Australian Government
Australian Radiation Protection and Nuclear Safety Agency

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Technical Report Series No. 173

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Technical Report 173
ISSN 0157-1400
June 2016

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ISSN: 0157-1400

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EXECUTIVE SUMMARY

The measurement efficiency of the alpha-spectrometers used in ARPANSA's Radioanalytical Services (RAS) laboratory must be known in order to determine the chemical recovery for the measured sample. The software used in the RAS laboratory (Apex-Alpha) requires that the measurement efficiency be determined by comparing the measured count rate with the certified emission rate from a calibration source.

This methodology assumes that the sample and calibration source are exactly the same diameter. However, the samples prepared in the RAS laboratory are either 20 mm or 17 mm in diameter, while the commercially available calibration sources are either 24.1 mm or 7 mm in diameter.

This report details a procedure whereby standard diameter calibration sources may be used to determine the measurement efficiency for samples of non-standard diameters. The procedure takes advantage of the fact that, in alpha-spectrometry, the measurement efficiency is completely determined by the relative solid angle subtended by the source at the detector. The procedure calculates the relative solid angle subtended by sources of the diameters of interest using Ruby's formula, (Ruby & Rechen, 1958) and (Ruby, 1994). The procedure then modifies the activities of the calibration sources by the ratio of the relative solid angles. These modified activities are then input to the software so that it can calculate the measurement efficiency appropriate to the samples even though the count rate is produced by a calibration source of a different diameter.

The procedure was validated by using the measurement efficiencies produced by this methodology to calculate the chemical recovery for several hundred samples. The chemical recovery in these analyses was expected to be 100%. Those calculated from the measurement efficiencies produced by this procedure averaged 101%. This shows that the procedure is fit for the laboratory's purposes.

1. INTRODUCTION

The measurement efficiency of the alpha-spectrometers used in ARPANSA's Radioanalytical Services (RAS) laboratory must be known in order to determine the chemical recovery for the measured sample. The measurement efficiency is usually determined by comparing the measured count rate with the certified emission rate from a calibration source. However, this methodology requires that the calibration source is an accurate representation of the sample. In particular, the method assumes that the sample and calibration source are exactly the same diameter.

The samples measured in the RAS laboratory are produced by either an electrodeposition apparatus or an autodeposition apparatus. Each of these pieces of equipment was developed in-house by ARPANSA due to the absence of suitable commercial equipment. The electrodeposition unit produces samples that are 20 mm in diameter, while the autodeposition unit produces samples that are 17 mm in diameter.

Calibration sources must be purchased from a commercial supplier in order that they are traceable to an international standard of radioactivity. Unfortunately, the commercial supplier only provides sources of specific diameters, determined by the apparatus used in their manufacture. The sources obtained by ARPANSA are either 24.1 mm or 7 mm in diameter.

This report details a procedure whereby standard diameter calibration sources may be used to determine the measurement efficiency for samples of non-standard diameters.

2. METHODOLOGY

2.1 Geometric Efficiency

The procedure developed by ARPANSA takes advantage of the fact that, in alpha-spectrometry, the measurement efficiency is completely determined by the relative solid angle subtended by the source at the detector. This is because the Passivated Implanted Planar Silicon (PIPS®) detectors used in the RAS laboratory have extremely thin (<50 nm silicon-equivalent) entrance window. Such a thin window means that virtually none of the incident alpha-particles suffer measureable energy loss before entering the detector.

Ruby's formula, (Ruby & Rechen, 1958) and (Ruby, 1994), gives the relative solid angle, *G*, subtended at a disc source by a coaxial parallel-disc detector:

Equation 1:
$$G = \frac{R_D}{R_S}\int_0^{\infty} \frac{e^{-k\cdot d}}{k} \cdot J_1(k \cdot R_S) \cdot J_1(k \cdot R_D)\, dk$$

where R_D and R_S are the radii of the detector and source, respectively, d is the distance between the source and the detector and $J_1(x)$ is the Bessel function of first kind and order 1. While this integral cannot be solved analytically, the power of modern computers means that it can be calculated by numerical integration:

Equation 2:
$$G \cong \frac{R_D}{R_S}\sum_{k=0}^{K}\left[\frac{e^{-k\cdot d}}{k} \cdot J_1(k \cdot R_S) \cdot J_1(k \cdot R_D) \cdot \delta k\right]$$

Solving Ruby's formula using the measured values of the source diameter and source-detector separation, together with the diameter of the detector given by the manufacturer, would provide a value for the measurement efficiency. Unfortunately, the software used in the RAS laboratory (Apex-Alpha) is not able to accept direct entry of the measurement efficiency. Rather, the software requires measurement of a calibration source so that it can internally calculate the efficiency.

2.2 Activity Correction

The count rate, *R*, observed in the detector is related to the activity of the source at the time of the measurement, *A*, and the measurement efficiency, *ε*, via Equation 3 (L'Annnunziata, 2012) (please see Annex A: The Effect of Backscattering on Efficiency Measurements). It should be noted that Equation 3 assumes that the emission probability of the alpha-particle of interest is 100%.

Equation 3:
$$R = \varepsilon \cdot A$$

For a source of smaller diameter to give the same observed count rate as a source of larger diameter (R_1=R_2), and noting that the efficiency (ε) is equal to the relative solid angle (G), the relationship shown in Equation 4 must hold.

Equation 4:
$$G_1 \cdot A_1 = G_2 \cdot A_2$$

Rearranging Equation 4 shows that the activity of the smaller source, A_1, is related to the activity of the larger source, A_2, by the ratio of the relative solid angles:

Equation 5:
$$A_1 = \frac{G_2}{G_1} A_2$$

Equation 5 can be inserted into the formula normally used to calculate the measurement efficiency (noting that R_1=R_2):

Equation 6:
$$\varepsilon_1 = \frac{R_1}{A_1} = \frac{R_2}{\frac{G_2}{G_1} A_2}$$

Equation 6 shows that a source that is not the same diameter as the sample can be used to calculate the measurement efficiency for the sample so long as the activity is 'corrected' by the ratio of the relative solid angles.

2.3 Determining the distance between source and detector

In order to calculate the relative solid angle subtended at the source by the detector, the distance between the source and detector must be accurately known. Unfortunately, the experimental arrangement of the equipment used for alpha-spectrometry means that this distance cannot be directly measured. The reasons for this can be seen in Figure 1.

Figure 1: The arrangement of detector (silver) and source holder (red) in an alpha-spectrometry chamber (left) and the detectors used for alpha-spectrometry (right).

It is very difficult to insert a measuring instrument into the detector chamber, shown at the left of Figure 1, because it measures only 8.26 x 6.03 x 6.35 cm. The measurement of the distance between the source and the detector is further exacerbated by the fact that, for the measurements made in the RAS laboratory, the source holder is placed in the slot closest to the detector, so as to maximise detection efficiency for low-activity samples. The construction of the detector also creates difficulty in that the surface of the detector is recessed from the metal outer, as shown at the right of Figure 1. Furthermore, the face of the detector is fragile and should not be touched by anything that could scratch the surface. While the manufacturer of the equipment provides nominal distances between

the source and detector at each shelf position, this may not be accurate enough for the purposes of this procedure.

While the distance between the source and detector may not be accurately known, both the diameter of certified reference sources and the diameter if the detector are very well known. Therefore, by comparing calculations of relative solid angle at various source-detector separations to the measured efficiency for a certified calibration source, the distance between the source and detector may be accurately determined.

2.4 Calibration Procedure

Based on the discussion above, the proposed calibration procedure is:

1. Measure the efficiency for a calibration source placed at the same distance from the detector as a sample.
2. Determine the distance between the source and detector by comparison with calculated relative solid angles.
3. Calculate the relative solid angles for sources of same diameter as:
    a. the calibration source
    b. an electrodeposited sample
    c. an autodeposited sample
4. Calculate the 'corrected' activity of the calibration source such that it can be used to measure the efficiency of the electrodeposited or autodeposited sample.
5. Use the 'corrected' calibration source to determine the measurement efficiencies in the normal manner for Apex-Alpha.

3. APPLICATION OF THE PROPOSED CALIBRATION PROCEDURE

3.1 Determine the distance between source and detector

The count rate from a certified reference source was measured in a number of alpha-spectrometry chambers at the same shelf position as that used to measure samples. The source was comprised of Am-241 deposited over a circular area of 7 mm diameter. The certified emission rate from front surface of the source was 1.88 x 10^5 alpha particles per minute on February 15, 1990, with an uncertainty of 1%. This means that the 4π-emission rate, at the date of the measurements, was 6011 α/s, with an uncertainty of 1%.

The measured count rates and efficiencies are shown in Table 1. This table shows that the average measurement efficiency is 30.9%, with a standard deviation of 0.5%.

Table 1: Measured count rates and efficiencies for a selection of alpha-spectrometry chambers.

The quoted uncertainties indicate the 95% confidence interval				
Chamber	**Measured Rate (CPS)**		**Measured Efficiency**	
A1B	1854	± 11	30.9%	± 0.6%
A2A	1854	± 11	30.8%	± 0.6%
A5B	1866	± 11	31.0%	± 0.6%
B1A	1862	± 11	31.0%	± 0.6%
B3B	1823	± 11	30.3%	± 0.6%
B6A	1834	± 11	30.5%	± 0.6%
C2B	1833	± 11	30.5%	± 0.6%
C4A	1915	± 11	31.9%	± 0.7%
C6B	1895	± 11	31.5%	± 0.7%

The manufacturer of the alpha-spectrometry detectors used in the RAS laboratory states that the active diameter of these detectors is 23.9 mm. Using the values of 7 mm and 23.9 mm for the diameters of the source and detector, respectively, the relative solid angle was calculated for a range of source-detector separations. These calculations are summarised in Table 2.

The calculated relative solid angles listed in Table 2 may be compared with the average measured efficiency based on the data in Table 1. This comparison shows that the separation between the source and the detector is between 4.5 and 5.1 mm, with an average value of 4.8 mm. These values for source-detector separation are comparable to the manufacturer's nominal value of 5 mm. However, these results also show that using the nominal value would have introduced a significant systematic error into the rest of the procedure.

Table 2: Calculated relative solid angles for a range of source-detector distances.

The highlighted entry indicates the average source-detector separation	
Distance (mm)	**Relative Solid Angle**
4.0	33.7%
4.5	31.9%
4.6	31.6%
4.7	31.2%
4.8	30.9%
4.9	**30.5%**
5.0	30.2%
5.1	29.9%
5.5	28.6%
6.0	27.1%

3.2 The calculation of activity correction factors

The source used to calibrate the detectors used in the RAS laboratory comprised four radionuclides (U-238, U-234, Pu-239 and Am-241) distributed over a circular area of 24.1 mm diameter.

The samples produced by the autodeposition equipment in the RAS laboratory covers the entire surface of a 17 mm diameter silver disc with the element of interest.

The electrodeposition equipment used in the RAS laboratory utilises a 25 mm diameter stainless steel disc as the sample substrate. However, the sample is not deposited over the entire surface of the disc due to presence of an O-ring seal. Fortunately, the electrodeposition process sometimes darkens the area over which the source is deposited. Several darkened sample discs were measured to determine that the diameter of over which the sample is actually deposited is 20.0±0.1 mm.

These diameters, together with the source-detector separation determined in section 3.1 and the diameter of the detector listed by the manufacturer, enable the calculation of relative solid angles for each source, as shown in Table 3.

Table 3: The calculated relative solid angle for various diameter sources.

The diameter of the detector is 23.9 mm and source-detector distance is 4.8 mm. The 95% confidence interval for all distances is ±0.2 mm. The calculated 95% confidence interval for the relative solid angle is ±0.008.	
Source Diameter (mm)	**Relative Solid Angle**
24.1	24.0%
20.0	26.7%
17.0	28.2%
7.0	30.9%

3.3 Corrections to source activity

The ratios of the relative solid angles shown in Table 3 may be used to calculate 'correction factors' for the certified activities of the calibration sources such that they can be used to determine the measurement efficiency for the source diameters used in the RAS laboratory. The modified source activities are shown in Table 4.

Table 4: The modified activities such that the sources can be used to determine measurement efficiency for samples of 17 and 20 mm diameter.

The uncertainties quoted indicate a 95% confidence interval.				
Source # 0278	**U-238**	**U-234**	**Pu-239**	**Am-241**
24.1 mm Source Activity (Bq) [certified]	1.72	1.71	1.65	1.57
Uncertainty	2.4%	2.4%	2.3%	2.2%
20mm Source Activity (Bq) [modified]	1.54	1.54	1.48	1.41
Uncertainty	5.1%	5.1%	5.0%	5.0%
17mm Source Activity (Bq) [modified]	1.46	1.45	1.40	1.34
Uncertainty	5.0%	5.0%	4.9%	4.9%
Source # 0655	**U-238**	**U-234**	**Pu-239**	**Am-241**
24.1 mm Source Activity (Bq) [certified]	1.84	1.81	1.70	1.82
Uncertainty	3.1%	3.1%	3.1%	3.1%
20mm Source Activity (Bq) [modified]	1.65	1.63	1.53	1.64
Uncertainty	5.4%	5.4%	5.4%	5.4%
17mm Source Activity (Bq) [modified]	1.57	1.54	1.45	1.55
Uncertainty	5.4%	5.4%	5.4%	5.4%

Source # 0656	U-238	U-234	Pu-239	Am-241
24.1 mm Source Activity (Bq) [certified]	1.74	1.68	1.76	1.73
Uncertainty	3.1%	3.1%	3.1%	3.1%
20mm Source Activity (Bq) [modified]	1.56	1.51	1.58	1.56
Uncertainty	5.4%	5.4%	5.4%	5.4%
17mm Source Activity (Bq) [modified]	1.48	1.43	1.50	1.47
Uncertainty	5.4%	5.4%	5.4%	5.4%
Source #0657	**U-238**	**U-234**	**Pu-239**	**Am-241**
24.1 mm Source Activity (Bq) [certified]	1.90	1.87	1.86	1.88
Uncertainty	3.1%	3.1%	3.1%	3.1%
20mm Source Activity (Bq) [modified]	1.70	1.68	1.67	1.69
Uncertainty	5.4%	5.4%	5.4%	5.4%
17mm Source Activity (Bq) [modified]	1.61	1.59	1.58	1.60
Uncertainty	5.4%	5.4%	5.4%	5.4%

3.4 Determination of the measurement efficiencies

The modified activities listed in Table 4 were used to create 2 certificates, within the Apex-Alpha software, for each source: one certificate with the activities modified to represent a 20 mm diameter source and the other certificate with the activities representing a 17 mm diameter source. The measurement activity for each detector chamber was determined according to the procedure outlined in the software manual (Canberra Industries, 2011). The counting time for each measurement was set at 1800 s, so as to produce approximately 1000 counts in each peak in the spectrum.

The procedure calculated the measurement efficiencies according to Equation 3, where the count rate, *R*, is that produced by one of the sources and the source activity, *A*, is taken from the appropriate certificate, for each radionuclide. The average efficiency is then calculated from the results of the four radionuclides in the source.

The results for the efficiencies calculated from only the areas of the peaks due to uranium-238 are shown in Table 5. The results in the column for a source of 24.1 mm diameter are those calculated based on the uncorrected activity of the source used and, so, represent what would be used if no corrections for solid angle were made. The next two columns list the efficiencies calculated from source activity modified so as to represent a source of 20 or 17 mm diameter. These results show that simply using a source that is not the same diameter as the sample would have introduced a systematic error that may be as large as 20%.

The right-hand column of Table 5 shows the measured solid angle for the 24.1 mm sources. This calculation uses Equation A2, where the backscatter fraction is that given in Table A1. These values compare well with the value of 24.0%, calculated according to Equation 2.

Table 5: The measured efficiencies and relative solid angle (RSA) for each of the alpha-spectrometry chambers used in the Radioanalytical Services laboratory at ARPANSA.

The count time for all measurements was 1800 s. The diameter of the sample is listed in column header. The relative solid angle is that for a 24.1 mm diameter source.						
Chamber	Source ID	U-238 Peak Area	Measured Efficiency			Measured RSA
			Ø=24.1mm	Ø=20mm	Ø=17mm	
A1A	278	965	31.3%	34.8%	36.7%	23.9%
A1B	655	1006	30.4%	33.8%	35.7%	23.2%
A2A	656	968	31.0%	34.5%	37.5%	23.7%
A2B	657	968	31.0%	34.5%	37.5%	23.7%
A3A	278	947	30.7%	34.1%	36.0%	23.4%
A3B	655	962	29.1%	32.3%	34.1%	22.2%
A4A	656	1015	32.5%	36.2%	39.3%	24.8%
A4B	657	947	27.7%	30.9%	32.6%	21.2%
A5A	278	985	31.9%	35.5%	37.5%	24.4%
A5B	655	947	28.6%	31.8%	33.6%	21.8%
A6A	656	906	29.0%	32.3%	35.1%	22.1%
A6B	657	958	28.1%	31.2%	33.0%	21.4%
B1A	278	1005	32.6%	36.2%	38.3%	24.9%
B1B	655	1011	30.5%	34.0%	35.9%	23.3%
B2A	656	900	28.8%	32.1%	34.9%	22.0%
B2B	657	997	29.2%	32.5%	34.3%	22.3%
B3A	278	1008	32.7%	36.3%	38.4%	24.9%
B3B	655	927	28.0%	31.2%	32.9%	21.4%
B4A	656	835	26.7%	29.7%	32.4%	20.4%
B4B	657	898	26.3%	29.3%	30.9%	20.1%
B5A	278	991	32.1%	35.7%	37.7%	24.5%
B5B	655	910	27.5%	30.6%	32.3%	21.0%
B6A	656	890	28.5%	31.7%	34.5%	21.8%
B6B	657	1002	29.4%	32.7%	34.5%	22.4%
C1A	278	985	31.9%	35.5%	37.5%	24.4%
C1B	655	1006	30.4%	33.8%	35.7%	23.2%
C2A	656	849	27.2%	30.2%	32.9%	20.8%
C2B	657	955	28.0%	31.1%	32.9%	21.4%
C3A	278	969	31.4%	34.9%	36.9%	24.0%
C3B	655	1062	32.1%	35.7%	37.7%	24.5%
C4A	656	945	30.3%	33.7%	36.6%	23.1%
C4B	657	976	28.6%	31.8%	33.6%	21.8%
C5A	278	999	32.4%	36.0%	38.0%	24.7%
C5B	655	1058	32.0%	35.6%	37.6%	24.4%
C6A	656	999	32.0%	35.6%	38.7%	24.4%
C6B	657	1030	30.2%	33.6%	35.5%	23.0%
Average			**29.9%**	**33.3%**	**35.4%**	**22.8%**

4. EXPERIMENTAL VALIDATION

As stated in the introduction, the measurement efficiency is used to determine the chemical recovery, *C*, for the measured sample. This is done by comparing the activity of the tracer radionuclide, as calculated from the count rate in the peak due to that radionuclide, R_T, (via a re-arrangement of Equation 3), with the known activity added to the sample prior to radiochemical treatment, A_T:

Equation 7: $$C = \frac{R_T}{\varepsilon \cdot A_T}$$

The chemical recovery would be expected to be 100% for those cases where no losses are expected in the from radiochemical procedures. This is the case for the analysis of uranium in water. Several hundred such samples have been analysed in the RAS laboratory. The chemical recovery, calculated according to Equation 7, for such samples are shown in Figure 2.

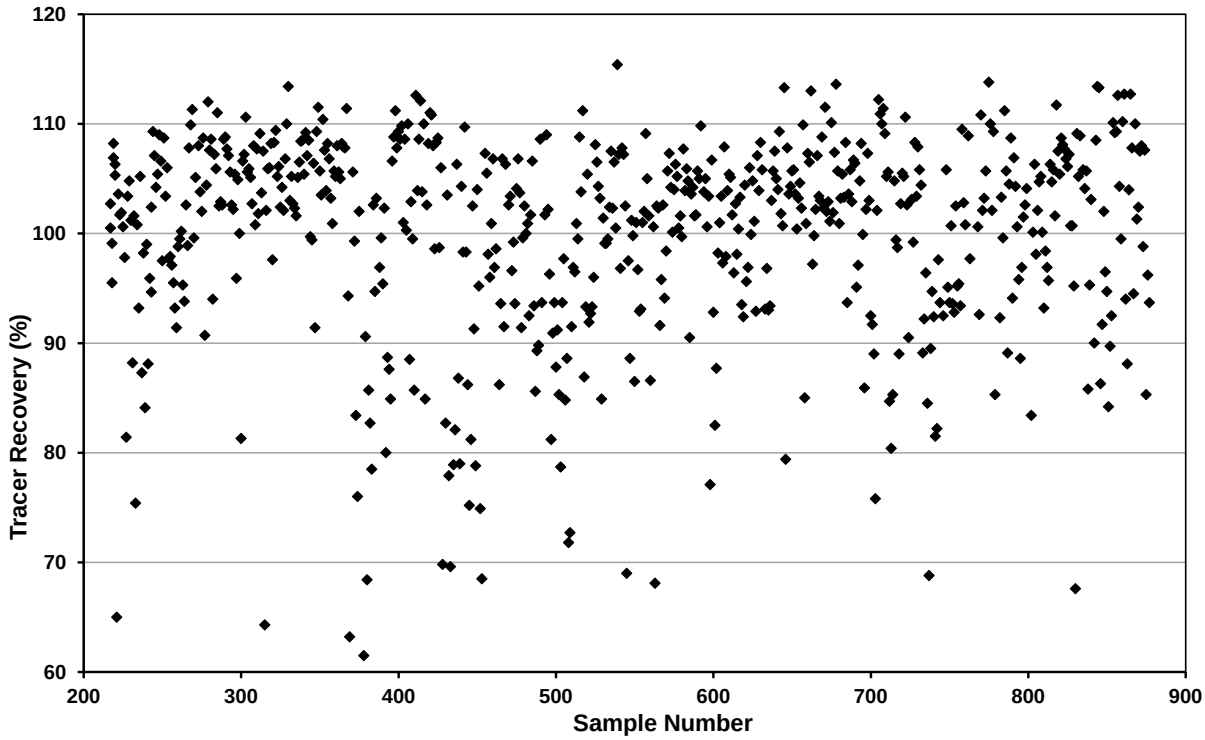

Figure 2: The chemical recovery of calculated for water samples analysed for uranium content.

Figure 2 shows that, in general, the chemical recovery for these analyses is about 100%. For the 610 samples with a recovery greater than 80%, the average chemical recovery was 101%. If only the 550 samples with recoveries greater than 90% are considered, the average recovery is 103%.

If the efficiency had been measured using the certified activities of the calibration sources, the efficiency values, as shown in Table 5, would have been 10% smaller and, therefore, the chemical recovery would have averaged 10% larger, i.e. 110%.

That the chemical recovery averages that expected, indicates that procedure used to modify the activities of the calibration sources produces measured efficiencies that accurately represent the true measurement efficiency of the systems.

5. CONCLUSION

The limited calibration procedures within the alpha-spectrometry software used in the RAS laboratory would normally require that the calibration source be the same diameter as the samples. A procedure was developed that enabled standard diameter calibration sources to be used to determine the measurement efficiency of non-standard diameter samples.

The procedure uses Ruby's formula, (Ruby & Rechen, 1958) and (Ruby, 1994), to produce 'correction factors' for the activity of the source such that the ratio of the observed count rate to the modified activity produces the measurement efficiency of source with the same diameter as the sample.

The proposed procedure was used to calibrate the measurement equipment used in the RAS laboratory. The results of the calibration were used to determine the chemical recovery of analyses expected to have no elemental losses. That the average chemical recovery is close to 100% indicates that the procedure is fit for purpose.

ANNEX A: THE EFFECT OF BACKSCATTERING ON EFFICIENCY MEASUREMENTS

An alpha-particle source is usually constructed of a thin layer of alpha-emitting radionuclide deposited on a metallic substrate. Intuitively, one would expect that the emission rate of alpha-particles from the front surface to be simply half of the decay rate of the radionuclide. That is, one expects that half of the alpha-particles would be emitted forward of the substrate and half emitted towards the substrate. However, the alpha-particles emitted towards the substrate are not simply absorbed. A significant fraction of these particles undergo elastic collisions with the atoms in the substrate and are reflected backwards. These reflected particles are not absorbed in the very thin layer of radioactive material and, so, enter the space forward of the substrate. Therefore, the actual emission rate into the space forward of the source, *E'*, will be greater than half the decay rate of radionuclide, *A*:

Equation A1: $$E' = \frac{A \cdot (1 + K_{BS})}{2}$$

where K_{BS} is the fraction of alpha particles elastically backscattered forward of the substrate. It should be noted that Equation A1 assumes that the emission probability of the alpha-particle of interest is 100%.

The backscattered alpha-particles may enter the detector. So, if a radioactive source is used to measure the relative solid angle subtended by the detector at the source, one must take into account the back-scattered particles:

Equation A2: $$G = \frac{R}{2 \cdot E'} = \frac{R}{A \cdot (1 + K_{BS})}$$

It should be noted that the determination of the source-detector separation, as detailed in section 3.1, used a source with a certified emission rate, rather than a certified activity. So the first part of Equation A2 was used and the determination of the separation was not affected by backscatter.

Equation 3 does not explicitly incorporate a correction for backscattering. This is because Equation 3 details the relationship between the activity of the source and the observed count rate, including the counts due to backscattered alpha-particles. This means that the measured efficiency will, in general, be greater than the relative solid angle subtended by source at the detector.

The relationship shown in Equation 4 can be more explicitly cast as:

Equation A3: $$G_1 \cdot E'_1 = G_2 \cdot E'_2$$

Substituting the relationship of Equation A1 into Equation A3 gives:

Equation A4: $$G_1 \cdot A_1 \cdot \left(1 + K_{BS_1}\right) = G_2 \cdot A_2 \cdot \left(1 + K_{BS_2}\right)$$

In the case that sources 1 and 2 are of different diameters but the same activity, then Equation A4 can be re-arranged to give the fraction of alpha particles elastically backscattered forward of the substrate for source 2 in terms of the relative solid angles and the backscatter fraction for source 1:

Equation A5: $$\left(1 + K_{BS_2}\right) = \frac{G_1}{G_2}\left(1 + K_{BS_1}\right)$$

The certificate for the source used to determine the source-detector separation details not only the emission rate forward of the substrate but also an estimate of the fraction of alpha particles elastically backscattered forward of the substrate. The certificate states that '*the ratio of the disintegration rate to the rate of emergence of alpha-particles was estimated to be 1.96*'. Substituting this value into Equation A1, the fraction of alpha particles elastically backscattered forward of the substrate, K_{BS}, for this 7 mm diameter source is estimated to be 2%.

Using the value of K_{BS} for the 7mm source and the calculated relative solid angles for sources of various diameters, the fraction of alpha particles elastically backscattered forward of the substrate, K_{BS}, for each source may be estimated via Equation A5. These estimates are shown in Table A1.

Table A1: Calculated relative solid angle and backscatter fraction for sources of various diameters placed 4.8 mm from a 23.9 mm diameter detector.

Note that the backscatter fraction listed for the 7mm diameter source is that estimated by the manufacturer.		
Source Diameter (mm)	**Relative Solid Angle**	**Backscatter Fraction (K_{BS})**
24.1	0.240	31%
20.0	0.267	18%
17.0	0.282	12%
7.0	0.309	2%

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