

**A Second Comparison of Procedures for the Assay of Low Levels
of Gamma-emitters in Nuclear Site Waste**

JULIAN DEAN

JULY 2010

A Second Comparison of Procedures for the Assay of Low Levels of Gamma-emitters in Nuclear Site Waste

Julian Dean
Acoustics and Ionising Radiation Division

ABSTRACT

A second 'drum comparison' exercise has been run by NPL. This involved two samples: Drum A and Drum B. Each sample consisted of a 200 L drum loaded with plastic bottles and vermiculite. Within each sample, one bottle was loaded with ion-exchange resin containing a standardized mixture of ^{241}Am , ^{137}Cs and ^{60}Co . The activity concentrations (averaged across the drum contents) were approximately 0.3 Bq g^{-1} for Drum A and 9 Bq g^{-1} for Drum B.

The participants were required to report the activity concentrations of the individual radionuclides in one (or both) samples. They were initially told which radionuclides were present, and were given ranges for the total activity concentration and details of the drum itself and the chemical composition of the contents. They were told neither the location of the activity in the drums nor the number of bottles containing the activity (the 'hot volume'). Drum A was measured by 8 UK and 1 overseas laboratory and Drum B was measured by 12 UK and 2 overseas laboratories. After the initial results deadline, NPL revealed the location and size of the 'hot volume' in each drum and participants were invited to submit additional data. The exercise was followed by a workshop for discussion of the results.

Approximately 78 % of the initial results for Drum A were in agreement with the NPL value. After disclosure of the 'hot volume' details, a subset of the participants who had measured Drum A submitted additional data and approximately 56 % of these data agreed with NPL (n.b. approximately 76 % of the data submitted by this subset prior to disclosure agreed with NPL). For Drum B, the corresponding figures were: 56 % of initial data agreed with NPL; after disclosure, 74 % (from a subset of participants) agreed with NPL; 77 % of the initial data from the subset agreed with NPL.

The main findings were that (i) different approaches to efficiency modelling of the drums had been adopted and had produced different results (even using the same software) and (ii) the use of Segmented Gamma Scanners had in some cases produced discrepant results. Like the previous exercise, the participants again found the exercise a valuable test of their efficiency models and measurement systems.

Queen's Printer and Controller of HMSO, 2010

ISSN 1754-2952

National Physical Laboratory
Hampton Road, Teddington, Middlesex, TW11 0LW

Extracts from this report may be reproduced provided the source is acknowledged and the extract is not taken out of context.

Approved on behalf of the NPLML by Dr B Zeqiri, Knowledge Leader
Acoustics and Ionising Radiation Division

CONTENTS

1	INTRODUCTION	1
2	BACKGROUND.....	1
2.1	BRIEF SUMMARY OF 2007 EXERCISE.....	1
2.2	PLAN FOR 2007 EXERCISE.	2
3	PRIMARY AND SECONDARY STANDARDISATION	3
4	PREPARATION OF STANDARD SOURCES	3
5	CIRCULATION OF DRUM AND REPORTING OF DATA.....	4
6	RESULTS AND DATA ANALYSIS	5
7	DISCUSSION.....	8
7.1	AMERICIUM-241.....	8
7.1.1	Americium-241 in Drum A.	8
7.1.2	Americium-241 in Drum B.....	9
7.2	COBALT-60	10
7.2.1	Cobalt-60 in Drum A.....	10
7.2.2	Cobalt-60 in Drum B.	10
7.3	CAESIUM-137.	11
7.3.1	Caesium-137 in Drum A.....	11
7.3.2	Caesium-137 in Drum B.....	11
8	CONCLUSIONS.....	11
9	ACKNOWLEDGEMENTS	13
10	REFERENCES	13
11	TABLES	14
12	FIGURES	28
	APPENDICES	90

1. INTRODUCTION

As the UK nuclear industry continues to move further into the site decommissioning and remediation phase, there is a continuing need for the accurate measurement of radioactivity in a wide range of potentially contaminated (or activated) materials such as concrete and soft waste to ensure the materials are sentenced with confidence to the correct waste streams in accordance with national legislation. This is important not only for public safety reasons but also to minimise the amount of RSA Exempt material sentenced as Low Level Waste - vital as the UK's LLW storage capacity diminishes.

The National Physical Laboratory (NPL) recently established a national 'measurement infrastructure', funded by NPL's government sponsors, to support users in this field by providing standard sources, comparison exercises and advice on good measurement. As part of this infrastructure project, NPL had run a 'drum comparison' exercise [Dean, 2007] to enable laboratories involved in the clearance and sentencing of bulk γ -emitting waste to test their existing measurement protocols.

The exercise proved very useful to the participating laboratories, especially for testing measurement methods where the γ detection efficiencies had been calculated using mathematical modelling techniques – it gave the users a means of validating their modelling. A second exercise was requested, preferably involving a drum containing a 'hotspot'. However, it was also noted the specification of the 2007 standard drum had not been ideal; some users had found that the activity was below their limit of detection, whilst others found that the 'layered' internal structure of the drum made analysis difficult when certain detector types (e.g. Segmented Gamma Scanners) were used.

A second exercise was therefore run in 2009, this time using two 'standard drums' with specifications designed to address the above problems. This report covers:

- The background to the second exercise;
- Briefly, the methods used to standardize the radioactive starting solutions;
- The preparation and certification of the 'standard drums';
- The conduct of the exercise;
- The results obtained and how they were analysed;
- Discussion and conclusions, taking into account discussions from the follow-up workshop.

2. BACKGROUND

2.1 Brief summary of 2007 exercise

In 2005, NPL ran a 'Workshop on Metrology for Decommissioning and Site Clearance' to determine users' needs for standards and reference materials for measurements of radioactivity in concrete, steel, soil and other materials. 'Standard 200 L drums' of concrete or soft waste 'spiked' with γ -emitting radionuclides were identified as a priority, and as a result NPL ran a 'drum comparison' in 2007. The aim of the exercise was to enable laboratories to evaluate a 'prototype' standard drum and at the same time test their measurement systems with a traceable standard. Full details are given in the NPL report [Dean, 2007].

Briefly, the stages of the 2007 exercise were as follows:

- Production and certification of a 200 L drum containing a low-density matrix (approximately 300 kg m^{-3}) spiked with γ -emitters;
- Circulation to participating laboratories;
- Data analysis at NPL;
- A follow-up workshop to discuss the results.

The drum was prepared by partially filling each of a set of 240 plastic bottles (volume 500 mL each) with ion-exchange resin, spiking each bottle with a standard solution of the radionuclides of interest and then loading the bottles into a 200 L drum, choosing a resin mass per bottle which would yield the required overall contents density. Prior to loading, the sealed bottles were shaken to ensure the activity was homogeneously distributed throughout each bottle. The radionuclides present, and their activity concentrations averaged over the drum's contents were approximately as follows:

- ^{241}Am : 0.3 Bq g^{-1}
- ^{60}Co : 0.04 Bq g^{-1}
- ^{137}Cs : 0.03 Bq g^{-1}

Feedback from the 2007 post-exercise workshop indicated that:

- The drum had effectively consisted of a sequence of 'layers' of air and resin, presenting unusual difficulties for the laboratories who had used Segmented Gamma Scanners;
- The activities of some of the radionuclides present (^{60}Co and ^{137}Cs) had been too low to measure;
- A useful format for a second exercise would be to concentrate the activity present into a hotspot (although there was no consensus on what proportion of activity should be in the hotspot).

2.2 Plan for 2009 exercise

In order to overcome the above problems, and to ensure heterogeneity (in terms of activity), NPL decided to prepare two samples for the second exercise, to be coded 'Drum A' and 'Drum B'. Drum A would contain an array of plastic bottles (500 mL each) as before, but with only one bottle of resin (containing all the activity present). The remaining bottles and all voids would be filled with inactive vermiculite. Concentrating the activity in this way would test the participants' ability to measure an 'extreme' example of a heterogeneous sample. The activity concentration in Drum A (i.e. the activity in the 'active bottle' divided by the mass of the drum's contents) would be just below the current UK exemption limit of 0.4 Bq g^{-1} .

Drum B would be identical to Drum A, except:

- The activity concentration would be of the order of 10 Bq g^{-1} ;
- The proportions of the radionuclides present would be different from Drum A.

Drum A would cater for those laboratories who could measure below 0.4 Bq g^{-1} and for whom an exempt package would be preferable for on-site logistical reasons. Drum B would cater for those needing higher activities. It would be an excepted radioactive package (rather than a Type A package, again to try and minimise any on-site logistical problems).

Both samples would contain ^{241}Am , ^{60}Co and ^{137}Cs .

3. PRIMARY AND SECONDARY STANDARDISATION

NPL, as the UK's National Measurement Institute, carries out metrology-related research and provides measurement standards, reference materials, calibration services and support to users (e.g. via guidance documents, training, comparison exercises and workshops) across some 20 generic areas of science and engineering. At the international level, NPL forms part of the international measurement system, being one of many National Measurement Institutes (NMIs) worldwide who work together under the auspices of organisations such as the BIPM (Bureau International des Poids et Mesures) to share resources and to harmonise standards.

NPL's Radioactivity Metrology Group holds the laboratory's expertise in radioassay, and has provided support to UK measurements in this field for many years. As part of its role, the group develops and maintains primary and secondary systems for the measurement of radioactivity. Some of these systems were used (directly or indirectly) in the development of the standard sources for this exercise and they are very briefly described below. By using these systems, NPL was able to produce sources directly traceable to national standards of radioactivity.

The normal method for the primary standardization of β/γ -emitting radionuclides (including those used in this exercise) is the 4π β/γ -coincidence counting technique [NCRP, 1985]. The activity concentrations quoted by NPL for the drums are ultimately traceable to this technique. However, the technique is highly labour-intensive and is normally impractical for routine measurements; secondary standard instruments are therefore used instead. Solutions standardised by the coincidence counting method are used to calibrate instruments such as re-entrant ionization chambers using standard glass ampoules or other measurement geometries. Solutions of unknown activity can then be assayed on an ionization chamber if they are in one of these geometries. These chambers are the instruments of choice for most applications, their only disadvantage being a larger overall measurement uncertainty than is attainable using the primary method. The NPL secondary standard ionisation chamber has been calibrated for some 60 radionuclides, including those of interest in this exercise (^{241}Am , ^{60}Co and ^{137}Cs).

High-resolution γ -spectrometry is a commonly-used technique for the assay of γ -emitting radionuclides, and is described in many standard texts. The method is frequently used at NPL for:

- (i) Determining levels of impurity (e.g. to correct ionization chamber responses);
- (ii) Carrying out comparative measurements on two or more solutions (e.g. to verify gravimetric dilution factors);
- (iii) Assay (when a calibration has been derived traceable to primary standards).

4. PREPARATION OF STANDARD SOURCES

The following materials were procured:

- Standardized radionuclide solutions of ^{241}Am , ^{60}Co and ^{137}Cs (two per radionuclide, one at 2 – 4 kBq g⁻¹ and one at 100 – 300 kBq g⁻¹);
- Dowex 'Marathon C' Cation Exchange Resin* (approx. 1 kg);
- Inactive vermiculite (approx. 18 kg);
- 235 x 500 mL HDPE bottles;
- 1 x 200 L drum.

* The Dow Chemical Company, Michigan, USA. Use of this product is not meant to imply that NPL recommends it over other similar products.

The individual radionuclide solutions (except one) were assayed on the NPL secondary standard ionisation chamber. Each ampoule was also measured using a γ -spectrometer to check for the presence of any γ -emitting impurities. The one ampoule not measured using the ionisation chamber (one of the ^{241}Am ampoules) had been previously standardized in a traceable manner and was measured by γ -spectrometry to confirm its activity concentration.

The inactive resin was dried using a freeze-drying technique and 300 g (nominal) was dispensed to each of two 500 ml plastic bottles. The fill height was approximately 10 cm. One 'resin bottle' was spiked with weighed aliquots (approximately 1 g) of each of the 'lower-activity' radionuclide solutions ($2 - 4 \text{ kBq g}^{-1}$). This resulted in a total activity in the resin bottle of approximately 11 kBq. This bottle would be used in Drum A. The second resin bottle was similarly spiked with each radionuclide, but using the higher-activity ampoules ($100 - 300 \text{ kBq g}^{-1}$). This gave a total activity of approximately 320 kBq. This bottle would be used for Drum B. The resin bottles were allowed to surface-dry before being sealed, loaded into a mechanical mixer and then mixed for 6 hours to ensure within-bottle homogeneity of the activity.

The first resin bottle was loaded into a 200 L drum along with 234 bottles of inactive vermiculite. Each vermiculite bottle was completely full and all voids between bottles were filled with additional vermiculite. There were five layers of bottles in the drum and the resin bottle was positioned in the second layer down (i.e. the bottle lid was approximately 16.8 cm below the drum lid) and touching the wall of the drum. The drum was weighed empty, then after addition of the resin bottle, then after addition of the remaining bottles and the vermiculite. Double weighing was used (i.e. the resin bottle, the empty bottles and the vermiculite were all weighed separately on another balance before being loaded into the drum). The total mass of the drum's contents was approximately 33.9 kg. The drum was then certificated (see Table 1), designated as 'Drum A' and circulated to the participants as detailed in Section 5 below.

After Drum A had been circulated and returned to NPL, it was opened and the resin bottle was removed. The drum and its remaining contents were weighed. The second resin bottle was inserted (again in the second layer down but 'one bottle in' from the drum wall, the distance from the wall to the side of the bottle being approximately 7.4 cm) and the drum was reweighed. Again, double weighing was used. The drum was re-sealed, designated 'Drum B', certificated (see Table 2) and circulated as detailed below.

5. CIRCULATION OF DRUM AND REPORTING OF DATA

A mailshot was prepared (see Appendix A) and circulated to around 440 contacts in the nuclear industry. The mailshot gave full details of the exercise. Key points made in the mailshot were:

- There would be two drum types available (A and B), with activity concentrations in the following ranges:
 - Drum A: $0.10 - 0.35 \text{ Bq g}^{-1}$
 - Drum B: $5 - 20 \text{ Bq g}^{-1}$
- The radionuclides present in both drum types would be ^{241}Am , ^{60}Co and ^{137}Cs ;
- The drums would contain resin and vermiculite (as explained in Section 2 above);
- The number of active bottles present in each drum type (or their locations in the drums) would not be disclosed until after the deadline for submission of results. There would then be a 'second deadline', for submission of additional (but not replacement) results derived from refined efficiency models.

A total of 15 laboratories expressed interest in participating and it was possible to accommodate 14 of them. At one laboratory, the drums were measured by two groups, so the total number of groups was 15. The contacts are listed in Appendix B. There were two overseas participants (one in France and one in Italy).

Drum A was dispatched to the first participant; thereafter, it was moved directly from one UK participant to the next according to a pre-determined timetable with NPL organising the courier service each time. Each site was given approximately seven working days (excluding the delivery and pick-up days) in which to carry out their measurements. For logistic reasons, Drum A was returned to NPL prior to dispatch to the Italian laboratory, which was the final laboratory in the schedule.

Drum A was repacked (as detailed in Section 4 above) to produce Drum B. Drum B was similarly circulated to the participants 'in series'. Each participant was given 1 working day (excluding the delivery and pick-up days) to measure the drum and again the drum was returned to NPL prior to dispatch to the overseas participants.

A spreadsheet (see Appendix C) was sent to the participants for reporting of their data. Also, the participants were provided with the following details:

- A supplier's 'Certificate of Packaging Performance' for the drum;
- Details of the ion-exchange resin, the vermiculite and the bottles (obtained from the suppliers);
- The fill height in the active bottle(s) (approximately 10 cm);
- The mass of each sample (including the empty drum mass). These values were nominally:
 - Drum empty: 16.4 kg
 - Drum A full: 50.3 kg
 - Drum B full: 51.1 kg

6. RESULTS AND DATA ANALYSIS

To preserve anonymity, each participant was assigned a code between 1 and 15, and their results were coded accordingly. Some of the laboratories had also participated in the 2007 exercise and their codes were changed for the current exercise.

On receipt of the results, NPL carried out data analyses using the method described below [Harms, 2009(a)].

Firstly, the deviation from the assigned (NPL) value of each laboratory value was calculated, given by:

$$D = 100 \frac{L - N}{N} = 100 \left(\frac{L}{N} - 1 \right) \quad [1]$$

where:

D = deviation from the NPL value (%)

L = the participant's value (Bq g⁻¹)

N = the NPL value (Bq g⁻¹)

The deviations are given in Tables 3 – 14 and are plotted in Figures 1, 5, 9, 13, 17, 21, 25, 29, 33, 37, 41 and 45. The error bars in the graphs represent the standard uncertainty ($k=1$) of the deviation:

$$u_D = 100 \frac{L}{N} \sqrt{\left(\frac{u_L}{L}\right)^2 + \left(\frac{u_N}{N}\right)^2} \quad [2]$$

where:

u_D = standard uncertainty of the deviation (%)

u_L = standard uncertainty of the participant's value (Bq g^{-1})

u_N = standard uncertainty of the assigned value (Bq g^{-1})

The results were evaluated using three tests:

$$\zeta = \frac{L - N}{\sqrt{u_L^2 + u_N^2}} \quad [3]$$

$$R_L = \frac{u_L}{L} \quad [4]$$

$$z = \frac{L - N}{R_{\text{med}} N} \quad [5]$$

where:

ζ = zeta score

R_L = relative uncertainty of the participant's value

z = z-score

R_{med} = median of the values of R_L

The zeta and z-scores were used to determine whether the difference between the participant's value and the NPL value was significantly different from zero. An IQR (Inter-Quartile Range) outlier test was used to determine whether a particular R_L value was significantly larger than the other values in a data set; the IQR was used to calculate a limiting value of R_L , ' R_{lim} ', for each data set. The zeta scores, z-scores and outlier results are given in Tables 3 - 14. The zeta scores are plotted in Figures 2, 6, 10, 14, 18, 22, 26, 30, 34, 38, 42 and 46. The R_L values are plotted in Figures 3, 7, 11, 15, 19, 23, 27, 31, 35, 39, 42 and 47.

Results for which the absolute values of the zeta score and the z-score were both ≤ 2.576 (corresponding to a significance levels of $\alpha = 0.01$) and for which the relative uncertainty R_L was not significantly larger than the other values in the data set were regarded as being 'in agreement' with NPL. These are marked in dark blue in the deviation plots. If either (i) the relative uncertainty R_L was significantly larger than the other values in the data set, (ii) the result passes the zeta test but not the z-test (i.e. large deviation from the NPL value combined with a large uncertainty), or (iii) the result passes the z-test but not the zeta test (small deviation from the NPL value combined with a small uncertainty), the participant's value is classified as 'questionable' (these are given in yellow in the deviation plots). If the absolute values of both the zeta score and the z-score are > 2.576 , then the participant's value is

classified as ‘discrepant’ from the NPL value (red points), regardless of the value of the relative uncertainty R_L . The findings for all the data are given in Tables 3 - 14.

Now, the zeta score and the z-score are related by:

$$\zeta = \frac{\sigma_p}{\sqrt{u_L^2 + u_N^2}} z \quad [6]$$

(where $\sigma_p = R_{\text{med}}N$, the standard uncertainty for proficiency assessment (Bq g^{-1})).

This can be rewritten as:

$$\frac{z^2}{\zeta^2} - \frac{u_N^2}{\sigma_p^2} = \frac{u_L^2}{\sigma_p^2} \quad [7]$$

The relative uncertainty of the laboratory R_L and the z-score are related by:

$$\frac{u_L}{R_L} = z\sigma_p + N \quad [8]$$

This can be rewritten as:

$$R_L^2 \left\{ z + \frac{N}{\sigma_p} \right\}^2 = \frac{u_L^2}{\sigma_p^2} \quad [9]$$

Consequently, comparison data can be neatly summarised by plotting the square of the ratio of u_L to σ_p against the z-score, and superimposing one parabola to represent a zeta score of 2.576, a second parabola representing the outlier limit R_{lim} of the relative laboratory uncertainty R_L , and vertical lines representing a z-score of 2.576. These are known as ‘Kiri’ plots [Harms, 2009(b)], and are given in Figures 4, 8, 12, 16, 20, 24, 28, 32, 36, 40, 44 and 48. Data points that are inside the zeta parabola (i.e., for which $\text{zeta} \leq 2.576$), within the z-score lines and outside the R_{lim} parabola (i.e., for which $R_L \leq R_{\text{lim}}$) are ‘in agreement’ with NPL. Data points which fail either the z-test, the zeta test or the relative uncertainty outlier test (but not both the z-test and zeta test), are classified as ‘questionable’ and are either:

- (i) inside the zeta parabola with a z-score < -2.576 or > 2.576 , or
- (ii) outside the zeta parabola with a z-score > -2.576 or < 2.576 ; or
- (iii) inside zeta parabola with a z-score > -2.576 or < 2.576 , but inside the R_{lim} parabola.

All other data points are classified as ‘discrepant’.

The deviations from the NPL value are also plotted in participant order (see Figures 49 - 75).

Finally, the NPL assigned value for each radionuclide in each sample was compared with the weighted mean of the Largest Consistent Subset (LCS) of the participants’ results. This method is based on a paper by Cox (Cox, 2007). The results are summarised in Table 15.

7. DISCUSSION

This discussion takes into account discussions from the post-comparison workshop. Minutes of the workshop are given in Appendix D.

Ten participants received Drum A, and all ten reported data. Six of these reported additional data after the hot volume details had been disclosed. Fifteen participants received Drum B, and all submitted data. Five of these also reported data 'post-disclosure'. Ten of the participants who measured Drum B had also measured Drum A.

n.b. For convenience, the terms 'pre-disclosure' and 'post-disclosure' are used in the discussion and conclusions to differentiate between results submitted before and after disclosure of the size and location of the 'hot volume' in each sample.

Some participants reported results for more than one detector, or reported more than one result for one detector (e.g. by using different efficiency models). Apart from one participant (who used a plastic scintillator), all performed γ -spectrometry to measure the drums. Of these, seven used 'open geometry' hyperpure Ge crystals, four used Segmented Gamma Scanners, a further two used both methods, and two carried out low-resolution gamma spectrometry using three-crystal (NaI(Tl)) detector systems.

For γ -spectrometry, source-to-detector distances varied from 25 to 200 cm but were typically 50 to 60 cm.

A variety of detector efficiency models and calibration standards was used. Four participants used ISOCS (adopting a variety of templates), two used GENIE 2000 / NDA 2000, one used ISOTOPIC, one used SNAP and one used Winner/Track software. Four participants derived calibrations using standard drums prepared 'in house' and one participant used 'point source' calibrations. A further four participants used the manufacturers' calibrations.

Six participants used standard drums of various kinds to validate calibrations.

Four participants used γ -probes to locate the 'hot volume' prior to measurement.

The results of the exercise were presented at the post-exercise workshop. It became apparent that NPL had not included all the data submitted by Participant 9. However, all their data are included in Sections 11 and 12, and are discussed below.

7.1 Americium-241

Measurement of the ^{241}Am component is difficult because the attenuation within the drum wall is much greater than for the other radionuclides present. Small differences between the actual composition of the drums and their composition assumed in efficiency models will also be much more important.

7.1.1 Americium-241 in Drum A

Pre-disclosure:

Fourteen results were submitted for Drum A pre-disclosure. Eleven were 'in agreement' with the NPL assigned value, but the results from Participants 2, 4 and 8 were 'questionable'.

Participant 2 used ISOCS to model the detection efficiency (using a 'complex pipe' template), and it is interesting to compare the results of users of this software. Participant 9 was another

ISOCS user. They submitted six results, coded 9A to 9F. Results 9A to 9C were obtained using three different HPGe detectors, all assuming a uniform distribution of activity within the drum. Results 9D to 9F were derived using the same three crystals but using a ‘sphere within a cylinder’ ISOCS model template and using a γ -probe to locate the hotspot and to estimate uncertainties on model parameters. Although results 9D to 9F looked higher than 9A to 9C, they were all in agreement with NPL. Participant 3 used ISOCS to model a simple cylinder (assuming a uniform distribution of activity), and they also agreed with NPL. Participant 5 (result 5A) also used ISOCS. They provided only limited details of their method, but they too agreed with NPL.

Post-disclosure:

Eleven results were submitted for Drum A post-disclosure; 5 were in agreement with NPL, 4 were questionable and 2 were ‘discrepant’.

This time, Participant 9 submitted 7 results. Results 9A-9C were derived using the same three detectors as above but refining the model using the hot-volume details provided by NPL. Results 9D and 9E came from two of those detectors but without rotation of the drum in either case. Results 9F and 9G likewise came from two of the detectors (without drum rotation), but here they vertically aligned the detector to be in line with the hot volume location, and this resulted in a questionable result in both cases. The result from Participant 2 was again questionable. At the workshop they revealed this was because they needed a value for the density of the dry ion-exchange resin for their ISOCS efficiency model and NPL had not provided this information. After the workshop, NPL provided mass data for the resin bottle to all participants, and Participant 2 submitted an additional ^{241}Am result of $(0.119 \pm 0.016) \text{ Bq g}^{-1}$, in agreement with NPL. Curiously, Participant 3 was now discrepant, having been in agreement pre-disclosure. They had changed their efficiency model to a ‘pipe’ template.

Participant 1 (using ISOTOPIC software) had changed their efficiency model to one based on a single small volume within a cylinder, and this also resulted in a discrepant result.

7.1.2 Americium-241 in Drum B

Pre-disclosure:

Eighteen results were submitted for Drum B pre-disclosure, of which 12 were in agreement with NPL. The data followed a similar pattern to the Drum A data, but with a few large deviations, notably Participants 12 and 15 (+111 % and +134 % respectively). There is no obvious reason for these deviations, although note that both used SGSs (see also 7.2.1 below).

Turning to the ISOCS users, Participant 2 again submitted a high result but, as explained above, amended their model after the workshop and arrived at a value of $(4.27 \pm 0.52) \text{ Bq g}^{-1}$, in agreement with NPL. Participant 9 submitted 6 results, 9A to 9F. Results 9A – 9C were from the same detectors as used for Drum A (again assuming a uniform activity distribution) and results 9D – 9F again were derived from using a γ -probe and ‘sphere within a cylinder’ modelling. All six results were in agreement with NPL. Note Participant 3 was now discrepant, but Participant 5 (result 5A) was in agreement.

Note that the weighted mean of the Largest Consistent Subset (LCS) of the participants’ results did not agree with the NPL value.

Post-disclosure:

Nine results were submitted for Drum B post-disclosure, and the outcome was very similar to that observed for Drum A. This time, Participant 9 submitted 5 results; 9A to 9C were from the same detectors (but with different source-to-detector distances) as for Drum A, whereas 9D and 9E resulted from vertical alignment of hot volume and detector and with no drum rotation.

In this case, the weighted mean of the LCS of the results did agree with NPL.

7.2 Cobalt-60

7.2.1 Cobalt-60 in Drum A

Pre-disclosure:

Twelve out of the 18 results submitted for this radionuclide pre-disclosure were in agreement with the NPL assigned value. The three lowest values submitted (coded 5B, 7A and 10A) were all discrepant and it is worth noting that these were all from SGS detectors. However, in the case of result 7A, note that the instrument used to obtain the result is not normally configured to measure these γ -energies; moreover, the quoted uncertainty was due to counting statistics only.

It is worth noting that, in the 2007 exercise, a ‘low’ result from an SGS measurement of ^{60}Co had been attributed to the low activity present; in some segments, only one of the two main gamma peaks had been identified and the software had been set to discount these activities in the absence of a confirming peak. It is possible in the current exercise that the active bottle in Drum A had been ‘split’ between adjacent segments in some cases and a similar effect had occurred.

Post-disclosure:

Post-disclosure, 8 out of 13 results were in agreement with NPL. Participants 1 and 3 were again discrepant, as were the two results from Participant 2; they later submitted additional results of $(0.116 \pm 0.009) \text{ Bq g}^{-1}$ (1173 keV) and $(0.114 \pm 0.009) \text{ Bq g}^{-1}$ (1333 keV), both in agreement with NPL. Participant 10’s result 10B was discrepant, but note they had used a plastic scintillator detector and that in normal operation it would be used not to determine individual radionuclides but would attribute all detectable γ -pulses to a single radionuclide within a waste fingerprint.

Unlike the pre-disclosure dataset, the weighted mean of the LCS of the results did not agree with NPL.

7.2.2 Cobalt-60 in Drum B

Pre-disclosure:

Twelve out of the 23 results submitted were in agreement with NPL and the pattern was similar to Drum A. Curiously, result 10A was now higher than the NPL assigned value (having been lower for Drum A). Participants 12 and 15 submitted discrepant (low) and questionable (high) results respectively, both using SGSs.

Participant 2's results were again discrepant but they later submitted additional results of $(2.388 \pm 0.190) \text{ Bq g}^{-1}$ (1173 keV) and $(2.273 \pm 0.180) \text{ Bq g}^{-1}$ (1332 keV), in agreement with NPL.

Post-disclosure:

Post-disclosure, 7 out of 9 results were in agreement with NPL. Note that Participant 1 was now in agreement. As for Drum A, the weighted mean of the LCS of the results did not agree with NPL.

7.3 Caesium-137

7.3.1 Caesium-137 in Drum A

Pre-disclosure:

Fifteen out of the 17 results submitted for ^{137}Cs pre-disclosure were in agreement with the NPL assigned value.

Post-disclosure:

Strangely, the dataset post-disclosure was less good, with only 7 out of 12 results being in agreement. The result 10B (obtained using a plastic scintillator) was highly discrepant, with a positive deviation of over 200 %. Again, Participant 2 added a revised value after the workshop of $(0.113 \pm 0.01) \text{ Bq g}^{-1}$, in agreement with NPL. Note that Participant 3 was again discrepant.

The weighted mean of the LCS of the results did not agree with NPL.

7.3.2 Caesium-137 in Drum B

Pre-disclosure:

The dataset for Drum B pre-disclosure was less good than for Drum A, with only 11 of the 22 results being in agreement with NPL. The results actually show a very similar pattern to the data for ^{60}Co in the same drum. The revised value from Participant 2 was $(2.945 \pm 0.52) \text{ Bq g}^{-1}$, in agreement with NPL.

Participants 13 and 14 both used three-crystal NaI(Tl) detectors and obtained questionable results.

Post-disclosure:

After disclosure of the hot volume, 8 out of 9 results agreed with NPL.

8. CONCLUSIONS

In the final dataset for Drum A, 78 % of the data submitted prior to disclosure of the size and location of the 'hot volume' were in agreement with the NPL assigned value, whereas only 56 % of the data submitted after disclosure were in agreement. The post-disclosure data came

from a subset of the participants who had submitted data pre-disclosure, and 76 % of the subset's pre-disclosure data was in agreement with NPL.

The reduction in overall performance post-disclosure was partly due to some results being less accurate after efficiency models had been revised. Note also that one participant submitted no data pre-disclosure but did submit discrepant data post-disclosure, which slightly 'worsened' the overall dataset.

For Drum B, the overall performance figures were:

- Percentage pre-disclosure data in agreement with NPL = 56 %
- Percentage post-disclosure data in agreement with NPL = 74 %
- Percentage pre-disclosure data in agreement with NPL (post-disclosure participants only) = 77 %

In this case, there was some overall improvement in performance for the subset of participants who had submitted data both pre- and post-disclosure, but also there were problems with the results from some of the other participants. Some SGS measurements did not agree with NPL, and 3 of the 4 results submitted using triple NaI(Tl) detectors were questionable.

The comparison (for the various datasets) of the weighted mean of the LCS of the results with the NPL value gave variable results. It was difficult to identify any trends because of the small number of datapoints in the datasets.

A key finding of the exercise (as in the 2007 exercise) was that the participants who had used ISOCS had obtained different results, some in agreement with NPL and others not. It may be significant that these participants had used different model templates and had made various assumptions about the drums and their contents. These results underline the importance of accurate input data in obtaining accurate assay results with modelling software, and there is perhaps a need for additional guidance in the use of software of this type.

Some participants who had used Segmented Gamma Scanners obtained discrepant data. These deviations may have been due to other causes, but possibly the highly heterogeneous nature of the samples was a factor.

NPL is happy to advise participants with outstanding discrepancies on how to resolve them.

It is worth noting that the two drums in this exercise were provided to the participants with a great deal of information on the drums themselves and their contents. This amount of information may often not be available for 'real' samples. It would be very interesting to run a comparison using a drum provided with 'minimal' information – better still, one containing 'real' items - and this was suggested by some of the participants at the post-exercise workshop.

The exercise had been useful to the participants in enabling them to test their efficiency models and/or measurement systems. Another comparison of this type is scheduled for 2011.

9. ACKNOWLEDGEMENTS

The author wishes to thank:

The participating organisations for the time and effort they have put into analysing the drums and contributing to the workshop, and for the information they have provided;

Dr A V Harms for his advice on data analysis and for checking this report;

Mr A Stroak, Mrs L J Keightley, Mr A Fenwick and Mr A Pearce for helping prepare and certificate the drums;

Miss J Wong for organising the transport of the drums.

Finally, the author gratefully acknowledges the financial support of the National Measurement System.

10. REFERENCES

Cox, M.G. The Evaluation of Key Comparison Data: Determining the Largest Consistent Subset. *Metrologia*, 44, 187-200, 2007.

Dean, J.C.J. A Comparison of Procedures Used at UK Nuclear Sites for Gamma Assays of Potentially Contaminated or Activated Materials. NPL Report IR2, 2007.

Harms, A.V. A New Approach for Proficiency Test Exercise Data Evaluation. *Accreditation and Quality Assurance*, 14, 253-261, 2009(a).

Harms, A.V. Visualisation of Proficiency Test Exercise Results in Kiri plots. *Accreditation and Quality Assurance*, 14, 307-311, 2009(b).

NCRP Report 58. Fundamental or Direct Measurement of Activity in Radioactive Decay. A Handbook of Radioactivity Measurements. NCRP, Bethesda, National Council on Radiation Protection and Measurements, 1985 (2nd edition), 74-139.

11. TABLES

Table 1 - Principal radionuclides in Drum A

Radionuclide	Decay mode	Activity concentration (averaged across contents of drum) @ 1200 UTC* 01/03/09 (Bq g ⁻¹)	Standard uncertainty ($k=1$) (Bq g ⁻¹)	Standard uncertainty ($k=1$) (%)
²⁴¹ Am	α/γ	0.1144	0.0007	0.62
⁶⁰ Co	β^-/γ	0.1127	0.0005	0.39
¹³⁷ Cs	β^- , β^-/γ	0.1085	0.0008	0.74

Table 2 - Principal radionuclides in Drum B

Radionuclide	Decay mode	Activity concentration (averaged across contents of drum) @ 1200 UTC 01/03/09 (Bq g ⁻¹)	Standard uncertainty ($k=1$) (Bq g ⁻¹)	Standard uncertainty ($k=1$) (%)
²⁴¹ Am	α/γ	4.022	0.017	0.43
⁶⁰ Co	β^-/γ	2.428	0.006	0.23
¹³⁷ Cs	β^- , β^-/γ	2.888	0.020	0.70

* Universal Time, Co-ordinated. This replaced Greenwich Mean Time in 1972.

Table 3 – Reported results for ^{241}Am for Drum A with ‘hot volume’ not knownNPL activity concentration = $(0.1144 \pm 0.0007) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{241}Am activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	0.134	0.029	17.13	0.87	0.68	Not outlier	In agreement
2	0.176	0.026	53.85	2.73	2.37	Not outlier	Questionable
3	0.07607	0.01499	-33.51	-1.70	-2.55	Not outlier	In agreement
4	0.192	27.32 %	67.83	3.44	1.48	Not outlier	Questionable
5A	0.109	19.4 %	-4.72	-0.24	-0.26	Not outlier	In agreement
6	0.108	54 %	-5.59	-0.28	-0.11	Not outlier	In agreement
7B	0.139	0.022	21.50	1.09	1.12	Not outlier	In agreement
8	0.087	0.01	-23.95	-1.22	-2.73	Not outlier	Questionable
9A	0.106	0.021	-7.34	-0.37	-0.40	Not outlier	In agreement
9B	0.113	0.023	-1.22	-0.06	-0.06	Not outlier	In agreement
9C	0.099	0.021	-13.46	-0.68	-0.73	Not outlier	In agreement
9D	0.134	0.032	17.13	0.87	0.61	Not outlier	In agreement
9E	0.15	0.036	31.12	1.58	0.99	Not outlier	In agreement
9F	0.145	0.038	26.75	1.36	0.81	Not outlier	In agreement

Table 4 – Reported results for ^{241}Am for Drum A with ‘hot volume’ knownNPL activity concentration = $(0.1144 \pm 0.0007) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{241}Am activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	0.058	0.005	-49.30	-3.54	-11.17	Not outlier	Discrepant
2	0.165	0.023	44.23	3.17	2.20	Not outlier	Questionable
3	0.06301	0.01273	-44.92	-3.22	-4.03	Not outlier	Discrepant
8	0.087	0.01	-23.95	-1.72	-2.73	Not outlier	Questionable
9A	0.133	0.034	16.26	1.17	0.55	Not outlier	In agreement
9B	0.13	0.033	13.64	0.98	0.47	Not outlier	In agreement
9C	0.119	0.027	4.02	0.29	0.17	Not outlier	In agreement
9D	0.149	0.037	30.24	2.17	0.93	Not outlier	In agreement
9E	0.133	0.032	16.26	1.17	0.58	Not outlier	In agreement
9F	0.162	0.04	41.61	2.98	1.19	Not outlier	Questionable
9G	0.167	0.056	45.98	3.30	0.94	Not outlier	Questionable

Table 5 – Reported results for ^{60}Co for Drum A for with ‘hot volume’ not knownNPL activity concentration = $(0.1127 \pm 0.0005) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{60}Co activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	0.132	0.0164	17.13	1.91	1.18	Not outlier	In agreement
2A	0.161	0.015	42.86	4.78	3.22	Not outlier	Discrepant
2B	0.157	0.014	39.31	4.38	3.16	Not outlier	Discrepant
3	0.09629	0.004737	-14.56	-1.62	-3.45	Not outlier	Questionable
4	0.090	17.94 %	-20.14	-2.24	-1.41	Not outlier	In agreement
5A	0.114	4.3 %	1.15	0.13	0.26	Not outlier	In agreement
5B	0.0831	2.8 %	-26.26	-2.93	-12.44	Not outlier	Discrepant
6	0.114	22 %	1.15	0.13	0.05	Not outlier	In agreement
7A	0.0804	0.0071	-28.66	-3.19	-4.54	Not outlier	Discrepant
7B	0.122	0.007	8.25	0.92	1.33	Not outlier	In agreement
8	0.11	0.007	-2.40	-0.27	-0.38	Not outlier	In agreement
9A	0.107	0.015	-5.06	-0.56	-0.38	Not outlier	In agreement
9B	0.112	0.016	-0.62	-0.07	-0.04	Not outlier	In agreement
9C	0.108	0.015	-4.17	-0.46	-0.31	Not outlier	In agreement
9D	0.117	0.017	3.82	0.43	0.25	Not outlier	In agreement
9E	0.127	0.019	12.69	1.41	0.75	Not outlier	In agreement
9F	0.135	0.021	19.79	2.20	1.06	Not outlier	In agreement
10A	0.069	15.8 %	-38.78	-4.32	-4.00	Not outlier	Discrepant

Table 6 – Reported results for ^{60}Co for Drum A with ‘hot volume’ knownNPL activity concentration = $(0.1127 \pm 0.0005) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{60}Co activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	0.140	0.009	24.22	3.26	3.03	Not outlier	Discrepant
2A	0.154	0.014	36.65	4.94	2.95	Not outlier	Discrepant
2B	0.151	0.013	33.98	4.58	2.94	Not outlier	Discrepant
3	0.08320	0.004152	-26.18	-3.53	-7.05	Not outlier	Discrepant
8	0.11	0.012	-2.40	-0.32	-0.22	Not outlier	In agreement
9A	0.116	0.019	2.93	0.39	0.17	Not outlier	In agreement
9B	0.115	0.017	2.04	0.27	0.14	Not outlier	In agreement
9C	0.111	0.016	-1.51	-0.20	-0.11	Not outlier	In agreement
9D	0.119	0.018	5.59	0.75	0.35	Not outlier	In agreement
9E	0.111	0.017	-1.51	-0.20	-0.10	Not outlier	In agreement
9F	0.133	0.02	18.01	2.43	1.01	Not outlier	In agreement
9G	0.134	0.025	18.90	2.55	0.85	Not outlier	In agreement
10B	0.141	4 %	25.11	3.38	5.00	Not outlier	Discrepant

Table 7 – Reported results for ^{137}Cs for Drum A for with ‘hot volume’ not knownNPL activity concentration = $(0.1085 \pm 0.0008) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{137}Cs activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	0.121	0.015	11.52	1.00	0.83	Not outlier	In agreement
2	0.159	0.017	46.54	4.03	2.97	Not outlier	Discrepant
3	0.09047	0.006206	-16.62	-1.44	-2.88	Not outlier	Questionable
4	0.091	18.88 %	-16.13	-1.40	-1.17	Not outlier	In agreement
5A	0.128	6.3 %	17.97	1.56	1.15	Not outlier	In agreement
5B	0.0953	4.1 %	-12.17	-1.05	-2.11	Not outlier	In agreement
6	0.114	25 %	5.07	0.44	0.32	Not outlier	In agreement
7A	0.0932	0.0133	-14.10	-1.22	-1.15	Not outlier	In agreement
7B	0.107	0.006	-1.38	-0.12	-0.25	Not outlier	In agreement
8	0.107	0.007	-1.38	-0.12	-0.21	Not outlier	In agreement
9A	0.099	0.021	-8.76	-0.76	-0.45	Not outlier	In agreement
9B	0.107	0.022	-1.38	-0.12	-0.07	Not outlier	In agreement
9C	0.102	0.021	-5.99	-0.52	-0.31	Not outlier	In agreement
9D	0.117	0.025	7.83	0.68	0.34	Not outlier	In agreement
9E	0.129	0.027	18.89	1.64	0.76	Not outlier	In agreement
9F	0.135	0.031	24.42	2.12	0.85	Not outlier	In agreement
10A	0.094	29.1%	-13.36	-1.16	-0.53	Not outlier	In agreement

Table 8 – Reported results for ^{137}Cs for Drum A with ‘hot volume’ knownNPL activity concentration = $(0.1085 \pm 0.0008) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{137}Cs activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	0.131	0.008	20.74	2.57	2.80	Not outlier	Questionable
2	0.152	0.014	40.09	4.96	3.10	Not outlier	Discrepant
3	0.07658	0.005322	-29.42	-3.64	-5.93	Not outlier	Discrepant
8	0.107	0.015	-1.38	-0.17	-0.10	Not outlier	In agreement
9A	0.113	0.026	4.15	0.51	0.17	Not outlier	In agreement
9B	0.115	0.024	5.99	0.74	0.27	Not outlier	In agreement
9C	0.11	0.022	1.38	0.17	0.07	Not outlier	In agreement
9D	0.118	0.025	8.76	1.08	0.38	Not outlier	In agreement
9E	0.118	0.025	8.76	1.08	0.38	Not outlier	In agreement
9F	0.129	0.028	18.89	2.34	0.73	Not outlier	In agreement
9G	0.133	0.035	22.58	2.79	0.70	Not outlier	Questionable
10B	0.333	3 %	206.91	25.61	22.40	Not outlier	Discrepant

Table 9 – Reported results for ^{241}Am for Drum B with ‘hot volume’ not knownNPL activity concentration = $(4.022 \pm 0.017) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{241}Am activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	4.5	0.9	11.88	0.71	0.53	Not outlier	In agreement
2	7.344	1.232	82.60	4.92	2.70	Not outlier	Discrepant
3	2.16	0.4735	-46.30	-2.76	-3.93	Not outlier	Discrepant
4	4.767	25.1 %	18.52	1.10	0.62	Not outlier	In agreement
5A	3.39	9.5 %	-15.71	-0.94	-1.96	Not outlier	In agreement
5B	1.90	5.4 %	-52.76	-3.15	-20.40	Not outlier	Discrepant
6	2.96	47 %	-26.40	-1.57	-0.76	Not outlier	In agreement
7B	5.14	0.74	27.80	1.66	1.51	Not outlier	In agreement
8	2.48	0.15	-38.34	-2.29	-10.21	Not outlier	Questionable
9A	3.002	0.602	-25.36	-1.51	-1.69	Not outlier	In agreement
9B	3.245	0.652	-19.32	-1.15	-1.19	Not outlier	In agreement
9C	2.851	0.572	-29.11	-1.74	-2.05	Not outlier	In agreement
9D	4.358	0.956	8.35	0.50	0.35	Not outlier	In agreement
9E	4.644	1.02	15.46	0.92	0.61	Not outlier	In agreement
9F	4.091	0.866	1.72	0.10	0.08	Not outlier	In agreement
11	3.51	0.34	-12.73	-0.76	-1.50	Not outlier	In agreement
12	8.50	10.6 %	111.34	6.64	4.97	Not outlier	Discrepant
15	9.43	19.1 %	134.46	8.02	3.00	Not outlier	Discrepant

Table 10 – Reported results for ^{241}Am for Drum B with ‘hot volume’ knownNPL activity concentration = $(4.022 \pm 0.017) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{241}Am activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	1.63	0.12	-59.47	-6.09	-19.74	Not outlier	Discrepant
3	2.287	0.4441	-43.14	-4.42	-3.90	Not outlier	Discrepant
8	2.48	0.16	-38.34	-3.93	-9.58	Not outlier	Discrepant
9A	4.271	0.946	6.19	0.63	0.26	Not outlier	In agreement
9B	4.413	0.943	9.72	1.00	0.41	Not outlier	In agreement
9C	4.059	0.883	0.92	0.09	0.04	Not outlier	In agreement
9D	5.294	1.184	31.63	3.24	1.07	Not outlier	Questionable
9E	4.759	1.141	18.32	1.88	0.65	Not outlier	In agreement
11	3.38	0.33	-15.96	-1.63	-1.94	Not outlier	In agreement

Table 11 – Reported results for ^{60}Co for Drum B with ‘hot volume’ not knownNPL activity concentration = $(2.428 \pm 0.006) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{60}Co activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	3.2	0.4	31.80	5.43	1.93	Not outlier	Questionable
2A	3.355	0.266	38.18	6.52	3.48	Not outlier	Discrepant
2B	3.177	0.252	30.85	5.27	2.97	Not outlier	Discrepant
3	1.84	0.1925	-24.22	-4.14	-3.05	Not outlier	Discrepant
4	1.821	17.15 %	-25.00	-4.27	-1.94	Not outlier	Questionable
5A	2.47	2.0 %	1.73	0.30	0.84	Not outlier	In agreement
5B	2.06	0.9 %	-15.16	-2.59	-18.88	Not outlier	Discrepant
6	2.35	21 %	-3.21	-0.55	-0.16	Not outlier	In agreement
7A	1.718	0.062	-29.24	-4.99	-11.40	Not outlier	Discrepant
7B	2.54	0.11	4.61	0.79	1.02	Not outlier	In agreement
8	2.22	0.13	-8.57	-1.46	-1.60	Not outlier	In agreement
9A	2.079	0.294	-14.37	-2.45	-1.19	Not outlier	In agreement
9B	2.129	0.302	-12.31	-2.10	-0.99	Not outlier	In agreement
9C	2.122	0.3	-12.60	-2.15	-1.02	Not outlier	In agreement
9D	2.449	0.348	0.86	0.15	0.06	Not outlier	In agreement
9E	2.505	0.36	3.17	0.54	0.21	Not outlier	In agreement
9F	2.483	0.357	2.27	0.39	0.15	Not outlier	In agreement
10A	2.9	2.6 %	19.44	3.32	6.24	Not outlier	Discrepant
11	2.38	0.13	-1.98	-0.34	-0.37	Not outlier	In agreement
12	1.56	1.4 %	-35.75	-6.10	-38.32	Not outlier	Discrepant
13	2.82	0.33	16.14	2.76	1.19	Not outlier	Questionable
14	2.70	0.42	11.20	1.91	0.65	Not outlier	In agreement
15	2.73	1.90 %	12.44	2.12	5.78	Not outlier	Questionable

Table 12 – Reported results for ^{60}Co for Drum B with ‘hot volume’ knownNPL activity concentration = $(2.428 \pm 0.006) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{60}Co activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	2.9	0.2	19.44	2.46	2.36	Not outlier	In agreement
3	1.788	0.1413	-26.36	-3.34	-4.53	Not outlier	Discrepant
8	2.22	0.24	-8.57	-1.08	-0.87	Not outlier	In agreement
9A	2.422	0.343	-0.25	-0.03	-0.02	Not outlier	In agreement
9B	2.499	0.355	2.92	0.37	0.20	Not outlier	In agreement
9C	2.482	0.353	2.22	0.28	0.15	Not outlier	In agreement
9D	2.777	0.406	14.37	1.82	0.86	Not outlier	In agreement
9E	2.698	0.396	11.12	1.41	0.68	Not outlier	In agreement
11	1.97	0.11	-18.86	-2.39	-4.16	Not outlier	Questionable

Table 13 – Reported results for ^{137}Cs for Drum B with ‘hot volume’ not knownNPL activity concentration = $(2.888 \pm 0.020) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{137}Cs activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	3.6	0.4	24.65	3.93	1.78	Not outlier	Questionable
2	4.257	0.42	47.40	7.56	3.26	Not outlier	Discrepant
3	2.32	0.283	-19.67	-3.14	-2.00	Not outlier	Questionable
4	2.225	20 %	-22.96	-3.66	-1.66	Not outlier	Questionable
5A	2.84	4.5 %	-1.66	-0.27	-0.11	Not outlier	In agreement
5B	2.84	1.8 %	-1.66	-0.27	-0.17	Not outlier	In agreement
6	2.70	24 %	-6.51	-1.04	-0.46	Not outlier	In agreement
7A	2.242	0.081	-22.37	-3.57	-7.74	Not outlier	Discrepant
7B	2.91	0.13	0.76	0.12	0.17	Not outlier	In agreement
8	2.67	0.16	-7.55	-1.20	-1.35	Not outlier	In agreement
9A	2.421	0.485	-16.17	-2.58	-0.96	Not outlier	Questionable
9B	2.594	0.519	-10.18	-1.62	-0.57	Not outlier	In agreement
9C	2.425	0.485	-16.03	-2.56	-0.95	Not outlier	In agreement
9D	2.986	0.603	3.39	0.54	0.16	Not outlier	In agreement
9E	3.213	0.659	11.25	1.79	0.49	Not outlier	In agreement
9F	2.98	0.614	3.19	0.51	0.15	Not outlier	In agreement
10A	3.55	3.6 %	22.92	3.65	5.12	Not outlier	Discrepant
11	2.87	0.18	-0.62	-0.10	-0.10	Not outlier	In agreement
12	1.94	2.1 %	-32.83	-5.23	-20.89	Not outlier	Discrepant
13	3.6	0.33	24.65	3.93	2.15	Not outlier	Questionable
14	3.39	0.22	17.38	2.77	2.27	Not outlier	Questionable
15	3.14	2.20 %	8.73	1.39	3.50	Not outlier	Questionable

Table 14 – Reported results for ^{137}Cs for Drum B with ‘hot volume’ knownNPL activity concentration = $(2.888 \pm 0.020) \text{ Bq g}^{-1}$ ($k=1$)

Reference time = 1200 UTC 1 March 2009

Participant code	Reported ^{137}Cs activity concentration (Bq g^{-1})	Reported uncertainty ($k=1$) (Bq g^{-1} , unless indicated)	Deviation from NPL value (%)	Z score	Zeta score	Result of outlier test	Conclusion
1	3.3	0.2	14.27	1.48	2.05	Not outlier	In agreement
3	2.280	0.2205	-21.05	-2.18	-2.75	Not outlier	Questionable
8	2.67	0.37	-7.55	-0.78	-0.59	Not outlier	In agreement
9A	2.944	0.592	1.94	0.20	0.09	Not outlier	In agreement
9B	3.172	0.638	9.83	1.02	0.44	Not outlier	In agreement
9C	2.951	0.596	2.18	0.23	0.11	Not outlier	In agreement
9D	3.537	0.737	22.47	2.32	0.88	Not outlier	In agreement
9E	3.356	0.703	16.20	1.68	0.67	Not outlier	In agreement
11	2.57	0.16	-11.01	-1.14	-1.97	Not outlier	In agreement

Table 15 – Comparison of NPL Assigned Values with Largest Consistent Subset of participants' results
(‘D’ denotes discrepant outcome)

Radionuclide	Assigned value (Bq g ⁻¹)	WM LCS (Bq g ⁻¹)	Size of the LCS (%)	Zeta score	Critical value
Drum A					
²⁴¹ Am	0.1144(7)	0.106(6)	100	−1.43	3.01
⁶⁰ Co	0.1127(5)	0.118(3)	78	1.89	2.88
¹³⁷ Cs	0.1085(8)	0.103(3)	100	−1.87	2.88
Drum AH					
²⁴¹ Am	0.1144(7)	0.111(8)	82	−0.39	3.17
⁶⁰ Co	0.1127(5)	0.134(4)	92	6.04 D	3.05
¹³⁷ Cs	0.1085(8)	0.127(6)	83	3.47 D	3.11
Drum B					
²⁴¹ Am	4.022(17)	3.49(15)	78	−3.43 D	2.90
⁶⁰ Co	2.428(6)	2.41(4)	78	−0.58	2.81
¹³⁷ Cs	2.888(20)	3.00(5)	86	2.19	2.76
Drum BH					
²⁴¹ Am	4.022(17)	3.38(23)	78	−2.83	3.36
⁶⁰ Co	2.428(6)	2.06(7)	89	−5.01 D	3.36
¹³⁷ Cs	2.888(20)	2.76(10)	100	−1.25	3.36

12. FIGURES

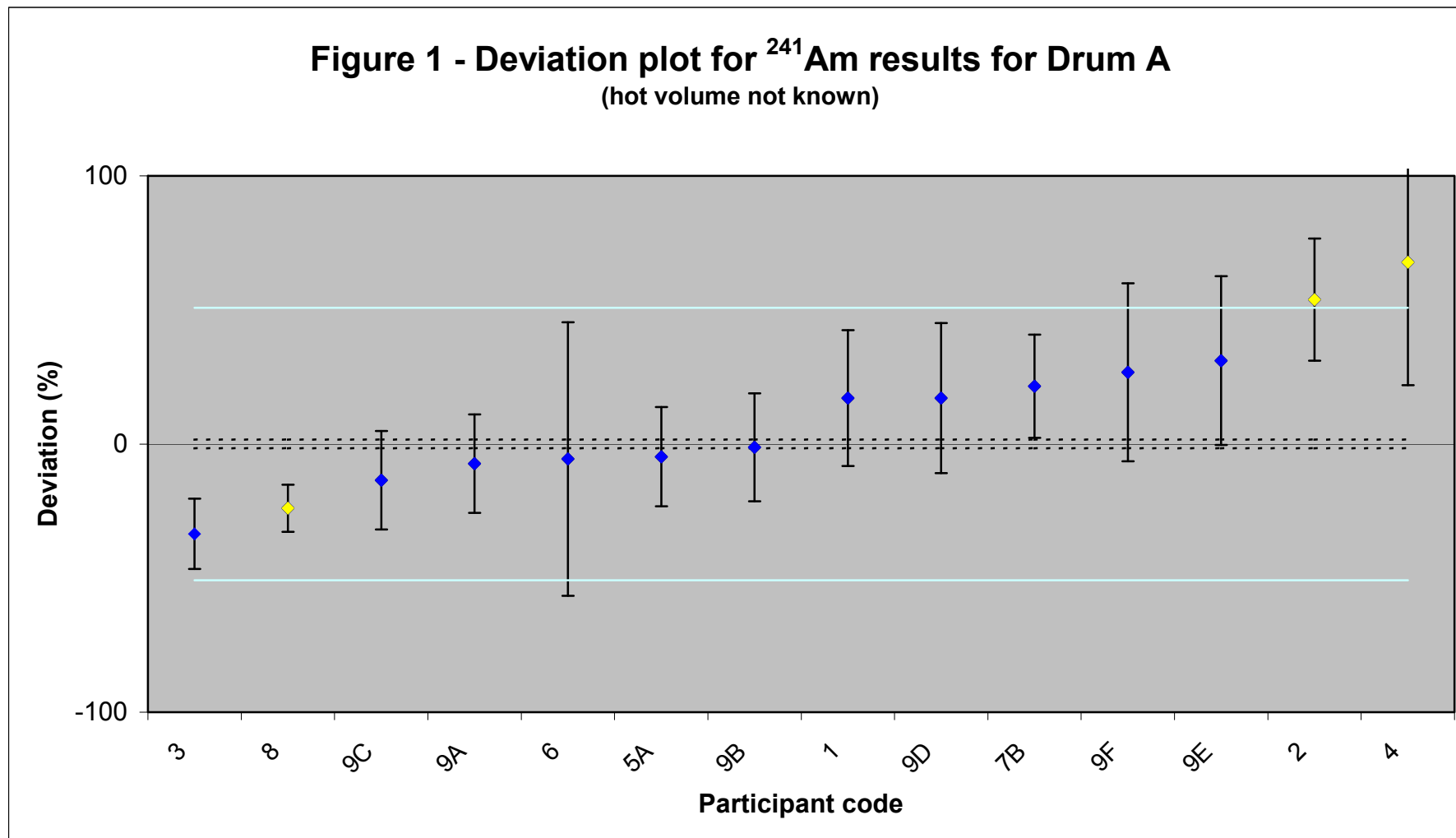


Figure 2 - Zeta-score plot for ^{241}Am results for Drum A
(hot volume not known)

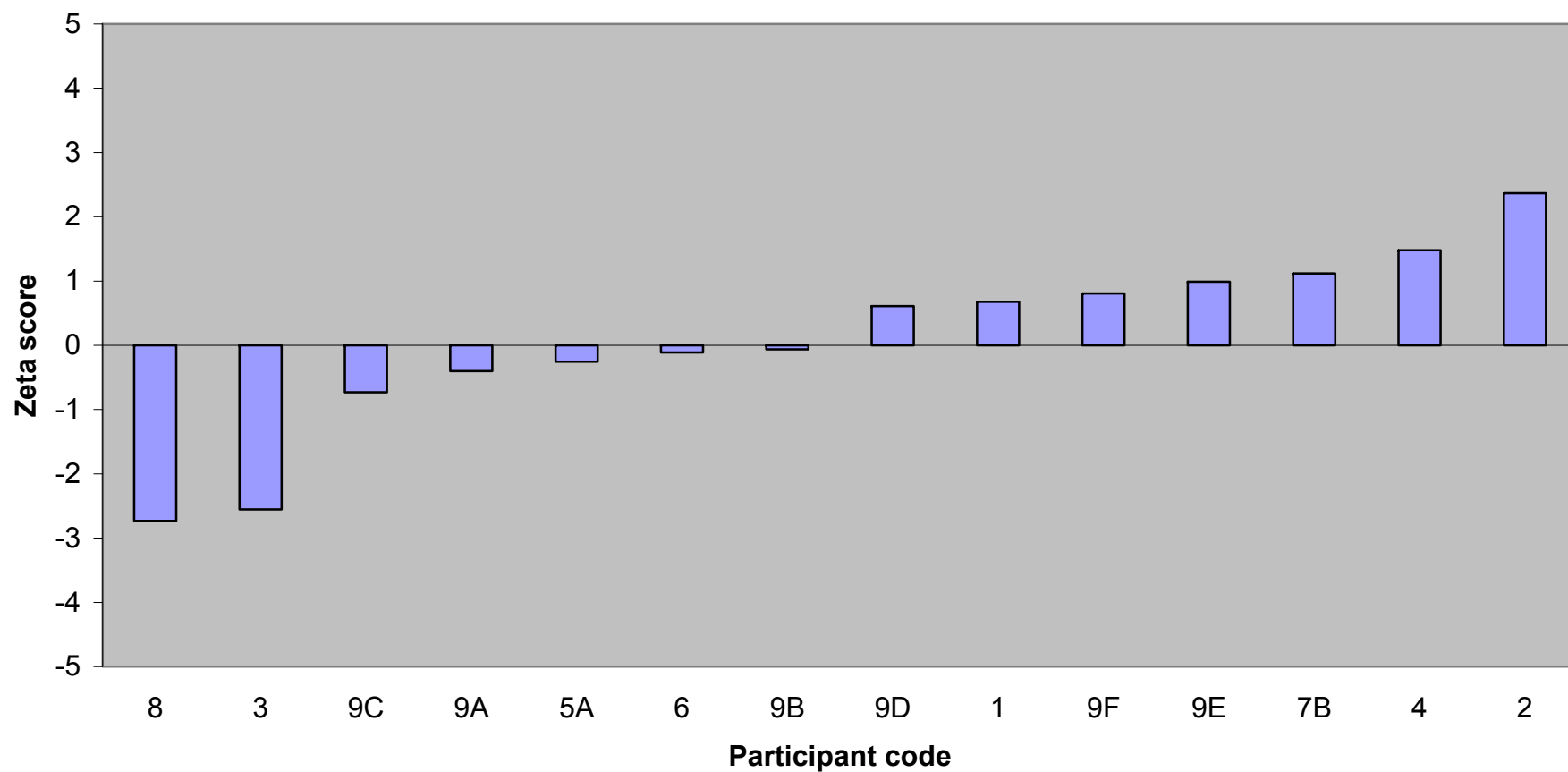


Figure 3 - Relative uncertainty plot for ^{241}Am results for Drum A
(hot volume not known)

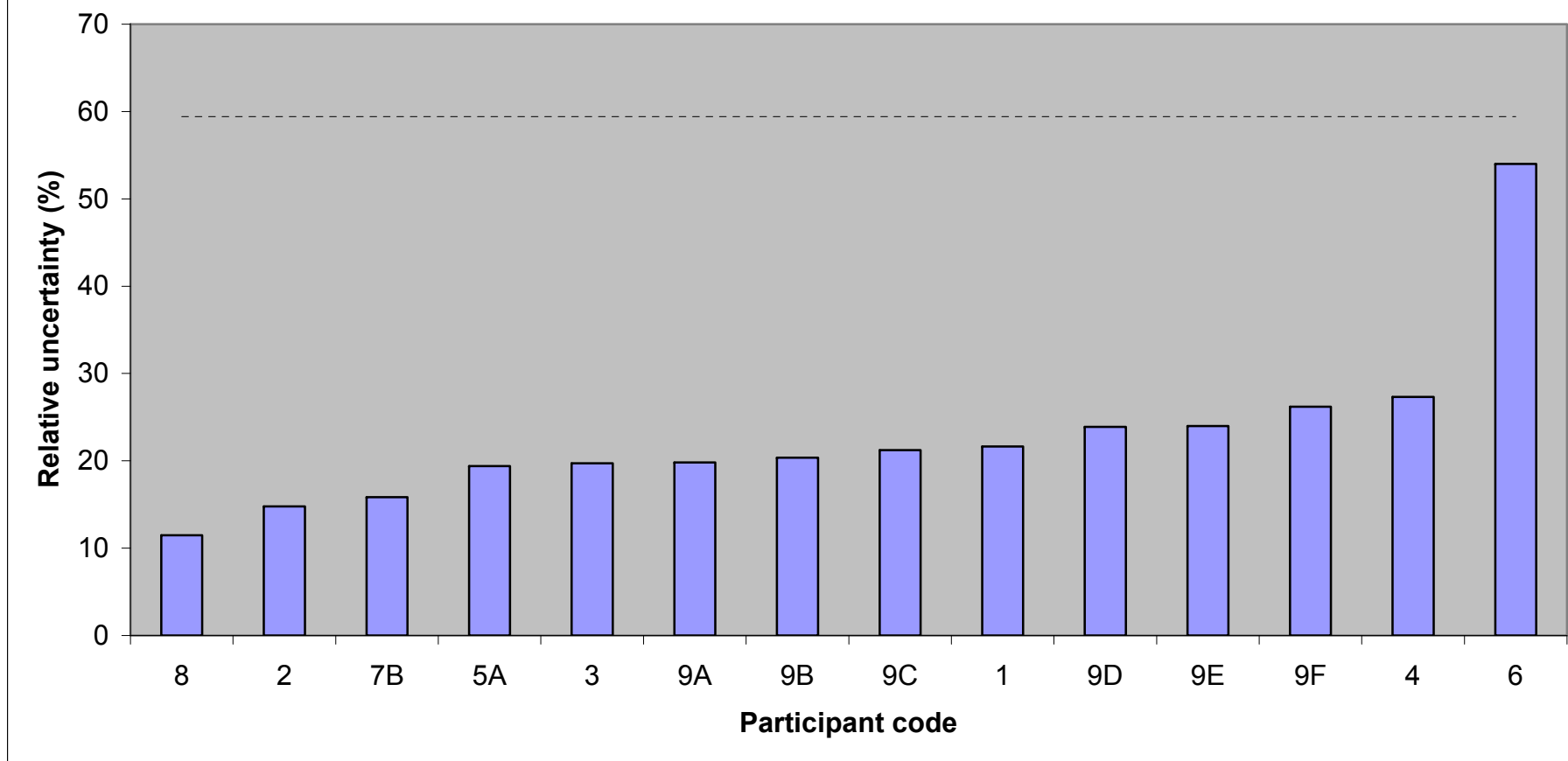
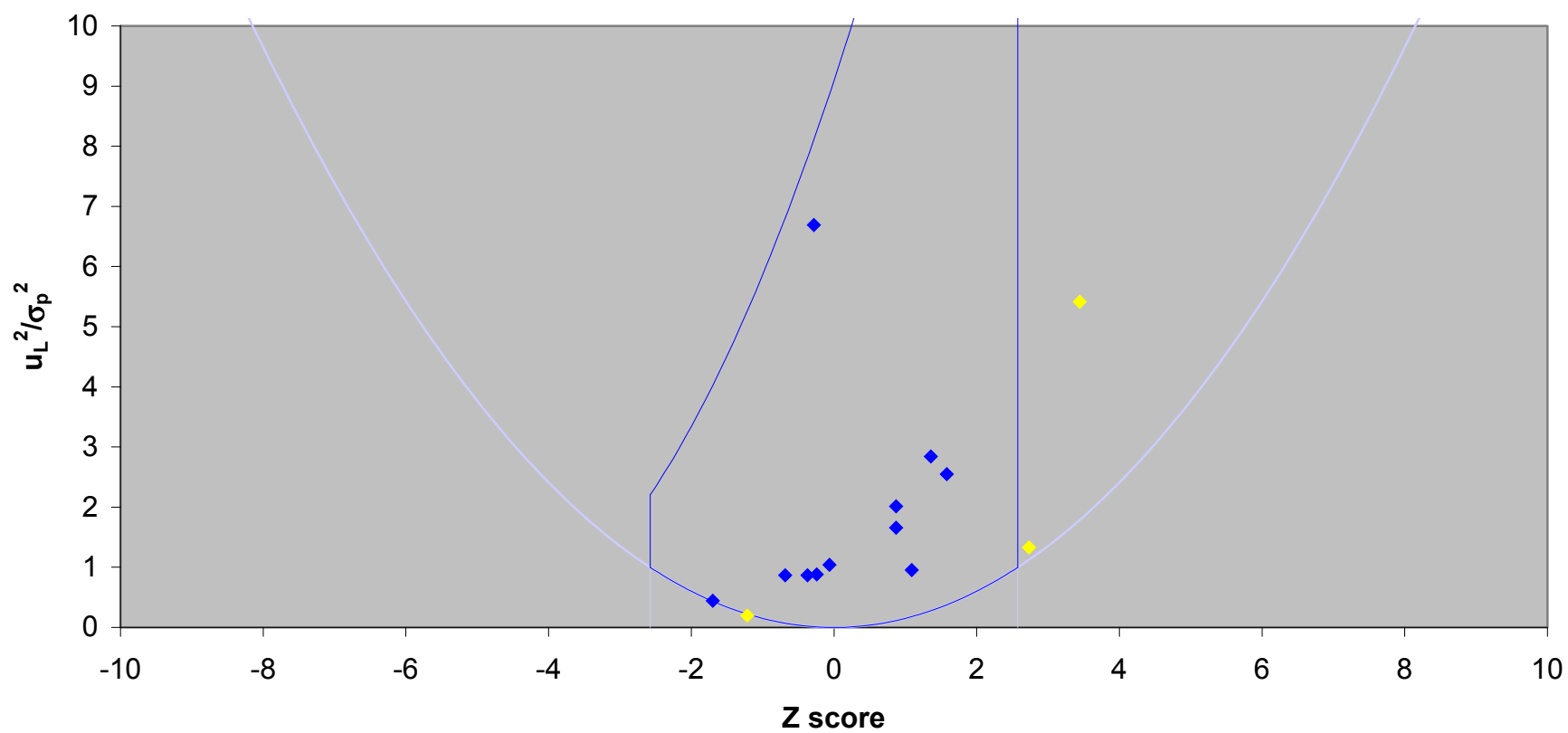


Figure 4 - 'Kiri' plot for ^{241}Am results for Drum A
(hot volume not known)



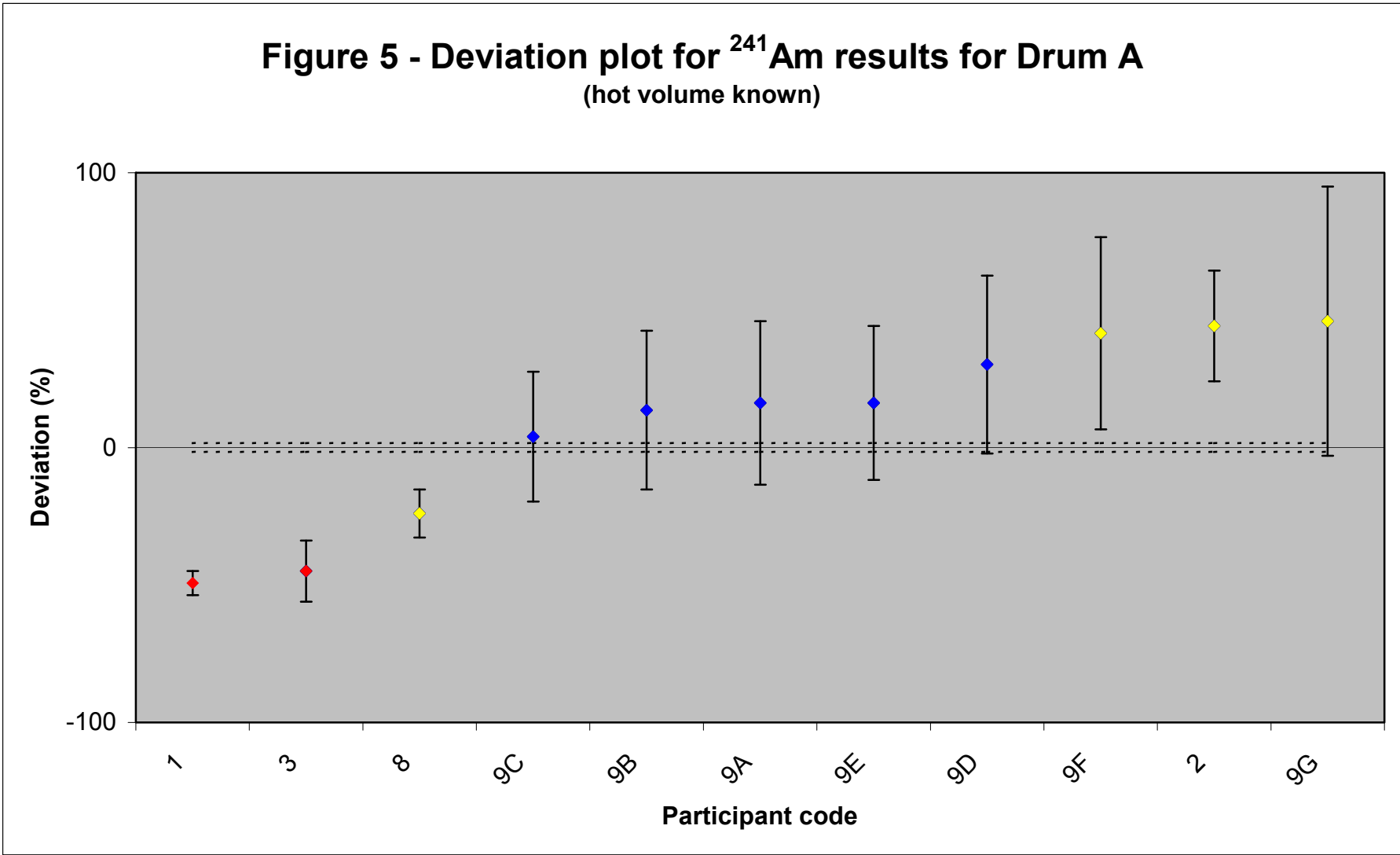


Figure 6 - Zeta-score plot for ^{241}Am results for Drum A
(hot volume known)

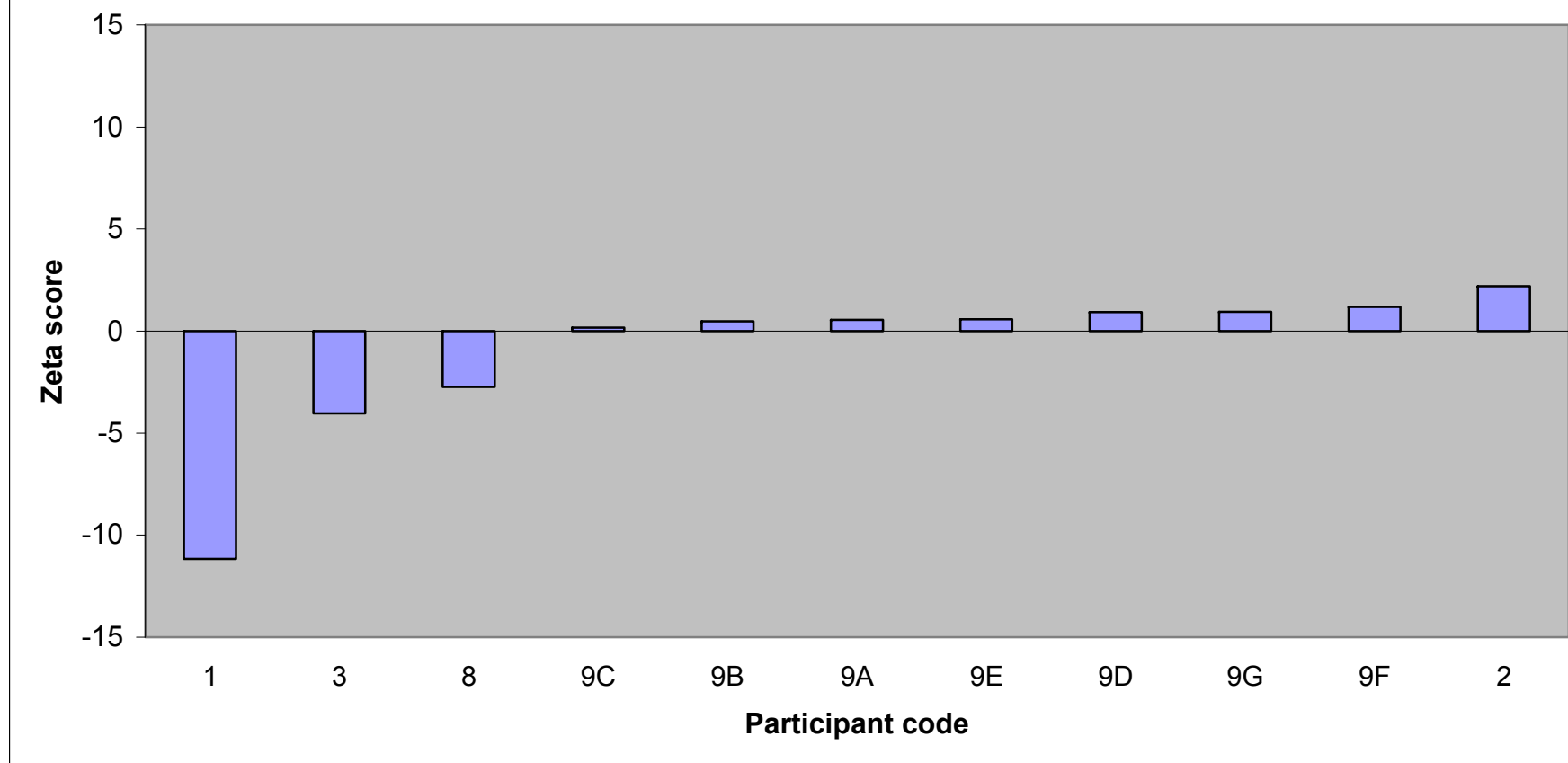


Figure 7 - Relative uncertainty plot for ^{241}Am results for Drum A
(hot volume known)

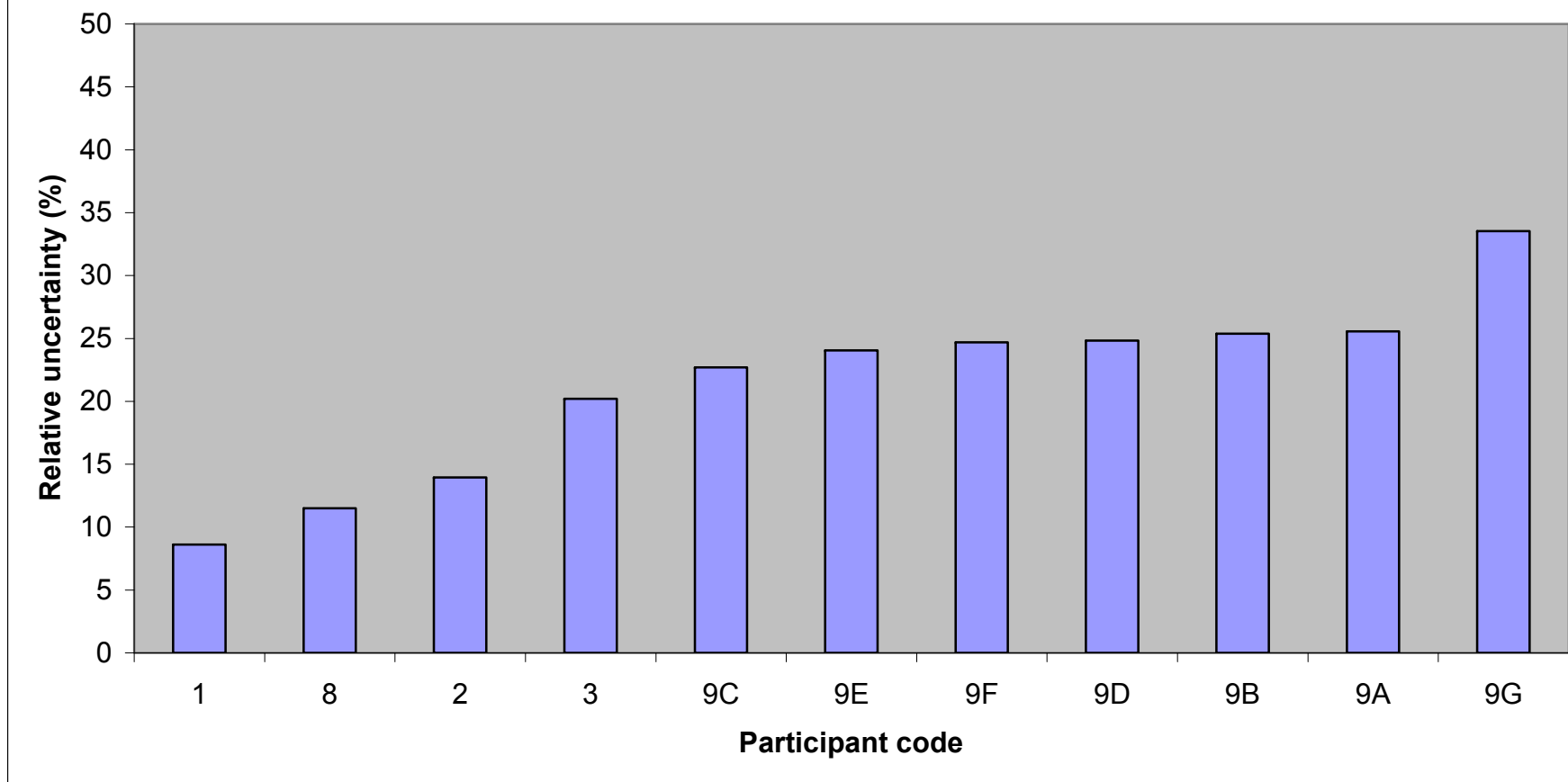


Figure 8 - 'Kiri' plot for ^{241}Am results for Drum A
(hot volume known)

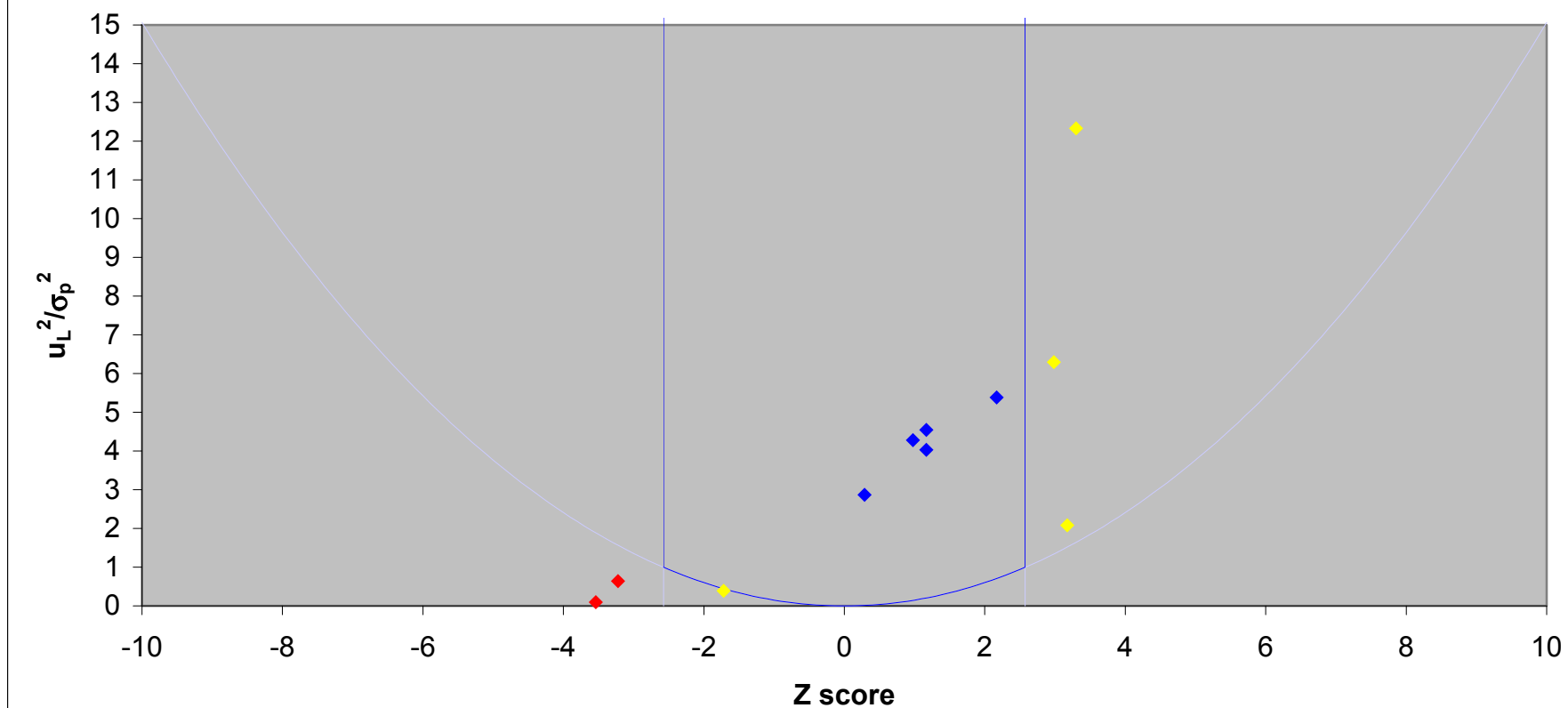


Figure 9 - Deviation plot for ^{60}Co results for Drum A
(hot volume not known)

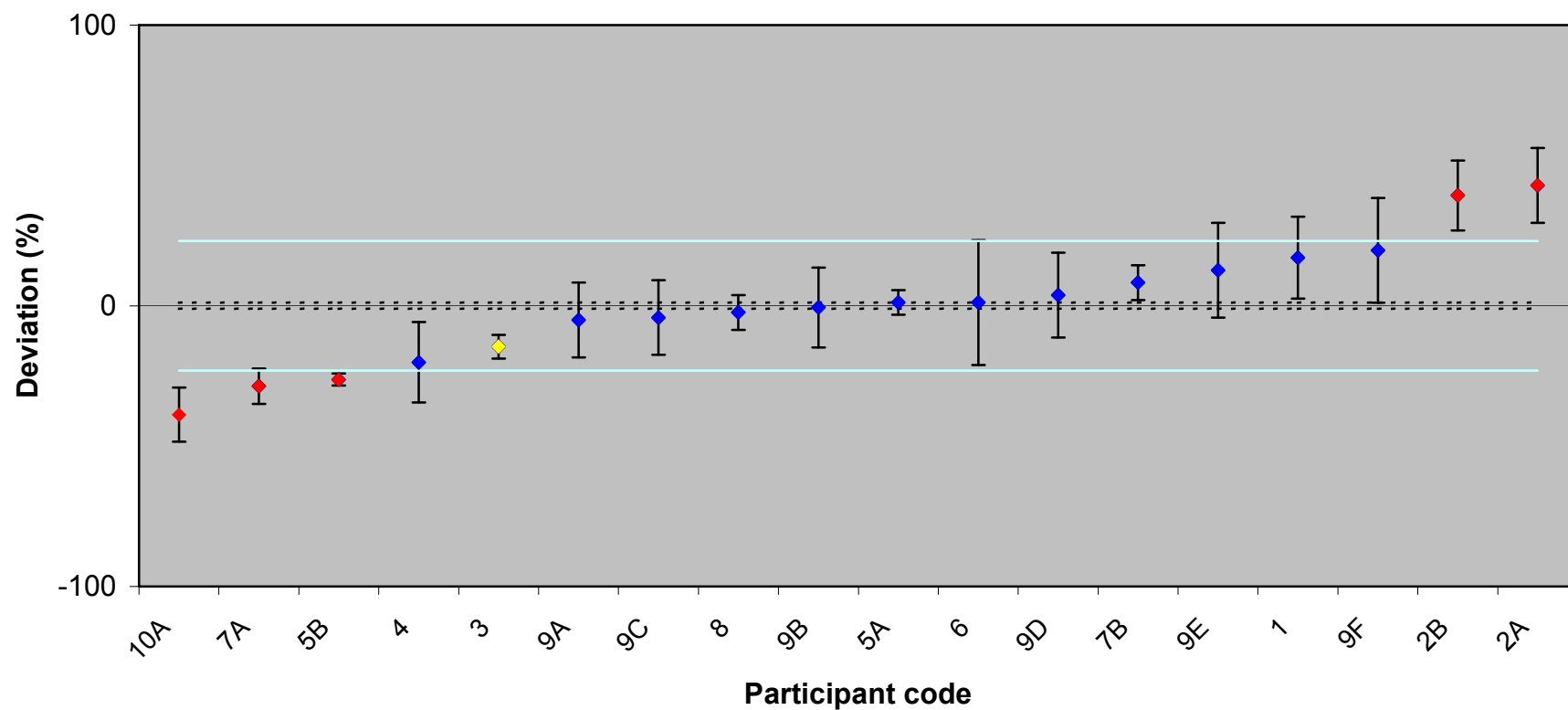


Figure 10 - Zeta-score plot for ^{60}Co results for Drum A
(hot volume not known)

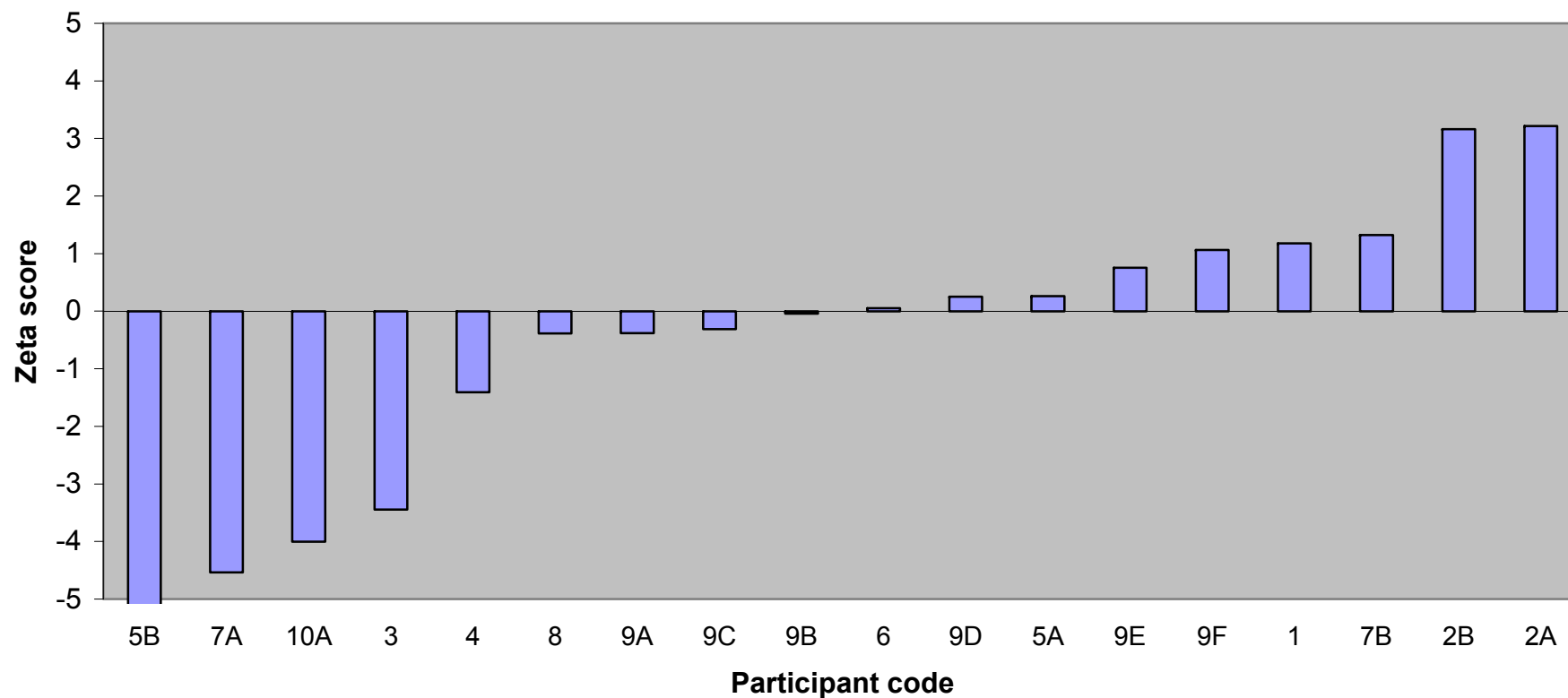


Figure 11 - Relative uncertainty plot for ^{60}Co results for Drum A
(hot volume not known)

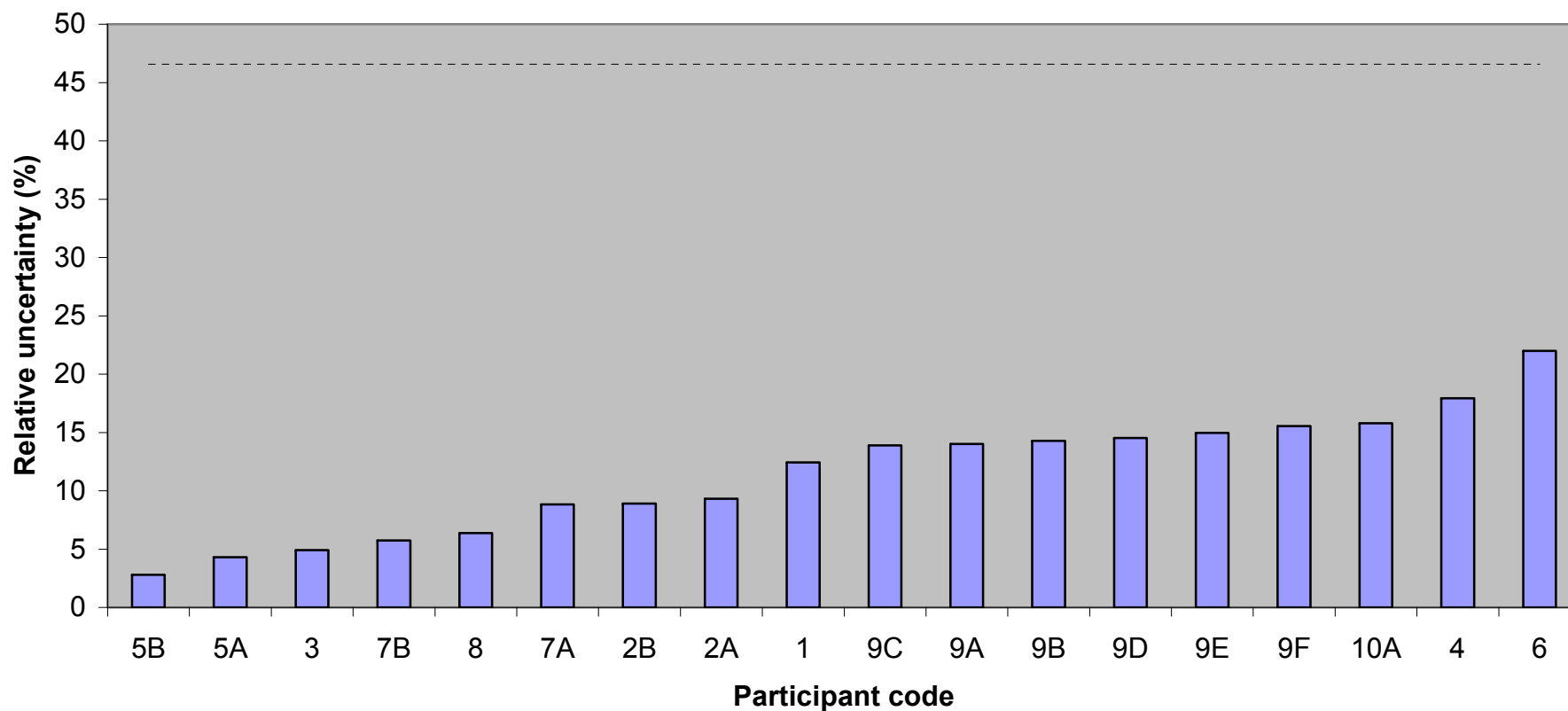
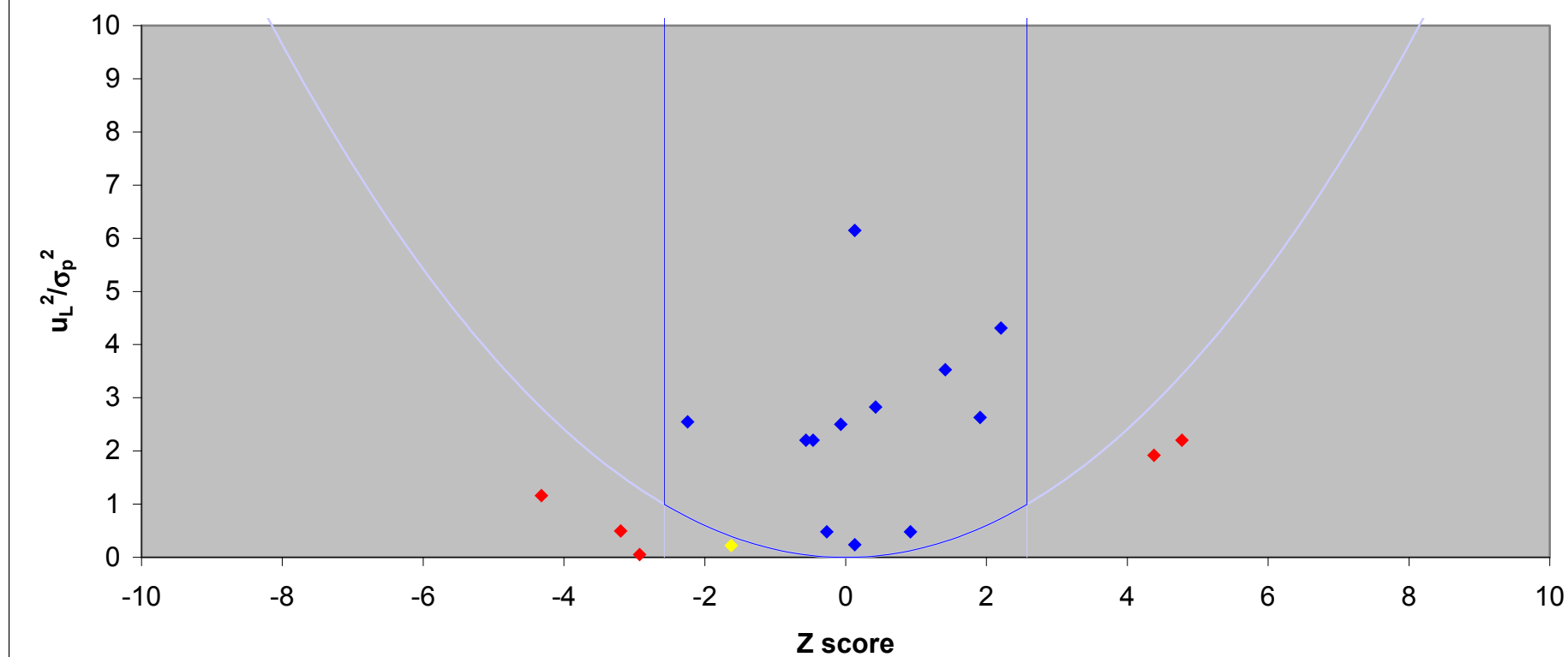


Figure 12 - 'Kiri' plot for ^{60}Co results for Drum A
(hot volume not known)



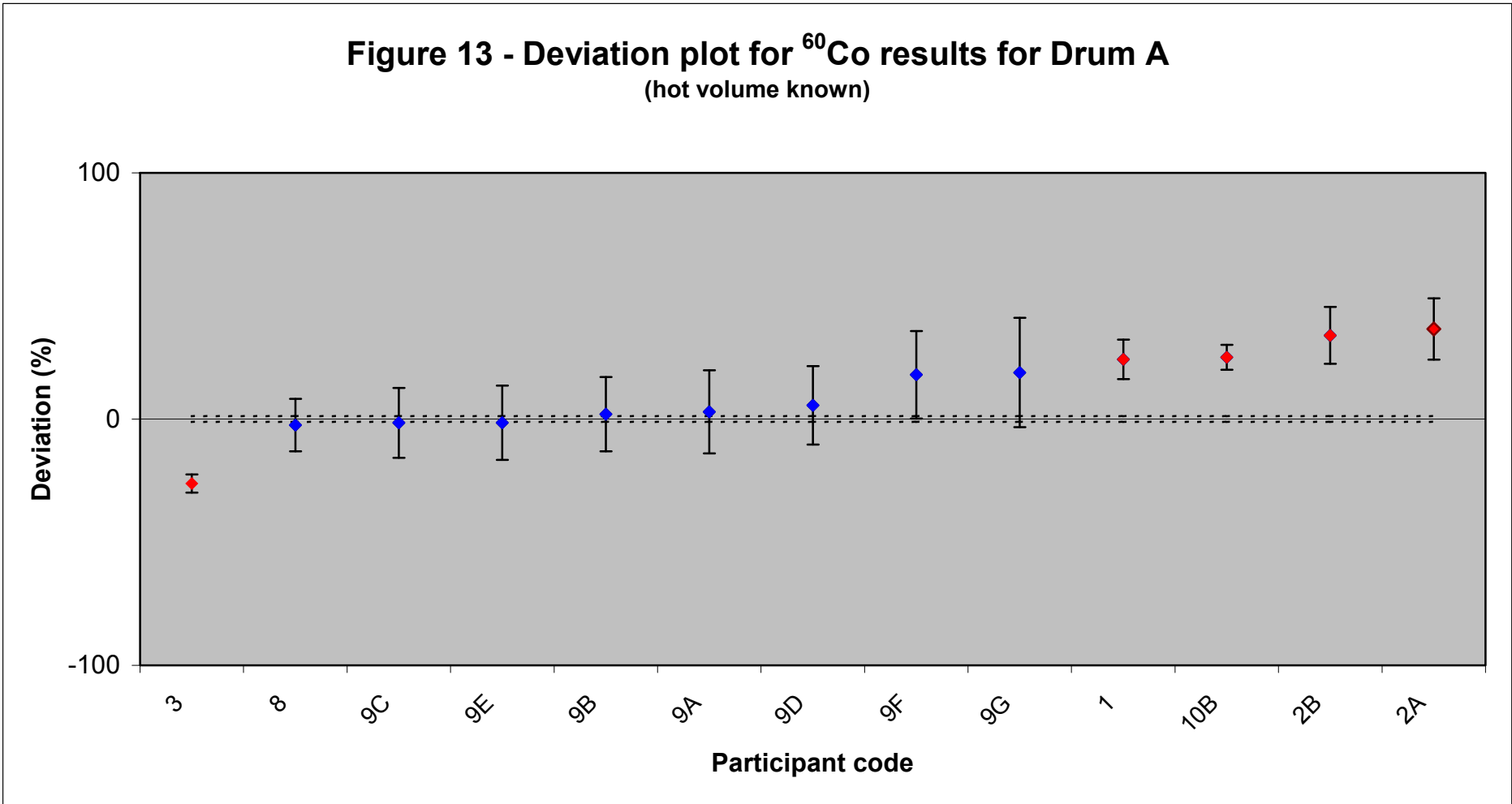


Figure 14 - Zeta-score plot for ^{60}Co results for Drum A
(hot volume known)

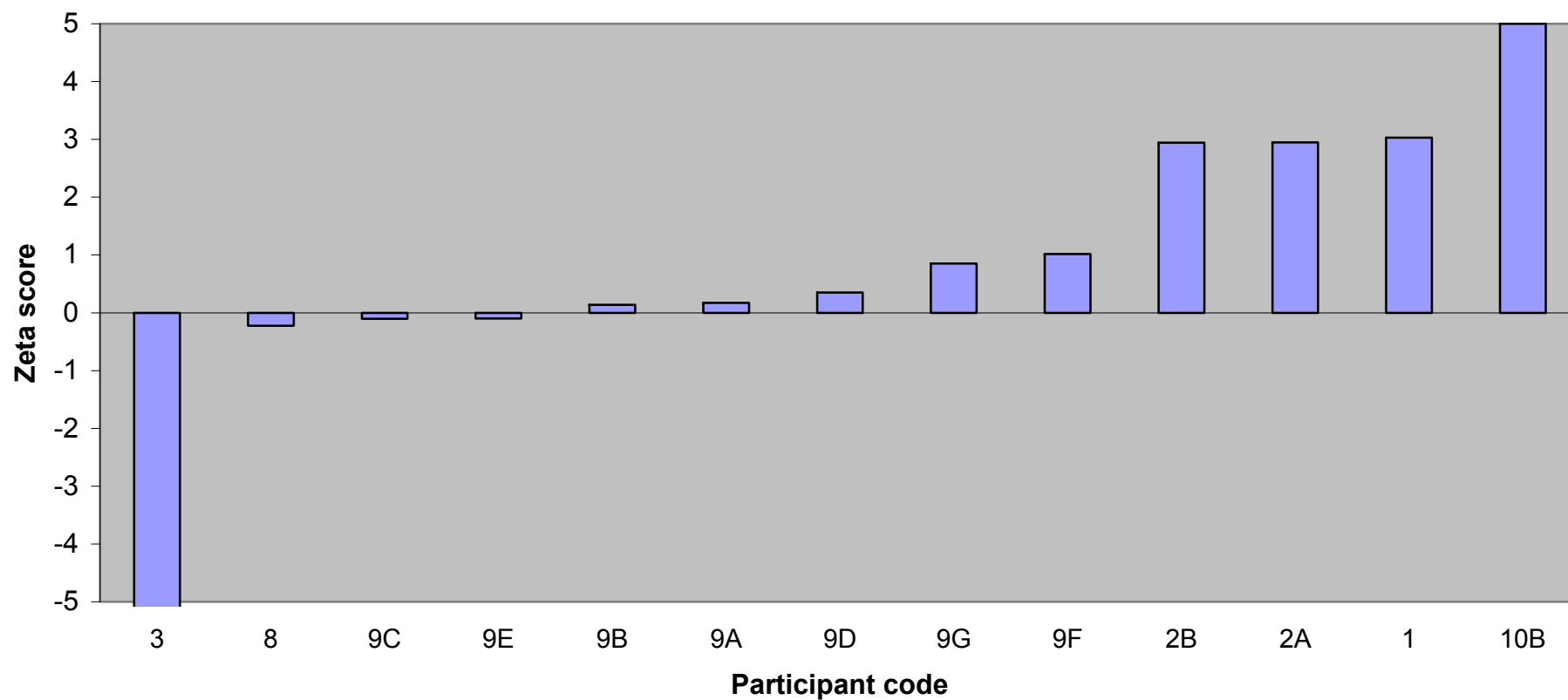


Figure 15 - Relative uncertainty plot for ^{60}Co results for Drum A
(hot volume known)

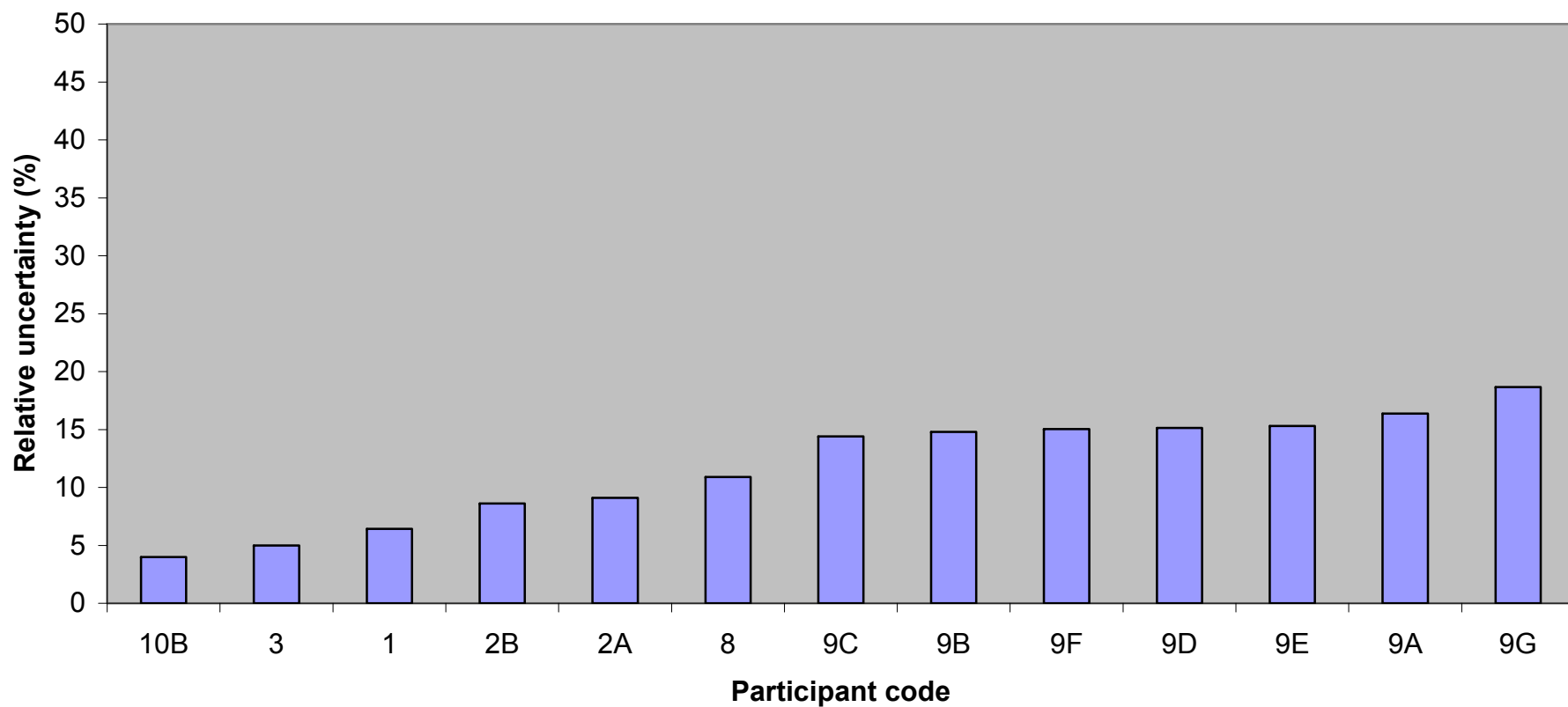


Figure 16 - 'Kiri' plot for ^{60}Co results for Drum A
(hot volume known)

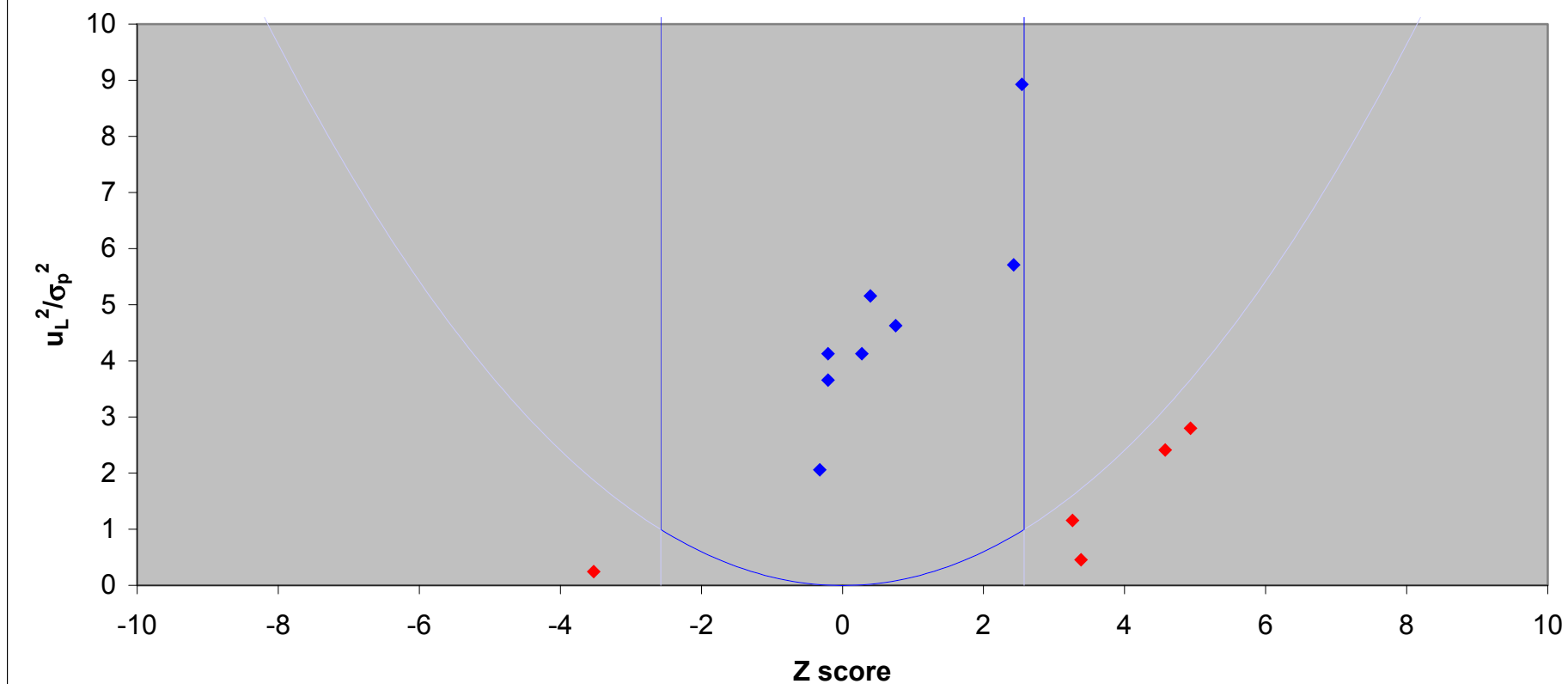


Figure 17 - Deviation plot for ^{137}Cs results for Drum A
(hot volume not known)

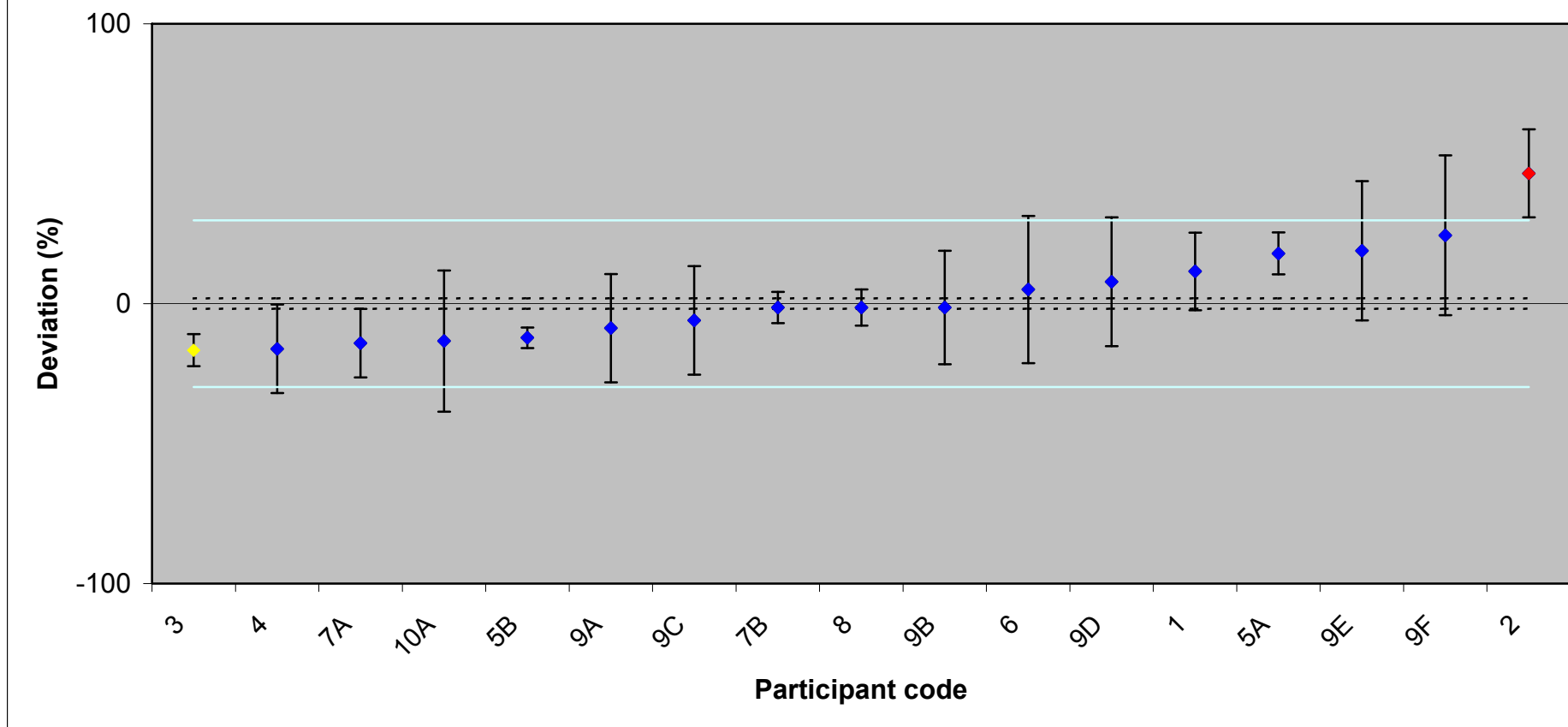


Figure 18 - Zeta-score plot for ^{137}Cs results for Drum A
(hot volume not known)

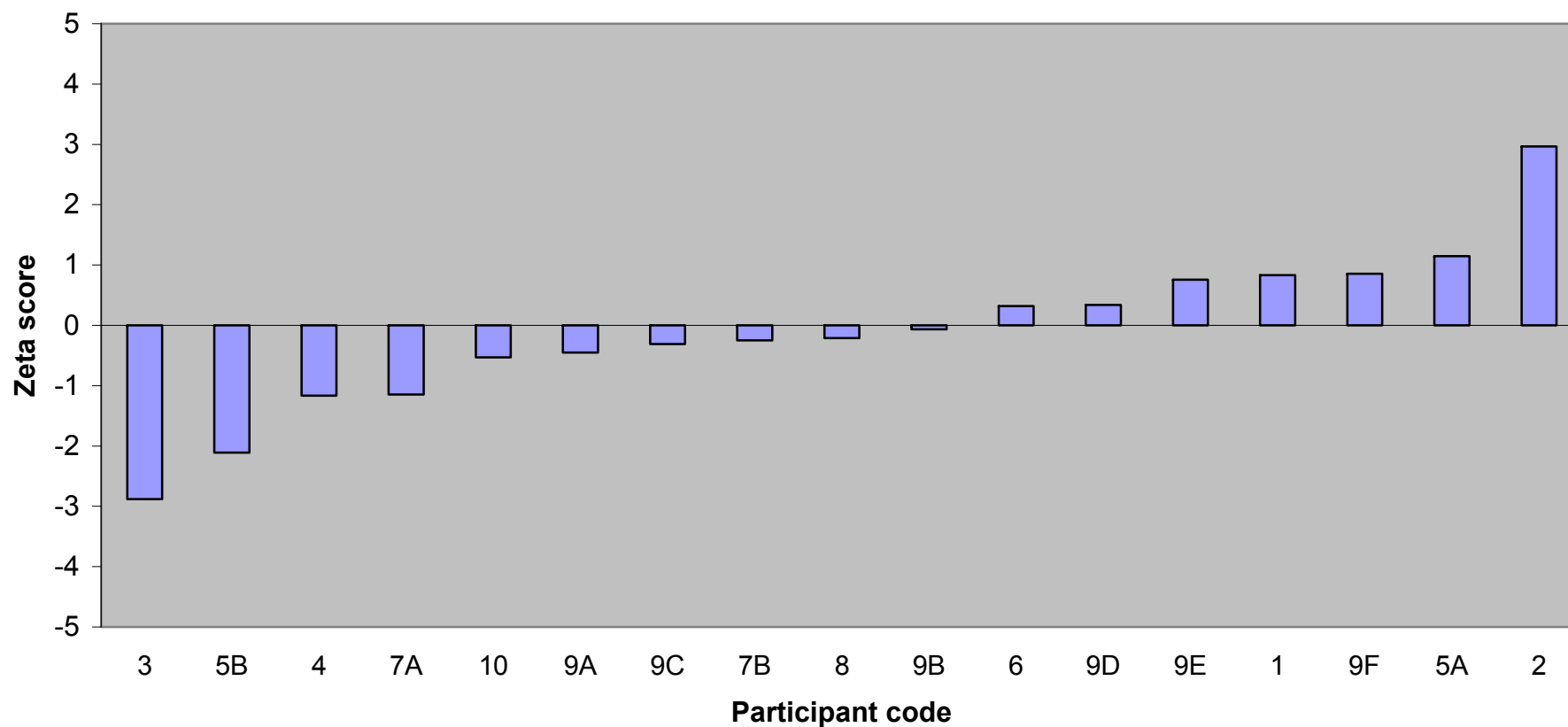


Figure 19 - Relative uncertainty plot for ^{137}Cs results for Drum A
(hot volume not known)

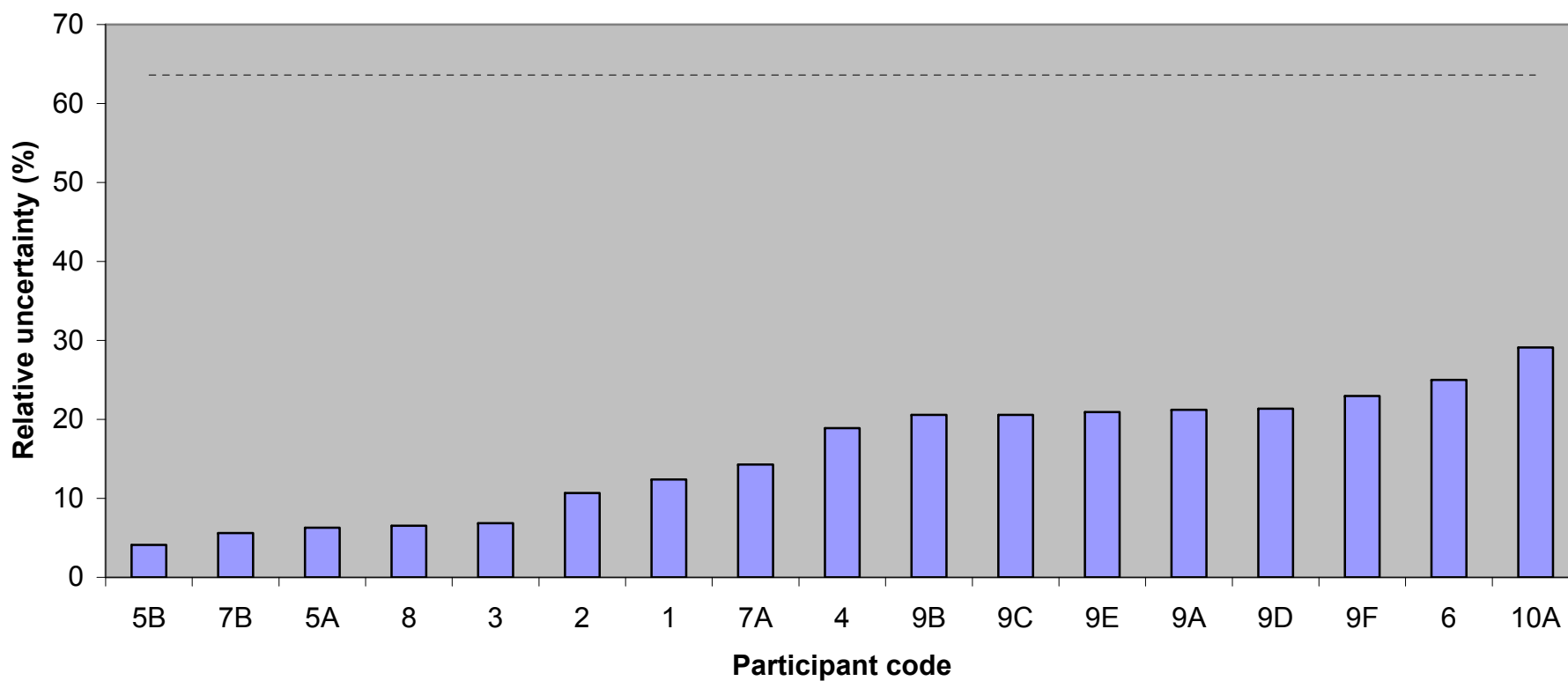


Figure 20 - 'Kiri' plot for ^{137}C results for Drum A
(hot volume not known)

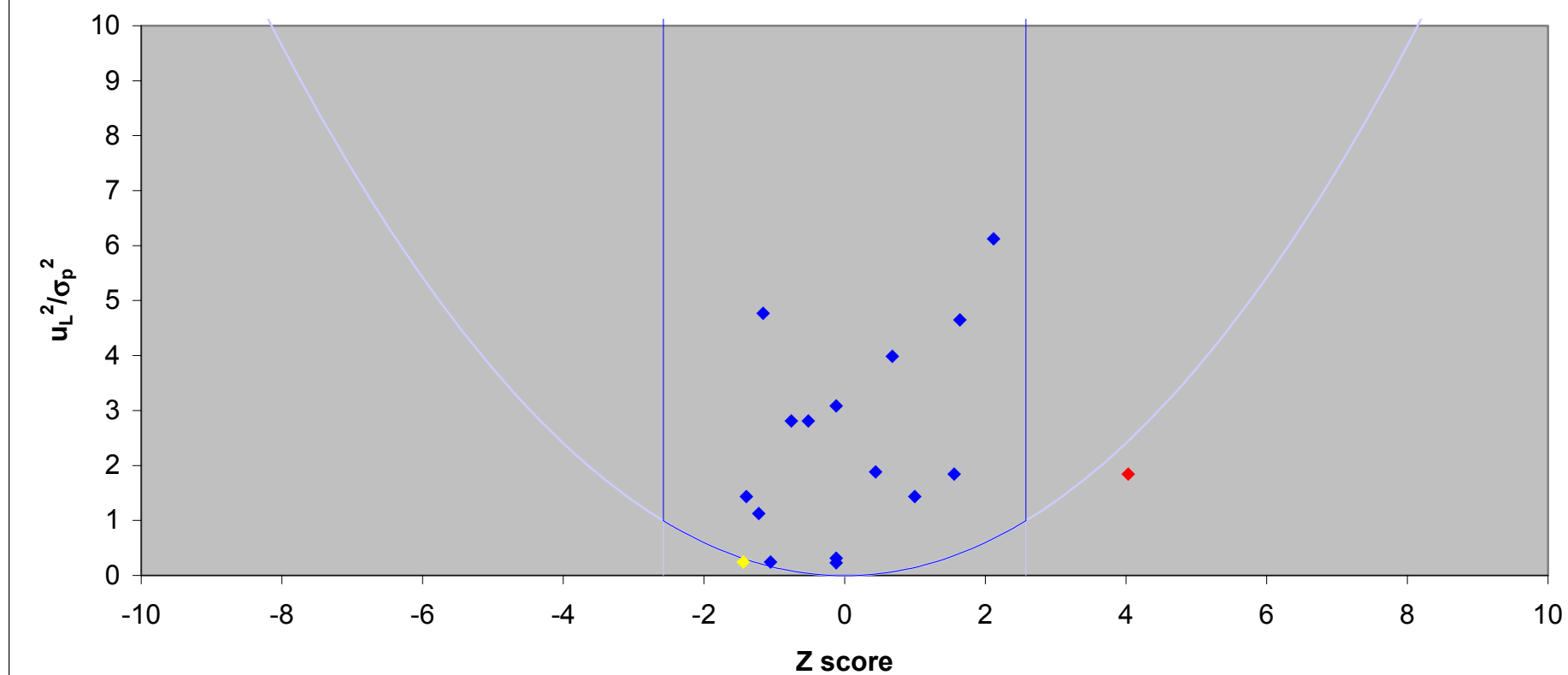


Figure 21 - Deviation plot for ^{137}Cs results for Drum A
 (hot volume known) (1 result above scale)

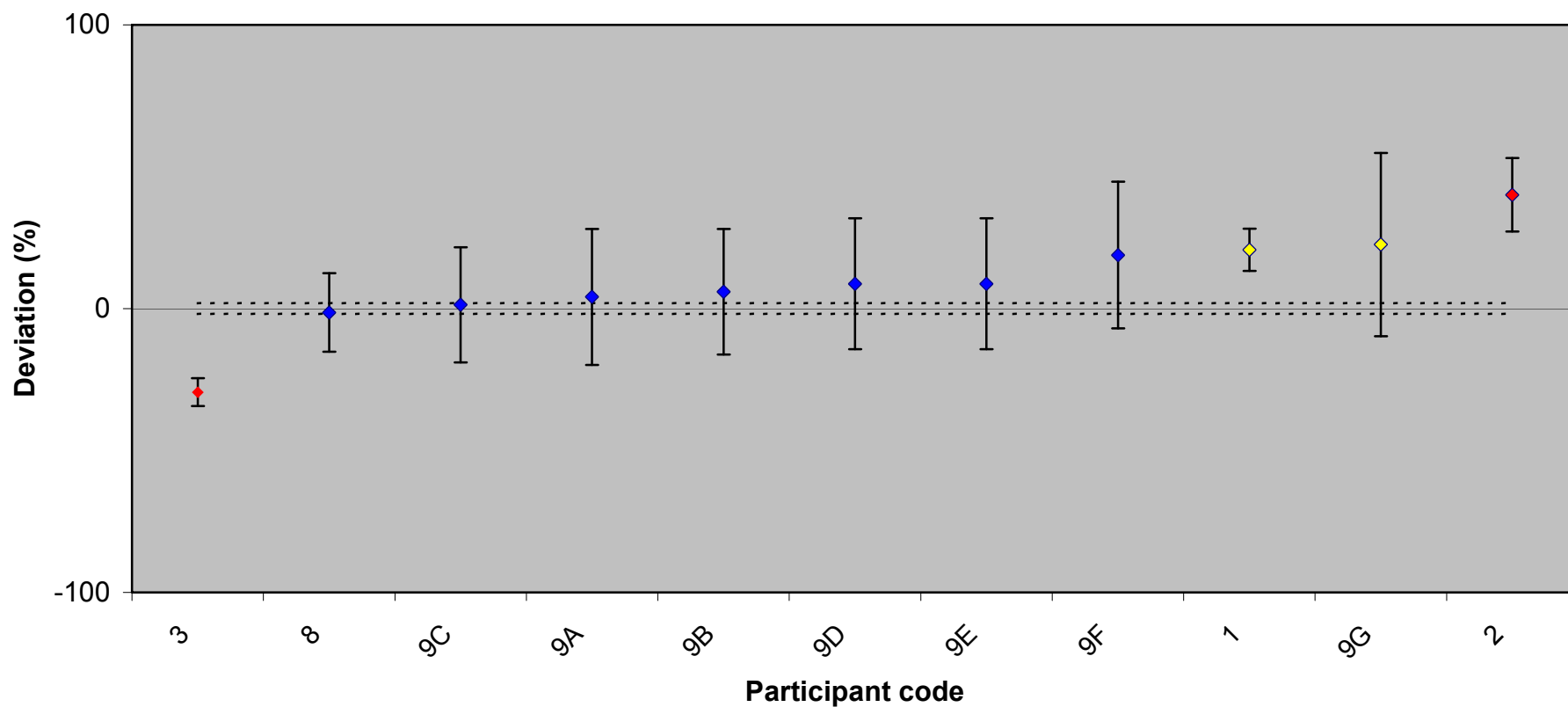


Figure 22 - Zeta-score plot for ^{137}Cs results for Drum A
(hot volume known)

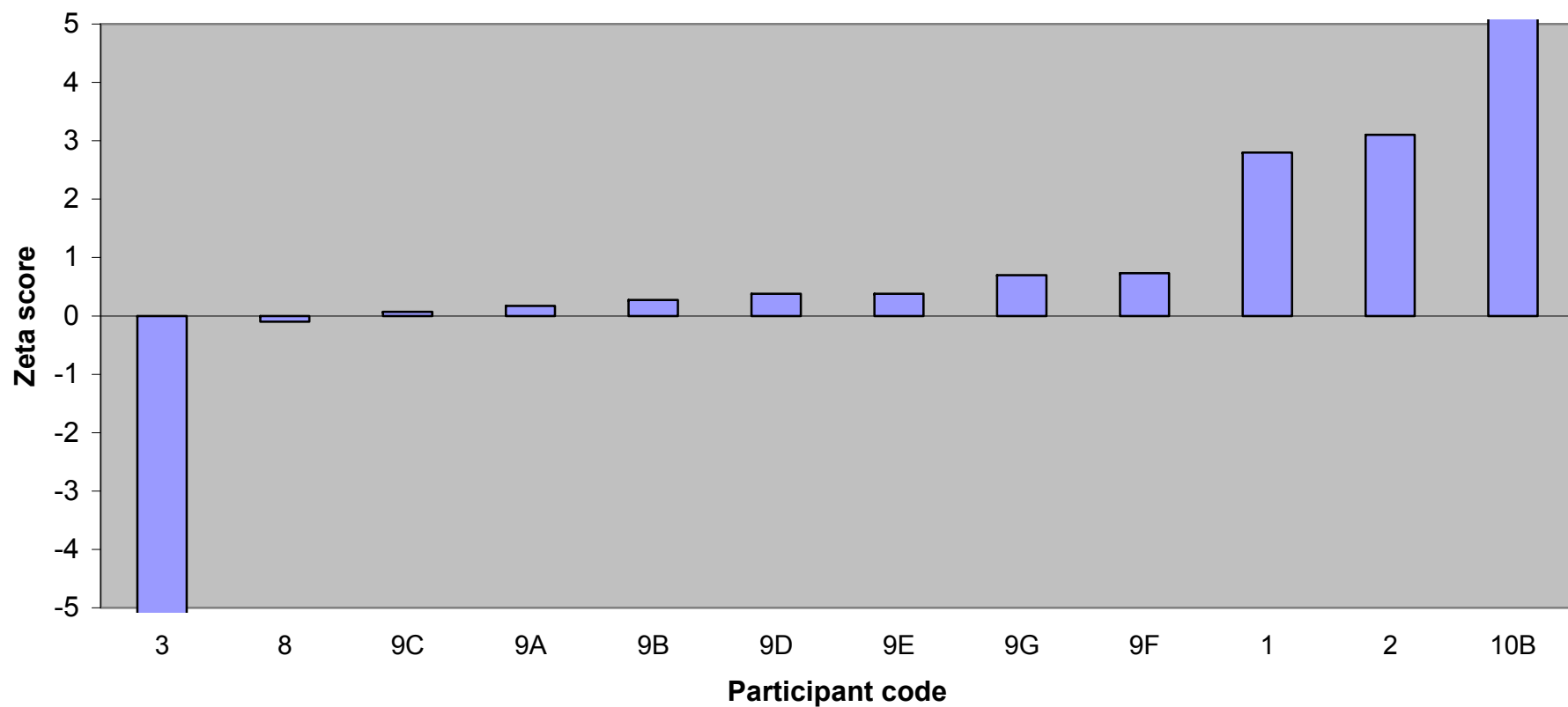


Figure 23 - Relative uncertainty plot for ^{137}Cs results for Drum A
(hot volume known)

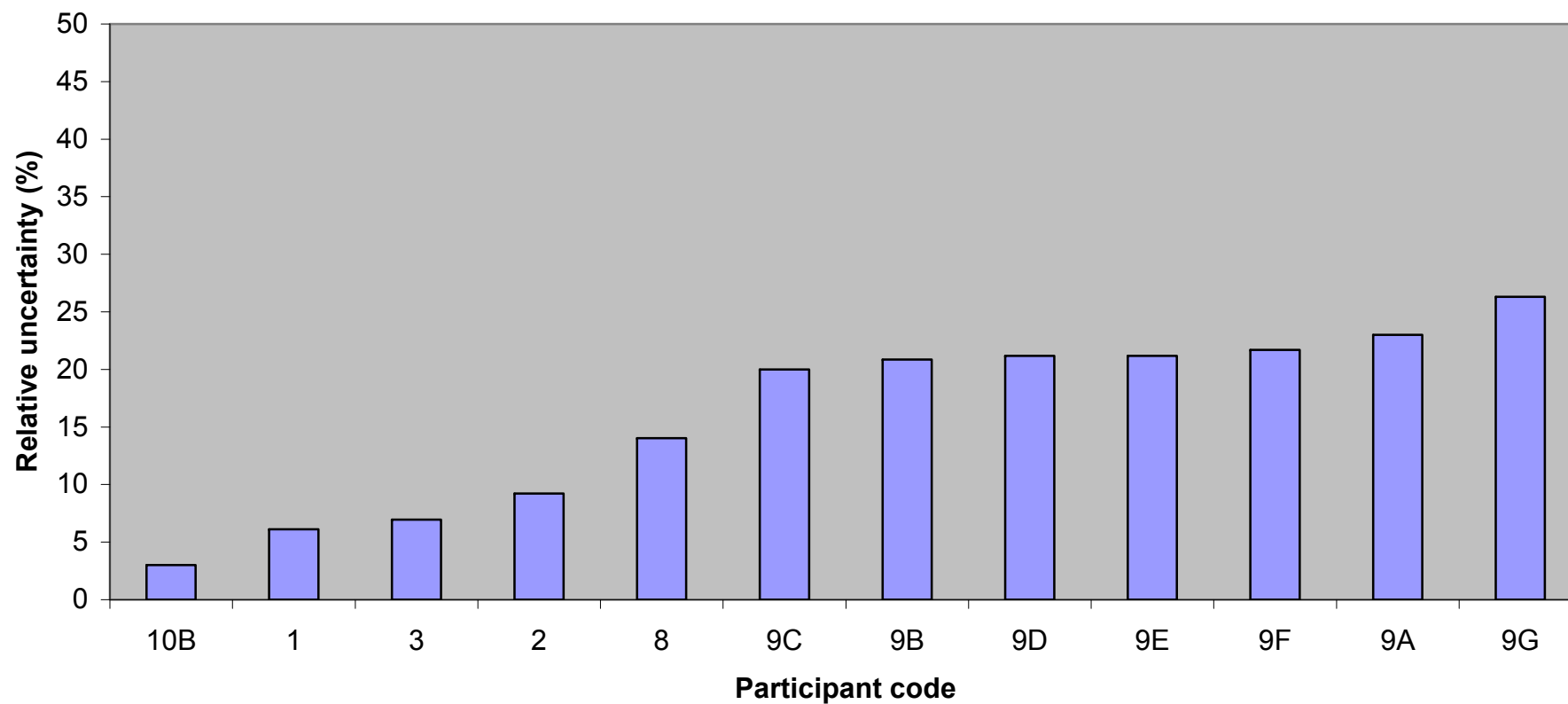
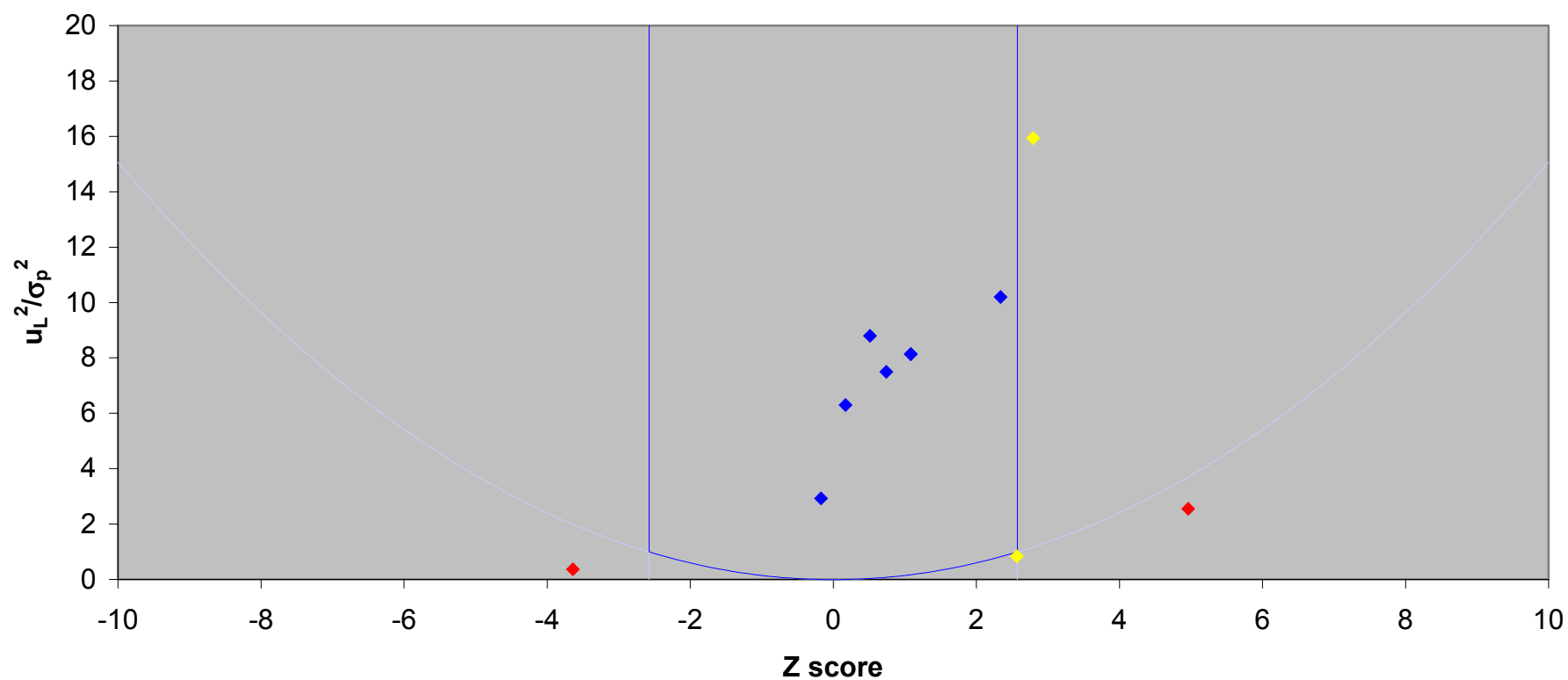


Figure 24 - 'Kiri' plot for ^{137}Cs results for Drum A
(hot volume known) (1 result to right of scale)



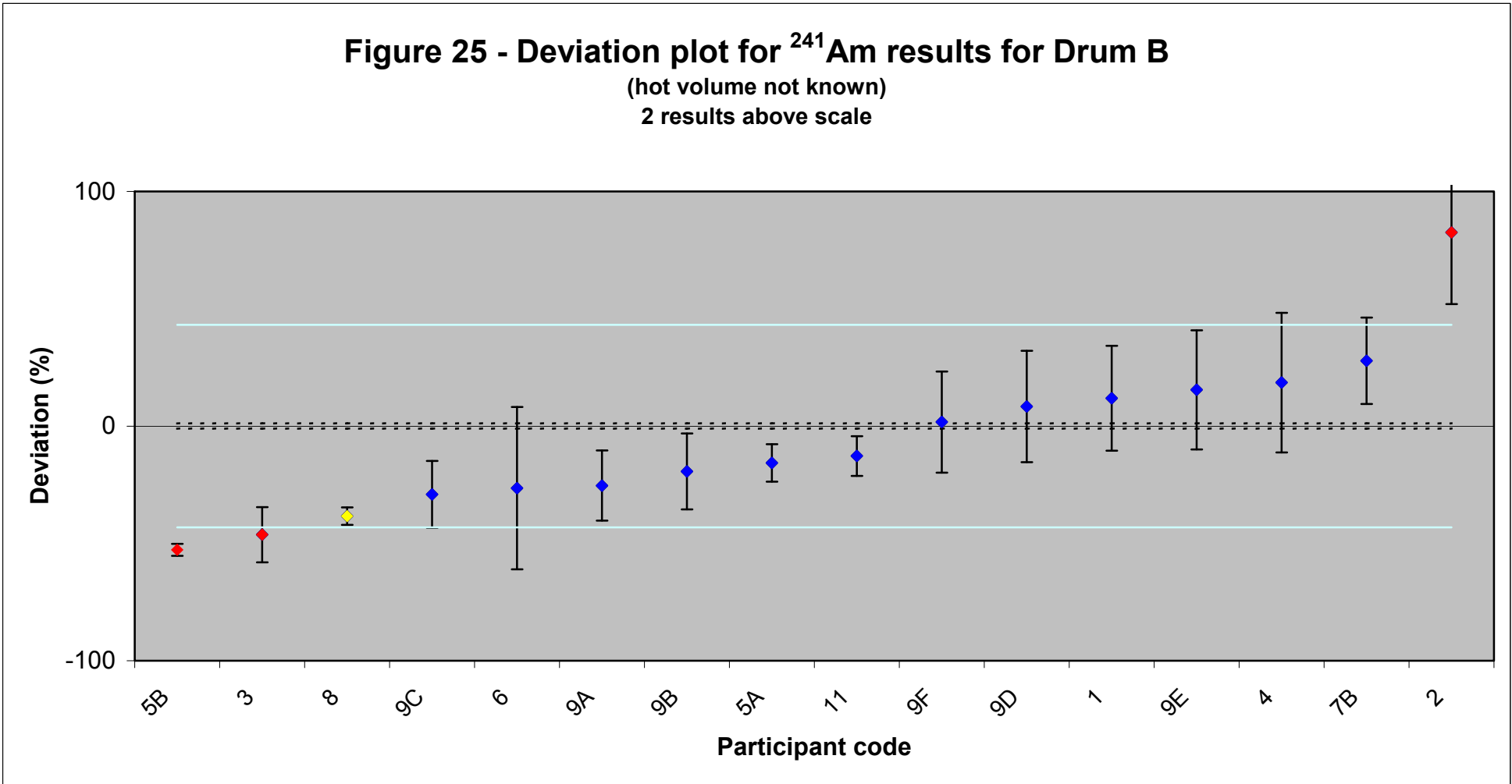


Figure 26 - Zeta-score plot for ^{241}Am results for Drum B
(hot volume not known)

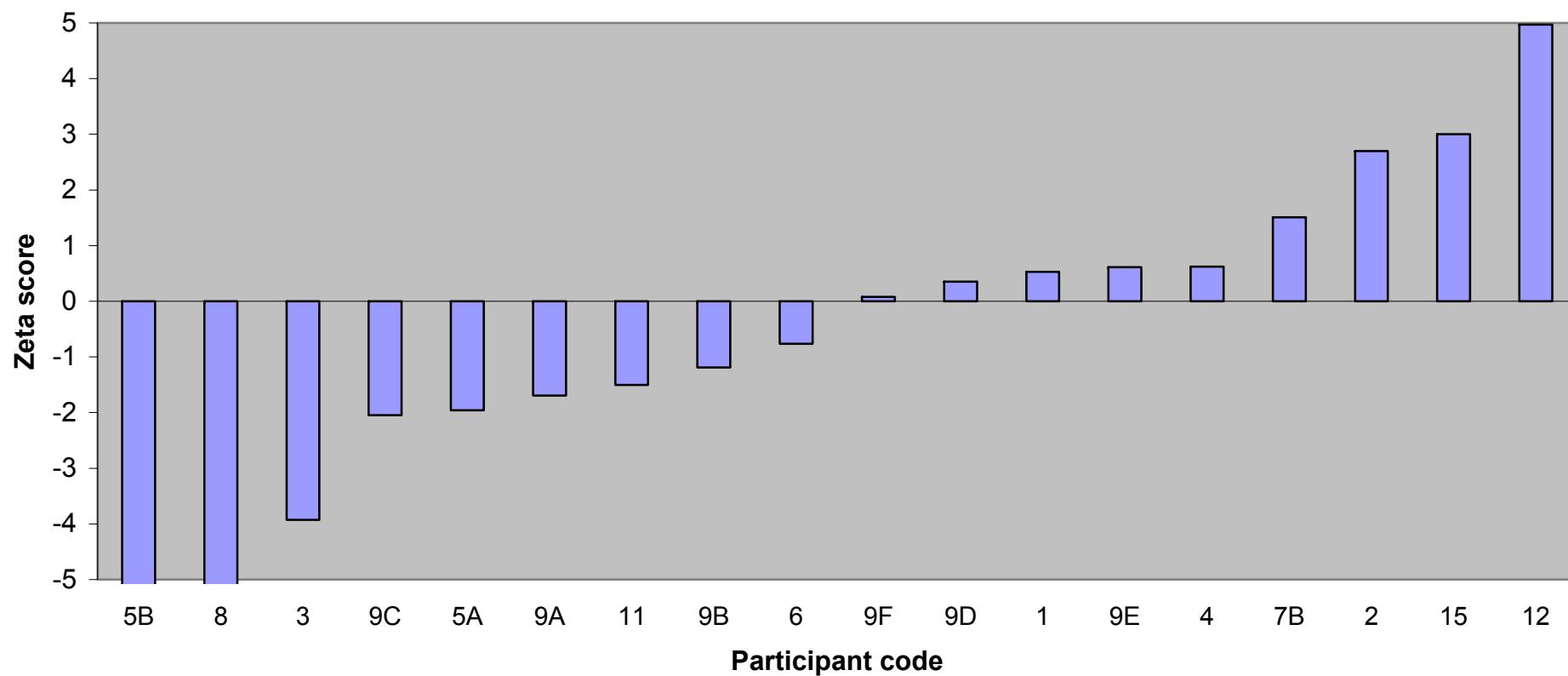


Figure 27 - Relative uncertainty plot for ^{241}Am results for Drum B
(hot volume not known)

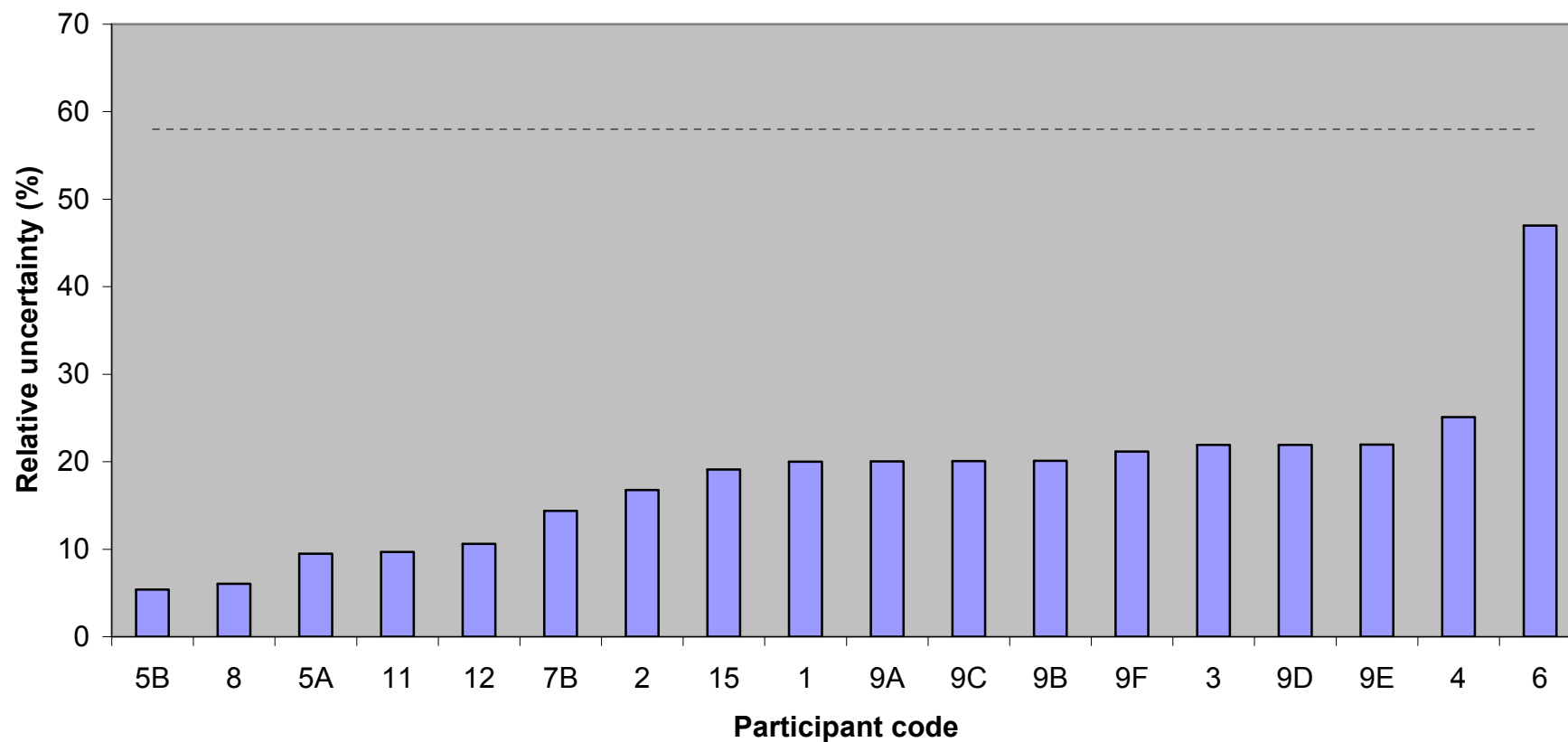


Figure 28 - 'Kiri' plot for ^{241}Am results for Drum B
(hot volume not known)

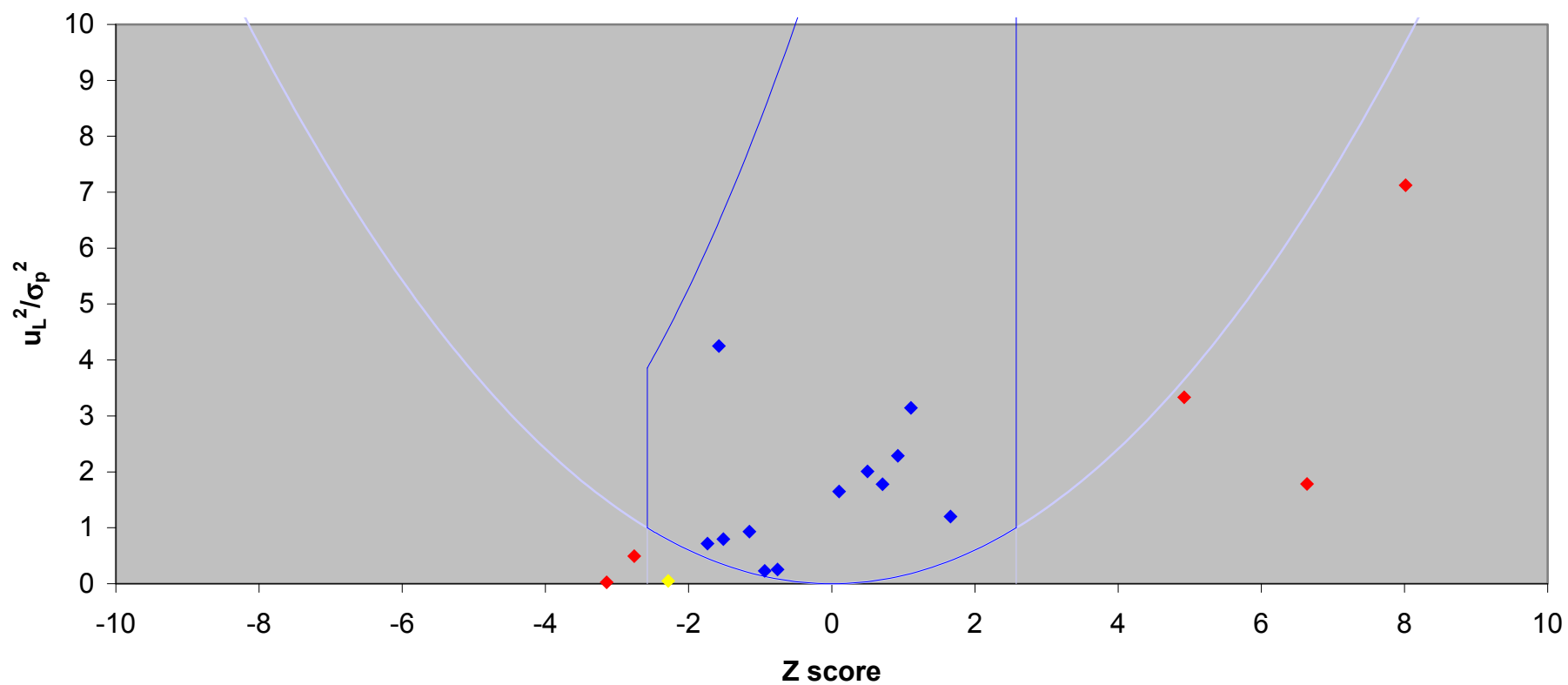


Figure 29 - Deviation plot for ^{241}Am results for Drum B
(hot volume known)

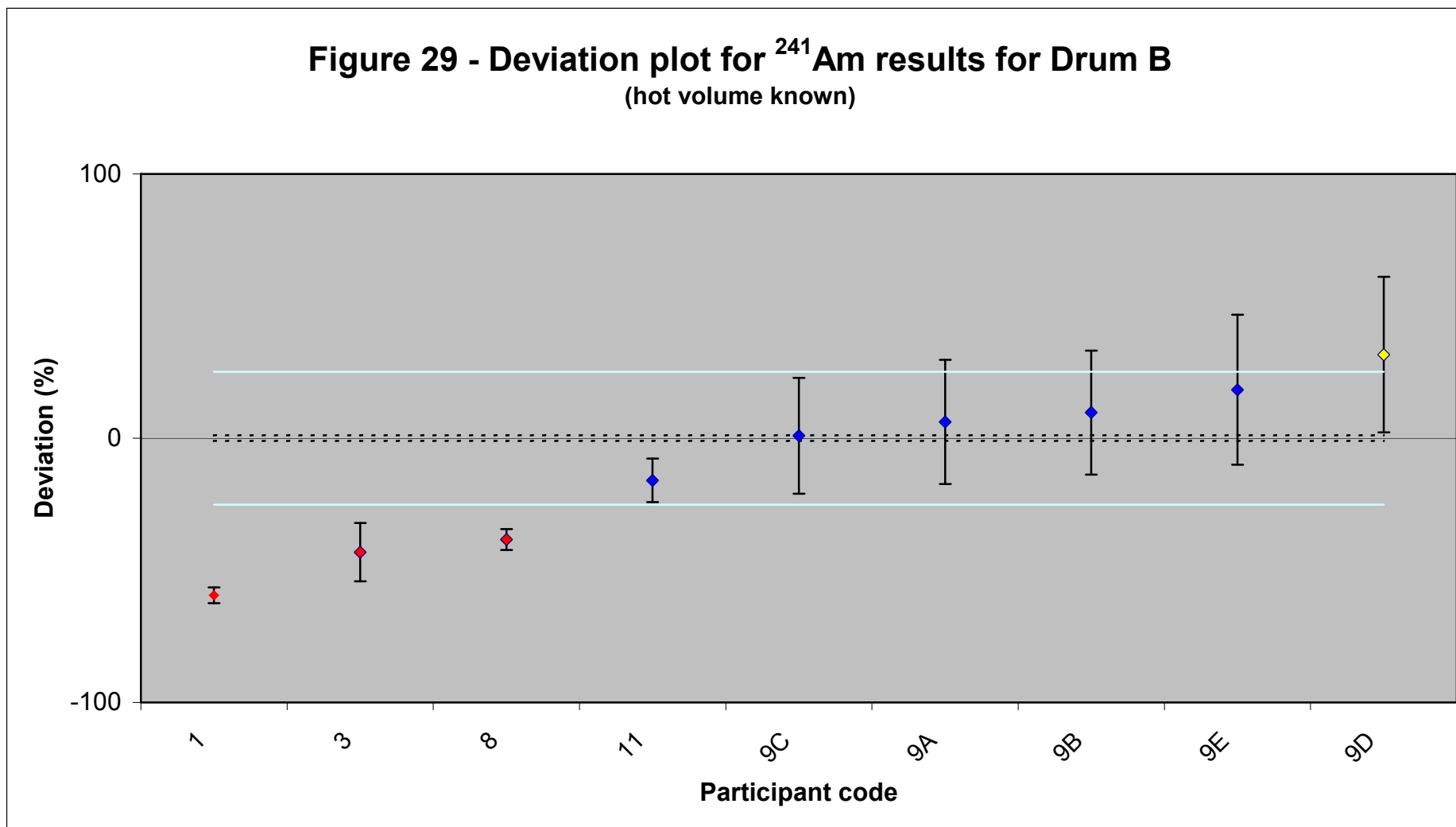


Figure 30 - Zeta-score plot for ^{241}Am results for Drum B
(hot volume known)

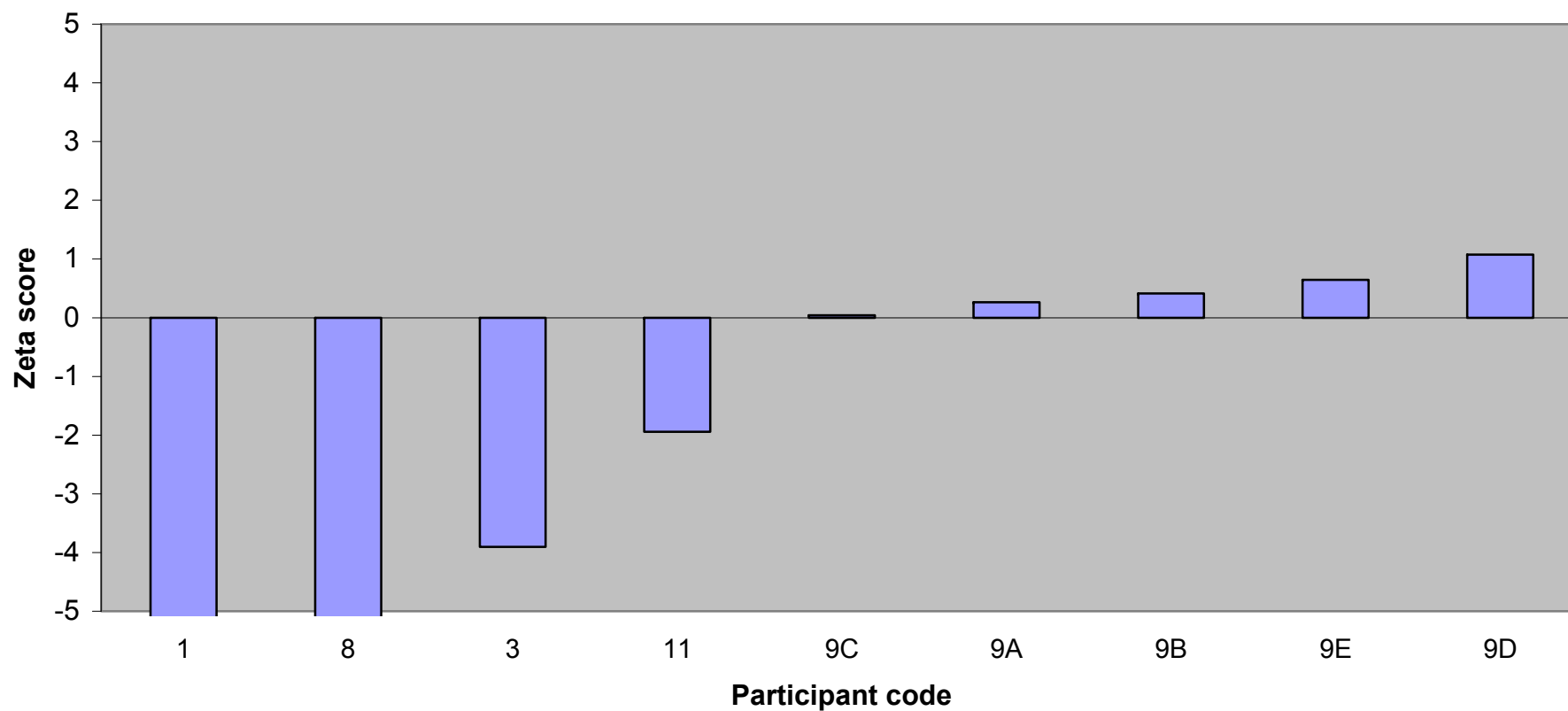


Figure 31 - Relative uncertainty plot for ^{241}Am results for Drum B
(hot volume known)

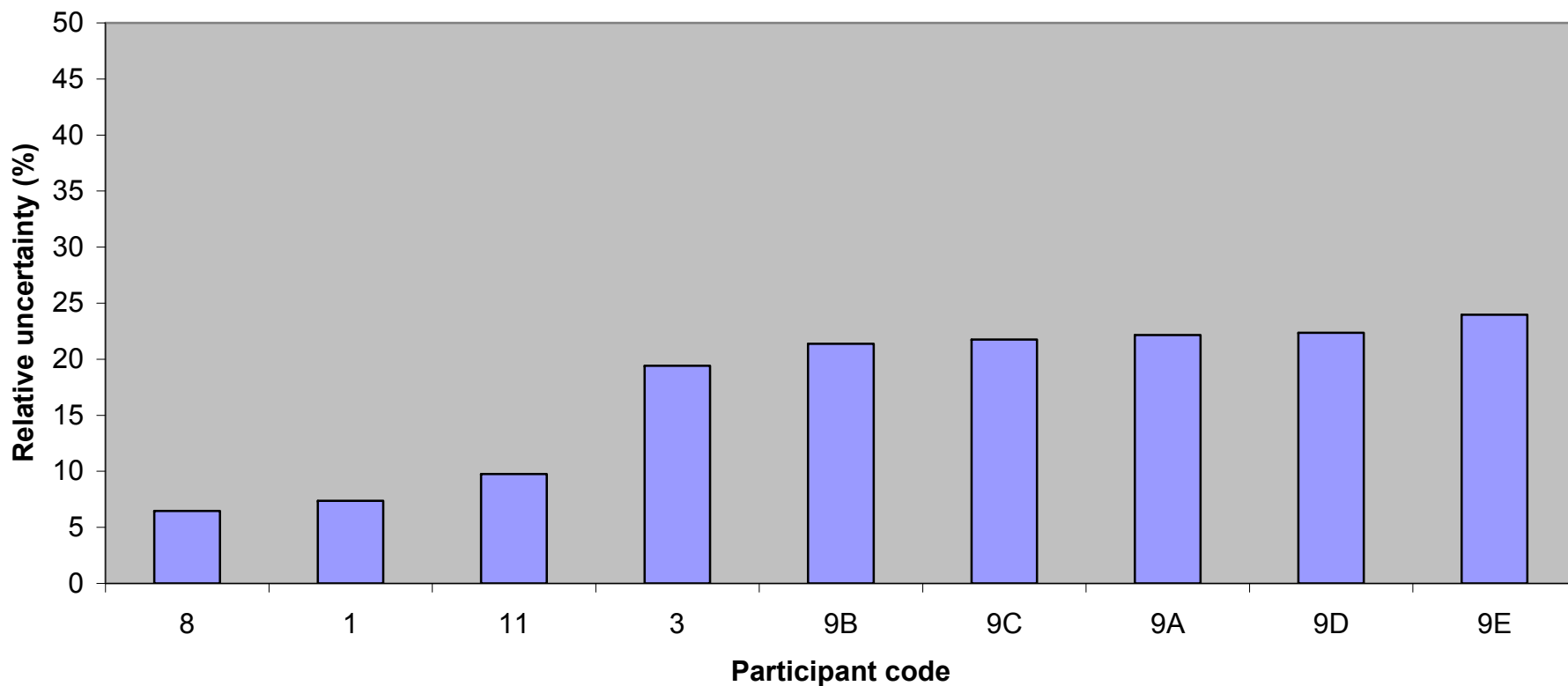


Figure 32 - 'Kiri' plot for ^{241}Am results for Drum B
(hot volume known)

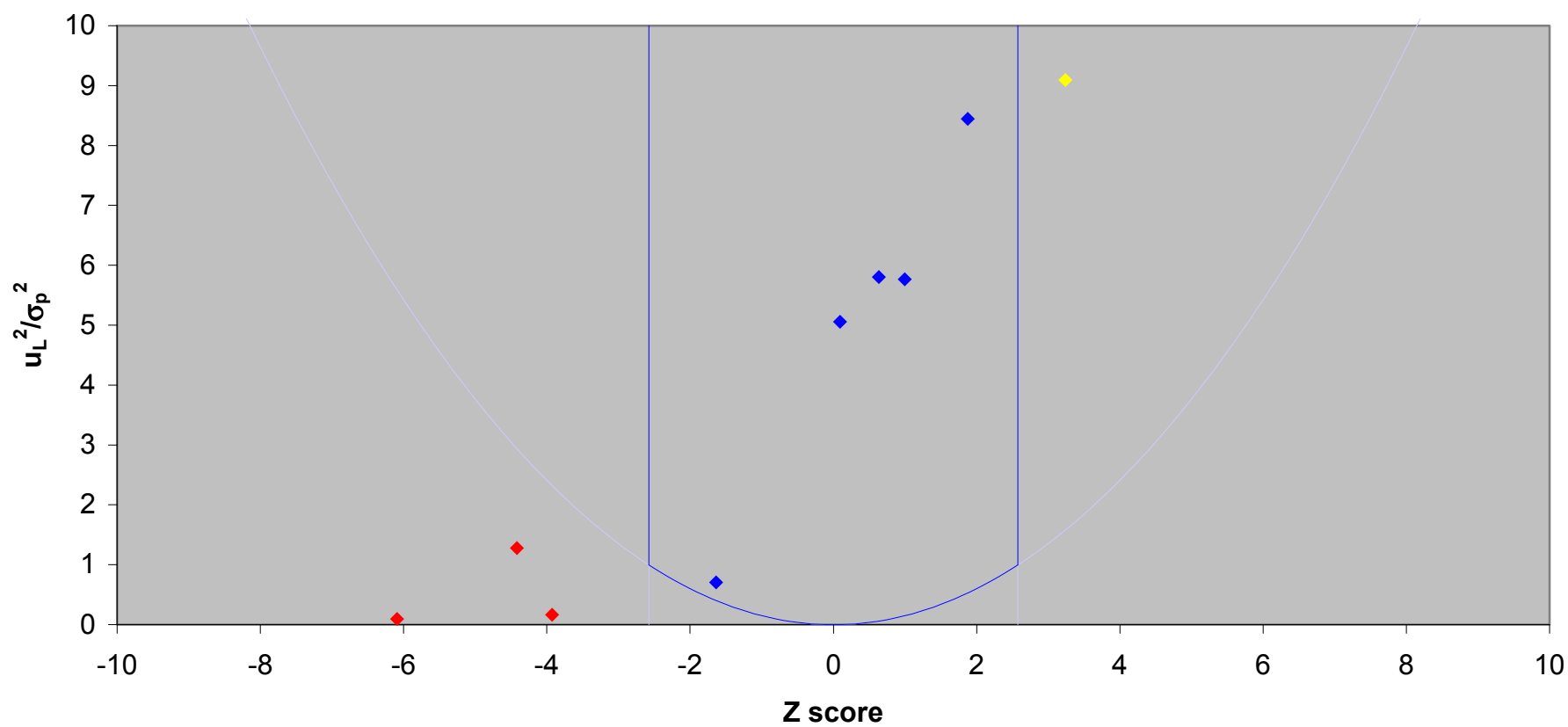


Figure 33 - Deviation plot for ^{60}Co results for Drum B
(hot volume not known)

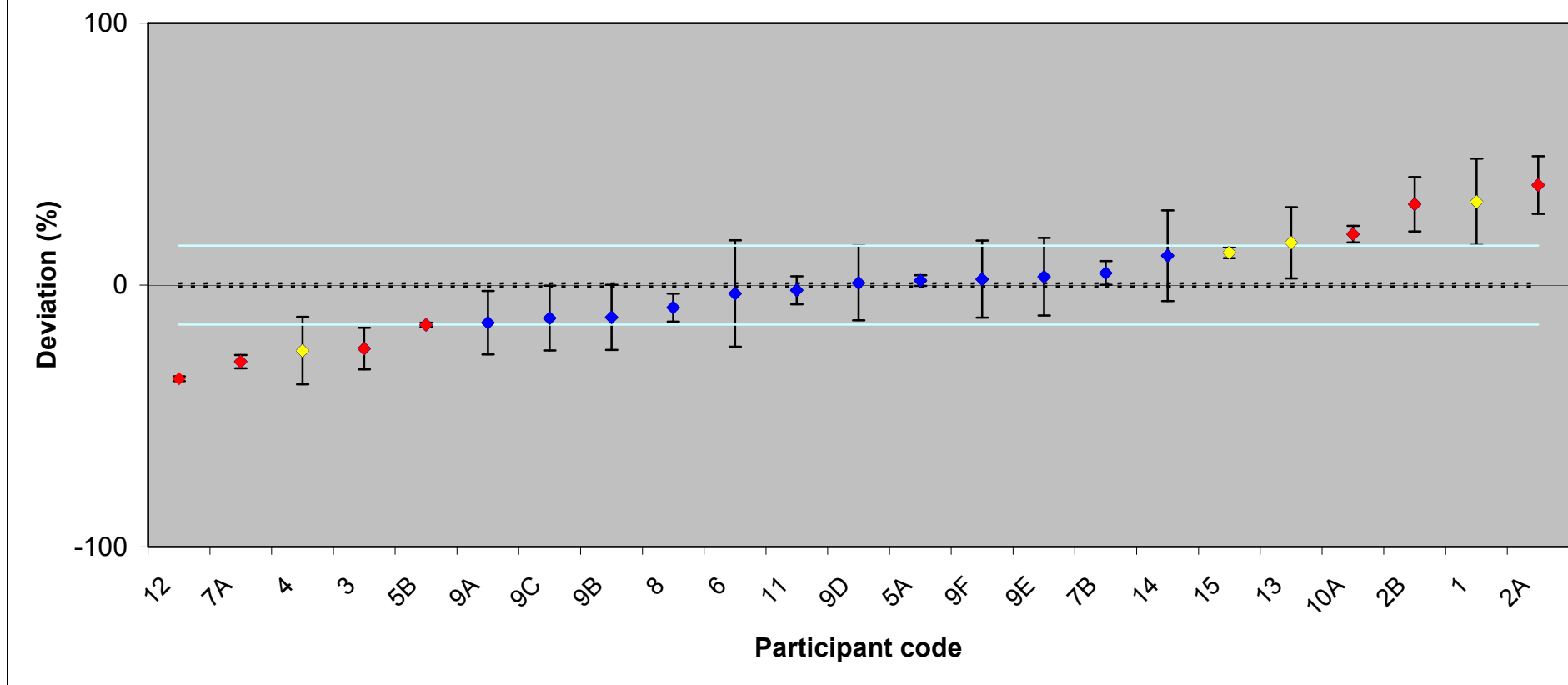


Figure 34 - Zeta-score plot for ^{60}Co results for Drum B
(hot volume not known)

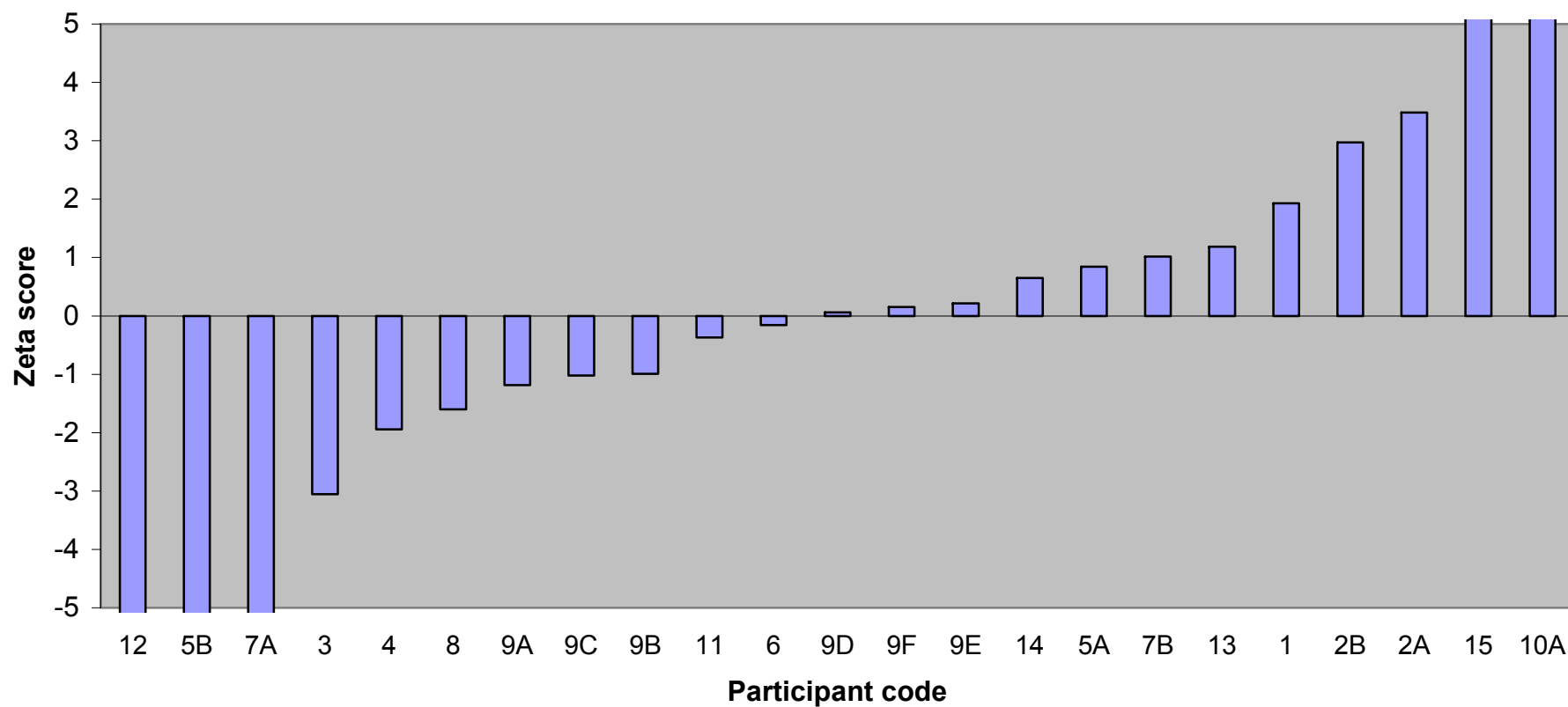


Figure 35 - Relative uncertainty plot for ^{60}Co results for Drum B
(hot volume not known)

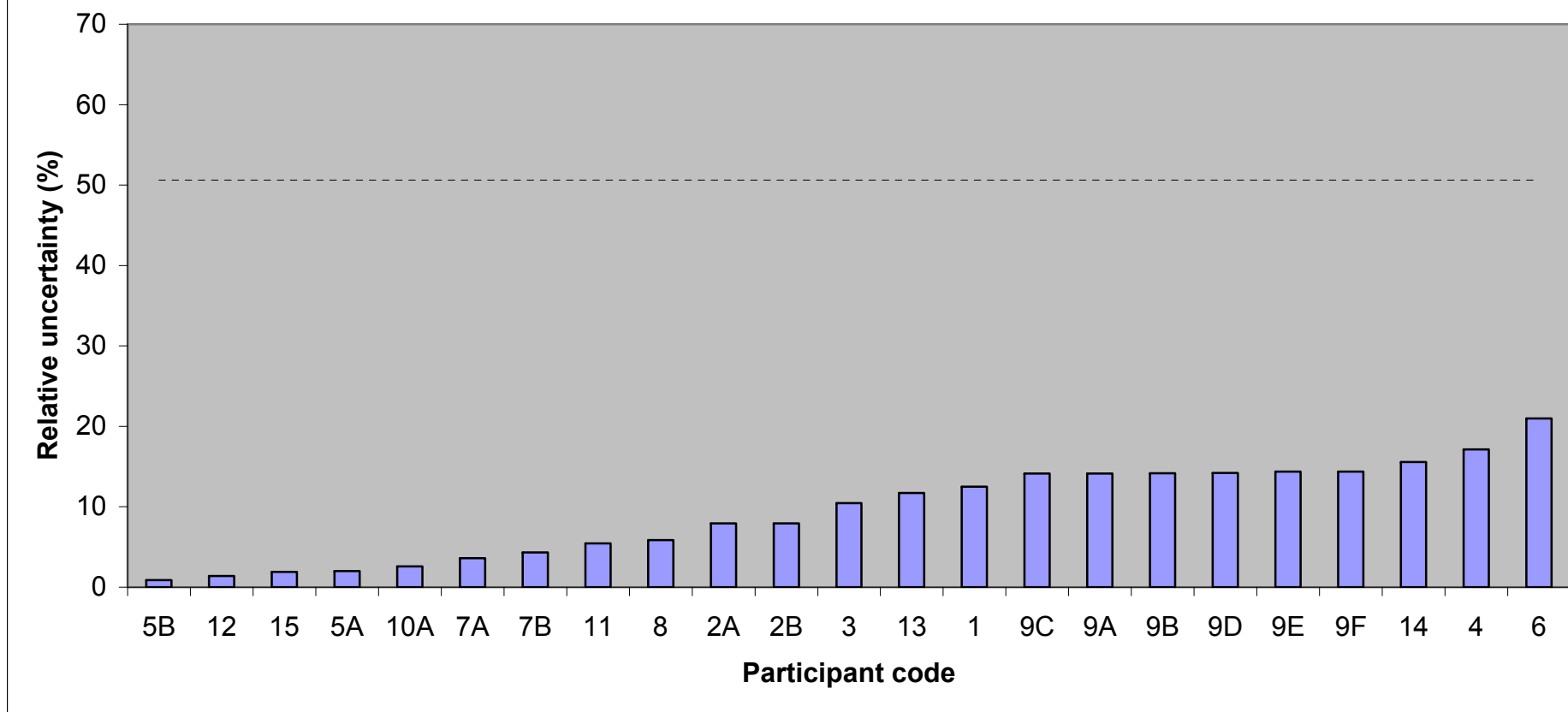


Figure 36 - 'Kiri' plot for ^{60}Co results for Drum B
(hot volume not known)

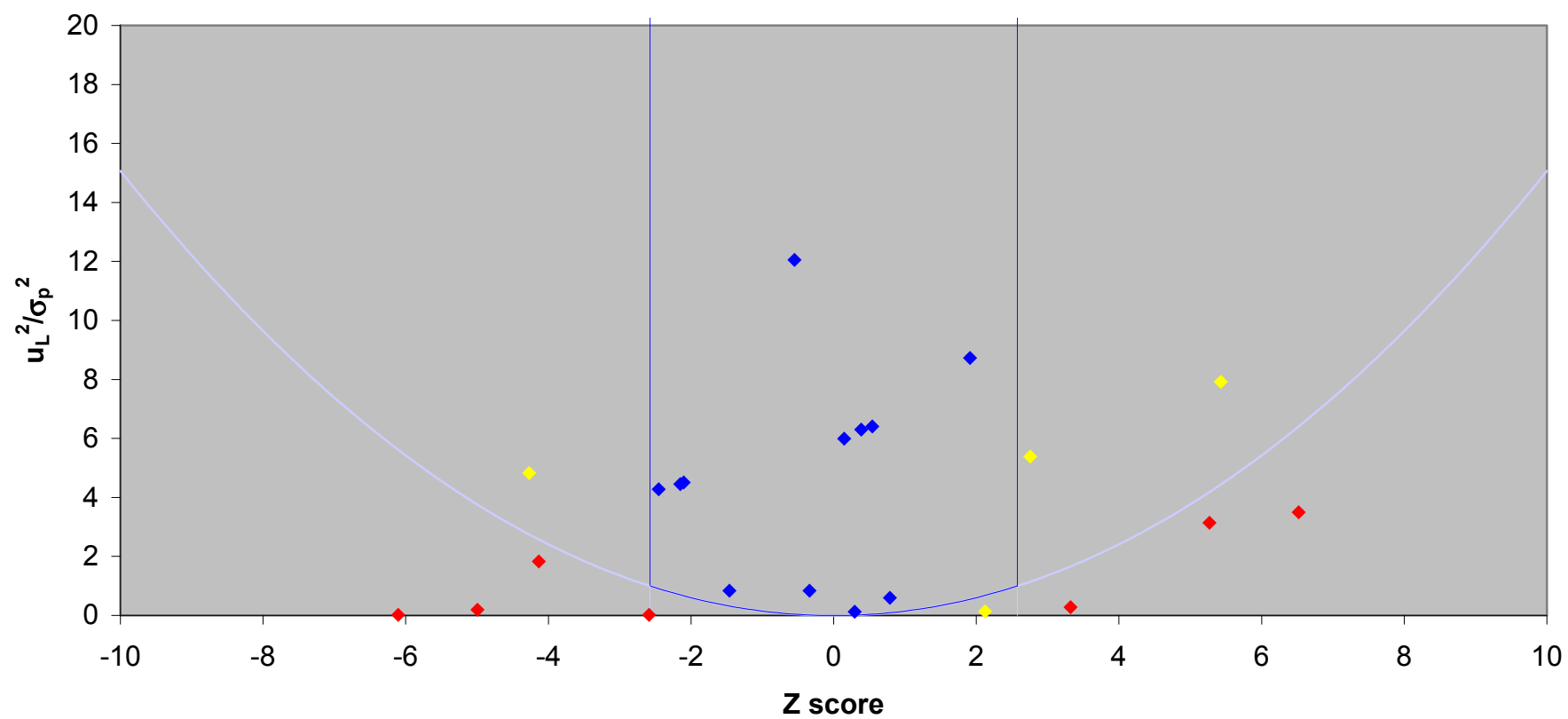


Figure 37 - Deviation plot for ^{60}Co results for Drum B
(hot volume known)

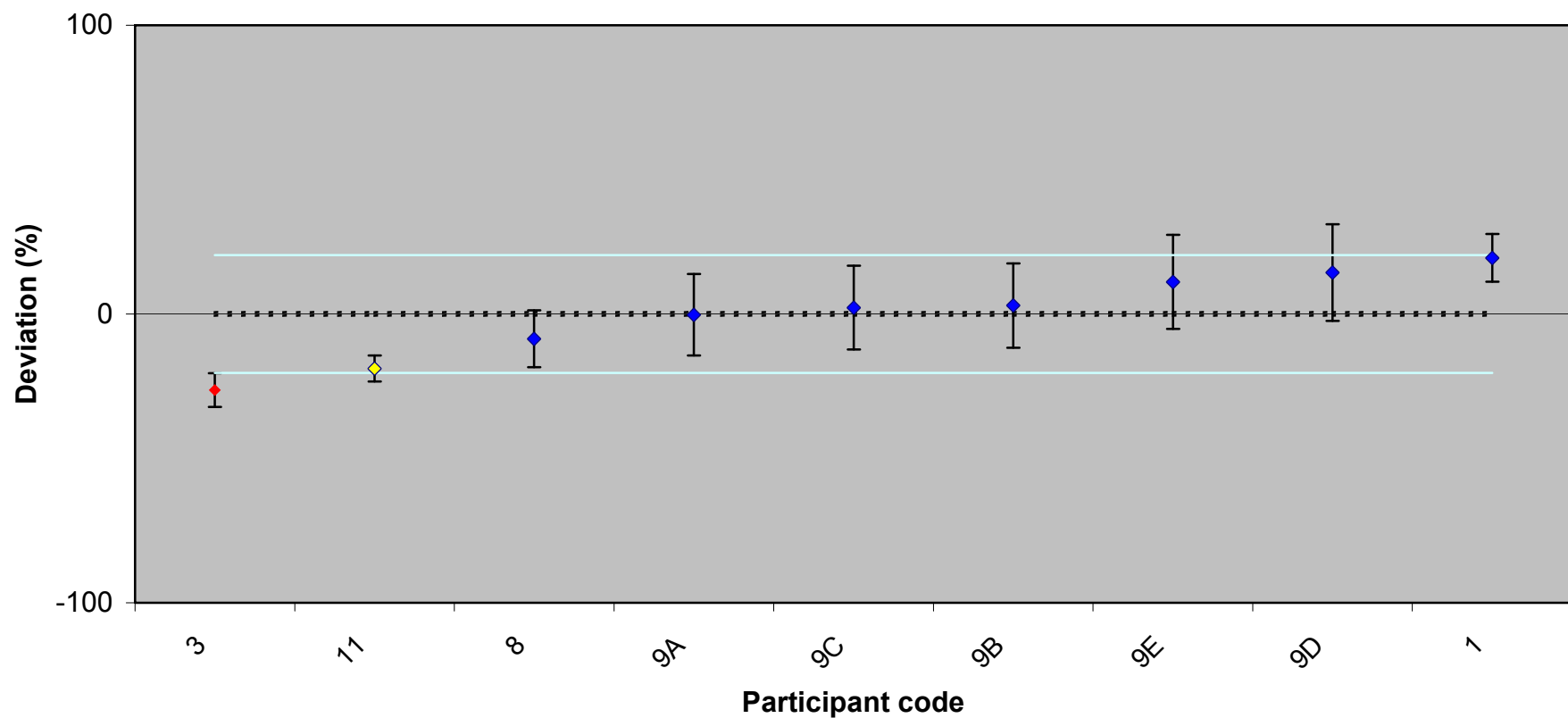


Figure 38 - Zeta-score plot for ^{60}Co results for Drum B
(hot volume known)

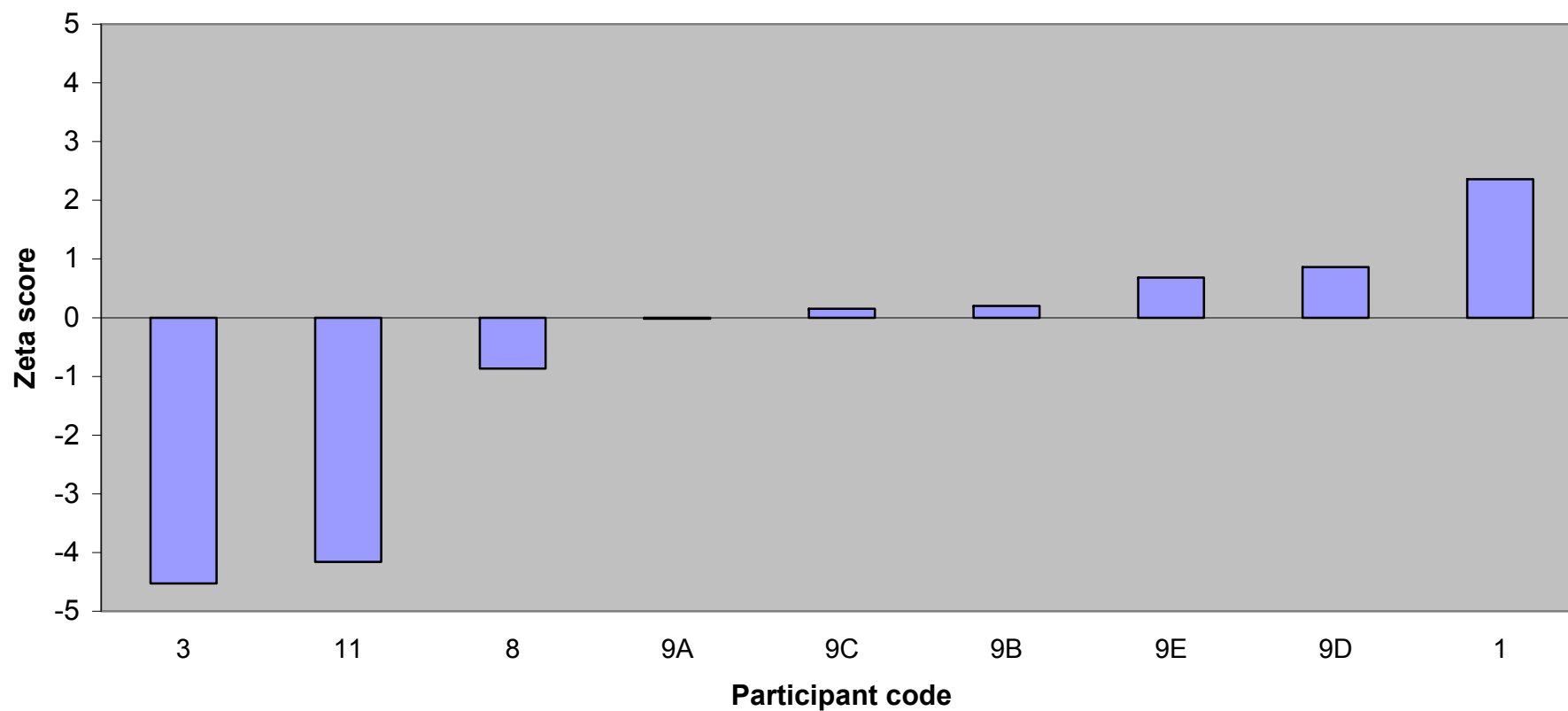


Figure 39 - Relative uncertainty plot for ^{60}Co results for Drum B
(hot volume known)

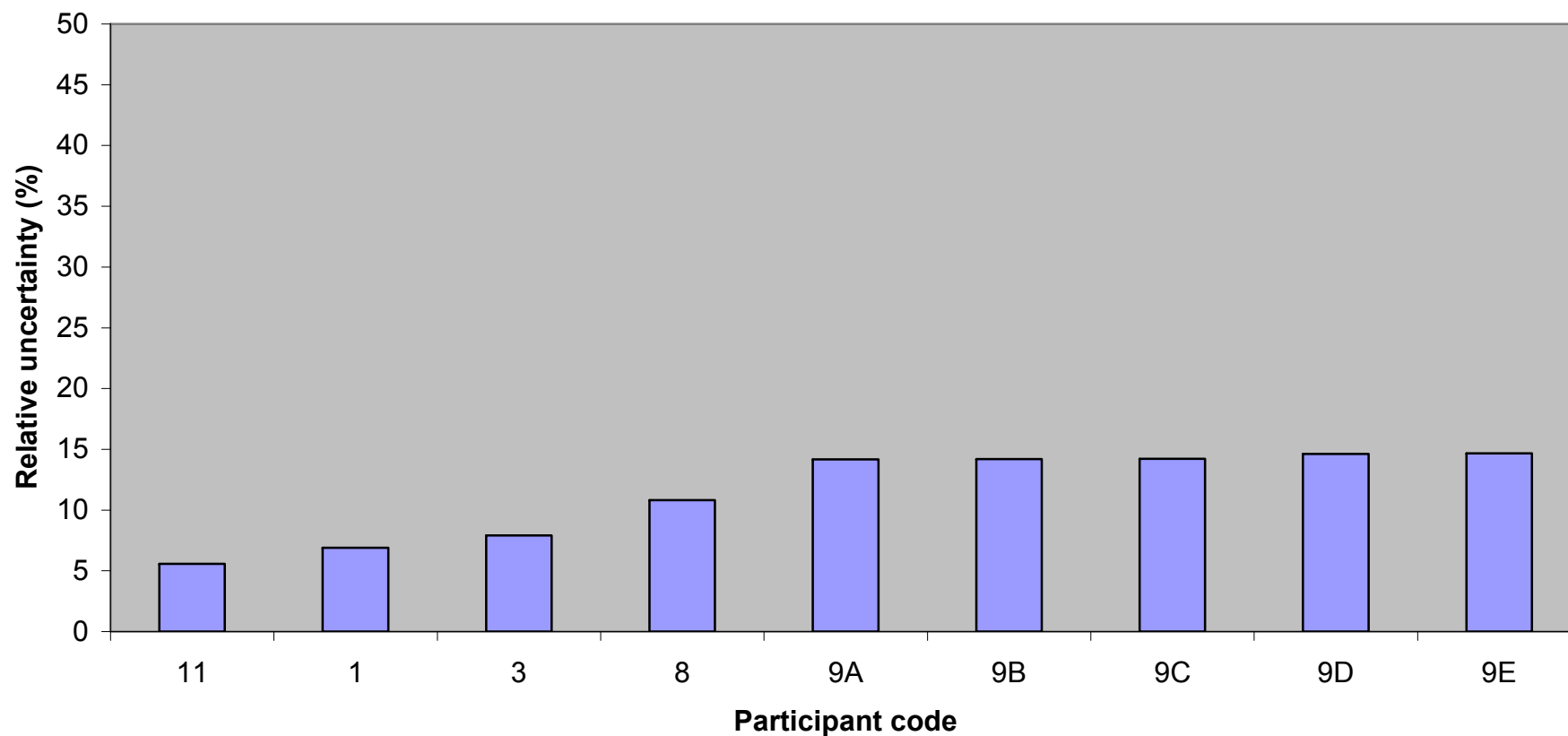


Figure 40 - 'Kiri' plot for ^{60}Co results for Drum B
(hot volume known)

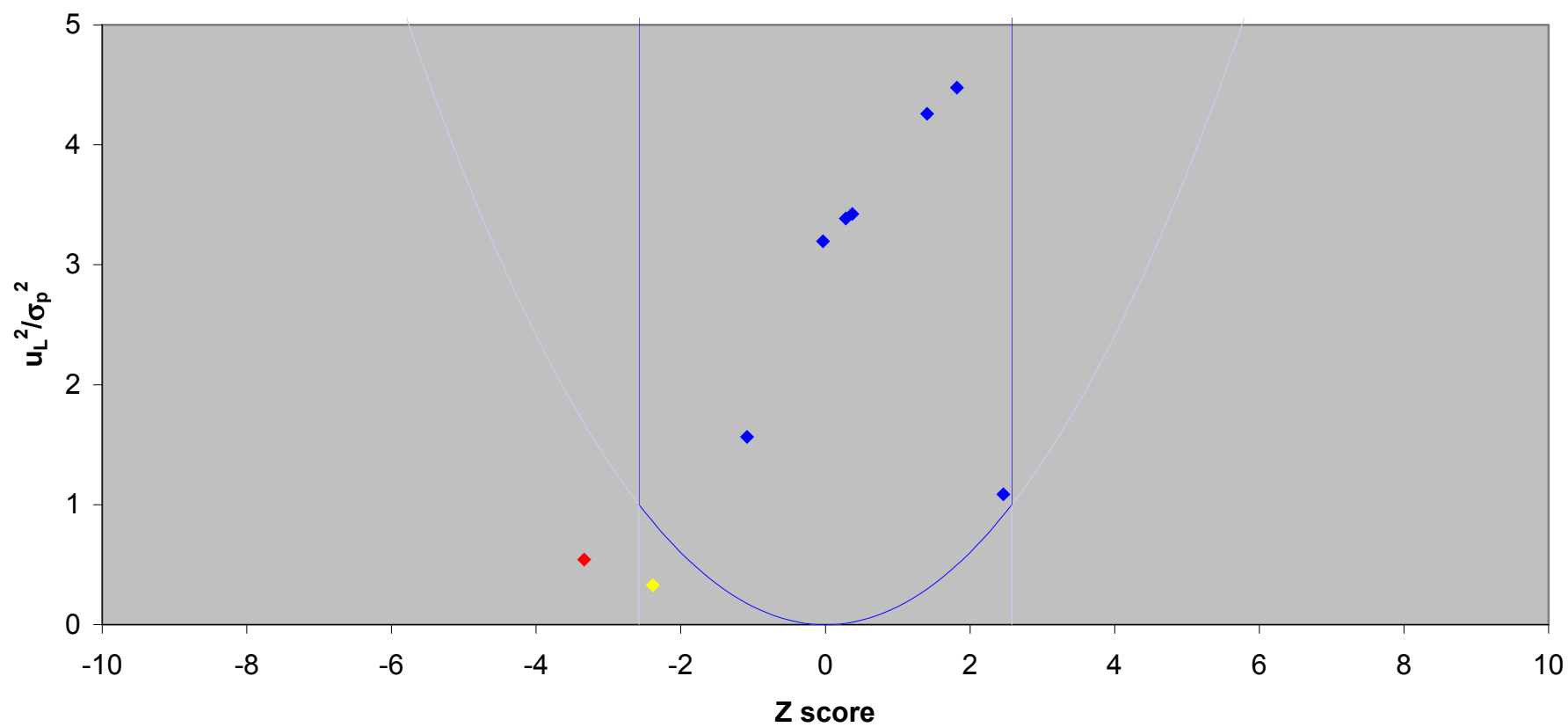


Figure 41 - Deviation plot for ^{137}Cs results for Drum B
(hot volume not known)

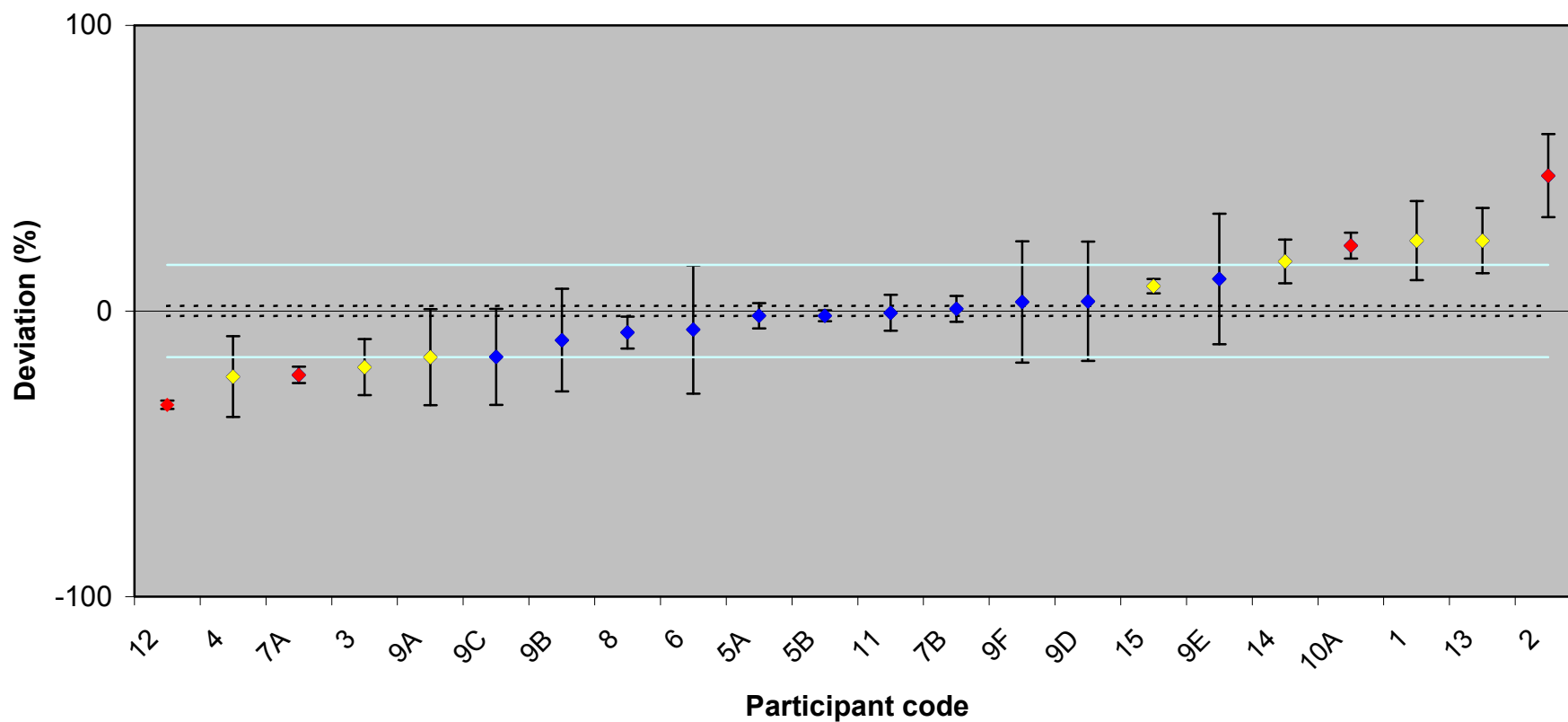


Figure 42 - Zeta-score plot for ^{137}Cs results for Drum B
(hot volume not known)

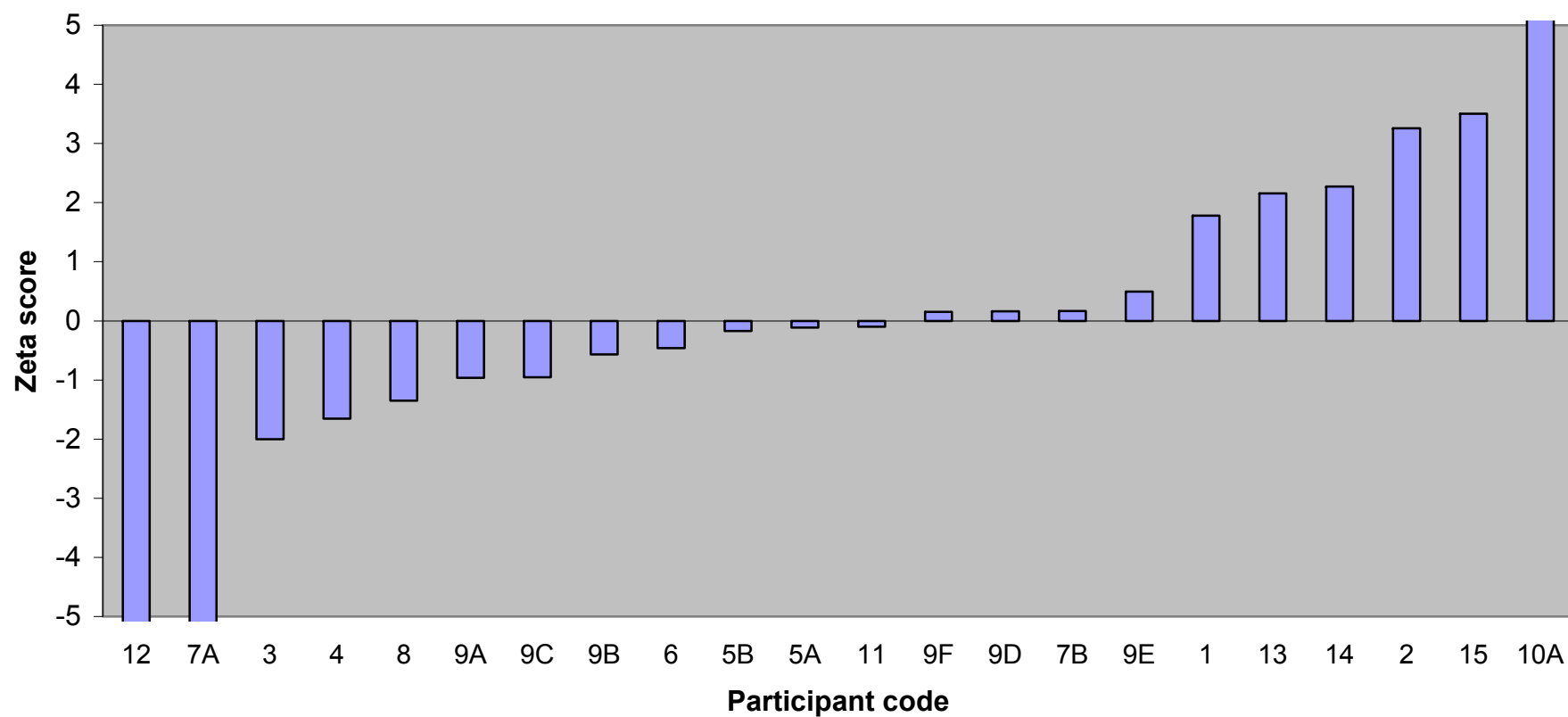


Figure 43 - Relative uncertainty plot for ^{137}Cs results for Drum B
(hot volume not known)

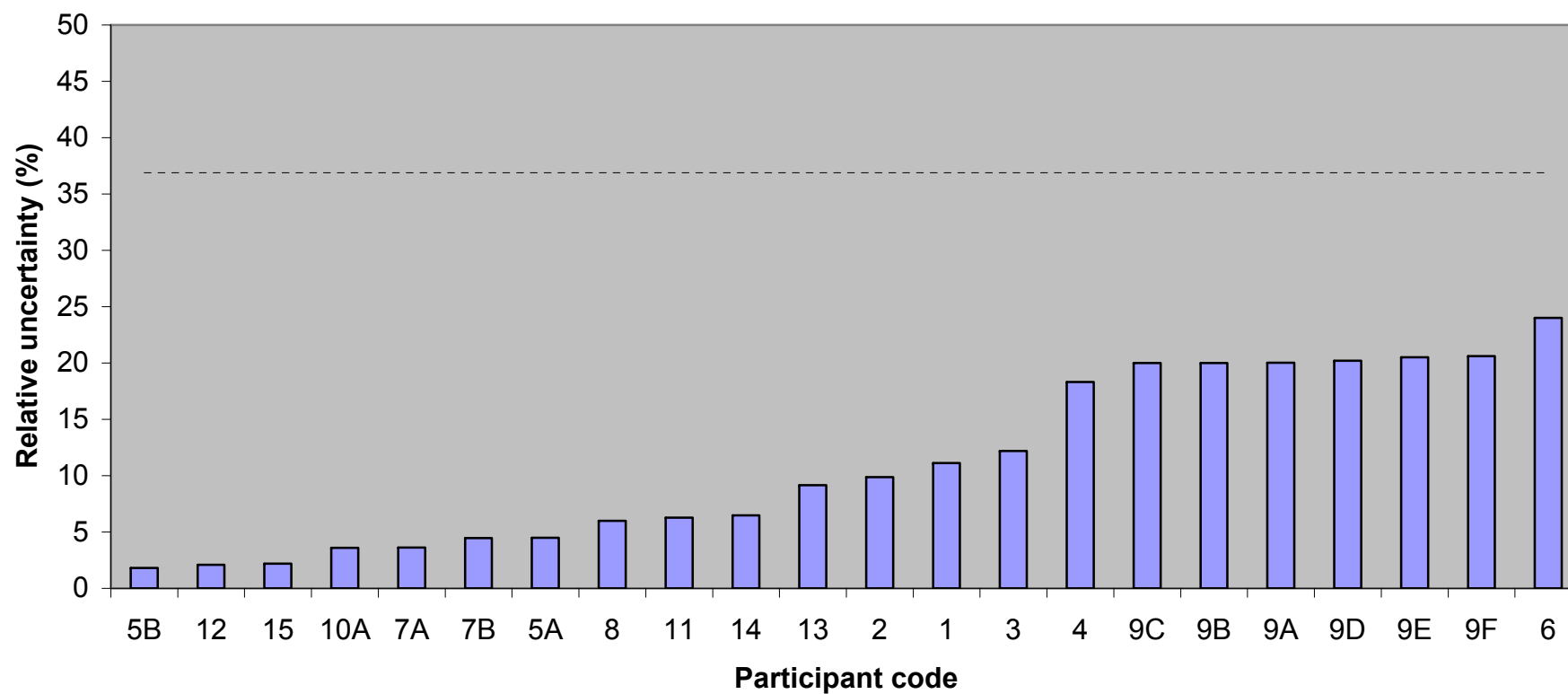


Figure 44 - 'Kiri' plot for ^{137}C results for Drum B
(hot volume not known)

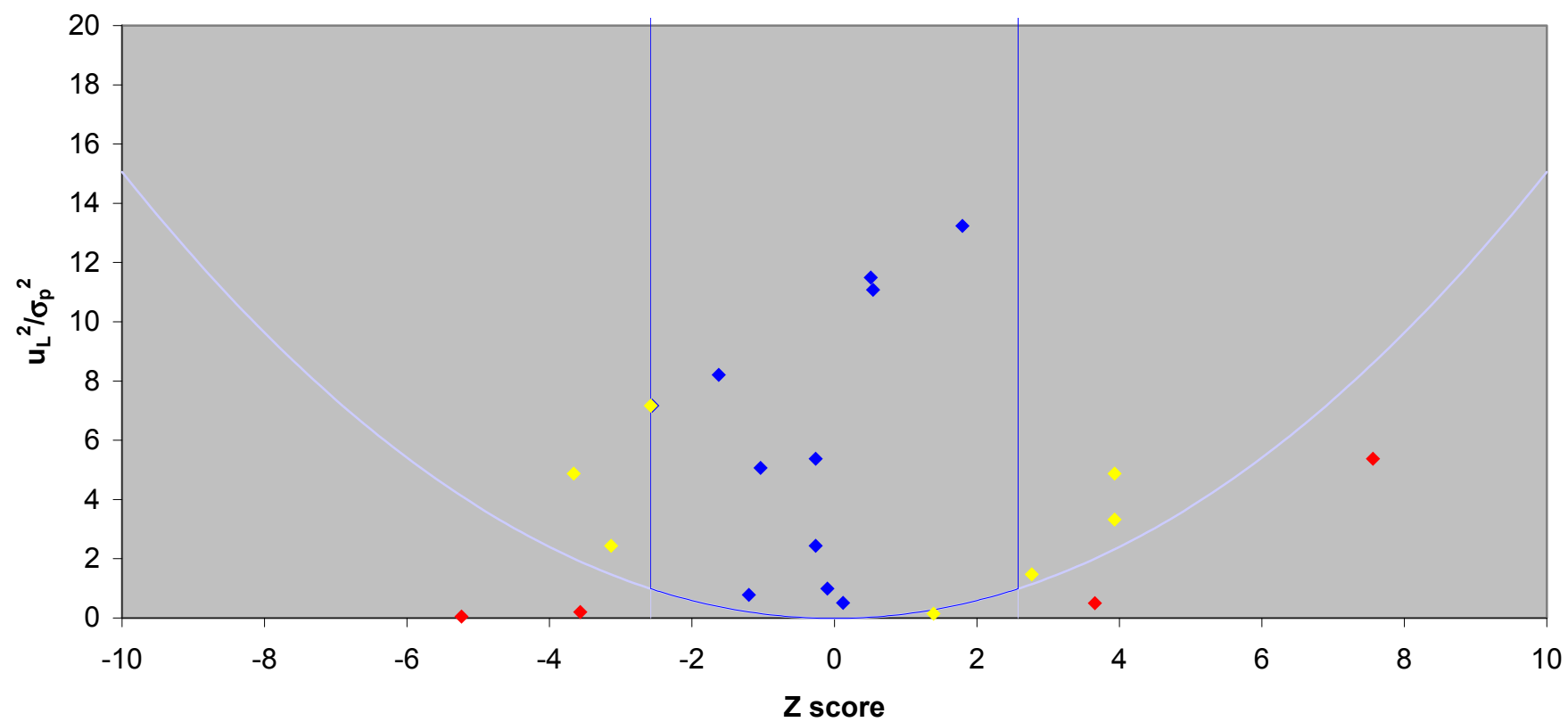


Figure 45 - Deviation plot for ^{137}Cs results for Drum B
(hot volume known)

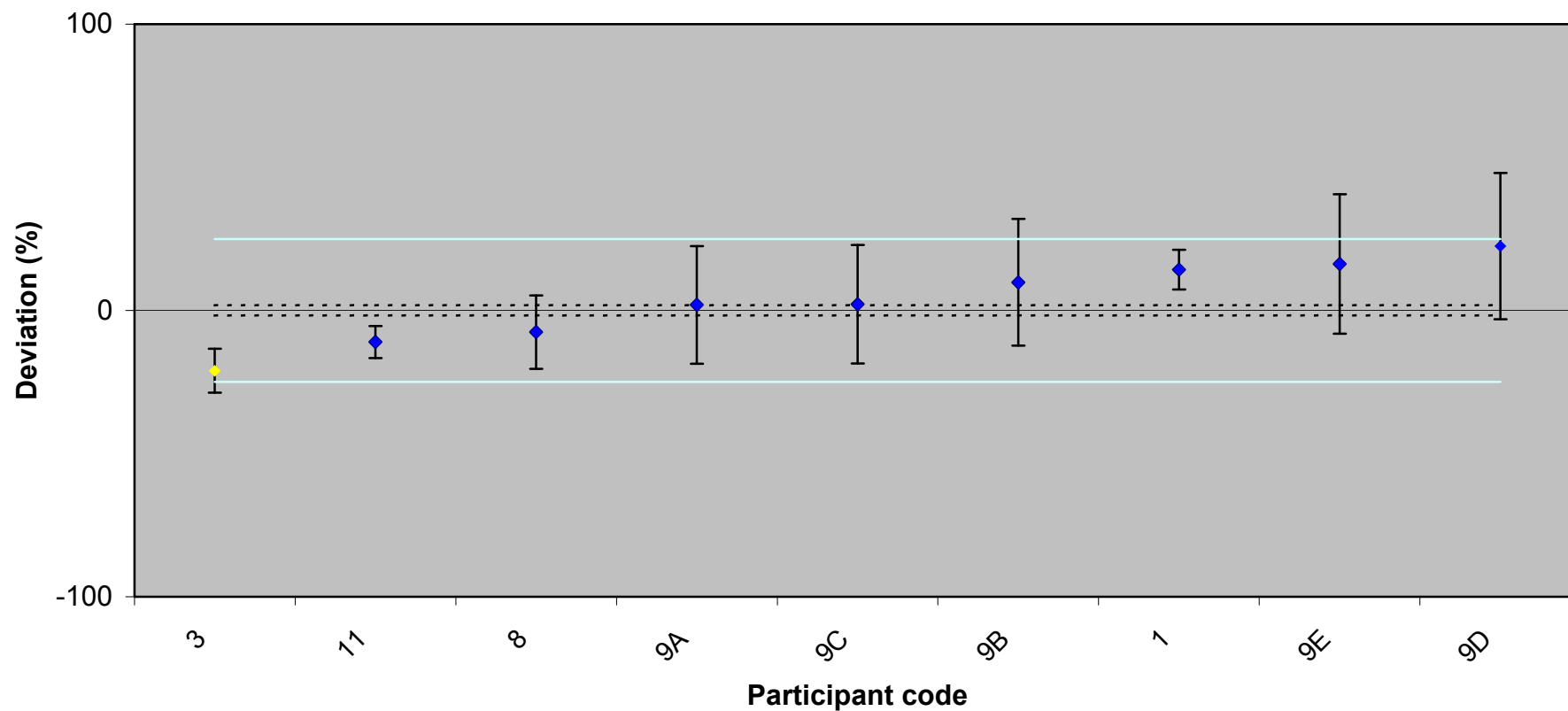


Figure 46 - Zeta-score plot for ^{137}Cs results for Drum B
(hot volume known)

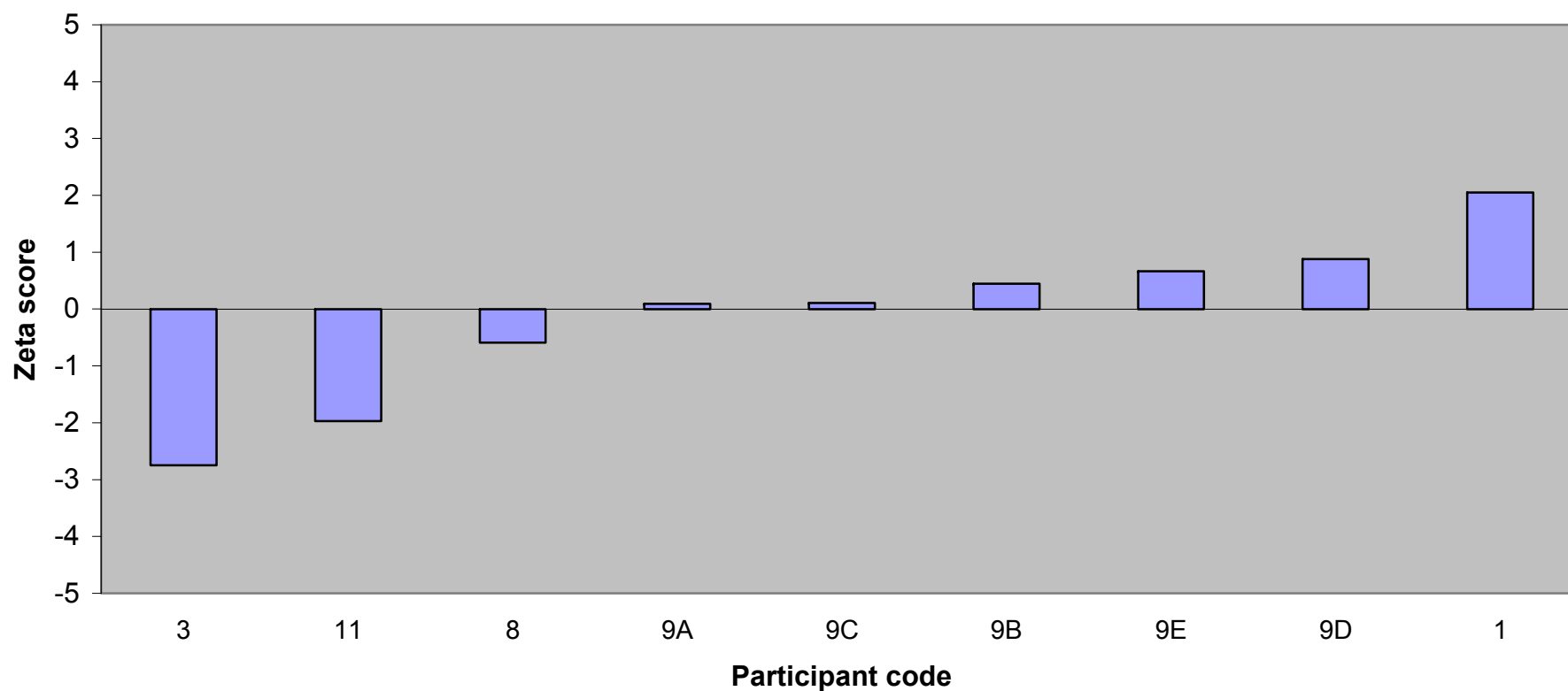


Figure 47 - Relative uncertainty plot for ^{137}Cs results for Drum B
(hot volume known)

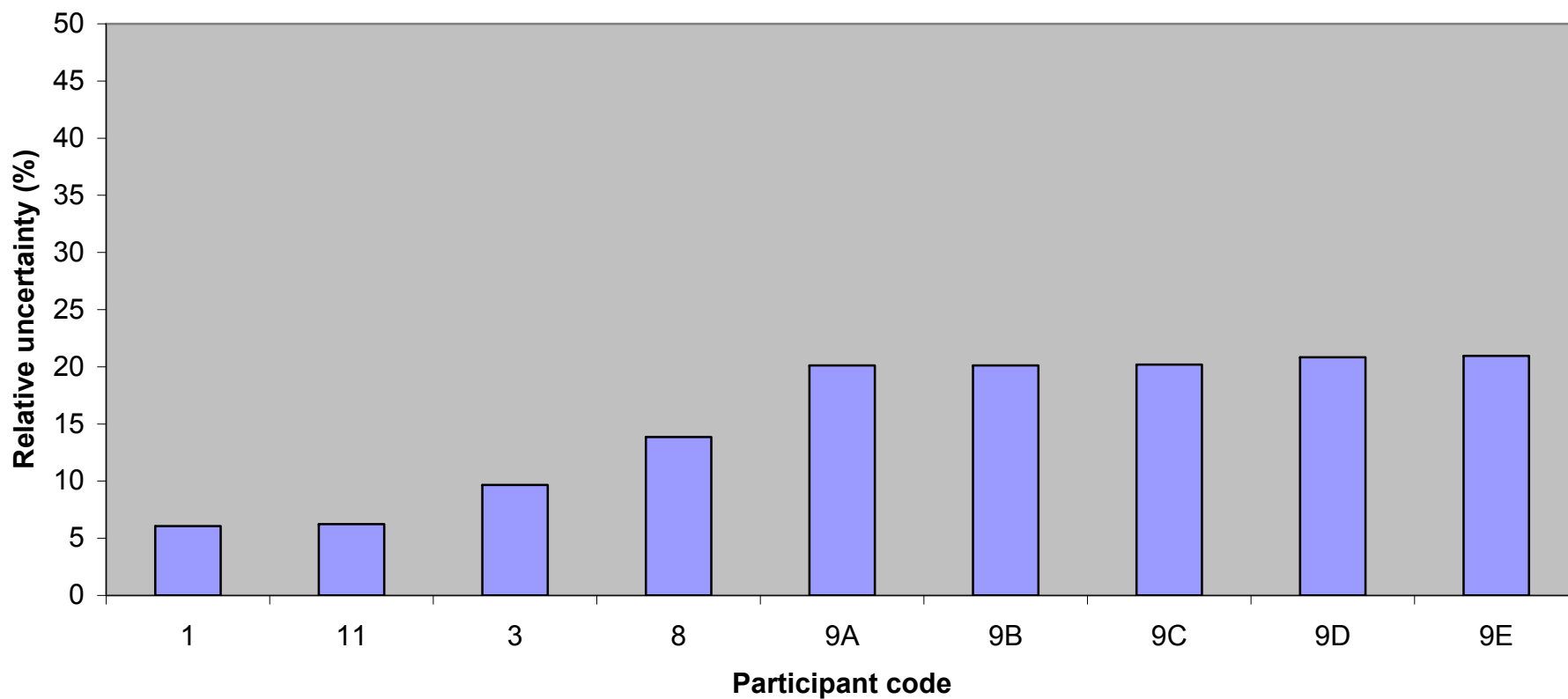
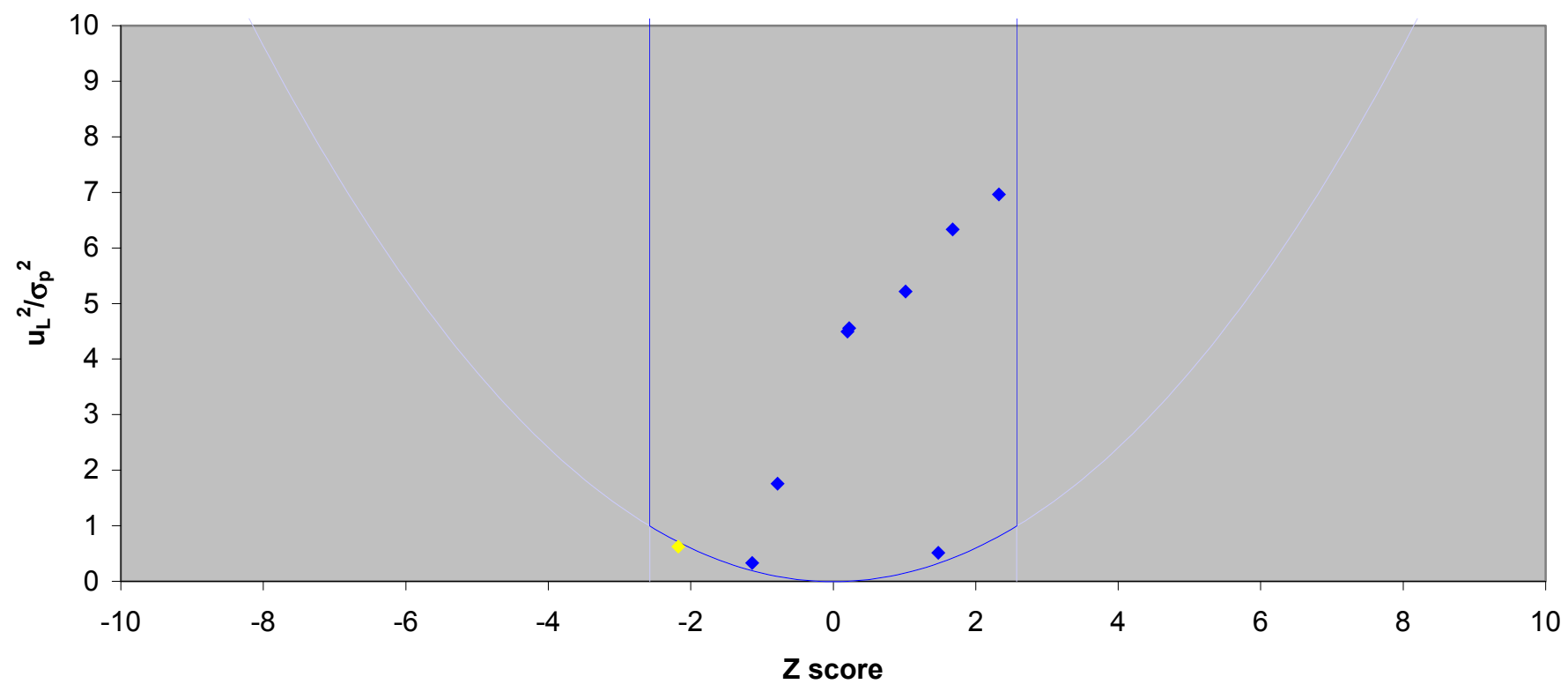
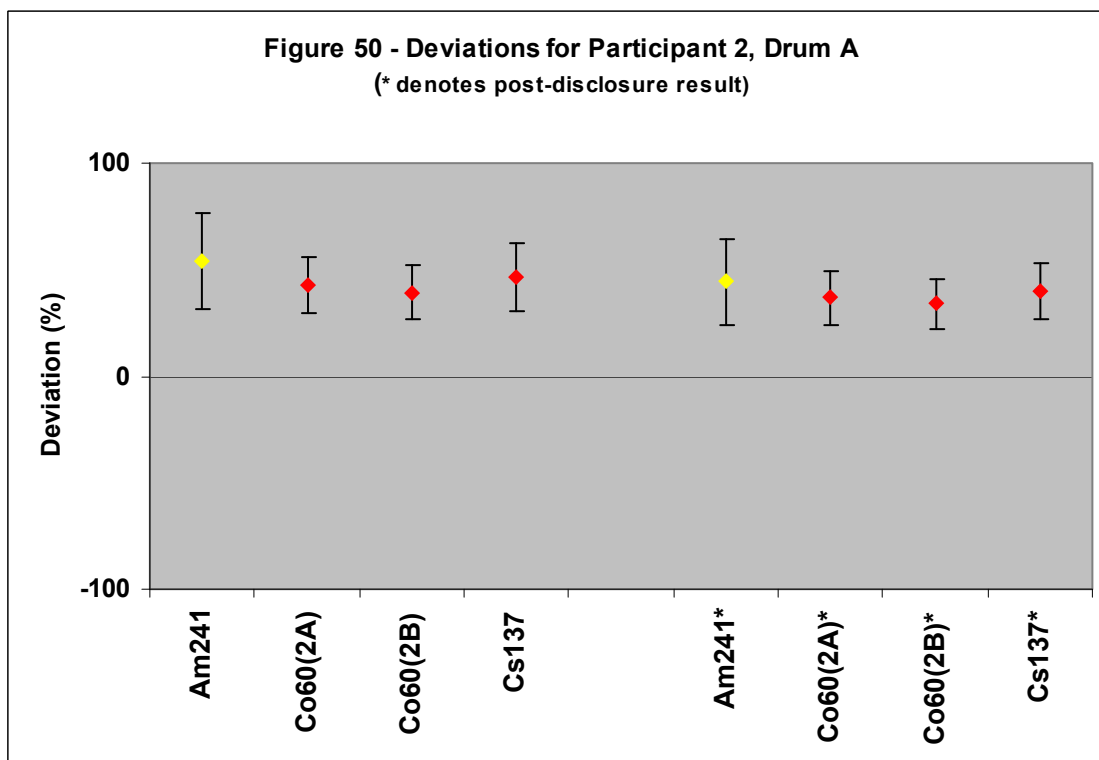
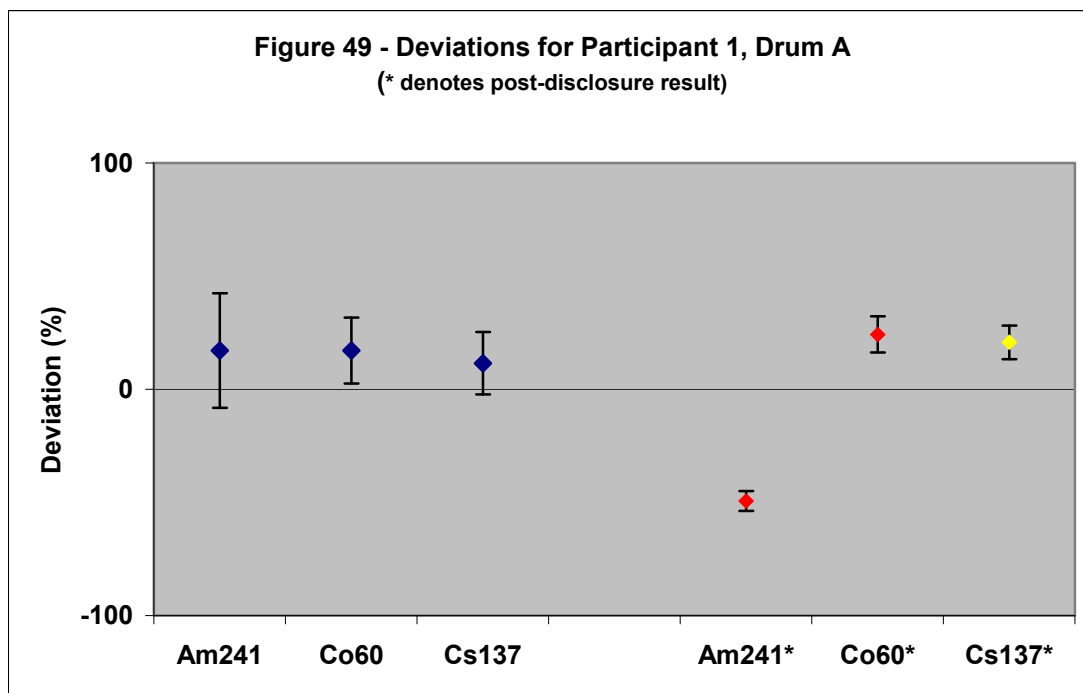
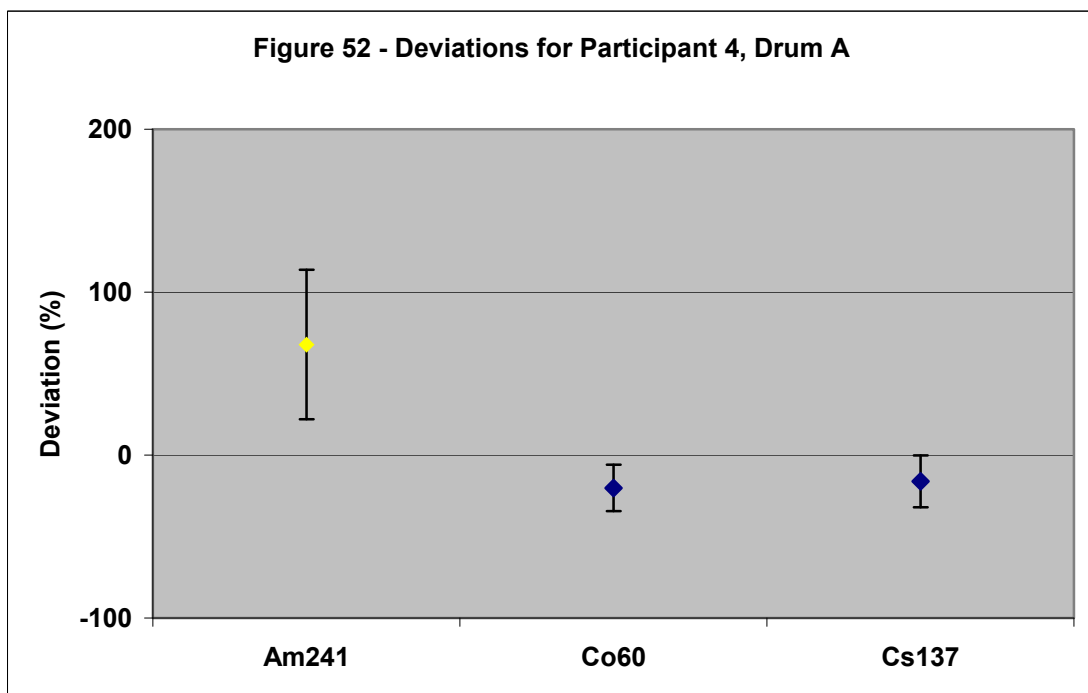
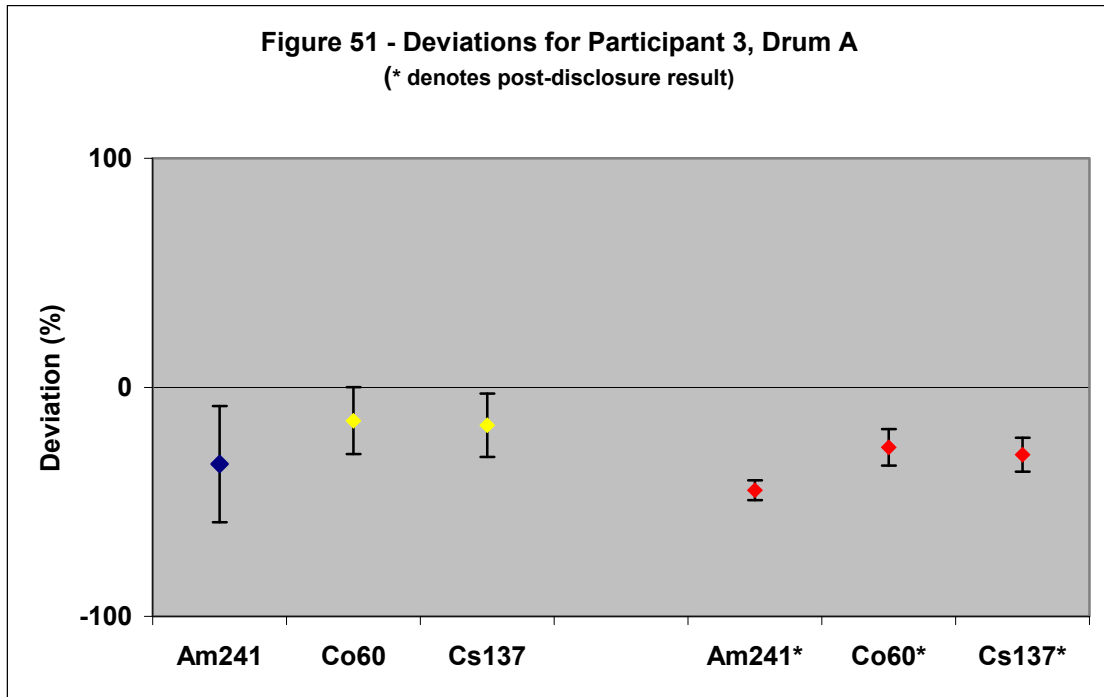
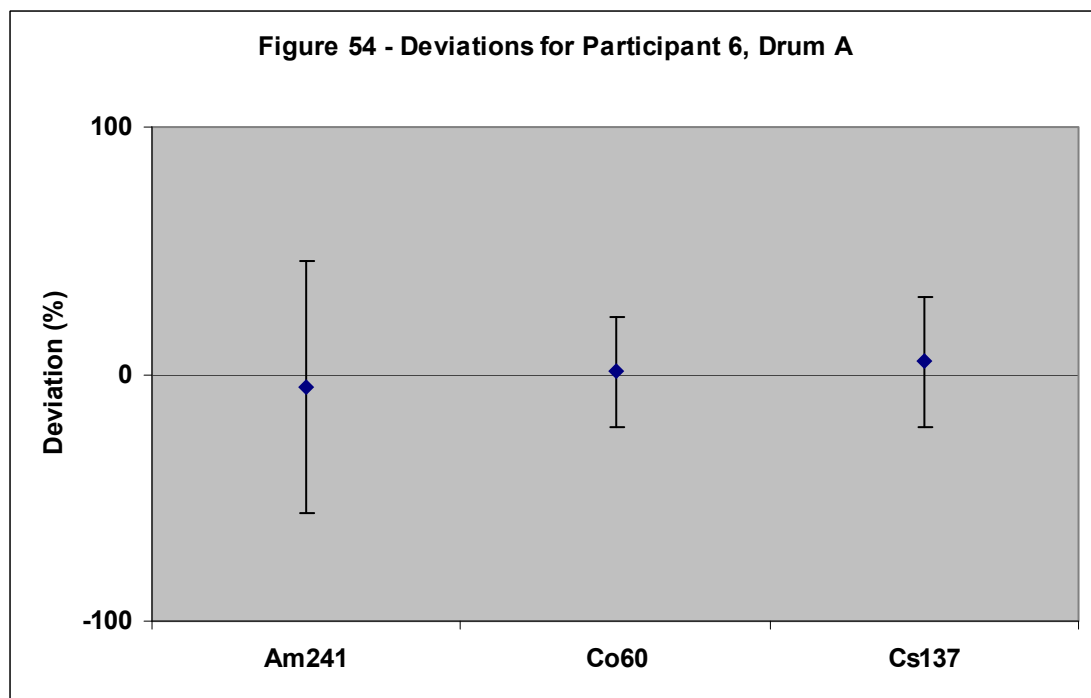
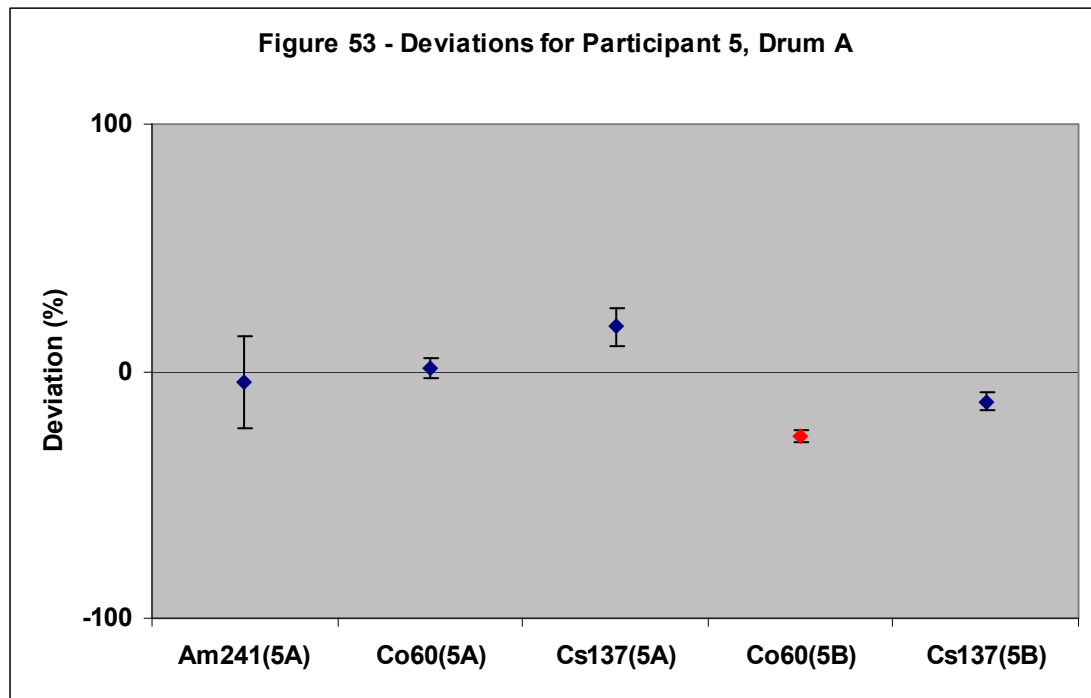


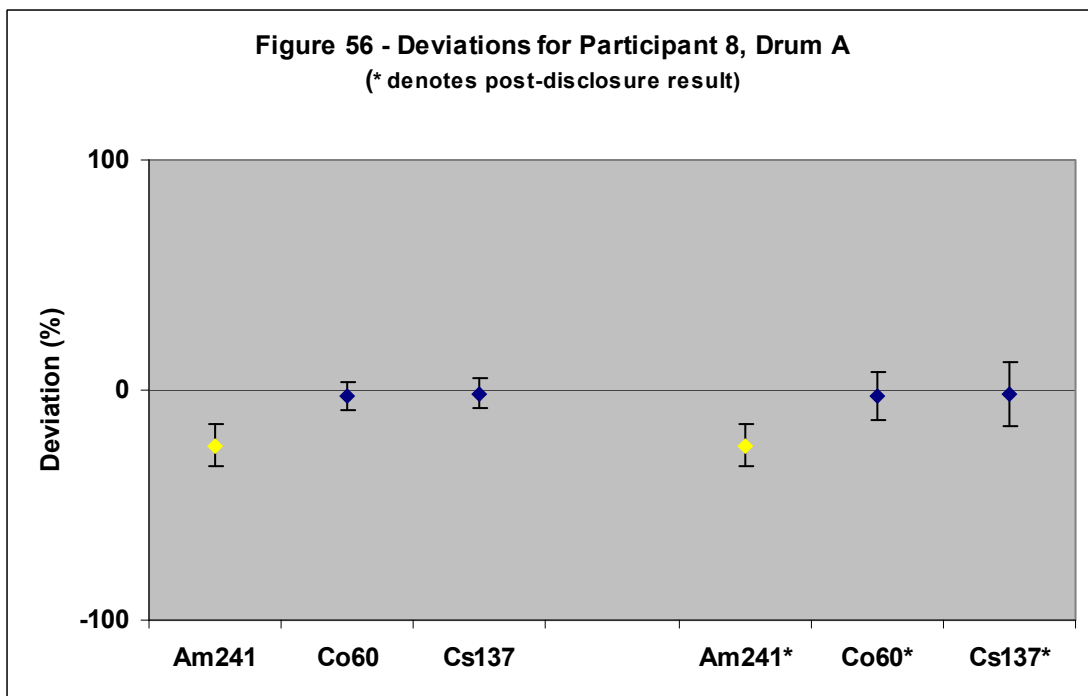
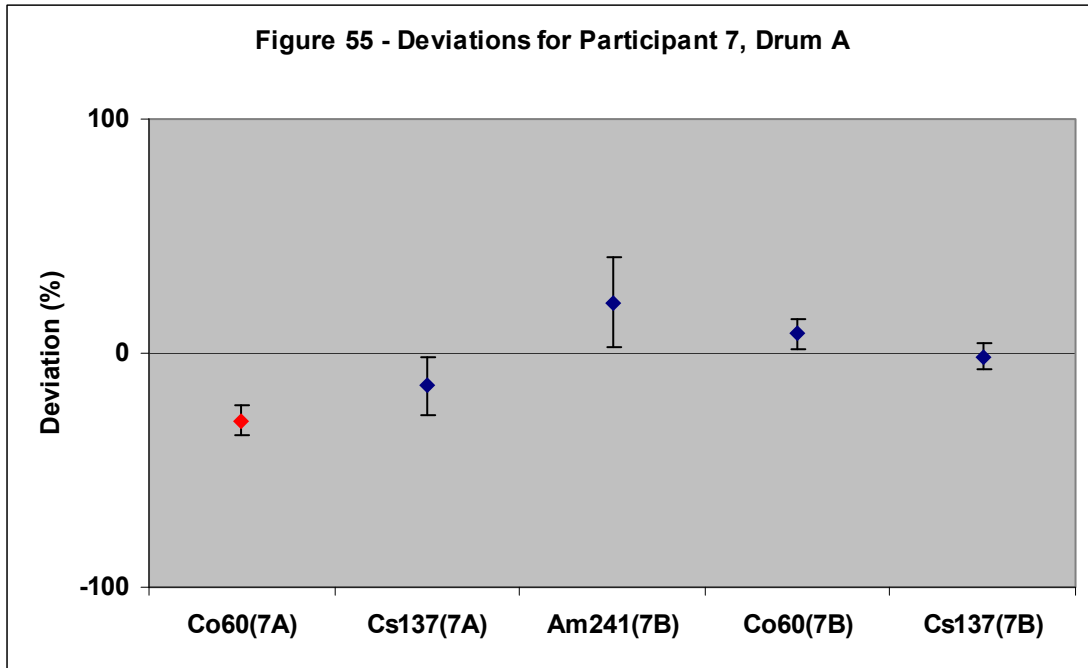
Figure 48 - 'Kiri' plot for ^{137}Cs results for Drum B
(hot volume known)

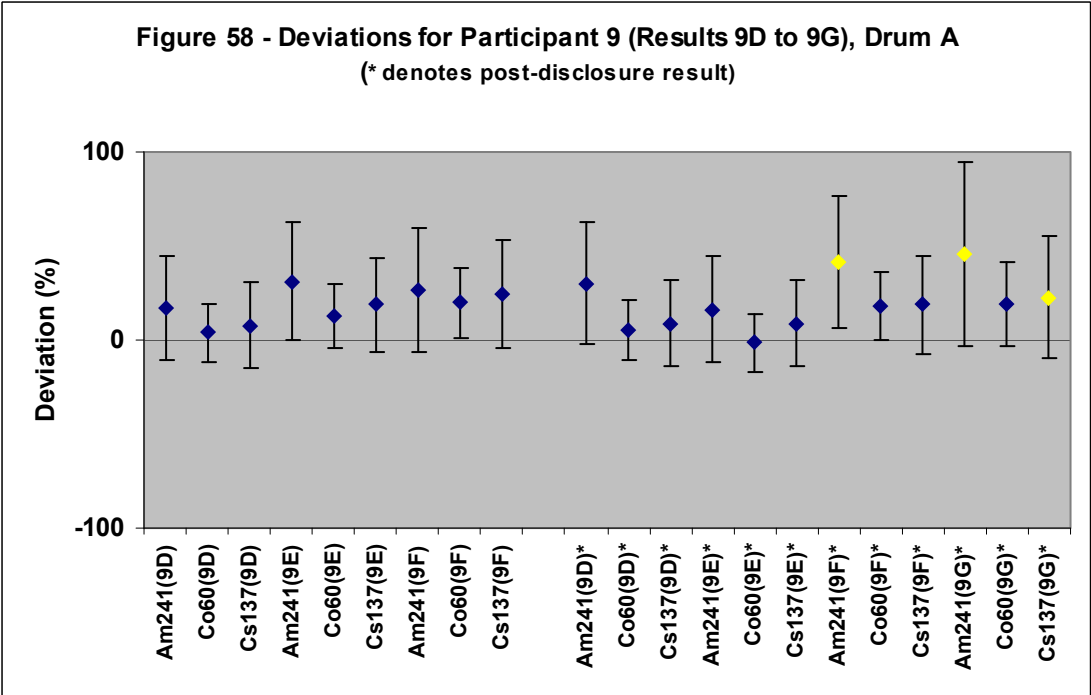
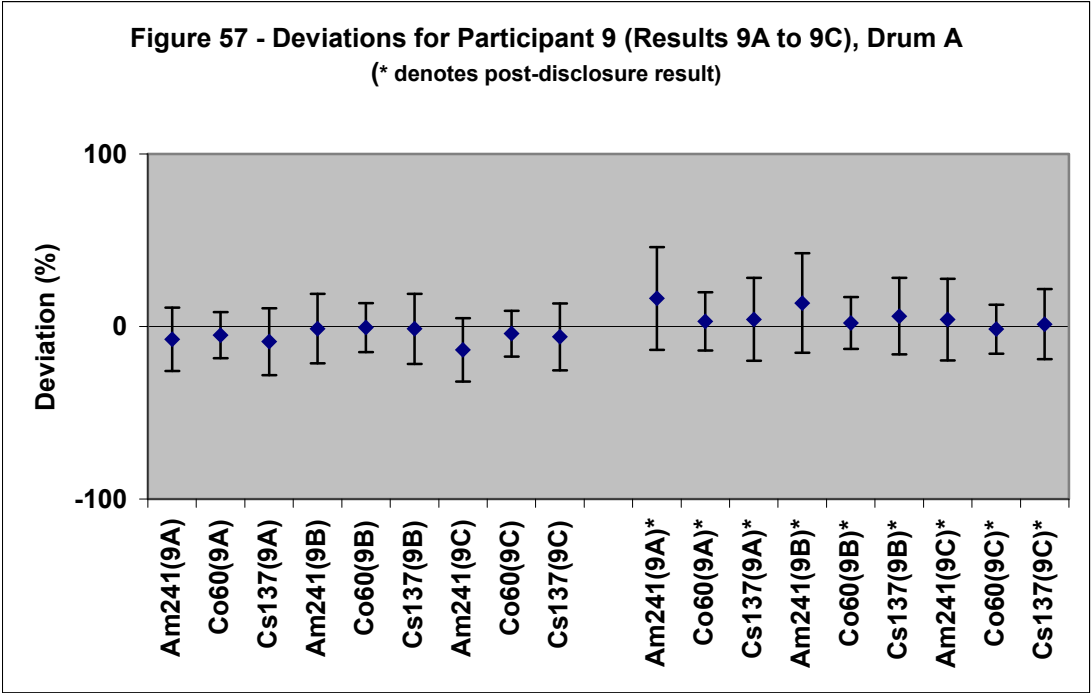


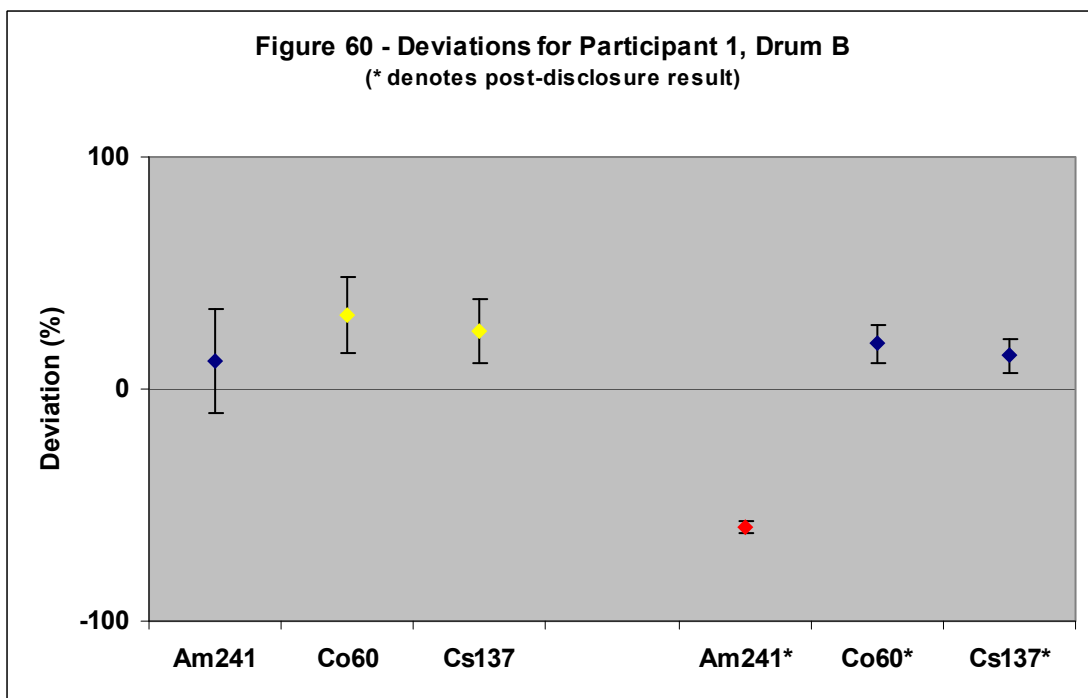
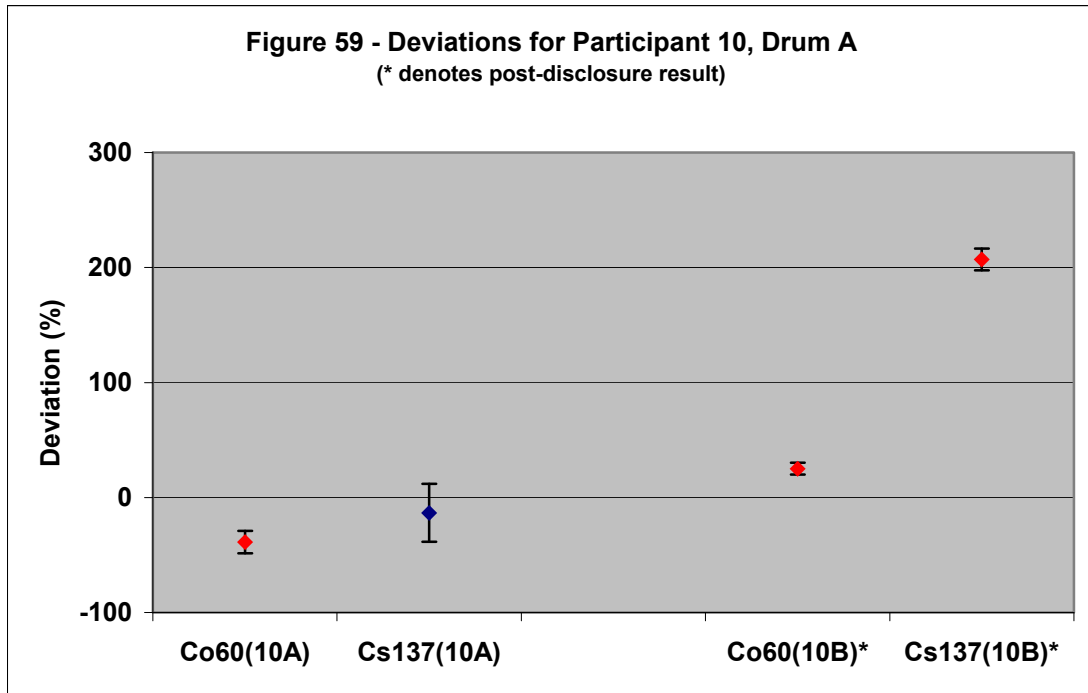


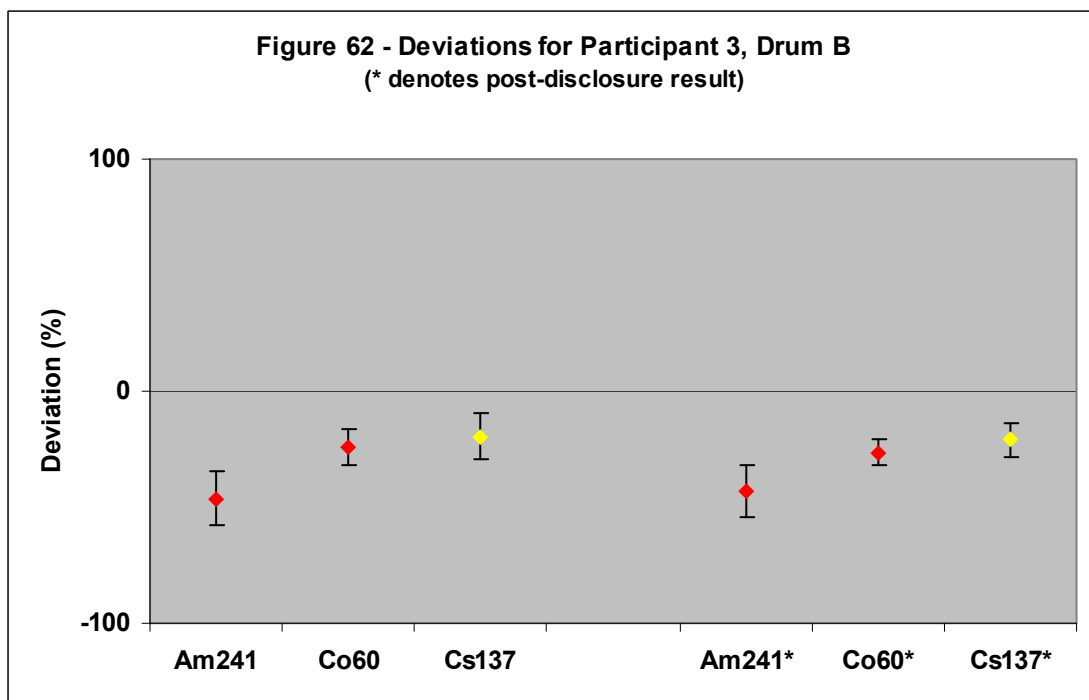
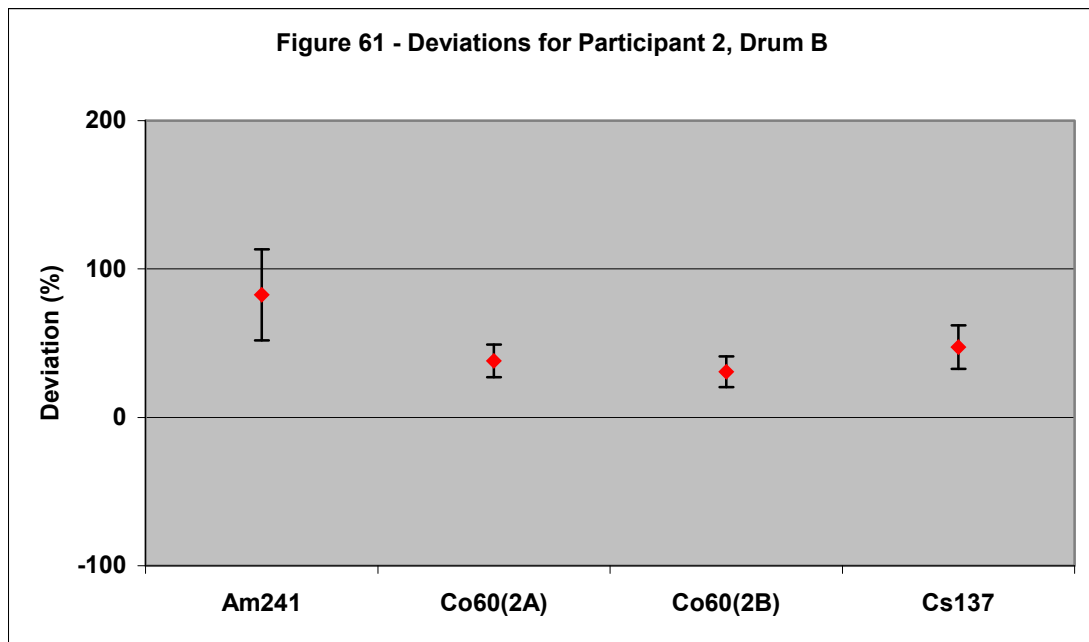


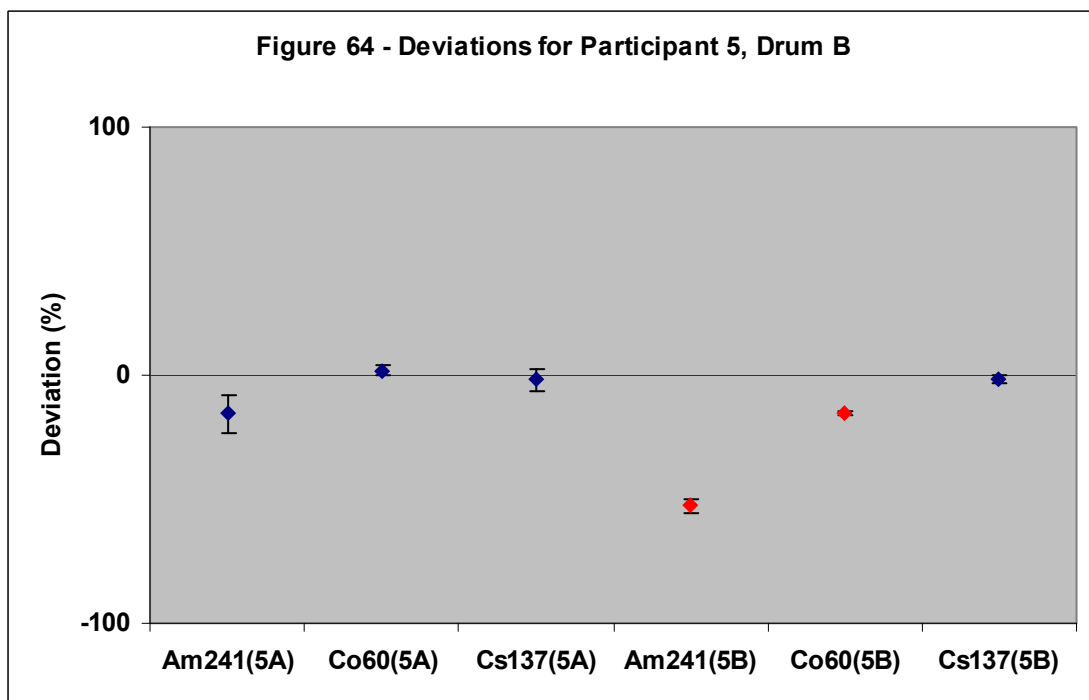
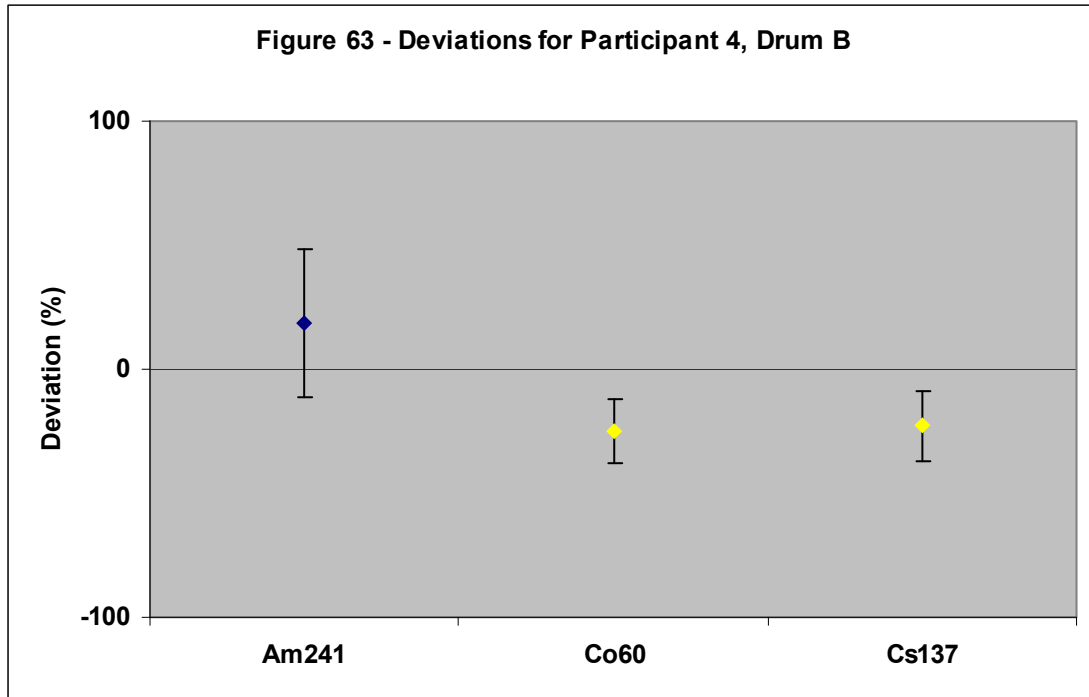


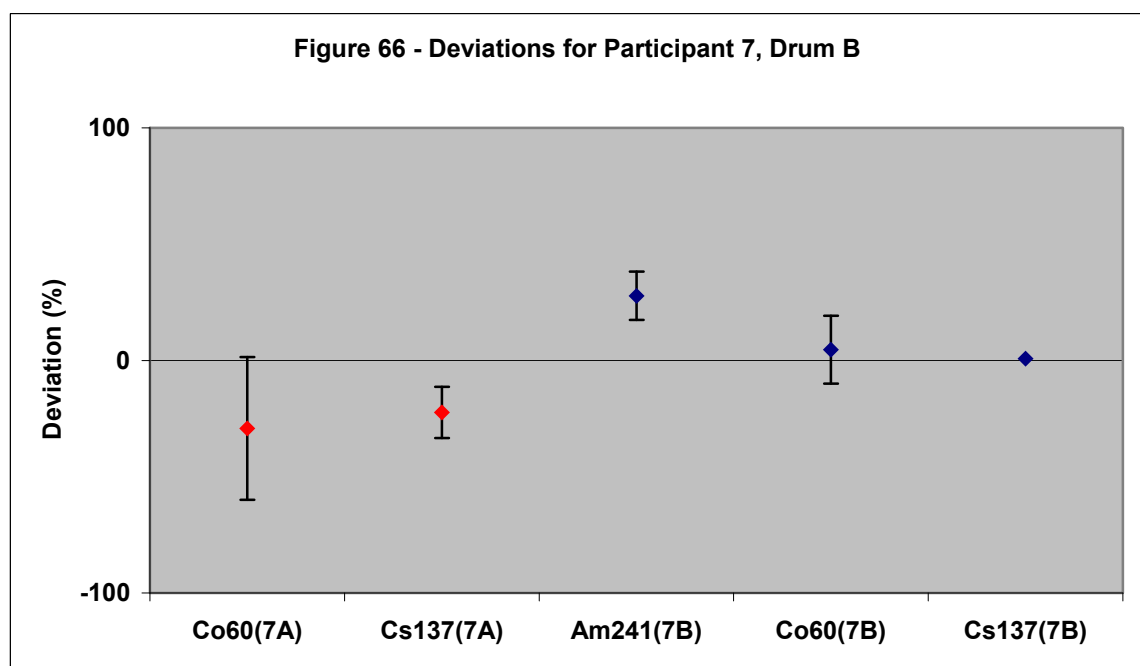
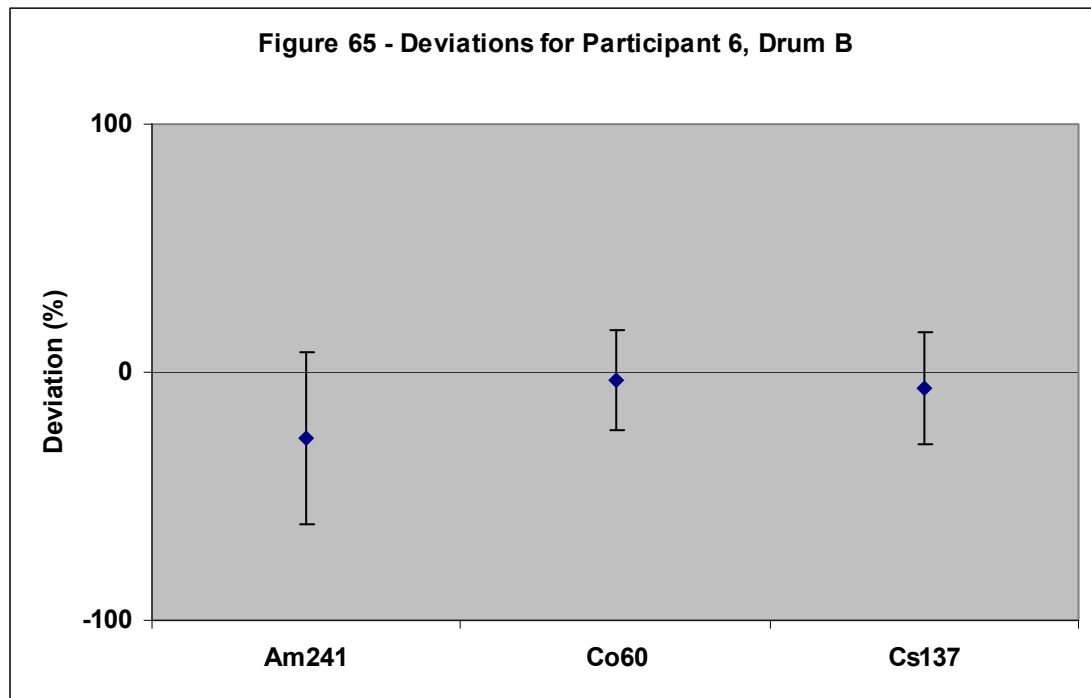


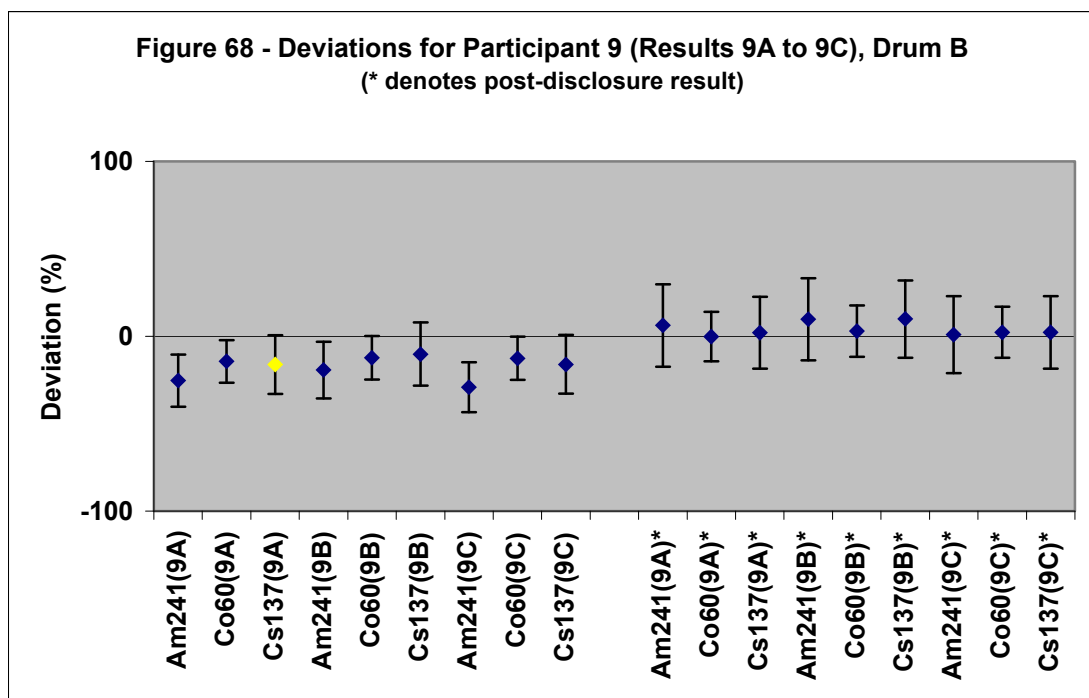
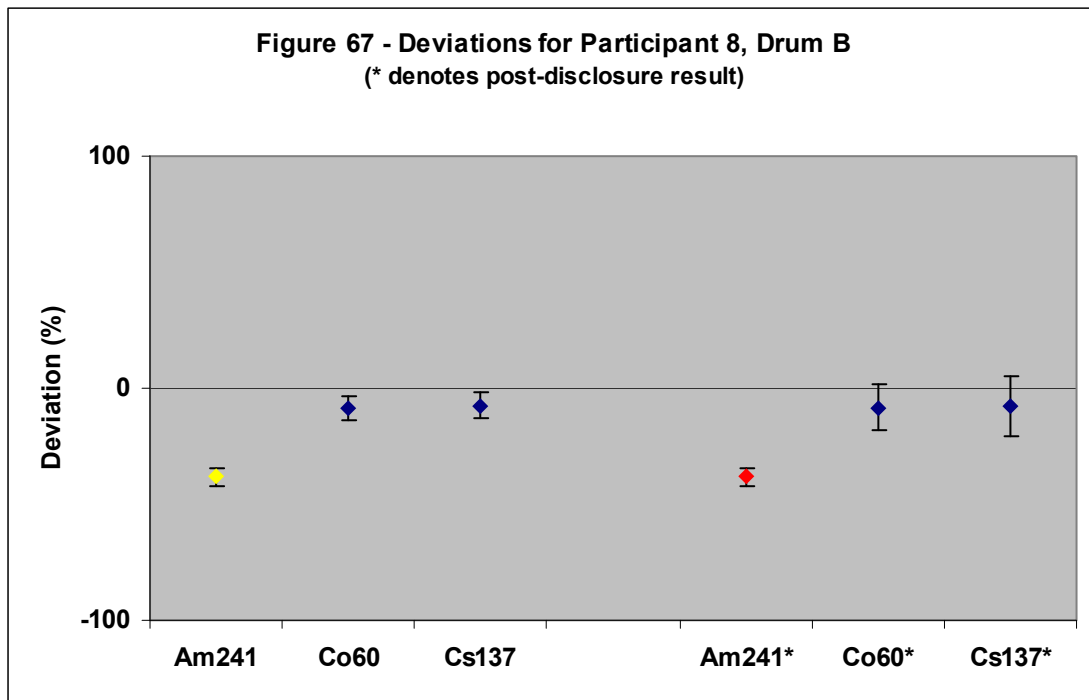


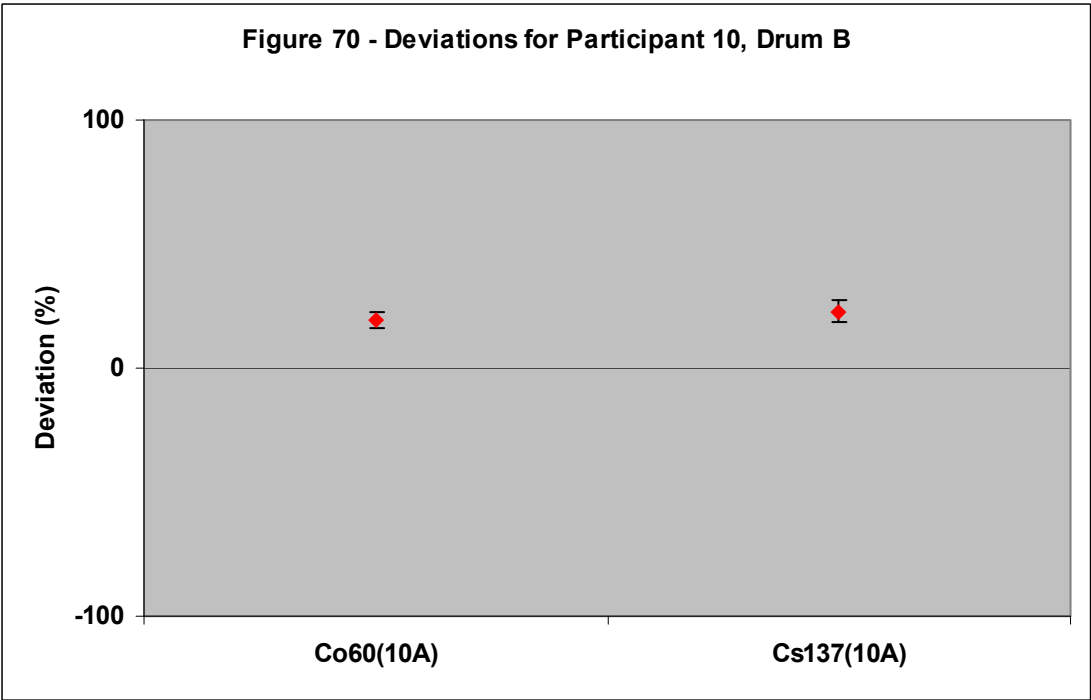
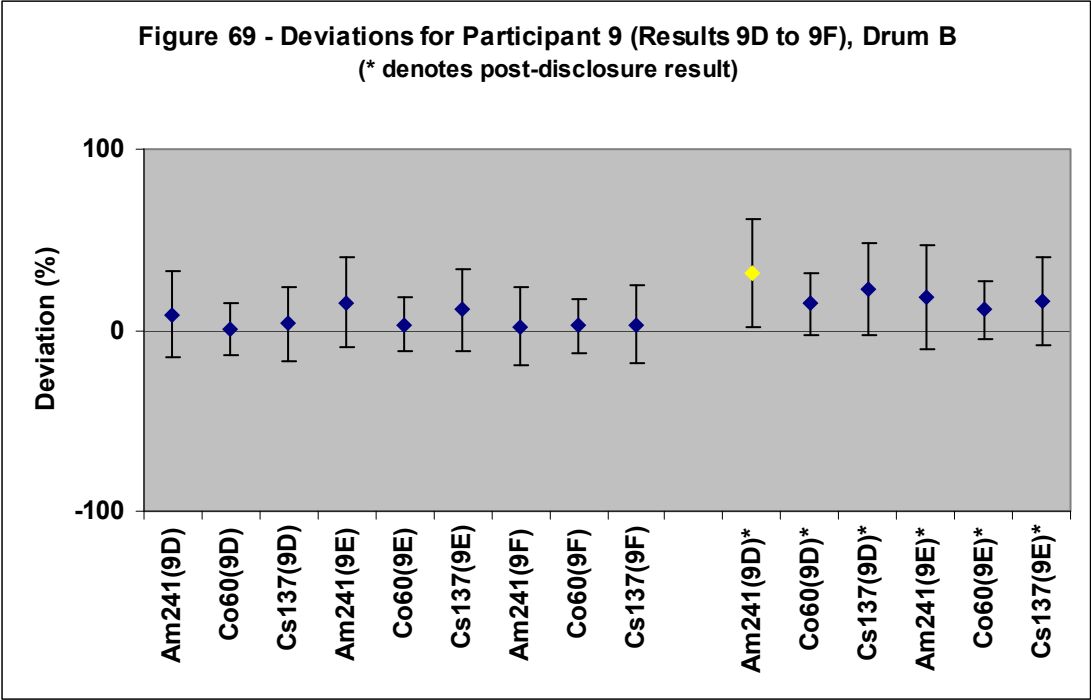


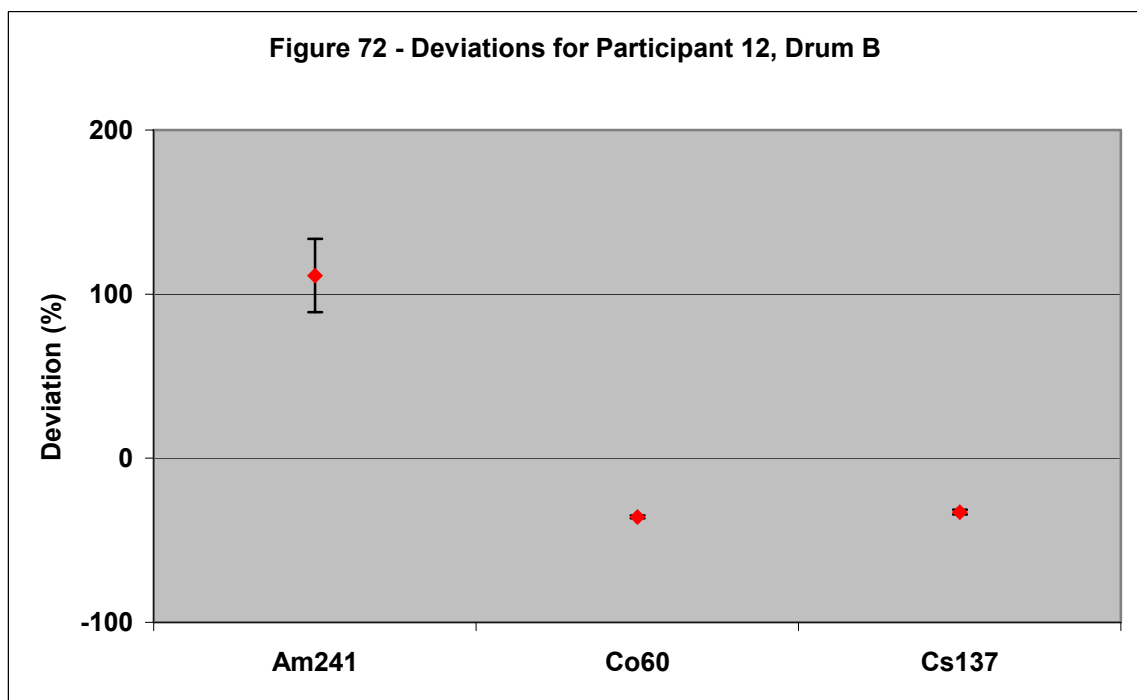
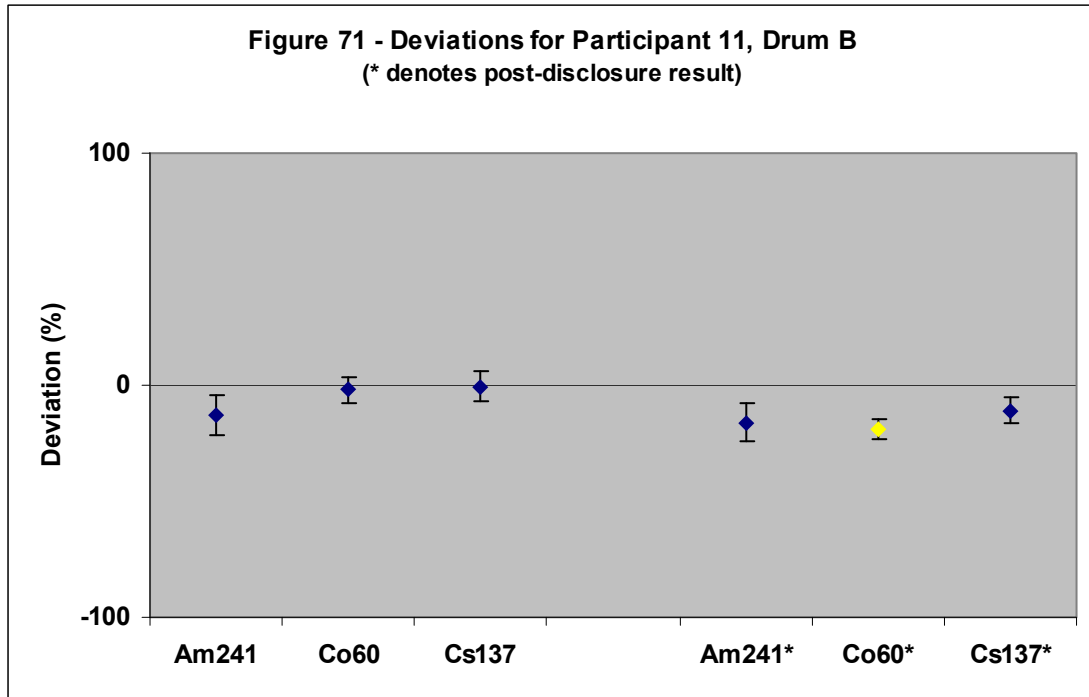


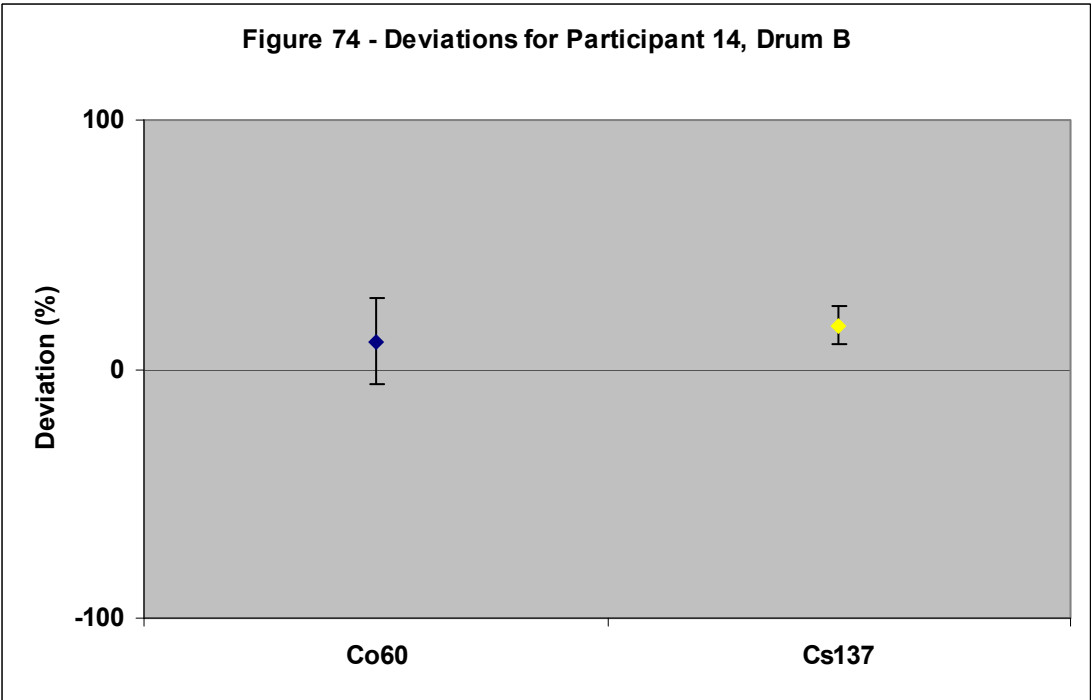
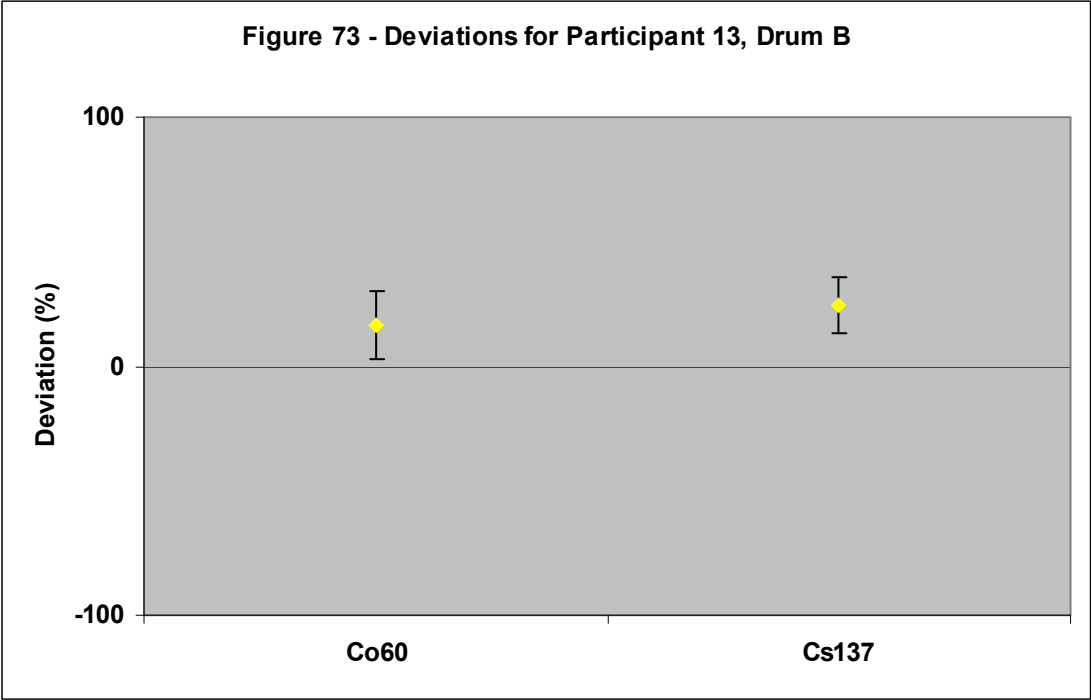


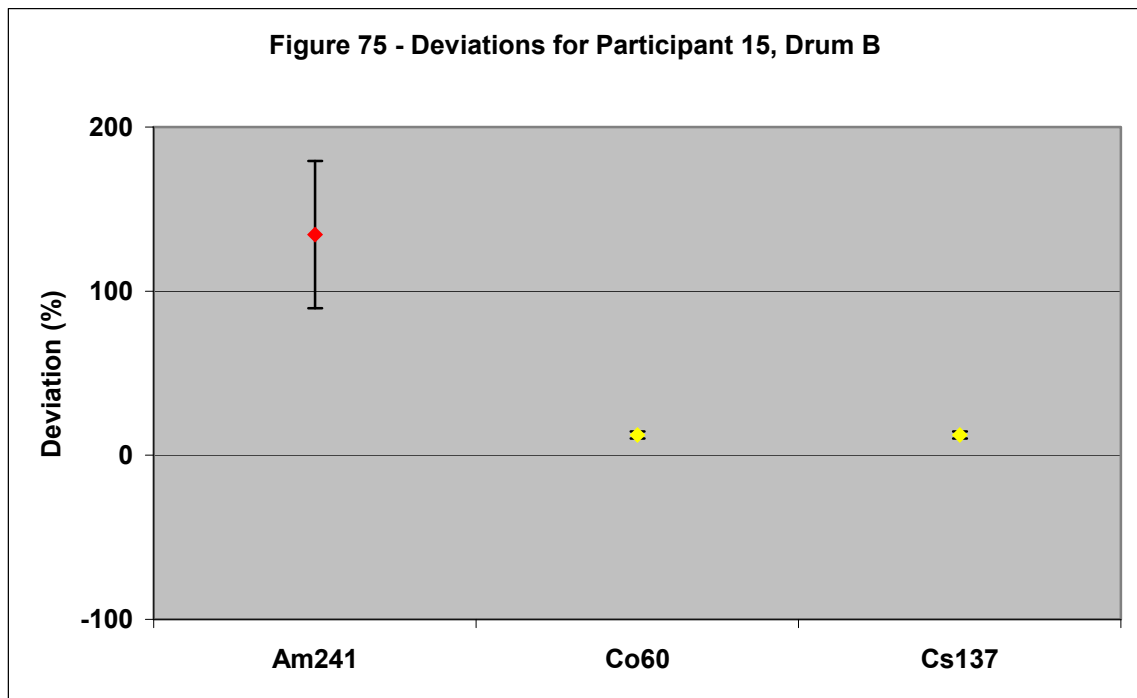












APPENDIX A

Initial mailshot

Dear Colleague,

Second NPL 200-litre Drum Comparison

In 2007, NPL ran a radioactivity comparison exercise to enable UK laboratories involved in decommissioning and site clearance to test their bulk-waste gamma measurement procedures. NPL prepared a 200 litre drum containing bottles of resin spiked with accurately-known activities of radionuclides directly traceable to UK primary standards of radioactivity. This was circulated as a 'blind sample' to the participants. The results were discussed at a follow-up workshop and published in NPL Report IR2 (available from the NPL website www.npl.co.uk).

I am now writing to invite you to participate in a second comparison.

Briefly, NPL will prepare two 200-litre drum sources:

- Drum A, with an activity concentration in the range 0.10 - 0.35 Bq g⁻¹
- Drum B, with an activity concentration in the range 5 - 20 Bq g⁻¹

The radionuclides ²⁴¹Am, ⁶⁰Co and ¹³⁷Cs will be present in both drums. The activity in each will be located in a small 'hot volume' consisting of one or more bottles of spiked resin, the rest of the drum being filled out with inactive material.

The drums will be circulated between March and November 2009 and participants will be asked to report the activity concentrations of the individual radionuclides. A draft report will be issued in January 2010, followed by a workshop in February and the final report in March.

If you are interested in participating, please click [here](#) for full details including fees.

Please note that requests to participate must be received by Friday 27 February 2009. There will be a limited number of places in the delivery schedule so please do not submit a Purchase Order until NPL confirms that your laboratory can be accommodated.

The project is part of the ongoing development of a UK radioactivity measurement infrastructure for nuclear decommissioning and site clearance. We hope you will be able to participate in the comparison and we look forward to hearing from you soon.

Yours faithfully

Julian Dean

January 2009

Second NPL drum comparison – Full details

A) Plan

NPL will prepare two drums:

- **Drum A**, with an activity concentration* (all nuclides combined) in the range 0.10 - 0.35 Bq g⁻¹
- **Drum B**, with an activity concentration in the range 5 - 20 Bq g⁻¹

Drum B will be prepared by modifying Drum A, so only one drum will be available for circulation at any one time.

Drum A will contain an array of 240 plastic bottles (500 ml each) in 5 layers of 48 bottles each. Nearly all of the bottles will be filled with ‘unspiked’ vermiculite, but a very small number will be filled with cation-exchange resin and will each contain a mixture of ²⁴¹Am, ⁶⁰Co and ¹³⁷Cs. If more than one ‘active bottle’ is present, the nuclide ratio and total activity per bottle will be constant and the bottles will be positioned together in a single location. There will therefore be a single ‘hot volume’ within the drum. The air gaps between bottles will be filled out with more vermiculite.

Drum B will be identical to Drum A except:

- the active bottle (or bottles) will contain significantly more activity
- the nuclide ratio may be different from Drum A
- the position of the active bottle(s) may be different from in Drum A

Participants may opt to measure either one or both drums. Drum A will be prepared first and then circulated to the participants wishing to measure at that level. It will then be returned to NPL and modified to produce Drum B. This will then be circulated and finally returned to NPL. Participants wishing to measure Drum A will be allocated 7 working days to do so (excluding arrival and dispatch days). Those measuring Drum B will be given 1 working day, again excluding arrival and dispatch days. NPL will arrange all drum movements within the UK and will arrange dispatches to overseas participants (overseas participants would have to arrange return shipments themselves).

Note that NPL will not initially disclose either the number of active bottles present in a drum or their location; however, participants will be given (prior to measurement) the dimensions of the drum and its mass (empty and full), along with manufacturer’s details of the resin and the vermiculite.

Participants will be asked to report their measurements of the individual radionuclide activity concentrations on reporting forms provided, along with details of calibration procedures and instrumentation used, by the ‘first deadline’ (see Timetable below). NPL will then disclose, for each drum type, the number of active bottles present and their location in the drum; participants will then have the opportunity to submit (by a second deadline) additional results based on this more detailed knowledge of the ‘hot volumes’. Note that revisions to earlier results (i.e. those submitted before the hot volume details were disclosed) will not be accepted after the first deadline.

* i.e. The activity present divided by the total mass of the drum’s contents, but excluding the mass of the drum itself.

NPL will then prepare a draft report (including all results, coded for anonymity), issue it to all participants and run a follow-up one-day workshop to discuss the exercise and decide on a forward plan (e.g. another comparison or maybe investigations into any calibration or measurement problems arising). A final report will then follow.

B) Timetable

Action	Dates
Interested laboratories to reply to this mailshot	By Friday 27 February 2009
NPL to contact each laboratory, advise if they can be accommodated, and, if so, agree receipt and dispatch dates	By 27 March 2009
Participants to submit Purchase Orders	Within two weeks of notification of receipt and dispatch dates
NPL to prepare Drum A	By 27 March 2009
Participants to measure Drum A	Within agreed dates between 30 March 2009 and 31 July 2009
NPL to prepare Drum B	By 7 August 2009
Participants to measure Drum B	Within agreed dates between 10 August 2009 and 13 November 2009
Participants to submit results (<u>not knowing</u> details of 'hot volumes')	By Friday 20 November 2009 (First deadline)
NPL to declare size, shape and location of hot volumes in both drum types	Monday 23 November 2009
Participants to submit any additional results (<u>knowing</u> details of hot volumes)	By Friday 4 December 2009 (Second deadline)
NPL to disclose activity concentrations	Monday 14 December 2009
NPL to issue draft report	January 2010
NPL to hold one-day workshop	February 2010 (date to be advised)
NPL to issue final report	March 2010

C) How to participate

Please contact Julian Dean by **27 February 2009** (at julian.dean@npl.co.uk) indicating:

- (i) Whether you are interested in measuring **Drum A**, **Drum B** or **both** drum types;
- (ii) For those wishing to measure **Drum A**, dates between **30 March 2009 and 31 July 2009** inclusive when it would **NOT** be possible for you to receive the drum, measure it and allow a courier on site to collect it;
- (iii) Likewise, for those wishing to measure **Drum B**, dates between **10 August 2009 and 13 November 2009** inclusive when it would **NOT** be possible for you to receive, measure and allow collection.

IMPORTANT: If the exercise is oversubscribed we shall deal with requests for inclusion on a first-come-first-served basis, so **PLEASE DO NOT SUBMIT A PURCHASE ORDER AT THIS STAGE**. We shall contact you in early March to confirm whether or not we can accommodate you, and if so we will agree receipt and dispatch dates with you and ask you to submit your order at that time. Participants must ensure Purchase Orders are submitted within two weeks to ensure that they receive the drum(s) on the allocated date(s).

Overseas colleagues should note that, because this exercise is funded primarily for the benefit of the UK measurement community, we must give priority to UK laboratories in the delivery schedule. However, we would very much value your participation and we shall do all we can to accommodate you.

D) Fees

The fees will be as follows:

- Participation fee (which covers attendance at the workshop): **£125**
- Loan of either Drum A or Drum B: **£250**
- Loan of both Drum A and Drum B: **£500**

Delivery charges will be added to the above fees. Within the UK, these will normally be £115 but will be £140 for some sites. Overseas charges will vary.

For UK participants, NPL will arrange for a courier to both deliver and collect, on the agreed dates. For overseas participants, NPL will arrange delivery on the agreed delivery date but the participant will be required to arrange return shipment by the agreed return date and to pay their own courier directly for that return shipment.

APPENDIX B

List of participants

Dr S J Allinson
National Nuclear Laboratory
A709 Springfields
Salwick
Preston
Lancashire PR4 0XJ

Mr N M Baghini
Imperial College London
Imperial College Reactor Centre
Silwood Park Campus
Buckhurst Road
Ascot
Berks SL5 7TE

Mr P W Clarke
Magnox North Ltd
Oldbury Power Station
Oldbury Naite
Thornbury
South Gloucestershire BS35 1RQ

Mr C Dale
AMEC Nuclear UK Ltd
Waste Quality Checking Laboratory
RSRL Winfrith Site
Building A512
Dorchester
Dorset DT2 8WQ

Mr S Defour
IRSN/DEI/SIAR
Route du Panorama
92262 Fontenay-aux-Roses
France

Mr S Holloway
AWE Aldermaston
Reading
Berkshire RG7 4PR

Mr K Hoyle
Magnox South Ltd
Berkeley Site
Berkeley
Gloucestershire GL13 9PB

Mr M Jackson
c/o Building 24
The Grove Centre
White Lion Road
Little Chalfont
Amersham HP7 9LL

Dr S Judge
Magnox South Ltd
Dungeness A
Romney Marsh
Kent TN29 9PP

Mr L Malpass
Magnox South Ltd
Berkeley Centre
Berkeley
Gloucestershire GL13 9PB

Dr T J Miller
AWE Aldermaston
Reading
Berkshire RG7 4PR

Mr I Pearman
Nuvia Ltd
B351.15
Harwell Science and Innovation Centre
Harwell
Didcot
Oxfordshire OX11 0TQ

Mr F Rizzo
Nucleco S.p.A.
Via Anguillarese 301
00123 S.M. di Galeria (Rm)
Rome
Italy

Mrs A Ross
DRSL
Dounreay
Thurso
Caithness KW14 7TZ

Mr M Rushby
Canberra UK Ltd
B528.10 Unit 1
Harwell Science and Innovation Campus
Didcot
Oxfordshire OX11 0TF

NPL Report IR 19

Dr S Tucker
Babcock Marine
Devonport Royal Dockyard
Devonport
Plymouth PL1 4SG

APPENDIX C

Reporting form

**NPL 200 L DRUM COMPARISON EXERCISE 2009 – RESULTS FOR DRUM A/B
(Hot volume known/not known)****Name:****Organisation:****Date of submission of results:**

Details of measurement method (please include, e.g., detector type/model, source-to-detector distance, background correction method, counting time, scan pattern, collimation, density correction routine, etc.)

--

Calibration method (please include, e.g., standard sources used, traceability of standards, uncertainties on standards, any geometrical correction factors used, all associated uncertainties, etc.)

--

Efficiency models (please include, e.g., details of any software used, and any validations carried out)

--

Any other information or comments

--

NPL 200 L DRUM COMPARISON EXERCISE 2009 – RESULTS FOR DRUM A/B
(Hot volume known/not known)

Nuclide: Am-241

Reference time 1 March 2009 12:00 GMT

	(Bq/g)
Activity concentration	
Combined standard uncertainty ($k=1$)	

Uncertainty budget components	Relative uncertainty (%)
Uncertainty component associated with net count rate of nuclide	
Uncertainty component associated with detector counting efficiency	
Uncertainty component associated with emission probability	
Uncertainty component associated with decay/ingrowth correction	
Other uncertainty components (please specify)	
Combined standard uncertainty	

Nuclear data used	
Half-life (days)	
Emission probability (%)	keV
Emission probability (%)	keV

Limit of Detection	
---------------------------	--

NPL 200 L DRUM COMPARISON EXERCISE 2009 – RESULTS FOR DRUM A/B
(Hot volume known/not known)

Nuclide: Cs-137

Reference time 1 March 2009 12:00 GMT

	(Bq/g)
Activity concentration	
Combined standard uncertainty ($k=1$)	

Uncertainty budget components	Relative uncertainty (%)
Uncertainty component associated with net count rate of nuclide	
Uncertainty component associated with detector counting efficiency	
Uncertainty component associated with emission probability	
Uncertainty component associated with decay/ingrowth correction	
Other uncertainty components (please specify)	
Combined standard uncertainty	

Nuclear data used	
Half-life (days)	
Emission probability (%)	keV
Emission probability (%)	keV

Limit of Detection	
---------------------------	--

NPL 200 L DRUM COMPARISON EXERCISE 2009 – RESULTS FOR DRUM A/B
(Hot volume known/not known)

Nuclide: Co-60

Reference time 1 March 2009 12:00 GMT

	(Bq/g)
Activity concentration	
Combined standard uncertainty ($k=1$)	

Uncertainty budget components	Relative uncertainty (%)
Uncertainty component associated with net count rate of nuclide	
Uncertainty component associated with detector counting efficiency	
Uncertainty component associated with emission probability	
Uncertainty component associated with decay/ingrowth correction	
Other uncertainty components (please specify)	
Combined standard uncertainty	

Nuclear data used	
Half-life (days)	
Emission probability (%)	keV
Emission probability (%)	keV

Limit of Detection	
---------------------------	--

APPENDIX D

Minutes of post-comparison workshop

Minutes of the 2nd NPL Drum Comparison Workshop

National Physical Laboratory
Hampton Road
Teddington
Middlesex
TW11 0LW
United Kingdom

25th February 2010

Delegates

Nasser Baghini	Imperial College Reactor Centre
Chris Dale	AMEC Nuclear UK Ltd
Julian Dean	NPL
Gary Faulkner	Babcock Marine
Chris Gilligan	NPL
James Gregory	G E Healthcare
Arvic Harms	NPL
Steven Holloway	AWE plc
Keith Hoyle	Magnox South (Berkeley)
Sami Kafala	Imperial College Reactor Centre
Timothy Miller	AWE plc
Ian Pearman	Nuvia Ltd
Martin Rushby	Canberra UK Ltd
Steven Sharp	Sellafield Ltd

Session 1**Background to exercise, results and discussion – Julian Dean, NPL**

A total of 14 participants attended the meeting, from 10 different institutions. The meeting was opened and chaired by Julian Dean, the coordinator for the comparison.

The exercise had followed on from the first such exercise in 2007. Outcomes of the 2007 exercise had been (i) the 'layers' of resin in the drum had caused difficulties for SGS users, (ii) the activity in the drum had been too low for some users, and (iii) a second exercise would be useful and should include a 'hotspot' in the drum.

In the second exercise, therefore, two drums (approx. 0.4 Bq g⁻¹ and 10 Bq g⁻¹) were offered, each with the activity concentrated in a single bottle of ion-exchange resin and with the rest of the drum completely filled with bottles of vermiculite (and with vermiculite in the deadspaces). The two drums were designated A and B respectively. The same radionuclides were present in each drum (²⁴¹Am, ⁶⁰Co and ¹³⁷Cs) and the activities were directly traceable to primary standards. The logistics of the exercise were described. Two results deadlines were

imposed for each drum - one prior to NPL's disclosure of the location of the 'hot bottle', and one 'post-disclosure'.

Most participants had used single-crystal HPGe detectors, SGSs, or both. A wide range of efficiency models / calibration standards had been used. The results were presented as deviation plots by drum type and by radionuclide. Each individual value had been analysed to determine the 'level of agreement' with the NPL value and was colour-coded to indicate whether it was 'in agreement', 'questionable' or 'discrepant'. The majority of returned results were within 50 % of the NPL assigned value.

For Drum A, 78% of the 'pre-disclosure' results agreed with NPL. Five of the ^{60}Co results were discrepant. Three of these were low compared with NPL and JD pointed out these were all SGS measurements and asked might this be relevant. The other two were from one participant (No. 2) and were high (discrepant/questionable) for all three nuclides. However, this participant had had to assume a value for the density of the dried ion exchange resin as a 'dry' value had not been supplied by NPL.

Action JD to provide all with resin mass in active bottles.

Some participants also submitted Drum A data after disclosure of the hot-volume location, but only 56% of results were in agreement with NPL (n.b. 76% of the 'pre-disclosure' data submitted by the same group of participants agreed with NPL). Participant 3 had changed their calibration template to a 'pipe' but this resulted in discrepant results. Participant 10B's result for ^{137}Cs was > 200% high. They had used a plastic scintillator detector.

For Drum B, the overall figures were:

- 'Pre-disclosure' data (all participants): 57% agreed
- 'Post-disclosure' data (subset of participants): 74% agreed
- 'Pre-disclosure' data (same subset of participants): 77% agreed

Pre-disclosure, Participants 12 and 15 reported results > 100% high for ^{241}Am .

The lower level of agreement for Drum B is probably due to the lower uncertainties (mainly counting statistics) on the returned results, making it more difficult to pass the Zeta Test.

In summary, the individual nuclide performance for both drums was as follows:

Nuclide	Results in agreement for Drum A (pre-disclosure)	Results in agreement for Drum A (post-disclosure)	Results in agreement for Drum B (pre-disclosure)	Results in agreement for Drum B (post-disclosure)
^{241}Am	11 out of 14	5 out of 11	8 out of 14	5 out of 9
^{60}Co	12 out of 18	8 out of 13	9 out of 19	7 out of 9
^{137}Cs	15 out of 17	7 out of 12	8 out of 18	8 out of 9

The chairman suggested possible reasons for some of the observed results, e.g.:

- Traceability problems with 'in-house' standards
- Incorrect use of 'pipe' templates
- Incorrect calculation of drum composition
- SGSs 'splitting' active bottle gamma emissions between adjacent segments
- Settling of vermiculite in transit (8cm in Drum A and 3cm in Drum B)

One participant said there is more than one ISOCS pipe template and that participants should specify which one they used.

Action JD to ask Participant 3 which pipe template they used.

One of the participants who had used an SGS was unsure how their ^{60}Co efficiency had been derived – it may have been done by extrapolation. They are primarily interested in low gamma energies.

Keith Hoyle commented that this was a useful exercise particularly for clearance and exemption monitoring.

Another participant asked if there were any air gaps in the drums. JD confirmed the drums were completely filled (with bottles, vermiculite and resin). A small amount of vermiculite settling had occurred (see above), but was not thought to have had a significant effect on the measurements. To prevent settling in future exercises, one participant commented that perhaps polystyrene pieces or solid blocks would be better.

JD asked if the ‘two deadline’ schedule had been useful. The consensus was that it had. In a ‘real life’ situation the hot spot is not known, but it would be useful to have the information. Steven Holloway commented that he didn’t receive the hot spot location for Drum B (this may have been due to AWE email filtering system). He went on to suggest the information could be added to the NPL web site.

Action JD to check why AWE didn’t receive hotspot location.

Ian Pearman pointed out that some Nuvia data were missing from the draft report.

Action IP and JCJD to check data transmission/receipt.

NPL would accept additional data from all participants. The deadline is 12 March 2010. Any new data would be listed separately from the original data set in the final report.

Action All to submit any additional data by 12 March 2010.**Participant presentation – Sami Kafala, Imperial College**

Imperial College Reactor Centre (ICRC) is a national resource for neutrons for research. Their work includes teaching, calibration facilities, isotope production, neutron activation analysis and delayed neutron counting. They participated in the NPL drum comparison to help develop new analytical techniques. Also, they have legacy waste and decommissioning waste streams. ICRC is seeking UKAS accreditation for analytical work and participation in the NPL exercises is useful for demonstrating technical competence.

SK presented their new gamma spectrometry system which uses ‘ISOTOPIC’ software. This is for non-destructive measurements of radioactive items in various containers and configurations. Items up to 450 kg can be accommodated. They sit on a turntable rotating at 1 rpm which reduces the effects of inhomogeneity. A germanium detector is located at a fixed distance from the source. They used ‘mixed radionuclide’ type standard sources to derive an efficiency curve for this detector based on a single point source measurement. This calibration is extrapolated to match the actual samples. This model is based on ‘point kernel’ methods and the entire measurement problem is broken down into multiple matrix voxels (i.e. elements of volume) and their contribution to the composite spectrum are calculated and summed. The approach, which is similar to Monte-Carlo Methods of simulation, utilises detector parameters (e.g. crystal diameter and length) which the user supplies as part of the measurement configuration. Examples of analysis reports and spectra were given.

Session 2

Participant presentation – Steven Holloway, AWE

Atomic Weapons Establishment (AWE) is responsible for the design, manufacture and support of warheads for the United Kingdom's nuclear deterrent. AWE waste streams are likely to be dominated by low energy gamma emitting isotopes.

The NPL drums were measured using two techniques. The AWE Non Destructive Assay Group used a portable High Resolution Gamma Spectrometer (HRGS) and simple spreadsheet analysis, whereas the Waste Management Group using a HRSGS (High Resolution Segmented Gamma Scanner) with analysis using Canberra's NDA2K software.

The portable HRGS was used to measure both drums. Both were scanned using low-resolution handheld gamma detector to locate the hotspot and to determine its approximate extent. A gamma image was taken of Drum B to determine the distance of the activity into the drum. These measurements indicated a single small activity region near to the drum's surface.

HRGS measurements using HPGe detectors were carried out from opposing sides of drum at 50 cm from the drum wall for Drum A and 100 cm for Drum B. Transmission measurements were carried out using ^{241}Am , ^{137}Cs and ^{60}Co sources to determine the linear attenuation coefficient of the matrix. Spreadsheet model setups were used, and assumed a point source of activity at variable distance into drum, linear attenuation coefficient for the matrix, and a 1 mm thick steel drum wall. Least squares fitting was used to determine the effective distance 'into the drum' of the activity in Drum A. The estimated uncertainty contained components due to counting statistics, activity location, linear attenuation coefficients, drum wall thickness, and activity and 'standoff' of sources used to determine detector efficiency.

Drum B was also measured on a turntable and analysed assuming a uniform distribution of activity. The results were very similar.

The AWE high-resolution segmented gamma scanner (HRSGS) uses a collimated detector to effectively split the drum into eight vertical segments. The detector has a low energy filter preventing the measurement of ^{241}Am at low levels. At each position a transmission measurement is made and the results assumes uniform activity distribution within each slice. The uncertainty budget only contains counting statistics as the energies measured are not what the system is configured to measure. For Plutonium or Uranium, a spatial component would be included.

Next comparison and proposals for NPL research – Julian Dean

JD invited proposals for NPL projects to support the decommissioning industry in the UK, and for input to the format of the next 'drum comparison' (scheduled for October 2010 to September 2011). The matrix, activity level and nuclides could be similar or different from previous comparisons. NPL could make homogenous or heterogeneous sources (i.e. with one or more hotspots).

Suggestions for the next exercise included:

- A drum containing a number of spiked ‘real’ artefacts (e.g. metal bars or tools). It would be a useful exercise to send out the drum with no information on its contents. It would be important to prevent the components from moving around during transport.
- A relatively small sample (e.g. a metal canister of some kind containing the activity) which could be incorporated by the participants into their own ‘usual’ measurement geometry prior to measurement. This might enable a wider range of labs to participate.
- A drum containing uranium.

Action JD to canvass user base for further input.

Results for ‘synthetic sand’ sample in 2009 Environmental Radioactivity Proficiency Test Exercise – Arvic Harms, NPL

AVH presented the participants’ measurement results for the ‘synthetic sand’ samples issued as part of the 2009 Environmental Radioactivity PTE. The samples were prepared by combining a radioactive standard solution with tetraethyl orthosilicate using a sol-gel reaction. This method enables various radionuclides to be incorporated into a sand-like material. The 50 g ‘sand’ samples (SiO₂) contained 6 nuclides (⁵⁵Fe, ⁹⁰Sr, ¹³³Ba, ¹³⁴Cs, ¹³⁷Cs and ¹⁵²Eu) at between 0.8 and 12 Bq/g. The homogeneity of the batch of sources was confirmed using gamma spectrometry. Approximately 73% of participants’ results were in agreement with the NPL assigned values, 12% were questionable and 16% discrepant.

This method could be extended to the preparation of larger-volume samples (e.g. as an alternative to the ion-exchange resin used in the drum comparisons).

A Certified Reference Material based on neutron-irradiated concrete – Arvic Harms

At the previous drum comparison workshop, NPL reported the results of measurements of a neutron-activated ‘real’ concrete powder sample by the participants in the 2007 Environmental Radioactivity PTE. A similar sample was offered in the 2008 PTE and the participants’ results were used to calculate assigned values for some of the radionuclides present. The material was certified for these radionuclides and this work has now been presented to ICRM and published in Applied Radiation and Isotopes.

Summary – Julian Dean

NPL would aim to issue workshop minutes in March 2010 and the final report in April 2010.

Action JD/CG.

JD thanked the participants for their measurement results and for their presentations and other contributions to the workshop. The workshop was closed at around 3-30 pm.

Actions

- JD to provide participants with mass of ion-exchange resin in active bottles
- JD to ask Participant 3 which ISOCS ‘pipe template’ they used
- JD to check why AWE (NDA group) did not receive hotspot details
- IP to check if all Nuvia data were sent
- JD to check if Nuvia data were all received

NPL Report IR 19

- All participants to submit any additional data by 12 March 2010
- JD to canvass wider user base on format of third exercise
- JD to issue workshop minutes in March 2010
- JD to issue exercise report in April 2010

Chris Gilligan
Julian Dean

Radioactivity Group, NPL
15 April 2010



100 years of living science
100

NPL Drum Proficiency Exercise

25th February 2010

Sami Kafala

MAHPG
Imperial College Reactor Centre

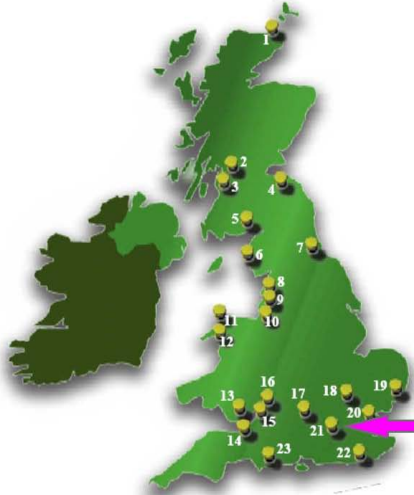
100 years of living science



100 years of living science
100

Location

Locations Of Civil Nuclear Sites



UK Civil RRs lost:

Harwell: Dido, Pluto, GLEEP

Winfrith: SGHWR, Nester, Dimple

Scottish Universities: UTR300

ICI: TRIGA..... etc.....

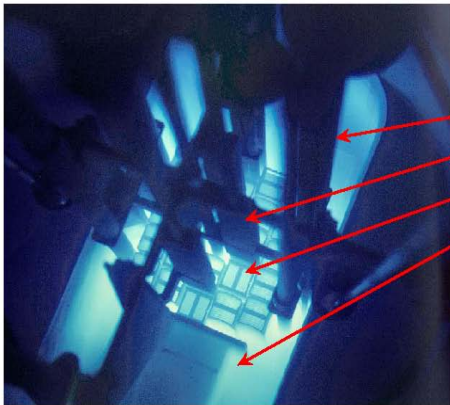
ICRC Mission:
 "to provide a
 national resource
 for research
 neutrons"



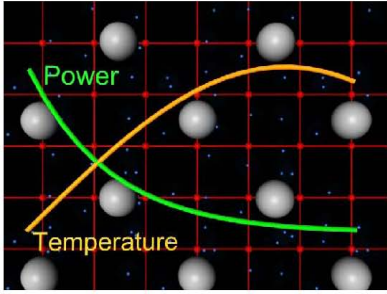
Imperial College
London

100 years of living science
100

Inside CONSORT



Irradiation Tube
 Control Absorber
 Fuel Assembly
 Water



Negative temperature coefficients are desirable from the safety point of view, because they help to avoid reactor excursions.

Imperial College
London

100 years of living science
100

CAPABILITIES RETAINED



Teaching (training students at under/postgraduate level, regulatory bodies, research and demonstration of reactor control)

Calibration facilities for neutron detectors using beam tube facilities (for testing of fission chambers/ion chambers for the power generating/defence sector)

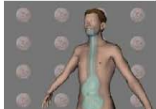
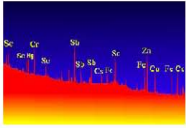




Isotope production Sm-153 (Pharmaceuticals)-Capability under development: Mo-99 (possible partnering with a UK organisation)



Trace element & radioactivity Analysis, etc. **NAA & DNC**

**Imperial College
London**

MAHPG Labs.

100 years of living science
100

**Gamma Spec
Systems**



**Prep
Room**

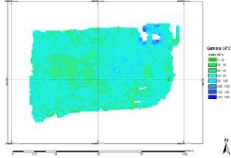


GPS/GIS



**Health
Phys**







**Beta Spec
Unit**



**Imperial College
London**

MAHPG Labs. Continue...

100 years of living science
100

Waste Assessment/Characterisation Systems




**Imperial College
London**

Objectives / Why participate in the NPL test exercises ?

100 years of living science
100

- Develop new analytical techniques for sample analysis, Environmental Radioactivity measurements & waste assessment.
- The ICRC has some legacy of radioactive waste on their sites (NLS & EAS) and Complete Silwood Park Legacy project to Regulator & Corporate satisfaction.
- Waste will be produced as a result of the decommissioning.
- Gain UKAS accreditation for analytical work.

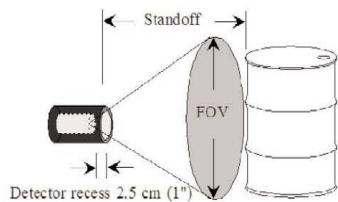
**Imperial College
London**

MAHPG Radioactive Waste Assay (NDA)

100 years of living science
100

ISOTOPIC

Non-destructive measurements of radioactive items in various containers and configurations

ISO-TURNABLE: To reduce measurement bias, 1 RPM, up to 450 kg capacity

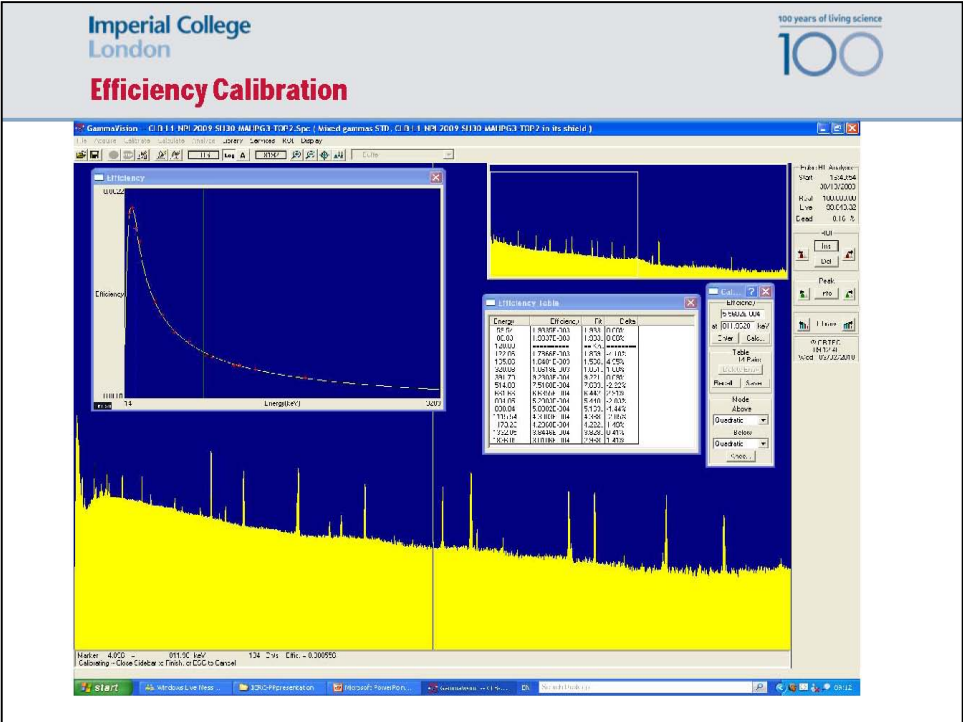
- The detector is characterised by a single point source measurement, with/out collimator.
- This primary calibration, which can be traced to a certified standard, for any detector, is extrapolated (modelled) to match the physical sample (container geometry, material, and matrix composition).
- The model is based on "point-kernel" methods in which the entire measurement problem is broken down into multiple matrix voxels (element of volume) and their contribution to the composite spectrum are calculated and summed.
- The approach, which is similar to Monte-Carlo Methods of simulation, utilises detector parameters (crystal diameter, crystal length, etc..) which the user supplies as part of the measurement configuration.

The activity of an isotope in a container is given by:

$$A_i = \frac{N_{meas} (CF_{item})(CF_{col})}{P_\gamma \cdot \epsilon_{det}}$$

Where,

- A_i : activity of the isotope to be reported (Bq),
 N_{meas} : measured peak area for a reference gamma ray of the isotope (c/s),
 CF_{item} : container, matrix, and sample self-attenuation correction factors
 CF_{col} : collimator correction factor,
 P_γ : gamma-ray Emission Probability,
 ϵ_{det} : detector efficiency (cps/Bq).



Imperial College
London

Some validation

100 years of living science
100

Samples received : 24th June 2009
Report issued : 26th June 2009
Our ref : AL03
Samples analysed as received

The reported expanded uncertainty is based on a standard uncertainty multiplied by a coverage factor $k=2$ providing a level of confidence of approximately 95%.

Method of Analysis	Container Type	Mass (g)	MAHPG No.	Sample	Sp. Activity (Bq/g)	Uncertainty (Bq/g)
GammaVision	75 ml Pot	72	AL0301	Walled Garden soil	3.6	0.2
	75 ml Pot	59	AL0302	Walled Garden soil	2.7	0.2
	75 ml Pot	58	AL0303	Walled Garden soil	8.2	0.5
	75 ml Pot	69	AL0304	Walled Garden soil	3.4	0.2
				Average	4.5	0.3
ISOTOPIC-Rotating	208 Litre Drum	160000	AL0301-04-Drum Rotating	Walled Garden soil	4.7	1.3
ISOTOPIC-Stationary	209 Litre Drum	160000	AL0301-04-Drum Stationary	Walled Garden soil	5.3	1.5

Imperial College London Silwood Legacy Wastes 100 years of living science 100 Undertaking work for the rest of the College associated with Academic Campus Legacy Waste - Reactor Centre asked to provide clearance of work area on behalf of the College  Walled garden May 2002 ¹³⁷Cs contaminated wastes from previous experimental work in 2002 (Soil uptake experiments were carried out). Re-assurance monitoring carried out at the time. However: area was not cleared to Industry best-practice levels. Undergoing final assessment and remediation (excavation/disposal off site)	
--	--

Imperial College London Covering Sheet 1 100 years of living science 100	<table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="text-align: center; font-weight: bold;">Reactor Centre</td> <td style="text-align: center; font-weight: bold;">Imperial College</td> </tr> <tr> <td style="text-align: center; font-weight: bold;">MAHPG Container Data Cover Sheet</td> <td style="text-align: center; font-weight: bold;">London</td> </tr> <tr> <td style="font-size: x-small;">Form ICRC/MP/5000</td> <td style="font-size: x-small;">Form Issue No.: 1 Date issued: 28/6/09</td> </tr> </table> MAHPG Batch No.: IL01 Date of Receipt: 15th July 2009 Description of Analysis: ISOTOPIC scan of NPL 200 Litre Drum A & B Competition Exercise 2009 Special Conditions: Analyse both in stationary and rotating modes Sample mass/volume: Whole sample Decay time: N/A Count time: 60000 seconds Authorised by: N. Baghini Date: 22/7/2009 Analysed by: N. Baghini Date: 31/7/2009	Reactor Centre	Imperial College	MAHPG Container Data Cover Sheet	London	Form ICRC/MP/5000	Form Issue No.: 1 Date issued: 28/6/09
Reactor Centre	Imperial College						
MAHPG Container Data Cover Sheet	London						
Form ICRC/MP/5000	Form Issue No.: 1 Date issued: 28/6/09						

<table border="1" style="width: 100%; border-collapse: collapse;"> <tr><td>Analysis range</td><td></td></tr> <tr><td>Start</td><td>130</td></tr> <tr><td>Stop</td><td>16000</td></tr> <tr><td>Uranium/Plutonium Tab</td><td></td></tr> <tr><td>Uranium isotopic weight fractions</td><td></td></tr> <tr><td>Use entered values</td><td>Leave blank</td></tr> <tr><td>Determined from spectrum</td><td>Checked</td></tr> <tr><td>Pu isotopic weight fractions</td><td></td></tr> <tr><td>Use entered values</td><td>Leave blank</td></tr> <tr><td>Determined from spectrum</td><td>Checked</td></tr> <tr><td>Report Tab</td><td></td></tr> <tr><td>Uncertainty reporting</td><td></td></tr> <tr><td>In activity units</td><td>Checked</td></tr> <tr><td>Total</td><td>Checked</td></tr> <tr><td>Confidence level</td><td>2</td></tr> <tr><td>Reporting options</td><td></td></tr> <tr><td>Library peaks</td><td>Checked</td></tr> <tr><td>Library peak matrix</td><td>Checked</td></tr> <tr><td>Unknown peaks</td><td>Checked</td></tr> <tr><td>Error estimation</td><td>Checked</td></tr> <tr><td>MDA's and correction factors</td><td>Checked</td></tr> <tr><td>Spectrum and interactive plots</td><td>Checked</td></tr> <tr><td>Print on completion</td><td>Leave blank</td></tr> <tr><td>Isotopic interactive plot</td><td>Checked</td></tr> <tr><td>ISOTOPIC SETTINGS DETECTORS</td><td></td></tr> <tr><td>Detector 1 of 1</td><td></td></tr> <tr><td>Name</td><td>MAHPG3</td></tr> <tr><td>Type</td><td>N Type</td></tr> <tr><td>Crystal dimensions (mm)</td><td></td></tr> <tr><td>Length</td><td>64.8</td></tr> <tr><td>Diameter</td><td>64.7</td></tr> <tr><td>Detector configuration</td><td></td></tr> <tr><td>Orientation</td><td>Side</td></tr> <tr><td>Weighting</td><td>Default 1</td></tr> <tr><td>Detector height (cm)</td><td>43.5</td></tr> <tr><td>Offset from edge (cm)</td><td>N/A</td></tr> <tr><td>Detector standoff (cm)</td><td></td></tr> <tr><td>Detector Collimator?</td><td>No</td></tr> <tr><td>Depth (Recess) (cm)</td><td>N/A</td></tr> <tr><td>Thickness (cm)</td><td>N/A</td></tr> <tr><td>Inner diameter (cm)</td><td>N/A</td></tr> <tr><td>Collimator material (check) 1% Fe or W</td><td>N/A</td></tr> <tr><td>Calibration file</td><td>c:\user\soeff\coll-MAHPG3-30-4mEU.clib</td></tr> <tr><td>PBC file</td><td>c:\user\pbc\luc00001 MAHPG3 detector C100700009 etc.</td></tr> <tr><td>Authorised By</td><td>N. Baghini</td></tr> <tr><td>Date</td><td>22nd July 2009</td></tr> </table>	Analysis range		Start	130	Stop	16000	Uranium/Plutonium Tab		Uranium isotopic weight fractions		Use entered values	Leave blank	Determined from spectrum	Checked	Pu isotopic weight fractions		Use entered values	Leave blank	Determined from spectrum	Checked	Report Tab		Uncertainty reporting		In activity units	Checked	Total	Checked	Confidence level	2	Reporting options		Library peaks	Checked	Library peak matrix	Checked	Unknown peaks	Checked	Error estimation	Checked	MDA's and correction factors	Checked	Spectrum and interactive plots	Checked	Print on completion	Leave blank	Isotopic interactive plot	Checked	ISOTOPIC SETTINGS DETECTORS		Detector 1 of 1		Name	MAHPG3	Type	N Type	Crystal dimensions (mm)		Length	64.8	Diameter	64.7	Detector configuration		Orientation	Side	Weighting	Default 1	Detector height (cm)	43.5	Offset from edge (cm)	N/A	Detector standoff (cm)		Detector Collimator?	No	Depth (Recess) (cm)	N/A	Thickness (cm)	N/A	Inner diameter (cm)	N/A	Collimator material (check) 1% Fe or W	N/A	Calibration file	c:\user\soeff\coll-MAHPG3-30-4mEU.clib	PBC file	c:\user\pbc\luc00001 MAHPG3 detector C100700009 etc.	Authorised By	N. Baghini	Date	22 nd July 2009	
Analysis range																																																																																													
Start	130																																																																																												
Stop	16000																																																																																												
Uranium/Plutonium Tab																																																																																													
Uranium isotopic weight fractions																																																																																													
Use entered values	Leave blank																																																																																												
Determined from spectrum	Checked																																																																																												
Pu isotopic weight fractions																																																																																													
Use entered values	Leave blank																																																																																												
Determined from spectrum	Checked																																																																																												
Report Tab																																																																																													
Uncertainty reporting																																																																																													
In activity units	Checked																																																																																												
Total	Checked																																																																																												
Confidence level	2																																																																																												
Reporting options																																																																																													
Library peaks	Checked																																																																																												
Library peak matrix	Checked																																																																																												
Unknown peaks	Checked																																																																																												
Error estimation	Checked																																																																																												
MDA's and correction factors	Checked																																																																																												
Spectrum and interactive plots	Checked																																																																																												
Print on completion	Leave blank																																																																																												
Isotopic interactive plot	Checked																																																																																												
ISOTOPIC SETTINGS DETECTORS																																																																																													
Detector 1 of 1																																																																																													
Name	MAHPG3																																																																																												
Type	N Type																																																																																												
Crystal dimensions (mm)																																																																																													
Length	64.8																																																																																												
Diameter	64.7																																																																																												
Detector configuration																																																																																													
Orientation	Side																																																																																												
Weighting	Default 1																																																																																												
Detector height (cm)	43.5																																																																																												
Offset from edge (cm)	N/A																																																																																												
Detector standoff (cm)																																																																																													
Detector Collimator?	No																																																																																												
Depth (Recess) (cm)	N/A																																																																																												
Thickness (cm)	N/A																																																																																												
Inner diameter (cm)	N/A																																																																																												
Collimator material (check) 1% Fe or W	N/A																																																																																												
Calibration file	c:\user\soeff\coll-MAHPG3-30-4mEU.clib																																																																																												
PBC file	c:\user\pbc\luc00001 MAHPG3 detector C100700009 etc.																																																																																												
Authorised By	N. Baghini																																																																																												
Date	22 nd July 2009																																																																																												

[illegible]



Analysis Report

100 years of living science



Sample Name: IL0101 Rotating Count 1

Spectrum File Name: C:\user\IL0101 Rotating Count 1\B00000 MAHPG3 Detector\B000001 MAHPG3 Detector.Axi

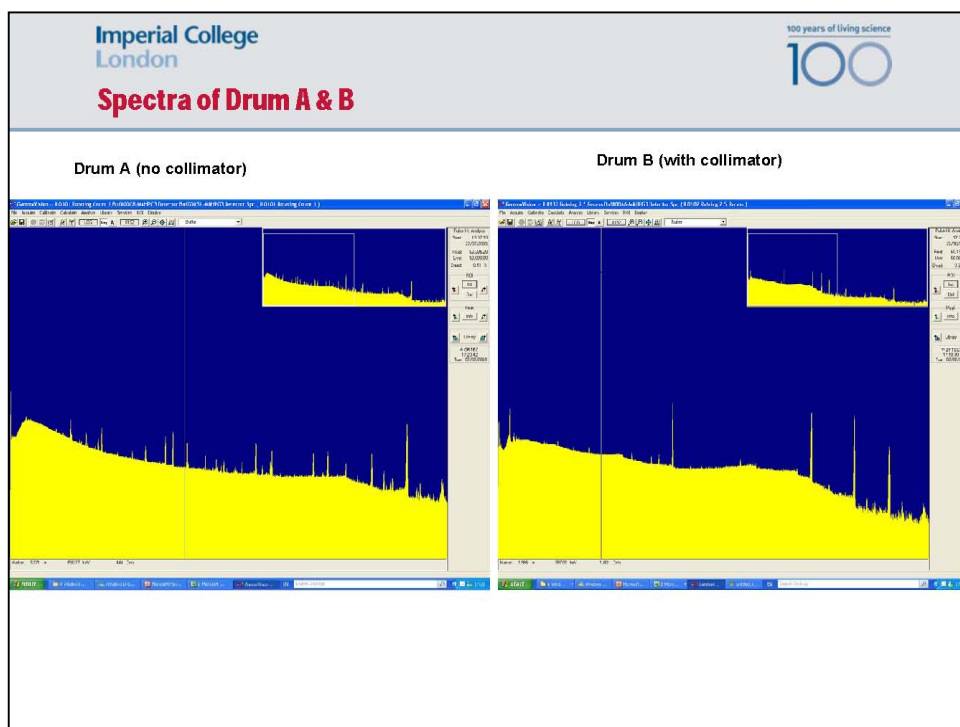
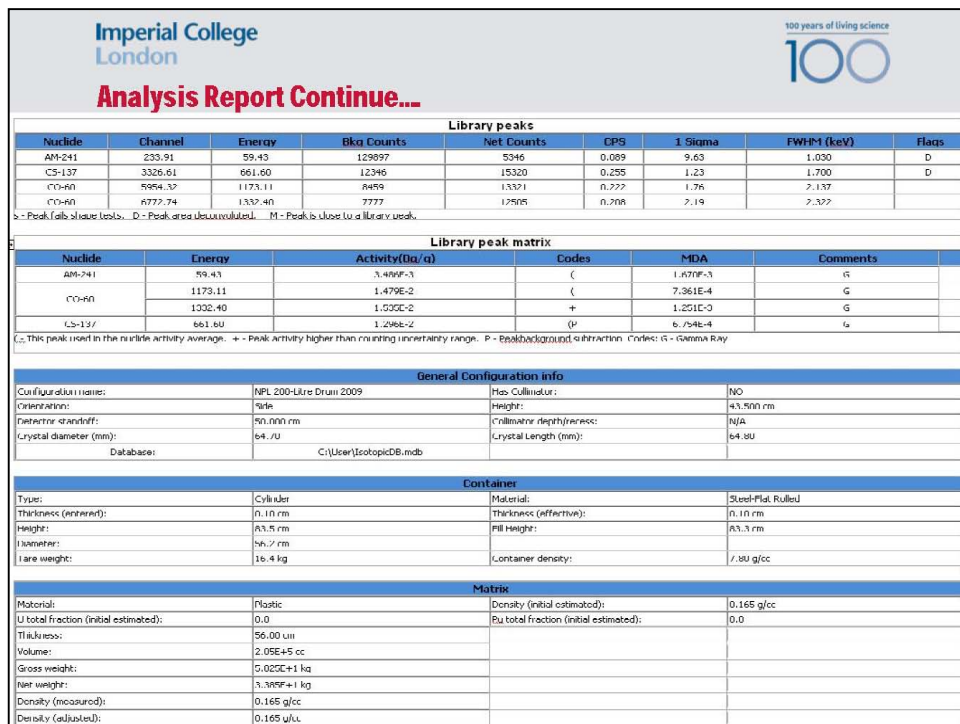
Acquisition information			
Live time:	60000	7/11/2009 13:07:53	Real time:
Detector:	UK3 MAHPG3 Detector		Dead time (%):
			0.51

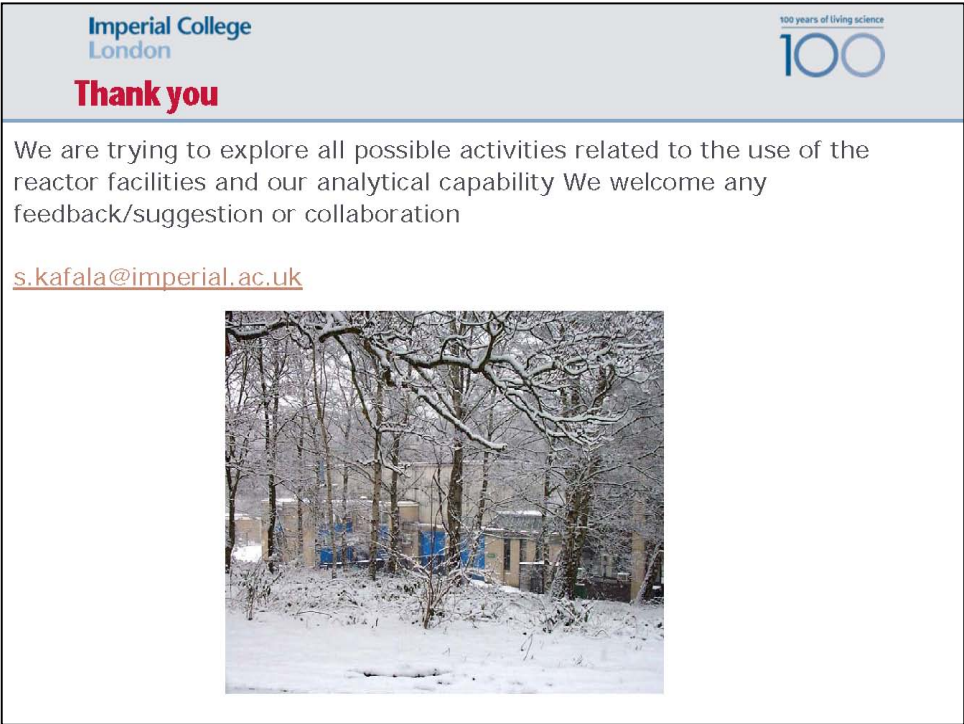
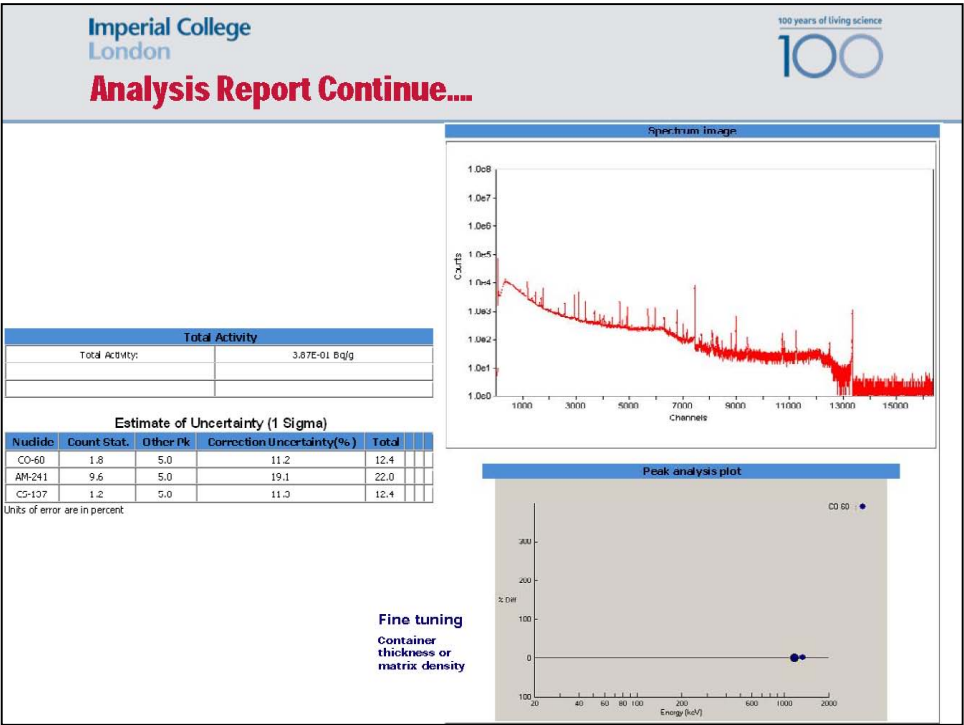
Calibration			
File name:	C:\user\CLB L1 NPL2009 SH30 MAHPG3 TOP2.Cb		
Energy calibration	Created:	7/11/2009 09:45:08	
	Zero offset:	0.2651113	
	Gain:	0.1947367	
	Quadratic:	-0.1436E-09	
Efficiency calibration	Created:	7/11/2009 09:45:18	
	Type:	Quadratic	
	Uncertainty Above Knee:	1.60%	
	Uncertainty Below Knee:	0.00%	
	Coefficients (Above the Knee):	-5.19996E4, 7.79340E-02, -6.140724E-02	
	Coefficients (Below the Knee):	-11.27073, 2.408789, -0.2834525	
	Deviation calibration point from reference (keV):	30.0 keV	

Library	
Main analysis library:	C:\user\IL0101\Library.Lib
Library match with:	0.5

Analysis parameters			
Quiet channel:	100	Stop channel:	10000
Peak cutoff (%):	20.0	Peak search sensitivity:	3
MFA type:	"RTPC" Traditional	Background type:	K Peak
Base Activity Units:	Bq/g	Activity scaling factor:	1.0000E+01/1.0000E+0
Activity Label:	Bq	Quantity Label:	g
Relative error (%):	1.0	Synthetic error (%):	0.0
Directed fit:	No	Uncertainty in percent:	No
Counting uncertainty only:	No	Confidence level (sigma):	1

Spectral corrections		
Decay correct to date:	Yes	Decay correction date/time:
PBC correction:	Yes	PBC File:
		PBC: Iso000001-MAHPG3 Detector 01-07-2009.Pbc







AWE assay of NPL drum

S P Holloway
steven.holloway@awe.co.uk
www.awe.co.uk



Introduction

- Drums measured using two techniques
 - AWE Non Destructive Assay Group, using a portable High Resolution Gamma Spectrometer (HRGS) and a simple spreadsheet analysis
 - AWE Waste Management Group using a HRSGS (High resolution Segmented Gamma ray Scanner) and analysis using Canberra's NDA2K software



Portable HRGS



3



Portable HRGS - Drum A (low activity)

- Scanned using low resolution handheld gamma detector to locate hotspot and determine approximate extent – indicated single small activity region near to drum's surface
- HRGS measurements using a ~40% electrically cooled HPGe detector -
 - From opposing sides of drum at 50cm from wall
 - Transmission measurements (^{241}Am , ^{137}Cs & ^{60}Co sources)
 - Background
- Spreadsheet model setup for opposing measurements assuming -
 - Point source of activity at variable distance into drum
 - Average measured linear attenuation coefficient of matrix
 - 1mm steel drum wall
- Least squares fitting used to determine effective distance into drum of activity (to get the same answer from both sides)
- Uncertainty contains components due to -
 - Counting statistics
 - Fitted activity location
 - Linear attenuation coefficient measurements (dominates)
 - Drum wall thickness
 - Activity and standoff of sources used to calibrate detector efficiency

4



Portable HRGS - Drum B (high activity)

- Scanned using low resolution handheld gamma detector to locate hotspot and determine approximate extent – indicated single small activity region close to drum's surface
- Gamma image to determine distance of activity into drum
- HRGS measurements using a ~100% HPGe detector -
 - From opposing sides of drum at 100cm from wall
 - Background
- Model setup assuming -
 - Point source of activity at measured distance into drum
 - Linear attenuation coefficient of matrix calculated using opposing measurements
 - 1mm steel drum wall
- Uncertainty contains components due to -
 - Counting stats
 - Distance into drum of activity
 - Measured linear attenuation coefficient
 - Drum wall thickness
 - Activity and standoff of sources used to calibrate detector efficiency
- Drum B was also measured on a turntable and analysed assuming a uniform activity - the result was very similar

5



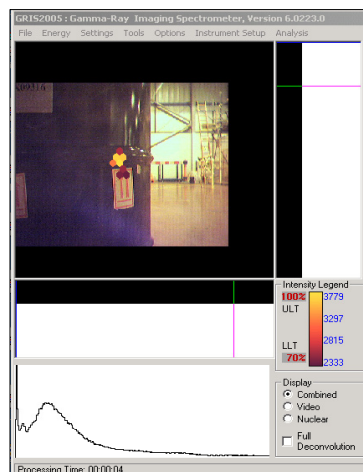
Gamma Imaging



6



Gamma Imaging



7



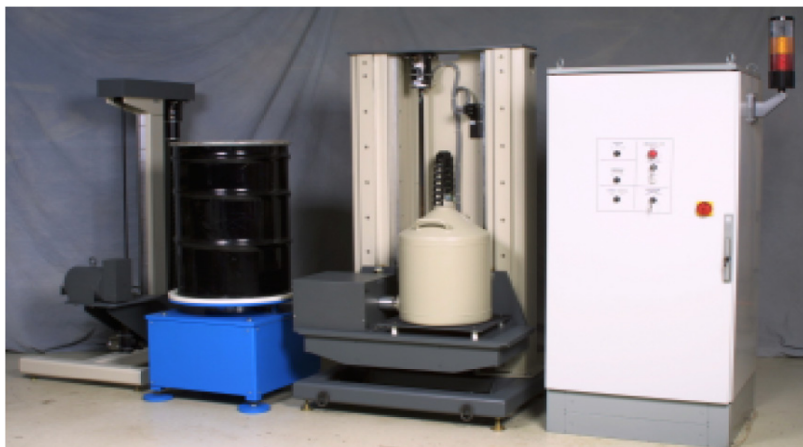
High Resolution Segmented Gamma Scanner

- Uses a collimated high resolution gamma detector to effectively split the drum into eight vertical slices
- A low energy filter prevents the measurement of ^{241}Am at low levels
- At each position a transmission measurement is also made
- Result assumes uniform activity distribution within each slice
- Uncertainty only contains counting statistics as the energies measured are not what the system is configured to measure - for Plutonium or Uranium a spatial component would be included

8



HRSGS



9

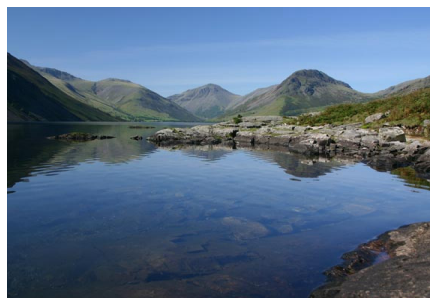
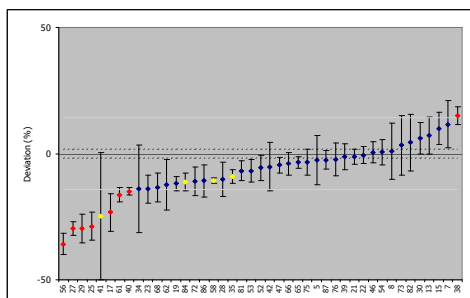


Results

Drum A (Bq/g)			
	NDA	SGS	NPL
Am	0.139 ± 0.022	not measured	0.1144 ± 0.0014
Co	0.122 ± 0.007	0.0804 ± 0.0071	0.1127 ± 0.0010
Cs	0.107 ± 0.006	0.0932 ± 0.0133	0.1085 ± 0.0016
Drum B (Bq/g)			
	NDA	SGS	NPL
Am	5.14 ± 0.74	not measured	4.022 ± 0.034
Co	2.54 ± 0.11	1.72 ± 0.06	2.428 ± 0.012
Cs	2.91 ± 0.13	2.24 ± 0.08	2.888 ± 0.040

10

S samples in the 2009 Environmental Radioactivity PTE



Arvic Harms and Chris Gilligan
National Physical Laboratory
25 February 2010

NPL Environmental Radioactivity PTEs

Started in 1989; 15 exercises organised so far (now on an annual basis)

Early exercises mainly UK participants

Activities traceable to NPL and ultimately to BIPM

Partly funded by the NMS and the participants

Latest completed exercise in 2009

73 participants (36 UK)

7 sample types (1 solid)

1214 results (71% in agreement)

Report will be published in April 2010



Radionuclides in S sample

6 nuclides:

^{55}Fe , ^{90}Sr , ^{133}Ba , ^{134}Cs , ^{137}Cs and ^{152}Eu

Participants: 26

Samples made by slow hydrolysis of a solution
resulting in solid SiO_2

S composition (Bq g^{-1})

	NPL	Labs	<i>t</i> -test	Ratio
^{55}Fe	4.38(13)	4.09(21)	-1.16	0.93
^{90}Sr	0.810(20)	0.757(17)	-2.01	0.93
^{133}Ba	4.91(13)	4.55(3)	-2.79 D	0.93
^{134}Cs	11.8(3)	11.05(5)	-2.38	0.94
^{137}Cs	5.05(13)	5.15(4)	0.78	1.02
^{152}Eu	4.84(13)	4.67(4)	-1.25	0.97

Homogeneity uncertainty

Measure each samples once:

$$u_{bb} = \sqrt{\frac{\sum_i (x_i - \bar{x}_i)^2}{n-1}} \left(\frac{100}{\bar{x}_i} \right)$$

Measure one sample m times:

$$u_{meas} = \sqrt{\frac{\sum_j (x_j - \bar{x}_j)^2}{m-1}} \left(\frac{100}{\bar{x}_j} \right)$$

Internal (counting) uncertainty:

$$u_{int} = 100 \text{ mean} \left(\frac{u_i}{x_i} \right)$$

Homogeneity uncertainty:

$$u_{hom}^2 = u_{bb}^2 - u_{meas}^2$$

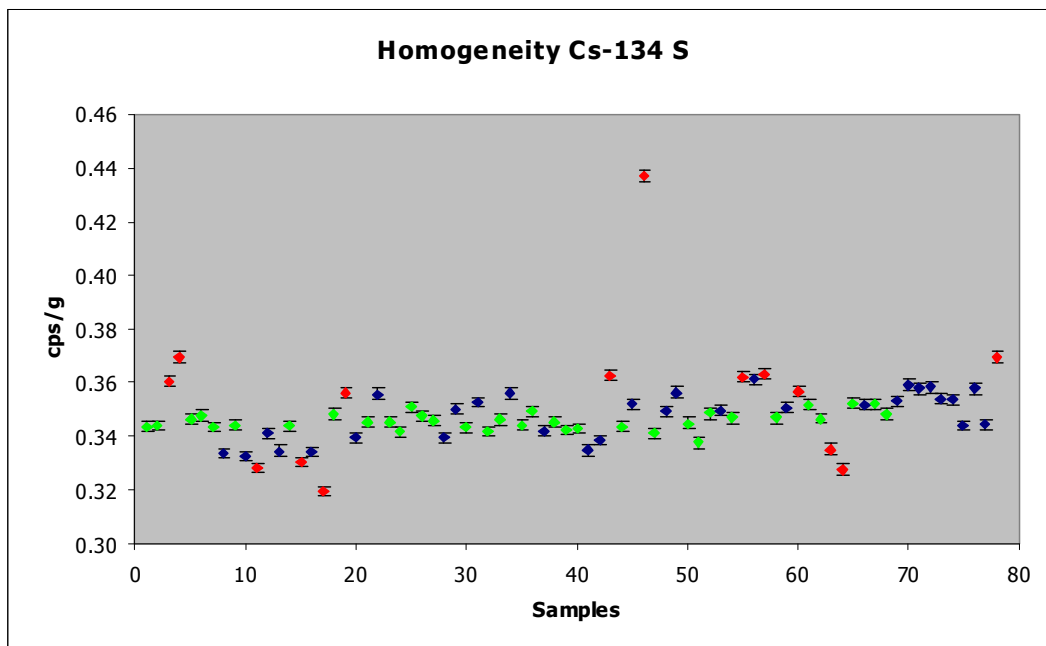
or

$$u_{hom}^2 = u_{bb}^2 - u_{int}^2$$

whichever gives the lowest result

Homogeneity uncertainty

	u_{bb} (%)	u_{meas} (%)	u_{int} (%)	u_{hom} (%)
^{133}Ba	1.34	1.61*	1.03	0
^{134}Cs	0.95	1.41*	0.55	0
^{137}Cs	1.19	1.73*	0.89	0
^{152}Eu	1.73	2.42*	1.42	0
	u_{N^*} (%)	u_{hom} (%)	u_{stab} (%)	u_N (%)
^{55}Fe	1.73	-	2.5	3.04
^{90}Sr	0.20	-	2.5	2.51
^{133}Ba	0.71	0	2.5	2.60
^{134}Cs	0.68	0	2.5	2.59
^{137}Cs	0.69	0	2.5	2.59
^{152}Eu	0.68	0	2.5	2.59



S in agreement (%)

(size of LCS)

⁵⁵ Fe	67	(67%)
⁹⁰ Sr	78	(78%)
¹³³ Ba	64	(82%)
¹³⁴ Cs	70	(78%)
¹³⁷ Cs	88	(88%)
¹⁵² Eu	68	(82%)

Results S

73% 'in agreement'
12% 'questionable'
16% 'discrepant'

