Intercomparison exercise in analysis of DTM in decommissioning waste

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January 2020

Abstract

An intercomparison exercise was carried out for difficult to measure ra-
dionuclides in activated reactor pressure vessel steel samples. The results
were analysed accoring to ISO 13528 standard. The performance as-
sessment was carried out using z score. Report includes an overview of
the radioanalytical procedures, preliminary and final results, and prefor-
mance assessments.

Key words

Decommissioning, Difficult-to-measure radionuclides, intercomparison

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Final Report from the NKS-B DTM-Decom activity
(Contract: AFT/B(19)3)

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Table of contents

1. Introduction

Increasing number of research and power reactors are being shut down and decommissioned within the Nordic countries. Currently, operators of research and power reactors are faced with a great challenge in handling of the decommissioning waste. Handling of the waste includes both characterisation and categorisation of the waste in order to separate exempted waste from radioactive waste. Handling of especially legacy waste has underlined the importance of proper characterisation and categorisation of the waste prior to formation of waste packages.

Both research and power reactors will have at least activated steel and concrete as decommissioning waste. Additionally, research reactors will have other materials, such as graphite and aluminium, in their inventory. Non-destructive techniques are available for determination of gamma emitting radionuclides whereas destructive analysis is needed for alpha and beta emitting, namely Difficult-To-Measure (DTM), radionuclides. The destructive analysis of DTM require radiochemical separation and purification methods, which often have several steps and depending on the material, the interfering radionuclides may cause significant bias in the results. For example, analysis of Ni-63 and Fe-55 in activated steel are heavily interfered with Co-60. Therefore, validation of the measurement method is compulsory in order to produce reliable results. In analytical chemistry, reference materials are used for the method validation. Radioactive reference materials are also available for analysis of radionuclides in environmental samples whereas there are no commercially available reference materials for DTM analysis in decommissioning waste. In cases, where reference materials are not available, one way of validation of the radiochemical method is to use spiked samples. Spiked samples are often valid for testing, but it has been shown for example in a previous NKS intercalibration [1] that spiked samples can behave very differently to real samples. Additionally, analysis of contaminated and activated wastes can be very different due to speciation of the DTMs.

DTM-Decom project was developed for validation of radiochemical analysis of DTM in activated steel via an intercalibration exercise. In total, seven laboratories from the Nordic countries participated and additionally one laboratory from a non-Nordic country joined self-funded.

2. DTMs of interest in activated steel

A survey was carried out on capabilities of the DTM analysis of the participant laboratories. After discussions based on the Table 1, the main DTMs of interest in the DTM-Decom intercomparison exercise were chosen to be Fe-55 and Ni-63. C-14 and Ni-59 analysis were optional and additionally, some laboratories also analysed Co-60.

Table 1. Capabilities of the participating laboratories on analysis of DTM in steel

Affiliation	Readiness of DTM analysis	DTMs of interest in steel
VTT	National project on C-14, Fe-55 and Ni-63 analysis in 2018, finalisation of the project spring 2019	C-14, Fe-55, Ni-63
IFE Kjeller	The method is under development	Fe-55 and Ni-63
DTU	Methods for the determination of DTM in steel have been routinely used for many years. DTU has series of materials for the analysis of steel samples based on the potential requirement in the decommissioning work. In addition to H-3, Fe-55, Ni-63, DTU also cover many others, i.e. Cl-36, Mo-93, Nb-94, Tc-99, Cs-135, I-129, and actinides. All these methods are ready for use.	Fe-55, Ni-63
CEA	Routine analysis for Fe-55, Ni-63, Ni-59 in steels but analysis under development for C-14 in steels. CEA has routine analysis for liquids and concretes.	Fe-55, Ni-63, Ni-59, C-14
HU	One functional method for determining Fe-55 and Ni-63 from steel samples containing a lot of 60Co was found in a master thesis at UH during the national project with VTT in 2018. That method will be used now, possibly with some adjustments.	Fe-55, Ni-63 (Ni-59, if our x-ray spectrometer is in operation)
Cyclife	Routine analysis for H-3 in steel samples. C-14, Cl-36, Fe-55, Ni-63 and I-129 analysis in steel under development.	C-14, Fe-55, Ni-63
Fortum	Routine analysis for Ni-63 and Fe-55 in spent resins, evaporation waste and swipe samples. The method has not been tested on other sample matrices. C-14 is measured in ventilation stack air.	Focus on Ni-63 and Fe-55, but C-14 would also be beneficial

3. Preparation, homogeneity and sending of the samples

3.1 Sample selection and preparation

One reactor pressure vessel steel bar (10 cm × 1 cm × 1 cm) was cut to approximately 0.2 mm slices using an electric discharge machine (EDM). The bar had received known amount of neutron radiation and the cooling time was also sufficiently long to lower the dose rate of the bar. Previous studies on a similar bar had shown that the bar should contain significant amounts of DTMs of interest [Leskinen et al. 2019]. After cutting of the slices, their oxidation layer was removed using an oxide removal solution (HCl:H_2O:hexamethyleneteramine) and dipped into ethanol. The samples were let to air dry. Each sample was given an identification number, placed in small bags and weight (Figure 1). Table 2 shows the participant's laboratory number (random order) and the sample numbers.

Figure 1. Example of a studied sample in a plastic bag.

Table 2. Numbers of the participating laboratories and the samples received.

Laboratory	Sample #
1	1-2
2	3-5
3	6-8
4	9-11
5	12-13
6	14-15
7	16-18
8	19-20

3.2 Homogeneity of the samples

Homogeneity studies were carried out using gamma spectrometry. Each sample was placed on an HPGe detector in an ISOCS system using a sample holder at 3 cm above the detector in order to reduce coincidence. Constant measurement geometry was ensured by placing the sample in a consistent position between petri dish covers and by placing a weight (water) on top (Figure 2). The results showed that the 20 samples distributed in the intercomparison exercise had 1.8% RSD for Co-60 (Figure 3). Therefore, the samples were considered to be homogenous.
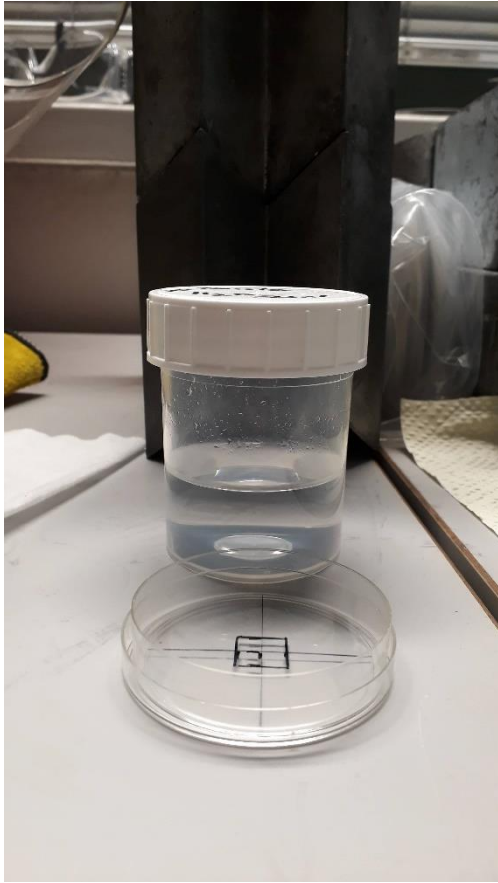
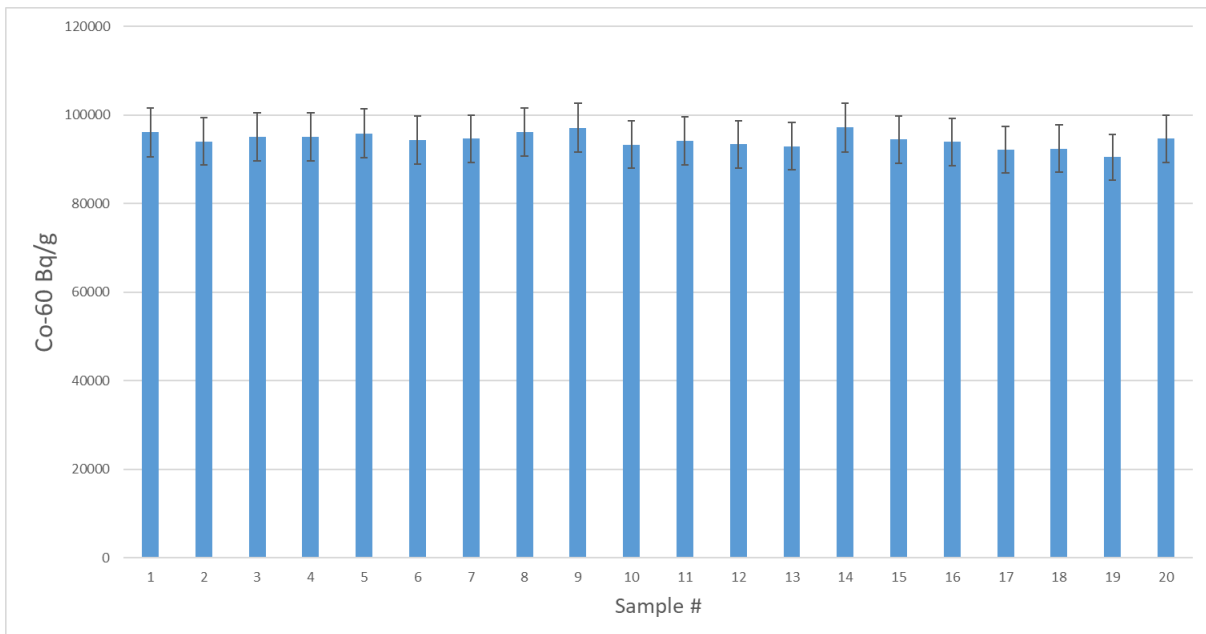**Figure 2.** Sample holder for gamma measurements

Figure 3. Co-60 activity concentrations in the 20 RPV steel samples measured at VTT

3.3 Sending of the samples

2-3 samples were packed inside a small lead shield, which was placed inside of a container and a cardboard box. The packages were sent to the participants as exempted packages (UN2910 shipment).

4. Radiochemical analysis

Participants carried out the radiochemical analyses based on either internal or published procedures [Brennetot, 2017; Gautier, 2015; Hazan & Korkisch, 1965; Hou, 2005, 2018; Hou et al., 2005a, 2005b, 2007; Leskinen et al., 2019]. However, all the procedures had similar components as discussed in sections 4.1-4.5, which summarise the main radioanalytical steps, namely decomposition, separation, purification and measurements.

4.1 Decomposition of the solid sample

In radiochemical analysis of Fe-55 and Ni-63, the decomposition of the solid matrix was carried out using different acid mixtures in a decanter on a hot plate (conc HNO_3:HCl with 2:1 ratio or aqua regia), in a round bottom flask on a heating mantle (5M H_2SO_4 + conc HNO_3 or H_2O:HNO_3:HCl + $HClO_4$) or in a microwave (HNO_3:HF:HCl). One laboratory decomposed the sample without heating in conc HNO3:HCl (3:1) and let the mixture react for 2 days. In this case, small black particles remained in the solution. Some laboratories added carriers and hold back carriers at the decomposition step (eg. Ni, Ag, Cs, Eu, Co, Mn, Sb, Sr, Zr).

In radiochemical analysis of C-14, the decomposition of the solid matrix was carried out using a round bottom flask on a heating mantle (5M H_2SO_4 + conc HNO_3 or H_2O:HNO_3:HCl + $HClO_4$) with and without a Na_2CO_3 carbon carrier. Equipment dedicated for volatile DTM analysis were an Oxidizer (combusted with cellulous pad and cellulous powder, trapping into CarboSorb) and a Pyrolyser (combusted for 10 hours with starting temperature of 50 °C rising up to 900 °C with Pt-alumina catalyst, addition of oxygen towards the end of the program and CO_2 trapped into CarbonTrap). The samples were measured using liquid scintillation counting (LSC) without further separation or purification.

4.2 Separation of DTM

Some laboratories added carriers and hold back carriers at the separation step. Additionally, some laboratories analysed an aliquot of the acid digested solution for the Fe-55 and Ni-63 analysis while some laboratories analysed the whole solution.

Separation of Fe and Ni species was carried out most often using hydroxide precipitation followed by an anion exchange resin. Majority of laboratories added NaOH or NH_3 to precipitate Ni and Fe at pH 8-9 followed by separation of Fe and Ni using AG 1x4 anion exchange resin[1]. One laboratory used Dowex 1x4 anion exchange resin[2] with acetone and 6 M HCl (9:1) mixture with low flowrate in order to remove Co effectively from the solution. One laboratory took first a 0.3 ml aliquot for Fe analysis and separated Fe from Ni using hydroxide precipitation at pH 4-5 in which majority of Fe is precipitated while majority of Ni remains in solution.

One laboratory removed first silver from the solution using evaporation of the solution to dryness, dissolving the residue to 0.1M HCl and then removal of silver via AgCl precipitation. Separation of Fe and Ni was carried out using TRU resin[3].

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[1] AG 1x4 anion exchange resin by Bio-Rad
[2] DOWEX 1x4 anion exchange resin by Dow Chemical Company
[3] TRU resin by Eichrom Technologies

4.3 Purification of DTM

Fe-55 fractions in 2 M HNO_3 after TRU resin separation were not further purified or evaporated but aliquots were taken from the eluate for the LSC measurements. One laboratory used a second anion exchange resin (AG 1x4) treatment for purification of Fe-55 fraction prior to evaporation whereas rest of the laboratories evaporated the fraction to dryness and dissolved the residue in 1 M H_3PO_4 or 0.5M HNO_3. Aliquots were taken for the LSC and yield measurements.

Ni-63 fractions were purified especially from Co-60 using Ni-resin[4] once or twice. One laboratory took an aliquot for the LSC measurement directly from the eluate and one laboratory diluted the eluate first. All the other laboratories evaporated the eluate first to 0.2 to 2 ml and then aliquots were taken for the LSC and yield measurements.

One of the laboratories reported inefficient purification of Ni-63 from Co-60. The upper spectrum of Figure 4 shows how the Ni-63 signal is disturbed by Co-60. However, further purification of the sample using a Ni-resin removed majority of Co-60 from the sample (lower picture of Figure 4). The remaining Co-60 interference was removed from the Ni-63 results by subtracting the Co-60 peak from the Ni-63.
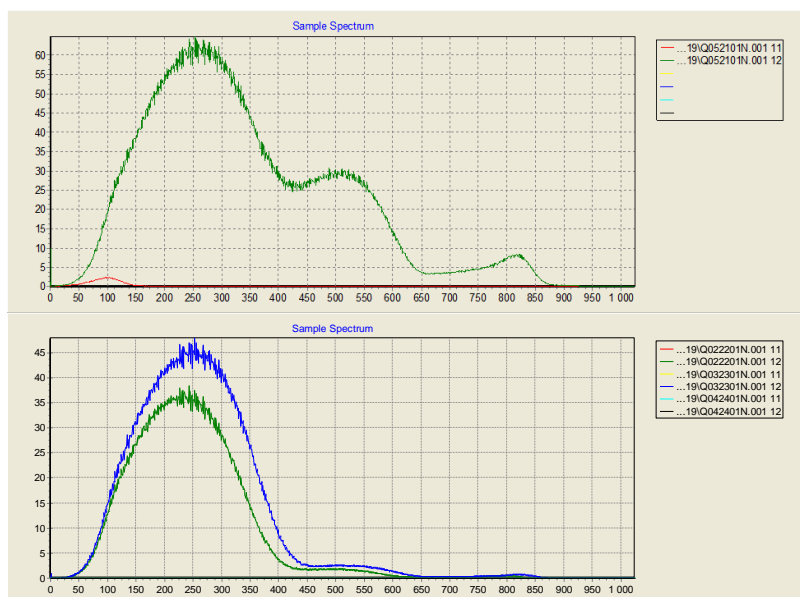
Figure 4. Example LSC spectrum of Ni-63 with significant amount of Co-60 (upper picture) and almost without Co-60 after a second Ni resin purification (lower picture)

4.4 LSC and yield measurements

1-8 ml of C-14, Fe-55 and Ni-63 fractions were dissolved into 10-19 ml liquid scintillation cocktail (Ultima Gold LLT, Gold Star, Carbon Count or HiSafe 3) and the measurements were carried out using Liquid Scintillation Counter (TriCarb, Quantulus, Wallac, Aloka or Hidex). Efficiency corrections were carried out using standard solutions or TDCR technique.

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[4] Ni-resin by Eichrom Technologies

Yield measurement for Fe-55 and Ni-63 fractions were carried out using ICP-MS[5], ICP-OES[6], MP-AES[7], or UV-VIS[8]. One laboratory reported using standard addition technique for Ni-63 analysis and estimated yield for Fe-55. C-14 yields were determined using standard solutions.

All analysis results were calculated to predefined reference date.

4.5 Analysis of Co-60

Some laboratories analysed also Co-60, even though it is not considered to be a DTM radionuclide. Some laboratories carried out the measurement in the solid form and some dissolved the sample first. All laboratories used HPGe detectors for the gamma measurements. Efficiency calibrations were carried out using calibration solutions, ISOCS or LabSOCS.

5. Methodology

Statistical analysis of the results was carried out using the ISO 13528 standard, which states that a variety of scoring strategies are available. Most often, however, participant's deviation from an assigned value is compared. The activated steel studied was not a reference material and thus a robust statistical method was utilised for development of assigned value based on the participant's results. Robust mean and robust standard deviation were calculated using Algorithm A, which transforms the original data by a process called winsorisation. This method provides alternative estimators of mean and standard deviation. Algorithm A is robust for outliers, when the expected proportion of outliers is less than 20%.

Performance assessment was carried out using z score. Z score (Eq. 1) is a recommended method in cases when participant's results are used for the calculation of assigned value. In cases where the robust standard deviation is large, another value for σ_{pt} can be used so that the results that are not fit for purpose will receive an action signal. The intercomparison exercise organiser can decide the value. In cases where the robust standard deviation was large ($1\sigma > 20\%$), the uncertainty of the assigned value (Eq. 2) was used as σ_{pt}. The analysis results with z score were marked as acceptable when $z \leq 2.0$, a warning signal was given for results with $2.0 < z < 3.0$, and results were unacceptable for $z \geq 3.0$.

$$z_i = (x_i - x_{pt}) / \sigma_{pt} \tag{1}$$

Where
x_{pt} = the assigned value
σ_{pt} = standard deviation for the proficiency assessment

$$u(x_{pt}) = 1.25 \times s^* / p^{0,5} \tag{2}$$

Where
s^* = robust standard deviation of the results
p = number of samples

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[5] Inductively Coupled Plasma Mass spectrometer
[6] Inductively Coupled Plasma Optical Emission Spectometer
[7] Microwave Plasma Atomic Emission Spectrometer
[8] Ultraviolet-visible spectrometer

6. Statistical analysis of the results

The methodology in section 5 was first applied to the preliminary results submitted prior to the final project meeting. During the final meeting, discussions were carried out for the differences in the results and possible corrective actions were suggested, such as correction of LSC efficiency curves. Clear blunders, such as calculation errors, were also corrected. Additionally, results which were calculated from replicates were instructed to be submitted as separate analysis results. In this section, the preliminary and final results are presented.

6.1 Fe-55 results

In total, 13 Fe-55 preliminary analysis results were submitted. The preliminary assigned value for the Fe-55 activity concentration was calculated to be 82.0 ± 29.4 kBq/g (2σ). Later, 21 final analysis results were submitted for the statistical analysis. The final assigned value for the Fe-55 activity concentration was calculated to be 73.7 ± 12.2 kBq/g (2σ). The preliminary and final Fe-55 activity concentration results and assigned values with 2σ uncertainties are shown in Figure 5. As the results show, the final assigned value was 10% lower compared to the preliminary value. Additionally, the final results showed less variation in the results since the uncertainty (2σ) had lowered by 60% compared to the preliminary uncertainty.

Corrective actions were subjected to the preliminary Fe-55 results for samples 1-2 (corrected quench curve), sample 3 (presentation of the replicate results), sample 14 (calculation correction from Bq to Bq/g and presentation of replica results) and 16-18 (correction of LSC efficiency using a CoreF function). Final results for samples 8 and 19-20 were new entries.

Figure 5. Fe-55 results (above preliminary, below final) in activated steel samples and assigned value with 2σ uncertainties.

The robust standard deviation for the preliminary and assigned values were large (1σ > 20%) and therefore uncertainties of the assigned values were used in the z score calculations. The z score results are shown in Table 3. The results show that in the preliminary results, 7 out of 13 entries were in acceptable range, 3 in warning signal range and 3 in unacceptable range whereas the final results had 12 out of 21 results in acceptable range, 2 in warning signal range and 7 in unacceptable range.

Table 3. Sample numbers and z scores of Fe-55 results. Acceptable results in green, warning signal in orange and unacceptable in red.

Sample #	Preliminary z score	Final z score
1	3,7	1,6
2	2,8	0,4
3	0,0	2,5
3	-	0,3
7	1,0	1,2
8	-	0,6
8	-	1,5
9	0,7	0,3
10	0,5	0,2
11	0,7	0,4
12	0,6	0,2
14	4,4	6,8
14	4,5	8,4
14	-	4,8
14	-	3,8
16	2,6	0,5
17	1,1	2,5
18	2,3	0,9
19	-	5,7
20	-	8,3
20	-	8,7

6.2 Ni-63 results

In total, 10 preliminary Ni-63 results were submitted. The preliminary assigned value for the Ni-63 activity concentration was calculated to be 87.1 ± 22.4 kBq/g (2σ). Later, 15 final analysis results were submitted for the statistical analysis. One result entry was rejected (sample 20) from the calculation of the final assigned value, since it was a clear outlier. The final assigned value for the Ni-63 activity concentration was calculated to be 86.4 ± 17.7 kBq/g (2σ). The preliminary and final Ni-63 activity concentration results and assigned values with 2σ uncertainties are shown in Figure 6. As the results show, the final assigned value and the uncertainty (2σ) were almost the same compared to the corresponding preliminary values (99% and 79%, respectively).

Corrective actions were subjected to the preliminary Ni-63 results for samples 3 (presentation of the replicate results) and 16-18 (correction of LSC efficiency using a CoreF function). Final results for samples 1-2 (removal of Co-60 using a second Ni resin, see Figure 3) and 19-20 were new entries.
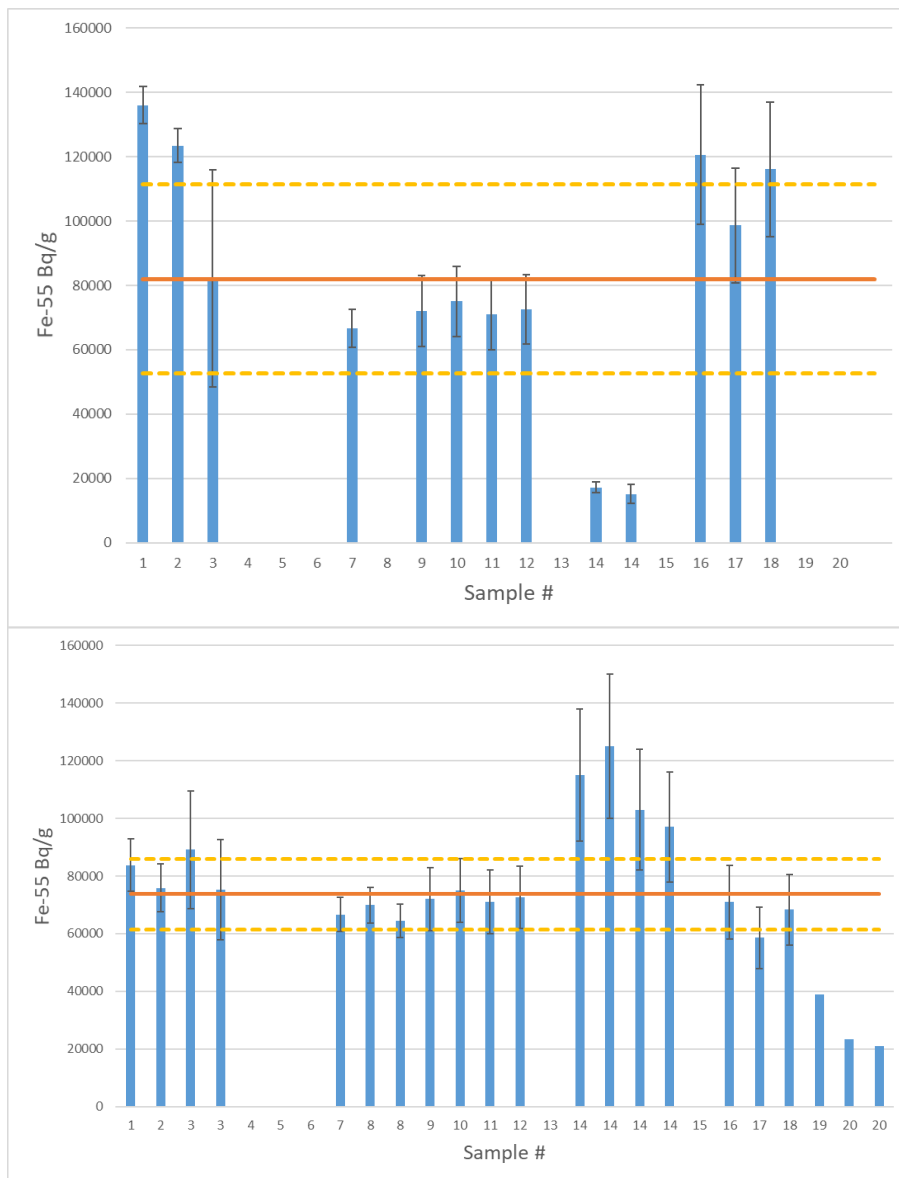
Figure 6. Ni-63 results (above preliminary, below final) in activated steel samples and assigned value with 2σ uncertainties.

The robust standard deviations for the preliminary and final assigned values were large (1σ > 20%) and therefore uncertainties of the assigned values were used in the z score calculations. The z score results are shown in Table 4. The results show that in the preliminary results, 7 out of 10 entries were in acceptable range, 0 in warning signal range and 3 in unacceptable range whereas the final results had 9 out of 15 results in acceptable range, 0 in warning signal range and 6 in unacceptable range.

Table 4. Sample numbers and z scores of Ni-63 results. Acceptable results in green, warning signal in orange and unacceptable in red.

Sample #	Preliminary z score	Final z score
1	-	0,8
2	-	0,2
3	0,6	0,6
3	-	1,1
7	0,1	0,1
9	3,6	4,5
11	5,2	6,5
12	0,2	0,1
14	1,5	1,9
14	1,5	1,5
16	0,6	0,9
17	3,1	4,4
18	1,9	3,2
19	-	3,4
20	-	9,6

6.3 C-14 results

In total, 6 preliminary results were submitted. The assigned value for the C-14 activity concentration was calculated to be 489 ± 328 Bq/g (2σ). Later, 7 final analysis results were submitted for the statistical analysis. The final assigned value for the C-14 activity concentration was calculated to be 541 ± 310 Bq/g (2σ). The preliminary and final C-14 activity concentration results and assigned values with 2σ uncertainties are shown in Figure 7. As the results show, the final assigned value increased 10% and uncertainty decreased 6% compared to the corresponding preliminary values.

Corrective actions were subjected to the preliminary C-14 results for samples 16-18 (correction of LSC efficiency using a CoreF function). Final result for sample 4 was a new entry.
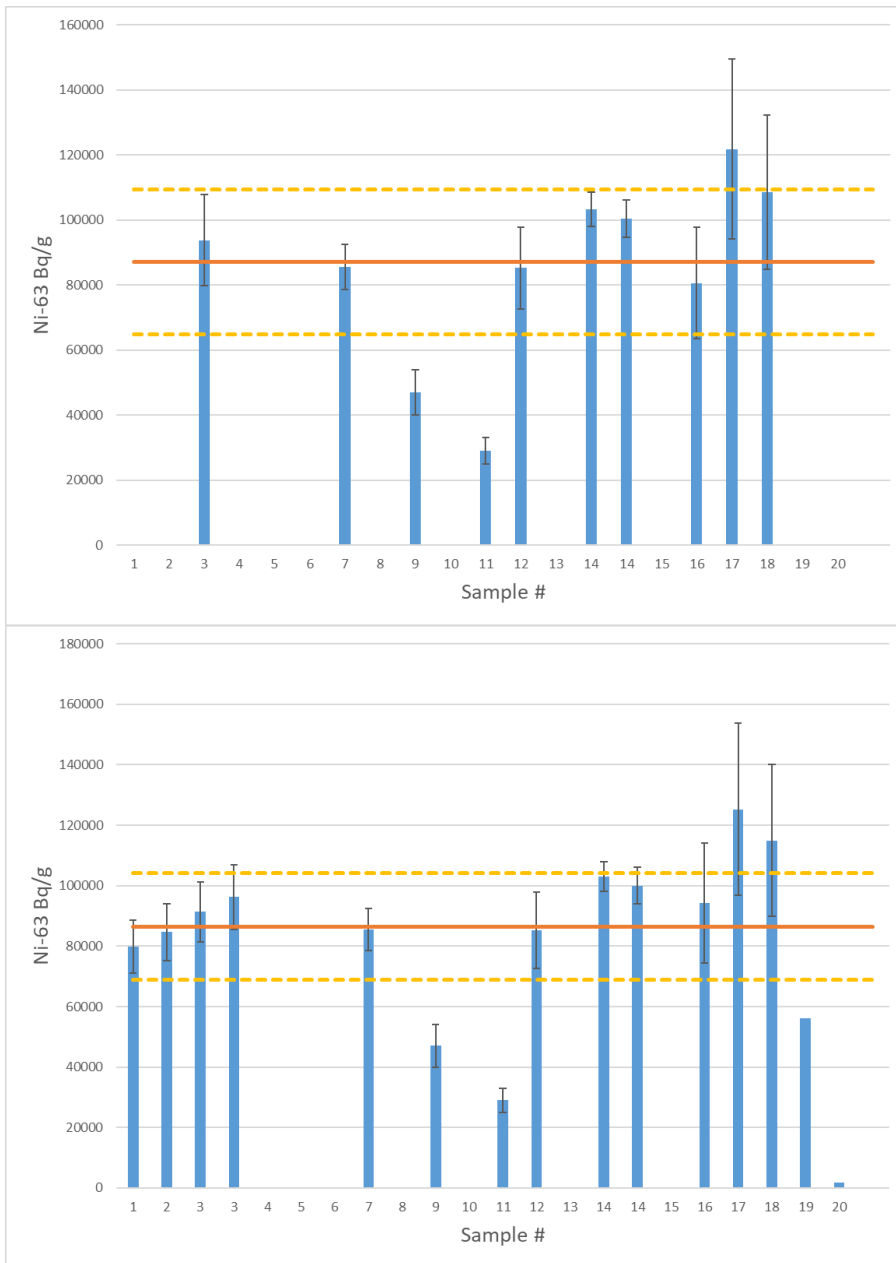
Figure 7. C-14 results (above preliminary, below final) in activated steel samples and assigned value with 2σ uncertainties.

The robust standard deviations for the preliminary and final assigned values were large (1σ > 20%) and therefore uncertainties of the assigned values were used in the z score calculations. The z score results are shown in Table 5. The results show that in the preliminary results, 6 out of 6 entries were in acceptable range whereas the final results had 3 out of 7 results in acceptable range and 4 in warning signal range.

Table 5. Sample numbers and z scores of C-14 results. Acceptable results in green, warning signal in orange and unacceptable in red.

Sample	Preliminary z score	Final z score
4	-	1,5
6	0,5	0,8
12	1,8	2,3
13	1,9	2,3
16	2,0	2,1
17	0,2	0,1
18	2,0	2,2

6.4 Co-60 results

In total, 15 Co-60 results were submitted. The assigned value for the Co-60 activity concentration was calculated to be 104.1 ± 10.6 kBq/g (2σ). Later, 17 final analysis results were submitted for the statistical analysis. The final assigned value for the Co-60 activity concentration was calculated to be 102.9 ± 9.6 kBq/g (2σ). The preliminary and final Co-60 activity concentration results and assigned values with 2σ uncertainties are shown in Figure 8. As the results show, the final assigned value and uncertainty decreased were almost the same compared to the corresponding preliminary values.

Corrective actions were subjected to the preliminary Co-60 results for sample 14 (measurements were done in solution). Final result for sample 19 was a new entry.

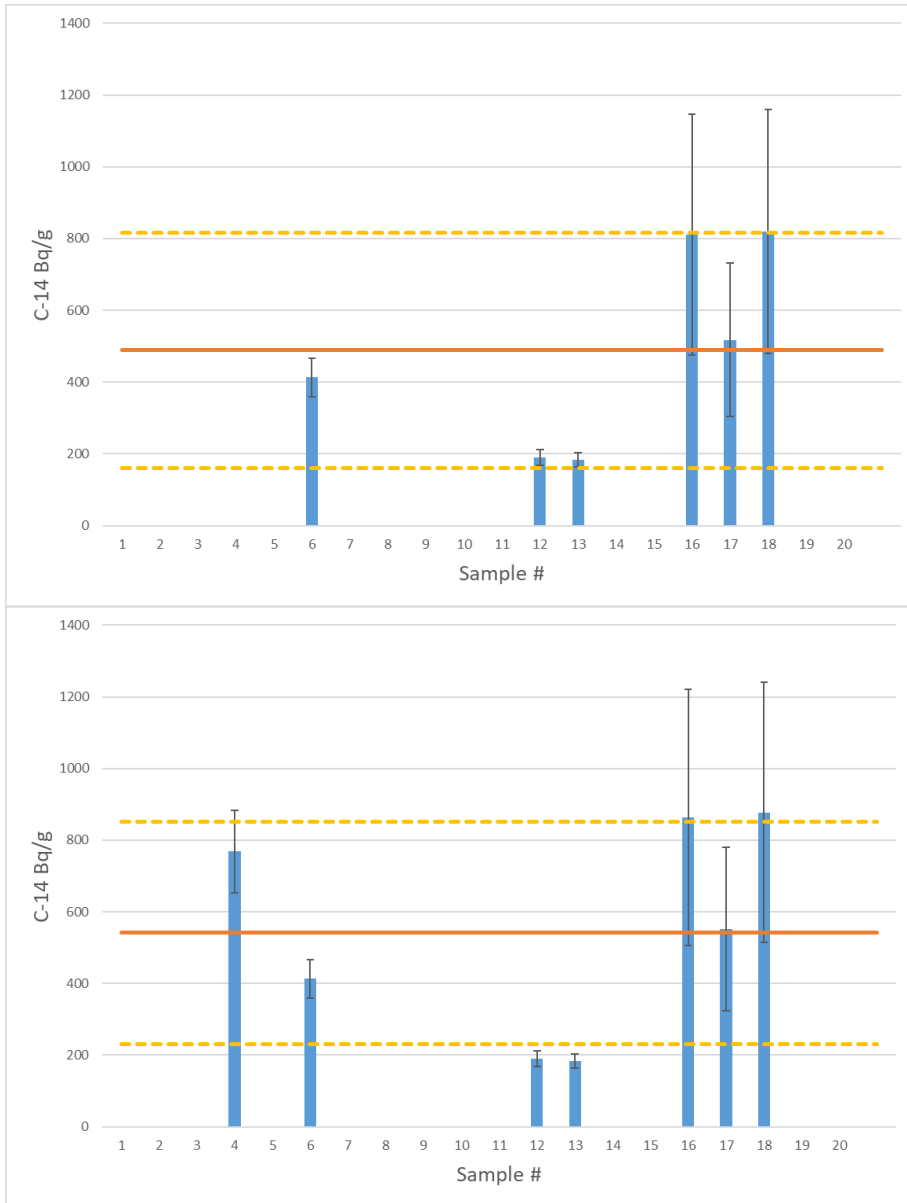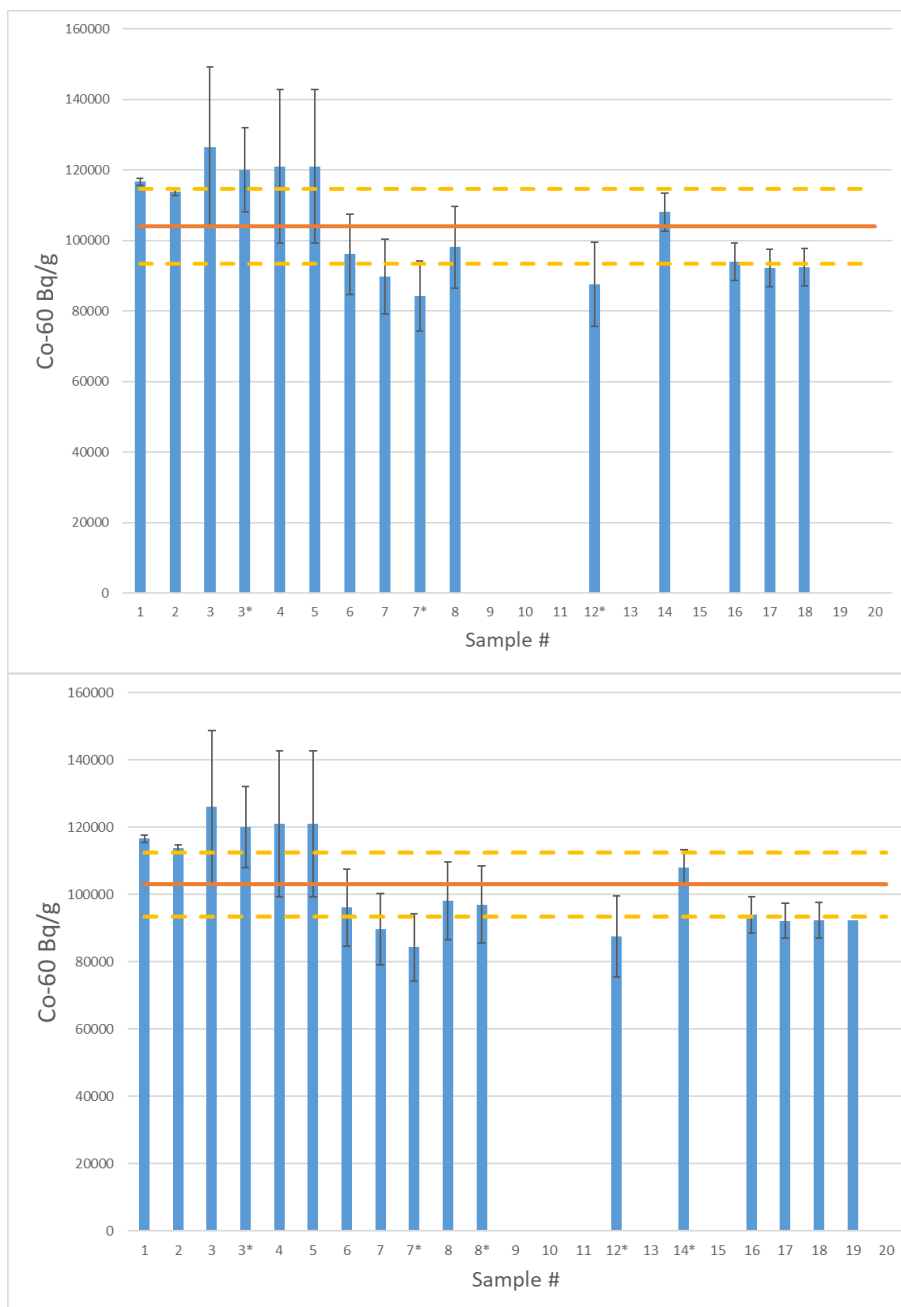**Figure 8.** Co-60 results (above preliminary, below final) in activated steel samples and assigned value with 2σ uncertainties. Samples measured in digested solutions are marked as *.

The robust standard deviations for the preliminary and final assigned values were not large (1σ < 20%) and therefore the standard deviations of the assigned values were used in the z score calculations. The z score results are shown in Table 6. The results show that in the preliminary and final results, all the entries were in the acceptable range.

Table 6. Sample numbers and z scores of Co-60 results. Acceptable results in green, warning signal in orange and unacceptable in red. Samples measured in digested solutions are marked as *.

Sample #	Preliminary z score	Final z score
1	0,8	0,9
2	0,6	0,7
3	1,4	1,5
3*	1,0	1,1
4	1,0	1,1
5	1,0	1,1
6	0,5	0,4
7	0,9	0,8
7*	1,2	1,2
8	0,4	0,3
8*	-	0,4
12*	1,0	1,0
14	0,2	-
14*	-	0,3
16	0,6	0,6
17	0,7	0,7
18	0,7	0,7
19	-	0,7

7. Conclusions

DTM analyses were carried out for 20 homogenous activated RPV steel samples. The results were analysed according to the ISO 13528 standard. The assigned value, to which results were compared, was calculated from participant's results. The performance was assessed using z score which indicates result's deviance from the assigned value.

The Fe-55 analysis was reported to be relatively easy without Co-60 interference. The z score results showed that 57% of the final Fe-55 results were in the acceptable range where as 10% were in the warning signal range. However, the uncertainties of both results in the warning signal range overlapped with the uncertainty of the assigned value. The final Fe-55 results in unacceptable range included replicas of sample 14 in which the yield was estimated to be 90%. This estimation of the yield may be the main reason for the deviance from the assigned value. Results of samples 19-20 also deviated significantly from the assigned value and it may be caused by problems in LSC analysis (inadequate quench curve) or/and in elemental analysis.

The Ni-63 analysis was reported to be significantly interfered by Co-60. However, diligent purification of Ni-63 fractions produced comparable results. The z score results showed that 60% of the final results were in the acceptable range whereas 40% were in unacceptable range. However, the uncertainties of 2 out of 6 results overlapped with uncertainty of the assigned value. Result entries of samples 19 and 20 did not include uncertainty assessment. Results of samples 9, 11 and 20 are clearly outside of the acceptable and warning signal range. Since the results are below the average trend, they are not interfered by Co-60 which would have increased the Ni-63 signal in LSC measurements. Deviance may be caused by inadequate LSC

quenching correction, elemental analysis of nickel (original concentration and yield) or yield and/or mass correction calculations.

The C-14 analysis had several major steps which affect the results significantly. Firstly, the sample had to be completely decomposed. Secondly, the oxidisation of carbon to CO_2 had to be complete. In this case, strong oxidising acids are needed to first decompose the sample and then to oxidise the carbon. Thirdly, efficient trapping of CO_2 is needed. The yield of trapping is thus often experimentally tested using a standard solution, for example, whereas behaviour of real sample may be different. Since there were only 7 entries for the final C-14 analysis, the uncertainty of the assigned value was relatively high. However, all the results were either in the acceptable or in warning signal range and more importantly within the same order of magnitude.

The Co-60 results were significantly more consistent compared to the DTMs, since the standard deviation (1σ) of the assigned value was below 20%. Also the uncertainty (2σ) of the final assigned value was only 9% and all the z score results were in the acceptable range. Additionally, Co-60 measurements in the solid and liquid form showed consistent results. The analysis results of Co-60 underline the difference between the analysis of difficult and easy to measure radionuclides.

As a conclusion, the DTM Decom project produced a very interesting set of results, which will be further analysed and discussed in another publication. In the upcoming publication, the results will be further studied by comparing the radioanalytically determined results with modelling results. Modelling results will provide a calculated assigned value based on the chemical composition of the original material, irradiation history and cooling time.

8. Acknowledgements

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Disclaimer

Title	Intercomparison exercise in analysis of DTM in decommissioning waste

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Abstract   An intercomparison exercise was carried out for difficult to measure
max. 2000 characters   radionuclides in activated reactor pressure vessel steel samples. The
                   results were analysed accoring to ISO 13528 standard. The
                   performance assessment was carried out using z score. Report
                   includes an overview of the radioanalytical procedures, preliminary
                   and final results, and preformance assessments.

Key words   Decommissioning,   Difficult-to-measure   radionuclides,
                   intercomparison