# Supplementary comparison CCRI(I)-S3 of standards for absorbed dose to water in $^{60}$Co gamma radiation at radiation processing dose levels

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## Abstract

A supplementary comparison of standards for absorbed dose to water in $^{60}$Co fields used for calibrations at radiation processing dose levels has been completed. Alanine dosimeters from the NIST and the NPL were used as transfer dosimeters and all participant irradiations were carried out in self-shielded irradiators. Irradiations were also carried out at the BIPM to allow direct comparison of dose ratios with the Key Comparison BIPM.RI(I)-K4. No significant difference was seen between the dose ratios obtained using the NPL and NIST alanine systems, no significant impact of mailing on dosimeter response was noted, and the adopted protocol limited any dose rate effects to the level of Type A uncertainties. The national standards of the participants are in agreement within the standard uncertainties, which are in the range from 1 to 2 parts in $10^2$.

## 1. Introduction

This comparison is the third overseen by the CCRI(I) to address the calibration and measurement capabilities of National Metrology and Designated Institutes for high-dose $^{60}$Co irradiations. This follows on from two previous comparisons (CCRI(I)-S1, Burns *et al*, 2006; CCRI(I)-S2, Burns *et al*, 2011). A significant difference from these earlier comparisons was that the protocol used here limited all irradiations to standardized calibration irradiators (i.e., not beams produced by commercial irradiation plants). The intent was not to compare realistic calibrations provided by the participants but rather the standards used to disseminate such CMCs, through the delivery of reference irradiations. The irradiator used was to be under the control of the participating laboratory, the use of any external client irradiator or irradiation plant was not permitted for this comparison. A second difference from the –S1 and –S2 comparisons was that a single dose level would be delivered by each participant. High-dose calibrations are generally provided in the range 5 kGy to 50 kGy, but for the reference irradiators, such as the Gammacell 220[1] used by many institutions, delivering such a range of doses is merely a matter of changing the irradiation time. Dose rate, therefore, is the fundamental quantity of interest. This allows a much narrower (and lower) range of irradiation doses and means that the performance of the transfer dosimeter itself (alanine) is not being tested. This latter point was an issue in the previous comparison, where apparent inter-lab differences were ultimately due to dose/dose rate effects of the alanine dosimeters (Desrosiers *et al*, 2008).

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## 2. Participants

Six laboratories participated in the comparison and these are listed in Table 1, together with other information relevant to the comparison. As in the previous comparison, the NIST and the NPL also acted as the laboratories issuing the transfer dosimeters. The BIPM provided reference irradiations to allow a link to be made with the BIPM.RI(I)-K4 key comparison (absorbed dose to water in $^{60}$Co). The NRCC acted as the organizing laboratory, analysing the data and producing the comparison report**.**

**Table 1: Summary of absorbed dose to water standards at participant laboratories**

| Institution | Country | Standard of absorbed dose to water in $^{60}$Co reference field | Transfer to high-dose irradiator | Nominal dose rate (kGy h$^{-1}$) | *u* (%) | Reference |
|---|---|---|---|---|---|---|
| CNEA-LDAD | Argentina | Graphite calorimeter (NPL) | Alanine dosimeter | 6 | 1.7 | ISO/ASTM 51607 |
| NIM | China | Fricke dosimeter | No transfer† | 1.5 | 0.9 | Zong *et al* (1998) |
| DTU-Risø | Denmark | Graphite calorimeter (NPL) | Alanine dosimeter | 5.4 | 1.4 | Risø HDRL Quality Manual, (2021) |
| NIST | United States of America | Water calorimeter | Alanine dosimeter | | 1.1 | NIST (2022) |
| NPL | United Kingdom | Graphite calorimeter | Alanine dosimeter | 9.5 | 1.1 | Kessler et al (2019). NPL (2022) |
| OAP | Thailand | Fricke Dosimeter | Alanine dosimeter | 2.25 | 1.3 | |
| BIPM | | Primary standard ionization chamber | No transfer† | 0.024 | 0.2 | Kessler and Burns (2018) Burns and Kessler (2018) |

† No irradiator employed; alanine transfer dosimeters irradiated directly in $^{60}$Co reference field

## 3. Transfer dosimeters, irradiations and measurements

### *3.1 Transfer dosimeters*

The NIST alanine dosimeters for use in the $^{60}$Co source comparisons were supplied in watertight cylindrical holders nominally 12 mm in diameter with a tolerance of 1 mm and 29 mm in length; each vial contained four alanine pellets (nominal dimensions 5 mm diameter, 3 mm thick). The NPL alanine dosimeters were supplied in cylindrical holders that could be made waterproof, 11.5 mm diameter (designed to fit a 12.0 mm hole) and 17 mm in length, containing 4 alanine pellets (nominal dimensions 5 mm diameter, 3 mm thick).

### *3.2 Irradiation procedure*

Each participating laboratory was issued a set of eight alanine transfer dosimeter vials from the NIST and a second set of eight dosimeters from the NPL, together with instructions on care and handling of the dosimeters. Participants were required to irradiate the vials to a dose constrained by the following:

i) Time of irradiation was to be in the range 10 minutes to 40 minutes to avoid any transit effects being significant and to simplify temperature control/monitoring.

ii) The dose delivered was to be in the range 1 kGy to 5 kGy, but ideally as close to 1 kGy as possible. This assumed a maximum dose rate for a reference irradiator of around 20 kGy $h^{-1}$.

iii) The same dose was to be delivered to each dosimeter (i.e., a range of doses was not required).

The NIST and the NPL also sent alanine dosimeters to the BIPM to be irradiated to 1 kGy in the BIPM therapy-level irradiator, as a reference. These were the only irradiations carried out in a low-dose rate beam.

An indirect comparison should be minimally impacted by any influence quantities of the transfer dosimeter being used, and this meant that for the alanine dosimeters used, all participants were to irradiate to similar dose values that should be close to the BIPM reference value of 1 kGy, within the constraints of (i) above. It was assumed that participants in this comparison had previously characterized fully their dose delivery system (i.e., validating the linearity of the irradiation timer, determining any transit dose) and therefore a single dose value was sufficient to compare different institutions.

After irradiation, the dosimeters were returned to the NIST and the NPL (as appropriate) for readout. The participant provided information on the date of irradiation, the average irradiation temperature, the temperature range and the nominal dose, but not the actual delivered dose. The nominal dose supplied to the NIST and the NPL is required to assist in the alanine measurement process. Formal dose declarations by the participants were submitted to the NRCC for compilation and analysis.

The irradiation geometry was not specified in detail in the protocol provided to the participants; it was preferred that each irradiating laboratory use their normal arrangement. This approach was taken so that the dose estimates would be representative of those routinely disseminated by each laboratory, rather than modified for the purpose of the comparison. The protocol did provide a number of recommendations:

i) The irradiations should be performed in a phantom large enough to achieve electronic equilibrium (preferably water-equivalent material, although water may be used).

ii) The radiation should be incident on the side of the cylindrical dosimeter in order to avoid a significant variation in dose along the stack of alanine pellets within the dosimeter.

iii) The irradiation geometry should be such that the irradiation temperature does not exceed 40 °C. Any conflict between these requirements and the normal arrangement used at each laboratory should be resolved well in advance of the irradiations.

It was not expected that there would be very large differences between laboratory protocols, since reference irradiators are very similar in their geometries.

From each batch of ten dosimeters sent to the participants, the two identified controls were to be kept un-irradiated and the instructions supplied with the dosimeters by the issuing laboratory were to be followed, particular in regard to storage temperature before and after irradiation (20 °C to 30 °C) and relative humidity (below 60 %).

### *3.3 Irradiations*

All irradiations took place between July and September 2019. Final information was supplied to the organizing laboratory in mid- 2021. The participants were asked to provide information on the conditions of irradiation, including the actual dose delivered according to their protocol, phantom material and size, the dose rate of the irradiating source, the derivation of the absorbed dose for each step of the calibration procedure, and a detailed list of uncertainty components.

### *3.4 BIPM irradiation procedure*

It is worth detailing the BIPM irradiation procedure as it is the necessary step to allow this supplementary comparison to be linked to the Key Comparison for absorbed dose to water in $^{60}$Co. The geometry is different from the self-shielded high-dose irradiators of the participants but was the same as used in the 2010 comparison (-S2). Four dosimeters (two from NPL, two from NIST) were irradiated simultaneously in PMMA sleeves at a depth of 5 g cm$^{-2}$ in the standard BIPM water phantom. The data were corrected for beam non-uniformity, for slight distance/depth differences, and the effect of the PMMA sleeve. The dose rate at the time of irradiation was around 0.024 kGy h$^{-1}$, resulting in an irradiation time of 40 hours to achieve approximately 1 kGy.

### *3.5 Alanine read-out*

The NPL dosimeters from all the irradiating laboratories, including those irradiated by the NIST, were measured at the NPL according to established procedures [NPL Technical Procedure QPDQL/B/D307] and the data sent to the organizing laboratory. A similar process was followed for the NIST dosimeters, including those irradiated by the NPL, according to the NIST procedures [Radiation Physics Division Quality System, Procedure 12].

## 4. Analysis of the results

The BIPM irradiations are important in providing a link between this comparison and the BIPM.RI(I)-K4 Key Comparison and also in removing any dependence on absolute dose measurements inherent to the NPL and NIST alanine systems. The significant difference in the dose rates between the participant fields and that of the therapy-level beam at the BIPM potentially impacts that normalization. However, in a recent two-way comparison of $^{60}$Co absorbed dose to water standards between the NPL and the NRCC (McEwen *et al*, 2019), irradiations were performed in both a therapy-level irradiator and a high-dose irradiator, and two independent alanine systems were used. Within the measurement uncertainties, there was no statistically significant difference in the results obtained with the two $^{60}$Co fields.

Although the delivered dose was intended to be close to 1 kGy, the protocol constraints did allow some variation. As a test of the sensitivity of alanine to delivered dose, two sets of NPL alanine dosimeters were irradiated in the Nordion Gammacell 220 high-dose $^{60}$Co irradiation field (traceable to the NRCC therapy-level primary standard). Nominal delivered doses were 1 kGy and 5 kGy, providing a test of linearity independent of the participants. The results indicated a maximum dose-dependent effect of 0.2% per kGy. It is not expected that there would be any different behaviour for the NIST alanine pellets, and so this sensitivity measurement is used to estimate a common uncertainty component.

The analysis of the participant results was made in two steps. In the first step, two sets of dose ratios were obtained:

$$R^i_{NPL} = \frac{D^i_{w,del}}{D^i_{w,meas,NPL}} \qquad\qquad R^i_{NIST} = \frac{D^i_{w,del}}{D^i_{w,meas,NIST}}$$

where the subscript *i* refers to a participant, *del* indicates the delivered dose reported by the participant, and *meas* indicates the alanine dose as measured by the NPL or NIST. For each participant there will be a maximum of 16 values of *R* (8 for NPL dosimeter, 8 for NIST dosimeters).

In the second step the dose ratios are normalized via the BIPM irradiations. For example, for the NIST dosimeters the equation is:

$$R^i_{BIPM\ (NIST)} = \overline{\frac{D^i_{w,del}}{D^i_{w,meas,NIST}}} \times \overline{\frac{D^{BIPM}_{w,meas,NIST}}{D^{BIPM}_{w,del}}}$$

where now the mean values of the various ratios are used. The equation is the same for the NPL dosimeters, resulting in two values for each participant, $R^i_{BIPM\ (NIST)}$ and $R^i_{BIPM\ (NPL)}$. The final result is obtained by taking the mean of these two values (note that for the NPL and the NIST, there is only a single dose ratio obtained with the other laboratory's alanine system).

## 5. Uncertainty budgets

Uncertainty budgets are given in Appendix A, as provided by the participants. The standard uncertainty in the delivery of dose to the alanine dosimeters is in the range of 1 % to 2 %.

## 6. Results

Mean values and standard deviations of the eight dosimeters are given in the tables below, rather than presenting results for each dosimeter vial irradiated. Significant figures beyond that justified by declared uncertainties are provided for information.

**Table 2: Results obtained using NPL alanine dosimeters**

| Participant | Temp. (°C) | $D_{w,meas}$ (Gy) | $D_{w,del.}$ (Gy) | $R_{NPL}$ | st dev (%) |
|---|---|---|---|---|---|
| CNEA | 17.5 | 868.7 | 880 | 1.013 | 0.39 |
| NIM | 24.9 | 1109.0 | 1106 | 0.997 | 0.22 |
| DTU | 30.0 | 1272.1 | 1270 | 0.998 | 0.44 |
| NIST | 23.0 | 986.0 | 1000 | 1.014 | 0.25 |
| OAP | 24.7 | 1008.5 | 1000 | 0.992 | 0.50 |
| BIPM | 19.4 | 972.7 | 978 | 1.006 | 0.15 † |

† Note that the BIPM irradiations involved fewer dosimeters

**Table 3: Results obtained using NIST alanine dosimeters**

| Participant | Temp. (°C) | $D_{w,meas}$ (Gy) | $D_{w,del.}$ (Gy) | $R_{NIST}$ | st dev (%) |
|---|---|---|---|---|---|
| CNEA | 17.5 | 890.0 | 880 | 0.989 | 0.47 |
| NIM | 24.9 | 1126.8 | 1103 | 0.979 | 0.33 |
| DTU | 30.0 | 1291.4 | 1270 | 0.983 | 0.19 |
| NPL | 25.0 | 1729.3 | 1703 | 0.985 | 0.14 |
| OAP | 24.0 | 1026.4 | 1000 | 0.974 | 0.43 |
| BIPM | 19.4 | 988.5 | 980 | 0.991 | 0.32 † |

† Note that the BIPM irradiations involved fewer dosimeters

**Table 4: Results normalized to BIPM reference irradiations**

| Participant | $R_{BIPM\ (NPL)}$ | $R_{BIPM\ (NIST)}$ | $R_{BIPM\ (combined)}$ | Difference (%) ‡ |
|---|---|---|---|---|
| CNEA | 1.007 | 0.998 | 1.003 | 0.9 |
| NIM | 0.992 | 0.988 | 0.990 | 0.4 |
| DTU | 0.993 | 0.992 | 0.992 | 0.1 |
| NIST | 1.009 | - | 1.009 | |
| NPL | - | 0.994 | 0.994 | |
| OAP | 0.986 | 0.983 | 0.985 | 0.3 |

‡ Defined as $R_{BIPM\ (NPL)} - R_{BIPM\ (NIST)}$

## 7. Investigation of influence quantities

A number of checks were used to confirm that influence quantities had not biased or invalidated the results. These various analyses provide additional confidence in the data given in Table 4.

7.1 In addition to the eight dosimeters sent to each participant for irradiation, NPL included one control dosimeter containing alanine pellets previously irradiated by the issuing laboratory to 1 kGy. The data for these NPL control dosimeters, read-out at the same time as the participant dosimeters, is given in Table 5. These measurements indicate no impact of the mailing process. Given the similarities of the NIST and NPL equipment and procedures, these results can be considered typical of the NIST system as well.

**Table 5: Measurement of NPL control dosimeters**

| Participant | Ratio ($D_{meas}/D_{del}$) |
|---|---|
| CNEA | 0.9998 |
| NIM | 1.0050 |
| DTU | 1.0013 |
| NIST | 0.9940 |
| OAP | 0.9992 |
| BIPM | 1.0050 |
| Mean | 1.0007 |
| Std dev | 0.0041 |

7.2 The values for the ratios $R_{NPL}^{DTU}$ and $R_{NPL}^{CNEA}$ should be close to unity, since DTU and CNEA obtain their traceability from the NPL. The ratios are found to be consistent with unity within the uncorrelated uncertainties.

7.3 The value of the NPL/NIST dose ratio obtained from Table 4 is within 0.5 % of the same ratio obtained for the previous high dose comparison, CCRI(I)-S2.

7.4 The standard deviations of the dose measurements given in Tables 2 and 3 (column 6) are consistent with expected values for the two read-out laboratories.

7.5 A comparison of the intra-dosimeter and inter-dosimeter variations is a useful quality-control check, and can indicate consistency of the irradiation procedure and/or any dose distribution along the axis of the dosimeter. The standard uncertainty of the doses measured by individual pellets within a single dosimeter, was similar to the standard deviation of the mean dose measured by the set of dosimeters, for a single participant (mean values of 0.20 % and 0.26 % respectively, combining NPL and NIST results). This indicates that using mean values, as shown in Tables 2 and 3, is appropriate.

## 8. Analysis and degrees of equivalence

Table 4 shows very good agreement between the ratios obtained using the two dosimeter systems from the NPL and the NIST, with a mean difference of 0.4% and a root-mean-square difference of 0.5%.

A comprehensive analysis to derive an overall uncertainty in each $R_{BIPM}^{i}$ value is not straightforward due to the multiple components, correlations between certain participants and the combining of NPL and NIST alanine data. In addition to the uncertainties stated by the participants in Table 1, three other components were identified as significant contributors – the standard uncertainty of the BIPM references irradiations (0.19%); the typical Type A standard uncertainty in the alanine read-out systems (0.25%, from column 6 of Tables 2 and 3); and the difference in the routes to obtain $R^i_{BIPM}$ via the two alanine systems (standard uncertainty = 0.4%, from column 5 of Table 4, based on a rectangular distribution). The overall uncertainty in the $R^i_{BIPM}$ values is given in Table 6.

**Table 6: Estimate of overall standard (*u*) and expanded uncertainty (*U*) of the values of $R^i_{BIPM}$**

| Participant | *u* (%) | $U_{total}$ (%) |
|---|---|---|
| CNEA-LDAD | 1.74 | 3.4 |
| NIM | 1.07 | 2.1 |
| DTU | 1.41 | 2.8 |
| NIST | 1.16 | 2.3 |
| NPL | 1.21 | 2.4 |
| OAP | 1.41 | 2.8 |

A coverage factor of 2 is used to determine *U*, representing a confidence level of approximately 95%.

The expanded uncertainty values in Table 6 are consistent with those obtained in the previous comparison, but the level of agreement seen in Table 4 suggests a possible overestimation of uncertainties by some participants.

Degrees of equivalence are not derived for supplementary comparisons but it can be seen that all the values in Table 4 are in the range ± 1%, indicating very good agreement between the participants. All of the dose ratios $R^i_{BIPM}$ are consistent with unity, given the stated standard uncertainties of each participant.

## 9. Conclusion

A supplementary comparison of standards for absorbed dose to water in $^{60}$Co fields used for calibrations at radiation processing dose levels has been completed. Alanine dosimeters from the NIST and the NPL were used as transfer dosimeters and all participant irradiations were carried out in self-shielded irradiators. The national standards are in agreement within the standard uncertainties, which are in the range from 1 to 2 parts in $10^2$.

## 10. Acknowledgements

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## Appendix A: Uncertainty budgets (all values given as relative standard uncertainties)

### Table A.1: CNEA-LDAD uncertainty budget

| Component of uncertainty | Type A (%) | Type B (%) |
|---|---|---|
| Dose rate calibration | | 1.10 |
| Mass of pellets | | 0.40 |
| LDAD Calibration curve | | 0.99 |
| Dosimeter-to-dosimeter variation | 0.50 | |
| Dose conversion | 0.30 | |
| Temperature of irradiation | | 0.30 |
| Resolution | 0.03 | |
| Combined standard uncertainty (*k*=1) | 1.67 | |

### Table A.2: NIM uncertainty budget

| Component of uncertainty | Type A (%) | Type B (%) |
|---|---|---|
| $G_{(Fe3+)}$ = 1.61(mol·$J^{-1}$) | | 0.65 |
| $\varepsilon_{(F3+)}$ @ 25°C | 0.27 | 0.21 |
| Conversion from $D_{\text{Fricke}}$ to $D_{\text{water}}$ | | 0.20 |
| Radiation spectrum | | 0.20 |
| Variation in spectrophotometer absorbance | 0.33 | 0.25 |
| Other | | 0.30 |
| Combined standard uncertainty (*k*=1) | 0.94 | |

### Table A.3: DTU-Risø uncertainty budget

| Component | Type A (%) | Type B (%) |
|---|---|---|
| Dose rate calibration | | 1.31 |
| Correction for $^{60}$Co half-life | | 0.01 |
| Irradiation time | | 0.05 |
| Geometry of dosimeters | | 0.1 |
| Transient dose | 0.02 | |
| Combined standard uncertainty (*k*=1) | 1.32 | |

**Table A.4: NIST uncertainty budget**

| Component of uncertainty | Type A (%) | Type B (%) |
|---|---|---|
| Reference dose rate | 0.37 | 0.56 |
| Alanine response | 0.60 | 0.15 |
| Dose rate effect | | 0.10 |
| Temperature correction | | 0.10 |
| Calibration curve | 0.5 | 0.10 |
| Combined standard uncertainty (*k*=1) | 1.05 | |

**Table A.5: NPL uncertainty budget**

| Component of uncertainty | Type A (%) | Type B (%) |
|---|---|---|
| Calibration of secondary standard ionisation chamber. | | 0.6 |
| Use of chambers to calibrate alanine dosimeters in therapy-level $^{60}Co$ beam* | 0.3 | |
| Transfer of alanine calibration from therapy-level beam to high dose irradiator beam | 0.3 | 0.5 |
| Use of high dose irradiators to irradiate customer dosimeters | 0.7 | |
| Timing | | 0.05 |
| Combined standard uncertainty (*k*=1) | 1.1 | |

* Theratron - NPL $^{60}Co$ therapy irradiator. Dose rate 0.9 Gy / min January 2019

**Table A.6: OAP uncertainty budget**

| Component of uncertainty | Type A (%) | Type B (%) |
|---|---|---|
| $^{60}Co$ irradiator | | 0.01 |
| Timing | | 0.07 |
| Dosimeter to dosimeter variation | 0.34 | |
| Positioning | 0.95 | |
| Temperature coefficient | 0.85 | |
| Combined standard uncertainty (*k*=1) | 1.32 | |

**Table A.7: BIPM uncertainty budget**

| Component of uncertainty | Type A (%) | Type B (%) |
|---|---|---|
| Reference dose rate | 0.04 | 0.19 |
| Dosimeter positioning | | 0.02 |
| Radial non-uniformity | | 0.03 |
| Effect of dosimeter holder | | 0.05 |
| Timer | | 0.01 |
| Combined standard uncertainty (*k*=1) | 0.19 | |