (216)	1382 (110)	1519 (262)
NORM6	Ra-226	49.7 (8.9)	43.9 (6.9)	54.0 (7.8)
	Th-232	56.4 (3.9)	53.3 (3.3)	58.7 (2.5)
	K-40	1221 (70)	1204 (100)	1234 (39)

1 Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 31. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants vibrated or compacted the test specimen, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

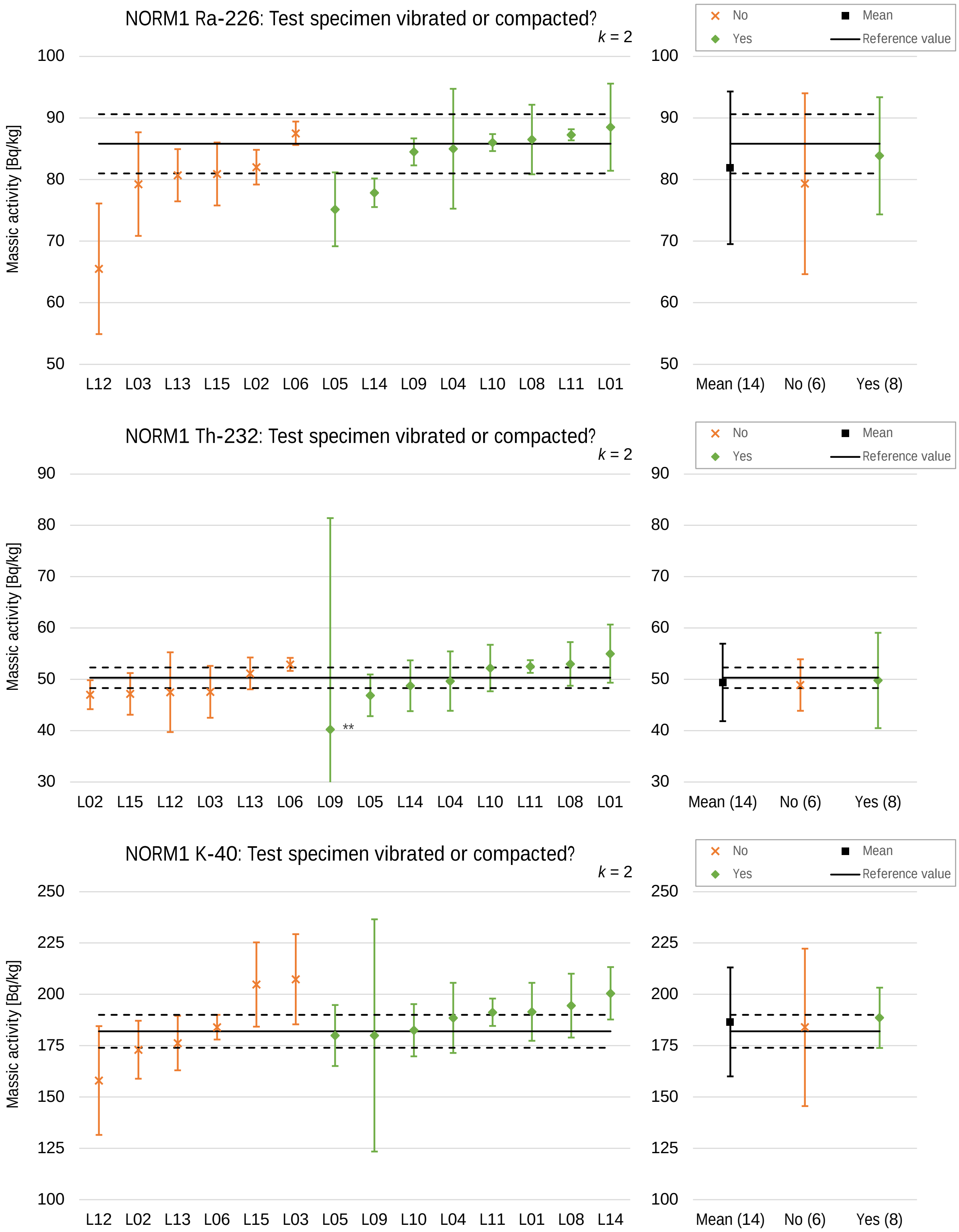


Figure 32. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants vibrated or compacted the test specimen, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

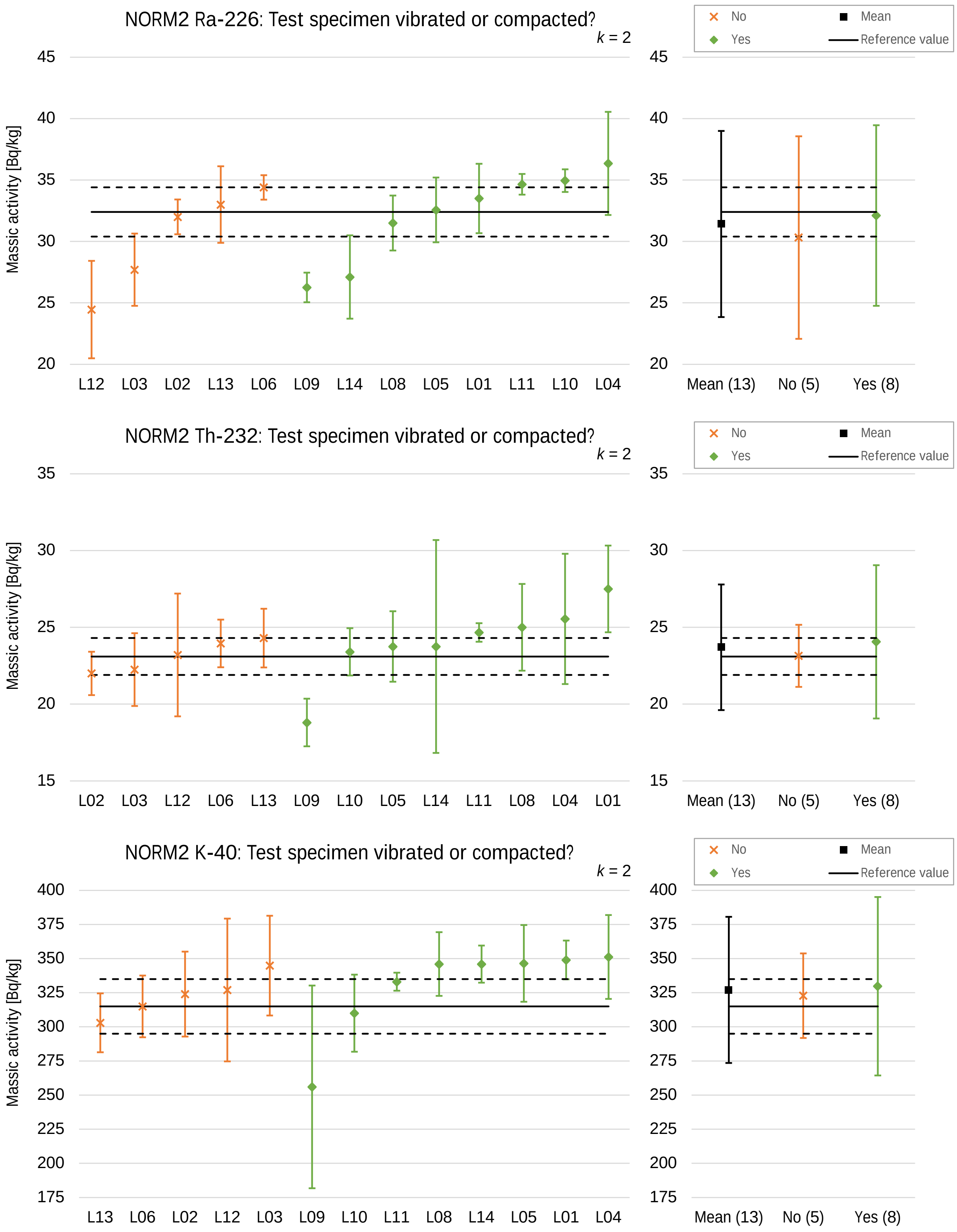
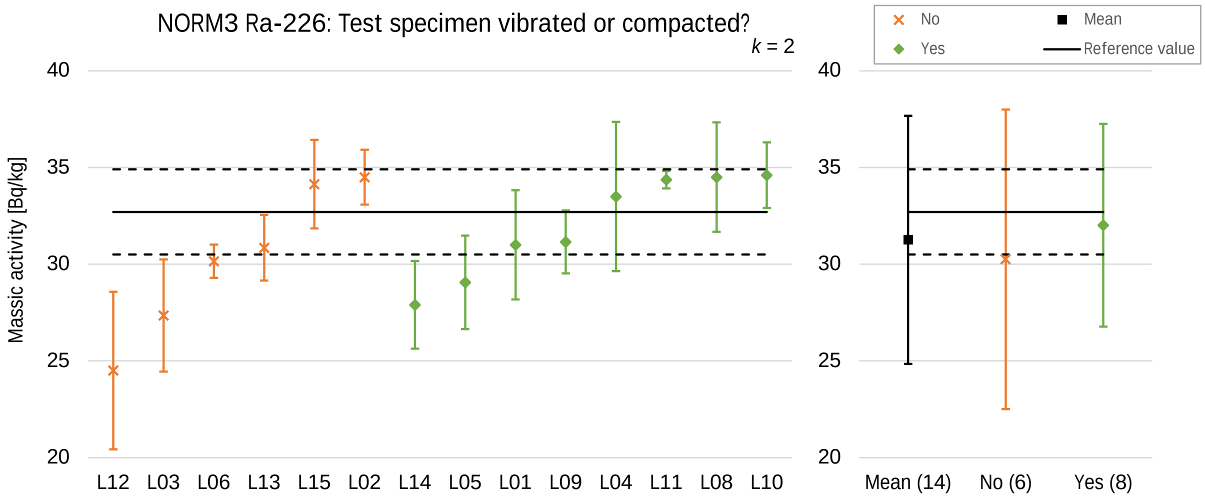
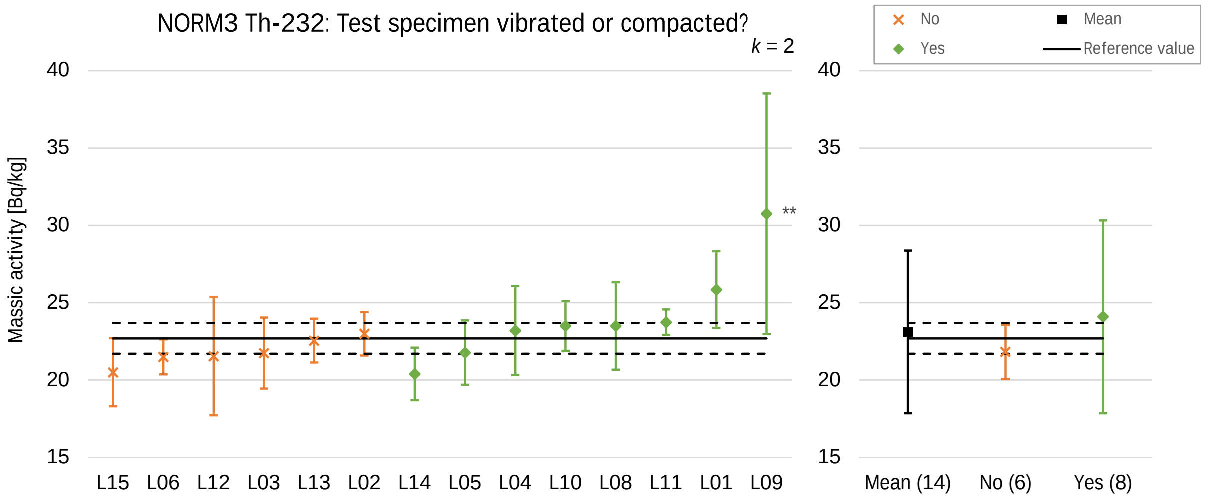


Figure 33. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants vibrated or compacted the test specimen, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.





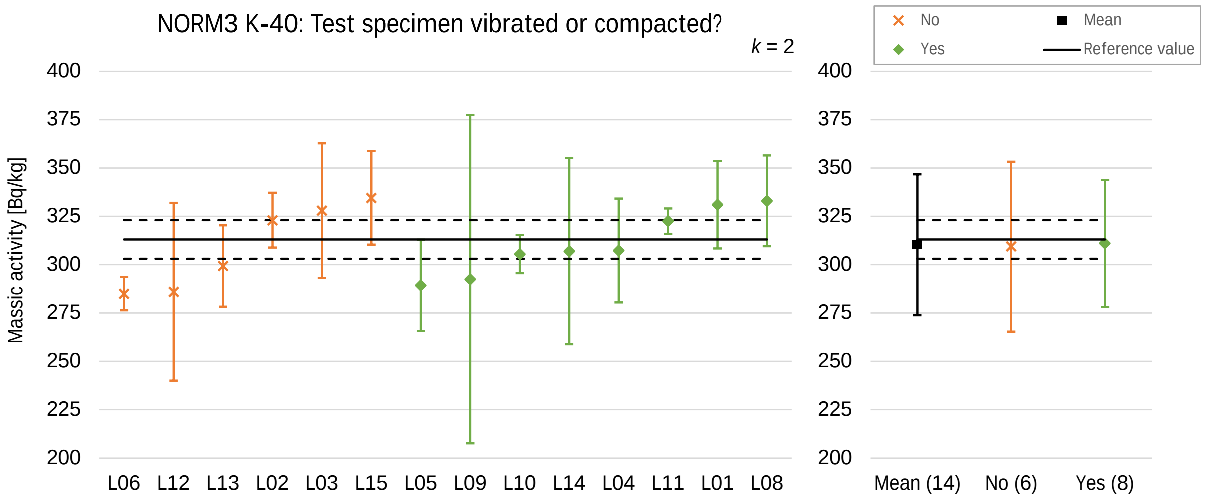


Figure 34. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants vibrated or compacted the test specimen, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

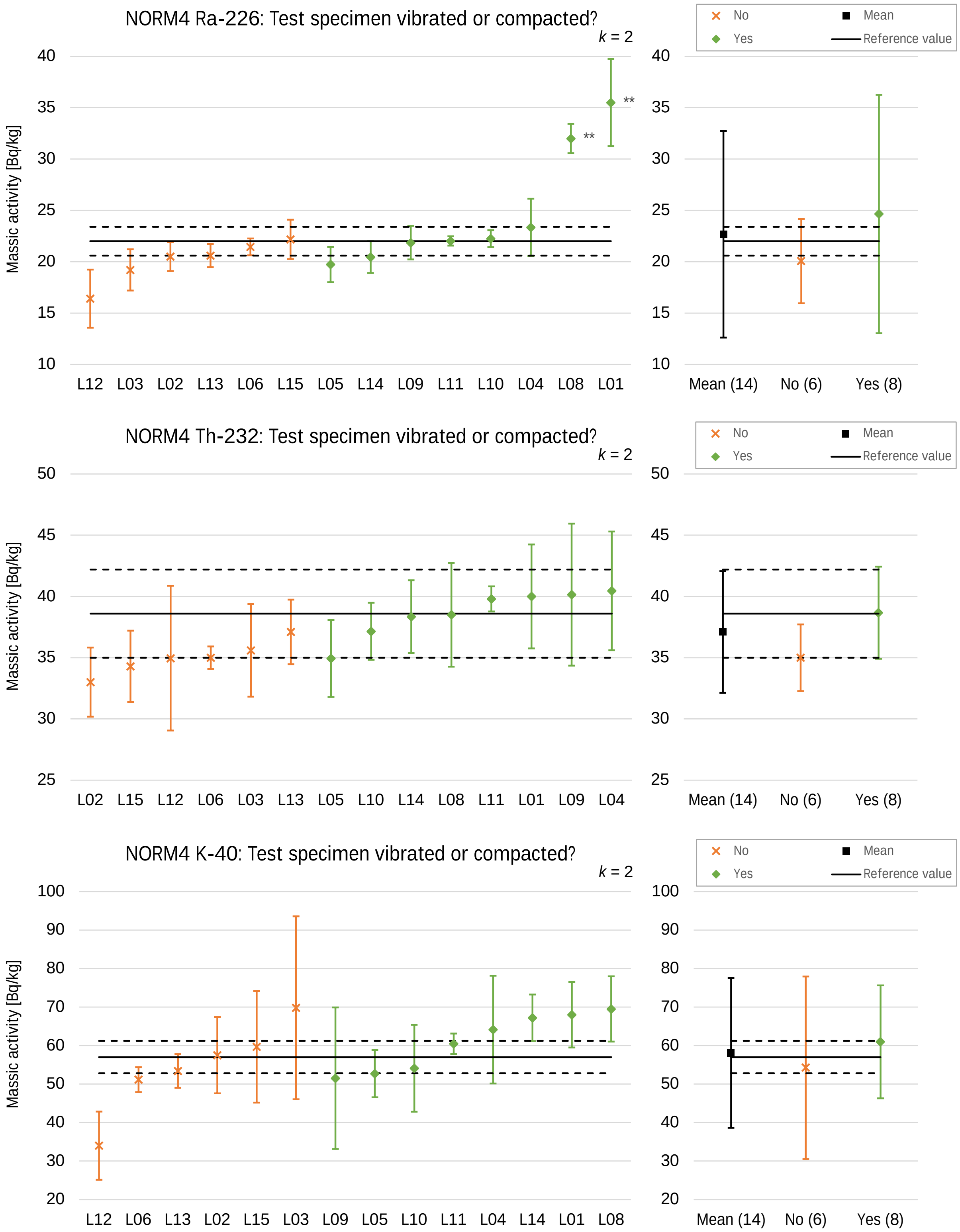
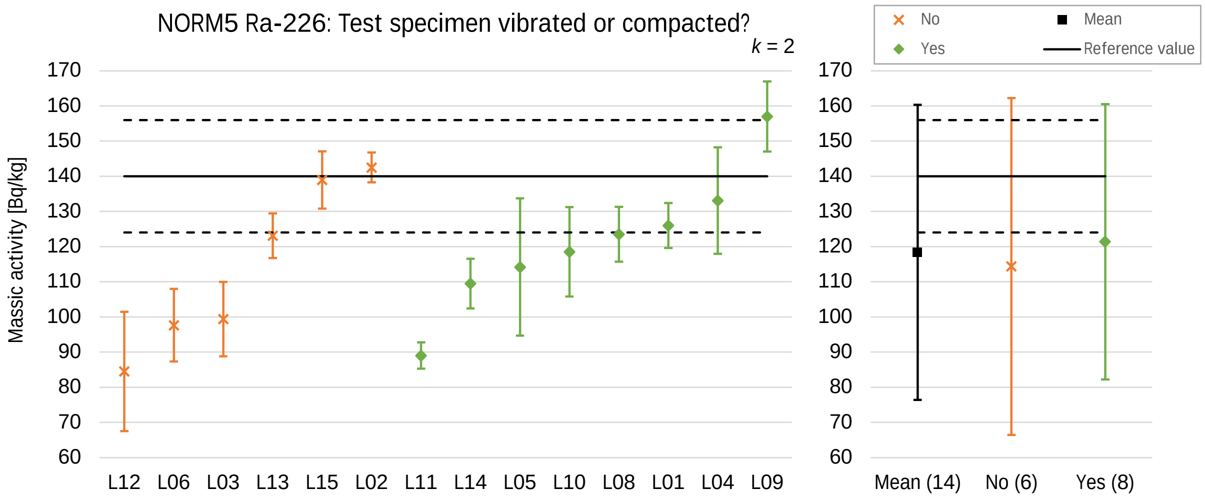
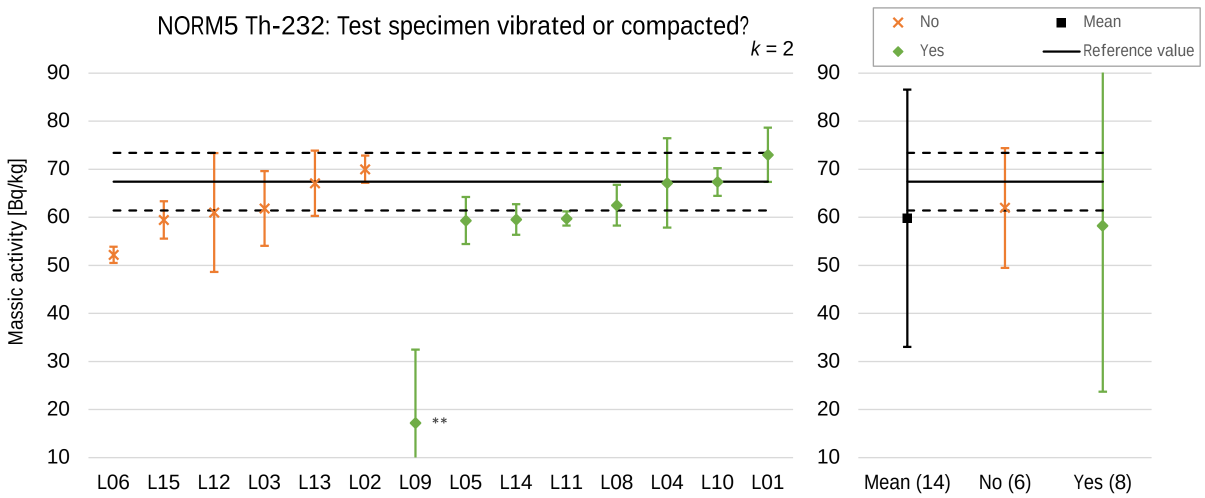


Figure 35. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants vibrated or compacted the test specimen, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.





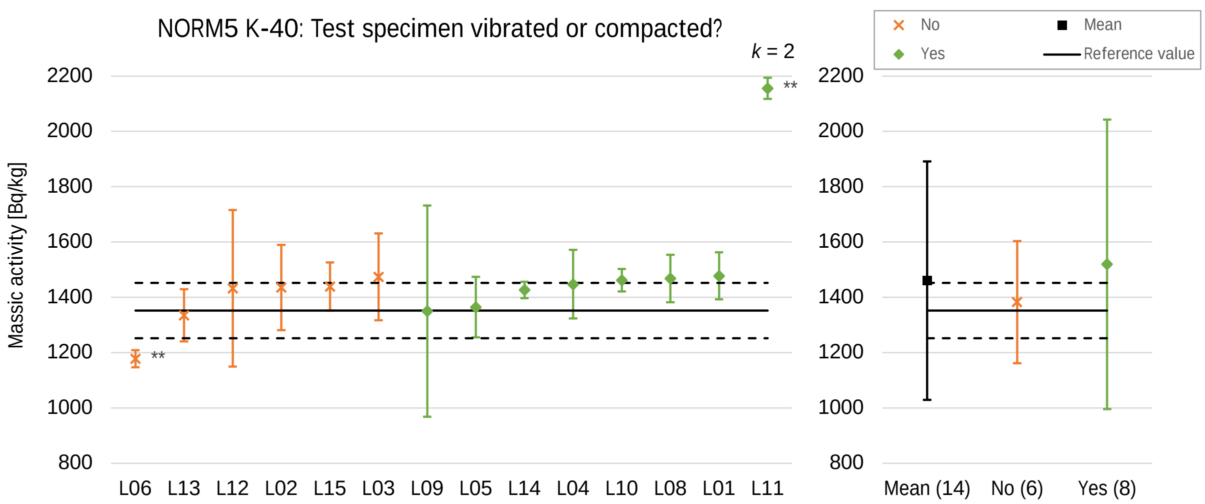


Figure 36. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants vibrated or compacted the test specimen, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

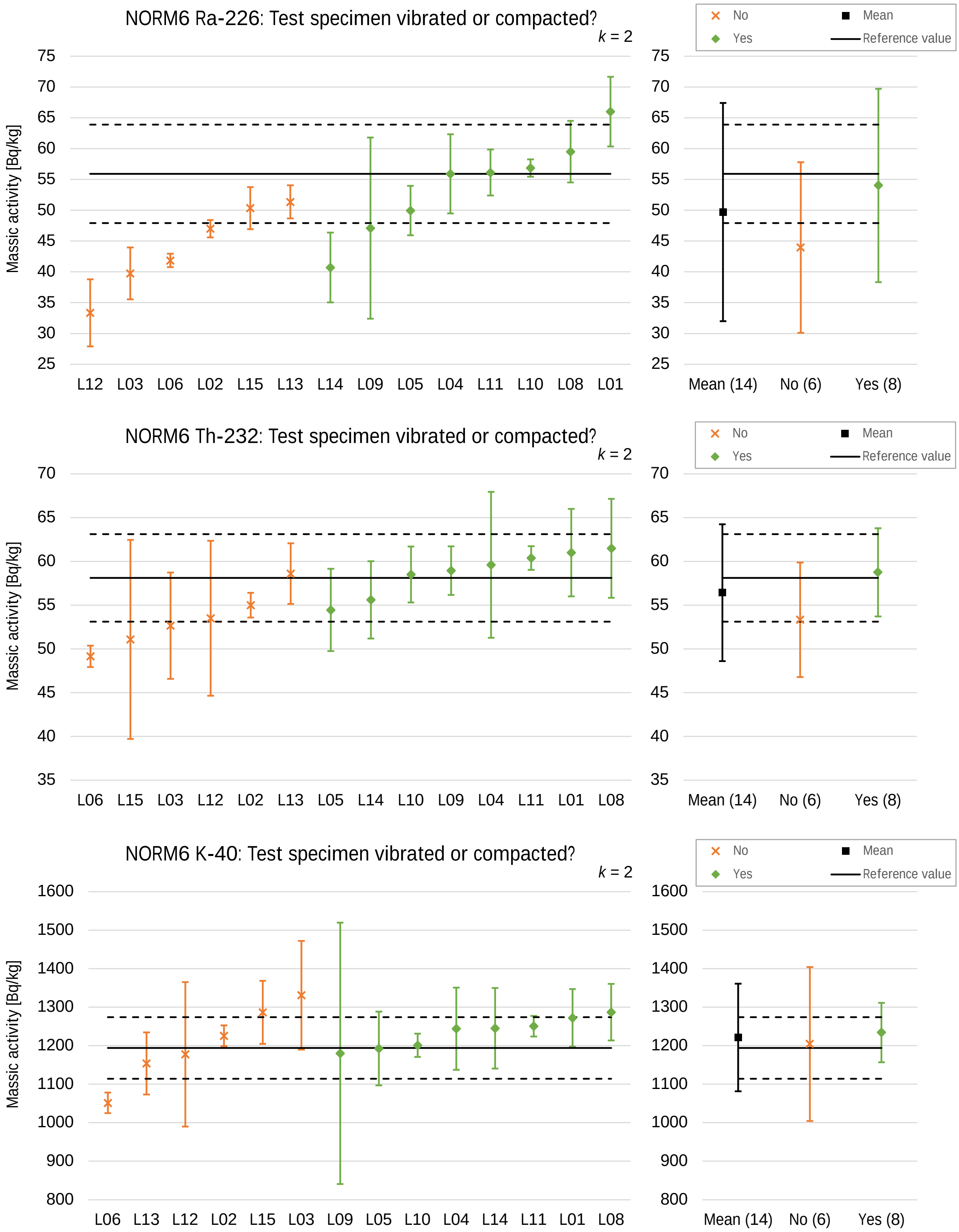


Table 28. Mean results from participants who indicated that they yes/no made the test specimen container radon tight.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Test specimen container made radon tight?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	86.1 (2.9)	80.2 (6.4)
	Th-232	49.4 (3.8)	52.0 (3.5)	48.4 (3.5)
	K-40	187 (13)	186 (10)	187 (15)
NORM2	Ra-226	31.4 (3.8)	32.9 (1.3)	30.8 (4.4)
	Th-232	23.7 (2.0)	24.6 (2.3)	23.3 (1.9)
	K-40	327 (27)	334 (17)	324 (31)
NORM3	Ra-226	31.3 (3.2)	32.5 (2.3)	30.7 (3.5)
	Th-232	23.1 (2.6)	23.5 (1.8)	23.0 (3.0)
	K-40	310 (18)	318 (22)	307 (17)
NORM4	Ra-226	22.7 (5.0)	27.4 (7.5)	20.8 (2.0)
	Th-232	37.1 (2.5)	36.6 (3.2)	37.3 (2.3)
	K-40	58 (10)	62 (9)	57 (10)
NORM5	Ra-226	118 (21)	122 (19)	117 (23)
	Th-232	60 (13)	64 (9)	58 (15)
	K-40	1460 (216)	1390 (142)	1489 (239)
NORM6	Ra-226	50 (9)	54 (11)	48 (8)
	Th-232	56.4 (3.9)	56.7 (5.8)	56.3 (3.3)
	K-40	1221 (70)	1209 (108)	1226 (55)

1 Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 37. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants made the test specimen container radon tight, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

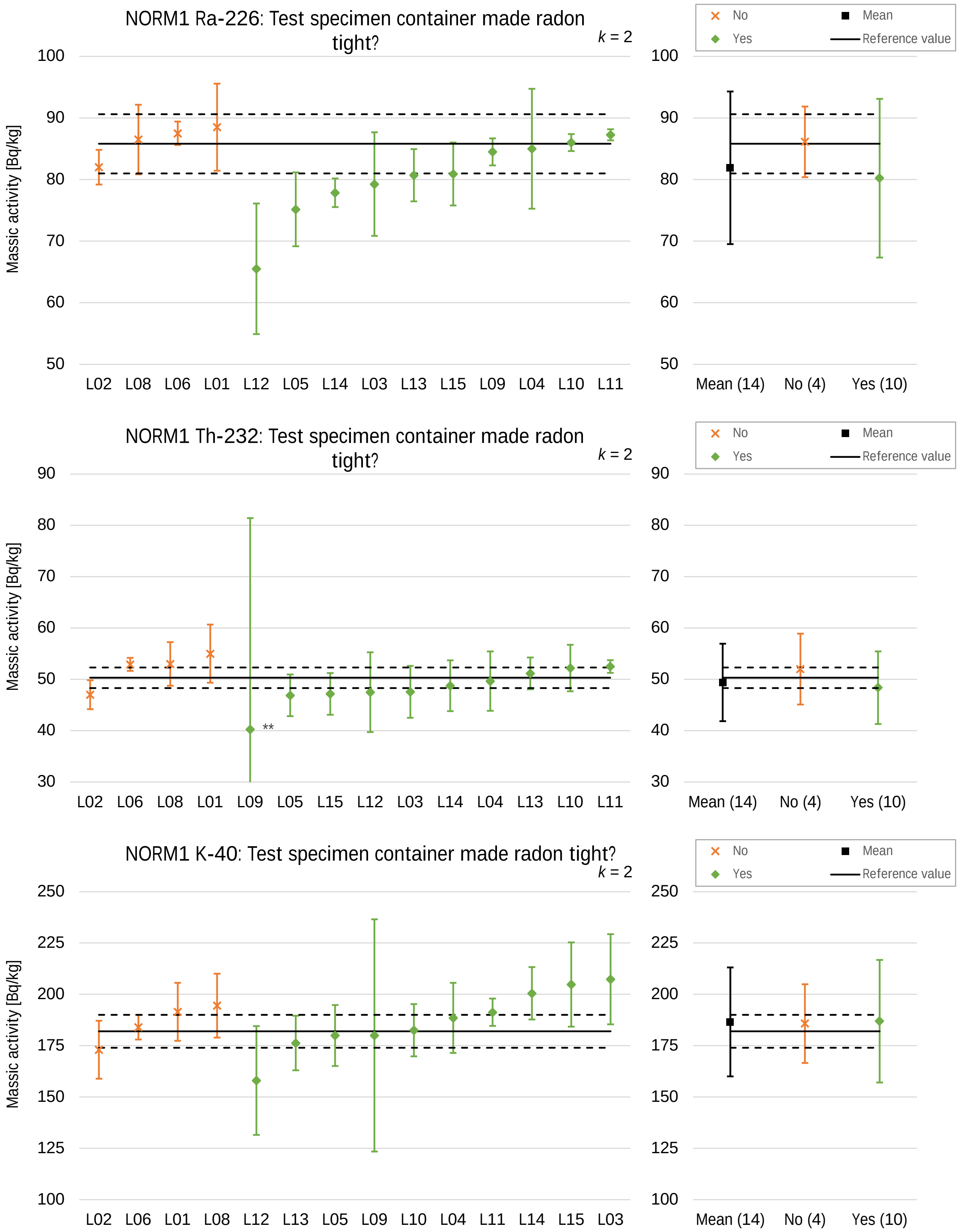


Figure 38. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants made the test specimen container radon tight, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

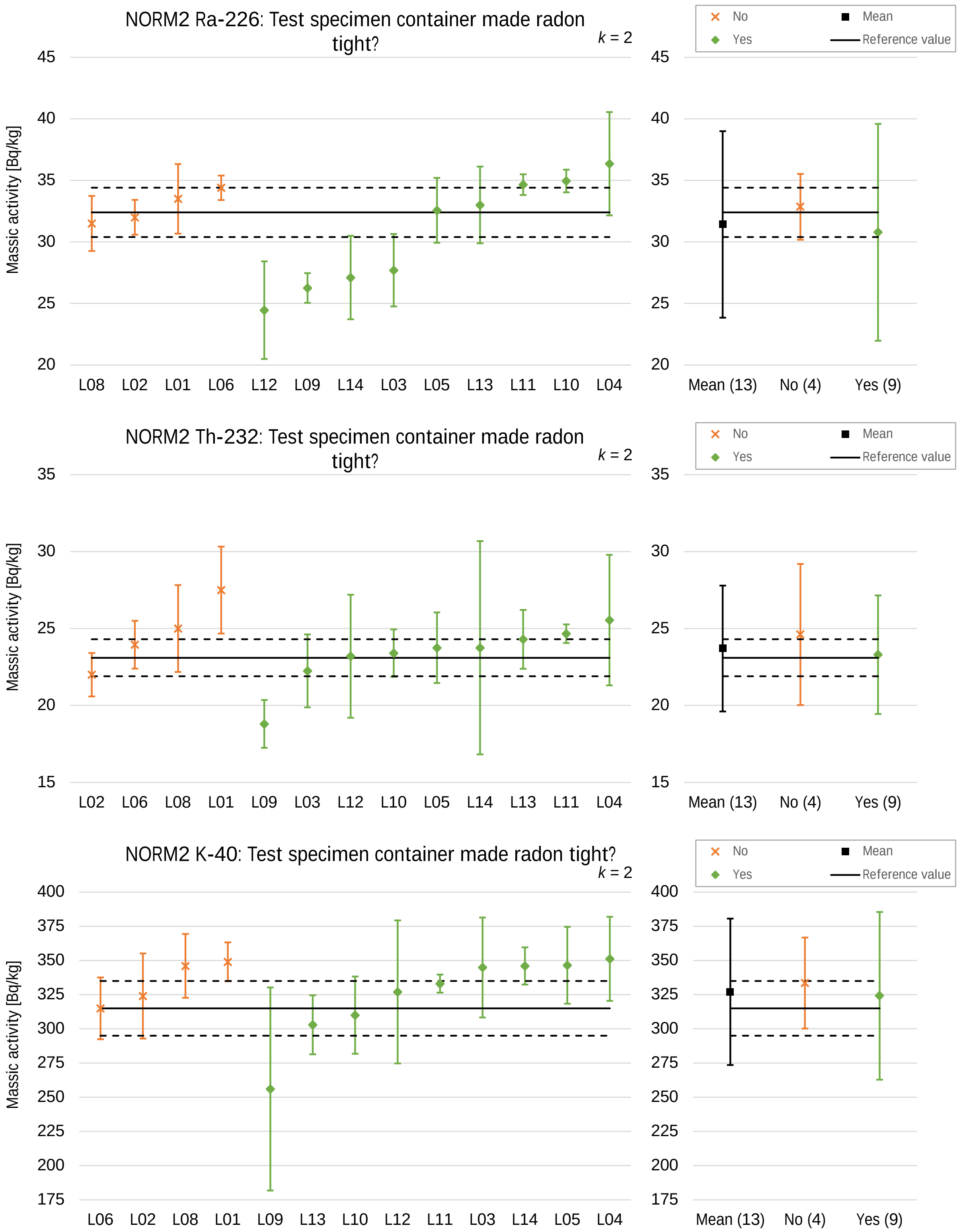


Figure 39. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants made the test specimen container radon tight, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

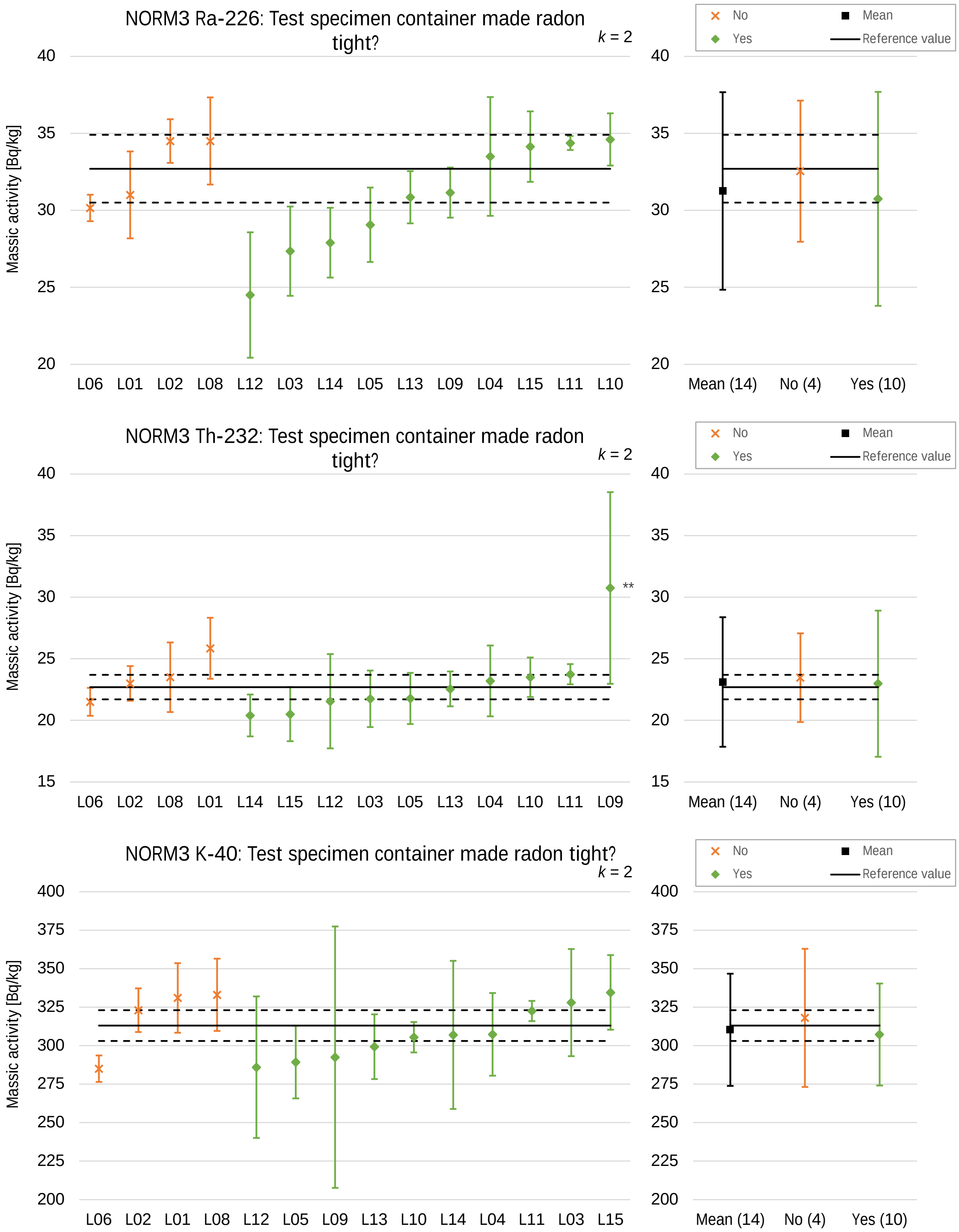


Figure 40. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants made the test specimen container radon tight, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

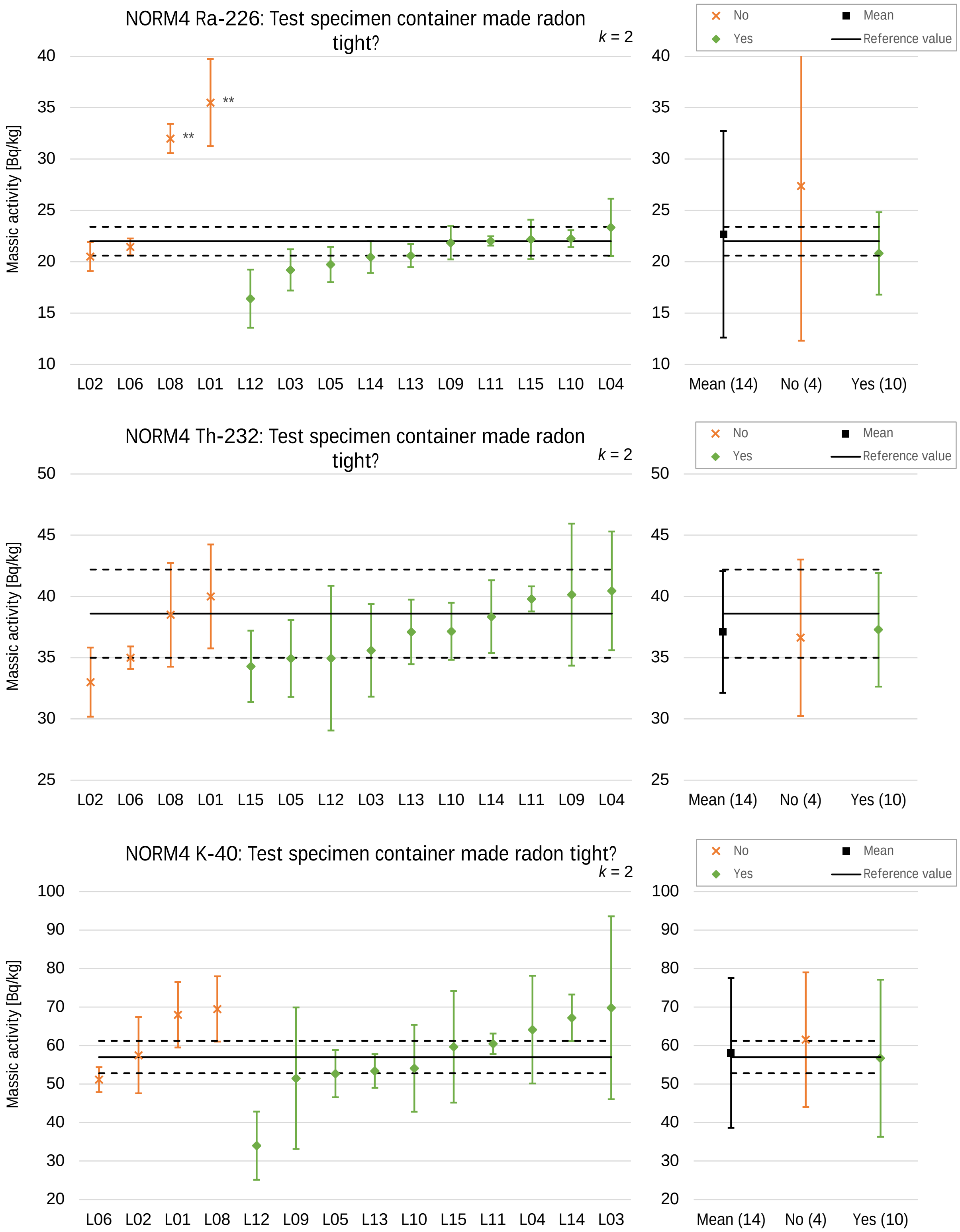


Figure 41. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants made the test specimen container radon tight, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

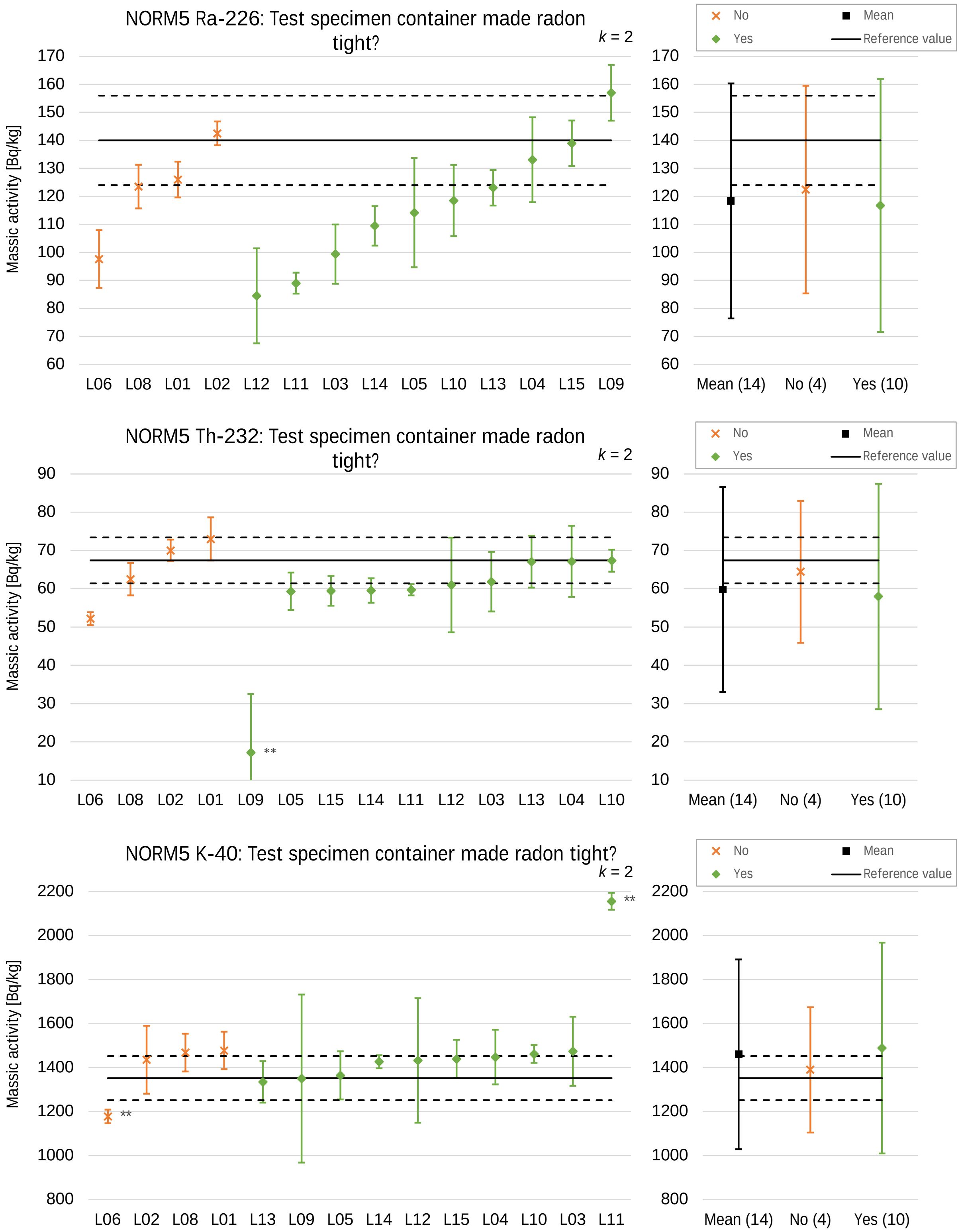
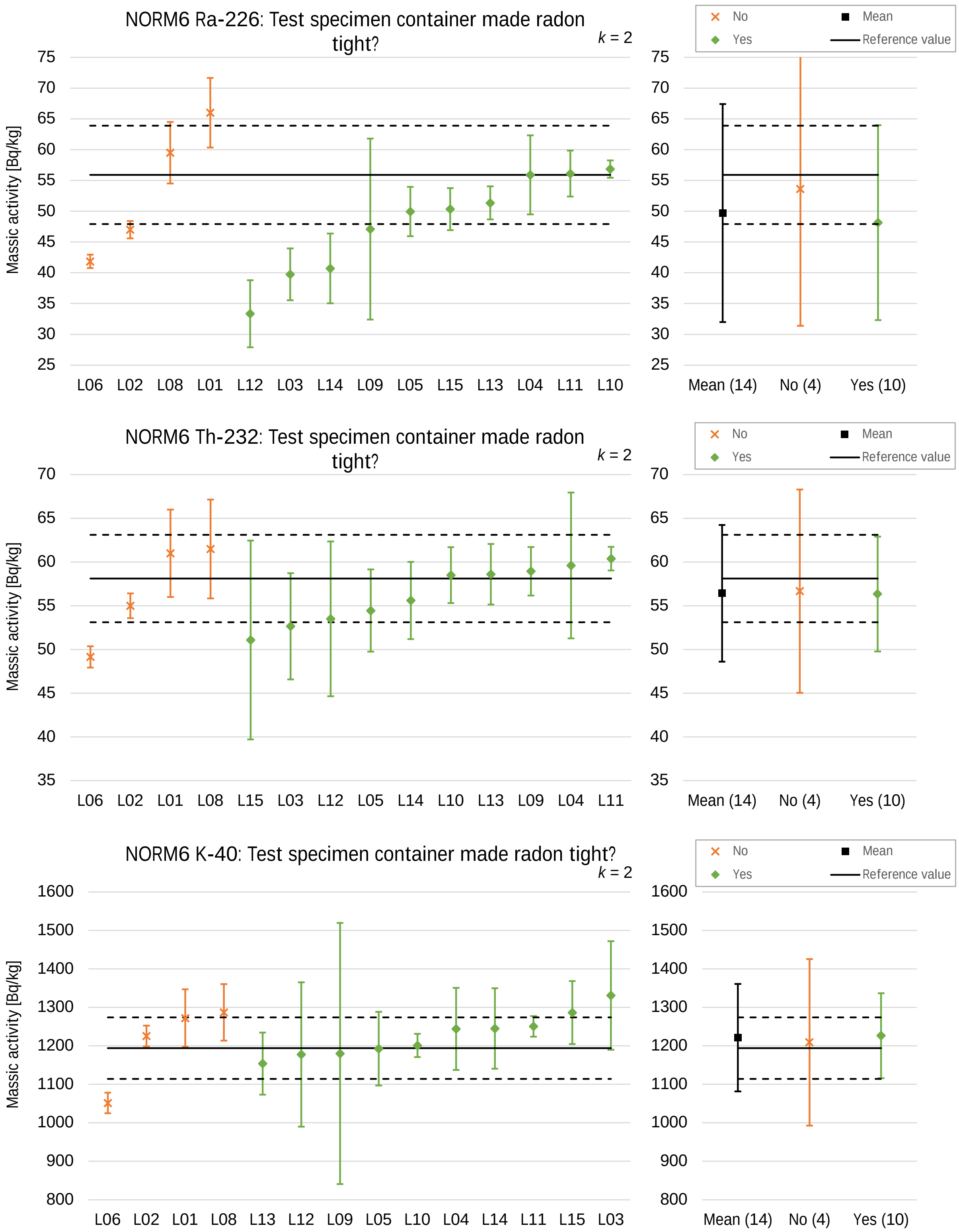


Figure 42. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants made the test specimen container radon tight, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.



6.4 Determination of dry matter

WD EN 17216 requires that the activity concentration or "massic activity" is reported on the dry matter. The dry matter shall be determined as in Clause 6.3.2.3, or the test specimen shall be dried completely following the procedure in Clause 7.2.3.3.

Q 3.1 of the reporting survey asked the participants if they followed closely section 6.3.2.3 for dry matter content determination, and if not, to describe deviations (See Table 29). Participants L10 and L11 did not determine the dry matter but dried the test specimen itself. Participant L13 applied Clause 6.3.2.3 to determine the dry matter for the two NORM3 samples only and another, unspecified method for the other samples. Participant L06 did not determine the dry matter correction factor, nor did the participant dry the test specimen. The participant was contacted and provided clarification. The participant assumed that the material was dry. Therefore, the results of L06 are for the material as provided, not taking into account possible moisture. Participant reported a correction factor for dry mass $\eta = 1$.

From the answers provided, it appears that it was not clear for all participants that they have the choice to determine the dry matter correction factor with a test portion or to fully dry the test specimen itself. The standard could be more explicit on this.

Equation (1) of WD EN 17216 defines the correction factor for dry mass η as the quotient of the dry mass by the fresh mass. Since the fresh mass is close to but slightly larger than the dry mass, numbers close to but slightly lower than 1 are expected for η. Nevertheless, two participants (L03 and L15) report a dry mass correction factor between zero and a few percent, while participant L09 reports a number between 0.001 and 0.024 without adding the percentage symbol. The numbers provided by the three participants were interpreted as moisture content (mass) and recalculated to correspond with the definition of η.

Table 29. Determination of the dry matter by the participants applying Clause 6.3.2.3. Cells are coloured green where the participants followed the normative requirements from the draft standard.

Participant	Q 3.1: Dry matter determined?	Comment
L01	YES	
L02	YES	
L03	YES	
L04	YES	
L05	YES	
L06	NO	Results not reported in dry mass.
L08	YES	
L09	YES	
L10	NO	Dried the test specimen following Clause 7.2.3.3
L11	NO	Dried the test specimen following Clause 7.2.3.3
L12	YES	
L13	YES (NORM3)	Another method applied for the other materials
L14	YES	
L15	YES	

Table 30 and Figure 43 presents the average correction factor for dry mass calculated for each of the materials based on the values reported by the participants, not including the reported value from participant L06. The mean results of participants who determined the dry mass correction factor compared with participant L06 are shown in Table 31 and Figure 44 to Figure 49.

Since participant L06 assumed that the materials were dry, one would expect that participant L06 reports a lower massic activity, especially for the materials with the largest moisture content (or smallest correction factor for dry mass). The materials with the largest moisture content are NORM3 and NORM6 and for these the laboratory bias of L06 is indeed negative. However, for NORM2 participant L06 shows a positive bias. For NORM4 the bias is negative as expected. NORM1 and NORM5 have a very low moisture content. The reported results for by participant L06 for NORM1 are acceptable, but the participant reported extremely low values for NORM5. This inconsistent behaviour of the results from participant L06 and the non-negligible variability between the participants together with the already low moisture content of the materials makes it difficult to draw conclusions on the impact of not applying the correction for dry mass.

based on the available data and low moisture content of the materials, the impact of not applying the correction for dry mass cannot be proven

Table 30. Correction factor for dry mass, averaged for each material over all participants except L09.

Material	Average correction factor for dry mass, $\boldsymbol{\eta}$ ([1])
NORM1	0.998 (0.004)
NORM2	0.985 (0.010)
NORM3	0.970 (0.015)
NORM4	0.989 (0.012)
NORM5	0.998 (0.003)
NORM6	0.980 (0.013)

1 The number between parentheses is the standard deviation between the reported values for that material.

Figure 43. Average correction factor for dry mass as reported by the participants. The average for each material is calculated not including the reported value of 1 by participant L06, who did not determine the dry mass. Data is sorted from higher moisture content to lower. Error bars correspond with two standard deviations ($k = 2$).

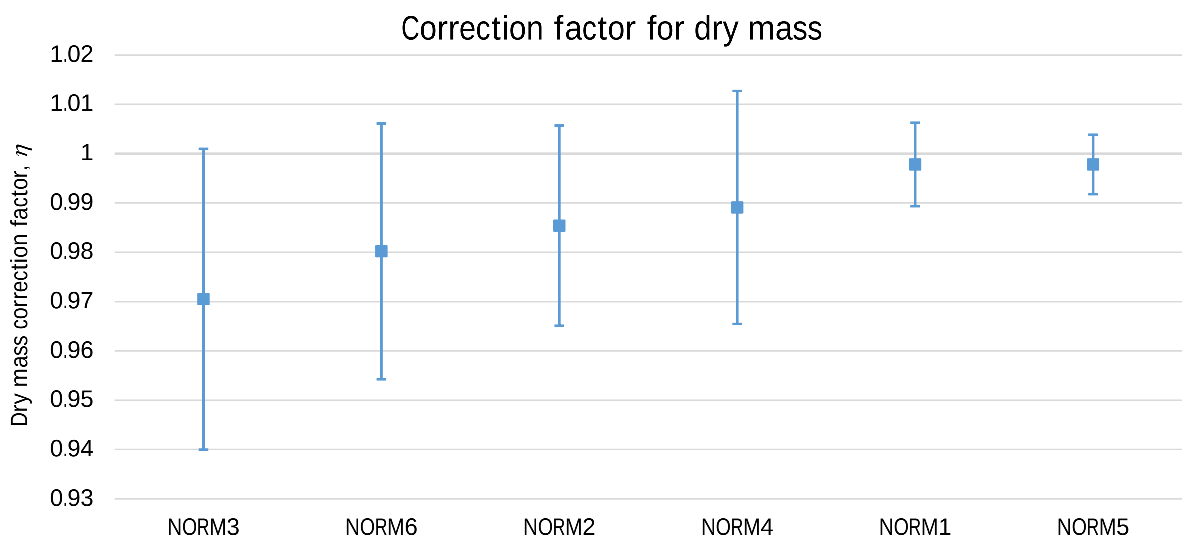


Table 31. Mean results from participants who indicated that they determined the dry matter or dried the test specimen completely.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Dry matter determined or test specimen completely dried?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	87.5 (1.0)	81.5 (6.2)
	Th-232	49.4 (3.8)	52.9 (0.6)	49.1 (3.8)
	K-40	187 (13)	184 (3)	187 (14)
NORM2	Ra-226	31.4 (3.8)	34.4 (0.5)	31.2 (3.8)
	Th-232	23.7 (2.0)	24.0 (0.8)	23.7 (2.1)
	K-40	327 (27)	315 (11)	328 (28)
NORM3	Ra-226	31.3 (3.2)	30.2 (0.4)	31.3 (3.3)
	Th-232	23.1 (2.6)	21.5 (0.6)	23.2 (2.7)
	K-40	310 (18)	285 (4)	312 (17)
NORM4	Ra-226	22.7 (5.0)	21.5 (0.4)	22.8 (5.2)
	Th-232	37.1 (2.5)	35.0 (0.5)	37.3 (2.5)
	K-40	58 (10)	51 (2)	59 (10)
NORM5	Ra-226	118 (21)	98 (5)	120 (21)
	Th-232	60 (13)	52 (1)	60 (14)
	K-40	1460 (216)	1178 (16)	1482 (208)
NORM6	Ra-226	49.7 (8.9)	41.9 (0.5)	50.3 (8.9)
	Th-232	56.4 (3.9)	49.2 (0.6)	57.0 (3.4)
	K-40	1221 (70)	1052 (13)	1234 (52)

[1] Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 44. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants determined the dry matter or dried the test specimen completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

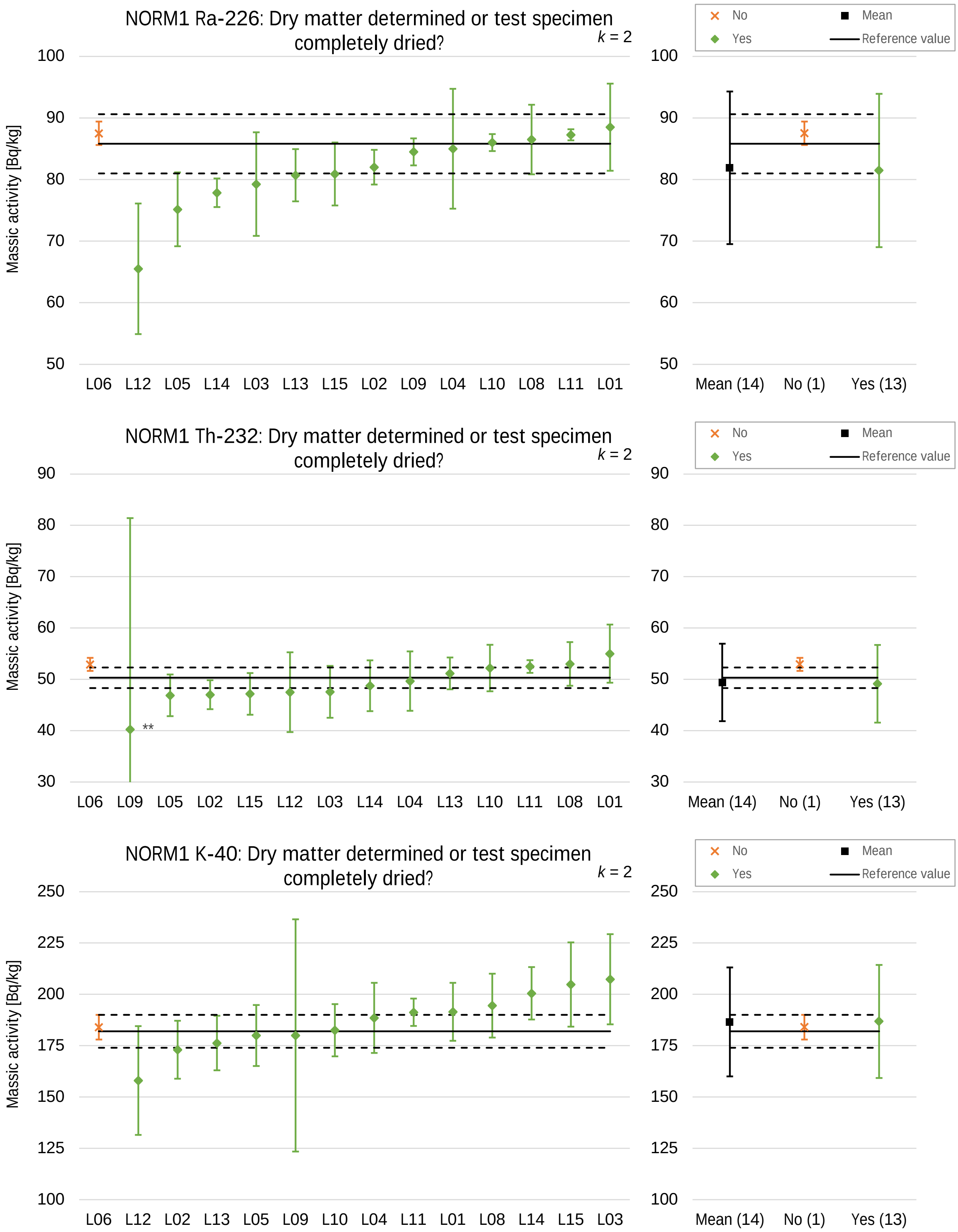


Figure 45. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants determined the dry matter or dried the test specimen completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

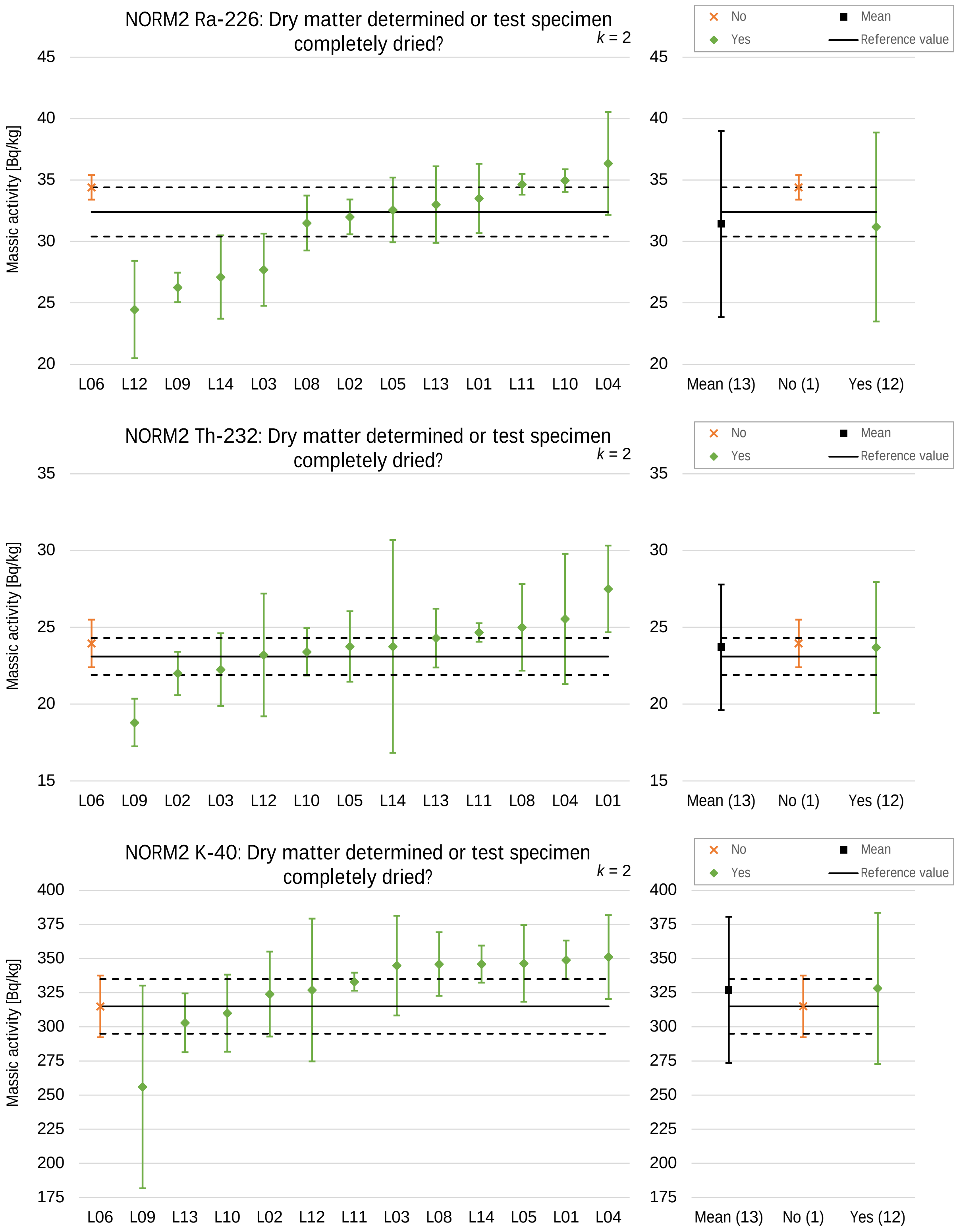


Figure 46. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants determined the dry matter or dried the test specimen completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

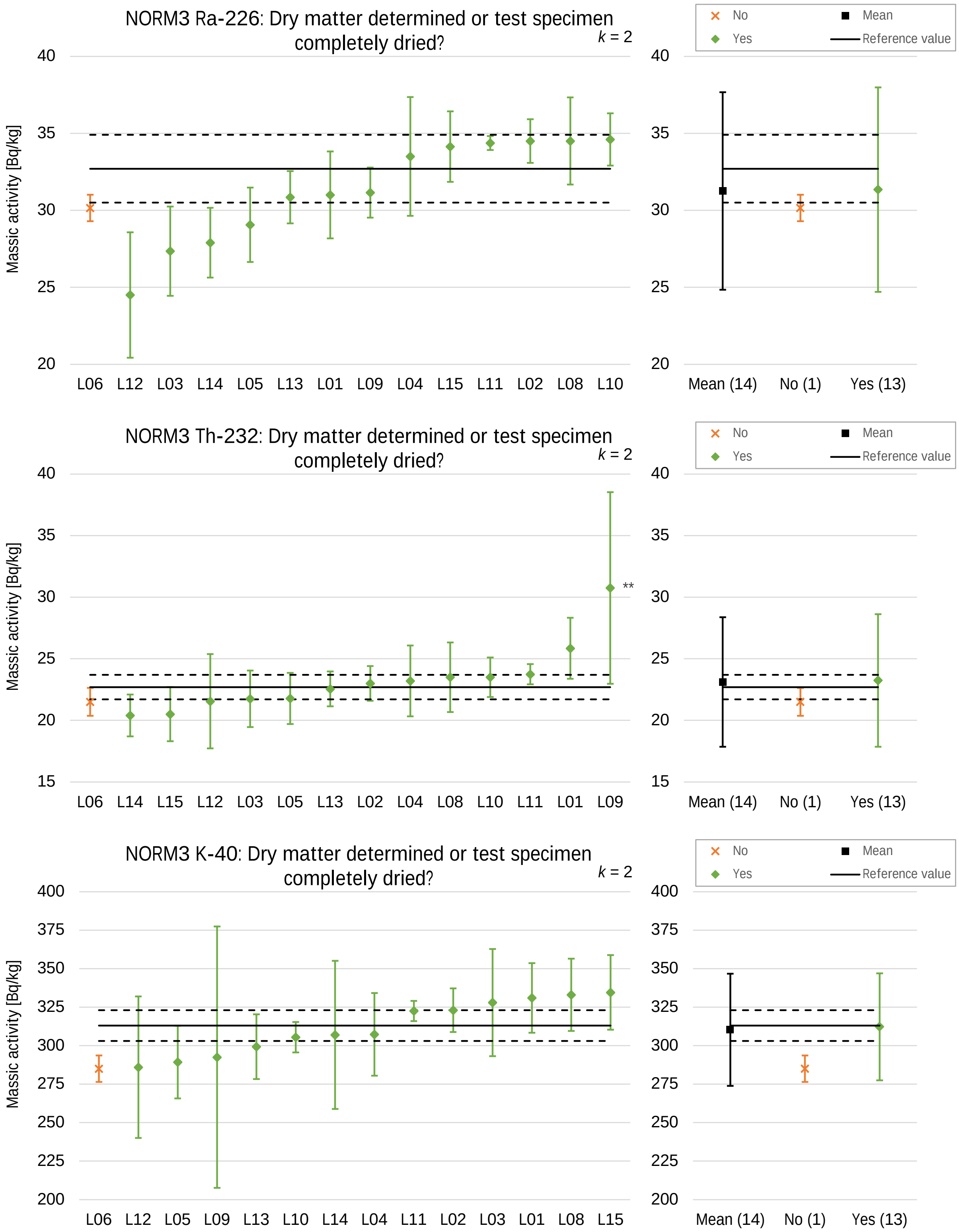


Figure 47. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants determined the dry matter or dried the test specimen completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

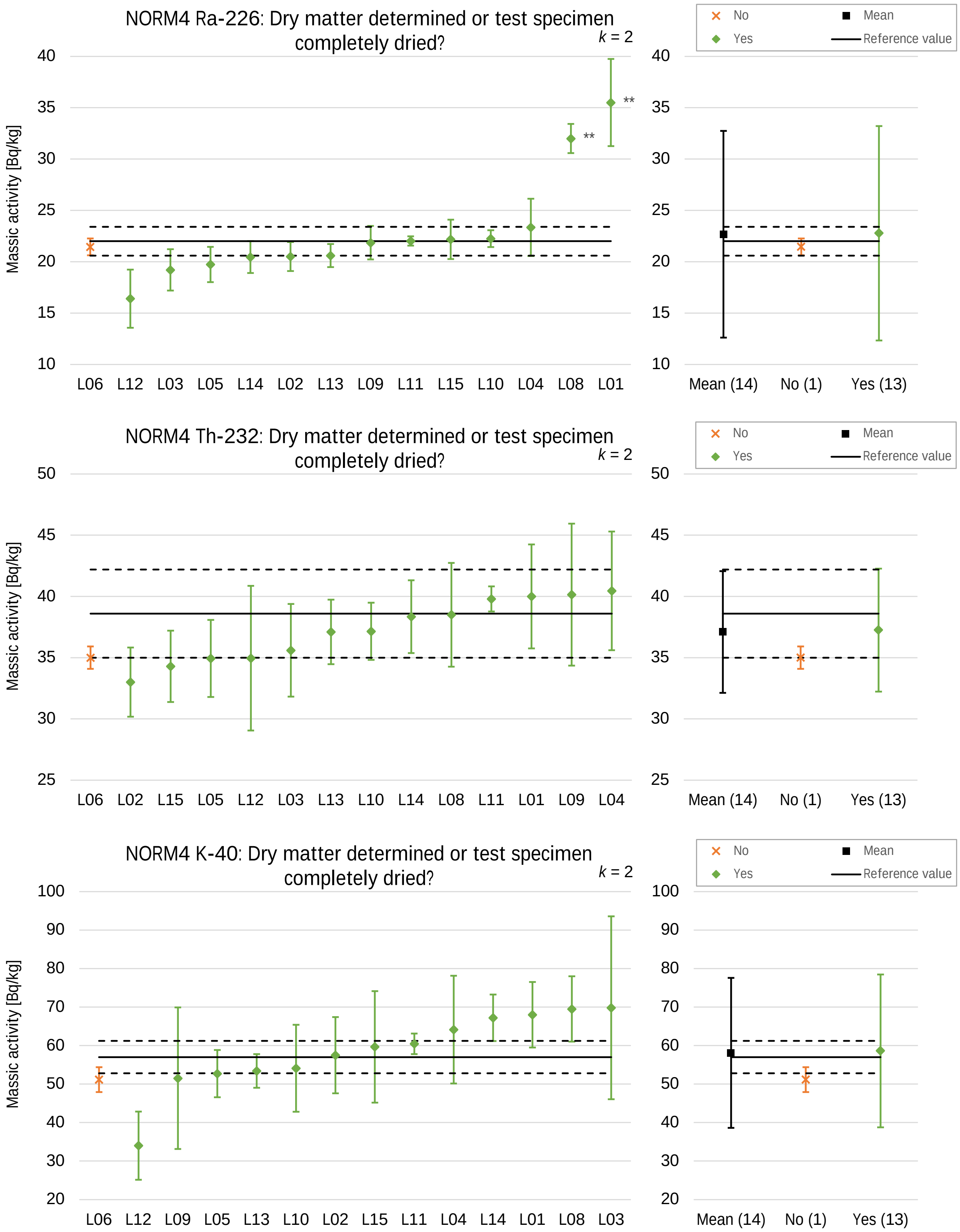


Figure 48. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants determined the dry matter or dried the test specimen completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

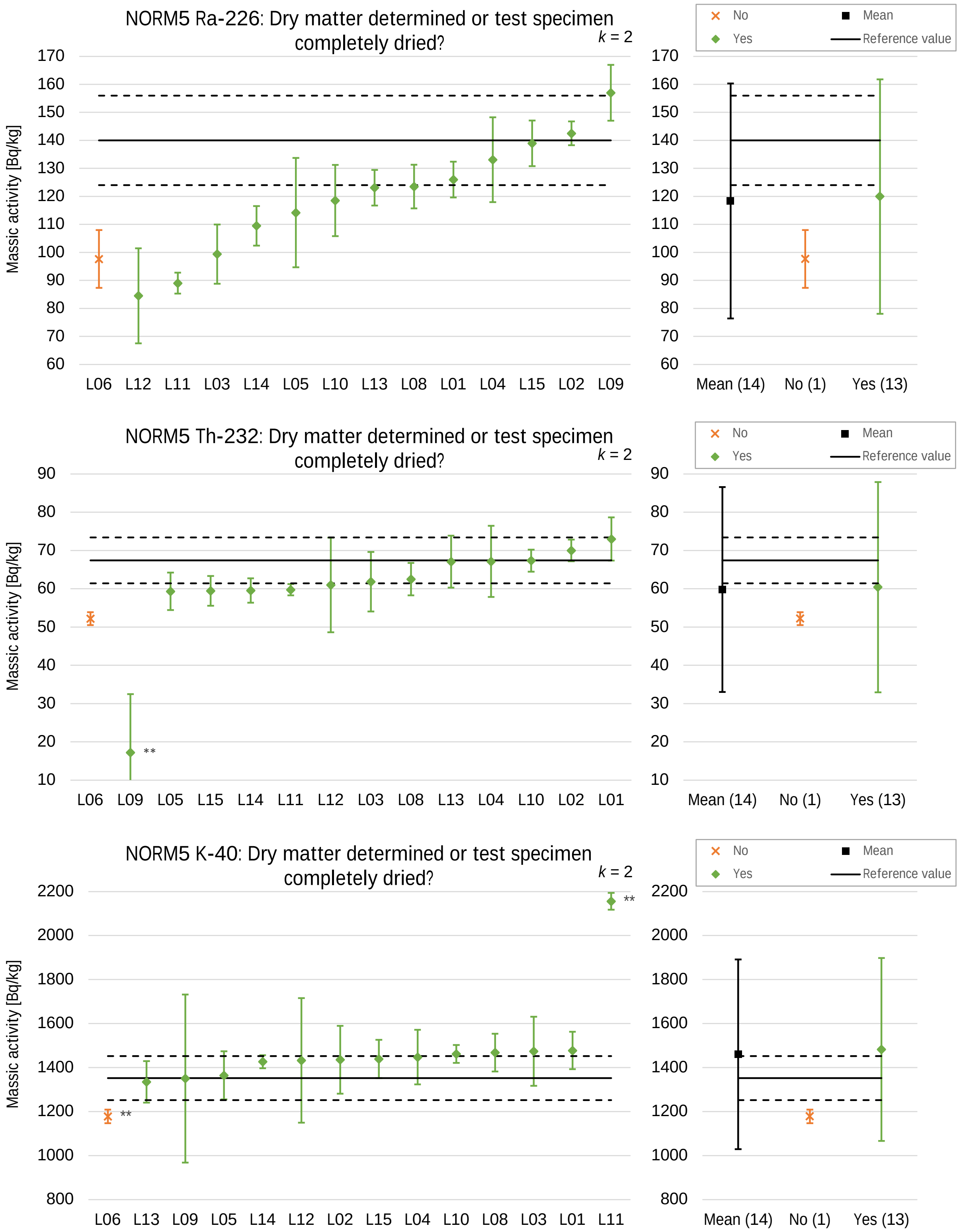
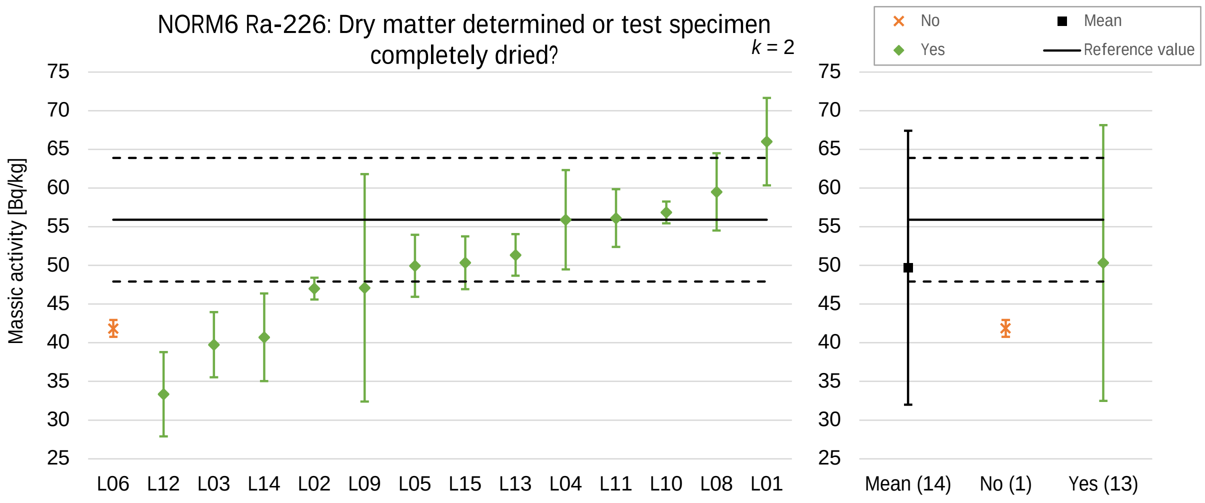
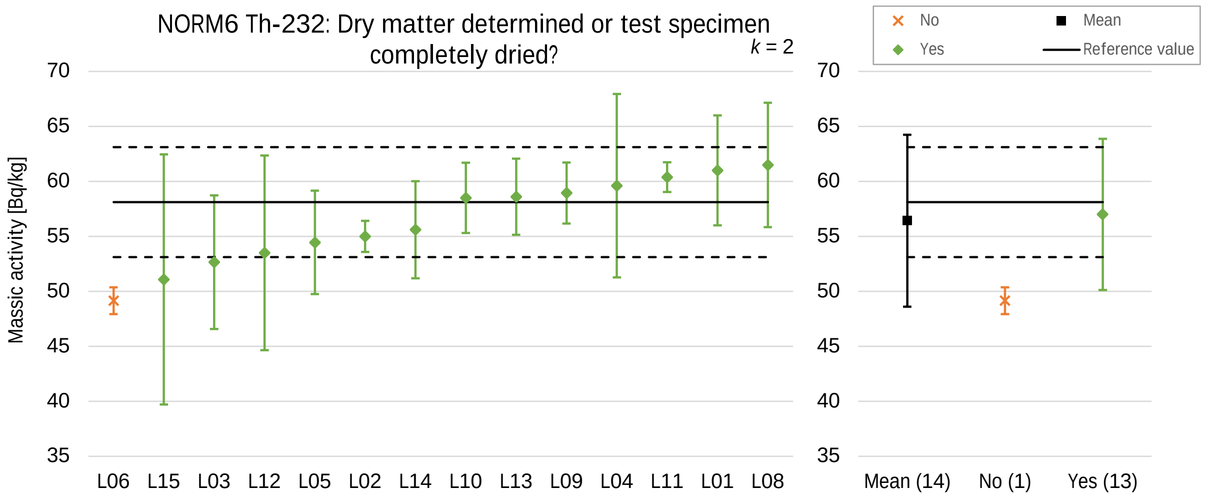
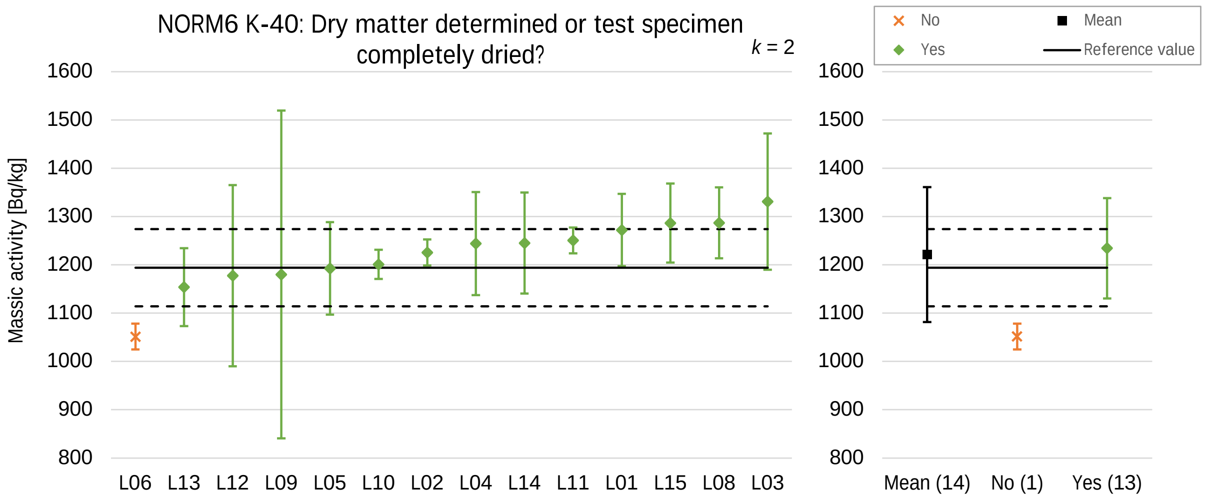


Figure 49. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants determined the dry matter or dried the test specimen completely, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.







6.5 Calibration of the detection efficiency

Clause 7.2.2.2 of WD EN 17216 allows the three methods specified in ISO 20042 [13] for the determination of the detection efficiency. The participants provided the answers as in Table 32. The most used method is the measurement of the full energy photopeak detection efficiency as a function of energy (65 %). 29 % compared with a calibration source and 21 % used Monte Carlo simulations or numerical models. L06 and L09 used two methods, but it is not clear how.

Table 32. Answers provided by the participants on how they performed the calibration of the de detection efficiency.

Participant	Q 4.8: Calibration of the detection efficiency		
	a) Direct comparison with a calibration source	b) Measurement of the full energy photopeak detection efficiency as a function of energy	c) Monte Carlo simulations or numerical models
L01		✓	
L02	✓		
L03		✓	
L04			✓
L05		✓	
L06 ([1])	✓	✓	
L08		✓	
L09 ([1])	✓	✓	
L10	✓		
L11			✓
L12		✓	
L13		✓	
L14		✓	
L15			✓
% of total	29 %	65 %	21 %

[1] Two participants indicated that they uses two methods.

For each test property, the mean results over the participants who used the same calibration method for the detection efficiency was determined. No significant differences could be observed between the mean results of the three different methods.

no significant differences could be observed between the mean results obtained by the three different methods for determining the detection efficiency

6.6 Calculation of the activity

Clause 8.3 of WD EN 17216 specifies how the activity is to be calculated, taking into account correction factors of attenuation, true-summing, dead-time, decay and positioning. The reporting questionnaire included three questions to assess how well the participants followed the specified method:

— Q 4.10 asked if attenuation corrections were made (correction factor f_1);

— Q 4.11 asked if true-summing corrections were made (correction factor f_2);

— Q 4.14 asked the participants if they calculated the activity according to Clause 8.3 of WD EN 17216, and if not, where they diverted.

The reporting survey did not include specific questions on the dead-time correction factor f_3, the decay correction factor f_4 and the correction factor f_5 related to positioning and/or filling height. However, the participants were asked if they took these effects into account in their uncertainty budget (See Section 6.7).

It must be noted that for the determination of the activities of Ra-226, Th-232 and K-40, no direct impact of true-summing is expected. However, when the calibration curve is established from measurements involving Co-60 and then extrapolated to K-40, true-summing may become relevant. Table 33 shows the replies on the three questions above.

Table 33. Calculation of the activity according to Clause 8.3 of the draft standard. Cells are coloured green where the participants followed the normative requirements from the draft.

Participant	Q 4.10: Attenuation corrections made?	Q 4.11: True-summing corrections made?	Q 4.14: Activity calculated according to Clause 8.3?	Comment
L01	YES	NO	NO	Barba-Lobo et al. (2021) was followed
L02	NO	NO	YES	
L03	NO	NO	YES	
L04	YES	YES	YES	
L05	YES	YES	YES	
L06	NO	YES	YES	
L08	YES	NO	YES	
L09	NO	NO	NO	For only one measurement per test specimen
L10	YES	YES	YES	
L11	YES	NO	YES	
L12	NO	NO	NO	True-summing, dead-time and positioning correction factors not applied
L13	YES	YES	YES	
L14	NO	NO	NO	Attenuation, true-summing, dead-time, decay and positioning correction factors not applied
L15	NO	NO	YES	

It should also be noted that not applying certain correction factors is only problematic when these factors have a significant impact on the measurement result. Some laboratories indeed mention that true-summing corrections are not necessary for certain radionuclides.

Also, the answers to Q 4.10 and Q 4.11 are somewhat inconsistent with the answers to Q 4.14, but this is due to the way the questions were posed. A participant who claims not to have made attenuation corrections may have assumed that the effect is negligible and hence $f_1 \approx 1$. Then indeed no correction is made.

Observing the data from Table 34 and Figure 50 to Figure 55, no significant difference could be observed between participants who claimed to have applied attenuation corrections and those who claimed not.

no significant difference could be observed between participants who claimed to have applied attenuation corrections and those who claimed not

Likewise, from Table 35 and Figure 56 to Figure 61, it is concluded that there is no significant difference between the participants who claimed to have applied true-summing corrections and those who did not.

no significant difference could be observed between participants who claimed to have applied true-summing corrections and those who claimed not

On the question if the activity was calculated as in Clause 8.3 of the standard, no difference could be observed between the participants who claimed to have followed the standard and those who did not (See Table 36 and Figure 62 to Figure 67)

no significant difference could be observed between participants who claimed to have calculated the activity as in Clause 8.3 and those who claimed not

Table 34. Mean results from participants who indicated that they made / not made attenuation corrections.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Attenuation corrections applied?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	79.6 (7.0)	84.2 (4.7)
	Th-232	49.4 (3.8)	47.3 (3.7)	51.5 (2.6)
	K-40	187 (13)	187 (18)	186 (7)
NORM2	Ra-226	31.4 (3.8)	28.7 (3.8)	33.8 (1.6)
	Th-232	23.7 (2.0)	22.3 (1.9)	24.9 (1.4)
	K-40	327 (27)	319 (33)	334 (20)
NORM3	Ra-226	31.3 (3.2)	30.0 (3.7)	32.6 (2.2)
	Th-232	23.1 (2.6)	22.8 (3.6)	23.4 (1.3)
	K-40	310 (18)	308 (21)	313 (17)
NORM4	Ra-226	22.7 (5.0)	20.3 (2.0)	25.1 (6.1)
	Th-232	37.1 (2.5)	35.9 (2.5)	38.3 (2.0)
	K-40	58 (10)	56 (12)	60 (7)
NORM5	Ra-226	118 (21)	119 (27)	118 (14)
	Th-232	60 (13)	54 (17)	65 (5)
	K-40	1460 (216)	1391 (101)	1530 (281)
NORM6	Ra-226	49.7 (8.9)	42.9 (5.7)	56.5 (5.3)
	Th-232	56.4 (3.9)	53.7 (3.2)	59.1 (2.4)
	K-40	1221 (70)	1214 (90)	1229 (48)

1 Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 50. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants applied attenuation corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

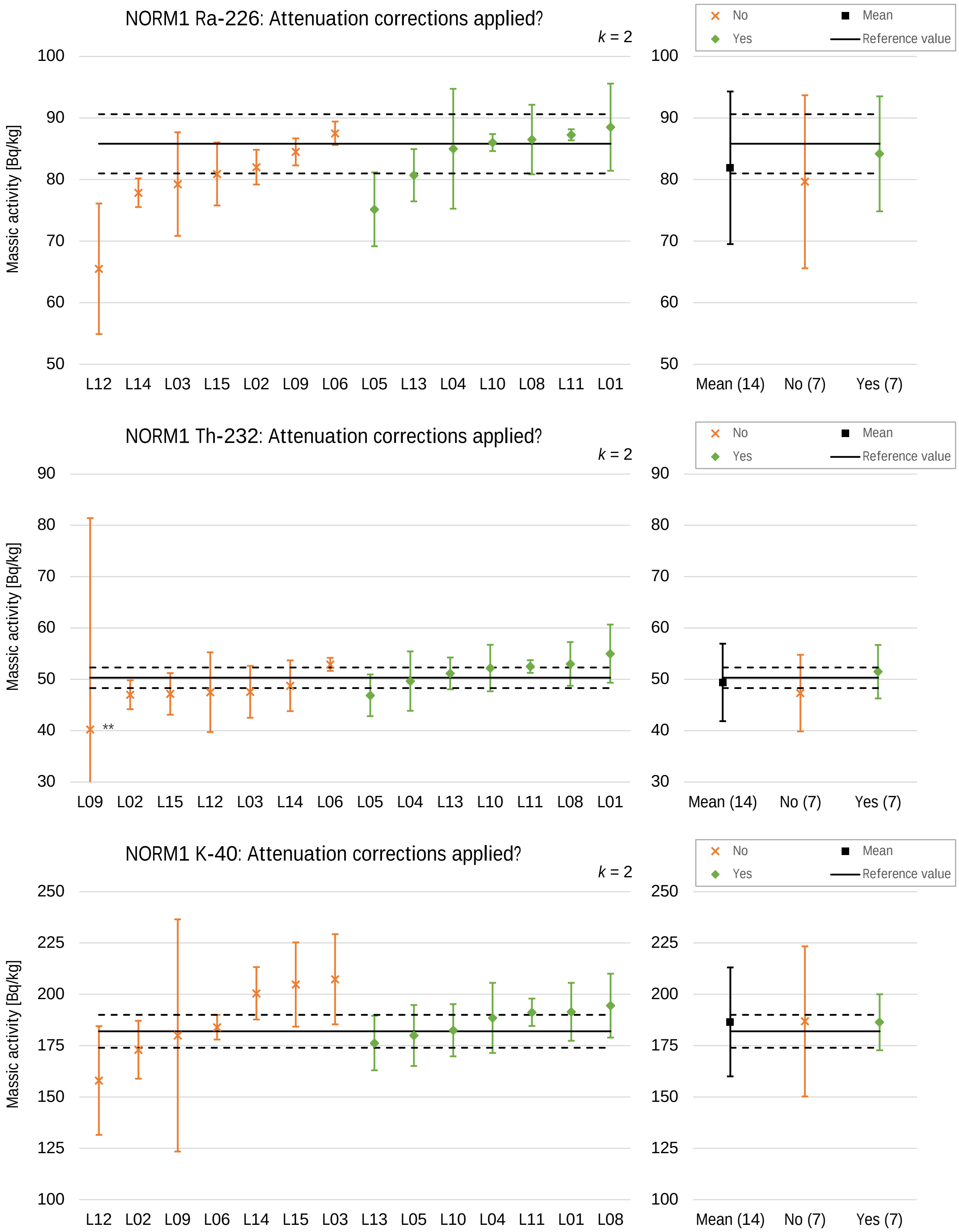
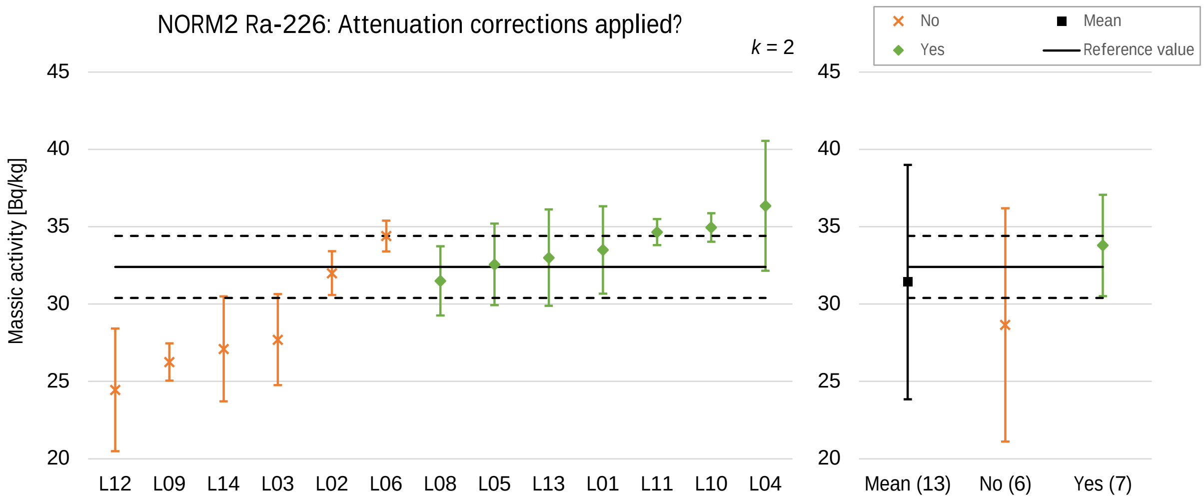
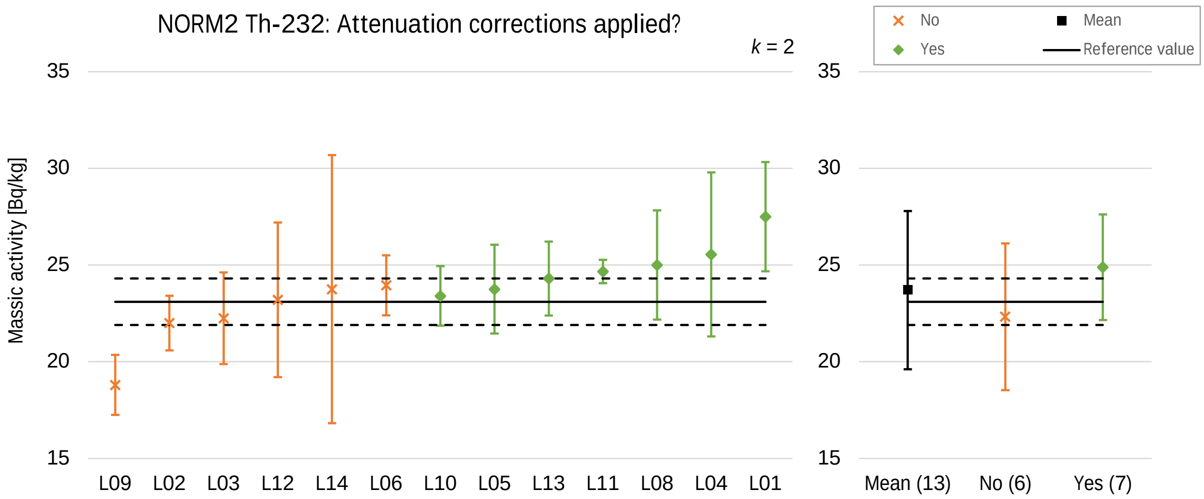


Figure 51. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants applied attenuation corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.





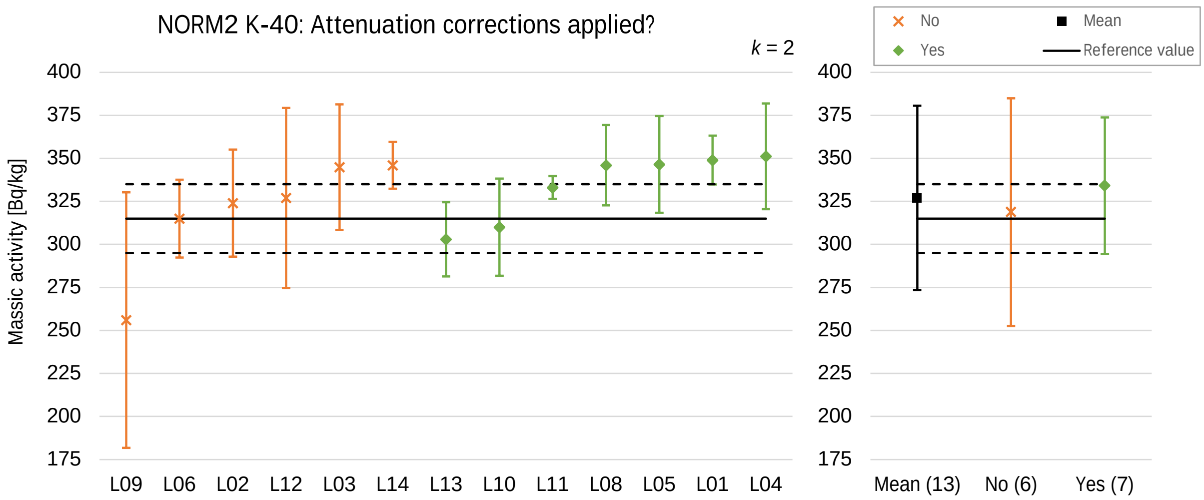


Figure 52. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants applied attenuation corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

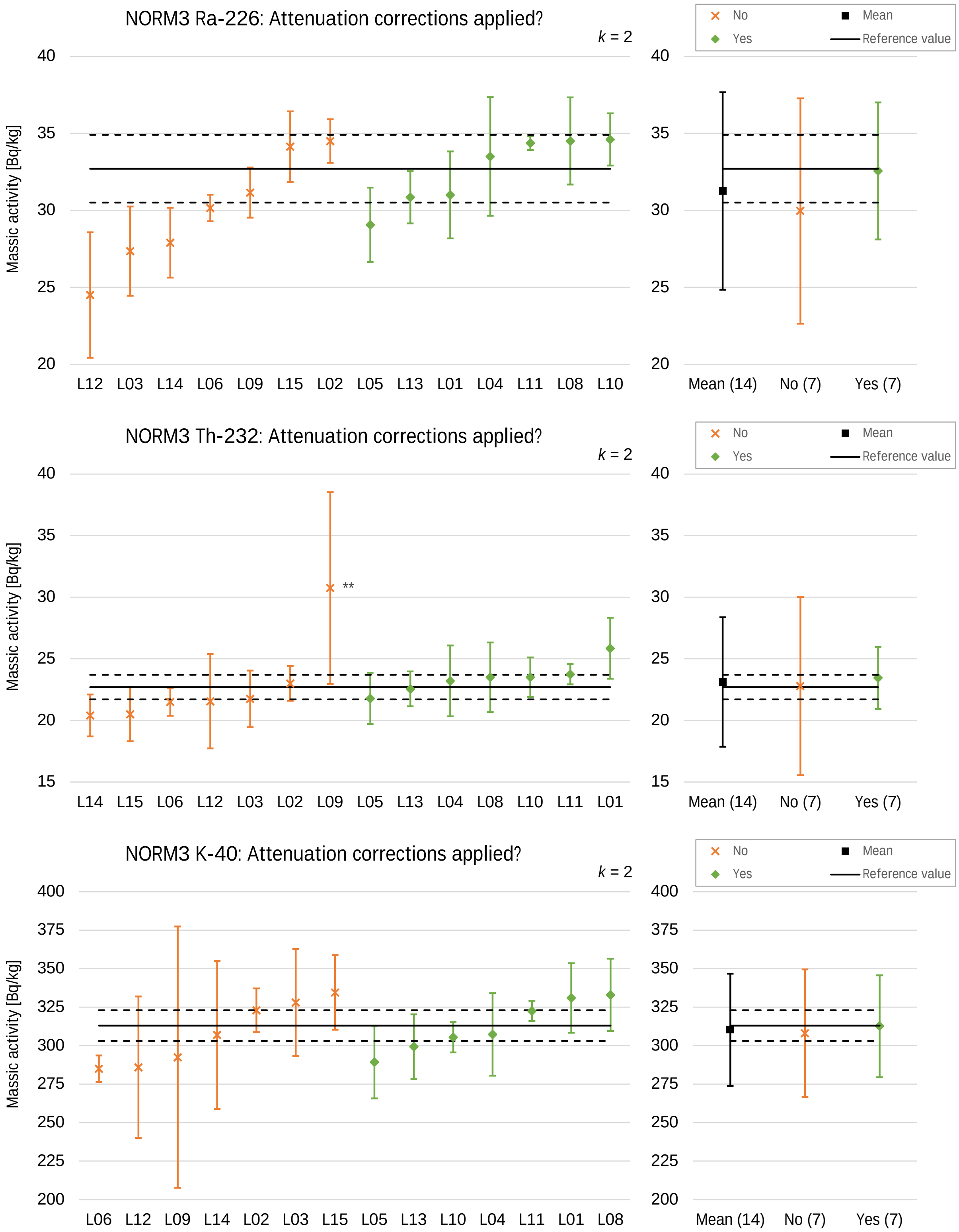


Figure 53. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants applied attenuation corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

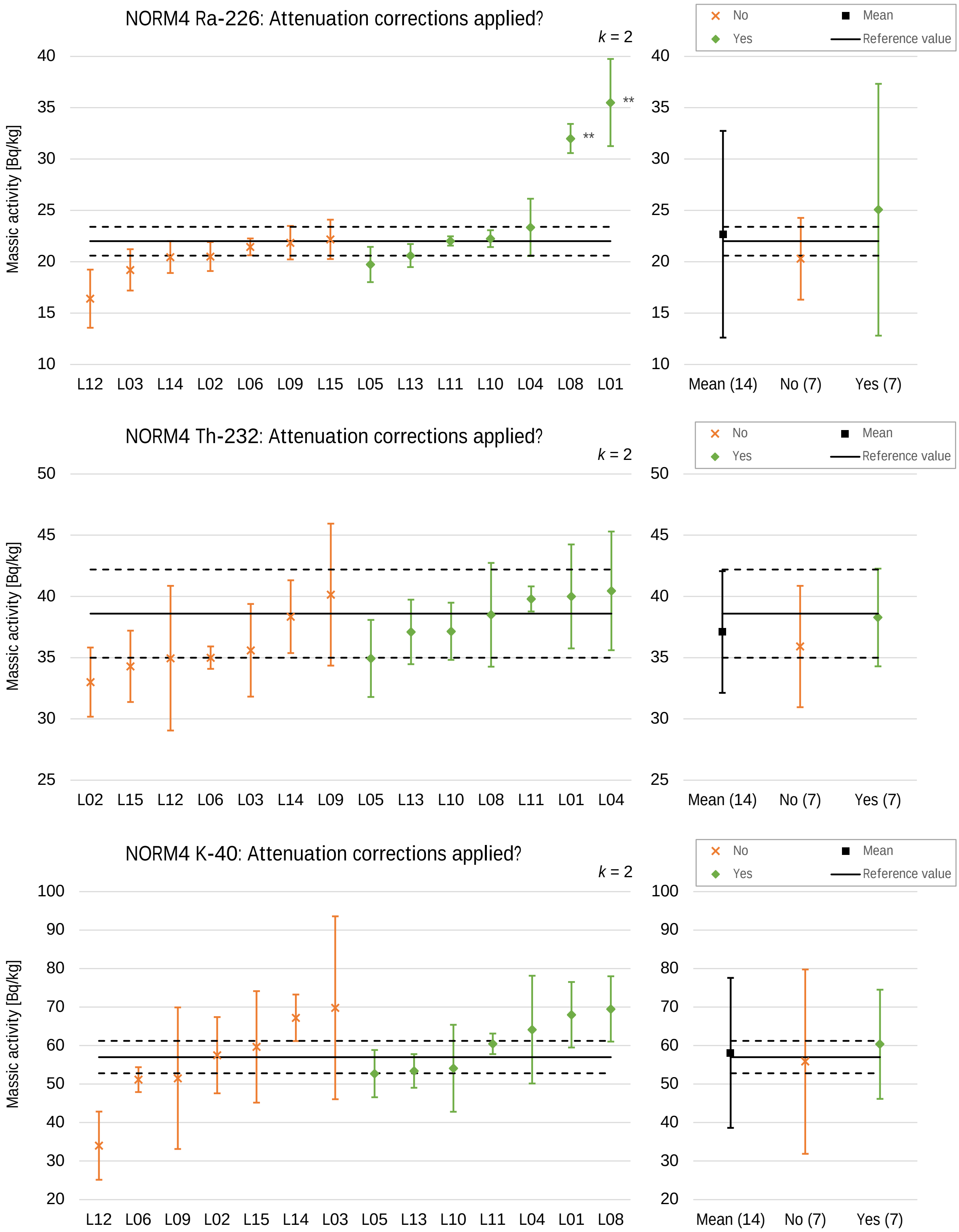


Figure 54. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants applied attenuation corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

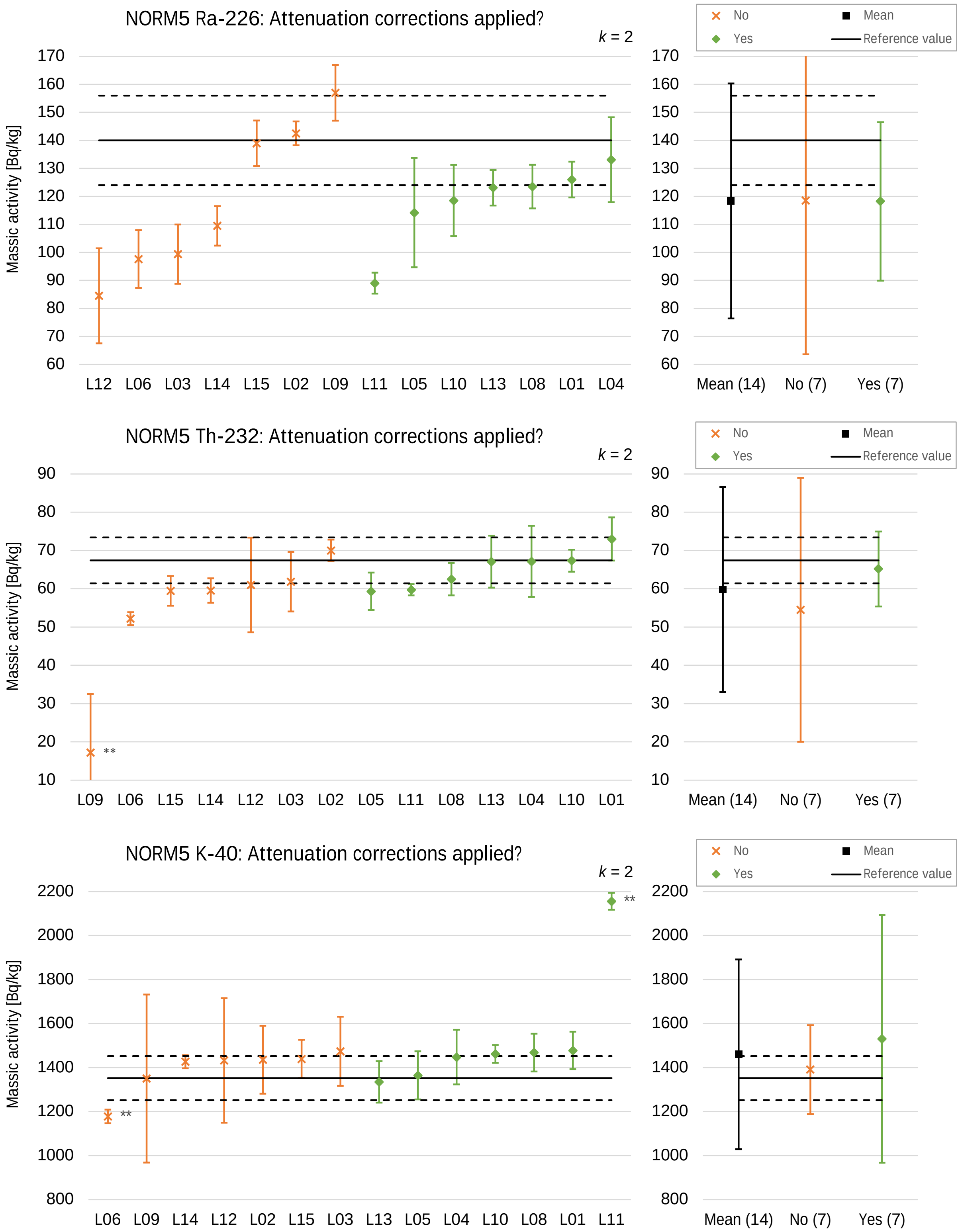


Figure 55. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants applied attenuation corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

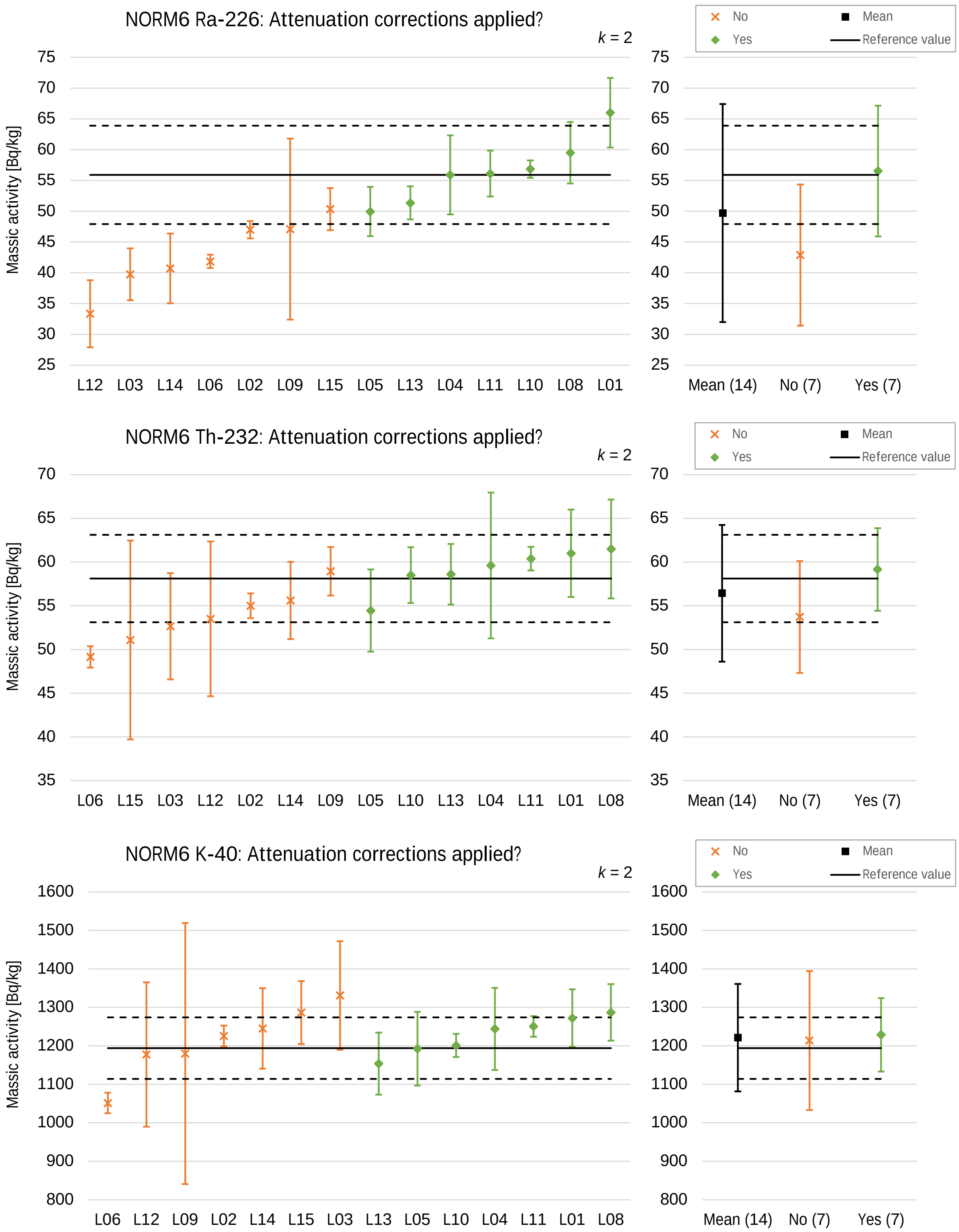


Table 35. Mean results from participants who indicated that they applied / not applied true-summing corrections.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	True-summing corrections applied?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	81.4 (7.0)	82.9 (5.0)
	Th-232	49.4 (3.8)	48.7 (4.4)	50.6 (2.4)
	K-40	187 (13)	189 (16)	182 (5)
NORM2	Ra-226	31.4 (3.8)	29.6 (3.7)	34.3 (1.5)
	Th-232	23.7 (2.0)	23.4 (2.6)	24.2 (0.8)
	K-40	327 (27)	328 (31)	325 (22)
NORM3	Ra-226	31.3 (3.2)	31.0 (3.7)	31.6 (2.3)
	Th-232	23.1 (2.6)	23.4 (3.2)	22.5 (0.9)
	K-40	310 (18)	318 (18)	297 (10)
NORM4	Ra-226	22.7 (5.0)	23.3 (6.2)	21.5 (1.4)
	Th-232	37.1 (2.5)	37.2 (2.7)	36.9 (2.2)
	K-40	58 (10)	60 (11)	55 (5)
NORM5	Ra-226	118 (21)	119 (25)	117 (13)
	Th-232	60 (13)	58 (16)	63 (7)
	K-40	1460 (216)	1518 (242)	1357 (114)
NORM6	Ra-226	49.7 (8.9)	48.9 (10.4)	51.2 (6.0)
	Th-232	56.4 (3.9)	56.6 (3.9)	56.1 (4.3)
	K-40	1221 (70)	1251 (51)	1169 (73)

[1] Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 56. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants applied true-summing corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

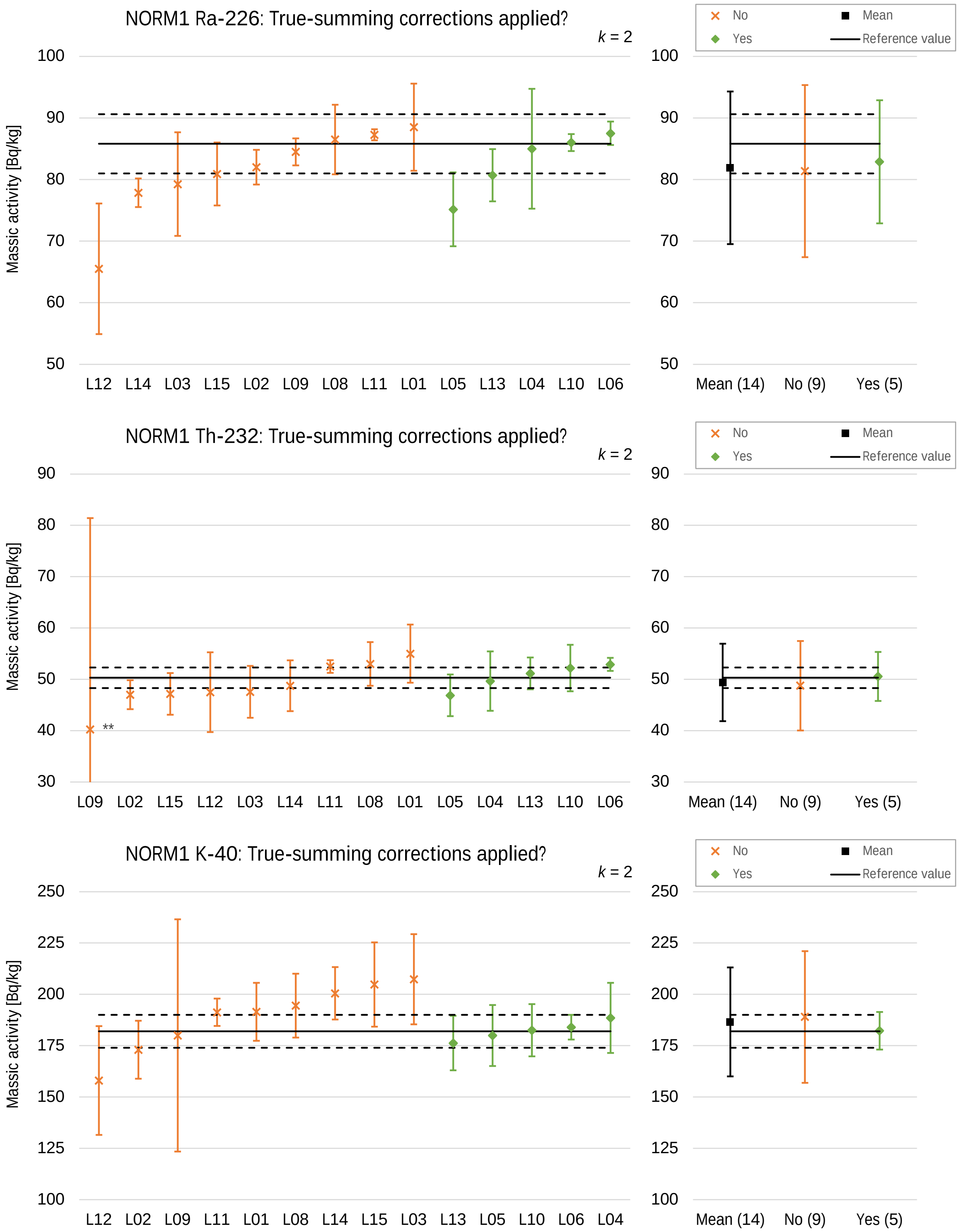


Figure 57. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants applied true-summing corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

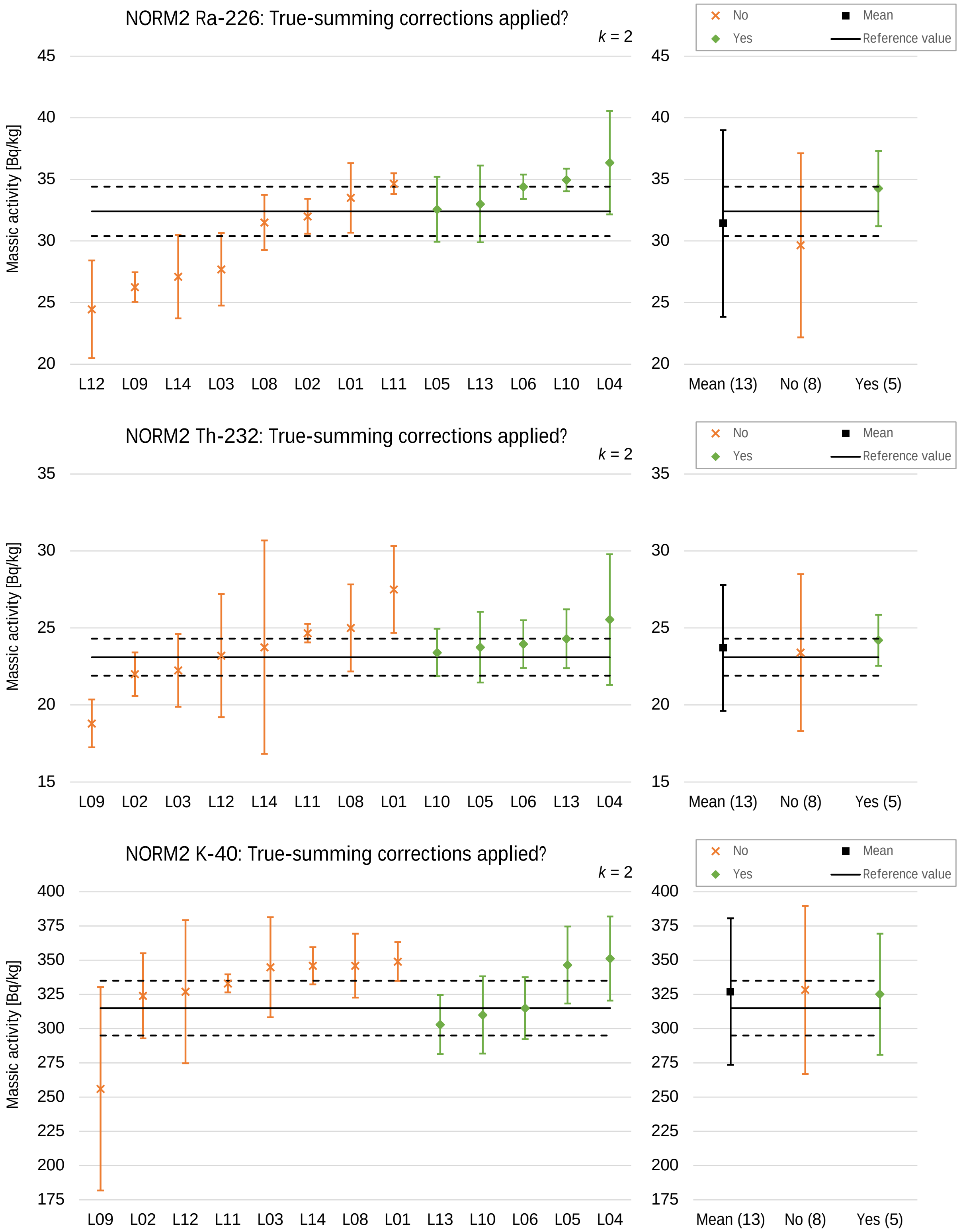


Figure 58. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants applied true-summing corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

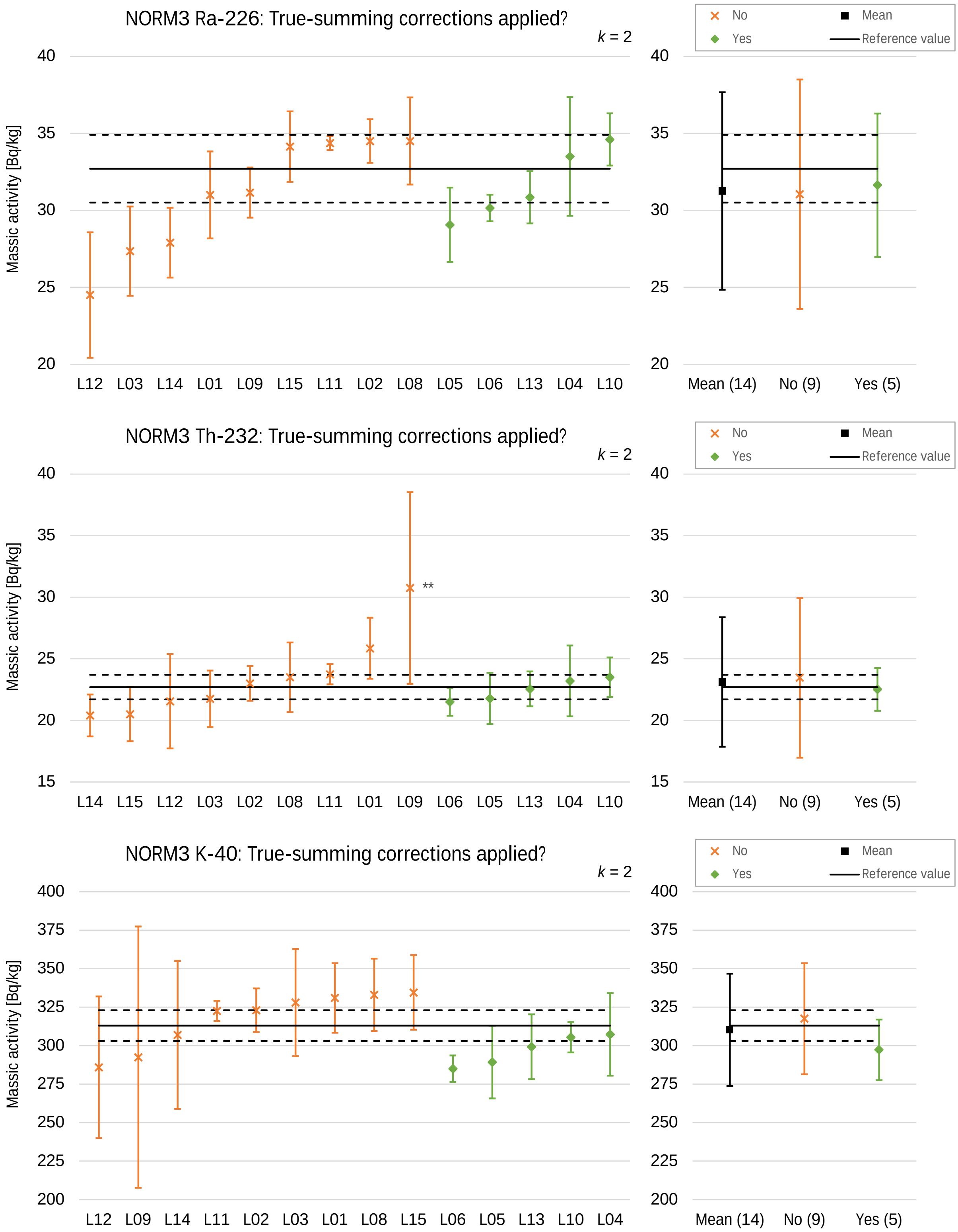


Figure 59. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants applied true-summing corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

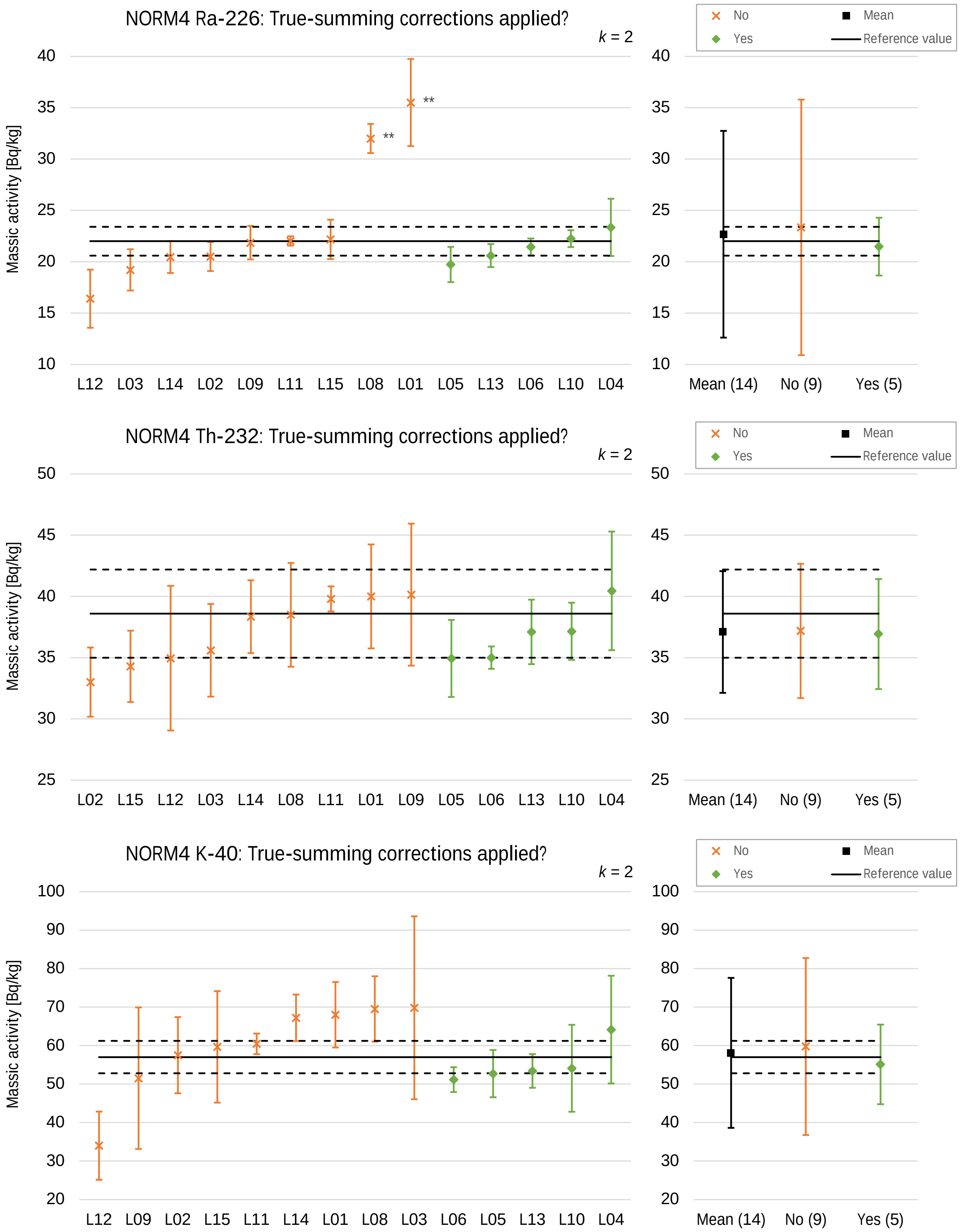


Figure 60. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants applied true-summing corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

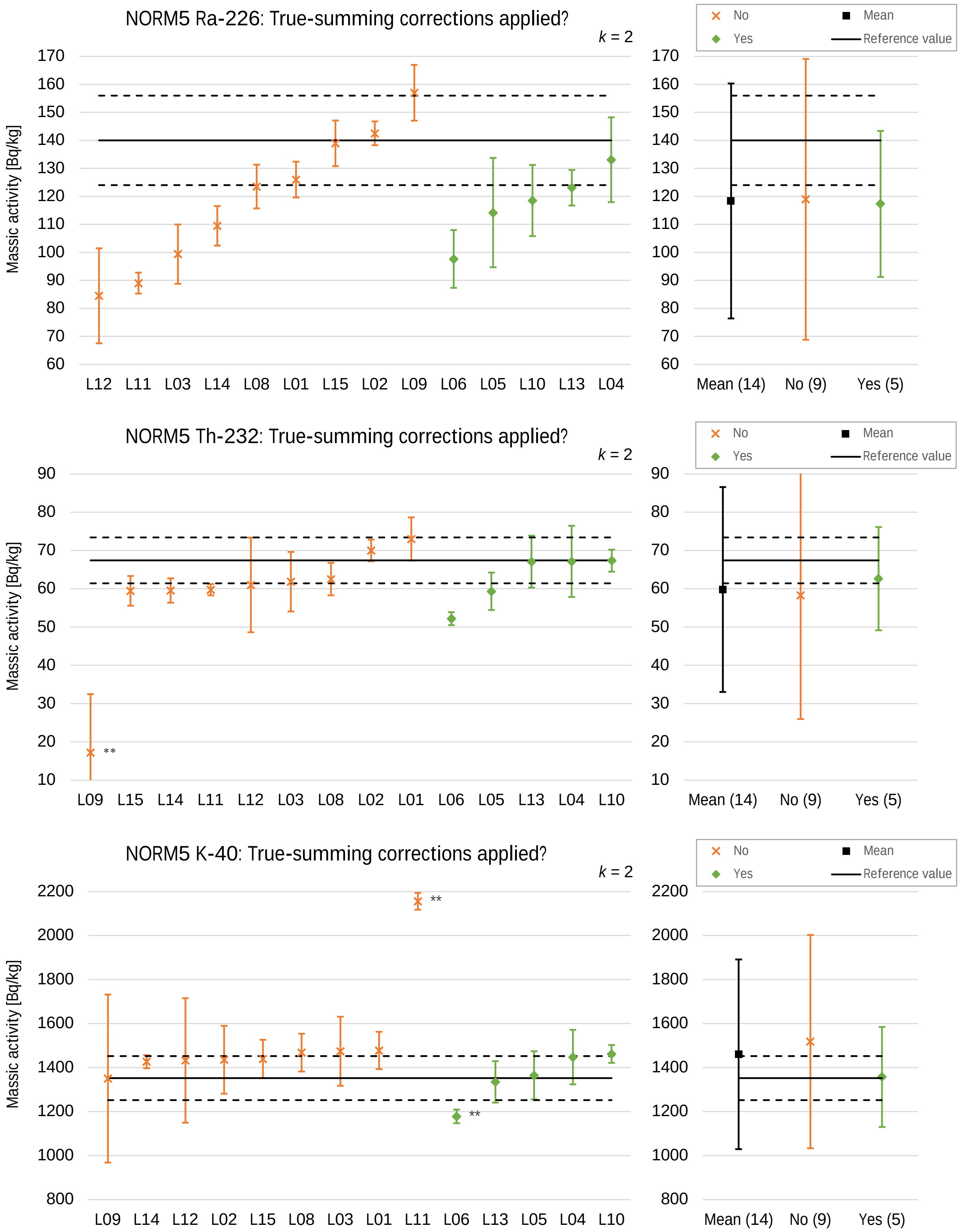


Figure 61. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants applied true-summing corrections, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

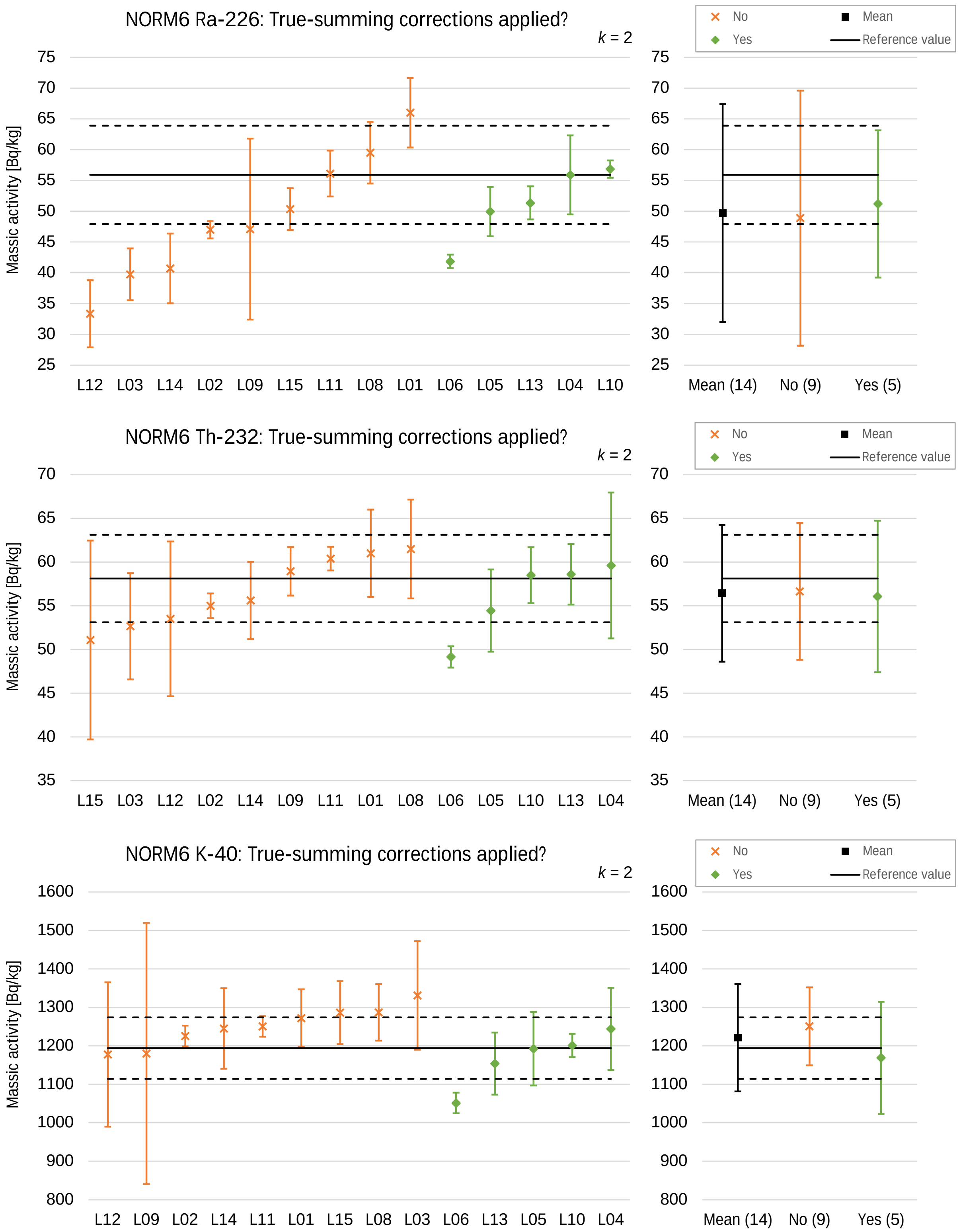


Table 36. Mean results from participants who indicated that they calculated / not calculated the activity as in Clause 8.3 of the draft standard.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Activity calculated as in Clause 8.3?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	79.1 (10.1)	83.0 (4.1)
	Th-232	49.4 (3.8)	47.9 (6.1)	50.0 (2.6)
	K-40	187 (13)	183 (18)	188 (11)
NORM2	Ra-226	31.4 (3.8)	27.8 (3.9)	33.0 (2.5)
	Th-232	23.7 (2.0)	23.3 (3.6)	23.9 (1.2)
	K-40	327 (27)	320 (43)	330 (18)
NORM3	Ra-226	31.3 (3.2)	28.6 (3.1)	32.3 (2.7)
	Th-232	23.1 (2.6)	24.6 (4.7)	22.5 (1.1)
	K-40	310 (18)	304 (20)	313 (18)
NORM4	Ra-226	22.7 (5.0)	23.6 (8.3)	22.3 (3.6)
	Th-232	37.1 (2.5)	38.4 (2.4)	36.6 (2.4)
	K-40	58 (10)	55 (16)	59 (7)
NORM5	Ra-226	118 (21)	119 (30)	118 (18)
	Th-232	60 (13)	53 (24)	63 (5)
	K-40	1460 (216)	1422 (60)	1476 (256)
NORM6	Ra-226	49.7 (8.9)	46.8 (14.0)	50.9 (6.5)
	Th-232	56.4 (3.9)	57.3 (3.4)	56.1 (4.2)
	K-40	1221 (70)	1219 (51)	1222 (79)

[1] Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 62. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants calculated the activity as in Clause 8.3 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

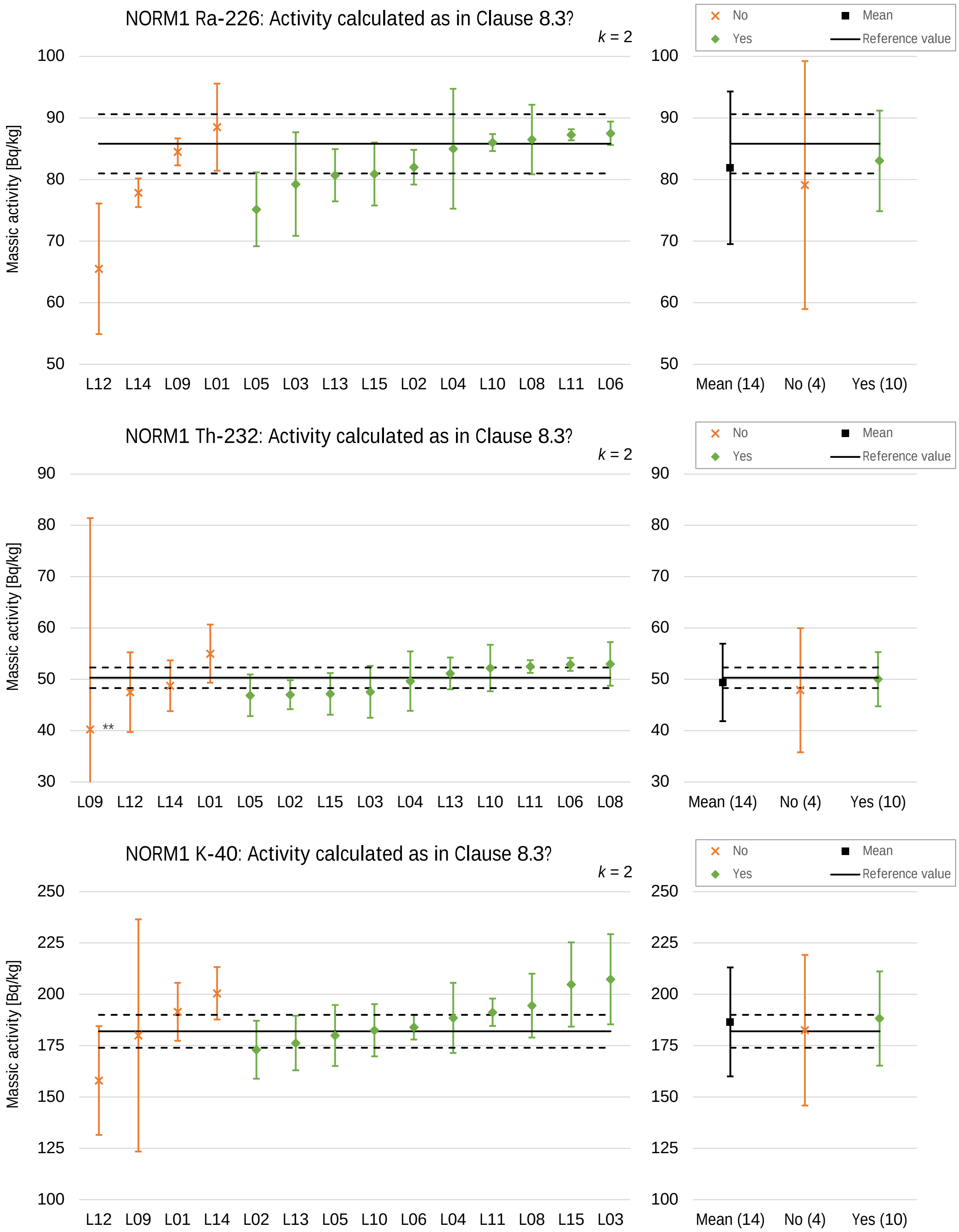


Figure 63. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants calculated the activity as in Clause 8.3 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

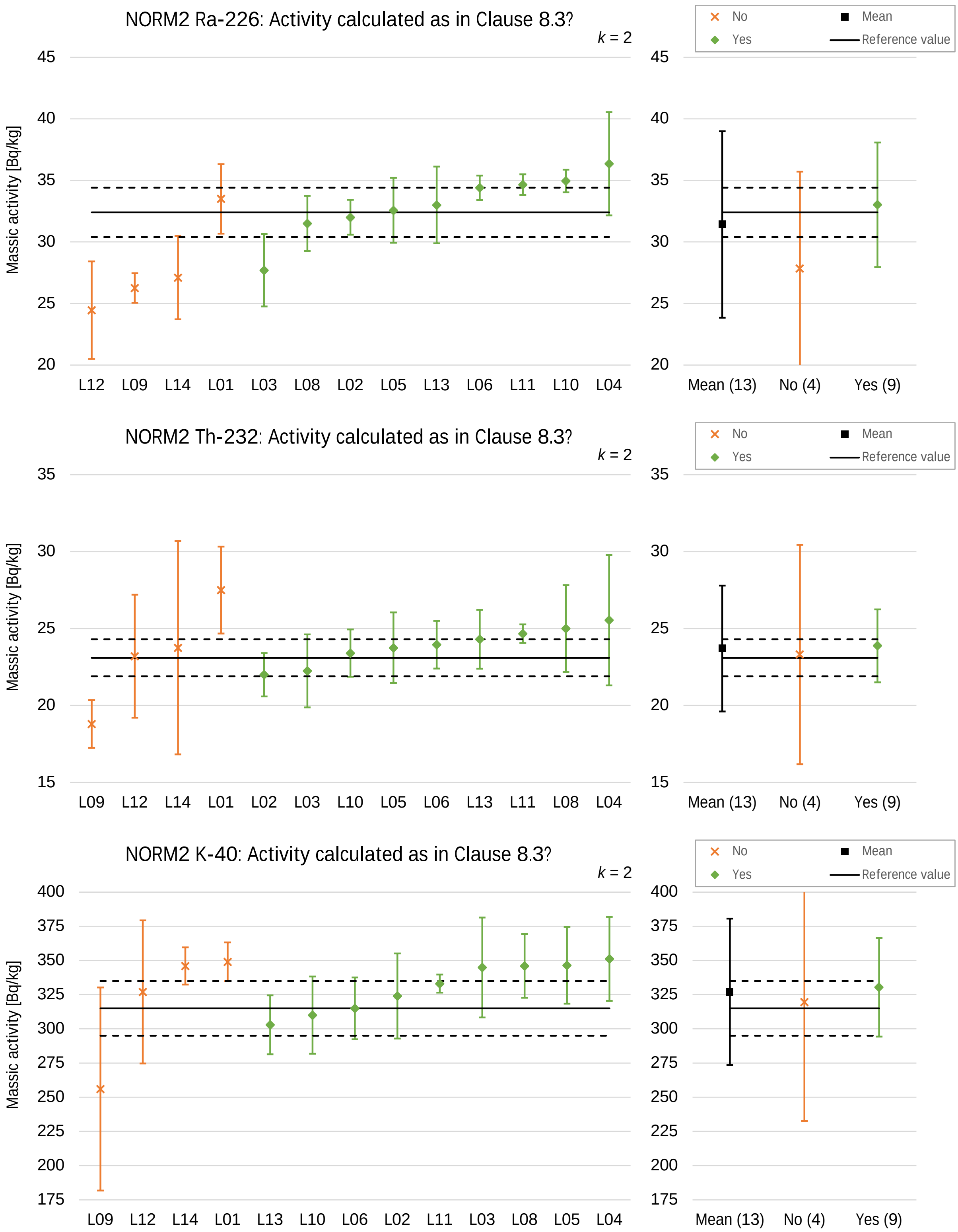


Figure 64. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants calculated the activity as in Clause 8.3 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

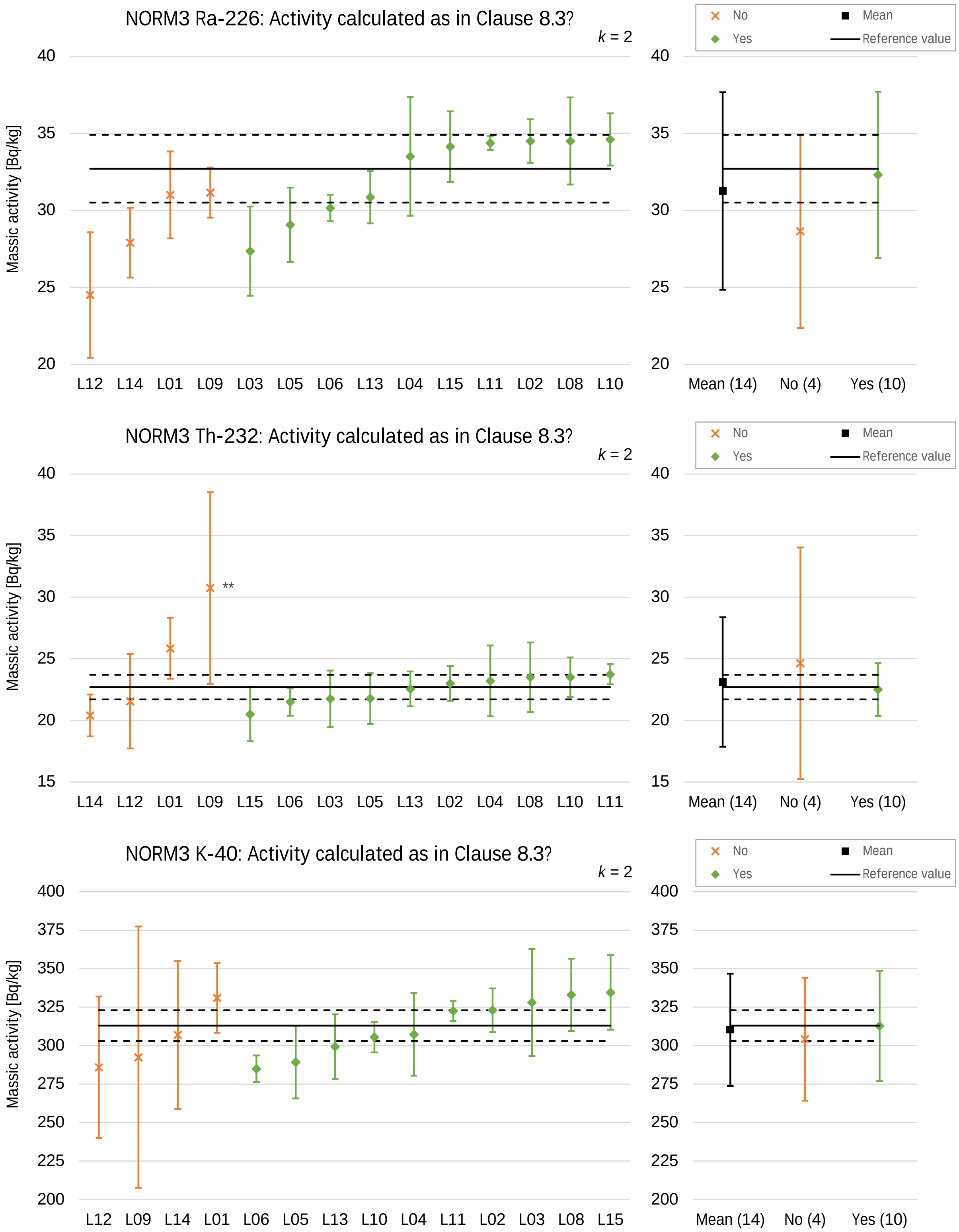


Figure 65. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants calculated the activity as in Clause 8.3 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

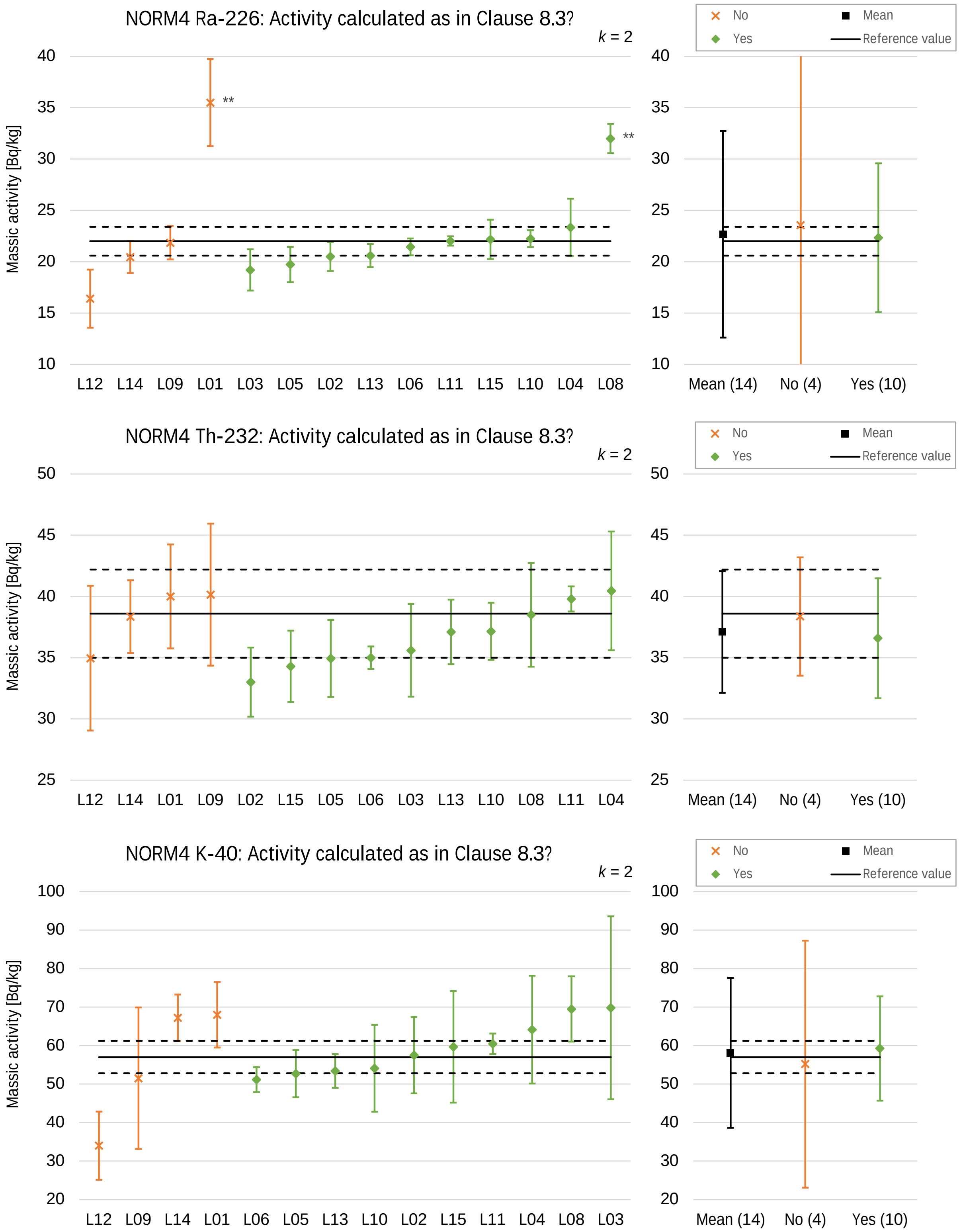


Figure 66. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants calculated the activity as in Clause 8.3 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

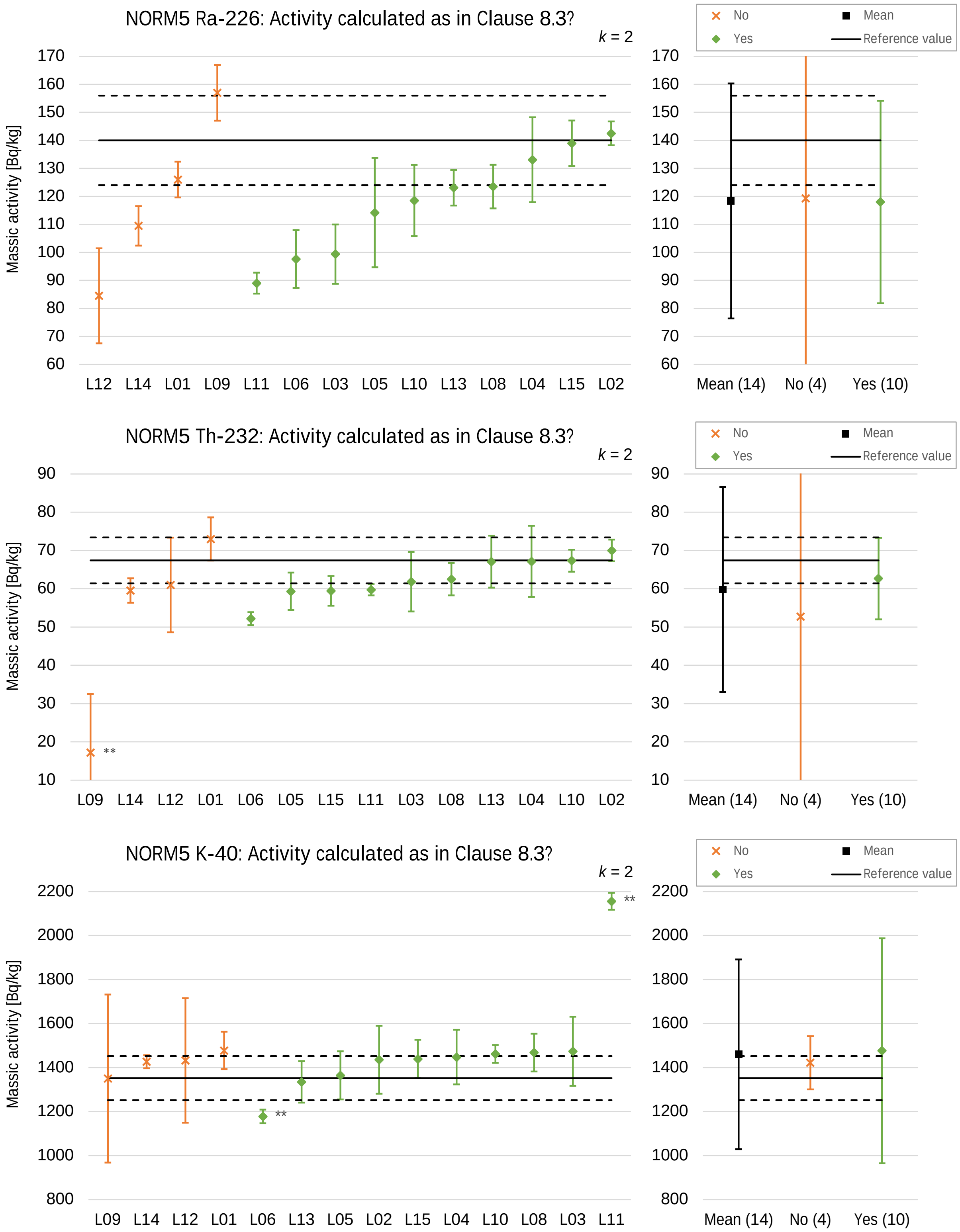
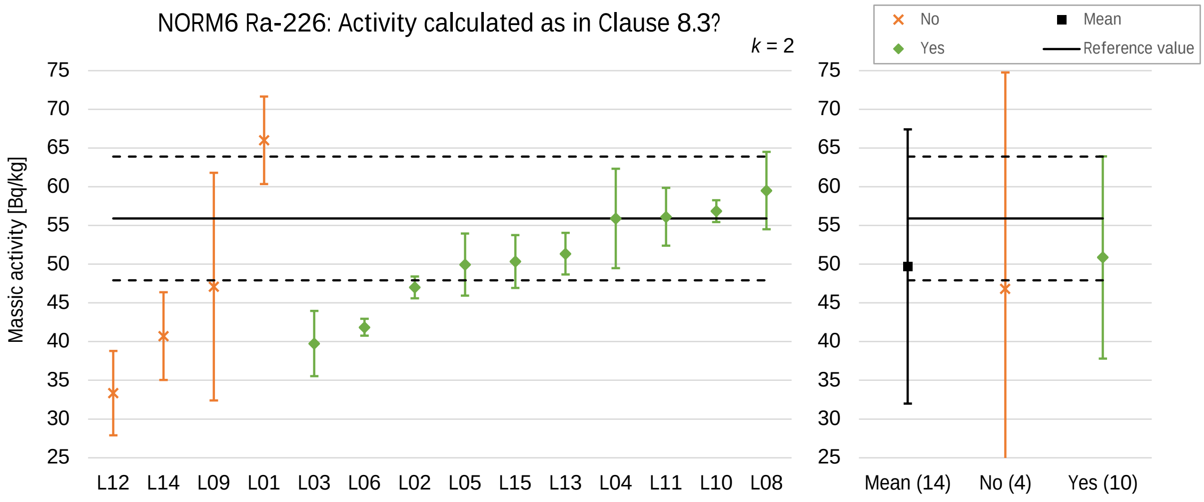
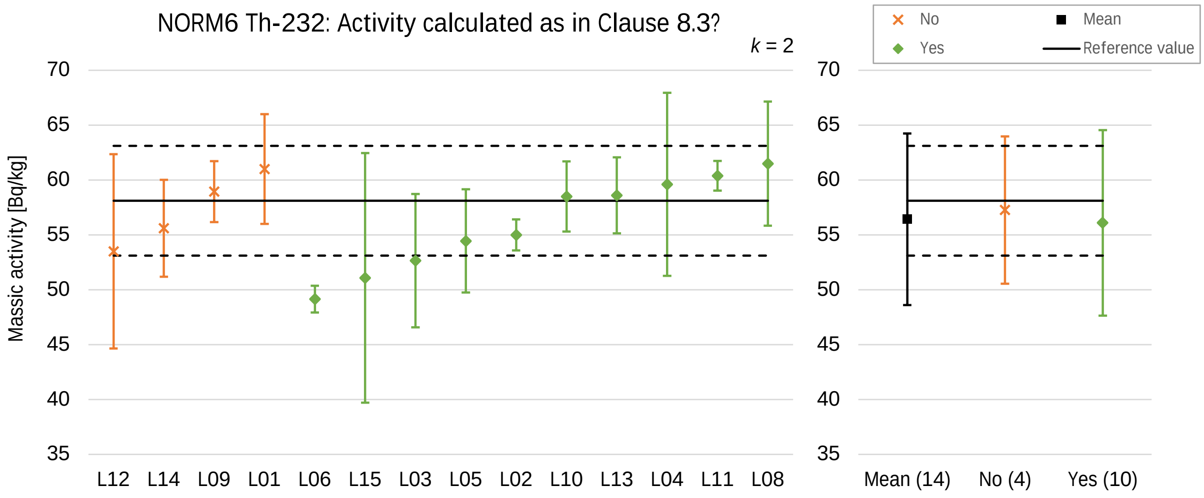
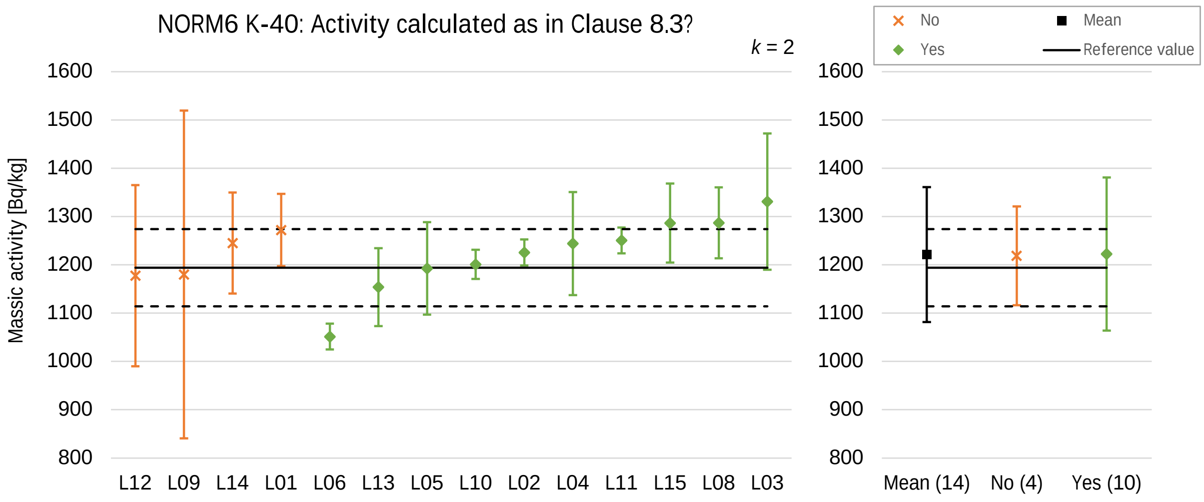


Figure 67. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants calculated the activity as in Clause 8.3 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.







6.7 Calculation of the uncertainty

Question Q 4.15 of the reporting survey asked the participants if they calculated the uncertainty following Clause 8.4 of WD EN 17216. The participants were asked to provide information on the sources of uncertainties that they took into account and their impact as a relative standard uncertainty (Q 4.16). Table 37 lists their replies. The table does not include the relative standard uncertainties since many of the labs did not provide this information and since the values very much depend on the conditions (radionuclide, geometry, counting time, etc.).

Only one of fourteen participants took the uncertainty due to radon leakage from the test specimen container into account, while this is a normative requirement specified in Annex A of WD EN 17216.

Table 37. Calculation of the uncertainty and sources of uncertainties considered. Cells are coloured green where the participants followed the normative requirements from the draft.

Participant	Q 4.15: Uncertainty calculated as in Clause 8.4?	Q 4.16: Peak area	Q 4.16: Background peak area	Q 4.16: Test specimen filling height	Q 4.16: Test specimen positioning	Q 4.16: Dry mass correction	Q 4.16: Fitting efficiency curve	Q 4.16: Corrections on the efficiency curve	Q 4.16: Calibration sources	Q 4.16: True summing	Q 4.16: Coincidences	Q 4.16: Dead time correction	Q 4.16: Radon leakage from test specimen container
L01	NO	YES	YES	NO	NO	YES	NO	NO	YES	NO	NO	YES	NO
L02	YES	YES	YES	NO	NO	NO	YES	YES	NO	NO	NO	NO	NO
L03	YES	YES	YES	NO	NO	YES	YES	NO	NO	NO	NO	NO	NO
L04	NO	YES	YES	YES	YES	NO	YES	YES	YES	YES	NO	YES	NO
L05	YES	YES	YES	NO	NO	YES	YES	YES	YES	YES	YES	YES	NO
L06	NO	YES	YES	YES	NO	YES	YES	YES	YES	NO	YES	YES	NO
L08	YES	YES	YES	NO	NO	YES	NO	NO	YES	NO	NO	YES	NO
L09	YES	YES	YES	NO	NO	YES	([1])	YES	YES	YES	YES	NO	YES
L10	YES	YES	YES	NO	NO	YES	YES	NO	YES	NO	NO	NO	NO
L11	YES	YES	YES	NO	NO	NO	YES	NO	NO	NO	NO	NO	NO
L12	NO	YES	YES	NO	NO	YES	YES	YES	YES	NO	NO	NO	NO
L13	YES	YES	YES	YES	YES	NO	YES	NO	NO	YES	NO	NO	NO
L14	NO	YES	YES	NO	NO	YES	YES	NO	YES	NO	NO	NO	NO
L15	YES	YES	YES	NO	NO	YES	NO	NO	NO	NO	NO	NO	NO

[1] The participant answered "Yes and No" and no clarification was asked.

The mean results of participants who did and who did not claim to have followed Clause 8.4 for the calculation of the uncertainty are shown in Table 38 and in Figure 68 to Figure 73. Also here, based on the observed data, no difference can be proven between the two.

no significant difference could be observed between participants who claimed to have calculated the uncertainty as in Clause 8.4 and those who claimed not

Table 38. Mean results from participants who indicated that they calculated / not calculated the uncertainty according to Clause 8.4 of the draft standard.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Uncertainty calculated according to Clause 8.4?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	80.9 (9.5)	82.5 (4.0)
	Th-232	49.4 (3.8)	50.8 (3.1)	48.6 (4.1)
	K-40	187 (13)	185 (16)	188 (12)
NORM2	Ra-226	31.4 (3.8)	31.2 (5.1)	31.6 (3.1)
	Th-232	23.7 (2.0)	24.8 (1.7)	23.0 (2.0)
	K-40	327 (27)	338 (16)	320 (31)
NORM3	Ra-226	31.3 (3.2)	29.4 (3.4)	32.3 (2.8)
	Th-232	23.1 (2.6)	22.5 (2.1)	23.5 (2.9)
	K-40	310 (18)	303 (19)	314 (18)
NORM4	Ra-226	22.7 (5.0)	23.4 (7.2)	22.3 (3.8)
	Th-232	37.1 (2.5)	37.8 (2.7)	36.7 (2.5)
	K-40	58 (10)	57 (14)	59 (7)
NORM5	Ra-226	118 (21)	110 (20)	123 (21)
	Th-232	60 (13)	63 (8)	58 (16)
	K-40	1460 (216)	1392 (121)	1498 (252)
NORM6	Ra-226	49.7 (8.9)	47.6 (13.1)	50.9 (6.0)
	Th-232	56.4 (3.9)	55.8 (4.8)	56.8 (3.6)
	K-40	1221 (70)	1198 (89)	1234 (59)

[1] Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 68. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants calculated the uncertainty according to Clause 8.4 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

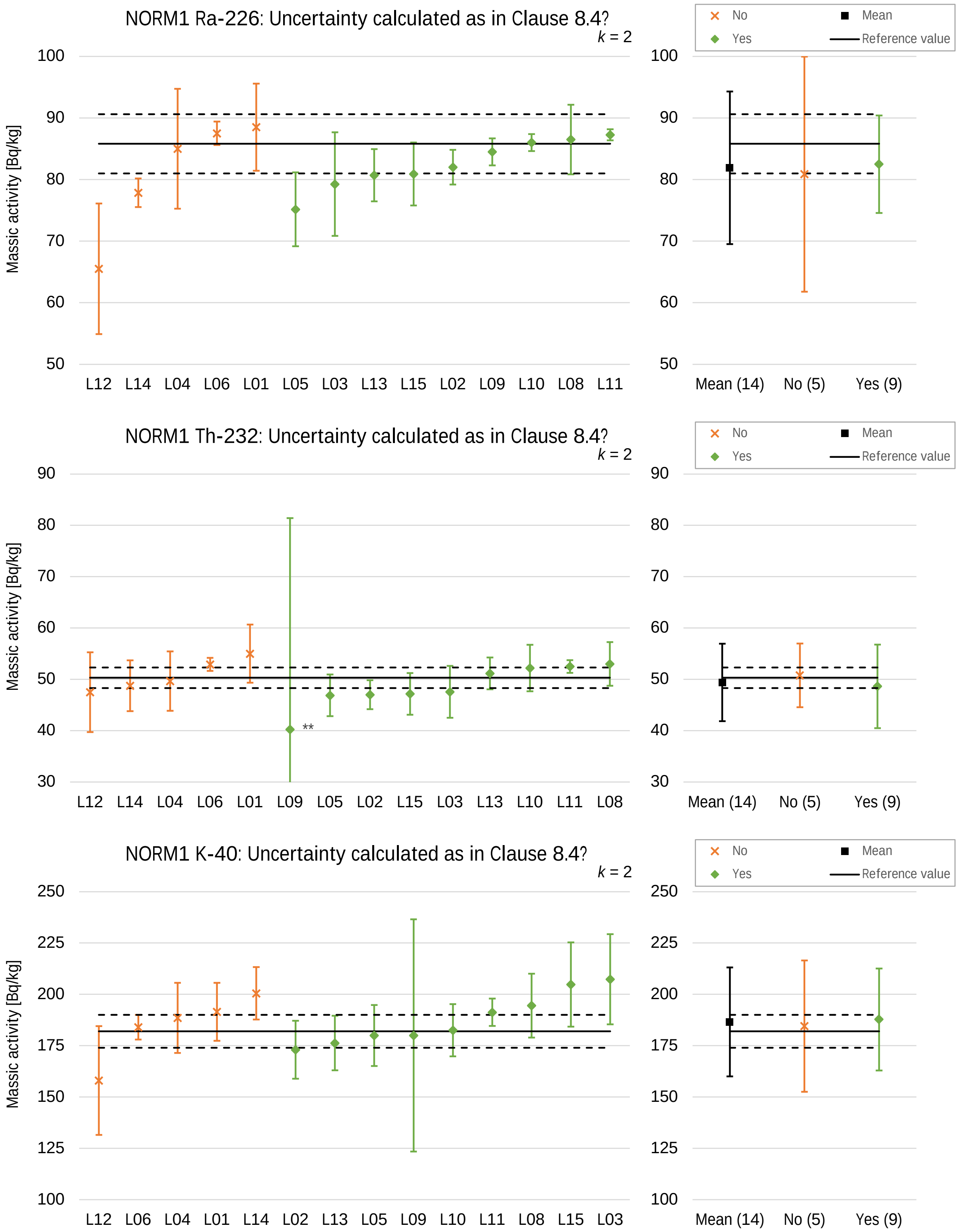


Figure 69. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants calculated the uncertainty according to Clause 8.4 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

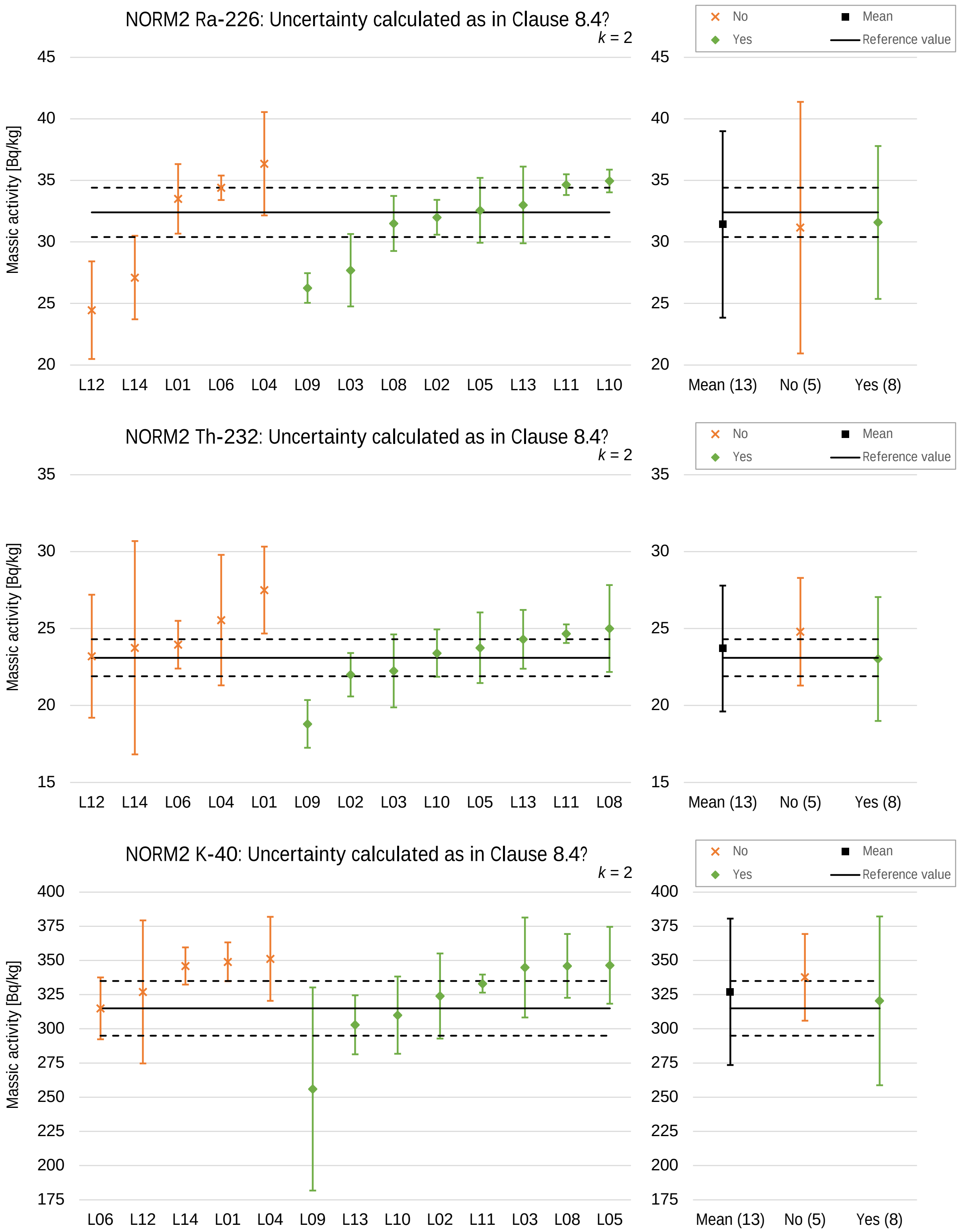


Figure 70. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants calculated the uncertainty according to Clause 8.4 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

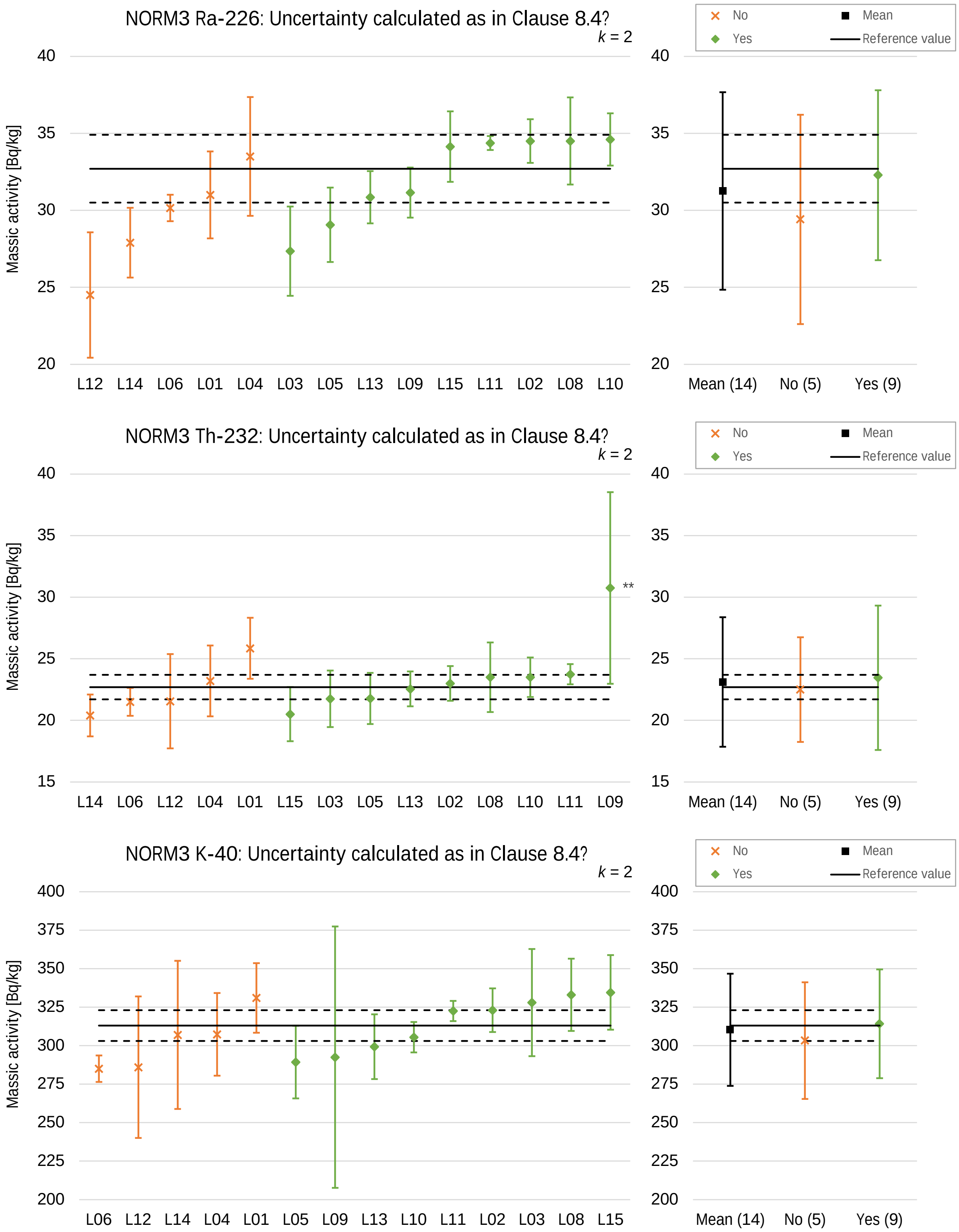


Figure 71. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants calculated the uncertainty according to Clause 8.4 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

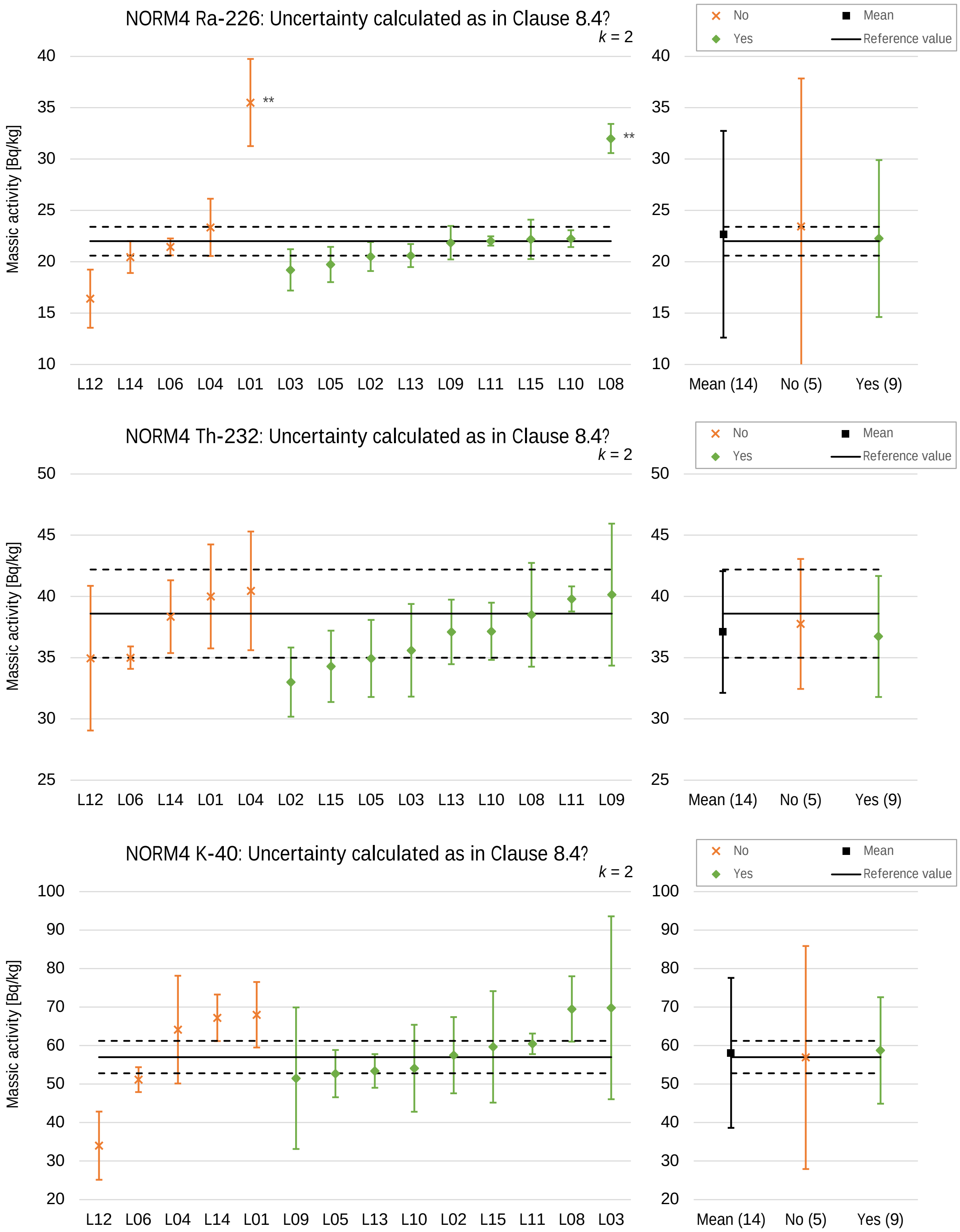


Figure 72. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants calculated the uncertainty according to Clause 8.4 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

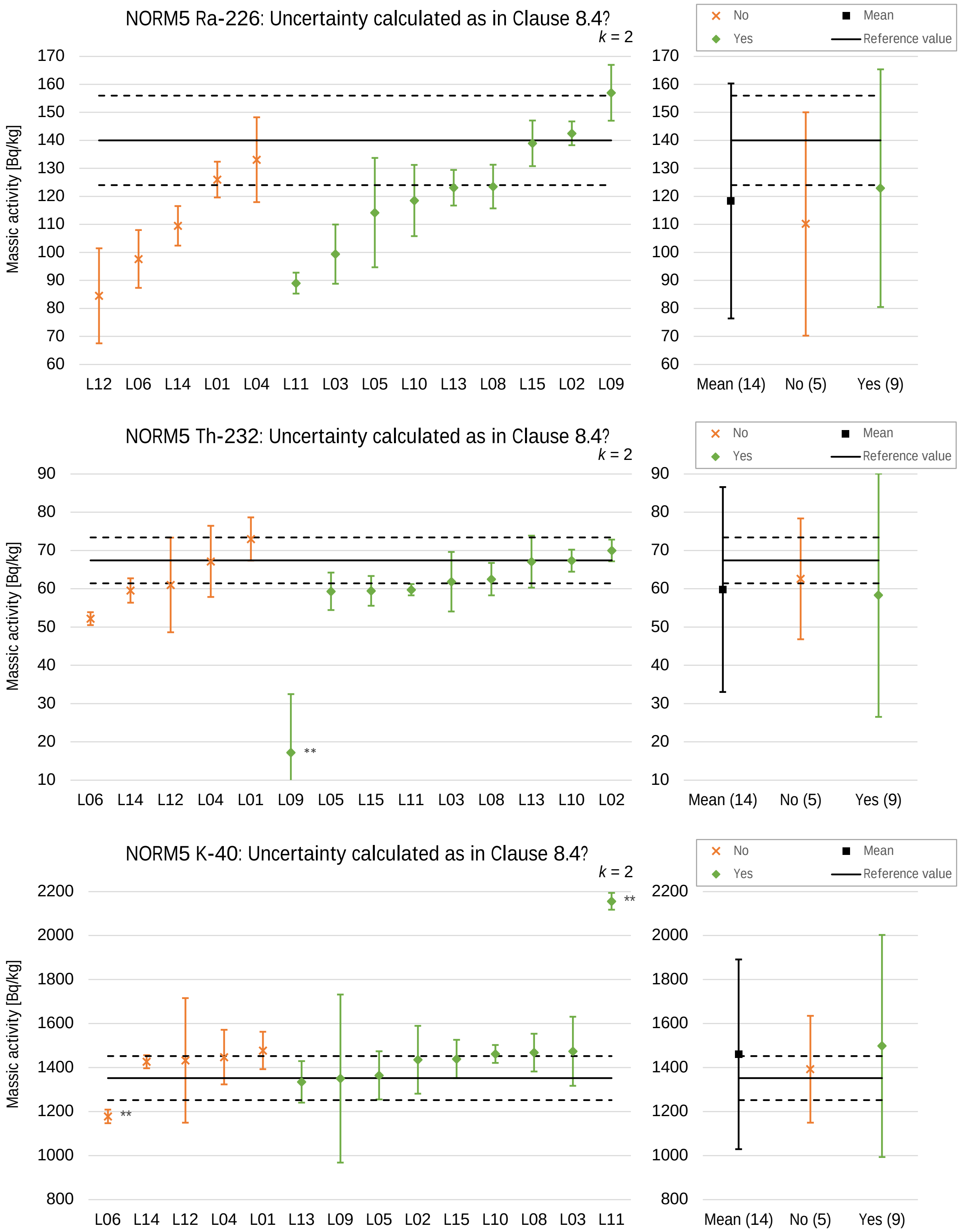
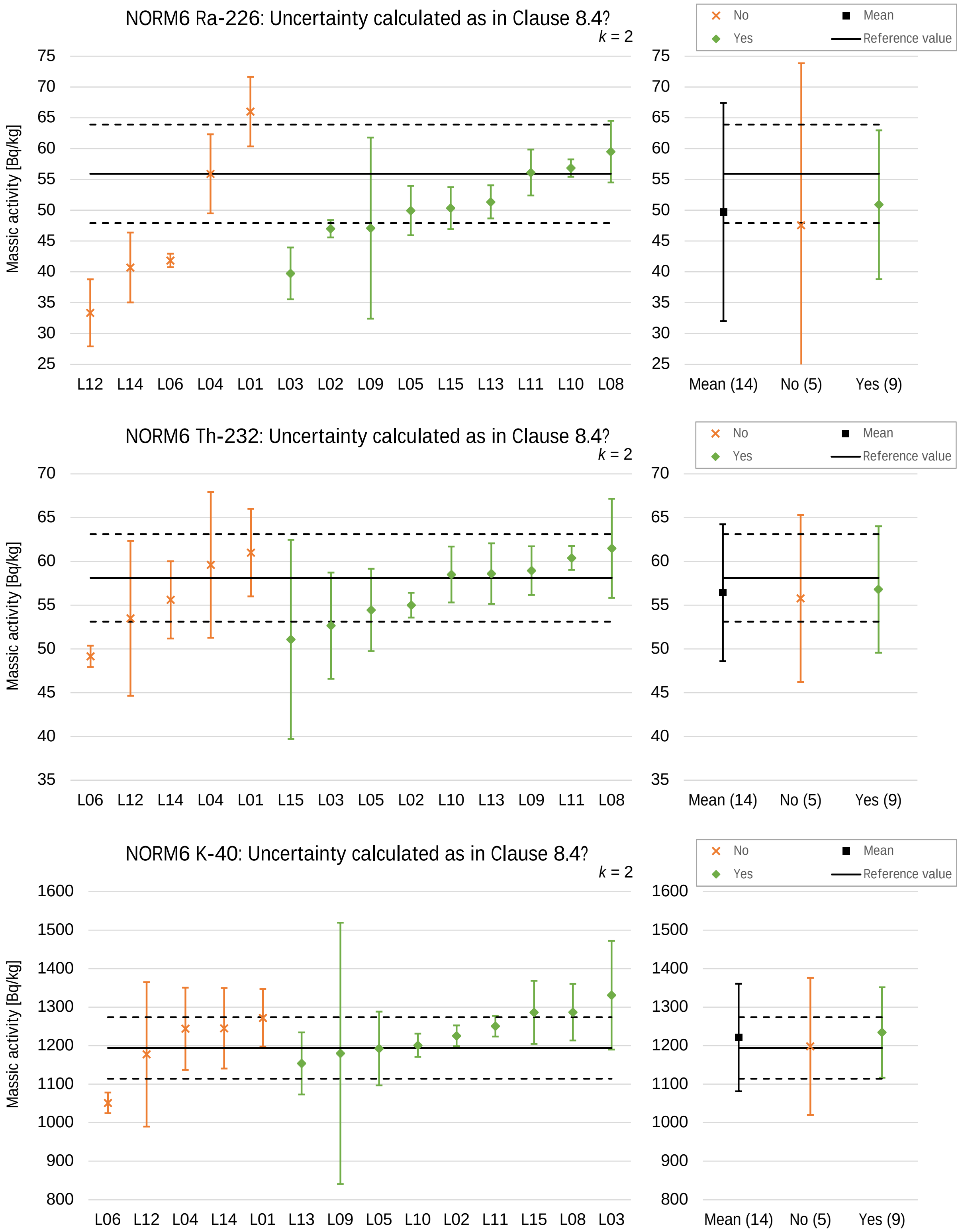


Figure 73. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants calculated the uncertainty according to Clause 8.4 of the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.



6.8 Strict application of WD EN 17216

Question 5.1 in the reporting survey polled the participants if they could you strictly follow WD EN 17216 and that it is unambiguous and clear. If they answered no to Q 5.1, the participants were asked to describe where it could not be followed or where it is not clear (Q 5.1.1). Additional comments or suggestions to improve the draft could be provided by filling in the commenting form (Q 5.2).

Table 39 presents the answers from the participants to question Q 5.1. The answers to Q 5.1.1 and Q 5.2 are included in Section 8 where the comments on the standard are provided.

Nine out of fourteen participants claim that they applied the method strictly. However, as discussed in Sections 6.2 to 6.7, ten questions from the reporting questionnaire enquired specific information on the application of ten normative requirements in the standard. As a rough quantitative indication and ignoring differences of their impact, Table 39 includes the count of how many of the ten requirements the participants claimed to have followed. As can be seen, only participant L05 claimed to have adhered to the ten requirements. Note that there are more than ten normative requirements in the standard, but not all were probed.

Table 39. Replies from the participants on the question if they could strictly apply the draft standard and if they have comments on it.

Participant	Q 5.1: Strict application of WD EN 17216?	Count /10 (1)
L01	YES	5
L02	YES	5
L03	NO	6
L04	YES	8
L05	YES	10
L06	NO	3
L08	YES	7
L09	YES	7
L10	YES	9
L11	YES	8
L12	NO	4
L13	YES	7
L14	NO	6
L15	NO	6

1 The count represents how many of the ten normative requirements probed in the reporting questionnaire were adhered to, as claimed by the participants.

The data shown in Table 40 and Figure 74 to Figure 79 allow a comparison between the mean results of participants who claimed to have strictly followed the draft standard and those who did not. Also in this case, a difference between the two groups could not be proven.

no significant difference could be observed between the result of the participants who claimed to have followed the draft method strictly and those who did not

For each test property, a hypothesis test was performed to test if the null hypothesis can be rejected (H_0: There is no correlation between the relative bias and the count of how many of the ten requirements a participant claimed to have followed). For all six materials and three radionuclides, the relationship is not statistically significant at a significance level of $\alpha = 0.05$. The null hypothesis cannot be rejected and the alternative hypothesis (H_1: There is a correlation) is insufficiently supported.

no correlation can statistically be observed between the count of how many of (a selection of) ten requirements a participant followed and the relative bias

Table 40. Mean results from participants who indicated if they strictly applied WD EN 17216.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	WD EN 17216 strictly followed?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	78.2 (8.0)	84.0 (4.1)
	Th-232	49.4 (3.8)	48.8 (2.4)	49.7 (4.5)
	K-40	187 (13)	191 (21)	184 (8)
NORM2	Ra-226	31.4 (3.8)	28.4 (4.2)	32.8 (2.9)
	Th-232	23.7 (2.0)	23.3 (1.1)	23.9 (2.4)
	K-40	327 (27)	333 (15)	324 (31)
NORM3	Ra-226	31.3 (3.2)	28.8 (3.6)	32.6 (2.1)
	Th-232	23.1 (2.6)	21.1 (0.6)	24.2 (2.7)
	K-40	310 (18)	308 (23)	311 (16)
NORM4	Ra-226	22.7 (5.0)	19.9 (2.3)	24.2 (5.6)
	Th-232	37.1 (2.5)	35.6 (1.6)	37.9 (2.6)
	K-40	58 (10)	56 (14)	59 (7)
NORM5	Ra-226	118 (21)	106 (20)	125 (19)
	Th-232	60 (13)	59 (4)	60 (17)
	K-40	1460 (216)	1390 (120)	1499 (252)
NORM6	Ra-226	49.7 (8.9)	41.2 (6.1)	54.4 (6.2)
	Th-232	56.4 (3.9)	52.4 (2.4)	58.7 (2.5)
	K-40	1221 (70)	1218 (109)	1223 (44)

[1] Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Figure 74. Results reported by the participants for NORM1. At the left, results are grouped whether or not the participants claimed to have strictly followed the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

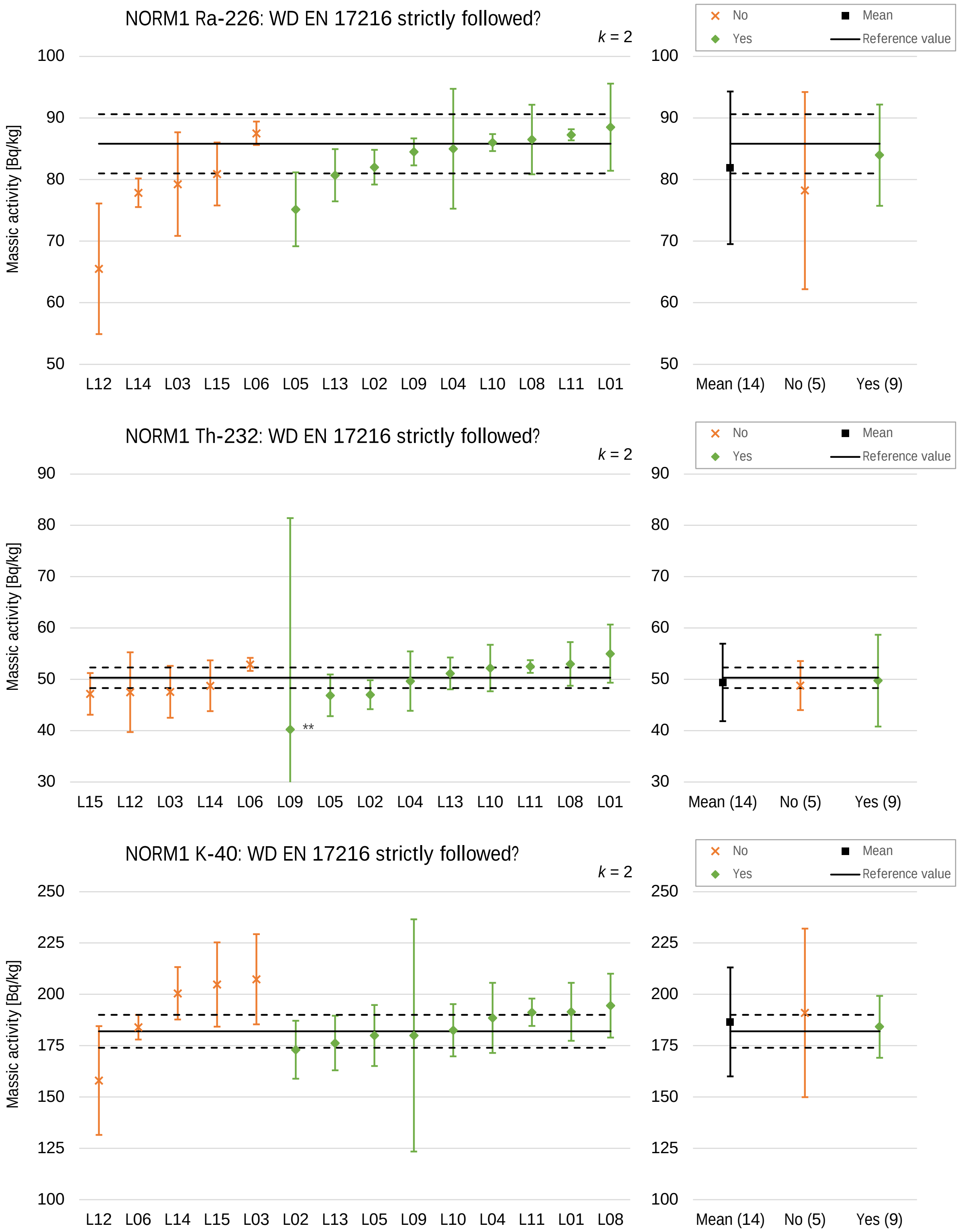


Figure 75. Results reported by the participants for NORM2. At the left, results are grouped whether or not the participants claimed to have strictly followed the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.

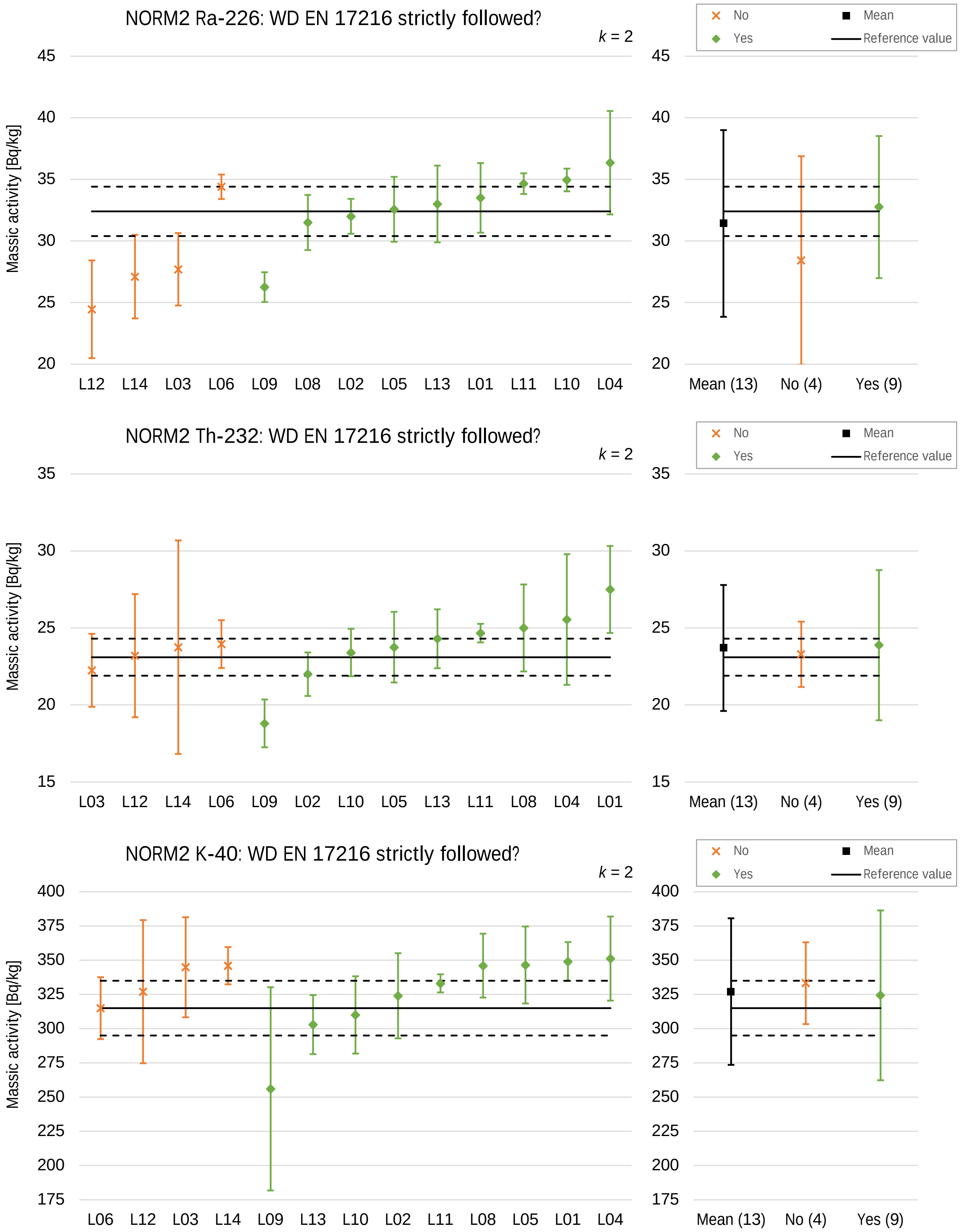


Figure 76. Results reported by the participants for NORM3. At the left, results are grouped whether or not the participants claimed to have strictly followed the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

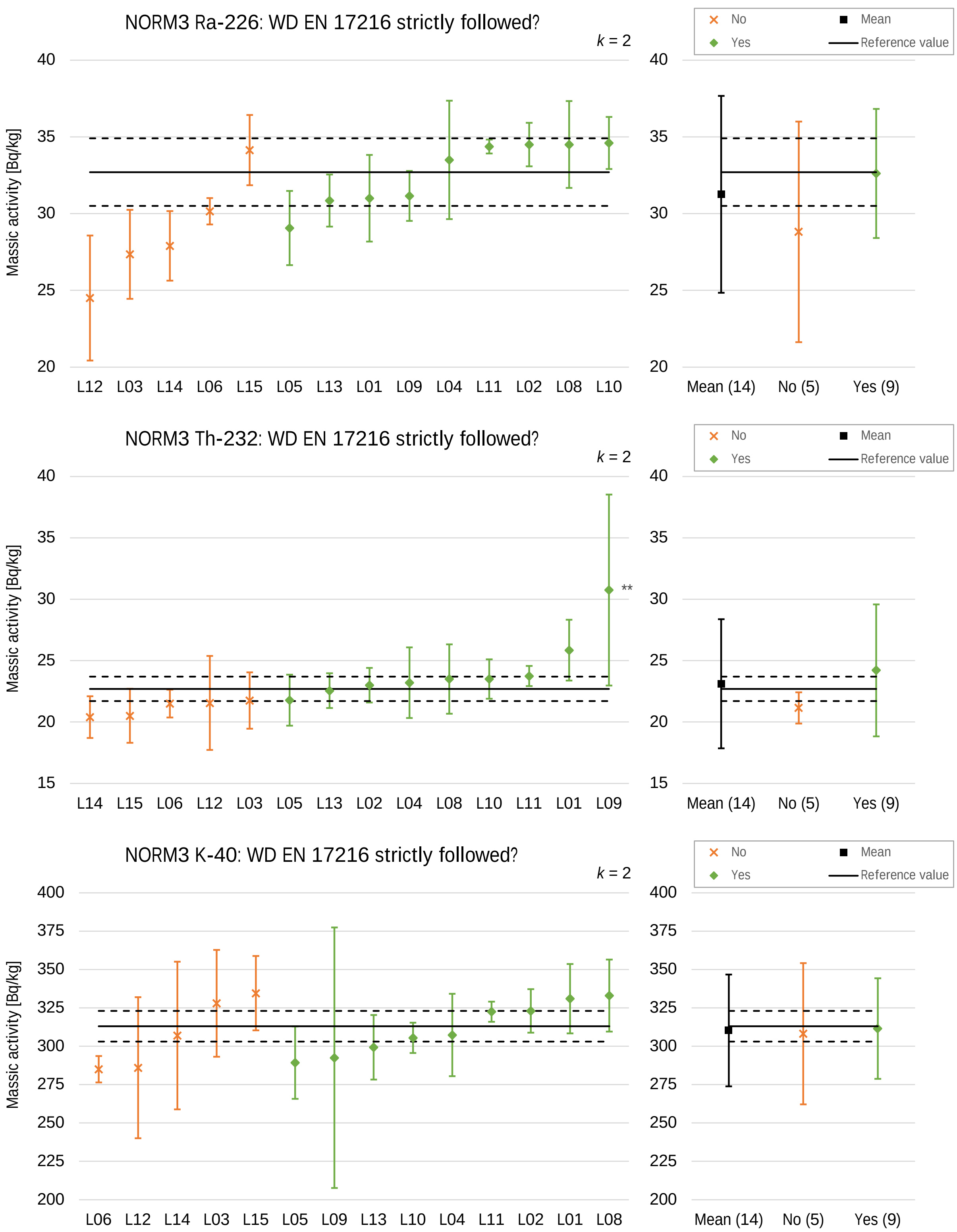


Figure 77. Results reported by the participants for NORM4. At the left, results are grouped whether or not the participants claimed to have strictly followed the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

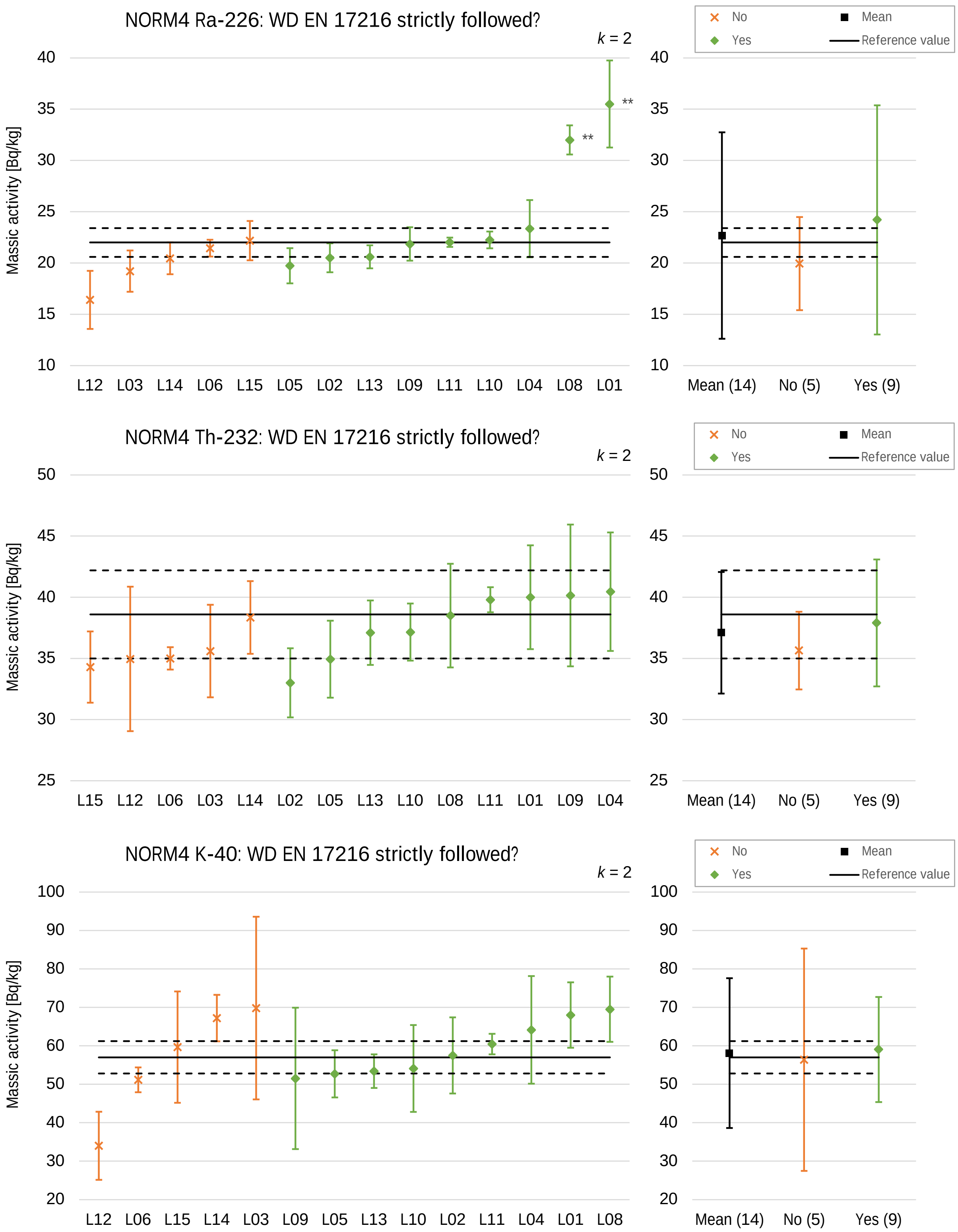


Figure 78. Results reported by the participants for NORM5. At the left, results are grouped whether or not the participants claimed to have strictly followed the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses. Outliers are identified with a double asterisk but included in the calculation of the mean.

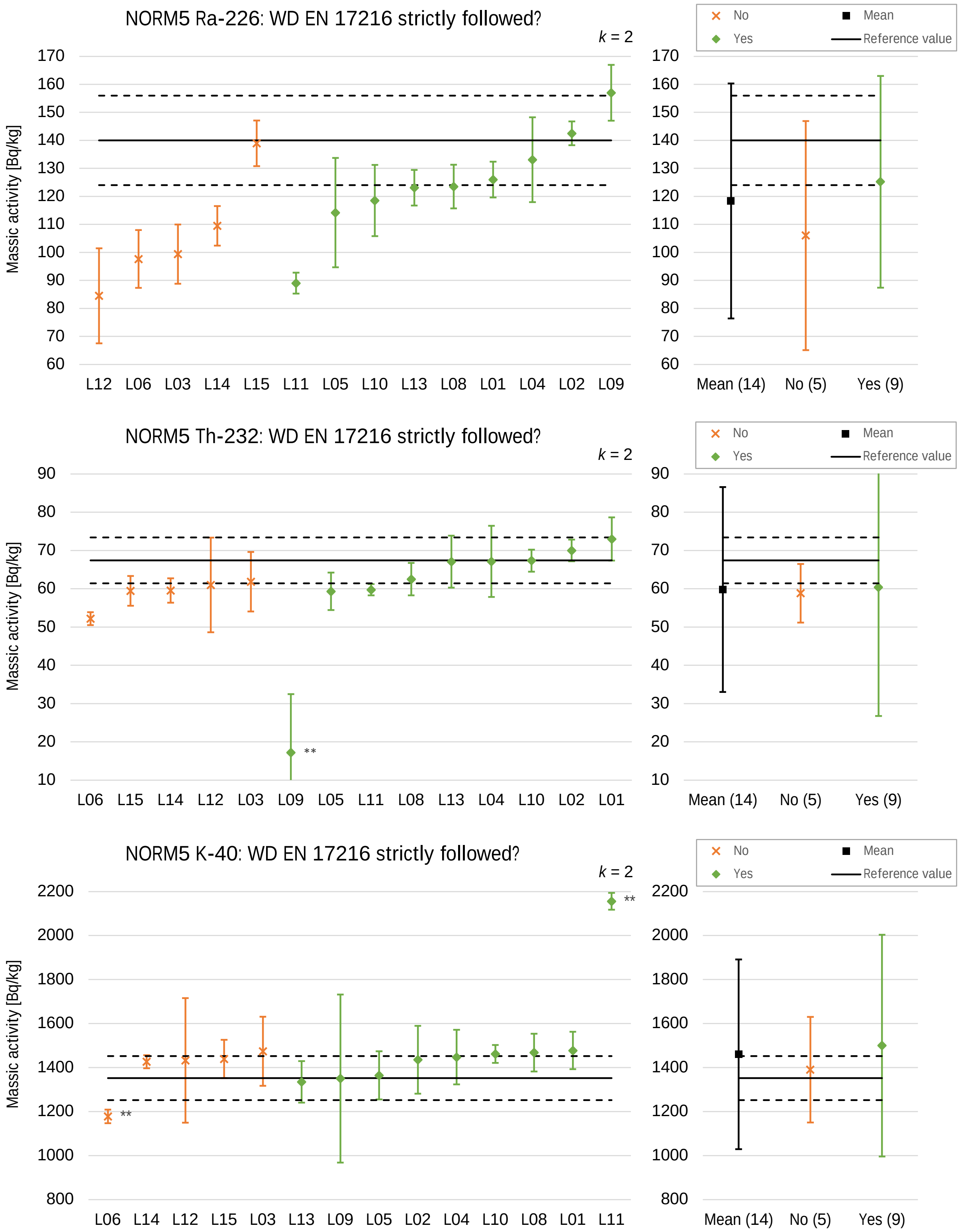
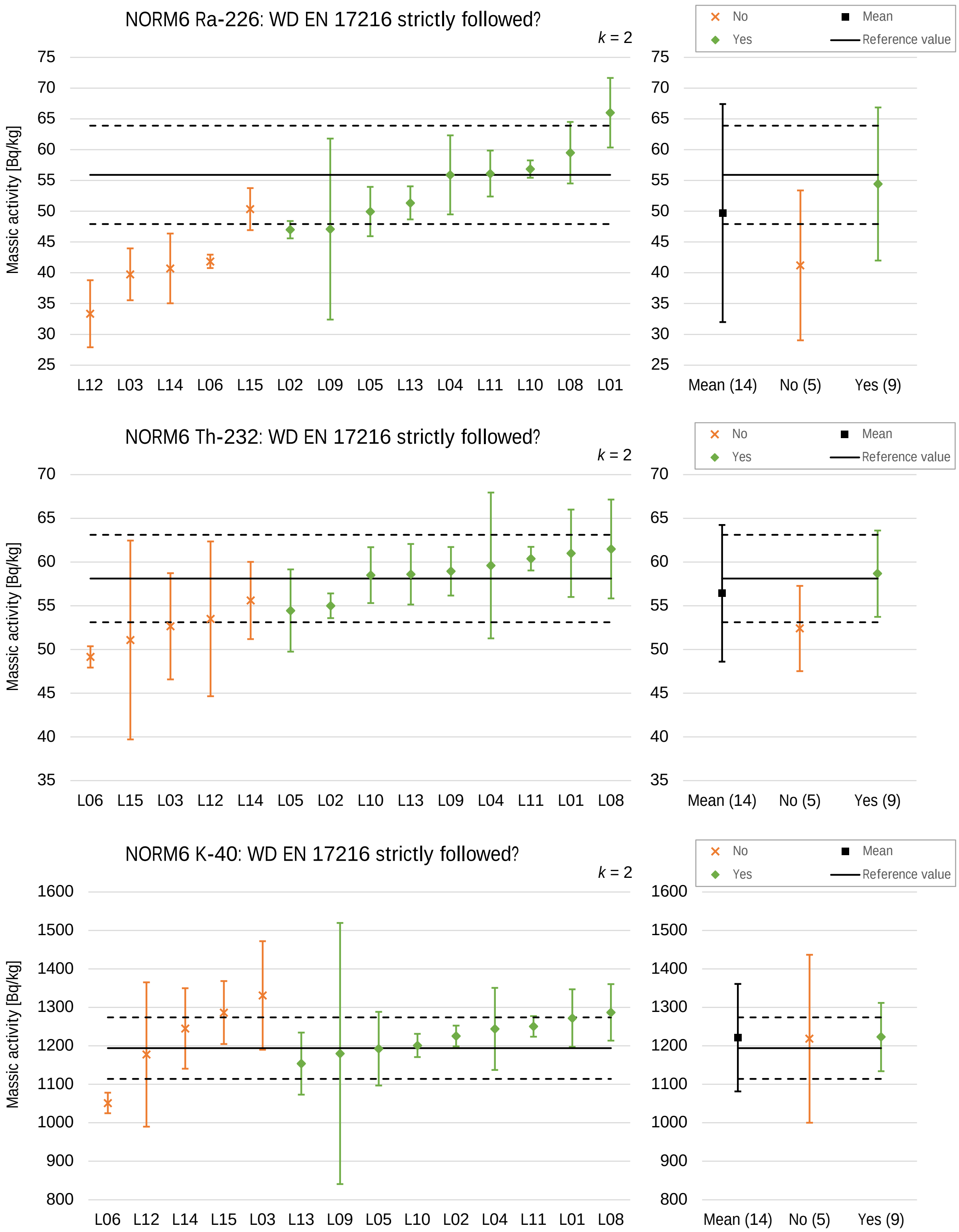


Figure 79. Results reported by the participants for NORM6. At the left, results are grouped whether or not the participants claimed to have strictly followed the draft standard, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. The number of participants is shown in parentheses.



6.9 Radon leakage rate of test specimen container

The determination of the radon leakage rate from the test specimen container and the determination of the relative uncertainty due to radon leakage is a normative requirement specified in Annex A of WD EN 17216.

However, only one of fourteen participants took the uncertainty due to radon leakage into account (See Section 6.7). From Section 6.8 it can be seen that participant L08 explicitly writes not to have followed Annex A. Participant L14 did not follow the Annex A since the participant claims to have an adequate sealing method. Participant L03 mentions Annex A in the comment on issues found in application of the method. Also, four participants (L01, L02, L06 and L08) claimed not to have applied sealing methods in addition to closing the lid of the test specimen container (See Section 6.3). Together, seven out of fourteen participants did not apply the requirements on sealing and determining radon leakage rate.

seven out of fourteen participants did not apply the requirements on sealing and determining radon leakage rate

6.10 Conclusions

In the subsections above, ten normative requirements from the standard were assessed by comparing the mean results provided by the laboratories who followed the requirement with those who did not. In the assessment, outliers earlier identified by Cochran's and Grubbs' test in Section 4 where included as well as the results from participant L09. Results from participant L07 were excluded since the participant did not use a semiconductor gamma-ray spectrometer. For none of the following normative requirements, a statistically significant difference between following and not following the requirement could be demonstrated:

— Crushing and sieving the material to a particle size less than 2 mm;

— Homogenising the material;

— Completely filling the test specimen containers;

— Vibrating or compacting the test specimen containers;

— Making the test specimen container radon tight;

— Determining the dry matter correction factor;

— Applying attenuation corrections;

— Applying true-summing corrections;

— Calculating the activity according to Clause 8.3 of the draft standard;

— Calculating the uncertainty according to Clause 8.4 of the draft standard.

Furthermore, based on the data from the collaborative assessment:

— The impact of shaking the test specimen could not be proven;

— No significant differences were observed between the three different methods of determining the detection efficiency;

— No significant differences were observed between participants who claimed to have applied WD EN 17216 strictly and those who claimed not;

— No correlation can statistically be observed between the relative laboratory bias and the count of how many of the ten selected requirements a participant followed.

For some of the test properties (materials and radionuclides), not following one or more normative requirements may have led to outliers identified by Cochran's and Grubbs' test in Section 4. However, the available data does not allow to make such a statement with a significant level of confidence.

7 Impact of other aspects

7.1 Introduction

Apart from enquiring how strictly the participants followed normative requirements in the draft standard to validate, both surveys included general questions on the level of experience of the participants and their routine activities. The impact of these aspects was analysed in the same way as the normative requirements discussed in Section 6. Also here, the data includes the outliers identified by Cochran's and Grubbs' test in Section 4 and the results provided by participant L09. This section presents the outcome of the analysis. Charts are only provided for the cases where there is an observable impact.

the data in this section includes participant L09 and the outliers identified earlier

7.2 Participants' routine activities

In the reporting questionnaire, ten participants mention that they are active in research and development. Eleven participants mention that they perform measurements for the monitoring of radioactivity in the environment (See Table 41). One participant mentions the monitoring of mineral-based construction materials. One participant mentions radon measurements and radiological evaluations in NORM industries. One participant mentions food monitoring. One participant mentions commercial measurements.

Table 41. Replies from the participants on general questions related to their routine activities.

Participant	Q 1.1: Performs R&D?	Q **1.1:** Monitors radioactivity in the environment?
L01	YES	NO
L02	YES	YES
L03	NO	YES
L04	YES	YES
L05	YES	YES
L06	YES	NO
L08	NO	NO
L09	YES	YES
L10	YES	YES
L11	YES	YES
L12	NO	YES
L13	YES	YES
L14	YES	YES
L15	NO	YES

Table 44 contains the means of the participants who yes/no perform R&D activities. For 13 of the 18 test properties, the mean over the participants who perform R&D shows a smaller bias than the mean over the other participants. For 11 of the 18 test properties, the spread between the participants who perform R&D is smaller than the spread between the other participants.

participants who perform R&D activities have a smaller bias and appear to have a smaller spread between their values than those who do not perform R&D

Table 45 provides the means over the participants who yes/no monitor radioactivity in the environment. A pattern demonstrating the impact on the bias and the spread between the participants is not apparent.

a difference between bias and spread of participants who yes/no perform environmental monitoring cannot be proven based on the available data

Table 42. Mean results from participants who indicated if they perform research and development.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Perform R&D?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	78.0 (8.9)	83.4 (4.4)
	Th-232	49.4 (3.8)	48.8 (2.8)	49.6 (4.2)
	K-40	187 (13)	191 (23)	185 (8)
NORM2	Ra-226	31.4 (3.8)	27.9 (3.5)	32.5 (3.3)
	Th-232	23.7 (2.0)	23.5 (1.4)	23.8 (2.3)
	K-40	327 (27)	339 (11)	323 (29)
NORM3	Ra-226	31.3 (3.2)	30.1 (5.0)	31.7 (2.4)
	Th-232	23.1 (2.6)	21.8 (1.2)	23.6 (2.9)
	K-40	310 (18)	320 (23)	306 (15)
NORM4	Ra-226	22.7 (5.0)	22.4 (6.8)	22.8 (4.6)
	Th-232	37.1 (2.5)	35.8 (1.9)	37.6 (2.6)
	K-40	58 (10)	58 (17)	58 (7)
NORM5	Ra-226	118 (21)	112 (24)	121 (20)
	Th-232	60 (13)	61 (2)	59 (16)
	K-40	1460 (216)	1453 (43)	1463 (259)
NORM6	Ra-226	49.7 (8.9)	45.7 (11.5)	51.3 (7.7)
	Th-232	56.4 (3.9)	54.7 (4.7)	57.1 (3.6)
	K-40	1221 (70)	1271 (65)	1202 (64)

1 Massic activity in dry mass. The standard uncertainty is shown in parentheses.

Table 43. Mean results from participants who indicated if they monitor radioactivity in the environment.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Monitors radioactivity in the environment?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	87.5 (1.5)	80.4 (6.1)
	Th-232	49.4 (3.8)	53.6 (1.2)	48.2 (3.4)
	K-40	187 (13)	190 (5)	186 (15)
NORM2	Ra-226	31.4 (3.8)	33.1 (1.5)	30.9 (4.2)
	Th-232	23.7 (2.0)	25.5 (1.8)	23.2 (1.9)
	K-40	327 (27)	337 (19)	324 (29)
NORM3	Ra-226	31.3 (3.2)	31.9 (2.3)	31.1 (3.5)
	Th-232	23.1 (2.6)	23.6 (2.2)	23.0 (2.8)
	K-40	310 (18)	316 (27)	309 (16)
NORM4	Ra-226	22.7 (5.0)	29.7 (7.3)	20.8 (1.9)
	Th-232	37.1 (2.5)	37.8 (2.6)	36.9 (2.6)
	K-40	58 (10)	63 (10)	57 (10)
NORM5	Ra-226	118 (21)	116 (16)	119 (23)
	Th-232	60 (13)	63 (10)	59 (14)
	K-40	1460 (216)	1375 (170)	1484 (228)
NORM6	Ra-226	49.7 (8.9)	55.8 (12.5)	48.0 (7.5)
	Th-232	56.4 (3.9)	57.2 (7.0)	56.2 (3.1)
	K-40	1221 (70)	1204 (132)	1226 (52)

1 Massic activity in dry mass. The standard uncertainty is shown in parentheses.

7.3 Experience and accreditation

In the first survey, four out of 15 participants answered that they do not have experience in measuring naturally occurring radionuclides in construction products using semiconductor gamma-ray spectrometric detectors. The four participants include L07. They answer that *they do have* experience in gamma-ray spectrometry, *but not necessarily* in measuring naturally occurring radionuclides in construction products, *or not necessarily* with semiconductor detectors. Participant L07 was rejected for not using a semiconductor. Seven laboratories are ISO 17025 accredited for gamma-ray spectrometry. The individual replies of the participants, except L07, are presented in Table 44.

Table 44. Replies from the participants on general questions related to their level of experience and ISO 17025 accreditation.

Participant	Experience in measuring naturally occurring radionuclides in construction products using semiconductor gamma-ray spectrometric detectors?	Q 1.2, Q 1.2.1: ISO 17025 accredited for gamma-ray spectrometry?
L01	YES	NO
L02	YES	NO
L03	YES	YES
L04	YES	YES
L05	YES	YES
L06	YES	NO
L08	YES	NO
L09	NO	NO
L10	YES	YES
L11	YES	NO
L12	NO	YES
L13	YES	YES
L14	YES	YES
L15	NO	NO

Table 45 shows the means between the participants that claim to have the experience and those who don't. Only for the measurement of Ra-226 in material NORM2, a significant difference is observed between the mean result over the two participants who do not have experience and the reference value (See Figure 80). Since NORM2 and NORM3 are from the same base material, issues with Ra-226 could also be expected for NORM3 and therefore the corresponding graph is included in Figure 80. For NORM3, two out of three results of the non-experienced participants correspond well with the reference value, while for NORM2 both results deviate. (Note that participant L15 did not analyse material NORM2.) Further investigation shows that participant L12 reported a significantly lower value for Ra-226 than the reference value *for all the materials*. For Th-232 and K-40 (except for NORM4), the results reported by participant L12 are in line with the reference value, for all the materials. Although the participant claimed to have sealed the test specimen container, the container was not completely filled and not vibrated or compacted. Hence, a lack of experience by L12 could be the cause of a significant negative bias in the measurement of Ra-226.

there are indications that a lack of experience will manifest itself in a significant negative bias for Ra-226

With respect to ISO 17025 accreditation, Table 46 presents the different means over the participants who are/are not accredited. The mean results over the participants who are ISO 17025 accredited for gamma-ray spectrometry is not systematically closer to the reference value than the mean over those participants who are not accredited. On the contrary, for 11 out of the 18 test properties, the bias of the average over the participants who are not accredited is smaller than the bias of the average over those that are accredited.

The impact on the mean differs from the impact on the spread. For 12 out of the 18 test properties, the spread between the results reported by participants who are accredited is smaller than the spread between the results reported by non-accredited participants. This is also demonstrated by the results of Cochran's and Grubbs' test in Section 4.6: None of the participants for which at least one outlier was identified is ISO 17025 accredited.

ISO 17025 accredited participants do not exhibit a significantly smaller bias, but the results are more consistent with other accredited participants

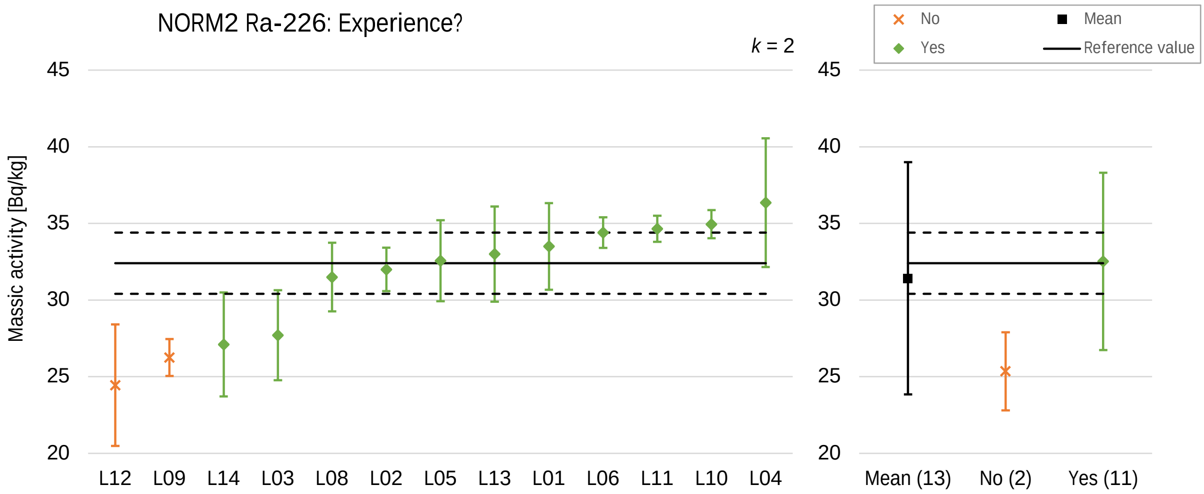
Table 45. Mean results from participants who indicated if they have experience in measuring naturally occurring radionuclides in construction products using semiconductor gamma-ray spectrometric detectors.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	Experience in measuring naturally occurring radionuclides in construction products using semiconductor gamma-ray spectrometric detectors?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	77.0 (10.1)	83.2 (4.5)
	Th-232	49.4 (3.8)	45.0 (7.0)	50.6 (2.8)
	K-40	187 (13)	181 (23)	188 (10)
NORM2	Ra-226 ([2])	31.4 (3.8)	25.4 (1.3)	32.5 (2.9)
	Th-232	23.7 (2.0)	21.0 (3.1)	24.2 (1.5)
	K-40	327 (27)	292 (50)	334 (18)
NORM3	Ra-226	31.3 (3.2)	29.9 (4.9)	31.6 (2.8)
	Th-232	23.1 (2.6)	24.3 (5.6)	22.8 (1.4)
	K-40	310 (18)	304 (26)	312 (17)
NORM4	Ra-226	22.7 (5.0)	20.1 (3.2)	23.4 (5.3)
	Th-232	37.1 (2.5)	36.5 (3.2)	37.3 (2.4)
	K-40	58 (10)	48 (13)	61 (7)
NORM5	Ra-226	118 (21)	127 (38)	116 (16)
	Th-232	60 (13)	46 (25)	64 (6)
	K-40	1460 (216)	1407 (81)	1475 (243)
NORM6	Ra-226	49.7 (8.9)	43.6 (9.0)	51.4 (8.4)
	Th-232	56.4 (3.9)	54.5 (4.0)	56.9 (3.9)
	K-40	1221 (70)	1215 (66)	1223 (74)

1 Massic activity in dry mass. The standard uncertainty is shown in parentheses.

2 Significant deviations were observed between the mean results over the participants who indicated that they do not have experience in measuring naturally occurring radionuclides in construction products using semiconductor gamma-ray spectrometric detectors and the reference value.

Figure 80. Results reported by the participants for Ra-226 in NORM2 and NORM3. At the left, results are grouped whether or not the participants claimed to have experience in measurements of naturally occurring radionuclides in construction products using semiconductor gamma-ray spectrometric detectors, and then sorted. At the right, the mean is shown for the two groups, next to the overall mean. Massic activity in dry mass. The number of participants is shown in parentheses.



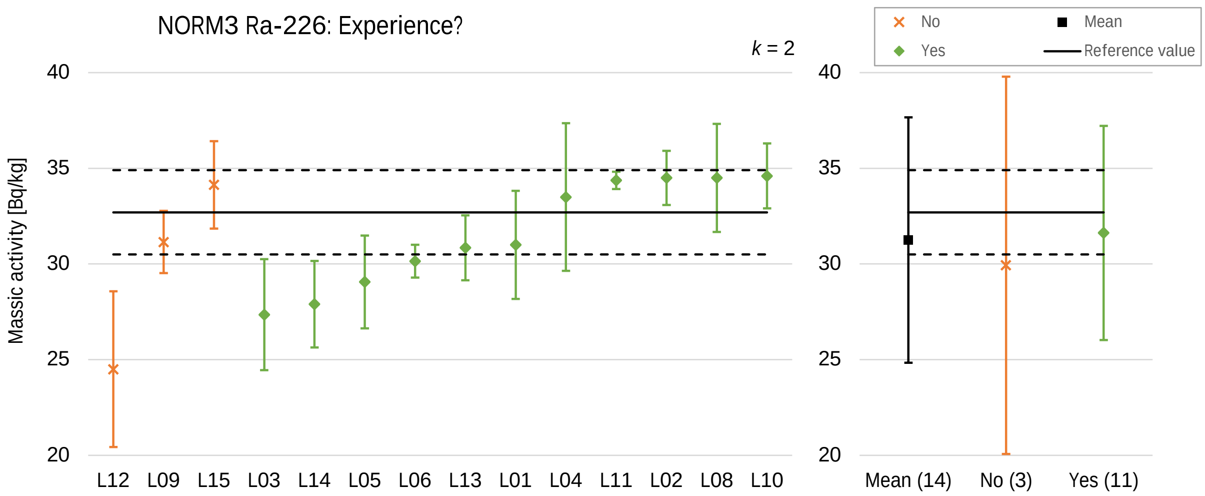


Table 46. Mean results from participants who indicated if they are ISO17025 accredited for performing gamma-ray spectrometry measurements.

Material	Radionuclide	Mean massic activity [Bq/kg] ([1])		
		Mean	ISO 17025 accredited for performing gamma-ray spectrometry measurement?	
			No	Yes
NORM1	Ra-226	81.9 (6.2)	85.3 (2.9)	78.5 (6.9)
	Th-232	49.4 (3.8)	49.7 (5.2)	49.1 (2.0)
	K-40	187 (13)	188 (10)	185 (16)
NORM2	Ra-226	31.4 (3.8)	32.1 (3.1)	30.9 (4.5)
	Th-232	23.7 (2.0)	23.7 (3.0)	23.7 (1.0)
	K-40	327 (27)	321 (34)	333 (20)
NORM3	Ra-226	31.3 (3.2)	32.8 (2.0)	29.7 (3.6)
	Th-232	23.1 (2.6)	24.1 (3.4)	22.1 (1.1)
	K-40	310 (18)	317 (20)	303 (14)
NORM4	Ra-226	22.7 (5.0)	25.1 (6.0)	20.3 (2.2)
	Th-232	37.1 (2.5)	37.2 (3.1)	36.9 (2.0)
	K-40	58 (10)	60 (7)	56 (12)
NORM5	Ra-226	118 (21)	125 (24)	112 (16)
	Th-232	60 (13)	56 (19)	63 (4)
	K-40	1460 (216)	1501 (307)	1420 (51)
NORM6	Ra-226	49.7 (8.9)	52.6 (8.4)	46.8 (9.0)
	Th-232	56.4 (3.9)	56.7 (5.0)	56.1 (2.8)
	K-40	1221 (70)	1222 (84)	1221 (59)

1 Massic activity in dry mass. The standard uncertainty is shown in parentheses.

8 Comments on WD EN 17216

8.1 Comments from participating laboratories on WD EN 17216

The participants in the collaborative assessment were given the opportunity to comment on the draft using the reporting questionnaire (Q 2.14, Q 5.1.1 and Q 5.2) and using the standardised commenting form used by ISO/IEC and CEN/CENELEC committee members.

— Q 2.14 asked the participating laboratories if they experienced difficulties related to the sample preparation. Table 47 shows their answers.

— Q 5.1 asked the participants if they could strictly follow WD EN 17216 and that it is unambiguous and clear. If they answered no, they were invited to comment (Q 5.1.1). (The impact of answering yes or no on the reported results is discussed in Section 6.8.) Table 48 presents the answers provided by the participants on the question where the standard could not be followed or where it was not clear (Q 5.1.1).

— Table 49 includes the comments and suggestions to improve the standard received via the reporting survey or via the commenting form. Note that the clause numbers on the reporting forms provided by the participants were adjusted to correspond with the version of the draft standard intended for being used in the collaborative trial of CEN/TS 17216 (prEN 17216, dated 2022-08, Reference [4]).

Table 47. Experienced difficulties by participants related to sample preparation.

Q 2.14: Difficulties related to the sample preparation
The slow compaction rate of the two samples of material NORM1 led to a reduction in sample height in the first week. The height did not change further.
Could not find a vibrating table on the market to automatically compact the sample.
Experienced difficulties in crushing the expanded clay blocks.

Table 48. Answers provided by the participants on the question where the standard could not be followed or where it was not clear.

Q 5.1.1: Where could the standard not be followed or where it was not clear?
Annex A
Section 6.3.2.1: The samples were not sieved. A vibrating table was not used, but the sample was stamped by hand several times. Section 6.3.2.2: The samples containers were not tested for radon leakage with the method described in Annex A. Section 6.3.2.3 and 7.2.3.3: The samples were not dried and the dry matter content was not determined. The samples arrived in airtight sealed containers. After the original containers were opened the sample material was immediately transferred to the measurement vessels and weighed. The weight was monitored for several days. As the weight did not change the dry matter content was assumed to be stable.
In principle, yes. The main problem is the use of commercial analysis programmes which, although they generally include the calculation of the activity according to this DRAFT, in relation to the calculation of uncertainty some of those included here do not appear, although the most modern versions have options to include them.
We calibrate with gamma cocktail and not adding each of the radionuclides. We don't determine the radon leakage.
It is difficult to do as described in Annex A.

Table 49. Comments and suggestions provided by the participants to improve the standard.

Clause (1)	Q5.2: Comments and suggestions to improve the standard
6.3.1.7	Comments: The possibility of sealing the container with radon-proof vacuum bags should be added.
7.2.1.3	Comments: The temperature of 103 ± 2 °C seems too strict and leads to increased qualification effort without leading to a more correct result. The quality requirements for ovens are specified in ISO 18589-2 (Measurement of radioactivity in the environment — Soil — Part 2: Guidance for the selection of the sampling strategy, sampling and pre-treatment of samples) as 105 ± 10 °C – we suggest these to be adopted. Proposed change: Drying oven, capable of maintaining a set temperature within ± 10 °C.
7.2.3.3	Comments: The temperature of 103 ± 2 °C seems too strict and leads to increased qualification effort without leading to a more correct result. The drying process is specified in ISO 18589-2 as 105 ± 10 °C to a constant weight. Proposed change: a) Place the test portion for the determination of the dry matter content in the drying oven at a temperature of (105 ± 10) °C.
A.2.2	Comments: We suggest the addition of water with high radon content as a radon source for the leak test.
Annex A	Comments: An alternative to the radon leakage test described in Annex A should be added, e.g., for laboratories using radon-proof vacuum bags. Proposed change: Radon tightness of the sample container sealed in a radon-proof vacuum bag can also be checked by determining the activity concentration of Ra-226 in a reference material after reaching secular equilibrium with its daughter products Pb-214 und Bi-214.
5	Comments: Is assumption of Th-232 in equilibrium with Ac-228 and Tl-208, same half-life should be used, that of Th-232
5	Comments: It takes several decades to achieve equilibrium between Th-232 and Ra-228. Assumption of equilibrium between Th-232 and Th-228 depends on how and when this construction material has been produced. If a long time has elapsed (several years) both Th isotopes will have different activities. Today, some building materials are made from wastes, with different origins.

Clause ([1])	Q5.2: Comments and suggestions to improve the standard
Table 1	Comments: Values of emission probabilities are slightly different to those commonly accepted of LNHB.
5	Comments: The possibility of determining Ra-226 directly from the 186 keV line is not mentioned. We suggest mentioning this possibility (see text on the right), which substantially simplifies the whole measurement process. Proposed change: Most natural materials contain the isotopes of uranium U-238 and U-235 in a natural isotopic mix. Therefore, the activity of Ra-226 in building materials, where the equilibrium in the uranium chain from U-238 to Ra-226 is not violated, can be assessed as follows: Subtracting the contribution of U-235 from the 186 keV peak created by Ra-226 photons. This approach does not require hermetically sealed samples.
6.3.1.2 6.3.2.1	Comments: Sifting separates fractions with different sizes, but the activity can vary from fraction to fraction! Proposed change: Using a sieve is not recommended as a method of sample preparation. Sifting should only serve as a confirmation that the granularity of the sample as a whole does not exceed 2 mm. It should be emphasized that sifting is not a method that should lead to the exclusion of fractions bigger than 2 mm.
6.3.1.4	Comments: Mechanical compacting device (i.e. vibrating table) can cause non-homogenous distribution of material in sample. Proposed change: Using a compacting device such as a vibrating table is suitable only for perfectly homogenous samples. Using such a device for a non-homogenous sample can lead to fraction separation.
7.2.3.3	Comments: … three consecutive weightings made 24 h apart is smaller than 0,1 % … We find it unnecessary to check for constant weight 3 times in the span of 3 days. This method seems unnecessarily expensive, both time-wise and financially-wise. Proposed change: Drying is carried out until a constant weight is reached. E.g. ISO 11465:1993 states a weight check after 4 hours.

Clause ([1])	Q5.2: Comments and suggestions to improve the standard
8.3.1	Comments: Possible notation error. The text states that activity should be calculated in test specimen “k”, but the indices in formula (3) are “i” for radionuclide and “l” for test specimen. Test specimens are labelled with “l” multiple times, but never with “k”. Proposed change: Fixing the notation (k → l)
8.6	Comments: Possible notation error (line 605). Another instance of unclear notation of test specimens. The live time tk is indexed with “k”, but the activity is indexed with “l” for test specimen. Proposed change: Fixing the notation (k → l)
9	Comments: We do not find it necessary to include the following: 1) dates of the preparation and testing of the test specimen; 2) the mass of the test specimen In the test report. Proposed change: Omit points f-1) and f-2)
9	Comments: Missing a point with information about the date, to which the activity is calculated. Should it be the day of sampling or the day of measuring? Proposed change: Expand the list of required test report information. Include the reference date and specify whether this date should be the day when the material was sampled (as stated by the customer) or whether it should be the day of the measurement.
Table D.1	Comments: Missing digit in the energy of Tl-208. It should be 2 615 keV!

Clause ([1])	Q5.2: Comments and suggestions to improve the standard
	Comments: As far as Draft EN 17216 (E) is concerned, the indications are clear and unambiguous. However, there are some aspects that I would like to comment on: - We did not determine radon leakage from the specimen container following Annex A, since the laboratory already uses an adequate sealing method to avoid leakage, a fact that is supported by the results of the interlaboratory exercises in which we have participated. - For calibration and determination of detection efficiency, the instructions in Annex B have not been followed as the laboratory uses an alternative way of calibration by preparing and measuring a large number of geometries with different densities using an NPL cocktail with a mixture of radionuclide standards of energies between 59 and 1836 keV, followed by the fitting of the corresponding efficiency curve (ISO 20042:2021)

[1] The clause numbers correspond with the version of the draft standard intended for being used in the collaborative trial of CEN/TS 17216 (prEN 17216, dated 2022-08, Reference [4]).

8.2 Comments from the JRC on WD EN 17216

The JRC provided comments on the method described in CEN/TS 17216 via e-mail communication, participation at meetings of TG 31 and bilateral discussions with members of TG 31. The Technical Group reached a consensus on the draft standard for distribution to the participants of the collaborative trial as WD EN 17216 [4].

The JRC provides the following additional comments on WD EN 17216:

- The number of test specimens under equations (8) and (9) should be written with a small n, not as N.
- The use of the squares is not necessary in Formula (11) since the uncertainties are positive values and the squares function is strictly increasing for positive real numbers. Easier is to write $u_{i;R} = \max\{u_{i;\mathrm{int}}, u_{i;\mathrm{ext}}\}$.
- It could be made clearer that the standard allows to determine the dry matter correction factor with a test portion or to fully dry the test specimen itself.
- Compacting by vibration or tapping may adversely impact homogeneity and lead to an increased bias, for example when smaller particles have another massic activity than bigger ones. This kind of segregation may be expected for samples such as concrete, consisting of hard aggregates and small, powder-like sand and cement particles. It is suggested to leave out vibrating and tapping. After homogenisation, the test specimen should be compacted by pressing down on its surface.
- The definition of the dry mass correction factor could be accompanied with a note that explains that a number close to, but lower than one is expected. Two of fourteen participants reported a dry mass correction factor between zero and a few percent, which cannot be correct when Equation (1) of the standard is applied.
- The standard should draw more attention on the interference of the 1459 keV line from Ac-228 in the determination of K-40. Especially for materials with relatively low activities of K-40 compared to Th-232 (which is determined via Ac-228). Not correcting for the interference results in a positive bias for K-40.
- Clause 9 of WD EN 17216 specifies that the test results shall be reported as $a_i \pm u_{i,\mathrm{tot}}$, where a_i is the massic activity of radionuclide i (Ra-226, Th-232 or K-40) and $u_{i,\mathrm{tot}}$ the total uncertainty of the massic activity of radionuclide i, both expressed in Bq/kg. However, from formula 12 of WD EN 17216, it is understood that $u_{i,\mathrm{tot}}$ is a standard uncertainty and therefore the notation with $\pm$ should be avoided as recommended by Clause 7.2.2 of the GUM [10]. It should be considered to add a recommendation on reporting values and uncertainties, e.g. by referring to Chapter 7 of the GUM [10] or [11].
- Some laboratories report an excessive number of digits. For example, L15 reported 337.85 (15.21) Bq/kg K40 in a NORM3 test specimen. It is generally sufficient to report a maximum of two significant digits for the uncertainty and to round the numerical value of the measurement result to the least significant figure in the value of the (expanded) uncertainty assigned to the measurement result. Applying this rule to the example above would result in 338 (15) Bq/kg, which is much more realistic.

9 Summary and conclusions

9.1 Policy question

The construction minerals sector is the largest among the non-energy extractive industries in the EU with respect to tonnage of extracted minerals, number of companies and employees, and turnover. Construction products may contain minerals with enhanced levels of natural radioactivity which may on a long term result in a non-negligible radiological exposure to humans. In the frame of the European Commission's Mandate M/366 from the Directorate-General for Internal Market, Industry, Entrepreneurship and SMEs (DG GROW), CEN/TC 351/WG 3 (dealing with the assessment of radiation from construction products) developed draft European Standard WD EN 17216 for the determination of the activity concentrations of Ra-226, Th-232 and K-40 in construction products. The standard is intended as a reference method in support of performance assessment (CE marking) of construction products.

The robustness of the method was validated earlier by an expert laboratory. Based on the results of the robustness testing, the committee developed a revised draft. This draft was tested for precision before CEN/TC 351 may decide to launch the CEN enquiry to publish the method as a European Standard. The assessment of the precision (repeatability and reproducibility) is the objective of the study.

9.2 Objectives of the study

The purpose of the study discussed in this report is to validate the precision (repeatability and reproducibility) of draft standard WD EN 17216 by means of a collaborative assessment. Participants were invited to apply the method mentioned in the draft standard for the analysis of a series of samples. They were asked to report the results to the JRC, who then scrutinised the results and determined the repeatability and reproducibility. Since reference values were available, the JRC also assessed the bias of the method.

It is not the aim of the study to evaluate the performance of the laboratories.

9.3 Summary of the collaborative assessment

Based on criteria agreed with CEN/TC 351/WG 3, five construction products were selected. From those products, JRC prepared six test materials, named:

- NORM1: Cement;
- NORM2: Pieces of expanded clay blocks;
- NORM3: Expanded clay blocks, in milled form;
- NORM4: Plaster;
- NORM5: Aggregates;
- NORM6: Concrete, in milled form.

The JRC determined the massic activity of Ra-226, Th-232 and K-40 in these materials. The materials were distributed over a number of samples. The homogeneity, short- and long-term stability of these samples was tested and discussed in a separate report [5]. The samples were found fit for purpose.

Fifteen participating laboratories received two samples of each of the materials. One participant asked and received four samples. The laboratories were asked to analyse the two samples in repeatability conditions thereby following the draft standard as close as possible and to report where they deviated from the method. For each of the six materials, they had to report the massic activity of Ra-226, Th-232 and K-40 of the dry mass, determined on both samples.

The data from one participant was rejected because of not using the required detector type. ISO 5725-2 was followed to determine the reproducibility and repeatability. Cochran's and Grubbs' tests were applied to detect stragglers and outliers in the means and spread of the reported results. Seven of 249 results were identified as outliers and not retained in the calculation of repeatability, reproducibility and bias. The results from one participant were excluded for their inconsistency. The repeatability and reproducibility were first calculated for each material and radionuclide. Then, the relationship between the precision and the mean was investigated, as well as the effect of sample heterogeneity. Finally, an average precision for each of the radionuclides was determined, making abstraction of the material. In a similar way, the bias of the method was determined following ISO 5725-4.

The draft standard contains a number of normative clauses. For the next ten requirements, the participants were asked if they followed or did not follow the requirement:

— Crushing and sieving the material to a particle size less than 2 mm;
— Homogenising the material;
— Completely filling the test specimen containers;
— Vibrating or compacting the test specimen containers;
— Making the test specimen container radon tight;
— Determining the dry matter correction factor;
— Applying attenuation corrections;
— Applying true-summing corrections;
— Calculating the activity according to Clause 8.3 of the draft standard;
— Calculating the uncertainty according to Clause 8.4 of the draft standard.

The results between groups of participants who followed / not followed the requirements were compared with each other and with the overall mean, to identify a possible systematic impact of not following the requirement. The outliers were included in the data.

In a similar way, the impact of a few other aspects was assessed, such as the experience, accreditation and routine activities of the participants.

The participants were given the opportunity to comment on the draft method. Their comments are complemented with a few remaining comments from the JRC.

9.4 Key findings

The screening of the materials demonstrated that the maximum uncertainty related to possible between-sample variation is not larger than 3 %. This value is a lower limit for repeatability.

For Ra-226, Th-232 and K-40, the repeatability relative standard deviation of the method is around 5 % over a limited validity range of massic activity.

The between-laboratory variability however, is considerably larger for Ra-226 than for the other two radionuclides, which is reflected in the larger reproducibility of 12.6 (5.4) % for Ra-226 compared with 7.7 (3.2) % for Th-232 and 8.3 (5.0) % for K-40. In general, it can be said that thirteen (out of fourteen) participants are consistent in performing the analysis of the two replicates. However, there is less consistency between the participants when it comes to the practice (how the work was conducted), especially for Ra-226.

Statistically, for all three radionuclides and six materials, at the 95 % confidence level, none of the biases observed is significant, with the exception of the bias of K-40 in material NORM5. The latter was found to be significant due to random effects which resulted in negative between-laboratory variance. This is contrary to the situation for Ra-226, where the poor between-laboratory consistency results in a larger estimate of the variation of the bias, which renders the bias insignificant at the 95 % confidence level.

Although no effect of the application of the requirements on sealing and determining the radon leakage rate could be demonstrated, the relative (but insignificant) bias of -7.5 (5.8) % for Ra-226 still raises concerns on radon leakage. Both, the bias for Th-232 (-1.4 (3.7) %) and for K-40 (3.2 (2.4) %), are insignificant at the 95 % confidence level, with exception of K-40 in NORM5 as mentioned. A positive bias for K-40 may be the result of not correcting for the interference of Ac-228 (peak at 1459.1 keV) in samples with a high activity of Th-232 compared to K-40 (with a peak at 1460.8 keV). Other causes of bias may be due to the extrapolation of the efficiency calibration curve after 1332 keV, inadequate coincidence summing correction of calibration sources or inadequate subtraction of background.

For none of the selection of ten normative requirements, a statistically significant difference between following / not following the requirement could be demonstrated. Furthermore, based on the data from the collaborative assessment:

- The impact of shaking the test specimen could not be proven;
- No significant differences were observed between the three different methods of determining the detection efficiency;
- No significant differences were observed between participants who claimed to have applied WD EN 17216 strictly and those who claimed not;
- No correlation can statistically be observed between the relative laboratory bias and how many of the ten selected requirements a participant followed.

For some of the materials and radionuclides, not following one or more normative requirements may have led to the outliers identified. However, the available data does not allow to make such a statement with a significant level of confidence.

The participants who perform R&D activities have a smaller bias and appear to have a smaller spread between their values than those who do not perform R&D. A difference between bias and spread of participants who perform / do not perform environmental monitoring cannot be proven based on the available data. There are indications that a lack of experience results in a significant negative bias for Ra-226. ISO/IEC 17025 accredited participants do not exhibit a significantly smaller bias, but the results are more consistent with other accredited participants.

9.5 Recommendations

For the assessment of the precision of WD EN 17216, fourteen laboratories were asked to apply the standard as strictly as possible. Nine of them claimed that they applied the method strictly, but when being specifically asked on the application of a selection of ten normative requirements, only one laboratory declares to have followed all ten requirements. Seven out of fourteen participants did not apply the requirements on sealing the sample containers and on determining the radon leakage rate, which is important for the measurement of Ra-226. Laboratories have difficulties applying the method in Annex A of the draft.

Despite the fact that not all laboratories followed the method rigorously, the results on precision and bias are still reasonable. Of the three evaluated radionuclides, the results of Ra-226 measurements showed the highest reproducibility and (absolute) bias, 12.6 (5.4) % and -7.5 (5.8) % respectively. An important remark on these numbers is that seven out of 249 results were rejected as outliers and that all results from one inconsistently performing laboratory were also rejected.

Since the precision and bias are reasonable, even when the method is not strictly followed, questions could be raised on the need for certain normative requirements of the standard. However, since the method aims at providing a reference method in support of performance assessment of construction products, the legal implications of a laboratory which is not able to demonstrate full compliance with the method may be considerable. Therefore, it may be expected that, as laboratories apply the method more rigorously while gaining experience, the precision and bias will improve. Also, for legal implications, it is not recommended to loosen the normative requirements in the method further. Alternatively, laboratories may benefit from support in applying the method.

Taking into account the size of the construction minerals sector and the soon to be finalised revision of the Construction Products Regulation (CPR), an increased interest of laboratories is expected to determine Ra-226, Th-232 and K-40 in construction products for performance assessment. Possible ways for the JRC to support these laboratories and to improve the quality of the measurement of natural radioactivity in construction products are the following:

- The JRC could assess the availability and the need of certified reference materials and may decide to develop these materials.
- The JRC could foster the exchange of good practices between laboratories via the Group of Notified Bodies under the framework of the CPR. For example, the JRC could regularly organise proficiency tests on construction products.

The role of the JRC in this matter should be further discussed with the Directorate-General for Energy and the Directorate-General for Internal Market, Industry, Entrepreneurship and SMEs.

References

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[2] Administrative Arrangement N° SI2.834008 - JRC.35292 signed between DG GROW and DG JRC, Ares(2020)5380111.

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List of abbreviations and definitions

CE	European Conformity
CEN	European Committee for Standardization
CENELEC	European Electrotechnical Committee for Standardization
CPR	Construction Products Regulation
DG ENER	Directorate-General for Energy
DG GROW	Directorate-General for Internal Market, Industry, Entrepreneurship and SMEs
EU	European Union
GIG	Główny Instytut Górnictwa, Silesian Centre for Environmental Radioactivity, Poland
GUM	Guide to the expression of uncertainty in measurement
HPGe	High-Purity Germanium
IEC	International Electrotechnical Commission
ILC	Interlaboratory Comparison
ISO	International Organization for Standardization
JRC	Joint Research Centre
NEN	Stichting Koninklijk Nederlands Normalisatie Instituut
SGDS	EC subgroup on dangerous substances
TC	Technical Committee
TG	Technical Group
TS	Technical Specification
WG	Working Group

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Annexes

Annex 1. Survey – Call for expression of interest

☑ Save a backup on your local computer (disable if you are using a public/shared computer)

Call for expression of interest to participate in an interlaboratory validation of EN 17216 draft standard

Fields marked with * are mandatory. ✖

✖

Disclaimer

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Pages | General information | Laboratory information | Analytical experience

Information to potential participants in interlaboratory validation of draft standard EN 17216

Construction products - Assessment of release of dangerous substances - Determination of activity concentrations of radium-226, thorium-232 and potassium-40 in construction products using semiconductor gamma-ray spectrometry

The interlaboratory validation study for the draft standard EN 17216 is planned for the beginning of 2022.

In this study each laboratory will receive 12 items for the measurements. Ten of them will be pulverised and two will be unprocessed. The two unprocessed items will need to be pulverised by the laboratory. For each item the massic activity (Becquerel per unit of dry mass) of K-40, Ra-226 and Th-232 will have to be measured using a semiconductor gamma-ray spectrometer and reported together with the detection limits, decision thresholds and detailed information on the uncertainty budget and the measurements themselves. Since the activity is to be specified for the dry material, the moisture content will need to be determined as well. All measurements will have to be conducted according to the draft standard EN 17216, which will be provided together with the samples. All measurements have to be conducted under repeatability conditions – using one detector by one operator within a short period of time. Laboratories will have three month for reporting results.

If your laboratory is interested in participating to this interlaboratory validation study please fill in this questionnaire. All replies will be evaluated and selected candidates will be contacted by the EC JRC-Geel.

Next

Pages | General information | Laboratory information | Analytical experience

Laboratory information

* Name of the institution

* Contact person

* Email address

@

* Phone number

* Street and number/Post box

* City

* Postal code

* Country

Previous | Next

Pages

General information | Laboratory information | Analytical experience

Analytical experience

* Do you have experience in measurements of naturally occurring radionuclides in construction products using semiconductor gamma-ray spectrometric detectors?

- (x) Yes
- ( ) No

* What type of construction products do you typically analyse?

* In which form do you receive these samples?

- ( ) As produced (e.g. cement, plaster, whole bricks, tiles)
- ( ) Pre-treated or processed (e.g. milled bricks)
- ( ) Both

* Do you have necessary equipment to process samples of construction products? (e.g. by crushing, pulverising, sieving)

- (x) Yes
- ( ) No

* Moisture content determination will be part of this study. Do you have necessary equipment to perform drying of samples up to 105 °C?

* What equipment do you have for sample processing? Please provide the kind of equipment and brand name/model number if possible.

* Approximately how many samples do you annually analyse for the presence of naturally occurring radionuclides in construction products?

* What is the typical sample mass used for a single measurement?

* How many years of experience do you have in these measurements?

* What type of detector(s) do you use for these analyses? Please list the manufacturer and model number of each detector, if possible.

* What type of efficiency calibration method do you use?

- ○ Reference material or source
- ○ Monte Carlo calculations only
- ○ Combination of Monte Carlo calculations and reference material or source measurements
- ◉ Other

Comments

Previous Submit

Annex 2. Participating laboratories (in random order)

1. National Radiation Protection Institute (SÚRO)
   Bartoškova 28
   14000 Praha 4
   Czechia

2. Federal Office for Radiation Protection
   Köpenicker Allee 120-130
   10318 Berlin
   Germany

3. University of the Basque Country - Ingeniería Energética - LMBA
   Plaza Ingeniero Torres Quevedo
   48013 Bilbao
   Spain

4. University of Barcelona - Laboratorio de Radiologia Ambiental
   Marti I Franques 1
   08028 Barcelona
   Spain

5. University of Extremadura - Applied Physics - Faculty of Veterinary - LARUEX
   Av. de la Universidad, s/n
   10003 Cáceres
   Spain

6. Health Institute Carlos III - Radiological Protection
   Ctra. Majadahonda-Pozuelo km.2
   28220 Majadahonda
   Spain

7. Research Centre of Natural Resources, Health and the Environment - Faculty of Experimental Sciences
   Dept. of Integrated Sciences
   Campus El Carmen s/n
   21007 Huelva
   Spain

8. Mark och Miljökontroll AB
   Bolshedens Industriväg 28
   42750 Billdal
   Sweden

9. Universidad de Sevilla - Física Aplicada II. E.T.S.A.
   Avenida Reina Mercedes, 2
   41012 Seville
   Spain

10. Universidad de Salamanca - Laboratorio de Radiaciones Ionizantes
    C. Espejo 2
    Edificio de I+D+i
    37007 Salamanca

11. AGES GmbH
    Spargelfeldstraße 191
    1220 Vienna
    Austria

12. Universidad de Valencia - Edificio de Investigación
Laboratorio de Radiactividad Ambiental
Calle Dr. Moliner, 50
46100 Valencia

13. NORM Technology Consulting S.L.
Facultad CC. Integradas - Facultad CC. Experimentales
Campus de El Carmen
21007 Huelva
Spain

14. Laboratorio de Radiactividad Ambiental - Universitat Politècnica de València
Camino de Vera s/n
46022 València
Spain

15. University of Granada - Faculty of Sciences - Inorganic Chemistry - Environmental Radiochemistry
Campus de Fuentenueva s/n
18071 Granada
Spain

Annex 3. Accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate G – Nuclear Safety and Security
Nuclear Data and Measurement Standards

Geel, 9 March 2023

Dear participant,

Thank you for participating in the validation study of the draft standard *WD EN 17216 (E): Construction products: Assessment of release of dangerous substances – Determination of activity of radium-226, thorium-232 and potassium-40 in construction products using semiconductor gamma-ray spectrometry*. The aim of this part of the validation study is to determine what differences can occur in actual testing.

For the validation of the method, you receive in total 12 samples to be analysed in your laboratory. These samples consist of 2 samples of 6 materials. Please store them at normal room temperature prior to analysis. Together with the samples, you receive a copy of the draft standard WD EN 17216 (E) to follow.

The purpose of the exercise is to discover the extent to which results can routinely vary in practise. For the analysis, the draft standard WD EN 17216 (E) shall be followed as close as possible. The same operator, who is routinely performing the gamma-ray spectrometry measurements, shall perform all the measurements. We would like you to analyse the 2 samples of identical materials as good as possible in the same (repeatable) conditions (i.e. same sample preparation, same analyst, same analytical equipment used, same calibration used, short interval between the analysis, etc). If in the course of the measurements changes in conditions occur (e.g. different operator, different detector used, etc), this shall be reported in the online questionnaire. The massic activity for each of the twelve samples received shall be the result of one measurement rounded up to 0.1 Bq/kg or smaller.

In a separate e-mail, you will receive the instructions for the reporting of the analytical results with their uncertainty, filling out the associated questionnaire and the commenting form on the draft standard. Based on your experience you can make suggestions to correct or improve the standard. The technical committee of CEN will evaluate them and implement them if found appropriate.

Please respect the reporting deadline of **21 June 2023**.

We remain at your disposal for any questions or comments at the following email: JRC-GEE-REM-COMPARISONS@ec.europa.eu.

With our best regards,

Raf VAN AMMEL/Jan PAEPEN	Petya MALO
Project Coördinators	Logistics assistant

Enclosure: WD EN 17216 (E)

Commission européenne/Europese Commissie, Retieseweg 111, 2440 Geel, BELGIQUE/BELGIË – Tel. direct line +32 14571-1286

JRC-GEE-REM-COMPARISONS@ec.europa.eu

Annex 4. Reporting questionnaire

Milc questionnaire

Comparison for Interlaboratory validation of draft EN 17216

The reporting of your measurement results and the associated questionnaire will be done online. Try to answer all the questions. Questions marked with an asterisk (*) are mandatory. The deadline for the submission of the results and the questionnaire is 21 June 2023. After submitting the report and the questionnaire, please download the pdf and send it as an attachment by email to JRC-GEE-REM-COMPARISONS@ec.europa.eu, as explained in the instructions that you received by email.

Submission Form

1. **General**

1.1. Which are the main activities carried out in your laboratory? E.g. Monitoring of radioactivity in the environment, Research and development, etc. *

1.2. Is your laboratory Certified (ISO 9000), Accredited (ISO 17025), Authorised, Other? *

1.2.1. If accredited, which method(s) is/are accredited? (e.g. Gamma ray spectrometry of environmental samples, etc.)

1.3. When were the samples received? dd/mm/yyyy *

(dd/MM/yyyy)

1.4. Were the samples received in good condition? *

1.5. How were the samples stored before analysis? *

2. **Sample preparation**

2.1. How were the test samples crushed to a particle size of 2mm according EN 17216 (E)? *

2.2. Which equipment was used to crush the test samples? (Type, Manufacturer, Model) *

2.3. Was the crushed material sieved? Which sieve was used? *

2.4. How was the sieved material homogenised? *

2.5. Describe the test specimen containers used? (Brand, Type, Shape, Material) *

2.6. Provide the dimensions of the test specimen containers (Height, Diameter in mm). *

2.7. Were the test specimen containers completely filled with test specimen? *

2.8. Was the test specimen vibrated to achieve a constant compacted sample? *

2.9. Which balance was used to determine the test specimen mass? (Manufacturer, Model, Latest calibration date) *

2.10. Specify the mass of the test specimen used (fill in Table 1. Masses of test specimen). *

See table **Table 1. Masses of test specimen** at bottom

2.11. How was the test specimen container made radon tight? *

2.12. Which material was used to tighten the test specimen container?

2.13. How long were the test specimen stored in the container before gamma-ray spectrometry measurement? *

2.14. Comments/difficulties encountered related to the sample preparation?

3. **Dry matter content determination**

3.1. Did you follow closely the section 6.3.2.3 for dry matter content determination? If not, please describe the deviations? *

3.2. Which test portion mass was used for dry matter content determination (g) and what is the correction factor eta, as defined in section 7.2.3.3 e)? (fill in Table 2. Dry matter content determination) *

See table **Table 2. Dry matter content determination** at bottom

4. **Measurements**

4.1. Which type of detector was used to analyse the test specimen? (Brand, Type, Nominal relative efficiency) *

4.2. How was the test specimen container positioned on the detector? (e.g. Centred by eye, Centred using sample holders, other) *

4.3. How long was a typical measurement time for one test specimen (s)? *

4.4. How was the background measurement(s) taken? (Typical duration, Container used)

4.5. What was the distance between the test specimen container and the endcap of the detector (mm)? *

4.6. Which data acquisition software was used? *

4.7. Which peak analysis software was used? *

4.8. How was the detection efficiency calibration determined? *

- ☐ a) Monte Carlo Simulations or numerical models
- ☐ b) Direct comparison with a calibration source
- ☐ c) LabSOCS
- ☐ d) Measurement of the full energy photopeak detection efficiency as a function of energy
- ☐ e) Other

4.8.1. If other, please describe *

4.8.2. If Monte Carlo code was used, specify the software used? *

4.9. If calibration source(s) was/were used, describe them? (Radionuclides present, activity + uncertainty, matrix composition) *

4.10. Were attenuation corrections made to the full energy photopeak detection efficiency curve? If so, how? *

4.11. Were true-summing corrections applied? If so, how? *

4.12. Which software was used for the activity calculation? *

4.13. Are the nuclear data in section 7.2.2.2 of EN 17216 (E) used in the calculations? *

4.14. Is the activity calculated according to section 8.3 of EN 17216 (E)? If not, which were the deviations? *

4.15. Is the uncertainty calculated following section 8.4 of EN 17216 (E)? *

4.16. Which typical sources of uncertainty are included in the calculations? (fill in Table 3. Sources of uncertainty) *

See table **Table 3. Sources of uncertainty** at bottom

4.17. Provide the measurement date and the operator for each measurement in Table 4. Measurement date and operator. *

See table **Table 4. Measurement date and operator** at bottom

5. **Additional information**

5.1. Could you strictly follow the Draft EN 17216 (E)? Is it unambiguous and clear? *

- a) Yes
- b) No

5.1.1. If not, describe where it could not be followed or it is not clear? *

5.2. Do you have any comments/suggestions to improve EN 17216 (E)? If so, make them in the Commenting_Form_draft prEN 17216.doc.

Table 1. Masses of test specimen

Questions/Response table	*Sample number*	*Test speciment mass (g)*
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		

Table 2. Dry matter content determination

Questions/Response table	*Sample number*	*Test portion mass (g)*	*Correction factor for dry mass (eta)*
1			
2			
3			
4			

Questions/Response table	*Sample number*	*Test portion mass (g)*	*Correction factor for dry mass (eta)*
5			
6			
7			
8			
9			
10			
11			
12			

Table 3. Sources of uncertainty

Questions/Response table	*Taken into account (y/n)*	*Relative standard uncertainty (%)*
Peak area		
Background peak area		
Test specimen filling height		
Test specimen positioning		
Dry mass correction		
Fitting efficiency curve		
Corrections on the efficiency curve		
Calibration sources		
True summing		

Questions/Response table	*Taken into account (y/n)*	*Relative standard uncertainty (%)*
Coincidences		
Dead time correction		
Radon leakage from test specimen container		
Total uncertainty		

Table 4. Measurement date and operator

Questions/Response table	*Sample number*	*Measurement date (dd/mm/yyyy)*	*Operator*
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			

Note: Page 11 of 11 of the reporting questionnaire is empty and not reproduced here.

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