

NPL REPORT IR 60

NUCLEAR INDUSTRY
PROFICIENCY TEST EXERCISE 2021

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Nuclear Industry Proficiency Test Exercise 2021

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ABSTRACT

An eighth Nuclear Industry Proficiency Test Exercise has been run by NPL. One sample was prepared, consisting of a mild steel drum (volume 200 L nominal) loaded with 240 plastic bottles (volume 500 mL nominal each) stacked in 5 layers of 48 bottles. Each bottle was filled with inactive vermiculite. Three plastic vials (20 mL volume) each containing ion-exchange resin (ca. 15 g) were inserted into three of the 500 mL bottles. These three bottles were placed centrally in the second layer i.e. the layer one up from the bottom of the drum. Each plastic vial had previously been spiked with known masses of standard solutions of 60 Co, 137 Cs and 241 Am, and the total mass of the drum's contents was known. The activity per unit mass of each radionuclide present was therefore known, being approximately 5.9 Bq g⁻¹, 11.9 Bq g⁻¹ and 18.9 Bq g⁻¹, respectively.

The participants reported their measured activity per unit mass for the individual radionuclides. The participants were told which radionuclides were present and a range for the activity per unit mass of each radionuclide, along with details of the empty drum (e.g. mass and dimensions) and the material type present. After the initial reporting deadline, the location of the activity within the drum was disclosed by NPL and participants were invited to submit additional results before a second reporting deadline.

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Department

Assigned Values (reference time 2021-06-01 12:00 UTC)

Nuclide	Assigned Value (Bq g ⁻¹)
⁶⁰ Co	5.903 ± 0.043
¹³⁷ Cs	11.89 ± 0.18
²⁴¹ Am	18.91 ± 0.15

UNCERTAINTIES

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

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1. INTRODUCTION

The accurate measurement of radioactivity in potentially active waste materials generated in nuclear decommissioning is essential for correct waste categorisation. This is important for public safety, to reduce the industry's costs and to minimise volumes of material being sent to the UK's LLW repository. The National Physical Laboratory (NPL) runs Nuclear Industry Proficiency Test Exercises ('drum comparisons') [Dean, 2007, 2010, 2012, 2013, 2015, 2017, 2019] to enable laboratories involved in the clearance and sentencing of bulk gamma-emitting waste to test their measurement procedures. The exercises provide a check on the techniques used for calculating detection efficiencies and enable participants to demonstrate measurement capability to third parties.

This report describes the eighth 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 drum from the previous exercise (2019) was repurposed for the 2021 exercise. The active vials were removed from the top layer of 48×500 mL HDPE bottles. These vials were disposed and the remaining inactive bottles were removed from the drum and placed aside for reuse.

Standard solutions of ⁶⁰Co, ¹³⁷Cs and ²⁴¹Am were identified for use. These individual radionuclide solutions were standardised via second standard ionisation chamber measurements. Ion-exchange resin (15 g nominal) was dispensed to three empty 20 mL plastic liquid scintillation vials. The resin in each of the three plastic vials was spiked with the standardised solutions of ⁶⁰Co, ¹³⁷Cs and ²⁴¹Am using a pycnometer and the masses of solution dispensed was recorded. The target dispensed activity for each of the vials was equal but the activity dispensed to each vial did vary due to differences in the masses dispensed. Each of the three spiked vials were then sealed and packed into one of the original 500 mL HDPE bottles with inactive vermiculite. In each case, the vial was positioned in the neck of the 500 mL bottle, close to the lid.

The empty mass of the drum was recorded and then the 240 × 500 mL HDPE bottles including the three containing the active vials were placed back in the drum. The 240 bottles formed five layers. The three bottles containing the activity were placed centrally in the second layer, i.e. the layer one up from the bottom of the drum. The lid was placed back on the drum and the mass of the full drum was recorded. The activity per unit mass of each radionuclide present was calculated.

3. CIRCULATION OF DRUM AND REPORTING OF DATA

To gauge the interest in another exercise, a letter was sent out to past participants to register their interest (Appendix B). A follow-up invitation letter (Appendix C) was then sent out to those who registered their interest accompanied by an enquiry form.

On receipt of enquiry forms from interested laboratories, NPL then agreed receipt and dispatch dates with all participants in advance. The timetable was later amended by two weeks to include an additional participant.

During the measurement period of the drum, the following were provided to each participant:

- Reporting Form (Appendix D);
- Techniques Form (Appendix E);
- Information Sheet, including confirmation of the revised timetable for the exercise (Appendix F).

The information provided included:

- The range of activities per unit mass for each radionuclide (3 − 30 Bq g⁻¹);
- The mass of the drum empty (19.55 kg nominal);
- The mass of the drum's contents (29.166 kg nominal);

The participants were required to report their measurement results by the 'first deadline', after which NPL disclosed the location of the activity within the drum and invited the participants to submit additional data by a 'second deadline'. The two data sets were analysed separately as described below.

A list of the participants is given in Appendix G.

4. TREATMENT OF DATA

To preserve anonymity, each participant was assigned a laboratory number, and their results were coded accordingly. This laboratory number is unique to this exercise and does not link back to previous exercises.

The data were analysed using the same methods as described in Harms et al. 2009.

The deviation 'D' from the assigned value from each laboratory value was calculated from:

$$D = \frac{L - N}{N} = \left(\frac{L}{N} - 1\right) \tag{1}$$

The standard uncertainty (k=1) ' u_D ' of the deviation was calculated from:

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

The quantities zeta (ζ), the relative uncertainty of a laboratory's value (R_L) and the z-score were calculated from:

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

$$R_L = \frac{u_L}{L} \tag{4}$$

$$z = \frac{L - N}{\sigma_p} = \frac{L - N}{0.05823 \, N} \tag{5}$$

where:

L is the participant's value;

N is the Assigned Value;

 $u_{\rm L}$ is the standard uncertainty of the participants' value;

 $u_{\rm N}$ is the standard uncertainty of the Assigned Value;

 σ_{p} is the standard uncertainty for proficiency assessment.

The value of the standard uncertainty for proficiency assessment σ_p is chosen by perception (viz. ISO 13528:2015 paragraph 6.3). It corresponds to a level of performance that NPL would wish laboratories to be able to achieve. It corresponds to a deviation D of 15 % (at a 99 % confidence level). In other words, any result with a deviation D smaller than \pm 15 % will pass the z-test.

Note that the z-score presented is as defined in ISO 13528:2015 rather than the commonly understood z-score and is used to reject results on the basis of a maximum percentage deviation.

The zeta and z-scores were used to determine whether the difference between the participant's value and the Assigned Value was significantly different from zero. The Interquartile Range outlier test (Harms and Gilligan, 2011) was used to determine whether the relative uncertainty $R_{\rm L}$ was significantly larger than the other values in the data set. Note that this test is unable to identify outliers if the data set is smaller than 7.

Results for which the absolute values of the zeta score and the z-score are both \leq 2.576 and for which R_L is not significantly larger than the other values in the data set are taken to mean that the participant's value is 'in agreement' with the Assigned Value. These results are plotted in white in this report.

If (i) R_L is significantly larger than the other values in the data set, or (ii) the result passes the zeta test but not the z-test (i.e., there is a large deviation from the Assigned Value combined with a large uncertainty), or (iii) the result passes the z-test but not the zeta test (where there is a small deviation from the Assigned Value and a small uncertainty), the participant's value is classified as 'questionable' (plotted in yellow).

If the absolute values of both the zeta score and the z-score are greater than 2.576, then the participant's value is classified as 'discrepant' from the Assigned Value (plotted in red), regardless of the value of R_L .

Table 1 Summary of data classification criteria

zeta test	<i>R</i> ∟ test	z test	Classification
pass	pass	pass	in agreement
pass	fail	pass	questionable
fail	pass	pass	questionable
pass	-	fail	questionable
fail	-	fail	discrepant

5. SUMMARY OF PARTICIPANTS RESULTS

The summary of participant results for the exercise are presented in Tables 2 and 3. The tables show the power moderated mean (PMM), deviation from the NPL assigned value, and the zeta. These terms are described in section 4 of the report, titled treatment of data.

The reference time is 2021-06-01 12:00 UTC.

Table 2 Summary of participant results for the first deadline.

Nuclide	NPL Assigned Values (Bq g ⁻¹)	` · · · · ·		Zeta
⁶⁰ Co	5.903 ± 0.043	5.65 ± 0.11	- 4.3	- 2.16
¹³⁷ Cs	11.89 ± 0.18	10.57 ± 0.15	- 11.1	- 5.60
²⁴¹ Am	18.91 ± 0.15	21.05 ± 0.66	11.3	3.14

Table 3 Summary of participant results for the second deadline.

Nuclide	NPL Assigned Values (Bq g ⁻¹)			Zeta
⁶⁰ Co	5.903 ± 0.043	5.55 ± 0.17	- 6.0	- 2.03
¹³⁷ Cs 11.89 ± 0.18		11.08 ± 0.48	- 6.8	- 1.57
²⁴¹ Am	²⁴¹ Am 18.91 ± 0.15		- 3.8	- 0.70

The deviation plots (Figures 1 - 6) for participants of the exercise and the results tables (Tables 3 - 9) are presented on the following pages. Some participant results have not been plotted as they fall outside of the range of the chart (± 50 %).

Deviation (%) of 60Co in Drum

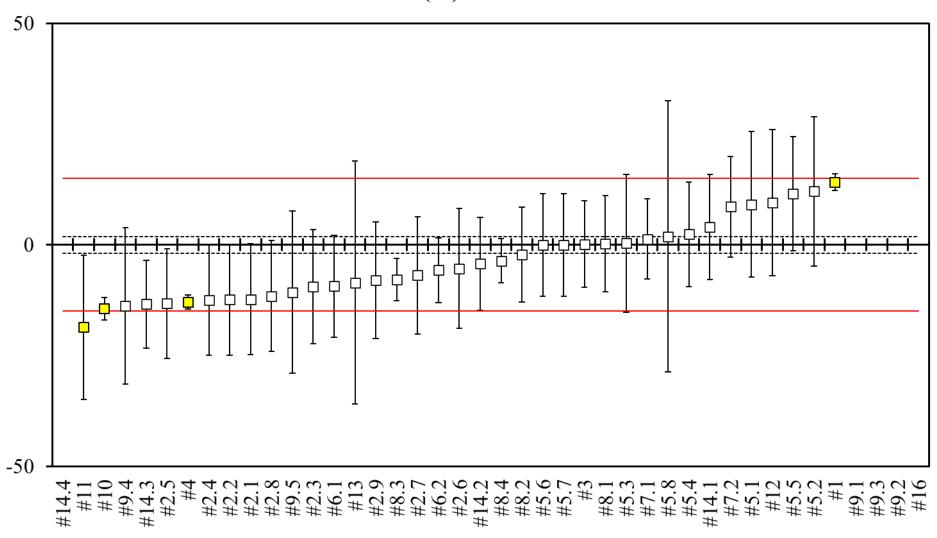


Figure 1 Deviation plot ⁶⁰Co (first deadline).

NPL assigned activity per unit mass for 60 Co = 5.903 \pm 0.043 Bq g $^{-1}$

Reference Time = 2021-06-01 12:00 UTC

Table 4 Reported results for ⁶⁰Co (first deadline).

	Laboratory		Dalatina Harantainta (0)	7-4- 0	70	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at <i>k</i> =1 (Bq g ⁻¹)	Relative Uncertainty (%)	Zeta Score	Z Score	(%)	Result
1	6.7374	0.1	1.5	7.67	2.43	14.14	Questionable
2.1	5.177743019	0.736172295	14.2	-0.98	-2.11	-12.29	In agreement
2.2	5.17169237	0.735370973	14.2	-0.99	-2.13	-12.39	In agreement
2.3	5.34712821	0.761067948	14.2	-0.73	-1.62	-9.42	In agreement
2.4	5.168323299	0.735886929	14.2	-1.00	-2.14	-12.45	In agreement
2.5	5.120317386	0.728788749	14.2	-1.07	-2.28	-13.26	In agreement
2.6	5.587416356	0.79555889	14.2	-0.40	-0.92	-5.35	In agreement
2.7	5.49496797	0.78141843	14.2	-0.52	-1.19	-6.91	In agreement
2.8	5.220776398	0.738416973	14.1	-0.92	-1.98	-11.56	In agreement
2.9	5.429545767	0.773820476	14.3	-0.61	-1.38	-8.02	In agreement
3	5.91	0.575309735	9.7	0.01	0.02	0.12	In agreement
4	5.14	0.09	1.8	-7.65	-2.22	-12.93	Questionable
5.1	6.441453619	0.966733436	15.0	0.56	1.57	9.12	In agreement
5.2	6.616492958	0.993523426	15.0	0.72	2.08	12.09	In agreement
5.3	5.923807181	0.916640973	15.5	0.02	0.06	0.35	In agreement
5.4	6.044561978	0.695504081	11.5	0.20	0.41	2.40	In agreement

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	Laboratory			Zata Caava	Z Score	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at <i>k</i> =1 (Bq g ⁻¹)	Relative Uncertainty (%)	Zeta Score	2 Score	(%)	Result
5.5	6.586729794	0.758708303	11.5	0.90	1.99	11.58	In agreement
5.6	5.901405307	0.680506046	11.5	0.00	0.00	-0.03	In agreement
5.7	5.901405307	0.680506046	11.5	0.00	0.00	-0.03	In agreement
5.8	6.015626055	1.804692676	30.0	0.06	0.33	1.91	In agreement
6.1	5.35	0.68	12.7	-0.81	-1.61	-9.37	In agreement
6.2	5.5657	0.4289	7.7	-0.78	-0.98	-5.71	In agreement
7.1	5.98	0.53	8.9	0.14	0.22	1.30	In agreement
7.2	6.41	0.67	10.5	0.76	1.47	8.59	In agreement
8.1	5.92	0.64	10.8	0.03	0.05	0.29	In agreement
8.2	5.77	0.63	10.9	-0.21	-0.39	-2.25	In agreement
8.3	5.44	0.28	5.1	-1.63	-1.35	-7.84	In agreement
8.4	5.69	0.29	5.1	-0.73	-0.62	-3.61	In agreement
9.1	18.1	3.63	20.1	3.36	35.48	206.62	Discrepant
9.2	19	4.76	25.1	2.75	38.10	221.87	Discrepant
9.3	19	4.12	21.7	3.18	38.10	221.87	Discrepant
9.4	5.09	1.04	20.4	-0.78	-2.37	-13.77	In agreement
9.5	5.27	1.08	20.5	-0.59	-1.84	-10.72	In agreement
10	5.0543	0.1443	2.9	-5.64	-2.47	-14.38	Questionable

	Laboratory		Dolotivo Unocutointo (9/)	Zeta Score	Z Score	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at k=1 (Bq g ⁻¹)	Relative Uncertainty (%)	Zeta Score	Z Score	(%)	Result
11	4.8	0.96	20.0	-1.15	-3.21	-18.69	Questionable
12	6.46380645	0.969570968	15.0	0.58	1.63	9.50	In agreement
13	5.4	1.62	30.0	-0.31	-1.46	-8.52	In agreement
14.1	6.137189009	0.700229857	11.4	0.33	0.68	3.97	In agreement
14.2	5.648620985	0.617786115	10.9	-0.41	-0.74	-4.31	In agreement
14.3	5.111175716	0.583407109	11.4	-1.35	-2.30	-13.41	In agreement
14.4	1.893444732	0.305394312	16.1	-13.00	-11.66	-67.92	Discrepant
16	23.3	1.7	7.3	10.23	50.61	294.71	Discrepant

Deviation (%) of ¹³⁷Cs in Drum

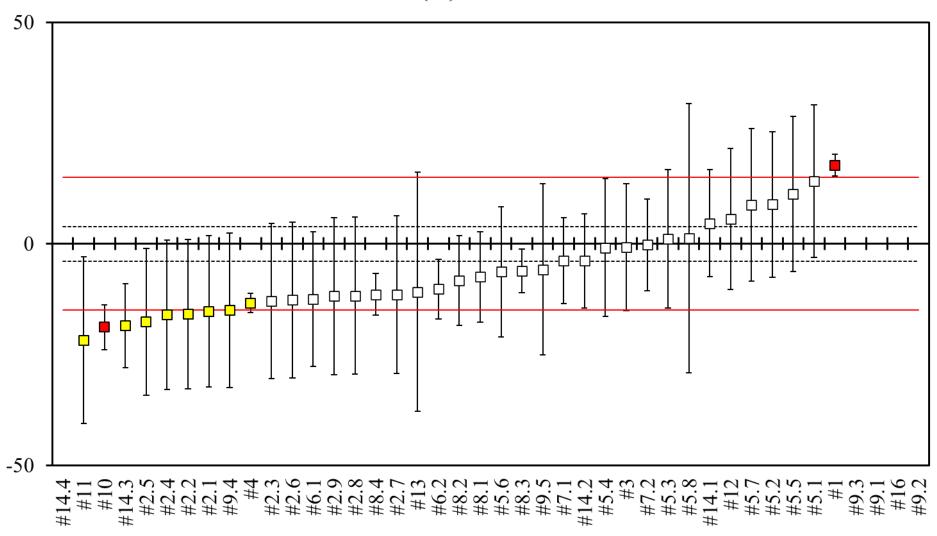


Figure 2 Deviation plot ¹³⁷Cs (first deadline).

NPL assigned activity per unit mass for 137 Cs = 11.89 ± 0.18 Bq g⁻¹

Reference Time = 2021-06-01 12:00 UTC

Table 5 Reported results for ¹³⁷Cs (first deadline).

	Laboratory		Relative Uncertainty (%)	Zoto Coore	Z Score	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at k=1 (Bq g ⁻¹)	ixerative officertainty (76)	Zeta Score	Z Score	(%)	Result
1	13.9976	0.2	1.4	7.83	3.04	17.73	Discrepant
2.1	10.07755739	2.019294757	20.0	-0.89	-2.62	-15.24	Questionable
2.2	9.997936082	2.00330956	20.0	-0.94	-2.73	-15.91	Questionable
2.3	10.35896588	2.075784736	20.0	-0.73	-2.21	-12.88	In agreement
2.4	9.984259757	2.00080394	20.0	-0.95	-2.75	-16.03	Questionable
2.5	9.788278505	1.962035046	20.0	-1.07	-3.04	-17.68	Questionable
2.6	10.38405212	2.081774335	20.0	-0.72	-2.18	-12.67	In agreement
2.7	10.53102023	2.110166779	20.0	-0.64	-1.96	-11.43	In agreement
2.8	10.49635295	2.09937133	20.0	-0.66	-2.01	-11.72	In agreement
2.9	10.48851691	2.102945067	20.0	-0.66	-2.02	-11.79	In agreement
3	11.8	1.692912041	14.3	-0.05	-0.13	-0.76	In agreement
4	10.3	0.2	1.9	-5.91	-2.30	-13.37	Questionable
5.1	13.57486163	2.037405844	15.0	0.82	2.43	14.17	In agreement
5.2	12.94292857	1.943433199	15.0	0.54	1.52	8.86	In agreement
5.3	12.01797542	1.851119356	15.4	0.07	0.18	1.08	In agreement
5.4	11.78328887	1.841901018	15.6	-0.06	-0.15	-0.90	In agreement

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	Laboratory		Deletine Here when to (0)	7-4- 0	Z Score	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at <i>k</i> =1 (Bq g ⁻¹)	Relative Uncertainty (%)	Zeta Score	Z Score	(%)	Result
5.5	13.22592053	2.068993464	15.6	0.64	1.93	11.24	In agreement
5.6	11.14032224	1.742759579	15.6	-0.43	-1.08	-6.31	In agreement
5.7	12.93840051	2.038518401	15.8	0.51	1.51	8.82	In agreement
5.8	12.03990048	3.611977296	30.0	0.04	0.22	1.26	In agreement
6.1	10.4	1.8	17.3	-0.82	-2.15	-12.53	In agreement
6.2	10.6766	0.786	7.4	-1.50	-1.75	-10.21	In agreement
7.1	11.43	1.14	10.0	-0.40	-0.66	-3.87	In agreement
7.2	11.86	1.22	10.3	-0.02	-0.04	-0.25	In agreement
8.1	11	1.2	10.9	-0.73	-1.29	-7.49	In agreement
8.2	10.9	1.19	10.9	-0.82	-1.43	-8.33	In agreement
8.3	11.17	0.56	5.0	-1.22	-1.04	-6.06	In agreement
8.4	10.53	0.54	5.1	-2.39	-1.96	-11.44	In agreement
9.1	41.6	8.75	21.0	3.39	42.91	249.87	Discrepant
9.2	48.7	11	22.6	3.35	53.17	309.59	Discrepant
9.3	35.5	7.46	21.0	3.16	34.10	198.57	Discrepant
9.4	10.1	2.07	20.5	-0.86	-2.59	-15.05	Questionable
9.5	11.2	2.29	20.4	-0.30	-1.00	-5.80	In agreement
10	9.6494	0.5803	6.0	-3.69	-3.24	-18.84	Discrepant

	Laboratory		Polotivo Uno ortainty (9/)	Zeta Score	Z Score	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at k=1 (Bq g ⁻¹)	Relative Uncertainty (%)	Zeta Score	Z Score	(%)	Result
11	9.3	2.23	24.0	-1.16	-3.74	-21.78	Questionable
12	12.55616253	1.883424379	15.0	0.35	0.96	5.60	In agreement
13	10.6	3.2	30.2	-0.40	-1.86	-10.85	In agreement
14.1	12.43838407	1.424395595	11.5	0.38	0.79	4.61	In agreement
14.2	11.43527595	1.253867977	11.0	-0.36	-0.66	-3.82	In agreement
14.3	9.690547857	1.113510157	11.5	-1.95	-3.18	-18.50	Questionable
14.4	3.631334008	0.742316897	20.4	-10.81	-11.93	-69.46	Discrepant
16	45.6	3.2	7.0	10.52	48.69	283.52	Discrepant

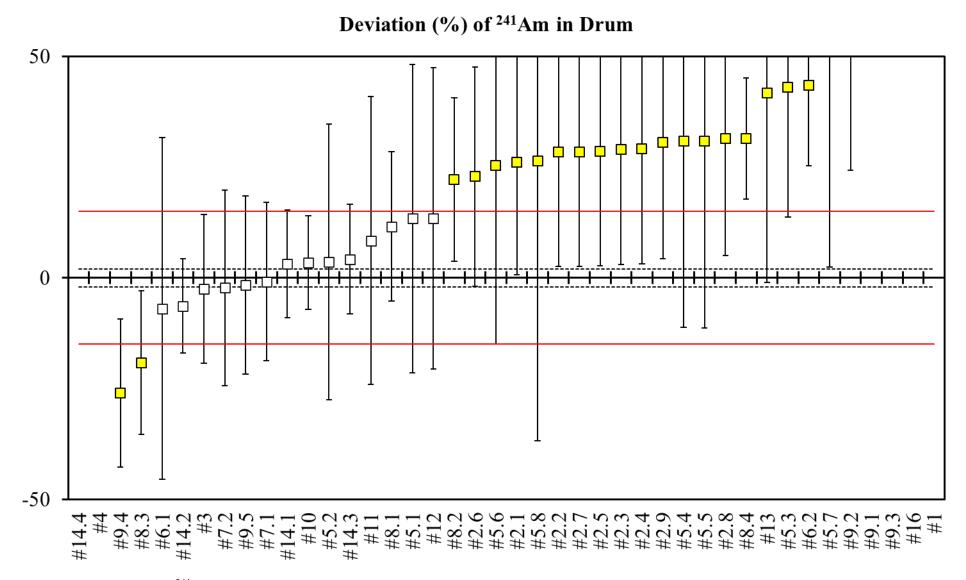


Figure 3 Deviation plot ²⁴¹Am (first deadline).

NPL assigned activity per unit mass for 241 Am = 18.91 ± 0.15 Bq g⁻¹

Reference Time = 2021-06-01 12:00 UTC

Table 6 Reported results for ²⁴¹Am (first deadline).

	Laboratory		Deletine Here when to (0/)	7-4- 0	7.0	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at k=1 (Bq g ⁻¹)	Relative Uncertainty (%)	Zeta Score	Z Score	(%)	Result
1	116.3468	4.3	3.7	22.65	88.49	515.27	Discrepant
2.1	23.82949183	4.784485334	20.1	1.03	4.47	26.02	Questionable
2.2	24.28454119	4.873889384	20.1	1.10	4.88	28.42	Questionable
2.3	24.38726942	4.896842467	20.1	1.12	4.97	28.96	Questionable
2.4	24.40990403	4.901023369	20.1	1.12	4.99	29.08	Questionable
2.5	24.31335949	4.879244422	20.1	1.11	4.91	28.57	Questionable
2.6	23.22575193	4.664363778	20.1	0.92	3.92	22.82	Questionable
2.7	24.28690199	4.877492932	20.1	1.10	4.88	28.43	Questionable
2.8	24.84006842	4.975926731	20.0	1.19	5.39	31.36	Questionable
2.9	24.67618606	4.952693214	20.1	1.16	5.24	30.49	Questionable
3	18.44	3.18	17.2	-0.15	-0.43	-2.49	In agreement
4	4.79	0.34	7.1	-38.00	-12.82	-74.67	Discrepant
5.1	21.43762815	6.586934431	30.7	0.38	2.30	13.37	In agreement
5.2	19.58480273	5.884213482	30.0	0.11	0.61	3.57	In agreement
5.3	27.04254375	5.537848147	20.5	1.47	7.39	43.01	Questionable
5.4	24.73168371	7.935791562	32.1	0.73	5.29	30.79	Questionable

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	Laboratory		Deletive Unequipierty (9/)	7.4.0	7.0	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at <i>k</i> =1 (Bq g ⁻¹)	Relative Uncertainty (%)	Zeta Score	Z Score	(%)	Result
5.5	24.73523712	7.972244416	32.2	0.73	5.29	30.81	Questionable
5.6	23.69728815	7.611580644	32.1	0.63	4.35	25.32	Questionable
5.7	28.6048928	9.242312334	32.3	1.05	8.80	51.27	Questionable
5.8	23.90109345	11.95073743	50.0	0.42	4.53	26.39	Questionable
6.1	17.6	7.3	41.5	-0.18	-1.19	-6.93	In agreement
6.2	27.1284	3.4407	12.7	2.39	7.46	43.46	Questionable
7.1	18.75	3.37	18.0	-0.05	-0.15	-0.85	In agreement
7.2	18.48	4.16	22.5	-0.10	-0.39	-2.27	In agreement
8.1	21.1	3.18	15.1	0.69	1.99	11.58	In agreement
8.2	23.1	3.48	15.1	1.20	3.81	22.16	Questionable
8.3	15.29	3.07	20.1	-1.18	-3.29	-19.14	Questionable
8.4	24.86	2.58	10.4	2.30	5.40	31.46	Questionable
9.1	58.9	16.8	28.5	2.38	36.32	211.48	Questionable
9.2	40.7	17.2	42.3	1.27	19.79	115.23	Questionable
9.3	59.8	12.6	21.1	3.25	37.13	216.23	Discrepant
9.4	14	3.16	22.6	-1.55	-4.46	-25.97	Questionable
9.5	18.6	3.8	20.4	-0.08	-0.28	-1.64	In agreement
10	19.5688	1.9932	10.2	0.33	0.60	3.48	In agreement

	Laboratory		Relative Uncertainty (%)	Zata Saara	Z Score	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at <i>k</i> =1 (Bq g ⁻¹)	Relative Uncertainty (%)	Zeta Score	2 Score	(%)	Result
11	20.5	6.15	30.0	0.26	1.44	8.41	In agreement
12	21.44409315	6.433227945	30.0	0.39	2.30	13.40	In agreement
13	26.8	8.1	30.2	0.97	7.17	41.72	Questionable
14.1	19.50418694	2.300493845	11.8	0.26	0.54	3.14	In agreement
14.2	17.7037991	2.010431423	11.4	-0.60	-1.10	-6.38	In agreement
14.3	19.70432105	2.340513262	11.9	0.34	0.72	4.20	In agreement
14.4	0.430093383	0.140030404	32.6	-90.06	-16.78	-97.73	Discrepant
16	77	6	7.8	9.68	52.75	307.19	Discrepant

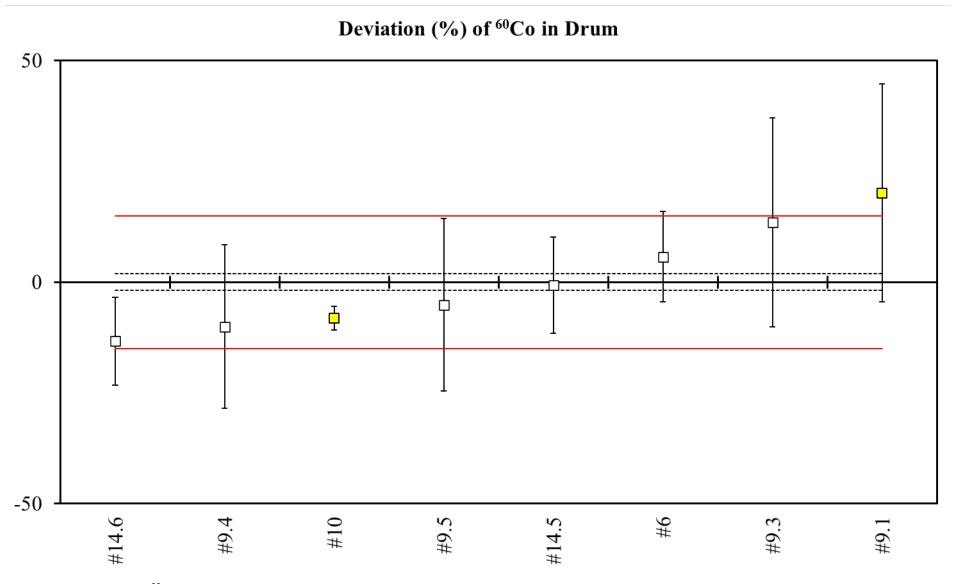


Figure 4 Deviation plot for ⁶⁰Co (second deadline).

NPL assigned activity per unit mass for 60 Co = 5.903 \pm 0.043 Bq g⁻¹

Reference Time = 2021-06-01 12:00 UTC

Table 7 Report results for ⁶⁰Co (second deadline).

	Laboratory		Relative Uncertainty (%)	Zeta Score	Z Score	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at <i>k</i> =1 (Bq g ⁻¹)	Relative Officertainty (%)	Zeta Score	2 3core	(%)	Result
6	6.24	0.6	9.6	0.56	0.98	5.71	In agreement
9.1	7.09	1.45	20.5	0.82	3.45	20.11	Questionable
9.3	6.7	1.39	20.7	0.57	2.32	13.50	In agreement
9.4	5.31	1.09	20.5	-0.54	-1.73	-10.05	In agreement
9.5	5.6	1.15	20.5	-0.26	-0.88	-5.13	In agreement
10	5.4226	0.1548	2.9	-2.99	-1.40	-8.14	Questionable
14.5	5.86183134	0.641104797	10.9	-0.06	-0.12	-0.70	In agreement
14.6	5.117645307	0.58414557	11.4	-1.34	-2.28	-13.30	In agreement

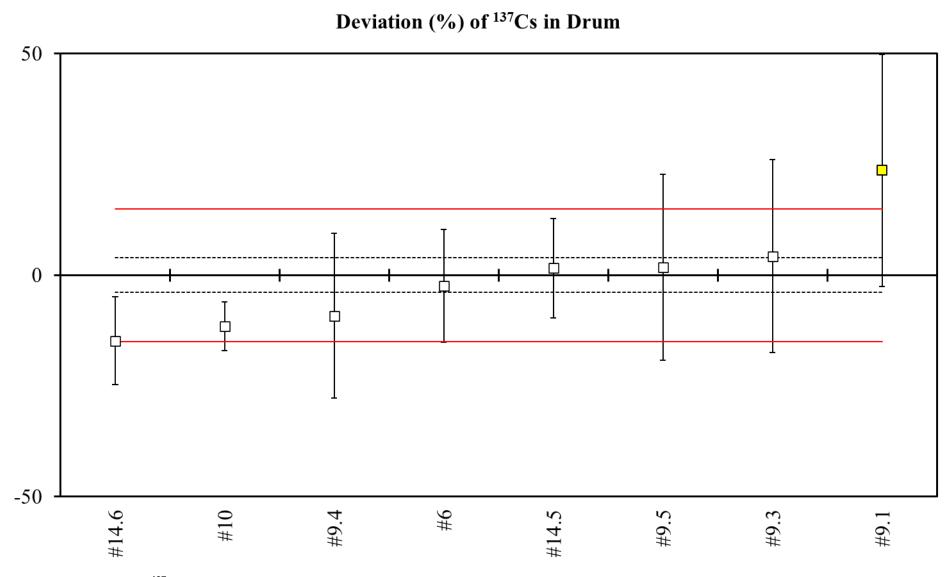


Figure 5 Deviation plot for ¹³⁷Cs (second deadline).

NPL assigned activity per unit mass for 137 Cs = 11.89 ± 0.18 Bq g⁻¹

Reference Time = 2021-06-01 12:00 UTC

Table 8 Reported results ¹³⁷Cs (second deadline).

	Laboratory		Relative Uncertainty (%)	Zeta Score	7.0000	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at <i>k</i> =1 (Bq g ⁻¹)	Relative Uncertainty (%)	Zela Score	Z Score	(%)	Result
6	11.6	1.5	12.9	-0.19	-0.42	-2.44	In agreement
9.1	14.7	3.1	21.1	0.90	4.06	23.63	Questionable
9.3	12.4	2.58	20.8	0.20	0.74	4.29	In agreement
9.4	10.8	2.2	20.4	-0.49	-1.57	-9.17	In agreement
9.5	12.1	2.48	20.5	0.08	0.30	1.77	In agreement
10	10.5225	0.6328	6.0	-2.08	-1.98	-11.50	In agreement
14.5	12.07665316	1.324194426	11.0	0.14	0.27	1.57	In agreement
14.6	10.12794716	1.163770326	11.5	-1.50	-2.55	-14.82	In agreement

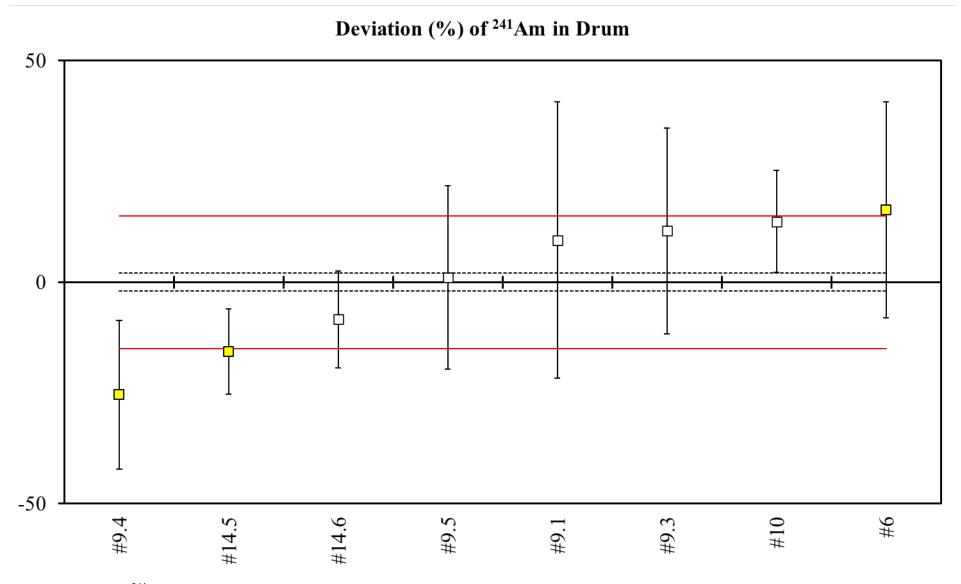


Figure 6 Deviation plot ²⁴¹Am (second deadline).

NPL assigned activity per unit mass for 241 Am = 18.91 ± 0.15 Bq g $^{-1}$

Reference Time = 2021-06-01 12:00 UTC

Table 9 reported results ²⁴¹Am (second deadline).

	Laboratory		Relative Uncertainty (%)	Zeta Score	Z Score	Deviation	Classification of
Code	Reported Activity per Unit Mass (Bq g ⁻¹)	Uncertainty at <i>k</i> =1 (Bq g ⁻¹)	Relative Officertainty (%)	Zeta Score	2 3core	(%)	Result
6	22	4.6	20.9	0.67	2.81	16.34	Questionable
9.1	20.7	5.89	28.5	0.30	1.63	9.47	In agreement
9.3	21.1	4.39	20.8	0.50	1.99	11.58	In agreement
9.4	14.1	3.17	22.5	-1.52	-4.37	-25.44	Questionable
9.5	19.1	3.91	20.5	0.05	0.17	1.00	In agreement
10	21.4985	2.1694	10.1	1.19	2.35	13.69	In agreement
14.5	15.94385414	1.810573267	11.4	-1.63	-2.69	-15.69	Questionable
14.6	17.31522862	2.05673274	11.9	-0.77	-1.45	-8.43	In agreement

6. DISCUSSION

The 2021 drum contained an inhomogeneous distribution of ⁶⁰Co, ¹³⁷Cs and ²⁴¹Am as three point-sources in the bottom third of the drum. Sixteen laboratories participated in the exercise and 15 submitted results by the first deadline with one laboratory unable to provide results due to issues related to their measurement instrument.

Seven of the laboratories chose to submit multiple results in order to compare different measurement methods. This resulted in a total of 42 sets of results were submitted for the first reporting deadline. Following the disclosure of the location of activity laboratories were invited to provide a second set of results, 4 of the 15 laboratories submitted.

All participants submitted at least one result obtained using HPGe detectors. These devices were manufactured by Ortec® (AMETEK Inc.), Mirion Technologies Inc., and one from ITECH Instruments. Two participants submitted additional results using CZT detectors from H3D and Kromek (participants 5.8 and 14.4 respectively). The number of detectors used ranged from one to nine. A range of detector window materials were used including carbon epoxy, carbon fibre, and most commonly aluminium (18). A number of participants used Mirion Technologies Inc. broad-energy germanium detectors (BEGe): 5 (5.2, 5.3, 5.4, 5.6 and 5.7), 7 (7.1 and 7.2), 11 and 12. Laboratories 5.2, 5.3, 5.4 and 5.6 opted for the BE5030 and laboratories 5.7 and 12 opted for the BE3830. The Ortec IDM-200-V designed for field applications was used for by laboratories 6.1, 8.1, 8.2, 9.5, 14.1 and 14.2. The trans-SPEX-DX-100T, an in-situ portable HPGe detector by Ortec, was used by laboratories 6.2 and 14.3.

Measuring distance from the drum ranged from 0.2 - 2 m and number of segments measured ranged from 1 to 24 with some with some participants opting for a helical-based scans of the drum (4, 8.3 and 8.4). Measurements at a single point were made by participants 2, 5 (5.4 to 5.8), 6 (6.1 and 6.2), 7.1, 8 (8.1 and 8.2), 9 (9.1, 9.4 and 9.5) 10, 11, 13 and 14 (14.1 - 14.3). Sequential measurements at various heights were made by participants 1, 5 (5.1 to 5.3), 7.2, 9 (9.2 and 9.3), 14.4 and 16, whilst participant 3, opted for three detectors covering the top middle and bottom of the drum.

Measurement acquisition software included Non-Destructive Assay Software (NDA 2000™, Mirion Technologies Inc.), Genie™ 2000 (Gamma Analysis Software, Mirion Technologies Inc.), Genie™ Bridge Spectrum Viewer (Mirion Technologies Inc.), GammaVision Gamma Spectrometry (Ortec®), Maestro 32 (Multichannel Analyser Emulation Software, Ortec®), ISOTOPIC (Gamma Spectrometry Waste Assay Measurement, Ortec®), Visualizer (H3D), InterWinner (ITECH Instruments) and SpectraLine (Laboratory of Spectrometry and Radiometry). A range of efficiency modelling methods and software were used, including In Situ Object Counting System (ISOCS) with MCNP modelling code, ISOTOPIC, SNAP, NDA and in-house methods.

Of the initial results (i.e. submitted by the first deadline), 59 % were 'in agreement' with the assigned value. The percentages 'in agreement' by nuclide were, 79 % for 60 Co, 64 % for 137 Cs and 33 % for 241 Am. This is an improvement for 60 Co and 137 Cs in level of agreement observed for the 2019 exercise where the percentages in agreement for each radionuclide were as follows, 60 Co, 67 %, 137 Cs 61 % and 241 Am 43 % The bias (relative to the assigned value) of the first deadline results were – 4.3 %, – 11.1 % and 11.3 % for 60 Co, 137 Cs and 241 Am respectively.

For the second round of reporting of results a total of eight sets of results were submitted by four independent laboratories. Of these results, 75 % were 'in agreement', 25 % were 'questionable' and there were no discrepant results. The collective bias for the second deadline results were -6.0 %, 6.8 % and -3.8 % for 60 Co, 137 Cs and 241 Am respectively.

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8. ACKNOWLEDGEMENTS

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9. APPENDICES

Appendix A – Techniques tables as submitted to NPL by participants who agreed to share their techniques. The information contained in the following tables detail the methods used for the drum measurement as submitted by participants. The contents of these tables have not been reviewed or amended by NPL.

Laboratory 1						
	Manufacturer	Canberra Harwell, now Mirion Technologies				
	Detector Type	The detector is a liquid nitrogen cooled coaxial high purity germanium detector. It is a 25% efficient (at 1.3 MeV relative to a 3" Nal crystal) p-type HPGe crystal with a transistor reset preamplifier for high count rate applications.				
	Crystal Type	Coaxial p-type HPGe				
	Number of Detectors	One				
Detector	Orientation/Arrangement of Detectors	Single detector at fixed distance (30 cm) from the drum surface. Detector is moved automatically to allow measurement across four segments of the drum.				
	Window Material	Aluminium				
	Collimated (if yes, provide details)	The detector is mounted in a lead collimator that restricts its field of view to a single quarter of the waste drum (220 mm in height at the axis of the drum). The solid lead collimator assembly provides all round shielding for the detector crystal.				
	Detector Shielding	The solid lead collimator assembly provides all round shielding for the detector crystal.				
	Acquisition Software	NDA2000 version 5.2				
	(Type and Version)	Genie2000 version 3.2.1				
	Distance from Drum (m)	30 cm				
Scanning Method	Rotation (Automatic/Manual)	The drum was constantly revolved on a turntable throughout the counting time (at 10 revolutions per minute). This is automatic.				

	Count of Vertical Segments	The second secon
	Count of Vertical Segments Measured	The analysis was performed over a series of four segments.
	Count Time	60 second transmission assay followed by 180 second drum assay, per segment. Total time: 960 seconds (16 minutes).
Matrix Density	Describe how the matrix density	Transmission correction, using a Sealed Eu-152
Correction	correction was applied	reference source (I6-097).
Modelling and Fitting	Modelling	
Software (if used)	Fitting	
Detector Calibration	Describe how the detector was calibrated and how this was applied	The system is subject to an efficiency and energy/resolution calibration procedure every year. The results from the calibration measurements are compared with the original calibration results and if they are found to be consistent, the system parameters are left unchanged. The efficiency and energy calibration is performed with a Eu-152 source (Y669). The source has a declared activity of 63,200 kBq (± 3.5%) on 1 October 1993. There is a Certificate of Calibration provided by Isotope Products Laboratories, USA. This is determined for an empty drum (i.e. containing no waste matrix). The measured spectrum is used as the input data to the Genie-2000 Efficiency Calibration utility. It calculates the efficiency at each of the Eu-152 gamma-ray energies from the background corrected count rate in each of the corresponding photopeaks, using the source certificate information to calculate the corresponding decay corrected emission rates. The system is also subject to transmission calibration procedures every year. The results from the

	calibration measurements are compared with the original calibration results and if they are found to be consistent, the system parameters are left unchanged. The transmission calibration is performed using the standard transmission source for the system. This is a Eu-152 (ID I6-097) source which had a declared activity of 74,000 kBq on 1 December 2011.
Additional Information	For declaration purposes current procedure at Winfrith only requires the quantification of Co-60 and/or Cs-137 in waste drums. These quantities are then applied to a well established waste stream fingerprint from which other radionuclides can be inferred. This includes Am-241. However, although not optimised to measure Am-241, direct measurements can be reported if Am-241 is detected. Results have been decay corrected to the 01 June 2021.

Laboratory 2					
	Manufacturer	Mirion (Canberra)			
	Detector Type	Liquid nitrogen/ Electrically cooled			
	Crystal Type	High purity Germanium			
Datastan	Number of Detectors	9			
Detector	Orientation/Arrangement of Detectors	Horizontal			
	Window Material	-			
	Collimated (if yes, provide details)	Lead/Tungsten, Copper, Aluminium			
	Detector Shielding	-			

	Acquisition Software (Type and Version)	Genie 2000 V3.4.1
	Distance from Drum (m)	0.503m
	Rotation (Automatic/Manual)	Both
Scanning Method	Count of Vertical Segments Measured	Mid-point
	Count Time	600 seconds
Matrix Density Correction	Describe how the matrix density correction was applied	As no mass percentages were provided for the 2021 measurement, and the setup was described as "as with the 2019 exercise" the same mass fraction as the 2019 measurement were used to give a bulk waste density
Modelling and Fitting	Modelling	ISOCS
Software (if used)	Fitting	Genie – non-linear square fit
Detector Calibration	Describe how the detector was calibrated and how this was applied	-
Additional Information		-

Laboratory 4				
Detector	Manufacturer	Ortec		
	Detector Type	GEM-series		
	Crystal Type	HPGe		
	Number of Detectors	1		
	Orientation/Arrangement of Detectors	Horizontal		
	Window Material	No window		

	Collimated (if yes, provide details)	Yes, variable collimator (1, 3.5, 14, 35, 70 mm openings; 70 mm opening used in this measurement)
	Detector Shielding	Lead, tungsten collimator
	Acquisition Software (Type and Version)	GammaVision 7.02.01
	Distance from Drum (m)	0,33-0,34
	Rotation (Automatic/Manual)	Automatic
Scanning Method	Count of Vertical Segments Measured	8-segment (helical scan)
	Count Time	800 s
Matrix Density Correction	Describe how the matrix density correction was applied	A collimated transmission source Eu-152, is scanned vertically over the drum and the detector follows the position of the transmission source. For each segment the attenuation of each gamma energy line is determined by comparing the measured value from the detector, with the value obtained without any intervening absorbing matrix.
Modelling and Fitting	Modelling	-
Software (if used)	Fitting	-
Detector Calibration	Describe how the detector was calibrated and how this was applied	Eu-152 source is placed in the middle of a calibration drum. It is applied automatically in the manufacturers assay software. Regular transmission measurements with an empty calibration drum are done to check transmission calculations.
Additional Information		Our measurement range is 100 keV onwards. Am-241 line 59,54 keV was visible on the spectrum and activity was calculated. However the efficiency calibration below 100 keV is not optimal and the measured Am-241 result is not considered reliable.

Laboratory 6.1		
	Manufacturer	ORTEC
	Detector Type	IDM-200-V
	Crystal Type	P-Type HPGe
	Number of Detectors	1
	Orientation/Arrangement of	Directly facing drum, with the centre of the detector
Detector	Detectors	49.5cm up from the drum base
20100101	Window Material	Aluminium
	Collimated (if yes, provide details)	No, but Copper/Tungsten collimator flush with detector face
	Detector Shielding	None
	Acquisition Software (Type and Version)	Maestro 32 (MCA Emulator) V6.06
	Distance from Drum (m)	91cm
	Rotation (Automatic/Manual)	Automatic
Scanning Method	Count of Vertical Segments Measured	1
	Count Time	600s
Matrix Density Correction	Describe how the matrix density correction was applied	Matrix density correction is applied in modelling software. Matrix modelled as 25% Vermiculite 75% paraffin by volume. (Based on 59% plastic 41% vermiculite by mass). Figures taken from 2019 NPL PTE
Modelling and Fitting	Modelling	SNAP V1.4
Software (if used)	Fitting	None
Detector Calibration	Describe how the detector was calibrated and how this was applied	Calibrated using Am-241, Ba-133, Co-57, Co-60, Eu- 152, Na-22 Intrinsic Efficiency calculated manually for each peak

	Efficiency Curve generated in Excel using equation for trendline from a graph of Ln(Energy) against Ln(Intrinsic efficiency) for input into SNAP.
Additional Information	Our normal measurements are counted at 60cm. The greater distance was to allow a different team to finish their count. Further measurements were taken of the drum @40cm in three segments that will be used once the detailed data is available.

Laboratory 6.2		
	Manufacturer	ORTEC
	Detector Type	Trans-Spec-DX-100T
	Crystal Type	N
	Number of Detectors	1
Detector	Orientation/Arrangement of Detectors	Mid-height and centred.
	Window Material	Aluminium
	Collimated (if yes, provide details)	N/A.
	Detector Shielding	N/A.
	Acquisition Software (Type and Version)	ORTEC GammaVision version 6.08
	Distance from Drum (m)	3
Scanning Method	Rotation (Automatic/Manual)	Automatic
	Count of Vertical Segments	1
	Measured	1
	Count Time	65950 s

Matrix Density Correction	Describe how the matrix density correction was applied	Material defined based on information provided by NPL on the materials in the matrix using a density based on the mass of the matrix equally distributed across the internal volume.
Modelling and Fitting	Modelling	Mirion ISOCS
Software (if used)	Fitting	N/A.
Detector Calibration	Describe how the detector was calibrated and how this was applied	Detector characterised by Mirion for ISOCS.
Additional Information		Activity assumed to be equally distributed across the bottom half of the matrix based on nearfield measurements.

Laboratory 7.1		
	Manufacturer	Mirion Technologies (Canberra UK) Ltd
	Detector Type	Mirion BdEGe Broad Energy Ge Detector
	Crystal Type	BEGe
	Number of Detectors	One
Detector	Orientation/Arrangement of Detectors	Detectors placed on a variable-height trolley ~50 cm from the surface of the Drum
	Window Material	Carbon fibre
	Collimated (if yes, provide details)	25 mm 90° Lead Collimator
	Detector Shielding	5 mm 90° Lead Collimator
	Acquisition Software (Type and Version)	GENIE™ 2000 Spectroscopy Software Version 3.4.1
Scanning Method	Distance from Drum (m)	50 cm
	Rotation (Automatic/Manual)	Automatic

	Count of Vertical Segments Measured	1
	Count Time	Multiple counts ranging from 1000 s to 5000 s.
Matrix Density Correction	Describe how the matrix density correction was applied	The influence of the matrix was accounted for using models generated using the Advanced ISOCS Uncertainty Estimator (A-IUE) software.
Modelling and Fitting	Modelling	ISOCS™ Calibration software and Advanced ISOCS Uncertainty Estimator (A-IUE) software.
Software (if used)	Fitting	-
Detector Calibration	Describe how the detector was calibrated and how this was applied	Energy and Peak shape calibration performed using a Na22/Eu155 calibration source. Efficiency calibration generated using ISOCS™ Calibration Software.
Additional Information		

Laboratory 7.2		
	Manufacturer	Mirion Technologies (Canberra UK) Ltd
	Detector Type	Mirion BEGe Broad Energy Ge Detector
	Crystal Type	BEGe
	Number of Detectors	One
	Orientation/Arrangement of	Detectors placed on a variable-height trolley ~20 cm
Detector	Detectors	from the surface of the Drum
	Window Material	Carbon fibre
	Collimated (if yes, provide details)	25 mm 180° Lead Collimator
	Detector Shielding	25 mm 180° Lead Collimator
	Acquisition Software	GENIE™ 2000 Spectroscopy Software Version 3.4.1
	(Type and Version)	GEINIE - 2000 Speciloscopy Soliware Version 3.4.1
Scanning Method	Distance from Drum (m)	20 cm

	Rotation (Automatic/Manual)	Manual (Drum not rotated during measurement). Measurements performed at 4 radial positions.
	Count of Vertical Segments Measured	3
	Count Time	2000 s for each count
Matrix Density Correction	Describe how the matrix density correction was applied	The influence of the matrix was accounted for using models generated using the ISOCS™ Calibration software.
Modelling and Fitting	Modelling	ISOCS™ Calibration Software
Software (if used)	Fitting	-
Detector Calibration	Describe how the detector was calibrated and how this was applied	Energy and Peak shape calibration performed using a Na22/Eu155 calibration source. Efficiency calibration generated using ISOCS™ Calibration Software.
Additional Information		

Laboratory 9.1		
	Manufacturer	Mirion Technologies (Canberra)
	Detector Type	n-type germanium-detector, model number GR2018, relative efficiency 23.9 % (measured)
	Crystal Type	Coaxial, diameter 51.5 mm, length 50 mm
Detector	Number of Detectors	one
	Orientation/Arrangement of	Detector is aligned with the vertical and radial middle of
	Detectors	the rotated drum.
	Window Material	Aluminium
	Collimated (if yes, provide details)	Detector is in a Pb-chamber with slit-collimator, slit height 10 cm, slit width 3 cm.

	Detector Shielding	Thickness of the Pb-chamber 5.1 cm forwards, 7.9 cm sidewards, 1 cm backwards, additionally a shielding wall made of steel behind the detector.
	Acquisition Software (Type and Version)	GENIE 2000 and NDA 2000
	Distance from Drum (m)	ca. 1.165 m
	Rotation (Automatic/Manual)	Automatic
Scanning Method	Count of Vertical Segments Measured	1
	Count Time	Real time 600 sec
Matrix Density Correction	Describe how the matrix density correction was applied	The matrix density correction results from the numerical efficiency calculation in the modelling program ISOCS.
Modelling and Fitting	Modelling	ISOCS Version 4.2.1
Software (if used)	Fitting	GENIE 2000
Detector Calibration	Describe how the detector was calibrated and how this was applied	Numerical Calibration based on point-source measured data.
Additional Information		

Laboratory 9.2		
Detector	Manufacturer	Mirion Technologies (Canberra)
	Detector Type	n-type germanium-detector, model number GR0518, relative efficiency 4.9 % (measured)
	Crystal Type	Coaxial, diameter 37.5 mm, length 31.5 mm
	Number of Detectors	One
	Orientation/Arrangement of Detectors	Segmented Gamma-Scan of a rotating drum. Detector is aligned with the radial middle of the drum.

	Window Material	Aluminium
	Collimated (if yes, provide details)	Rotatable Pb-Cylinder-Collimator, thickness 30 cm, length 200 cm; selectable boreholediameter 10 mm, 17.5 mm or 25 mm.
	Detector Shielding	At the side 10 cm Pb, additionally a shielding wall made of steel and Pb blocks around the detector.
	Acquisition Software (Type and Version)	InterWinner Version 7.10.3075
	Distance from Drum (m)	ca. 0.69 m
	Rotation (Automatic/Manual)	Automatic
Scanning Method	Count of Vertical Segments Measured	16 vertical segments
	Count Time	1620 sec Real time for the sum spectrum
Matrix Density Correction	Describe how the matrix density correction was applied	The matrix density correction results from the numerical efficiency calculation in the software InterWinner Version 7.10.3075.
Modelling and Fitting	Modelling	InterWinner Version 7.10.3075
Software (if used)	Fitting	InterWinner Version 7.10.3075
Detector Calibration	Describe how the detector was calibrated and how this was applied	Numerical Calibration based on point-source measured data.
Additional Information		

Laboratory 9.3		
	Manufacturer	Ametec / Ortec
Detector	Detector Type	n-type germanium-detector, model number GMX 30- Plus-S, relative efficiency 36 % (measured)
	Crystal Type	Coaxial, diameter 57.8 mm, length 70.1 mm

	Number of Detectors	one
	Orientation/Arrangement of Detectors	Segmented Gamma-Scan of a rotating drum. Detector is aligned with the radial middle of the drum.
	Window Material	Aluminium
	Collimated (if yes, provide details)	Pb-collimator, thickness 10 cm, length 214 cm, borehole diameter 60 cm (a Pb-collimator-slot with diameter 40 cm available in case of high dead times).
	Detector Shielding	At the side 10 cm Pb, additionally a shielding wall composed of heavy concrete blocks behind the detector.
	Acquisition Software (Type and Version)	GammaVision 8.10 and Scanner32 5.0.9.0
	Distance from Drum (m)	ca. 0.41 m
Scanning Method	Rotation (Automatic/Manual)	Automatic
	Count of Vertical Segments Measured	12 vertical segments
	Count Time	3896 sec Real time for the sum spectrum
Matrix Density Correction	Describe how the matrix density correction was applied	Adaptation of an active and a passive matrix ordered to radius and height. Each with a homogeneous density distribution.
Modelling and Fitting	Modelling	Scanner32 5.0.9.0
Software (if used)	Fitting	GammaVision 8.10
Detector Calibration	Describe how the detector was calibrated and how this was applied	Numerical Calibration based on point-source measured data.
Additional Information		

Laboratory 9.4		
	Manufacturer	Ametek / Ortec
	Detector Type	p-type germanium-detector, model number GEM15-S, relative efficiency 20 % (measured)
	Crystal Type	Semi planar, diameter 52.6 mm, length 43.4 mm
	Number of Detectors	one
Detector	Orientation/Arrangement of Detectors	Detector is aligned with the vertical and radial middle of the rotating drum.
	Window Material	Aluminium
	Collimated (if yes, provide details)	open geometry
	Detector Shielding	At the side 5 cm Pb, additionally a shielding wall made of steel and heavy concrete blocks around the detector.
	Acquisition Software (Type and Version)	DigiDART (portable digital MCA from ORTEC)
Scanning Method	Distance from Drum (m)	2.706 m
	Rotation (Automatic/Manual)	Automatic
	Count of Vertical Segments Measured	1
	Count Time	3600 sec Real time
Matrix Density Correction	Describe how the matrix density correction was applied	The matrix density correction results from the numerical efficiency calculation in the modelling program WinnerTrack.
Modelling and Fitting	Modelling	InterWinner Version 7.10.3063 with WinnerTrack
Software (if used)	Fitting	InterWinner Version 7.10.3063
Detector Calibration	Describe how the detector was calibrated and how this was applied	Numerical Calibration based on point-source measured data.
Additional Information		

Laboratory 9.5		
	Manufacturer	Ametek / Ortec
	Detector Type	p-type germanium-detector, model number IDM-200-V, relative efficiency 52.86 % (measured)
	Crystal Type	Coaxial, diameter 85 mm, length 32 mm
	Number of Detectors	one
	Orientation/Arrangement of	Detector is aligned with the vertical and radial middle of
	Detectors	the rotating drum.
Detector	Window Material	Aluminium
	Collimated (if yes, provide details)	Pipe-collimators with the length 2, 4 and 6 inch available. Due to high distance the drum is completely in the view field of the detector (open geometry).
	Detector Shielding	At the side 1.8 mm Cu, 1 mm Zn, 10 mm Steel and 27.5 mm Pb (pipe-collimator from in- to outside)
	Acquisition Software (Type and Version)	GammaVision Version 8.1.0
Scanning Method	Distance from Drum (m)	1 m
	Rotation (Automatic/Manual)	Manual (sum spectrum consists of four single spectra, each for one quarter of the drum surface)
	Count of Vertical Segments Measured	1
	Count Time	3600 sec Real time
Matrix Density Correction	Describe how the matrix density correction was applied	The matrix density correction results from the numerical efficiency calculation in the modelling program WinnerTrack.
Modelling and Fitting	Modelling	InterWinner Version 7.10.3063 with WinnerTrack
Software (if used)	Fitting	InterWinner Version 7.10.3063

Detector Calibration	Describe how the detector was calibrated and how this was applied	Numerical Calibration based on point-source measured data.
Additional Information		

Laboratory 11		
	Manufacturer	Canberra
	Detector Type	Broad energy germanium
	Crystal Type	n-type
	Number of Detectors	1
Detector	Orientation/Arrangement of Detectors	Drum was set on a rotating table, measured from 4 different angles and also while rotating at distances 0,5 m, 1 m and 2 m.
	Window Material	Carbon fibre
	Collimated (if yes, provide details)	No
	Detector Shielding	No
	Acquisition Software (Type and Version)	Genie2000, Canberra, V3.4.1
	Distance from Drum (m)	0.5, 1 and 2
	Rotation (Automatic/Manual)	Yes, automatic
Scanning Method	Count of Vertical Segments Measured	1
	Count Time	4000 s and 86000 s
Matrix Density Correction	Describe how the matrix density correction was applied	Geometry Composer
Modelling and Fitting	Modelling	Geometry Composer
Software (if used)	Fitting	Geometry Composer

Detector Calibration	Describe how the detector was calibrated and how this was applied	ISOCS
Additional Information		Source localisation was performed by highly sensitive Nal detector, outer surfaces were scanned at 5 different heights and at 8 different angles. Based on the measurements the source was assumed in the centre at 30 cm height from the bottom of the drum.

Laboratory 12		
	Manufacturer	Canberra (now Mirion)
	Detector Type	HpGE BEGE ISOCS
	Crystal Type	BE3830
	Number of Detectors	1
Detector	Orientation/Arrangement of Detectors	In front of the drum, middle height
	Window Material	Alu
	Collimated (if yes, provide details)	Lead, 5cm, 2x45°
	Detector Shielding	Lead, 5cm
	Acquisition Software (Type and Version)	Genie2000 V3.4.1
Scanning Method Count Measu	Distance from Drum (m)	0,6
	Rotation (Automatic/Manual)	Auto
	Count of Vertical Segments	
	Measured	-
	Count Time	-

Matrix Density	Describe how the matrix density	Geometry Composer
Correction	correction was applied	Geometry Composer
Modelling and Fitting	Modelling	Geometry Composer
Software (if used)	Fitting	-
	Describe how the detector was	
Detector Calibration	calibrated and how this was	ISOCS
	applied	
Additional Information		

Appendix B – Invitation to register interest sent to participants of previous exercises.



National Physical Laboratory Hampton Road Teddington Middlesex United Kingdom TW11 0LW Switchboard 020 8977 3222

www.npl.co.uk/contact

Version 1

8th February 2021

Dear Colleague,

NPL NUCLEAR INDUSTRY PROFICIENCY TEST EXERCISE 2021

NPL is planning to run its 8th drum-based bulk-waste proficiency test exercise to enable laboratories worldwide involved in decommissioning and site clearance to test their bulk-waste gamma measurement procedures. As in 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 likely contain 240 plastic bottles filled with vermiculite. A subset of the bottles will contain vials of resin spiked with gamma emitting radionuclides. The overall activity concentration in the drum will likely be in the range $3-30~{\rm Bg~g^{-1}}$. The drum for the last exercise contained $^{60}{\rm Co}$, $^{137}{\rm Cs}$ and $^{241}{\rm Am}$.

To ensure the ongoing relevance of this exercise, <u>NPL welcome any suggestions</u> regarding the drum contents such as 'mock waste' materials and radionuclides.

It is forseen that the drum will be available for measurement between June and September (inclusive) 2021 and participants will be asked to report the activity concentrations of the individual radionuclides in October. A short exercise report will be published in January 2022. All results will be coded and treated as confidential.

In previous exercises information linking specific measurement techniques to the corresponding result has been limited. For the upcoming exercise it is proposed that participants will have the option of including details on the measurement techniques used for participants to review. Before implementing this NPL is requesting feedback on whether this option would be of interest.

The fees are likely to be similar to previous exercises:

- Participation: Approximately £2500-3000
- Delivery: To be advised

NPL will arrange for a courier to deliver and pick-up on the agreed dates. It is planned that the drum would circulate Europe first in June-July and the UK after (August-September) but this is also dependent on the number and location of participating laboratories.

Appendix C - A follow-up invitation letter



National Physical Laboratory Hampton Road Teddington Middlesex United Kingdom TW11 0LW Switchboard 020 8977 3222

www.npl.co.uk/contact

Version 1

22nd April 2021

Dear Colleague,

NPL NUCLEAR INDUSTRY PROFICIENCY TEST EXERCISE 2021

Thank you for expressing an interest in the 8th drum-based bulk-waste Proficiency Test Exercise run by NPL. As in previous exercises, the purpose is to provide the user community with a voluntary, independent and confidential test of their bulk waste measurement procedures. We have taken your feedback into consideration and will deliver the exercise following the brief below.

In brief: NPL will prepare a single 'mock waste' sample in a 200-litre steel drum. The drum will contain 240 plastic bottles filled with vermiculite. A subset of the bottles will contain vials of resin spiked with 60 Co, 137 Cs and 241 Am. The overall activity concentration in the drum will be in the range 3-30 Bg g^{-1} .

The contents and mock-waste will be comparable to the 2019 exercise, with an inhomogeneous distribution and the same radionuclides at similar activity concentrations. Each participant will have 3 working days (excluding the arrival and despatch days) to analyse the drum. It is for this reason that the measurement period will be between July and November (inclusive) 2021. This year the measurement period has been split into for UK and non-UK participants. Following the measurement period participants will be asked to report the activity concentrations of the individual radionuclides. A short exercise report will be published by March 2022. As with previous exercises results will be coded and treated as confidential, but this year there will be an option on the techniques form to opt into a comparison table. This table will still be anonymised but will detail the measurement scheme aligned with the corresponding laboratory number. Another notable change is the inclusion of a workshop scheduled for April 2022. We encourage participants to attend this workshop and see it as an opportunity for participants to share their experiences, reflect on the exercise and provide feedback to influence future exercises. We envisage that this workshop will be hosted both online and at NPL to ensure it is inclusive as possible.

NPL Management Ltd is a company registered in England and Wales No. 2937881. Registered Office: NPL Management Ltd, Hampton Road, Teddington. Middlesex, United Kingdom TW11 OLW

Appendix D - Reporting Form

NOTES:

- 1] Uncertainties should be quoted as the absolute uncertainty at k = 1.
- 2] Please report your measurement techniques separately on the 'Techniques Form', available from the NPL webpage.
- 3] The reference time for all reported results is 2021-06-01 12:00 UTC
- 4] The reporting units for Drum samples are Bq/g.

WARNING:

Do not edit or modify this reporting form as this may result in the submitted results not being used in the final data evaluations.

LAB CODE:		
Source Type	Drum	
Radionuclide	Activity	Uncertainty
⁶⁰ Co	0.0000 Bq/g	± 0.0000 Bq/g
137Cs	0.0000 Bq/g	± 0.0000 Bq/g
²⁴¹ Am	0.0000 Bq/g	± 0.0000 Bq/g

Page 1 of 3

NPL Nuclear Industry PTE 2021 - Techniques Form

Participating Laboratory	
Contact Person	
Option to opt into information sharing:	I confirm that information shared in this form may be used as part of a comparison table in the final report. I am aware that that the results will still be anonymised, but details provided below could lead to laboratory identification:
	[INSERT NAME] on behalf of [LABORATORY NAME]

Please provide details of the following, additional comments may be added under "additional information":

Detector	Manufacturer	
	Detector Type	
	Crystal Type	
	Number of Detectors	
	Orientation/Arrangement of Detectors	
	Window Material	
	Collimated (if yes, provide details)	
	Detector Shielding	

Appendix F - Information Sheet

Version 1

NPL NUCLEAR INDUSTRY PTE 2021 INFORMATION SHEET UPDATES TO SCHEDULE IN RED

Technical

As with the 2019 exercise, the 200 L drum contains 240 HDPE bottles (in 5 'layers' of 48), each filled with vermiculite. A number of these bottles also contain a plastic vial containing ion-exchange resin. All of these 'resin vials' have been spiked with standardised radioactive solution.

The overall activity concentration of the drum (i.e. activity/total mass of drum contents) lies in the range of 3 - 30 Bg g⁻¹.

Nominal mass of empty drum = 19.55 kgMass of drum contents = $28.166 \pm 0.012 \text{ kg}$ (k = 1)

<u>Delivery</u>

The drum will be delivered to (and collected from) the participants on agreed dates as per the schedule below. The drum will be an Excepted Package. Participants must have arrangements in place for moving the drum off, from and onto the courier's van and must ensure that the drum bears the address label provided by NPL before it is returned to the courier.

Data reporting

Forms for data and method reporting have been sent out separately to participant technical contacts. All completed forms must be returned by the 'first deadline' which has been updated in the schedule below.

NPL will then disclose the locations of the 'resin vials' and invite participants to submit *additional* results (by the second deadline) should they wish. Note that 'first deadline' *results* and any *corrections* must be submitted prior to the disclosure of the 'resin vials' location.

Appendix G – Participation List

Note that this is an alphabetical list and does not reflect the laboratory numbers assigned to participating laboratories.

A Waterfall **AWE**

Aldermaston Reading

Berkshire RG7 4PR

UK

A Leskinen

VTT Technical Research Centre of

Finland Ltd. Otakaari 3 FI-02150 Espoo

Finland

B Wellens

Nuclear Engineering Seibersdorf GmbH,

Forschungszentrum, 2444 Seibersdorf.

Austria

C Nobs

United Kingdom Atomic Energy Authority,

Culham Science Centre,

Abingdon, **OX14 3DB**

UK

C Binnerslev

Mirion Technologies (Canberra UK) Ltd

207A Cavendish Place

Birchwood Park Warrington WA3 6WV

UK

E Mauro Nucleco S.p.A. Via Anguillarese 301 S M di Galeria (Rm)

00123 Rome

Italy

F Rodari

L.B. Servizi per Aziende s.r.l.

81, 00135 Rome

Italy

F Schwabenland

Kerntechnische Entsorgung Karlsruhe

GmbH,

Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen

Germany

H Beddow Nuvia Ltd The Library 8th Street

Harwell Science and Innovation Campus

Didcot OX11 0RL

UK

J Mason

A.N. Technology Limited

Unit 5 & 6 Thames Park Lester Way Wallingford

Oxfordshire OX10 9TA

UK

K Hostikka

Fortum Power and Heat Oy

Loviisa Power Plant

PL 751

00026 Basware

Finland

L Hayward Magnox Ltd.

Oldbury Technical Centre,

Oldbury Naite, Thornbury, Gloucestershire BS35 1RQ, UK

M Giacomelli

ZVD Zavod za varstvo pri delu d.o.o.,

Pot k izviru 6

1260 Ljubljana - Polje

Slovenia

S Fleck

VKTA Radiation Protection, Analytics,

Disposal

Bautzner Landstraße 400

Gebäude 885 01328 Dresden

NPL REPORT IR 60

Germany

T Steinhardt Jülicher Entsorgungsgesellschaft für Nuklearanlagen mbh (JEN) Welhelm-John-Straβe 52428 Jülich Germany

T Dieudonne IRE-ELIT Avenue de l'Esperance 1 B-6220 Fleurus Belgium