NPL Nuclear Industry Proficiency Test Exercise 2012

JULIAN DEAN

September 2013
NPL Nuclear Industry Proficiency Test Exercise 2012

Julian Dean
Acoustics and Ionising Radiation Division

ABSTRACT

A fourth Nuclear Industry Proficiency Test Exercise has been run by NPL. One sample was prepared, consisting of a nominal 200 L drum loaded with steel and Nylon sheets and plastic bottles containing coarse aggregate (with one containing ion exchange resin). The steel sheets and the resin bottle were spiked with known weights of standard solutions of $^{241}\text{Am}$, $^{60}\text{Co}$ and $^{137}\text{Cs}$. The activity concentrations (averaged across the drum contents) were approximately 2.8 Bq g$^{-1}$, 1.0 Bq g$^{-1}$ and 1.0 Bq g$^{-1}$ respectively.

The participants were required to report the activity concentrations of the individual radionuclides. Information disclosed to the participants initially included the radionuclides present, an activity concentration range, details of the empty drum (e.g. its mass and dimensions) and the percentage of each material type present. The drum was measured by 11 UK and 3 overseas laboratories. After the initial results deadline, further details of the distribution of materials and activity within the drum were disclosed and the participants were invited to submit additional data. It was pointed out that, during the transport process, the bottles in the drum had moved and it was concluded that the ‘active’ bottle could have been positioned anywhere in the base of the drum when measured by the participants (although probably within 25 cm of the base).

Approximately 44% of the ‘initial’ results were statistically ‘in agreement’ with the NPL value. This was partly due to the large proportion of questionable and discrepant data submitted for $^{241}\text{Am}$. As in earlier exercises, some of the results derived using Segmented Gamma Scanners were either low, questionable or discrepant. Four participants submitted additional results after NPL had disclosed the drum’s internal matrix structure; however, the overall proportion of results ‘in agreement’ with NPL was only 27% (compared with 53% for the same four participants ‘pre-disclosure’).
CONTENTS

1 INTRODUCTION ............................................................................................................................................. 1
2 PREPARATION OF STANDARD DRUM ........................................................................................................... 1
3 CIRCULATION OF DRUM AND REPORTING OF DATA .................................................................................. 2
4 RESULTS AND DATA ANALYSIS .................................................................................................................... 2
5 DISCUSSION .................................................................................................................................................... 4
6 ACKNOWLEDGEMENTS ................................................................................................................................. 4
7 REFERENCES .................................................................................................................................................... 4
8 TABLES ............................................................................................................................................................ 5
9 FIGURES .......................................................................................................................................................... 11
10 APPENDIX A – INITIAL MAILSHOT ............................................................................................................ 21
11 APPENDIX B – REPORTING FORM ............................................................................................................... 24
12 APPENDIX C – LIST OF PARTICIPANTS ...................................................................................................... 25
1 INTRODUCTION

The accurate measurement of radioactivity in potentially active materials produced in nuclear site decommissioning is essential for correct waste categorisation. This is important for public safety, to reduce the industry’s costs and to minimise LLW volumes. The National Physical Laboratory (NPL) runs Nuclear Industry Proficiency Test Exercises (‘drum comparisons’) [Dean, 2007, 2010, 2012] to enable laboratories involved in the clearance and sentencing of bulk γ-emitting waste to test their measurement procedures. The exercises provide a check on the modelling techniques used for calculating detection efficiencies and enable participants to demonstrate measurement capability to third parties.

This report describes the fourth PTE in this series, covering:

- the preparation of the ‘standard drum’;
- the circulation of the drum and reporting of data;
- the reported results and data analysis.

2 PREPARATION OF STANDARD DRUM

The following materials were procured / prepared:

- Standardised radionuclide solutions of $^{241}$Am, $^{60}$Co and $^{137}$Cs
- 1 x 500 ml HDPE bottle containing ion-exchange resin;
- 29 x 500 ml HDPE bottles containing coarse aggregate;
- 3 x filter papers (80 mm x 80 mm);
- 1 x 200 L steel drum;
- Approximately 10 kg of Nylon 6-6 (in sheets);
- Approximately 4 kg of 0.9 mm BS 1449 stainless steel sheet;

The activity concentrations of the radionuclide solutions had been previously determined by assay in glass ampoules in a re-entrant secondary standard ionisation chamber. Each ampoule had been measured using a γ-spectrometer to check for the presence of any γ-emitting impurities.

The remaining activity was contained within the single bottle of ion-exchange resin which had been previously standardised and which contained all three radionuclides. A ‘drum insert’ was prepared (see Figure 1). The base and two opposite sides consisted of Nylon 6-6 and the other two sides consisted of stainless steel. This formed a box-like structure with the top of the ‘box’ open. Weighed amounts of the standardised solutions were drop-deposited onto the filter papers, and after drying the filters were taped to the outward-facing walls of the steel plates, as illustrated. Each filter was spiked with a different nuclide. The bottle of resin was spiked with a mixture of all three radionuclides and was positioned in a bottom corner of the box. The bottles of coarse aggregate were then placed in a single layer at the bottom of the box, adjacent to the active bottle.

The drum was certificated for the activity concentrations of each radionuclide present (see Table 1) and was designated source X12152.
3 CIRCULATION OF DRUM AND REPORTING OF DATA

An initial mailshot was prepared (see Appendix A) and circulated to potential participants.

A Reporting Form (Appendix B) was sent to the participants for reporting their data. Also, the participants were provided with the following details:

- The activity concentration range (1 - 10 Bq/g);
- Details of the empty drum from the manufacturer;
- The mass of the drum empty (18.3 kg nominal) and full (54.9 kg nominal);
- The percentage by mass of the drum’s contents:
  - Coarse aggregate (56.1 % nominal)
  - Plastics (31.4 % nominal)
  - Metal (11.5 % nominal)
  - Ion-exchange resin (1.0 % nominal).

After the ‘first deadline’ (see Appendix A), participants were given details of the drum’s ‘internal structure’ and locations of the activity, plus more details of the individual materials, which were:

- Coarse aggregate: 20.54 kg nominal
- Nylon: 9.78 kg nominal
- Steel: 4.19 kg nominal
- HDPE: 1.71 kg nominal
- Resin: 0.32 kg nominal

Unfortunately, the bottles of coarse aggregate and resin were found to have moved in transit and it was not clear at which stage of the circulation of the drum this had occurred. The active bottle could have been positioned anywhere in the base of the drum when measured by the participants (although probably within 25 cm of the base of the ‘internal box’). However, the box structure and the filter papers had not moved in transit. Note also that the activity in the bottle was small compared with the total activity on the filter papers (< 10%).

Participants were invited to submit additional results based on the above information; four did so.

A list of participants is given in Appendix C.

4 RESULTS AND DATA ANALYSIS

To preserve anonymity, each participant was assigned a number, and their results were coded accordingly. Some of the laboratories had also participated in previous exercises and their numbers 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:

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

[1]
where:

\[ D = \text{deviation from the NPL value (\%) } \]
\[ L = \text{the participant’s value (Bq g}^{-1}) \]
\[ N = \text{the NPL value (Bq g}^{-1}) \]

The deviations are plotted in Figures 2 – 4 (results submitted prior to the first deadline, designated ‘pre-disclosure’) and Figures 8 – 10 (results submitted after first deadline, designated ‘post-disclosure’). The error bars in the graphs represent the standard uncertainty \((k=1)\) of the deviation:

\[ u_D = 100 \frac{L}{N} \sqrt{\frac{u_L^2}{L^2} + \frac{u_N^2}{N^2}} \] \[ [2] \]

where:

\[ u_D = \text{standard uncertainty of the deviation (\%)} \]
\[ u_L = \text{standard uncertainty of the participant’s value (Bq g}^{-1}) \]
\[ u_N = \text{standard uncertainty of the assigned value (Bq g}^{-1}) \]

The results were evaluated using three tests:

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

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

\[ z = \frac{L - N}{\sigma_p} = \frac{L - N}{0.05823 \times N} \] \[ [5] \]

where:

\[ \zeta = \text{zeta score} \]
\[ R_L = \text{relative uncertainty of the participant’s value} \]
\[ Z = \text{z-score} \]
\[ \sigma_p = \text{standard deviation for proficiency assessment} \]

The standard deviation for proficiency assessment represents the level of performance that NPL would wish laboratories to be able to achieve. It corresponds to a deviation ‘\(D\)’ of 15\% at the 99\% confidence level.

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 results are plotted in Figures 5 - 7. Note that no outlier test was carried out on the ‘post-disclosure’ data sets owing to the small numbers of results in each set – in these cases, all results are regarded as having passed the outlier test. The zeta scores, z-scores and outlier results are also given in Tables 2 - 7.
Results for which the absolute values of the zeta score and the z-score were both ≤ 2.576 (corresponding to a significance levels of α = 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 \( R_L \).

5 DISCUSSION

Overall, 44% of the results submitted pre-disclosure were in agreement with the NPL Assigned Values. If the proportion in agreement with NPL is broken down by nuclide, the percentages are: \(^{241}\text{Am} \) 14%, \(^{60}\text{Co} \) 48% and \(^{137}\text{Cs} \) 71%. Apart from one result, all the \(^{241}\text{Am} \) data were higher than NPL (with positive deviations between 6 % and 196 %), and it is not clear why.

It is interesting that many of the discrepant data for \(^{60}\text{Co} \) and \(^{137}\text{Cs} \) were from measurements using either Segmented Gamma Scanners (SGSs) or collimated detectors. The lowest results reported for \(^{60}\text{Co} \) (although mostly in agreement with NPL) were from SGS measurements and this is similar to the findings of previous exercises (Dean 2007, 2010, 2012).

Four participants submitted results post-disclosure. The overall proportion of results in agreement with NPL was only 27% (compared with 53% pre-disclosure for the same group of participants).

6 ACKNOWLEDGEMENTS

The author wishes to thank the participating organisations for the time and effort they have put into analysing the drum, and for the information provided. Also, thanks go to Daniel Ainsworth for organising the transport of the drum and Peter Ivanov for reviewing this report. Finally, the author gratefully acknowledges the financial support of the National Measurement System.

7 REFERENCES


8 TABLES

Table 1 - Principal radionuclides in X12152

<table>
<thead>
<tr>
<th>Radionuclide</th>
<th>Decay mode</th>
<th>Activity concentration (averaged across contents of drum) @ 2012-07-01 1200 UTC* (Bq g⁻¹)</th>
<th>Standard uncertainty (k=1) (Bq g⁻¹)</th>
<th>Standard uncertainty (k=1) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>²⁴¹Am</td>
<td>α/γ</td>
<td>2.762</td>
<td>0.010</td>
<td>0.4</td>
</tr>
<tr>
<td>⁶⁰Co</td>
<td>β/β</td>
<td>0.9649</td>
<td>0.0020</td>
<td>0.21</td>
</tr>
<tr>
<td>¹³⁷Cs</td>
<td>β, β/γ</td>
<td>1.022</td>
<td>0.007</td>
<td>0.6</td>
</tr>
</tbody>
</table>

*Universal Time, Co-ordinated. This replaced Greenwich Mean Time in 1972. Format of date is YYYY-MM-DD.
Table 2 - Reported results for $^{241}$Am (‘pre-disclosure’)

NPL activity concentration = $(2.762 \pm 0.010)$ Bq g$^{-1}$ ($k=1$)  
Reference time: 2011-03-01 1200 UTC

<table>
<thead>
<tr>
<th>Participant code</th>
<th>Reported activity concentration (Bq/g)</th>
<th>Reported uncertainty (%)</th>
<th>Zeta score</th>
<th>Z score</th>
<th>D (%)</th>
<th>Outlier test result</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1A</td>
<td>7.69</td>
<td>100</td>
<td>0.64</td>
<td>30.64</td>
<td>178.4</td>
<td>Outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>1B</td>
<td>3.35</td>
<td>12</td>
<td>1.46</td>
<td>3.66</td>
<td>21.29</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>2</td>
<td>3.14</td>
<td>11</td>
<td>1.09</td>
<td>2.35</td>
<td>13.69</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>3</td>
<td>3.94</td>
<td>18.9</td>
<td>1.58</td>
<td>7.32</td>
<td>42.65</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>4</td>
<td>3.8</td>
<td>19</td>
<td>1.44</td>
<td>6.45</td>
<td>37.58</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>5</td>
<td>3.36</td>
<td>50</td>
<td>0.36</td>
<td>3.72</td>
<td>21.65</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>6A</td>
<td>0.38</td>
<td>15.8</td>
<td>-39.13</td>
<td>-14.81</td>
<td>-86.24</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>6B</td>
<td>5.58</td>
<td>95.93</td>
<td>0.53</td>
<td>17.52</td>
<td>102.0</td>
<td>Outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>7</td>
<td>6.32</td>
<td>17.9</td>
<td>3.14</td>
<td>22.12</td>
<td>128.8</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>8A</td>
<td>4.613</td>
<td>20.24</td>
<td>1.98</td>
<td>11.51</td>
<td>67.02</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>8B</td>
<td>5.170</td>
<td>20.21</td>
<td>2.30</td>
<td>14.97</td>
<td>87.18</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>8C</td>
<td>4.675</td>
<td>20.21</td>
<td>2.02</td>
<td>11.89</td>
<td>69.26</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>9</td>
<td>2.93</td>
<td>5.5</td>
<td>1.04</td>
<td>1.04</td>
<td>6.083</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>10A</td>
<td>5.44</td>
<td>25.5</td>
<td>1.93</td>
<td>16.65</td>
<td>96.96</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>10B</td>
<td>5.04</td>
<td>25.4</td>
<td>1.78</td>
<td>14.16</td>
<td>82.48</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>10C</td>
<td>5.03</td>
<td>25.4</td>
<td>1.78</td>
<td>14.10</td>
<td>82.11</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>11A</td>
<td>8.19</td>
<td>10.20</td>
<td>6.50</td>
<td>33.75</td>
<td>196.5</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>11B</td>
<td>7.05</td>
<td>10.41</td>
<td>5.84</td>
<td>26.66</td>
<td>155.3</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>12</td>
<td>4.595</td>
<td>17.82</td>
<td>2.24</td>
<td>11.40</td>
<td>66.36</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>13</td>
<td>3.09</td>
<td>33</td>
<td>0.32</td>
<td>2.04</td>
<td>11.88</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>14</td>
<td>6.66</td>
<td>27.55</td>
<td>2.12</td>
<td>24.24</td>
<td>141.1</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
</tbody>
</table>
Table 3 - Reported results for $^{60}$Co (‘pre-disclosure’)

NPL activity concentration = (0.9649 ± 0.0020) Bq g$^{-1}$ (k=1)  
Reference time: 2011-03-01 1200 UTC

<table>
<thead>
<tr>
<th>Participant code</th>
<th>Reported activity concentration (Bq/g)</th>
<th>Reported uncertainty (%)</th>
<th>Zeta score</th>
<th>Z score</th>
<th>D (%)</th>
<th>Outlier test result</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1A</td>
<td>1.27</td>
<td>40</td>
<td>0.60</td>
<td>5.43</td>
<td>31.62</td>
<td>Outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>1B</td>
<td>2.36</td>
<td>12</td>
<td>4.93</td>
<td>24.83</td>
<td>144.6</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>2</td>
<td>1.23</td>
<td>7.3</td>
<td>2.95</td>
<td>4.72</td>
<td>27.47</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>3</td>
<td>1.04</td>
<td>2.7</td>
<td>2.67</td>
<td>1.34</td>
<td>7.783</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>4</td>
<td>1.4</td>
<td>12</td>
<td>2.59</td>
<td>7.74</td>
<td>45.09</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>5</td>
<td>1.07</td>
<td>7</td>
<td>1.40</td>
<td>1.87</td>
<td>10.89</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>6A</td>
<td>2.34</td>
<td>12.8</td>
<td>4.59</td>
<td>24.47</td>
<td>142.5</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>6B</td>
<td>1.08</td>
<td>14.79</td>
<td>0.72</td>
<td>2.05</td>
<td>11.93</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>7</td>
<td>0.878</td>
<td>13.5</td>
<td>-0.73</td>
<td>-1.55</td>
<td>-9.006</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>8A</td>
<td>1.101</td>
<td>14.44</td>
<td>0.86</td>
<td>2.42</td>
<td>14.11</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>8B</td>
<td>1.069</td>
<td>14.40</td>
<td>0.68</td>
<td>1.85</td>
<td>10.79</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>8C</td>
<td>1.049</td>
<td>14.39</td>
<td>0.56</td>
<td>1.50</td>
<td>8.716</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>9</td>
<td>1.040</td>
<td>4.0</td>
<td>1.80</td>
<td>1.34</td>
<td>7.783</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>10A</td>
<td>1.47</td>
<td>11.7</td>
<td>2.94</td>
<td>8.99</td>
<td>52.35</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>10B</td>
<td>1.38</td>
<td>11.6</td>
<td>2.59</td>
<td>7.39</td>
<td>43.02</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>10C</td>
<td>1.39</td>
<td>11.5</td>
<td>2.66</td>
<td>7.57</td>
<td>44.06</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>11A</td>
<td>0.92</td>
<td>10.03</td>
<td>-0.49</td>
<td>-0.80</td>
<td>-4.653</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>11B</td>
<td>1.39</td>
<td>11.70</td>
<td>2.61</td>
<td>7.57</td>
<td>44.06</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>12</td>
<td>1.066</td>
<td>14.72</td>
<td>0.64</td>
<td>1.80</td>
<td>10.48</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>13</td>
<td>1.08</td>
<td>8</td>
<td>1.33</td>
<td>2.05</td>
<td>11.93</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>14</td>
<td>1.48</td>
<td>27.35</td>
<td>1.27</td>
<td>9.17</td>
<td>53.38</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
</tbody>
</table>
Table 4 - Reported results for $^{137}\text{Cs}$ (‘pre-disclosure’)

NPL activity concentration = (1.022 ± 0.007) Bq g$^{-1}$ ($k=1$)  
Reference time: 2011-03-01 1200 UTC

<table>
<thead>
<tr>
<th>Participant code</th>
<th>Reported activity concentration (Bq/g)</th>
<th>Reported uncertainty (%)</th>
<th>Zeta score</th>
<th>Z score</th>
<th>D (%)</th>
<th>Outlier test result</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>1A</td>
<td>0.965</td>
<td>30</td>
<td>-0.20</td>
<td>-0.96</td>
<td>-5.577</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>1B</td>
<td>2.224</td>
<td>12</td>
<td>4.50</td>
<td>20.20</td>
<td>117.6</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>2</td>
<td>1.02</td>
<td>7.8</td>
<td>-0.03</td>
<td>-0.03</td>
<td>-0.1957</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>3</td>
<td>1.45</td>
<td>6.2</td>
<td>4.75</td>
<td>7.19</td>
<td>41.88</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>4</td>
<td>1.1</td>
<td>12</td>
<td>0.59</td>
<td>1.31</td>
<td>7.632</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>5</td>
<td>1.11</td>
<td>7</td>
<td>1.13</td>
<td>1.48</td>
<td>8.611</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>6A</td>
<td>3.25</td>
<td>12.7</td>
<td>5.40</td>
<td>37.44</td>
<td>218.0</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>6B</td>
<td>1.12</td>
<td>17.07</td>
<td>0.51</td>
<td>1.65</td>
<td>9.589</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>7</td>
<td>2.62</td>
<td>23.5</td>
<td>2.60</td>
<td>26.85</td>
<td>156.4</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>8A</td>
<td>0.933</td>
<td>20.36</td>
<td>-0.47</td>
<td>-1.50</td>
<td>-8.708</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>8B</td>
<td>0.976</td>
<td>20.28</td>
<td>-0.23</td>
<td>-0.77</td>
<td>-4.501</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>8C</td>
<td>0.925</td>
<td>20.32</td>
<td>-0.52</td>
<td>-1.63</td>
<td>-9.491</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>9</td>
<td>0.907</td>
<td>4.1</td>
<td>-0.31</td>
<td>-1.93</td>
<td>-11.25</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>10A</td>
<td>1.08</td>
<td>14.3</td>
<td>0.38</td>
<td>0.97</td>
<td>5.675</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>10B</td>
<td>1.06</td>
<td>14.1</td>
<td>0.25</td>
<td>0.64</td>
<td>3.718</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>10C</td>
<td>1.01</td>
<td>14.2</td>
<td>-0.08</td>
<td>-0.20</td>
<td>-1.174</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>11A</td>
<td>1.96</td>
<td>11.09</td>
<td>4.31</td>
<td>15.76</td>
<td>91.78</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>11B</td>
<td>1.16</td>
<td>14.20</td>
<td>0.84</td>
<td>2.32</td>
<td>13.50</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>12</td>
<td>0.9195</td>
<td>14.84</td>
<td>-0.75</td>
<td>-1.72</td>
<td>-10.03</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>13</td>
<td>1.13</td>
<td>15</td>
<td>0.64</td>
<td>1.81</td>
<td>10.57</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>14</td>
<td>1.33</td>
<td>27.36</td>
<td>0.85</td>
<td>5.18</td>
<td>30.14</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
</tbody>
</table>
Table 5 - Reported results for $^{241}$Am (‘post-disclosure’)

NPL activity concentration = (2.762 ± 0.010) Bq g$^{-1}$ ($k=1$)  
Reference time: 2011-03-01 1200 UTC

<table>
<thead>
<tr>
<th>Participant code</th>
<th>Reported activity concentration (Bq/g)</th>
<th>Reported uncertainty (%)</th>
<th>Zeta score</th>
<th>Z score</th>
<th>D (%)</th>
<th>Outlier test result</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>1.9</td>
<td>15</td>
<td>-3.02</td>
<td>-5.36</td>
<td>-31.21</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>6B</td>
<td>3.60</td>
<td>48.7</td>
<td>0.48</td>
<td>5.21</td>
<td>30.34</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>11A</td>
<td>10</td>
<td>10.20</td>
<td>7.10</td>
<td>45.00</td>
<td>262.06</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>11B</td>
<td>9.70</td>
<td>10.41</td>
<td>6.87</td>
<td>43.14</td>
<td>251.19</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>13</td>
<td>3.22</td>
<td>27</td>
<td>0.53</td>
<td>2.85</td>
<td>16.58</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
</tbody>
</table>

Table 6 - Reported results for $^{60}$Co (‘post-disclosure’)

NPL activity concentration = (0.9649 ± 0.0020) Bq g$^{-1}$ ($k=1$)  
Reference time: 2011-03-01 1200 UTC

<table>
<thead>
<tr>
<th>Participant code</th>
<th>Reported activity concentration (Bq/g)</th>
<th>Reported uncertainty (%)</th>
<th>Zeta score</th>
<th>Z score</th>
<th>D (%)</th>
<th>Outlier test result</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>1.1</td>
<td>11</td>
<td>1.12</td>
<td>2.40</td>
<td>14.00</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>6B</td>
<td>0.718</td>
<td>16.5</td>
<td>2.08</td>
<td>-4.39</td>
<td>-25.59</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>11A</td>
<td>0.92</td>
<td>10.03</td>
<td>-0.49</td>
<td>-0.80</td>
<td>-4.653</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>11B</td>
<td>1.41</td>
<td>11.70</td>
<td>2.70</td>
<td>7.92</td>
<td>46.13</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>13</td>
<td>1.09</td>
<td>11</td>
<td>1.04</td>
<td>2.23</td>
<td>12.97</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
</tbody>
</table>
Table 7 - Reported results for $^{137}$Cs (‘post-disclosure’)

NPL activity concentration = $(1.022 \pm 0.007) \text{ Bq g}^{-1} (k=1)$  
Reference time: 2011-03-01 1200 UTC

<table>
<thead>
<tr>
<th>Participant code</th>
<th>Reported activity concentration (Bq/g)</th>
<th>Reported uncertainty (%)</th>
<th>Zeta score</th>
<th>Z score</th>
<th>D (%)</th>
<th>Outlier test result</th>
<th>Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>0.83</td>
<td>11</td>
<td>-2.10</td>
<td>-3.23</td>
<td>-18.79</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
<tr>
<td>6B</td>
<td>2.23</td>
<td>17.6</td>
<td>3.08</td>
<td>20.30</td>
<td>118.2</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>11A</td>
<td>1.96</td>
<td>11.09</td>
<td>4.31</td>
<td>15.76</td>
<td>91.78</td>
<td>Not outlier</td>
<td>Discrepant</td>
</tr>
<tr>
<td>11B</td>
<td>1.15</td>
<td>14.20</td>
<td>0.78</td>
<td>2.15</td>
<td>12.52</td>
<td>Not outlier</td>
<td>In agreement</td>
</tr>
<tr>
<td>13</td>
<td>1.77</td>
<td>21</td>
<td>2.01</td>
<td>12.57</td>
<td>73.19</td>
<td>Not outlier</td>
<td>Questionable</td>
</tr>
</tbody>
</table>
Figure 1 - Schematic diagram of ‘drum insert’

NPL Nuclear Industry PTE 2012
Dimensions of insert for standard drum X12152

Left wall:
Nylon 10 mm thick

Base:
Nylon 10 mm thick

Front wall, back wall:
Both steel 0.9 mm thick

Right wall:
Nylon 10 mm thick

* $^{137}$Cs filter on back wall, on rear face
** $^{241}$Am and $^{60}$Co filters on front wall, on front face
Figure 2 - Deviation plot for $^{241}\text{Am}$ results, pre-disclosure
(6 results above area of plot)
Figure 3 - Deviation plot for $^{60}$Co results, pre-disclosure
(2 results above area of plot)
Figure 4 - Deviation plot for $^{137}$Cs results, pre-disclosure
(3 results above area of plot)
Figure 5 - Relative uncertainty plot for $^{241}$Am results, pre-disclosure
Figure 6 - Relative uncertainty plot for $^{60}\text{Co}$ results, pre-disclosure
Figure 7 - Relative uncertainty plot for $^{137}$Cs results, pre-disclosure
Figure 8 - Deviation plot for $^{241}$Am results, post-disclosure
(2 results above area of plot)
Figure 9 - Deviation plot for $^{60}$Co results, post-disclosure
Figure 10 - Deviation plot for $^{137}$Cs results, post-disclosure
(1 result above area of plot)
10  APPENDIX A – Initial Mailshot

Dear Colleague,

**FOURTH NPL NUCLEAR INDUSTRY PROFICIENCY TEST EXERCISE**

NPL is about to run its fourth bulk-waste proficiency test exercise (‘drum comparison’) to enable UK laboratories involved in decommissioning and site clearance to test their bulk-waste gamma measurement procedures. I am writing to invite you to participate in this exercise. As for previous exercises, the purpose is to provide the user community with a voluntary, independent and confidential test of their bulk waste measurement procedures.

**In brief:** NPL will prepare a single ‘mock waste’ sample in a 200-litre steel drum. The drum will contain metal, plastics, building material simulant (coarse aggregate) and ion-exchange resin. The radionuclides $^{241}$Am, $^{60}$Co and $^{137}$Cs will be present and the overall activity concentration (activity/total mass of material in drum) will be in the range 1 – 10 Bq g $^{-1}$. The activity will be distributed in a heterogeneous manner. The drum will be available for measurement between early August 2012 and mid-February 2013 and participants will be asked to report the activity concentrations of the individual radionuclides. A short exercise report will be published in March 2013. All results will be coded and treated as confidential.

n.b. Please note that, due to limited available funding, there will be no post-exercise meeting.

If you are interested in participating, please read the full details given on pages 2 to 3 below and then complete and return the PTE Enquiry Form. Please note that requests to participate must be received by close of business on 3 July 2012.

We hope you are interested and that you will be able to participate.

Yours faithfully

Julian Dean

June 2012
A) Plan for Fourth NPL Nuclear Industry PTE

NPL will prepare a single ‘mock waste’ sample in a 200-litre steel drum. The drum will contain a mixture of metal, plastics, building material simulant (coarse aggregate) and ion-exchange resin. The radionuclides $^{241}\text{Am}$, $^{60}\text{Co}$ and $^{137}\text{Cs}$ will be present and the overall activity concentration (activity/total mass of material in drum) will be in the range $1 - 10$ Bg g$^{-1}$. The activity will be distributed in a heterogeneous manner within the drum.

The drum will be delivered to (and collected from) the participants on agreed dates. Participants will be given 2 working days to measure the drum (excluding arrival and dispatch days). The drum will be an Excepted package.

Note that, prior to measurement by the participants, NPL will disclose only the above information plus:

- the percentage by mass of each material present;
- the dimensions of the drum;
- the mass of the drum (empty and full).

Participants will be asked to report their measurements of the individual radionuclide activity concentrations along with details of calibration procedures and instrumentation used, by a ‘first deadline’ (see Timetable below). NPL will then disclose:

- more details of the materials (e.g. which metal) and the ‘internal structure’ of the drum;
- the location(s) of the activity present.

Participants will then have the opportunity to submit (by a ‘second deadline’) additional results based on this more detailed knowledge of the drum. Note that revisions to earlier results (i.e. those submitted before the first deadline) will not be accepted during this period.

NPL will then prepare a short draft report (including all results, coded for anonymity) and issue to all participants.
B) Timetable

<table>
<thead>
<tr>
<th>Action</th>
<th>Dates</th>
</tr>
</thead>
<tbody>
<tr>
<td>Interested laboratories to reply to this mailshot</td>
<td>By 3 July 2012</td>
</tr>
<tr>
<td>NPL to contact each laboratory, advise if they can be accommodated, and, if so, agree receipt and dispatch dates</td>
<td>By 27 July 2012</td>
</tr>
<tr>
<td>Participants to submit Purchase Orders</td>
<td>Within two weeks of notification of receipt and dispatch dates</td>
</tr>
<tr>
<td>Participants to measure drum</td>
<td>Within agreed dates in the period 6 August 2012 to 15 February 2013</td>
</tr>
<tr>
<td>Participants to submit results (not knowing details of drum contents)</td>
<td>By 1 March 2013 (First deadline)</td>
</tr>
<tr>
<td>NPL to declare further details of drum contents</td>
<td>4 March 2013</td>
</tr>
<tr>
<td>Participants to submit any additional results (knowing details of drum contents)</td>
<td>By 15 March 2013 (Second deadline)</td>
</tr>
<tr>
<td>NPL to issue report</td>
<td>By 29 March 2013</td>
</tr>
</tbody>
</table>

C) How to participate

Please complete the enclosed Enquiry Form and return it by close of business on 3 July 2012 to: radioactivity@npl.co.uk

Please indicate on the form which weeks in the period 6 August 2012 to 15 February 2013 you would prefer for delivery of the sample drum to your site. Note that you will be allocated two full working days to measure the drum (excluding the arrival and dispatch days).

IMPORTANT: If the exercise is oversubscribed we shall deal with requests for inclusion on a first-come-first-served basis, so please PLEASE DO NOT SUBMIT A PURCHASE ORDER AT THIS STAGE. We shall contact you in July 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 on the allocated date.

D) Fees

The fees will be as follows:

- Participation: £1075
- Delivery: To be advised

NPL will arrange for a courier to deliver and pick-up on the agreed dates.
## NUCLEAR INDUSTRY PTE 2012 REPORTING FORM

<table>
<thead>
<tr>
<th>Radionuclide</th>
<th>Activity concentration (Bq g(^{-1}))</th>
<th>Type A uncertainty (% (k=1))</th>
<th>Type B uncertainty (% (k=1))</th>
<th>Combined standard uncertainty (% (k=1))</th>
</tr>
</thead>
<tbody>
<tr>
<td>(^{241})Am</td>
<td>RT 1200 UTC, 01/07/12</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(^{60})Co</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>(^{137})Cs</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Please provide brief details of the following:

<table>
<thead>
<tr>
<th>Detector type</th>
<th>Scanning method used (including, e.g., detector collimation, segmented scanning and rotation of drum)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Matrix density correction applied</td>
</tr>
<tr>
<td></td>
<td>Calibration standards used</td>
</tr>
<tr>
<td></td>
<td>Efficiency modelling and software</td>
</tr>
<tr>
<td></td>
<td>Other</td>
</tr>
</tbody>
</table>

NPL Report IR 30
12 APPENDIX C – List of Participants

Mr N M Baghini
MAHPG Laboratory
Imperial College London
Silwood Park Campus
Buckhurst Road
Ascot
Berkshire SL5 7TE

Dr H Beddow
Nuvia Ltd
B351.28
Fifteenth Street
Harwell Oxford
Didcot
Oxfordshire OX11 9AW

Mr W De Boeck
SCK-CEN
Boeretang 200
2400 Mol
Belgium

Mr J Cairns
Aurora Health Physics Services Ltd
3 The Terrace
Library Avenue
Harwell Oxford
Didcot OX11 0SG

Mr G Faulkner
Babcock International Group – Marine and Technology (Devonport)
Babcock International Group
PC956 N016
Devonport Royal Dockyard
Plymouth
Devon PL1 4SG

Mr T Hatt
ORTEC – Advanced Measurement Technology
Spectrum House
1 Millars Business Centre
Fishponds Close
Wokingham
Berkshire RG41 2TZ

Mr T Huys
Belgoprocess
Gravenstraat 73
2490 Dessel
Belgium

Mr R Major
AMEC
601 Faraday St
Birchwood
Warrington WA3 6GN

Mr A Waterfall
AWE
Aldermaston
Reading
Berkshire RG7 4PR

Mr N J O’Brien
AWE
Aldermaston
Reading
Berkshire RG7 4PR

Dr D Parvin
Babcock International Group
B14.1
Sellafield
Seascale
Cumbria

Mr M Rushby
Canberra UK Ltd
B528.10 Unit 1
Harwell Oxford
Didcot
Oxfordshire OX11 0DF

Mr G Simone
Nucleco S.p.A.
Via Anguillarese 301
Rome
Italy

Mr J Stephenson
D60
RSRL Winfrith
Winfrith Newburgh
Dorset DT2 8WG

Mr G Ward
Sizewell A Site - Magnox Ltd.
Nr Leiston
Suffolk IP16 4UE