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Australasian Gamma-ray Spectrometry Capability Exercise – 2013

Stephen Long

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by

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Executive Summary

International experience with significant radiological incidents has shown that extensive measurement of environmental samples is required (Japanese Ministry of Health, Labour and Welfare, 2013) (IAEA, 1988). Many of these samples would require analysis by gamma-spectrometry. ARPANSA's capacity to analyse samples could be overwhelmed by the response required by a significant radiological incident. Therefore, assistance from other laboratories might be required to respond to a significant radiological incident.

This exercise was conducted to ascertain the capability of Australasian laboratories with respect to general gamma-ray spectrometry. The purpose of the exercise was simply to determine whether the methods currently used by each participating laboratory produced acceptable results when applied to a sample containing radionuclides created in a nuclear reactor.

Six of the eight Australasian radioanalytical laboratories, identified in a previous report (Long & Sdraulig, 2014) as having gamma-spectrometry capabilities, participated in this exercise. Two laboratories did not participate because they specialised in analyses that did not include the radionuclides used in the exercise.

Participants were asked to analyse a solution obtained from an accredited supplier of Certified Reference Materials and Proficiency Testing products. The solution contained Am-241, Cs-134, Cs-37, Co-60 and Zn-65.

The reported results were assessed against the Performance Acceptance Limits defined by the supplier of the samples, for each radionuclide. The Performance Acceptance Limits approximate 95% confidence intervals of the performance that an experienced laboratory should achieve.

Twenty eight of the thirty reported results were within the Performance Acceptance Limits defined by the supplier.

Four of the six laboratories reported that they do not apply corrections for True Coincidence Summing (TCS). Two laboratories significantly overestimated the activity concentration of Zn-65 because they had not corrected the data from which they derive the detection efficiency for the effects of TCS. Three laboratories significantly underestimated the activity concentration of Cs-134 because they did not correct their measurement for the effect of TCS.

One participant was able to provide acceptable results for four of the five radionuclides despite having an efficiency calibration specifically designed for the analysis of naturally occurring radionuclides, as opposed to anthropogenic radionuclides.

Contents

1. Introduction

International experience with significant radiological incidents has shown that extensive measurement of environmental samples is required in response to the incident (IAEA, 1988) (Japanese Ministry of Health, Labour and Welfare, 2013). Many of these samples would require analysis by gamma-spectrometry. ARPANSA maintains a radiochemistry laboratory, in part, to respond to radiological incidents. Theoretically, this laboratory has the capacity to measure about 100 samples per day by gamma-spectrometry. However, even this capacity could be overwhelmed by the response required by a significant radiological incident. Therefore, assistance from other laboratories might be required to respond to such an incident.

A survey of Australasian radioanalytical laboratories (Long & Sdraulig, 2014) found that eight Australasian laboratories could provide gamma-spectrometry support to ARPANSA. However, this survey also found that there was considerable specialisation in the radionuclides analysed by these laboratories. This is because many of these laboratories exist to fulfil specific needs and are not funded or specifically tasked with providing analyses in the event of general radiological incident. Therefore, some laboratories may not be able to provide assistance for some radiological incidents, particularly those involving the dispersion of radioactive material created in a nuclear reactor.

This exercise was conducted to ascertain the capability of participating laboratories with respect to general gamma-ray spectrometry. It should be noted that this was not a Proficiency Test Exercise because participants were not judged as to the acceptability of their procedures. The procedures used by each laboratory are appropriate to their particular objectives. Rather, the purpose of the exercise was simply to determine whether the methods currently used by each participating laboratory produced acceptable results when used for another purpose.

2. Methodology

All eight Australasian radioanalytical laboratories (including ARPANSA) were asked to participate in the exercise. Six laboratories, including ARPANSA, agreed to participate. Two laboratories did not participate because they specialised in analyses that did not include the radionuclides used in the exercise.

ARPANSA purchased 6 vials of a standard concentrate from an accredited supplier of Certified Reference Materials and Proficiency Testing products. The concentrate was prepared by adding known amounts of Am-241, Cs-134, Cs-137, Co-60, Mn-54 and Zn-65 to de-ionised water and acidifying the solution with nitric acid to pH<2.

The supplier also provided a certificate of analysis stating the certified values of the 'made-to' activity concentration of each radionuclide after diluting 5 mL of the concentrate into 995 mL of 0.1M nitric acid. The certificate of analysis also included Performance Acceptance Limits approximating 95% confidence intervals of the performance that an experienced laboratory should achieve.

A vial was sent to each laboratory along with instructions for diluting the concentrate and a form for reporting their results. The participants were asked to use their regular procedures for measuring radionuclides in water by gamma-spectrometry and to report results within three weeks of receiving the vial.

2.1 Analysis of Results

For each radionuclide, ARPANSA calculated the Relative Bias (R) of the measured activity concentration (M), relative to the certified activity concentration supplied by the manufacturer (C):

Equation 1:
$$R = \frac{M-C}{C} \times 100$$

Due to the uncertainties in both the certified value (u_c) and the measured value (u_m), the Relative Bias for an individual radionuclide may not be statistically significant. Therefore, ARPANSA also calculated the U-test value (U):

Equation 2:
$$U = \frac{\sqrt{(M-C)^2}}{\sqrt{u_m^2 + u_c^2}}$$

The Relative Bias for an individual radionuclide is definitely statistically significant if U > 2.58 and may be statistically significant if U > 1.64.

If a participant under- or overestimated the uncertainty associated with a measurement, the U-test value will over- or underestimate the statistical significance of the Relative Bias. Therefore, ARPANSA also compared the measured value to the Performance Acceptance Limits provided by the supplier of the sample.

Each participant was provided with an individual report detailing the Relative Bias, U-test value and comparison with the Performance Acceptance Limits for each radionuclide. This report also indicated if there appeared to be systematic errors in the laboratory's measured values and indicated the potential sources of these errors, based on the results and responses from the Report Form.

The laboratories were given a designation based on the date they reported their results: laboratory 1 was the first to report, laboratory 2 was the second to report, and so on.

3. Results and Discussion

In interpreting the results of the survey, the information contained in Annex A may prove useful. The results for each radionuclide will be discussed separately because each tests specific issues for the laboratories.

3.1 Laboratory Calibration

As part of the reporting process, each laboratory was asked to supply details of how they calibrated their spectrometers. In particular, they were asked to report what radionuclides were used to derive the detection efficiencies. The calibration sources used by the participating laboratories can be characterised as either a mixed-gamma source or a naturally occurring radionuclides source.

Mixed gamma sources incorporate several anthropogenic radionuclides (Am-241, Cd-109, Cs-137 and Co-60) in a resin matrix. A mixed-gamma source may also include Co-57, Ce-139, Hg-203, Sn-113, Sr-5, Y-88, Cr-51, Mn-54 and Zn-65 to provide a broader data set. It should be noted that several of the radionuclides used in mixed-gamma sources exhibit True Coincidence Summing (TCS). These radionuclides are Ce-139, Co-60 and Y-88. As explained in Annex A, data derived from these radionuclides should be corrected for TCS or the detection efficiency will be underestimated at energies of 166, 898, 1173, 1332 and 1836 keV.

The radionuclides commonly incorporated in sources of naturally occurring radioactive materials (NORM) are Ra-226, Ra-228 and Th-228. The short-lived radioactive progeny of these radionuclides (particularly radioactive isotopes of Pb and Bi) are assumed to be in secular equilibrium with their parents and emit the gamma-rays used to derive the detection efficiency. As shown in Annex A, such sources exhibit considerable TCS and will generally underestimate the detection efficiency.

The type of source used to derive the detection efficiency by each laboratory is shown in Table 1. This table also lists whether the laboratories correct the data for TCS.

Table 1: Calibration sources used by each participant

Laboratory	Calibration Source	Correction for TCS Applied
1	Mixed-Gamma	Yes
2	Mixed-Gamma	No
3	Mixed-Gamma	Yes
4	NORM	No
5	Mixed-Gamma	No
6	Mixed-Gamma	No

While only two of the participants correct for TCS, it should be noted that many of these participants specialise in the analysis of specific radionuclides. TCS corrections may not be required for the particular radionuclides they specialise in. It should also be noted that tools to routinely calculate TCS corrections have only become available relatively recently and that many radioanalytical laboratories have not yet implemented these tools into their operations. As discussed in Annex A, this may cause problems in analysing the radionuclides used in this exercise.

3.2 Caesium-137

Five of the six participants use Cs-137 to generate the data used to determine the detection efficiency of their spectrometer. This radionuclide does not exhibit True Coincidence Summing. Furthermore, any parameterisation of efficiency with energy should match the measured value at the energy of the gamma-ray emitted by this radionuclide (662 keV) because any data at nearby energies are also unaffected by TCS.

The results of the participants are shown in Figure 1. In the figure (and the following four figures), the measured value is shown as a cross with the error bars indicating the 95% confidence interval of the measurement. The solid line indicates the reference value of the sample, while the upper and lower dashed lines indicate the upper and lower acceptance limits defined by the supplier.

All participants reported results within the acceptance limits defined by the supplier of the sample. Indeed, the 95% confidence interval for the measurement encompassed the reference value for all but one participant. When comparing the confidence interval of this participant to the rest, it appears that this participant may have underestimated the uncertainty associated with their measurement.

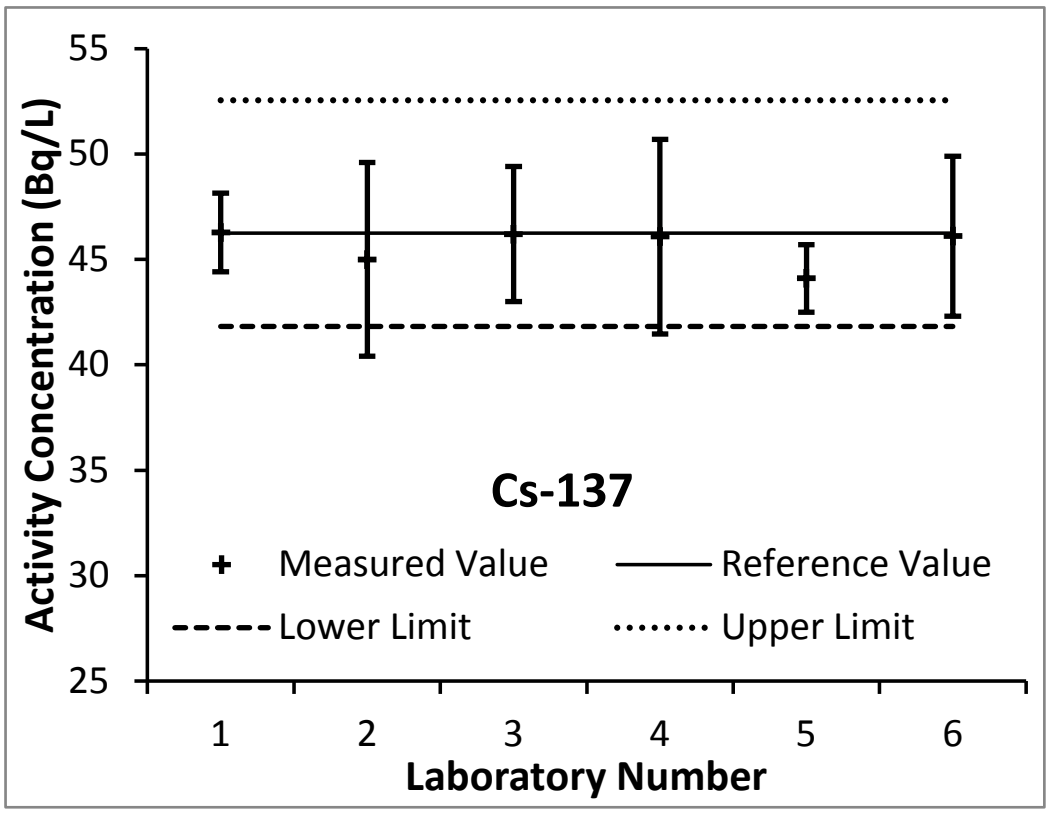

Figure 1: Reported results for caesium-137.
The error bars indicate the 95% confidence interval of the measurement.

3.3 Americium-241

Five of the six participants also use Am-241, which does not exhibit True Coincidence Summing, to calibrate their spectrometer. However, parameterisation of efficiency with energy may provide values significantly different from the measured value. This is because, at the energy of the gamma-ray emitted by this radionuclide (60 keV), the detection efficiency is rapidly changing with energy and there may be limited data on which to base the parameterisation in this energy region.

The results of the participants are shown in Figure 2. Five of the six participants reported values within the acceptance limits defined by the supplier of the sample. Laboratory 4 does not use Am-241 to derive the detection efficiency for their spectrometer. Indeed, the calibration source used by laboratory 4 provides very little data at low energies, so this deviation is to be expected. Two other laboratories reported 95% confidence intervals that did not encompass the reference value. The confidence interval reported by Laboratory 5 is significantly smaller than that reported by comparable laboratories, possibly indicating an underestimation their measurement uncertainty. With a U-test value of 1.85, the deviation shown by laboratory 3 might be statistically significant, but would require further testing to confirm this.

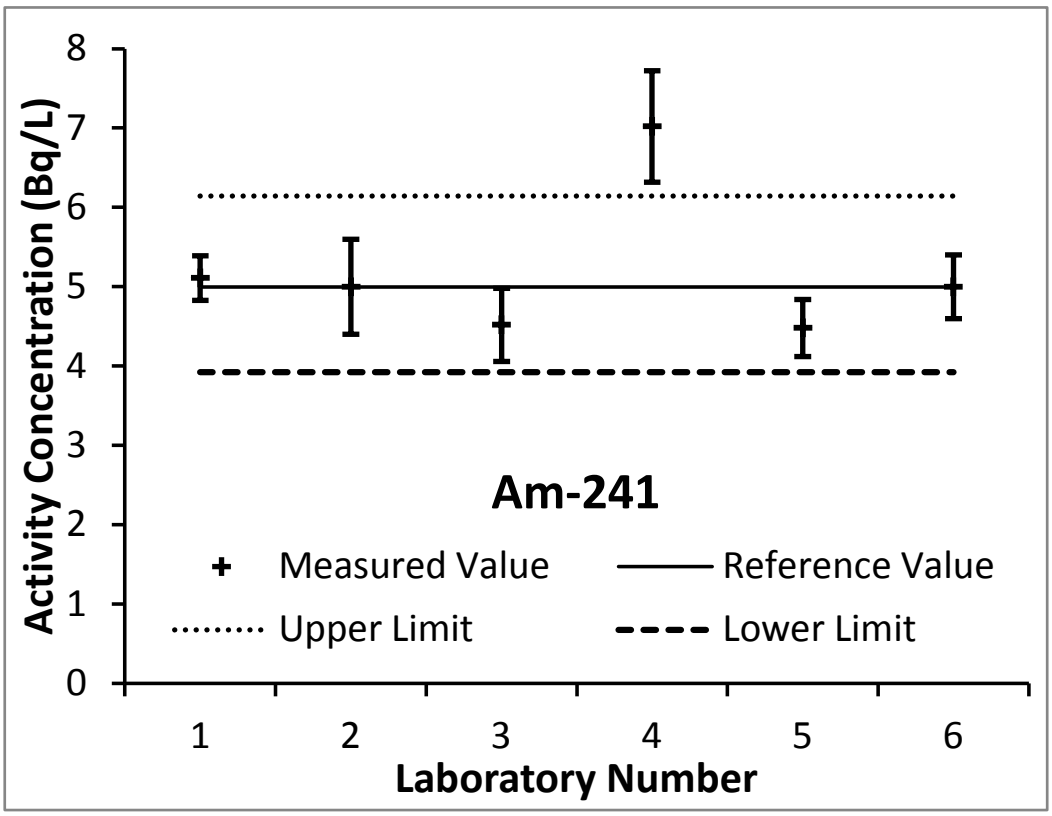

Figure 2: Reported results for americium-241.
The error bars indicate the 95% confidence interval of the measurement.

3.4 Cobalt-60

All but one of the participants use Co-60 to generate the data used to determine the detection efficiency of their spectrometer. Unlike Cs-137 and Am-241, this radionuclide does exhibit True Coincidence Summing. Therefore, the analysis for this radionuclide will test the effectiveness of the TCS correction procedures of the two laboratories that apply such corrections. For those laboratories that do not apply TCS corrections, their parameterisation of efficiency with energy should provide an effective efficiency value (see Equation 7) and, therefore, they should produce an acceptable result.

The results of the participants are shown in Figure 3. All of the participants reported values within the acceptance limits defined by the supplier of the sample. However, laboratory 4, which uses NORM radionuclides to derive their detection efficiencies, reported a result that had a relative bias of more than 10%. The uncertainty associated with the measurement was such that this bias may not be significant.

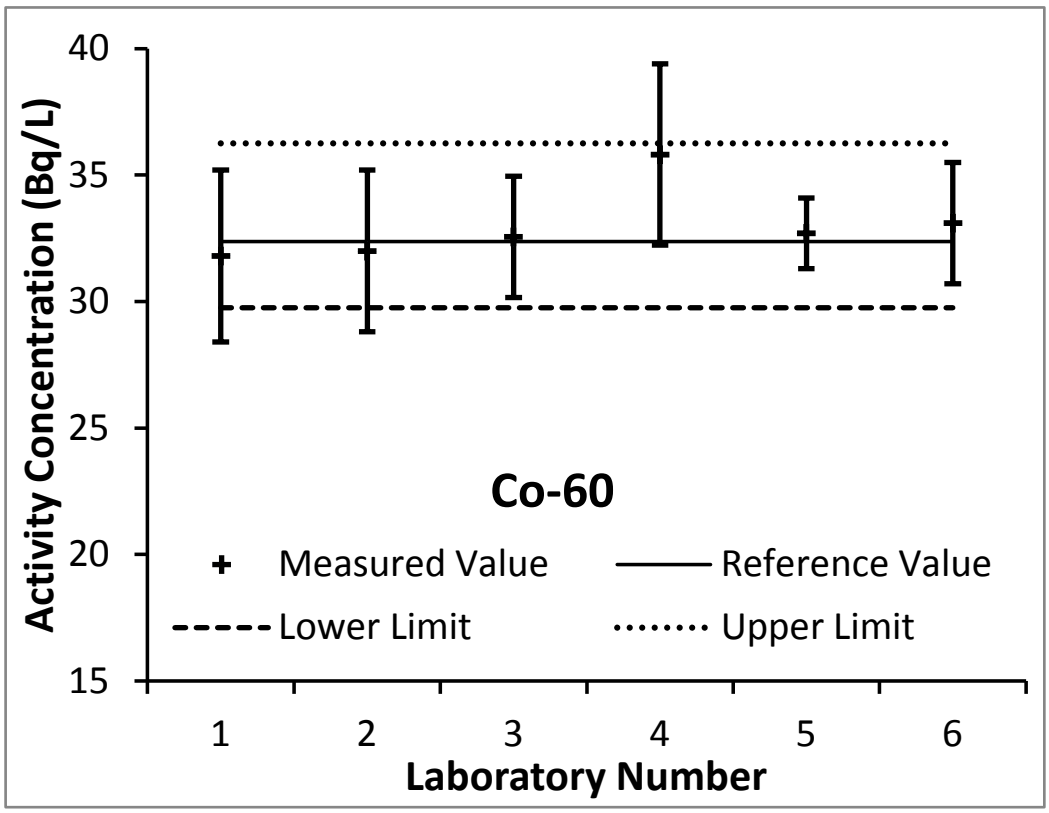

Figure 3: Reported results for cobalt-60.
The error bars indicate the 95% confidence interval of the measurement.

3.5 Zinc-65

The efficiency calibration sources used by only 3 laboratories contained Zn-65. While this radionuclide does not exhibit TCS, the radionuclides used to generate efficiency data at energies close to that emitted by Zn-65 (1116 keV) are affected by TCS (see Figure 7). For those laboratories that do not include Zn-65 in their efficiency calibration data, this may mean that they under-estimate the detection efficiency and, hence, overestimate activity. Two of the three laboratories that do include Zn-65 in their efficiency data set also correct for TCS effects, so their results indicate the effectiveness of the corrections they apply.

The results of the participants are shown in Figure 4. While 5 of the 6 participants reported values within the acceptance limits defined by the supplier of the sample, it should be noted that these acceptance limits are not equidistant from the reference value. The supplier states that 'the Performance Acceptance Limits closely approximate a 95% confidence interval of the performance that experienced laboratories should achieve using accepted environmental methods'. That is, the supplier recognises that many laboratories do not implement corrections for TCS and, therefore, overestimate the activity of this radionuclide. If failure to correct for TCS were considered an 'unacceptable' environmental method, laboratory 5 would have reported a value greater than the reduced acceptance limit.

Laboratories 4, 5 and 6 are those laboratories that do not include Zn-65 in their efficiency calibration data set. (It should be noted that the laboratory designation was determined by the reporting date and, therefore, that these laboratories are in numerical order is completely coincidental.) That these laboratories overestimate the activity of this radionuclide confirms that they are underestimating the detection efficiency in this energy region. In the case of laboratories 5 and 6, this underestimation is significant.

With a U-test value of 1.59, the bias exhibited by laboratory 1 is probably not significant. That is, it may be attributable to statistical variation in a single measurement, rather than systematic bias in their method.

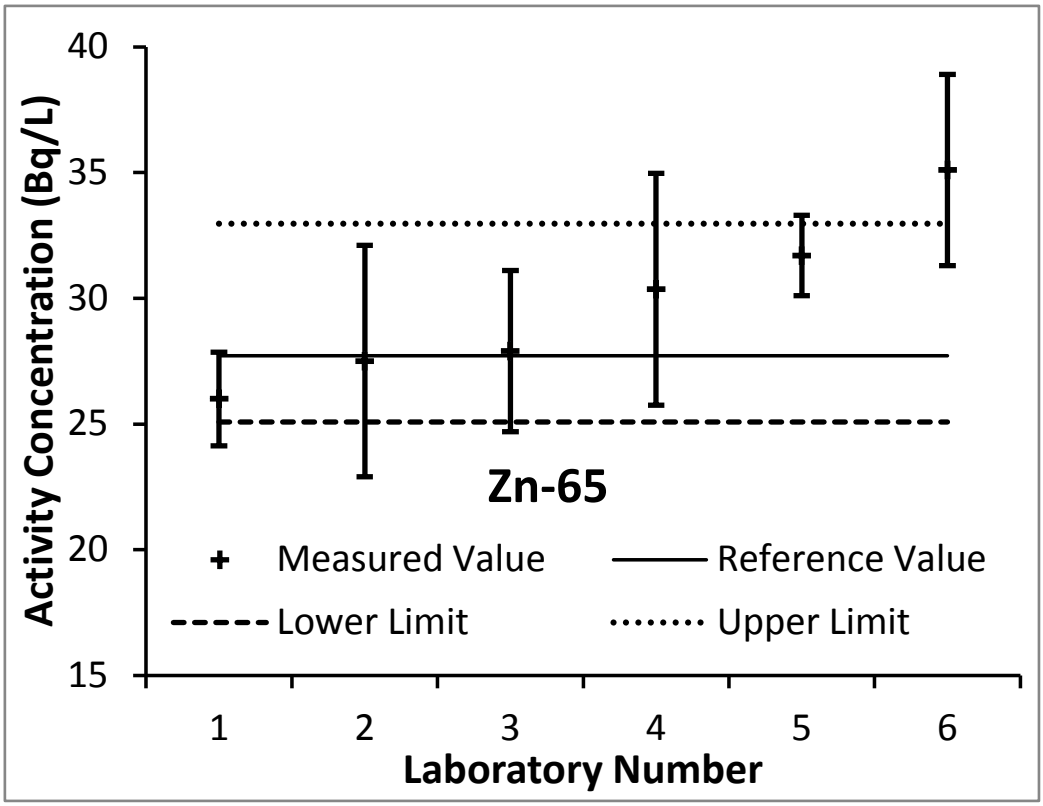

Figure 4: Reported results for zinc-65.
The error bars indicate the 95% confidence interval of the measurement.

3.6 Caesium-134

This radionuclide exhibits significant True Coincidence Summing and is not included in the efficiency calibration data set of any of the participants. For most participants, the efficiency calibration data in the energy region of the gamma-rays emitted by this radionuclide (600 – 800 keV) are unaffected by TCS. Therefore, most laboratories would have derived the activity using the actual detection efficiency in their analyses. However, the count rate of the Full-Energy Peaks associated with Cs-134 will be reduced by TCS effects. Therefore, those laboratories that do not apply a correction for TCS will underestimate the activity of this radionuclide. Accurate measurement of this radionuclide, therefore, poses a significant challenge to all of the participants.

The results of the participants are shown in Figure 5. It should be noted that, as was the case for Zn-65, the acceptance limits defined by the supplier of the sample are not equidistant from the reference value. In this case, the supplier recognises that many laboratories will underestimate the activity of this radionuclide. This is the case for laboratories 2, 5 and 6, all of whom would have reported values below a more rigorous acceptance range.

Laboratories 1 and 3 are those that apply TCS corrections to their results and both of these laboratories performed well.

It is worth noting that laboratory 4 uses NORM radionuclides to obtain its efficiency data and that these radionuclides are also affected by TCS. Therefore, this laboratory used an effective efficiency value to calculate the activity of this radionuclide. That their reported result was so close to the reference value indicates that the TCS corrections for the NORM radionuclides and for Cs-134 are approximately the same for that laboratory's measurement procedure. This may not be the case for other radionuclides (e.g. Rh-106).

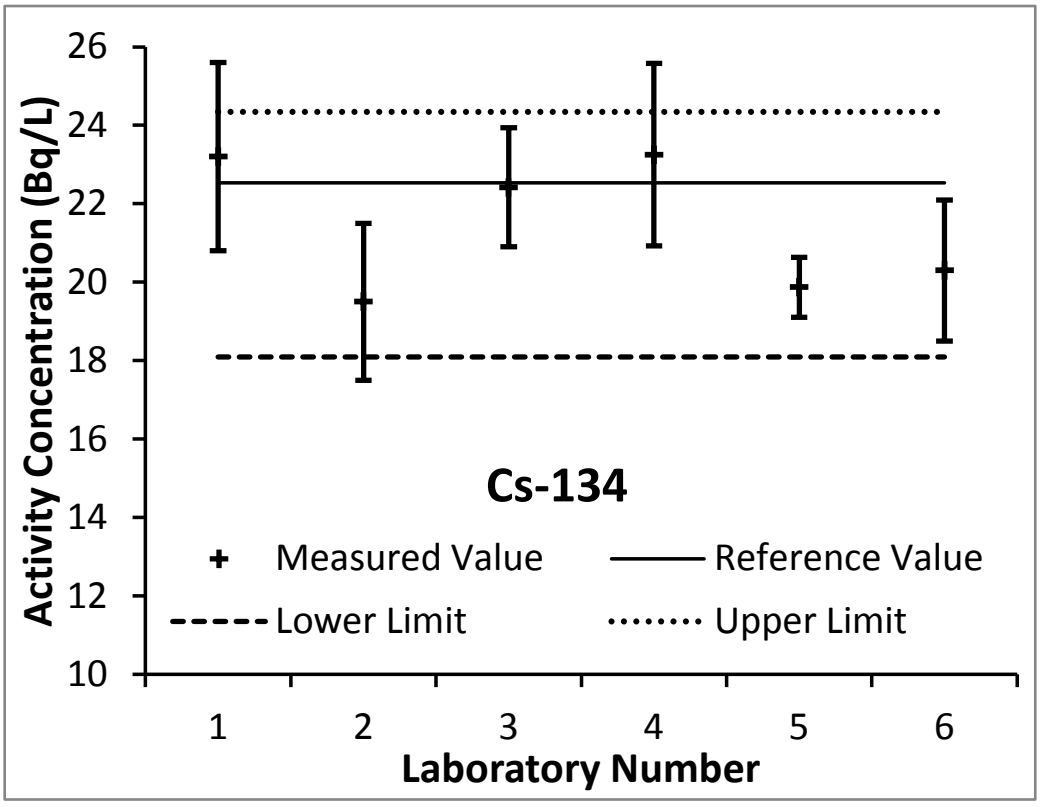

Figure 5: Reported results for caesium-134.
The error bars indicate the 95% confidence interval of the measurement.

4. Conclusion

This exercise was conducted to ascertain the capability of participating laboratories with respect to general gamma-ray spectrometry. It should be noted that this was not a Proficiency Test Exercise because participants were not judged as to the acceptability of their procedures. Rather, the purpose of the exercise was simply to determine whether the methods currently used by each participating laboratory produced acceptable results when applied to a sample containing radionuclides created in a nuclear reactor.

Six of the eight Australasian radioanalytical laboratories identified in a previous report (Long & Sdraulig, 2014) as having gamma-spectrometry capabilities participated in this exercise. The two laboratories that declined participation did so because their methods were specifically developed to analyse samples for radioisotopes from the uranium and thorium decay chains, rather than the anthropogenic radionuclides used in this exercise.

A vial of a standard concentrate from an accredited supplier of Certified Reference Materials and Proficiency Testing products was sent to each participant together with instructions for diluting the concentrate and reporting results. The laboratories were given a designation based on the date they reported their results: laboratory 1 was the first to report, laboratory 2 was the second to report, and so on.

In almost all cases, the participants reported results that would be considered acceptable by the supplier of the samples. That is, 28 of the 30 reported results were within the Performance Acceptance Limits defined by the supplier. Laboratory 4 reported an unacceptable value for the activity concentration of Am-241 and laboratory 6 reported an unacceptable value for Zn-65.

However, four of the six laboratories reported that they do not apply corrections for True Coincidence Summing. Two laboratories significantly overestimated the activity concentration of Zn-65 because they had not corrected the data from which they derive the detection efficiency for the effects of TCS. Three laboratories significantly underestimated the activity concentration of Cs-134 because they did not correct their measurement for the effect of TCS.

It is interesting to note that laboratory 4 produced acceptable results for four of the five radionuclides despite having an efficiency calibration specifically designed for the analysis of naturally occurring radionuclides, as opposed to anthropogenic radionuclides.

Annex A: Principles of Gamma-Ray Spectrometry (Gilmore, 2008)

The purpose of gamma-ray spectrometry is to both identify the radionuclides in a sample and to estimate the activity of those radionuclides. Identification of the radionuclides is based on relating the energy of the gamma-rays emitted by each radionuclide with the energy of the Full-Energy Peaks (FEPs) in the spectrum. The activity of the radionuclide is estimated by relating the count rate in the FEP with the emission rate of the corresponding gamma-ray.

Energy Calibration

The abscissa of a basic gamma-ray spectrum is the channel number of the Multi-Channel Analyser (MCA) used. The ordinate of the spectrum is the number of counts recorded in that channel during the counting period. The channel number of the MCA is proportional to the energy deposited in the detector by a detection event. The FEP is produced by gamma-rays of a particular energy depositing all of their energy in the detector, in individual events. So, energy calibration is done through the straightforward process of relating the channel number of the centroid of each FEP in a spectrum with the known energies of the gamma-rays emitted by a calibration source. In general, this relationship approximately linear:

Equation 3:
$$\mathbf{E = a + b \cdot N + c \cdot N^2}$$

Here *E* is energy, *N* is the MCA channel number and *a*, *b* and *c* are constants to be determined, where *c* is very small.

Efficiency Calibration

The relationship between the count-rate in the FEP and the activity of the radionuclide is:

Equation 4:
$$A = \frac{R}{\varepsilon \cdot \beta}$$

Here *A* is the activity, *R* is the count-rate, *ε* is the detection efficiency and *β* is the emission probability of the gamma-ray. The emission probability is a known nuclear property of the radionuclide of interest.

The detection efficiency is the probability that an emitted gamma ray will deposit all of its energy in the detector. This probability is dependent upon the energy of the gamma-ray, the particular detector used, the spatial relationship between the radioactive material and the detector and thickness and composition of any absorbing materials between the radioactive material and the detector (particularly the material through which the radionuclide is dispersed).

Measuring the detection efficiency can be as straightforward as measuring the count rate of the FEPs of interest produced by a source of known activity in the same geometric configuration and matrix as the samples to be measured. However, this assumes that the radionuclides of interest are limited in number and that calibrated sources are available. This is usually the case for naturally occurring radioactive materials.

Unfortunately, for anthropogenic radionuclides, there are only a limited number of calibration sources available. In order to calibrate for the several hundred anthropogenic radionuclides that may be of interest in general gamma-ray spectrometry, an empirical relationship between detection efficiency and energy is constructed based on a limited number of data points:

Equation 5: $$\mathbf{ln(\varepsilon) = \sum_i C_i \cdot (ln(E))^i}$$

The parameters, C_i, are determined by fitting the function to the measured data.

True Coincidence Summing

True Coincidence Summing (TCS) occurs when a radionuclide emits two or more gamma-rays within a few tens of nanoseconds of one-another. If both gamma-rays interact with the detector, it is recorded as a single detection event. In such a case, the energy deposited in the detector does not correspond to the energy of either gamma-ray. Therefore, this effect reduces the count rate in the FEP.

For radionuclides exhibiting TCS, an extra factor should be included in the relationship between activity and count rate:

Equation 6: $$\boldsymbol{A = \frac{R \cdot K_{TCS}}{\varepsilon \cdot \beta}}$$

Unfortunately, the correction for TCS, K_{TCS}, cannot be calculated analytically, except for the most trivial of cases. Fortunately, modern computing power enables routine calculation of the correction via Monte Carlo Modelling.

In cases where the calibration source used incorporates a radionuclide exhibiting TCS and no correction is made for it, the count-rate of the FEP will be underestimated. Therefore, the detection efficiency at that energy will also be underestimated. The effect can be seen in Figure 6. In the figure, the data affected by TCS are shown as unfilled circles.

The calibration sources usually used to derive the detection efficiency contain Y-88 and Co-60, both of which exhibit TCS, to generate data at higher energies. If this data is used to derive the relationship between energy and detection efficiency, without correction for TCS, then the derived relationship will predict detection efficiencies significantly lower than the actual detection efficiency. This is demonstrated in Figure 7. In this figure, the measurements affected by TCS are shown as unfilled circles, the fit to the uncorrected data as the solid line and the fit to corrected data as the dashed line. The fit to the uncorrected data significantly underestimates the detection efficiency.

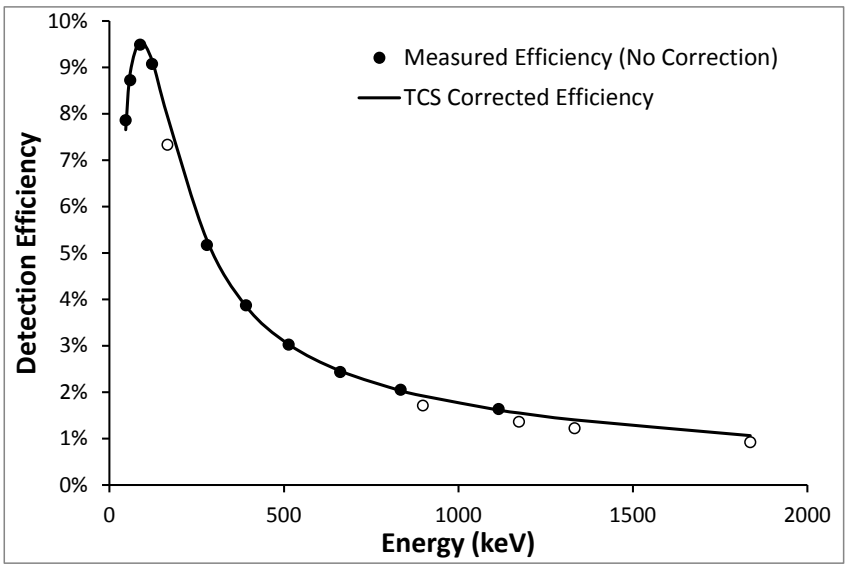

Figure 6: Measured detection efficiency showing the effect of TCS

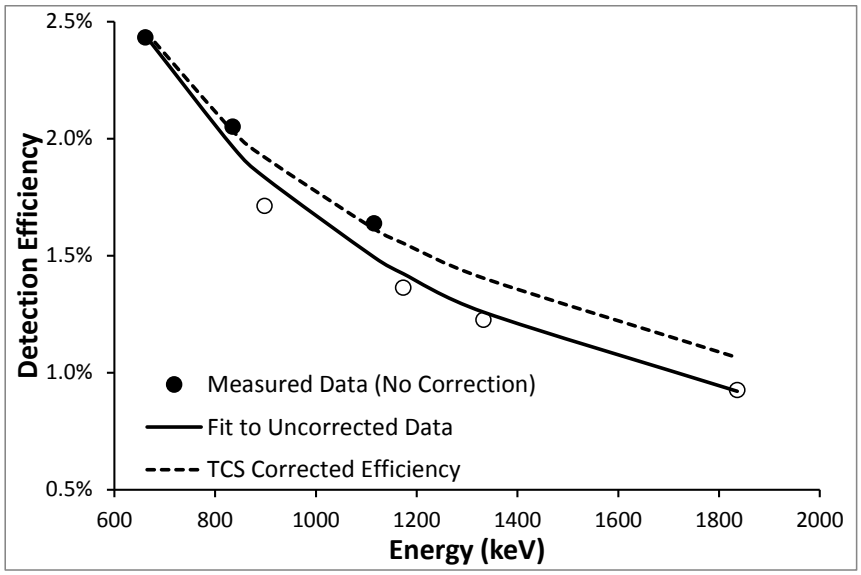

Figure 7: Underestimation of the detection efficiency at higher energies

Some laboratories use calibration sources containing naturally occurring (NORM) radionuclides (Ra-226, Ra-228 and/or Th-228) to generate efficiency measurements. Many of the radioactive progeny of these radionuclides exhibit TCS. Therefore, if no corrections are applied to these data, the measurements will significantly underestimate the detection efficiency. This can be seen in Figure 8.

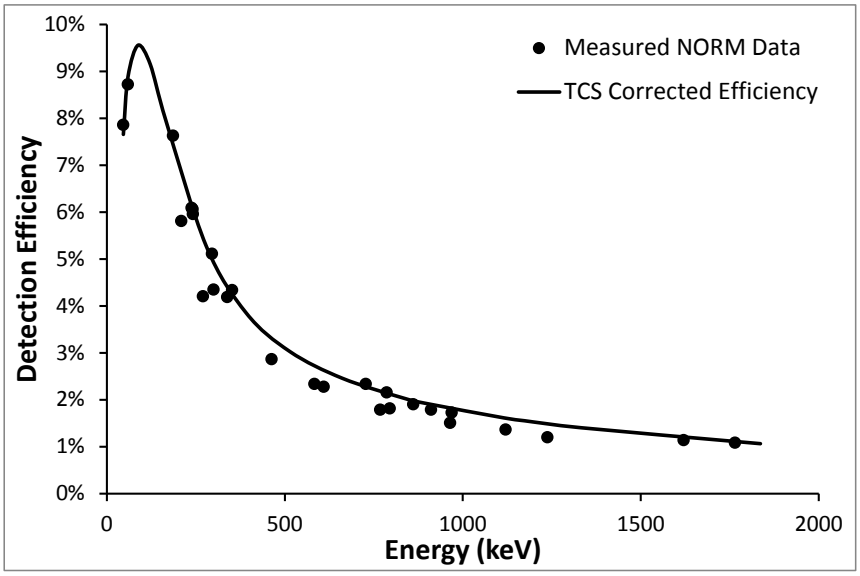

Figure 8: Efficiency data derived from NORM sources.

It should be noted that a correction for TCS need not be applied if the samples are analysed for only those radionuclides used in the calibration source. In such cases, the effective efficiency,ε', derived from the calibration source also includes the TCS correction factor:

Equation 7: $$A = \frac{R}{\varepsilon' \cdot \beta} \qquad \textbf{where} \qquad \boldsymbol{\varepsilon' = \frac{\varepsilon}{K_{TCS}}}$$

In such cases, a parameterisation, such as Equation 5 should not be used so as to ensure that exact values of ε' are used for each gamma-ray.

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