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Intercomparison exercise of ^{93}Mo and ^{59}Ni analysis in activated steel

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Abstract

An intercomparison exercise was carried out for Difficult-To- Measure (DTM) radionuclides in high activity steel samples. The steel sample was homogenized via acid digestion prior to distribution to the partners. Results were collected from radioactive and elemental analyses. The results were analysed according to the ISO 13528 standard. The performance assessment was carried out using z score. The report includes an overview of the radioanalytical procedures, preliminary and final results, and performance assessments.

Key words

Decommissioning, Difficult-to-measure radionuclides, intercomparison exercise, high activity steel, elemental analysis

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Intercomparison exercise of ETM and non-volatile DTM analysis in homogenised high activity steel

Final Report from the NKS-B MoNi activity (Contract: AFT/B(24)5)

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1 Introduction

Intercomparison exercises on radiochemical analysis of difficult to measure (DTM) radionuclides in real radioactive waste samples has been organised within the NKS community for 4 years (DTM Decom I-III, RESINA projects (Leskinen et al., 2020a, 2021a, 2022a, 2023a). Both Nordic and non-Nordic partners have been involved. In addition to the NKS-reports, the exercise results have been published also in peer reviewed articles (Leskinen et al., 2020b, 2021b, 2022b, 2023b). The focus in the DTM Decom I-III projects was on beta emitters (and to an also extend gamma emitters) whereas RESINA project focused on alpha emitters. The exercises were carried out and statistically analysed according to ISO 13528 standard (ISO, 2015).

The statistically analysed main DTMs in previous projects (Leskinen et al., 2020a, 2021a, 2022a, 2023a) were:

- ^{14}C, ^{55}Fe and ^{63}Ni in activated steel
- ^{3}H, ^{55}Fe, ^{63}Ni in activated concrete
- ^{55}Fe, ^{63}Ni, ^{90}Sr, ^{241}Pu, and alpha emitters in spent ion exchange resin

As the above mentioned list indicates, ^{55}Fe and ^{63}Ni have been exhaustively studied in the previous NKS projects. Additionally, the top three materials in decommissioning (i.e., steel, concrete, resin) have been covered. Therefore, MoNi project proposed to take a step further and study ^{59}Ni and ^{93}Mo which have not been given attention previously:

- ^{59}Ni is purified with ^{63}Ni. ^{63}Ni is generally analysed using liquid scintillation counting (LSC) and its signal overwhelms ^{59}Ni, which is a low X-ray emitter. ^{59}Ni can be measured using its X-rays, but ^{59}Ni standard solutions are expensive and consequently less accessible for method development. ^{55}Fe is also a low X-ray emitter and its use in preparation of standard curves have been reported in literature. Therefore, the challenges in ^{59}Ni analysis are related to procurement of a ^{59}Ni standard solution, suitability of ^{55}Fe use as surrogate, and detection of low energy X-rays. As purification of ^{59}Ni includes procedures in which ^{63}Ni and ^{55}Fe are also purified, these DTMs are also foreseen to be assessed in this project. However, the focus is on ^{59}Ni.
- ^{93}Mo is a long lived DTM with low activities in radioactive waste. This combination may cause constraints in the final disposal. Therefore, reliable radiochemical analysis of ^{93}Mo is especially important. Additionally, there are no commercially available ^{93}Mo standard solutions for method development. The laboratories may take different routes to carry out the method development, but the conclusion remains the same; intercomparison exercise is needed. The purification of ^{93}Mo may also include purification of ^{94}Nb and ^{93}Zr. In addition, there was an interest to study ^{94}Nb and ^{93}Zr.

The MoNi project content followed the same methodology as in the previous intercomparison projects:

- Kick-off meeting (February or early March) to discuss the analysis capabilities, to select the radionuclides of interest and discuss the material to be studied.
- Sending of the material to partners by May.
- Analysis time until early September.
- Discussion of preliminary results in a preliminary meeting held by middle of October.

- Revision of results until early November.
- Discussion of statistically analysed results by middle of November.
- Reporting of results in a NKS report by end of the year.

Young scientists were involved in the MoNi project at VTT (MSc), DTU (PhD student and Post doc), IFE (PhD student), NMBU (MSc student) and Isotoptech (PhD student).

2 Survey of capabilities in the beginning of the MoNi project

A survey of the DTM analysis capabilities of the partners was carried out in the beginning of the project. The results are shown in Table 1. The affiliation codes (A to J) and the sample numbers (1-10) in the following sections do not correlate to facilitate anonymous reporting. The capabilities of the partners show that use of variety of analytical methods and measurement equipment were foreseen. Additionally, some portfolios included routine radioanalytical methods for several radionuclides whereas some methods were under development or validation. The reporting includes several abbreviations which are explained in Table 2

Table 1 Capabilities of the participating laboratories on DTM analyses in high activity steel in the beginning of the project.

Affiliation code	Readiness	Overview of planned methods	Equipment
A	Method ready for purification of ^{55}Fe, ^{59}Ni and ^{63}Ni. Detection of ^{59}Ni being finalised. ^{93}Mo method under development.	Acid digestion of the sample. ^{14}C oxidised and trapped in NaOH solution. Use of AG resin and Ni resin in ^{55}Fe, ^{59}Ni and ^{63}Ni purification method. LSC for ^{14}C, ^{55}Fe and ^{63}Ni detection. BEGe for ^{59}Ni detection. ICP-OES for Fe and Ni yield determinations. ^{93}Mo purification tested using cation exchange, TEVA® and alumina resin columns. ^{93}Zr and ^{94}Nb purified as by products. Detection of ^{93}Mo using ICP-MS (primary) or LSC (secondary).	QQQ-ICP-MS (Agilent), ICP-OES (Agilent SVDV), LSC (Hidex), Mirion broad energy gamma detector
B	Method ready for ^{14}C, ^{55}Fe, ^{63}Ni, ^{59}Ni, ^{94}Nb, ^{36}Cl, ^{41}Ca). ^{93}Mo, ^{93}Zr and ^{79}Se methods under development. ^{41}Ca method to be validated.	Acid digestion of the sample. Use of resins to separate radionuclides. Measurement using LSC, gamma-spectrometry or ICP-MS.	LSC, gamma spectrometry, ICP-MS
C	Methods in place for ^{55}Fe, ^{63}Ni, ^{59}Ni. Chemistry known and measurement possible for ^{93}Mo, ^{93}Zr, ^{94}Nb, ^{79}Se, ^{41}Ca.	Microwave dissolution of sample. LSC for ^{55}Fe/^{63}Ni. γ-spectrometry for ^{94}Nb (depending on activity) ICP-MS for ^{41}Ca (possibly), ^{59}Ni, ^{63}Ni (possibly), ^{79}Se (possibly), ^{93}Zr and ^{94}Nb	ICP-MS Agilent 8900 triple quad HIDEX LSC Ortec HPGe
D	Routine analysis for ^{3}H, ^{14}C, ^{55}Fe, ^{63}Ni, ^{59}Ni, ^{93}Mo, ^{93}Zr, ^{94}Nb, ^{36}Cl and ^{41}Ca in radioactive samples at low and intermediate level activity (liquids, effluents, steels, concretes and resins etc.).	For ^{14}C, a pyrolyser decomposition is used. For ^{55}Fe, ^{63}Ni, ^{59}Ni, ^{93}Mo, ^{93}Zr, ^{94}Nb, ^{41}Ca and ^{36}Cl, acid digestion or alkaline fusion is used for desction of matrix. Purification of the nuclides using hydroxide or ammonia precipitation, anion exchange resin or Ni resin (for ^{63}Ni) or TRU® resin (for ^{55}Fe) or Sr resin (for ^{41}Ca) or DMG precipitation (^{59}Ni) or liquid-liquid extraction with BPHA (^{93}Zr, ^{94}Nb), or liquid-liquid extraction with trioctyl ammonium chloride (^{93}Mo), organic synthesis (for ^{36}Cl).	ICP-OES for Ca, Ni and Fe yields, ion chromatography for Cl yield, gravimetry for Ni, Mo and Nb yields. LSC for ^{14}C, ^{36}Cl, ^{41}Ca, ^{55}Fe and ^{63}Ni. X spectrometry for ^{93}Mo and ^{94}Nb. ICP-MS for ^{93}Zr. We can use AMS to detect ^{36}Cl and ^{41}Ca at low level.

E	Method ready for ^{14}C, ^{55}Fe, ^{63}Ni, ^{59}Ni, ^{93}Mo, ^{93}Zr, ^{94}Nb, ^{36}Cl, ^{41}Ca. ^{41}Ca method to be validated.	Acid digestion of the sample. ^{14}C separated by wet oxidation-acid stripping. Use of Dowex1x8 resin and DMG in ^{55}Fe and ^{59}Ni/^{63}Ni purification method. Use of AG1x4 resin in ^{36}Cl purification. LSC for ^{14}C, ^{55}Fe and ^{63}Ni detection. LEGe for ^{59}Ni detection. Low Background Alpha/Beta Counting System for ^{36}Cl (AgCl precipitation) detection. ICP-OES for Fe and Ni yield determinations. ^{93}Mo, ^{93}Zr and ^{94}Nb purification tested using TEVA® column. Detection of ^{94}Nb using HPGe, ^{93}Mo using LEGe and ^{93}Zr using LSC.	ICP-MS (Agilent 8900), ICP-OES (Varian 710-ES), LSC (PerkinElmer 4900), Mirion HPGe/LEGe(Low Energy Germanium detector)
F	Method ready for separation and analysis of ^{14}C, ^{36}Cl, ^{79}Se, ^{55}Fe, ^{59}Ni, ^{63}Ni, ^{94}Nb and ^{93}Zr from liquid waste and concrete matrices. Method for ^{93}Mo in liquid waste and concrete is under development.	Acid digestion of samples. Separation of radionuclides on extraction chromatographic resins. Measurement techniques: ^{93}Zr - ICP-MS and LSC; ^{93}Mo - LSC; ^{93m}Nb – X-ray spectrometry) ^{94}Nb - gamma spectrometry; ^{55}Fe (LSC); ^{63}Ni (LSC); ^{59}Ni (X-ray spectrometry); ^{36}Cl (LSC); ^{14}C (LSC/AMS); ^{79}Se (LSC)	ICP-MS (Agilent 8800); Gamma spectrometry: HPGe+LEGe detectors Canberra 16k MCA, Genie software; LSC: PerkinElmer TriCArb 2800; Agilent 4100 MP-AES.
G	Method ready for separation and analysis of ^{14}C, ^{36}Cl, ^{41}Ca, ^{55}Fe, ^{63}Ni, ^{93}Mo, ^{94}Nb. Methods for ^{79}Se and ^{93}Zr are under development.	Acid digestion of the sample. 14C oxidised and trapped in NaOH solution. Use of AG resin and Ni resin in ^{55}Fe and ^{59}Ni/^{63}Ni purification method. AgCl precipitation or AG1x4 resin in ^{36}Cl purification. $CaCO_3/Ca(OH)_2$ precipitation for 41Ca. Anion+Cation exchange +Alumina resin for ^{93}Mo. LSC for ^{14}C, ^{36}Cl, 41Ca, ^{55}Fe, ^{63}Ni, ^{93}Mo detection. ICP-OES for Fe and ICP-OES for Fe and Ni yield determinations. Gamma for ^{94}Nb.	LSC, gamma spectrometry, ICP-QQQ (Agilent 8800)
H	Method ready for: ^{93}Zr, ^{41}Ca (concrete), ^{55}Fe (separation method to be validated).	Acid-assisted digestion. ^{41}Ca, ^{59}Ni, ^{93}Mo, ^{36}Cl determination by ICP-MS. TK400® ^{55}Fe,	ICP-MS/MS (Agilent 8800), gamma spectrometry, LSC, alpha spectrometry
I	Method ready for ^{59}Ni. Method development for ^{93}Mo, ^{93}Zr.	Resin exchanges for separation and purification	LSC, gamma spectrometry, ICP-MS

J	Method ready for ^{63}Ni and ^{55}Fe. Method development for ^{59}Ni and ^{93}Mo.	HR-ICP-MS for ^{59}Ni and ^{93}Mo. LSC for ^{55}Fe and ^{63}Ni. γ-spectrometry for ^{93}Mo (16.6keV) and ^{60}Co.	Hidex Ulla Ultra Low Level Analyzer, Thermo Fisher Element Series HR-ICP-MS, Canberra HPGe detector

Table 2 Abbreviations

Abbreviation	
AMS	Accelerator Mass Spectrometry
BEGe	Broad Energy Germanium
BPHA	Bisphenol A
DMG	Dimethylglyoxime
DTM	Difficult-To-Measure
ETM	Easy-To-Measure
HPGe	High Purity Germanium
HR-ICP-MS	High Resolution Inductively Coupled Plasma Mass Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-MS/MS	Inductively Coupled Plasma tandem Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ISOCS	In-situ Object Counting System
ISOXRCE	ISOCS calibration source
LEGe	Low Energy Germanium
LSC	Liquid Scintillation Counting
MDA	Minimal Detectable Activity
MP-AES	Microwave plasma atomic emission spectrometer
RSD	Relative standard deviation
TDCR	Triple-to-Double Coincidence Ratio
QQQ-ICP-MS	Triple Quadrupole Inductively Coupled Plasma Mass Spectrometry

3 Sample selection

Three high activity samples were discussed in the kick-off meeting:

- Steel 1: with moderate dose rate (0.1 g sample with dose rate of ~100 µSv h^{-1} at 1 cm), having known chemical compositions of Mo and Ni (0.2 and 9 mass-%, respectively) and preliminary activation calculation results.
- Steel 2: with high dose rate (0.1 g sample with dose rate of ~1 mSv h^{-1} at 1 cm) and having known chemical compositions of Mo and Ni (2 and 11 mass-%, respectively).
- Steel 3: with very high dose rate (0.01 g sample with dose rate of ~6 mSv h^{-1} at 1 cm), and without known Ni or Mo content, although the material was known to contain Mo.

The samples were discussed based on the probabilities that measurable amounts of radionuclides of interest would be present in the sample in comparison with radiation safety. Based on the discussions, Steel 1 was selected. The sample amount was selected to be ~100 mg per partner. The main radionuclides of interest in MoNi were ETMs, ^{59}Ni and ^{93}Mo. Optional radionuclides to analyse were ^{55}Fe, ^{63}Ni, ^{93}Zr, ^{94}Nb and ^{93m}Nb.

In previous intercomparison exercises, the sample homogeneity was determined using gamma spectrometry. The relative standard deviations (RSD) were ~1-2 % (Leskinen et al. 2020a, 2021a, 2022a, 2023a). However, higher RSDs were encountered in a trilateral intercomparison study on high activity steel in 2023 (Leskinen et al. 2023c). The same Steel 1 material had been studied and 100 mg of small chips of the material was studied for homogeneity. The results showed an RSD of ~10 %. Therefore, homogenisation of the material via acid digestion was decided to be carried out prior to distribution of samples to the partners. The acids in the acid digestion were determined based on the partners' experience.

As acid digestion affects the volatile radionuclides (i.e., ^{14}C, ^{36}Cl and ^{79}Se), the project was divided into two exercises:

- MoNi project to study non-volatile radionuclides.
- MoNi II project to study volatile radionuclides (proposed for 2025).

4 Sample preparation

The Steel 1 sample was sub-sampled using a MeteCNC milling machine in a hot cell (Figure 1). The milling machine produced small chips of the Steel 1 (~ 1 g), which were taken outside the hot cell for further preparation. The Steel 1chips were dissolved in *Aqua Regia* (1:3 ratio of HNO_3 and HCl) with small amount of HF to keep Nb in solution and to be still acceptable for handling in some partner laboratories. The acid digestion solution was divided to ~ 6 g samples in plastic 20 ml liquid scintillation vials (Figure 2). Each sample was measured using gamma spectrometry and homogeneity study of each sample was carried out based on ^{60}Co activity. The ^{60}Co RSD was 2.7 % and consequently the samples were considered as homogenous. The samples were distributed to partners in April-May.

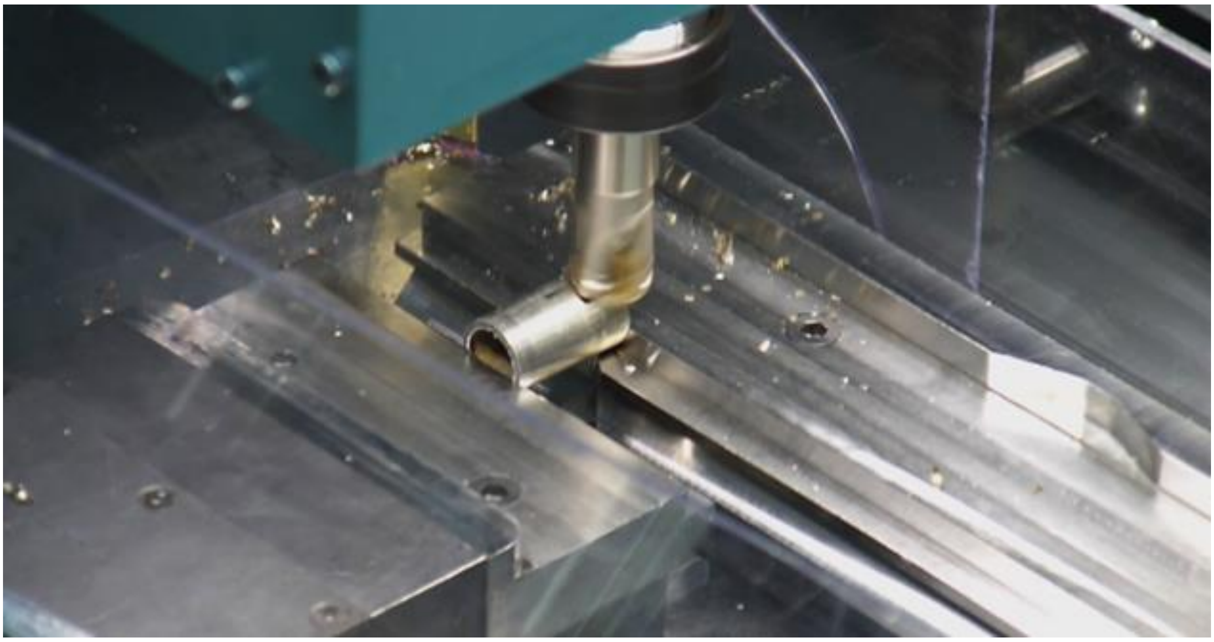

Figure 1. Sub-sampling of Steel 1 using CNC milling machine.

Figure 2. Example of an acid digested sample.

5 Preliminary meeting results

The analysis methods, yields and analysis result trends were discussed in the preliminary meeting in October. Seventy percent of the partners had submitted results by the preliminary meeting. Some partners were contacted to re-check the calculations in cases when a clear outlier was noticed prior to the preliminary meeting. No indication was given whether the deviation was positive or negative to the general trend. The reported radiochemical analysis yield results are shown in Table 3. The yield results show that the ^{55}Fe yields varied from approximately 10 to 100 %, ^{63}Ni yields varied from ~50-100 %, ^{59}Ni yields varied from approximately 50 to 100 %, ^{93}Mo yields varied from ~30-90 %, ^{93}Zr yields varied from 30-100 %, ^{94}Nb yields varied from ~45-100 %, and ^{93m}Nb yields were ~100 %. Some new yield results and updates are reported in Table 13.

Table 3. Radiochemical yields and uncertainties (*k = 2*) discussed in the preliminary meeting.

	Yield ± uncertainty [%] (*k =2*)						
	^{55}Fe	^{63}Ni	^{59}Ni	^{93}Mo	^{93}Zr	^{94}Nb	^{93m}Nb
1	-	-	-	-	-	-	-
2	~80	-	-	~30-75	~30-75	-	-
3	-	-	-	-	-	-	-
4	~55-60	~80	79±15	32±15	32±15	-	-
5	13 ± 10	82 ± 10	-	-	98 ± 10	98 ± 10	-
6	74 ± 15	61 ± 9	61 ± 9	93±1	99 ±1	97 ± 1	98 ± 1
7	98 ± 16	98 ± 16	80± 5	44±8	-	-	-
8	-	-	-	-	-	-	-
9	-	~50-60	50 ± 2	~70-90	~40-50	~45-60	-
10	101 ± 1	99 ± 1	99 ± 1	87 ± 1	99 ± 1	101 ± 2	101 ± 2

5.1 Preliminary analysis results of ETMs

The sample specific ETM analysis methodologies are reported in Table 4. The sample preparations contained mainly measurements in aliquots with or without dilutions. The measurement distance from the sample to the detector varied between 16 mm to 10 cm. Dead times varied from 0.09 % to 4.4 % and measurement times from 15 min to 22 hours. All measurements were carried out using High Purity Germanium detectors (HPGe) and efficiency calibrations were carried out mainly using ^{60}Co, ^{152}Eu or mixed standards and to lesser extent (2) with ISOCS/Labsocs. The uncertainty calculations were reported to be mainly combined uncertainties. Reported references were decay data for ^{60}Co (LNE-LNHB/CEA, 2010) and power moderated mean (Pomme & Keightley, 2015.)

The preliminary analysis replicate results are shown in Figure 3. The results show a good alignment. Sample 2 analyser reported presence of ^{137}Cs signal with ~ 48 % relative uncertainty (*k = 1*) and therefore a quantitative analysis result was not reported. Sample 10 analyser reported ^{94}Nb result measured in the original acid digested sample to be below minimal detectable activity (MDA) of ~1070 Bq g^{-1} which was much higher compared to the radiochemically purified ^{94}Nb results.

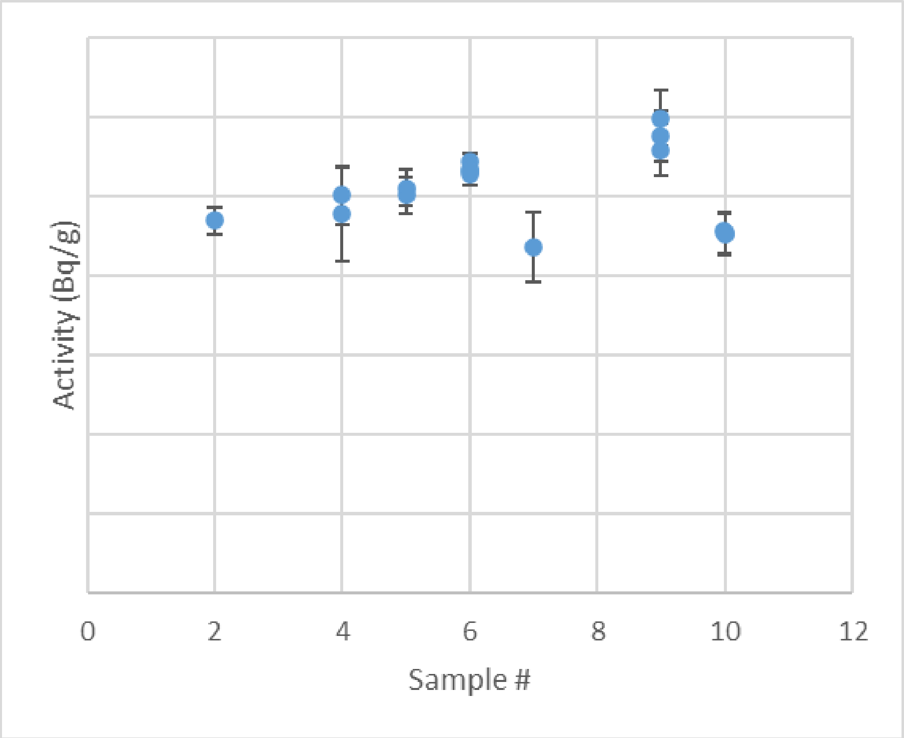

Figure 3. Preliminary ^{60}Co results in high activity steel. The uncertainties are stated with a coverage factor of *k = 2*. Analysis methodologies expressed in Table 4.

Table 4. ETM analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8	9	10
Sample preparation	-	Original sample diluted, and a subsample of this solution taken for γ-spectrometry	-	50μL of sample into 50mL HNO3 0.1M	A small aliquot of the sample was placed in a glass recipient and mixed with 1 M HNO_3 to a total volume of 5 ml.	Sample was diluted to 20mL.	Sample was measured as received using a sample mass of around 0.1 ml	-	0.5 mL aliquot weighted on an analytical balance as well as aliquots of the load solutions used for DTM measurements were measured.	Sample was measured as received using the provided mass information.
Measurement geometry	-	10 mL solution in 20 mL plastic scintillation vial	-	SG50 (2 devices were used)	Sample was measured at 8 cm distance on top of the detector	Sample was measured at 10 cm distance on top of the detector.	Sample was measured in a 50 mL vial at 16 mm distance on top of the detector.	-	Samples were diluted to 10 mL in LSC vials and measured at about 10 cm distance from the detector.	Sample was measured at 4 cm and 10 cm distance on top of the detector.
Dead time	-	0.86 %	-	0.16% / 0.09%	0.09 %	1.6%	0.16 %	-	0.50 %	At 4 cm 4.4%, at 10 cm 1.6%
Measurement time	-	80 000s	-	14400s / 18000s	30000 s	2hrs, three replicate measurements	10800s, one replicate	-	4000 s	3 times 15 min at 4 cm, 2 times 60 min at 10 cm.
Detector type and efficiency	-	p-type coaxial	-	HPGe 50.2% / HPGe 49.4%	BeGe (Mirion); BE3830 34% efficiency	Canberra HPGe 4020	Canberra SEGe GC3018; 30% relative efficiency	-	HPGe detector attached to Canberra 16k MCA with Genie software	Miron HPGe, 40% S/N-B19116 and Mirion HPGe, 15% S/N-B18067

Efficiency calibration	-	Direct comparison with ^{60}Co standard	-	by ISOCS/Labsocs	efficiency calibrated using a mixed gamma calibration standard (LEA)	Efficiency calibration using ^{152}Eu standard radiation source	same geometry efficiency calibration with multi-gamma source	-	Efficiency calculation was performed with a mixed standard solution.	ISOCS efficiency calibration using Geometry composer. ISOXRCE measured before and after the measurement campaign
Description of uncertainty calculations	-	Quadrature sum of net count rate, efficiency, emission probability and decay	-	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty: sample prep, efficiency, measurement uncertainty	Combined uncertainty with the sample preparation and measurement uncertainties.	Combined uncertainty with the sample preparation and measurement uncertainties	-	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty calculated using the ISOCS MDA result and other uncertainties arising from weighing.

5.2 Preliminary radiochemical ^{55}Fe results

The sample specific ^{55}Fe analysis methodologies are reported in Table 5. The overview of the radiochemical methods included different resin treatments (i.e., anion exchange, TRU®), precipitation, evaporation, and liquid-liquid extraction. The activities were measured using liquid scintillation counting (LSC) and yields by analysis of stable Fe using ICP-MS or ICP-OES. Stabilisation time for the LSC measurements varied from 15 min to 2-3 days. The efficiency calibrations were carried out using TDCR or ^{55}Fe quench curve. The uncertainty calculations were reported to be mainly combined uncertainties. Reported references were decay data for ^{55}Fe (LNE-LNHB/CEA, 2024), power moderated mean (Pomme, Kneightley, 2015), and radiochemical analyses (Gautier et al., 2020, Leskinen et al., 2020c).

The preliminary ^{55}Fe analysis replicate results are shown in Figure 4. The results show a somewhat scattered trend. Sample 2 and 6 were respectively the highest and lowest data entries.

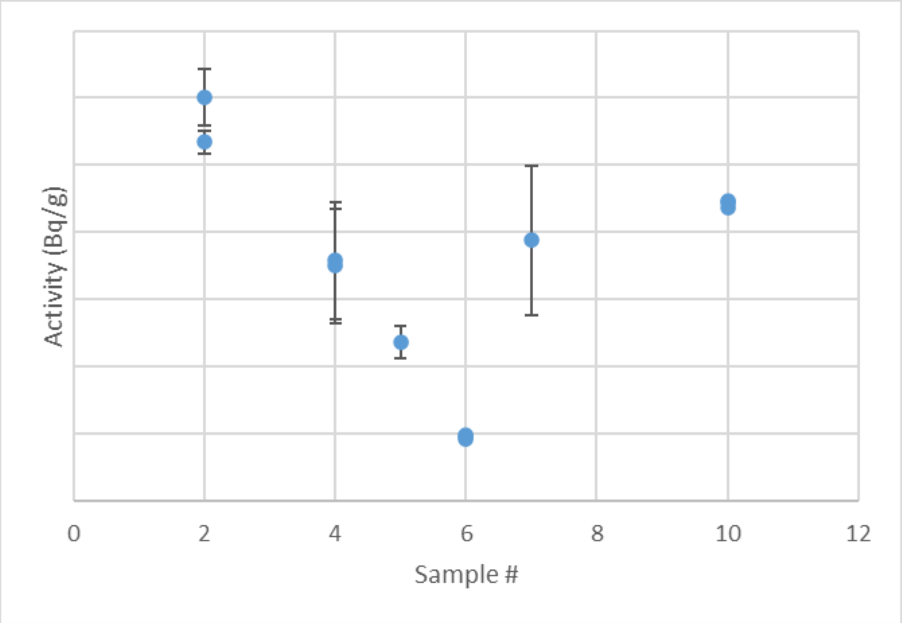

Figure 4. Preliminary ^{55}Fe results high activity steel. The uncertainties are stated with a coverage factor of $k = 2$. Analysis methodologies expressed in Table 5.

Table 5. Radiochemical ^{55}Fe analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8	9	10
Overview of the radiochemical method	-	^{55}Fe was separated from the 3 diluted sample aliquots using anion exchange, with no added stable carrier - approximate iron content ~500 µg. The columns was stripped with 10M HCl (Co and Ni), 6 M HCl (Zr), 3M HCl (Mo) and 0.1 M HNO3 (Fe). Stable iron content measured on a fourth diluted aliquot by ICP-MS.	-	Liquid-liquid extraction of 20µL or 50µL of sample dilute in 100mL deionized water. Add of stable Fe to follow the extraction (separation yield). Determination of the separation yield of ^{55}Fe by MP-AES.	Fe was separated and purified using an ion exchange resin	An aliquot was taken for the original chemical composition studies using ICP-OES first. ^{55}Fe was purified in conjunction with ^{59}Ni and ^{63}Ni. NaOH was added to precipitate most of the cations. Concentrated NH4OH was added to form Fe precipitates. Dowex 1 × 8 resin was then used to separate Fe from other impurities.	^{55}Fe was *not* purified in conjunction with ^{63}Ni. First, an aliquot was taken for the original chemical composition studies using ICP-OES. One aliquot sample was taken to contain approximately 1 mg of Fe according to the measured initial chemical composition results. ^{55}Fe was first precipitated with ammonia and separated using AG ion exchange resin followed by TRU® resin.	-	N/A	^{55}Fe was purified in conjunction with ^{59}Ni and ^{63}Ni. Three aliquot samples were taken to contain approximately 4 mg of Fe in each aliquot according to the submitted initial chemical composition results. 2 mg of Ni was added in each aliquot sample. Additionally, an aliquot was taken for the original chemical composition studies using ICP-OES. The aliquot samples were first evaporated to near dryness and dissolved in 9M HCl. ^{55}Fe was separated from ^{63}Ni and ^{59}Ni and purified using AG ion exchange resin.
Measurement information	-	Purified iron fraction precipitated as $Fe(OH)_3$, washed and dissolved in minimum of H_3PO_4 for LSC counting.	-	Mixture of 9,9mL sample and 10mL UltimaGold LLT liquid scintillation counting cocktail in a 20 mL plastic vial. Sample were let to stabilise 2 or 3 days to reduce luminescence. Samples were measured by 2 cycles of 60min	The purified Fe fraction was measured with LSC to determine ^{55}Fe	1 mL of purified ^{55}Fe fraction was mixed with 15 mL of Pico-Fluor Plus cocktail in a 20 mL plastic vial. Samples were measured 3 times 30mins. Yield was determined by measuring the stable Fe content in the purified fraction.	3 ml of purified ^{55}Fe fraction was mixed with 17 ml of UltimaGold LLT liquid scintillation counting cocktail in a 20 ml glass vial. Samples were let to stabilise minimum of 15 min to reduce luminescence. Sample was	-	N/A	1 ml of purified ^{55}Fe fraction was mixed with 10 ml of HiSafe 3 liquid scintillation counting cocktail in a 20 ml glass vial. Samples were let to stabilise minimum of 12 hours to reduce luminescence. Samples were measured 3 times 3600s. Yield was determined by measuring the stable Fe content in the purified fraction.

							measured once 1800s. Yield was determined by measuring the stable Fe content before and after separation by ICP-AES.			
Equipment	-	HIDEX 600 SL and Agilent ICP-QQQ 8900	-	Quantulus GCT6220 (Perkin Elmer) Liquid Scintillation	Quantulus 1220TM	PerkinElmer Tri-Carb 4910TR and Varian ICP-OES 710-ES	Ametek ICP-AES Spectro Blue for ICP. AccuFLEX LSC-8000 Hitachi for LSC	-	N/A	Hidex 300 SL liquid scintillation counting. Agilent ICP-OES 5100 SVDV. Mettler Toledo AX 504 balance.
Efficiency calibration	-	From TDCR values generated by HIDEX 600 SL	-	^{55}Fe efficiency curve	27 %	Using 5 vials of standard ^{55}Fe sources with the same activities but different tSIEs to fit an efficiency regression line on LSC.	Quench curve with ^{55}Fe source	-	N/A	Approximately 0.6 derived from ^{55}Fe TDCR with CoreF
Description of uncertainty calculations	-	Complex, but based on counting uncertainty (0,3%), TDCR (varies widely), yield (~2-3%) and decay (~0,04%). Means calculated as power moderated mean.	-	Combined uncertainty with the sample preparation and measurement uncertainties		Combined uncertainty with the sample preparation and measurement uncertainties, including the efficiency, counts, weight, recovery rates.	Combined uncertainty with the sample preparation, recovery yield and measurement uncertainties	-	N/A	Combined uncertainty with the sample preparation and measurement uncertainties

5.3 Preliminary radiochemical ^{63}Ni results

The sample specific ^{63}Ni analysis methodologies are reported in Table 6. The overview of the radiochemical methods included different resin or DMG treatments, precipitation, and evaporation. The activities were measured similarly to ^{55}Fe. The uncertainty calculations were reported to be mainly combined uncertainties. References were reported for the radiochemistry (Gautier et al., 2016, Leskinen et al., 2020 c).

The preliminary ^{63}Ni analysis replicate results are shown in Figure 5. The results show a good alignment, except Sample 6 seems to deviate from the general trend. Sample 2 analyser reported that meaningful results using QQQ-ICP-MS was not possible due to the interference from ^{63}Cu.

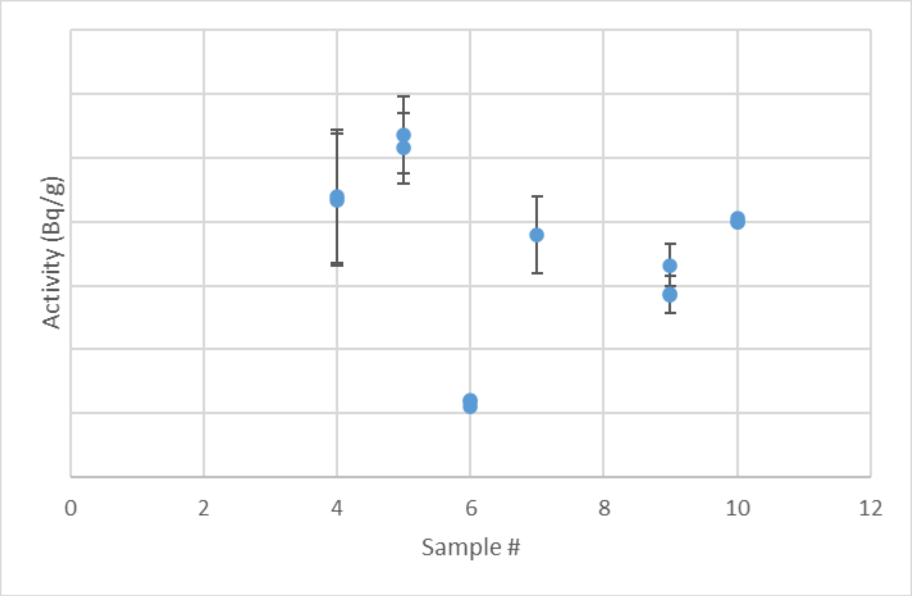

Figure 5. Preliminary ^{63}Ni results in high activity steel. The uncertainties are stated with a coverage factor of $k = 2$. Analysis methodologies expressed in Table 6.

Table 6. Radiochemical ^{63}Ni analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8	9	10
Overview of the radiochemical method	-	See ^{55}Fe	-	Purification of 20µL and 50µL of sample (dilute in 10mL deionized water) on anionic resin then DMG resin. Add of stable Ni before separation to follow the extraction yield. Determination of the separation yield of ^{63}Ni by MP-AES.	Ni-63 was separated before analysis by using an ion exchange resin followed by a separation on an extraction resin	An aliquot was taken for the original chemical composition studies using ICP-OES first. ^{59}Ni and ^{63}Ni were purified in conjunction with ^{55}Fe. NaOH was added to precipitate most of the cations. Concentrated NH4OH was added for Fe and Ni separation. Purification of Ni was carried out by adding ammonium citrate and dimethylglyoxime (DMG) for the formation of a complex of Ni.	Ni-63 was not purified in conjunction with Fe-55. First, an aliquot was taken for the original chemical composition studies using ICP-OES. 1.8 mg of Ni was added to the aliquot sample according to the measured initial chemical composition results. The aliquot sample was first evaporated to dryness and dissolved in 8M HCl. Ni-63 was first separated using AG ion exchange resin and then was purified using Ni-resin.	-	^{59}Ni and ^{63}Ni were purified in conjunction with ^{55}Fe and partially with other DTMs. Two aliquot samples of 1,83 g were taken to start processing together with other DTMs on cation exchanger, and another aliquot of 0,6 g was processed separately. Fe was removed using MIBK chromatography and Ni was separated on DMG column. Ni was precipitated as Ni-DMG2. One of the precipitates was used to measure ^{59}Ni by X-ray spectrometry. Finally, the precipitates were dissolved. Part of the solution was used for chemical recovery determination with MP-AES,	^{59}Ni and ^{63}Ni were purified in conjunction with ^{55}Fe. Three aliquot samples were taken to contain approximately 4 mg of Fe in each aliquot according to the submitted initial chemical composition results. 2 mg of Ni was added in each aliquot sample. Additionally, an aliquot was taken for the original chemical composition studies using ICP-OES. The aliquot samples were first evaporated to near dryness and dissolved in 9M HCl. Fe-55 was separated from ^{59}Ni and ^{63}Ni using AG ion exchange resin. ^{59}Ni and ^{63}Ni fraction was purified using Ni-resin.

									and part was used for LSC measurement of ^{63}Ni.	
Measure-ment information	-	See ^{55}Fe	-	Mixture of 9,9mL sample and 10mL UltimaGold LLT liquid scintillation counting cocktail in a 20 mL plastic vial. Sample were let to stabilise 2 days to reduce luminescence. Samples were measured by 2cycles of 30min	^{63}Ni was measuerd with LSC; 0,5 ml diluted Ni fraction was mixed with HiSafe 3 liquid scintillation counting cocktail in a 20 ml glass vial	1 mL of purified ^{63}Ni fraction was mixed with 15 mL of Pico-Fluor Plus cocktail in a 20 mL plastic vial. Samples were measured 3 times 30mins. Yield was determined by measuring the stable Ni content in the purified fraction.	5 ml of purified ^{63}Ni fraction was mixed with 15 ml of UltimaGold LLT liquid scintillation counting cocktail in a 20 ml glass vial. Samples were let to stabilise minimum of 15 min to reduce luminescence. Sample was measured once 1800s. Yield was determined by measuring the stable Ni content before and after separation by ICP-AES.	-	An aliquot of the purified ^{59}Ni and ^{63}Ni fraction was evaporated and dissolved in 1 mL 1M HNO3 and mixed with 10 ml of Gold star liquid scintillation cocktail in a 20 ml glass vial. Samples were measured 2 times for 600s. Yield was determined by measuring the stable Ni content using MP-AES and ICP-MS.	0.1 ml of purified ^{59}Ni and ^{63}Ni fraction was mixed with 10 ml of HiSafe 3 liquid scintillation counting cocktail in a 20 ml glass vial. Samples were let to stabilise minimum of 12 hours to reduce luminescence. Samples were measured 3 times 3600s. Yield was determined by measuring the stable Ni content in the purified fraction.
Equipment	-	HIDEX 600 SL and Agilent ICP-QQQ 8900	-	Quantulus GCT6220 (Perkin Elmer) Liquid Scintillation	Quantulus 1220TM	PerkinElmer Tri-Carb 4910TR and Varian ICP-OES 710-ES	Ametek ICP-AES Spectro Blue for ICP. AccuFLEX LSC-8000 Hitachi for LSC	-	N/A	Hidex 300 SL liquid scintillation counting. Agilent ICP-OES 5100 SVDV. Mettler Toledo AX 504 balance.
Efficiency calibration	-	From TDCR values generated	-	^{55}Fe efficiency curve	27 %	Using 5 vials of standard ^{55}Fe sources with the same activities but	Quench curve with ^{55}Fe source	-	N/A	Approximately 0.8 derived from ^{63}Ni TDCR with CoreF

		by HIDEX 600 SL				different tSIEs to fit an efficiency regression line on LSC.				
Description of uncertainty calculations	-	Complex, but based on counting uncertainty (0,3%), TDCR (varies widely), yield (~2-3%) and decay (~0,04%). Means calculated as power moderated mean (see note).	-	Combined uncertainty with the sample preparation and measurement uncertainties		Combined uncertainty with the sample preparation and measurement uncertainties, including the efficiency, counts, weight, recovery rates.	Combined uncertainty with the sample preparation, recovery yield and measurement uncertainties	-	N/A	Combined uncertainty with the sample preparation and measurement uncertainties

5.4 Preliminary radiochemical ^{59}Ni results

The sample specific ^{59}Ni analysis methodologies are reported in Table 7. The overview of the radiochemical methods included different Ni-resin or DMG treatments, liquid-liquid extractions, and precipitation. The sample measurements were carried out in liquid or solid form (i.e., precipitate, evaporation residue). The measurements were carried out using different types of detectors i.e., GL2015, LEGe, HPGe with Be window and BEGe. The efficiency calibrations were carried out using ^{59}Ni standard solution, multinuclide efficiency calibration or ^{55}Fe surrogate. The uncertainty calculations were reported to be mainly combined uncertainties. No radiochemistry related references were reported as the in-house methods had not been published.

The preliminary ^{59}Ni analysis replicate results are shown in Figure 6. The results did not show a clear trend even though Samples 6, 7 and 10 are somewhat aligned. Sample 4 result is below limit of detection whereas all other results are quantitative with higher activity concentration compared to Sample 4. Additionally, Sample 9 result is above the Sample 6, 7, and 10 results. Sample 2 analyser reported that meaningful results using QQQ-ICP-MS was not possible due to interference from ^{59}Co.

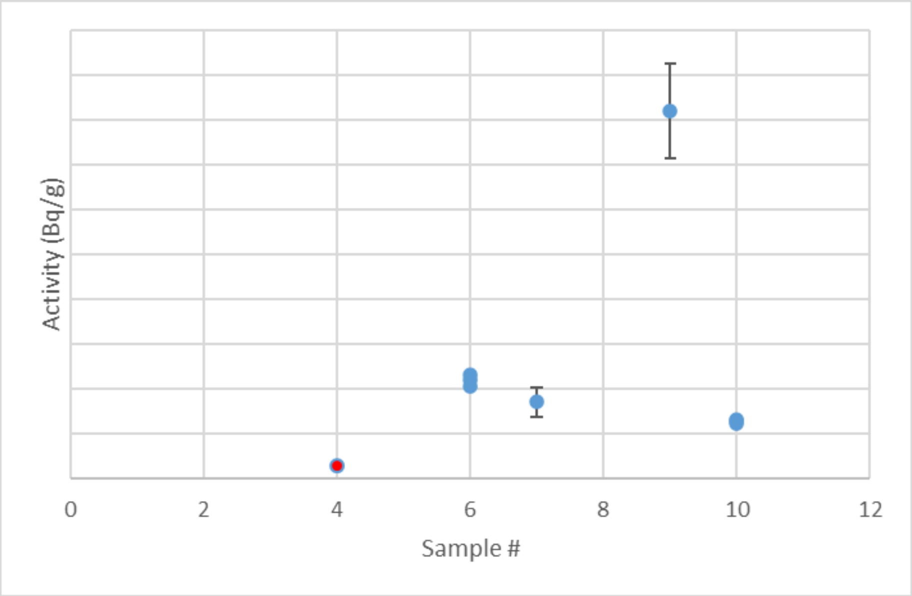

Figure 6. Preliminary ^{59}Ni results in high activity steel. The uncertainties are stated with a coverage factor of $k = 2$. Red dot indicates a result under limit of detection. Analysis methodologies expressed in Table 7.

Table 7. Radiochemical ^{59}Ni analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8	9	10
Overview of the radiochemical method	-	See ^{55}Fe	-	Purification of 20µL and 50µL of sample (dilute in 10mL deionized water) on anionic resin then DMG resin. Add of stable Ni before separation to follow the extraction yield. Determination of the separation yield of ^{63}Ni by MP-AES.	-	Purification of 20µL and 50µL of sample (dilute in 10mL deionized water) on anionic resin then DMG resin. Add of stable Ni before separation to follow the extraction yield. Determination of the separation yield of ^{63}Ni by MP-AES.	The purification protocol is not the same as for ^{63}Ni. Precipitation was first performed with ammonium to precipitate iron. After recovery of the supernatant, a first precipitation with DMG was done followed by liquid-liquid extraction based on nitroso 1 naphtol 2 in chloroform. Then, liquid-liquid extraction based on DMG in chloroform was performed and a final precipitation with DMG was done.	-	^{59}Ni and ^{63}Ni were chemically processed together as long as the Ni-DMG2 precipitate was formed.	^{59}Ni and ^{63}Ni was purified in conjunction with ^{55}Fe. Three aliquot samples were taken to contain approximately 4 mg of Fe in each aliquot according to the submitted initial chemical composition results. 2 mg of Ni was added in each aliquot sample. Additionally, an aliquot was taken for the original chemical composition studies using ICP-OES. The aliquot samples were first evaporated to near dryness and dissolved in 9M HCl. ^{55}Fe was separated from ^{59}Ni and ^{63}Ni using AG ion exchange resin. ^{59}Ni and ^{63}Ni fraction was purified using Ni-resin.
Measurement information	-	See ^{55}Fe	-	Mixture of 100µL sample+9,9mL HCl 0,1M in a plastic vial analyzed by X spectrometry (350 000s)	-	Mixture of 100µL sample+9,9mL HCl 0,1M in a plastic vial analyzed by X spectrometry (350 000s)	Around 10 mg Ni-DMG precipitate is finally obtained. Yield was determined by weighing the precipitate. X-spectrometry for ^{59}Ni	-	The Ni-DMG2 precipitate was filtered on a membrane and covered after drying with a plastic foil, that was placed in a standard geometry close (about 2 cm) to the detector. Counting time was	1 ml of purified ^{59}Ni and ^{63}Ni fraction was let to dry on a membrane filter. Samples were measured 3 times 2 d. Yield was determined by measuring the stable Ni content in the purified fraction.

									234000 s with dead time of 0,09%	
Equipment	-	See ^{55}Fe	-	Canberra detector GL2015 (7500SL), measurement subcontacted to CEA LARC Laboratory	-	Canberra detector GL2015 (7500SL), measurement subcontacted to CEA LARC Laboratory	Ametek ICP-AES Spectro Blue for initial Ni content. Mettler Toledo XP 504 balance for final Ni content. Canberra LEGe GL0210: measurement during 86400 s.	-	HPGe detector with 25 µm Be window attached to Canberra 16k MCA with Genie software	Mirion Broad Energy HPGe. Agilent ICP-OES 5100 SVDV. Mettler Toledo AX 504 balance.
Efficiency calibration	-	See ^{55}Fe	-	^{59}Ni	-	^{59}Ni	Equivalent geometry multi-nuclide efficiency calibration & sample/standard attenuation correction	-	Counting efficiency is approximately 0.07. ^{55}Fe source was prepared by evaporating a ^{55}Fe standard solution on a membrane. The source was covered with the plastic foil and was placed in the standard geometry close to the detector.	^{55}Fe surrogate efficiency curve
Description of uncertainty calculations	-	See ^{55}Fe	-	Combined uncertainty with the sample preparation and measurement uncertainties	-	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty with the sample preparation, recovery yield and measurement uncertainties	-	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties

5.5 Preliminary radiochemical ^{93}Mo results

The sample specific ^{93}Mo analysis methodologies are reported in Table 8. The overview of the radiochemical methods included several methods i.e., liquid-liquid extractions, resin treatments, evaporations, and precipitations. The sample measurements were carried out in liquid or solid form. The measurements were carried out using different types of techniques i.e., EGM-10-ER, LEGe, ICP-MS and LSC. The mass spectrometric measurement calibrations were carried out using mono- and multielement standards. The efficiency calibrations were carried out using ^{55}Fe, ^{93m}Nb and multielement standard solutions or modified ^{3}H TDCR measurement. The uncertainty calculations were reported to be mainly combined uncertainties. Reported references included ^{93}Mo half-life information (Kajan et al., 2021), power moderate mean (Pomme & Keigtley, 2015), and radiochemistry (Shimada et al., 2014, Shimada & Kameo, 2016).

The preliminary ^{93}Mo analysis replicate results are shown in Figure 7. Sample 5 analyser assumed that the m/z signal is 100 % from ^{93}Mo and therefore, the approach clearly gives an overestimated result. When the Sample 5 result is excluded from the data set, the remaining results show a good alignment of results above limit of detection. Samples 4 and 6 are below limit of detection but correctly above the quantitative Sample 2, 7 and 9 results. Sample 10 analyser reported that limit of detection could not be reported as no reasonable LSC efficiencies were available.

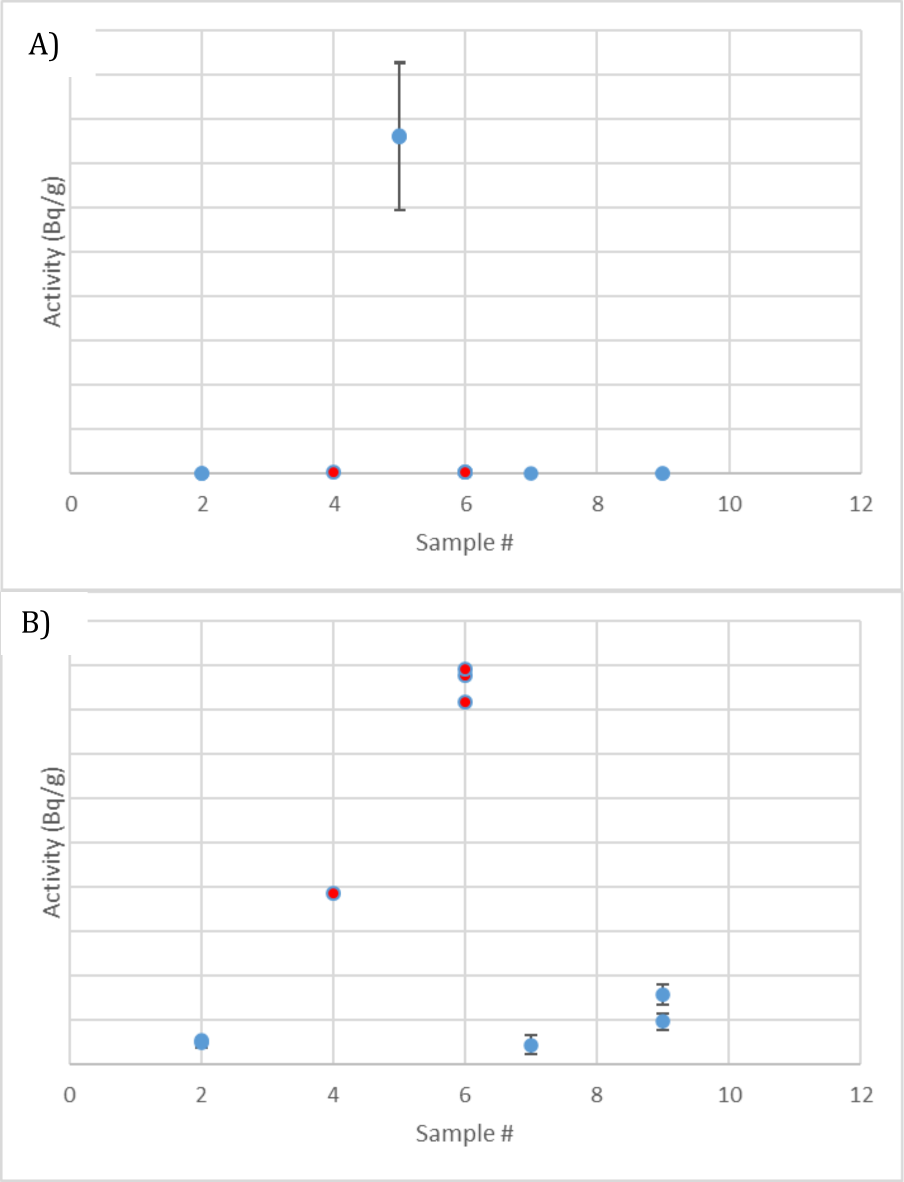

Figure 7. Preliminary ^{93}Mo results in high activity steel. Figure A with all results and Figure B with all except Sample 5 results. The uncertainties are stated with a coverage factor of $k = 2$. Analysis methodologies expressed in Table 8.

Table 8. Radiochemical ^{93}Mo analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8	9	10
Overview of the radiochemical method	-	See ^{55}Fe	-	Purification of 50µL of sample (dilute in 20mL HCl 0,3M) on cationic resin to eliminate ^{60}Co, then liquide-liquide extraction with ABO/CHCl$_3$, then NH$_4$OH. Add of stable Mo before separation to follow the extraction yield. Determination of the separation yield of ^{93}Mo by MP-AES.	Sample was diluted in 2% v/v HNO3 and 0.05% v/v HF and analyzed using ICP-MS. The reported result is a combination of analytes at mass-to-charge 93 (excluding ^{93}Zr) and thus interferences from ^{93}Nb and polyatomic interferences are still present.	0.2 mg of Nb, Zr, Mo were added in each aliquot sample. The aliquot samples were first evaporated to near dryness and dissolved in 0.1M HF. ^{93m}Nb, ^{94}Nb, ^{93}Zr, ^{93}Mo were separated using TEVA® ® Resin.	Mo is first precipitated with hydroxy-quinoline. After dissolution of the precipitate, a purification on alumina column is performed. A liquid-liquid extraction step with Aliquot 336 (methyltrioctylammonium chloride) is then applied. Finally, Mo is precipitated with hydroxy-quinoline	-	1.5 ml aliquot was evaporated to dryness in a teflon beaker, then converted to fluoride and dissolved in 20 ml 0.02 M HF. Co, Ni, Mn were separated on a cation resin column (14 cm, Ø 10 mm). The effluent was evaporated and dissolved in 60mL 4M HCl/0.25M Na$_2$SO$_3$ and passed through a DGA resin (25 mm, Ø 17 mm) column. Mo is stripped with 4M HNO$_3$ (after Nb removal). Mo is purified on Alumina column (50 mm, Ø 7 mm) loaded from 10 mL 1M HNO$_3$, Mo is stripped with 10 mL 25% NH$_3$. Mo source is prepared in 10 mL 0.1M HCl/0.01M HF.	^{93}Mo was purified in conjunction with ^{93}Zr, ^{93m}Nb and ^{94}Nb. Three aliquot samples were taken to contain approximately 4 mg of Fe in each aliquot according to the submitted initial chemical composition results. 1 mg of Mo, Zr and Nb was added in each aliquot sample. Additionally, an aliquot was taken for the original chemical composition studies using ICP-OES. The aliquot samples were first evaporated to near dryness and treated with conc. HF several times before dissolving in dilute HF. Mo was separated from cationic species using AG cation exchange resin. Mo fraction was purified using TEVA® resin.
Measurement information	-	Purified molybdenum fraction evaporated and dissolved in 5% HNO$_3$	-	Mixture of 9mL sample+1mL NH$_4$OH 4M in a plastic vial analyzed by X spectrometry (350 000s)	Dilution. Contribution of ^{93}Zr was omitted based on ^{93}Zr analysis results.	2 mL of purified Mo fraction were dropped onto 1-inch filter paper and were evaporated to dryness. Samples were measured by LEGe for 2 hours. Yield was determined by measuring the Nb	A mass of around 10 mg precipitate is finally obtained. Yield was determined by weighing the precipitate. X-spectrometry for ^{93}Mo.	-	Half of the purified Mo solution is mixed with 10 mL Gold star cocktail and measured by LSC, from the other half chemical recovery is determined by ICP-MS and aliquot is used for ICP-MS measurement of ^{93}Mo.	1 ml of purified ^{93}Mo fraction was mixed with 10 ml of HiSafe 3 liquid scintillation counting cocktail in a 20 ml glass vial. Samples were let to stabilise minimum of 16 hours to reduce luminescence. Samples were measured 2 times 3600s. Yield was determined by measuring the stable Mo content in the purified fraction.

						content with ICP-OES.				
Equipment	-	Agilent ICP-QQQ 8900	-	Canberra detector EGM-10-ER, measurement subcontacted to CEA LARC Laboratory	Agilent 7800 ICP-MS	LEGe CANBERRA GL0210	Ametek ICP-AES Spectro Blue for initial Mo content. Mettler Toledo XP 504 balance for final Mo content. Canberra LEGe GL0210: measurement during 400000s	-	Perkin Elmer TriCarb 2800, Agilent 8800 TripleQuad	Hidex 300 SL liquid scintillation counting. Agilent ICP-OES 5100 SVDV. Mettler Toledo AX 504 balance.
Efficiency calibration	-	Calibration with stable molybdenum and mass bias interpolated	-	^{93m}Nb	External calibration using monoelemental standard of natural Nb. Signal of ^{93}Nb was used to determine the mass fraction of analytes at mass-to-charge 93. A multielemental standard was used for quality control of the calibration.	Calibrated by ^{93m}Nb standard source	Equivalent geometry multi-nuclide efficiency calibration and sample/standard attenuation correction	-	LSC efficiency: approximately 0.2 calibrated by the use of ^{55}Fe standard solution. ICP-MS sensitivity is regarded equal to those of stable Mo isotopes (e.g., ^{98}Mo): 10E5 cps/ppb ^{98}Mo	With LSC could not be determined from TDCR with CoreF
Description of uncertainty calculations	-	Complex, but based on measurement uncertaint	-	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty including sample preparation and	Combined uncertainty with the sample preparation and measurement uncertainties.	Combined uncertainty with the sample preparation, recovery yield and measurement uncertainties	-	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties

		y (varies), yield (varies) and half-life (20%). Means calculated as power moderated mean (see note).			measurement uncertainty	(including uncertainty of the counting rates, counting efficiency, weights, pipets, and the chemical recovery)				

5.6 Preliminary radiochemical ^{93}Zr results

The sample specific ^{93}Zr analysis methodologies are reported in Table 9. The overview of the radiochemical methods included resin treatments (i.e., UTEVA®, TK400®, AG cation exchange) and evaporations. The sample measurements were carried out in liquid form. The measurements were carried out using mainly LSC but also mass spectrometry. The mass spectrometric measurement calibrations were carried out using monoelement standards. The LSC efficiency calibrations were carried out using ^{63}Ni surrogate or ^{63}Ni TDCR measurement. The uncertainty calculations were reported to be mainly combined uncertainties. One reference on radiochemistry was reported (Shimada & Kameo, 2016).

The preliminary ^{93}Zr analysis replicate results are shown in Figure 8. The results did not show a clear trend. Actually, the results seem to form two sets of results i.e., Samples 5 and 6 in one group and Samples 4, 9 and 10 in the other group.

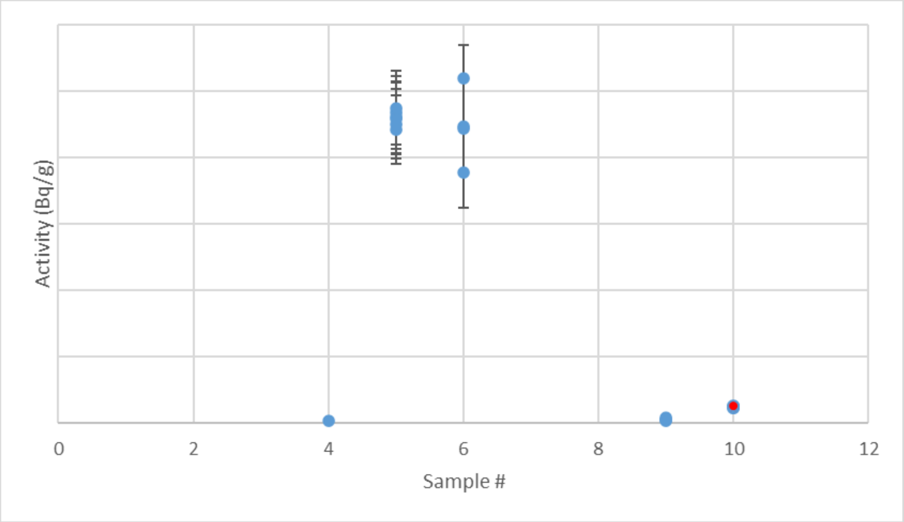

Figure 8. Preliminary ^{93}Zr results in high activity steel. The uncertainties are stated with a coverage factor of $k = 2$. Red dot indicates a result under limit of detection. Analysis methodologies expressed in Table 9.

Table 9. Radiochemical ^{93}Zr analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8	9	10
Overview of the radiochemical method	-	See ^{55}Fe	-	Purification of 2mL aliquot (from a mixture of 1mL sample and 1,5mL HNO^{3} 9M/1ppm of ^{90}Zr) on UTEVA® resin and TK400® resin (from Triskem). Determination of the separation yield of ^{90}Zr by QQQ-ICP-MS	Zr was separated and purified using an ion exchange resin. Sample was diluted in 2% v/v HNO_3 and 0.05% v/v HF and analyzed using ICP-MS.	0.2 mg of Nb, Zr, Mo were added in each aliquot sample. The aliquot samples were first evaporated to near dryness and dissolved in 0.1M HF. ^{93m}Nb, ^{94}Nb, ^{93}Zr, ^{93}Mo were separated using TEVA® resin.	-	-	Purification of 2mL aliquot (from a mixture of 1mL sample and 1,5mL HNO_3 9M/1ppm of ^{90}Zr) on UTEVA® resin and TK400® resin (from Triskem). Determination of the separation yield of ^{90}Zr by QQQ-ICP-MS	^{93}Zr was purified in conjunction with ^{93}Mo, ^{93m}Nb and ^{94}Nb. Three aliquot samples were taken to contain approximately 4 mg of Fe in each aliquot according to the submitted initial chemical composition results. 1 mg of Zr, Mo and Nb was added in each aliquot sample. Additionally, an aliquot was taken for the original chemical composition studies using ICP-OES. The aliquot samples were first evaporated to near dryness and treated with conc. HF several times before dissolving in dilute HF. Zr was separated from cationic species using AG cation exchange resin. Zr fraction was purified using TEVA® resin.
Measurement information	-	See ^{55}Fe	-	Mixture of 8mL sample and 12mL UltimaGold AB liquid scintillation counting cocktail in a 20 mL plastic vial. Sample were let to stabilise minimum of 1 hour to reduce luminescense. Samples were measured 2 times 6h	m/z 93	1 mL of purified Zr fraction was mixed with 15 mL Pico-Flour LLT and 4 mL n-hexane. Samples were measured by LSC for 30 minutes. Yield was determined by measuring the Zr content with ICP-OES.	-	-	Mixture of 8mL sample and 12mL UltimaGold AB liquid scintillation counting cocktail in a 20 mL plastic vial. Sample were let to stabilise minimum of 1 hour to reduce luminescense. Samples were measured 2 times 6h	1 ml of purified Zr-93 fraction was mixed with 10 ml of HiSafe 3 liquid scintillation counting cocktail in a 20 ml glass vial. Samples were let to stabilise minimum of 16 hours to reduce luminescence. Samples were measured 3 times 3600s. Yield was determined by measuring the stable Zr content in the purified fraction.
Equipment	-	See ^{55}Fe	-	^{63}Ni surrogate efficiency curve	External calibration using monoelemental standard of natural Nb. A multielemental standard was	^{63}Ni surrogate efficiency curve	-	-	^{63}Ni surrogate efficiency curve	Approximately 0.8 derived from ^{63}Ni TDCR with CoreF

					used for quality control of the calibration.					

5.7 Preliminary radiochemical ^{94}Nb results

The sample specific ^{94}Nb analysis methodologies are reported in Table 10. The overview of the radiochemical methods included resin treatments (i.e., ion exchange, TEVA®, DGA®) and evaporations. The sample measurements were carried out in liquid form using gamma spectrometry (i.e., BeGe, HPGe). The efficiency calibrations were carried out using ISOCS or ^{152}Eu and mixed sources. The uncertainty calculations were reported to be mainly combined uncertainties. One reference on radiochemistry was reported (Shimada & Kameo, 2016).

The preliminary ^{94}Nb analysis replicate results are shown in Figure 9. The Sample 2 analyser reported that ^{94}Nb was not detected in the original solution using gamma spectrometry due to high Compton background from ^{60}Co. Additionally, the Sample 10 results show the clear benefit in purification of ^{94}Nb as the directly from the original sample analysis result is below limit of detection whereas quantitative results were obtained in the purified fraction. The ^{94}Nb results in purified fractions show a relatively good trend. Sample 2 analyser reported that mass spectrometric analysis was not carried out due to large difference between predicted and observed values for ^{93}Nb.

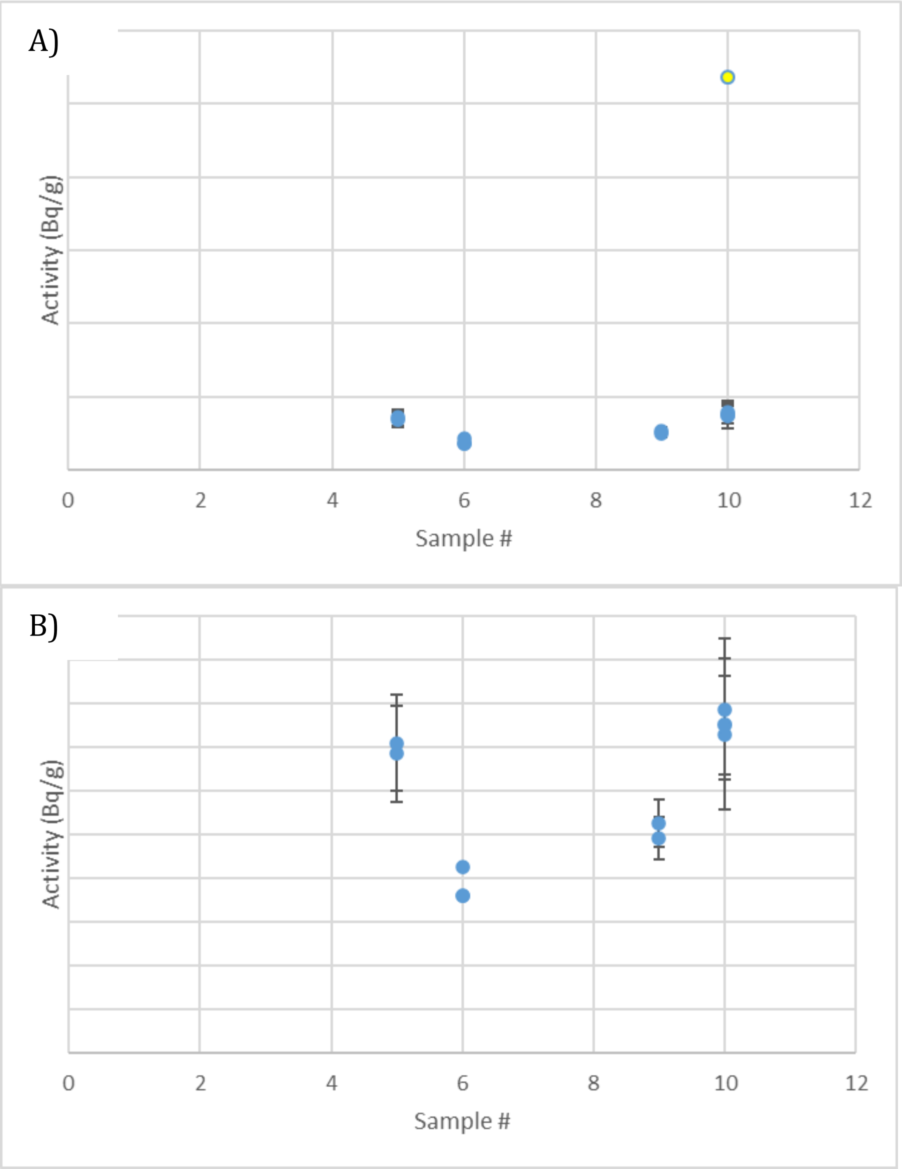

Figure 9. Preliminary ^{94}Nb results in high activity steel. The uncertainties are stated with a coverage factor of $k = 2$. Yellow dot indicates analysis result which was carried out in original solution without radiochemical purifications. Figure A shows all results and figure B results for radiochemically purified samples. Analysis methodologies expressed in Table 10.

Table 10. Radiochemical ^{94}Nb analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8	9	10
Overview of the radiochemical method	-	See ^{55}Fe	-	-	^{94}Nb was separated on an ion exchange resin. Separation yield was determined by measurement of natural Nb before and after separation.	0.2 mg of Nb, Zr, Mo were added in each aliquot sample. The aliquot samples were first evaporated to near dryness and dissolved in 0.1M HF. ^{93m}Nb, ^{94}Nb, ^{93}Zr, ^{93}Mo were separated using TEVA® Resin.	-	-	1.5 ml aliquot was evaporated to dryness in a teflon beaker, then converted to fluoride and dissolved in 20 ml 0.02 M HF. Co, Ni, Mn were separated on a cation resin column (14 cm, Ø 10 mm). The effluent was evaporated and dissolved in 60mL 4M HCl/0.25M Na_2SO_3 and passed through a DGA resin (25 mm, Ø 17 mm) column. Nb gets into the effluent. Nb is purified on TEVA® column (40 mm, Ø 7 mm) loaded from 20 mL 11M HF, Nb is stripped with 10 mL 7M HNO_3. Nb source is prepared in 10 mL 0.1M HCl/0.01M HF.	^{94}Nb was purified in conjunction with ^{93}Mo and ^{93}Zr. Three aliquot samples were taken to contain approximately 4 mg of Fe in each aliquot according to the submitted initial chemical composition results. 1 mg of Nb, Zr and Mo was added in each aliquot sample. Additionally, an aliquot was taken for the original chemical composition studies using ICP-OES. The aliquot samples were first evaporated to near dryness and treated with conc. HF several times before dissolving in dilute HF. Nb was separated from cationic species using AG cation exchange resin. Nb fraction was purified using TEVA® resin.
Measurement information	-	See ^{55}Fe	-	-	Analysis with gamma spec after separation; determination of separation yield by ICP-MS	Purified Nb was dispensed to HDPE bottle. Samples were measured by HPGe for 16 hours. Yield was determined by measuring the Nb content with ICP-OES.	-	-	The solution is measured in LSC vial by gamma spectrometry. Aliquot is taken to determine chemical recovery by ICP-MS.	Total purified fraction (20 ml LSC vial) measured with a gamma spectrometer at a distance of 3-4 cm. Measurement times 45 h, 51 h, 100 h.
Equipment	-	See ^{55}Fe	-	-	Mirion BeGe detector BE3830; 35% efficiency	HPGe MIRION GC4020	-	-	HPGe detector attached to Canberra 16k MCA with Genie software	Mirion HPGe. Agilent ICP-OES 5100 SVDV. Mettler Toledo AX 504 balance.
Efficiency calibration	-	See ^{55}Fe	-	-	efficiency calibrated using a mixed gamma calibration standard (LEA)	Calibrated by Eu-152 standard source.	-	-	Efficiency calculation was performed with a mixed standard solution.	ISOCS efficiency calibration using Geometry composer. ISOXSRCE checks regularly.

Description of uncertainty calculations	-	See ^{55}Fe	-	-	combined uncertainty: sample prep, efficiency, measurement uncertainty	Combined uncertainty with the sample preparation and measurement uncertainties. (including uncertainty of the counting rates, counting efficiency, weights, pipets, and the chemical recovery)	-	-	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties

5.8 Preliminary radiochemical ^{93m}Nb results

The sample specific ^{93m}Nb analysis methodologies are reported in Table 11. The overview of the radiochemical methods included dilutions, evaporations, and resin treatments (i.e., AG cation exchange, TEVA®). The sample measurements were carried out in liquid and solid forms (evaporate residue). The detections were carried out using mass spectrometry, gamma spectrometry and LSC. The LSC efficiency calibration was carried out using ^{93m}Nb standard solution, the gamma spectrometric LEGe efficiency calibration was carried out using a ^{93m}Nb standard source. The mass spectrometric calibration was carried out using a monoelemental standard. The uncertainty calculations were reported to be mainly combined uncertainties. One reference on radiochemistry was reported (Shimada & Kameo, 2016).

The preliminary ^{93m}Nb analysis replicate results are shown in Figure 10. The Sample 2 analyses reported that ^{93m}Nb was not possible to be measured using QQQ-ICP-MS and Sample 5 analyser selected not to report their results due to interfering nuclides still being present in the sample. The remaining two analysis results seem to be comparable.

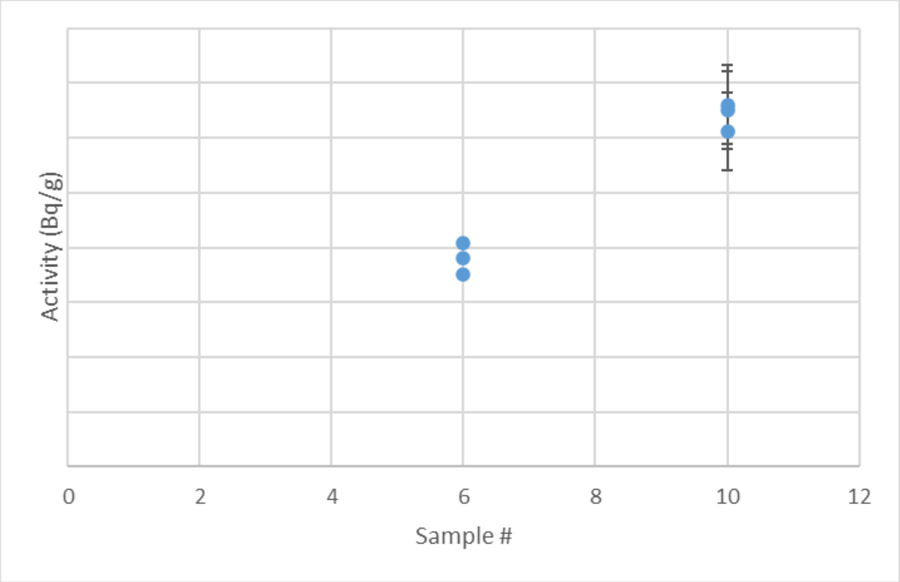

Figure 10. Preliminary ^{93m}Nb results in high activity steel. The uncertainties are stated with a coverage factor of *k = 2*. Analysis methodologies expressed in Table 11.

Table 11. Radiochemical ^{93m}Nb analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8	9	10
Overview of the radiochemical method	-	See ^{55}Fe	-	-	-	0.2 mg of Nb, Zr, Mo were added in each aliquot sample. The aliquot samples were first evaporated to near dryness and dissolved in 0.1M HF. ^{93m}Nb, ^{94}Nb, ^{93}Zr, ^{93}Mo were separated using TEVA® ® Resin.	-	-	-	^{93m}Nb was purified in conjunction with ^{93}Mo and ^{93}Zr. Three aliquot samples were taken to contain approximately 4 mg of Fe in each aliquot according to the submitted initial chemical composition results. 1 mg of Nb, Zr and Mo was added in each aliquot sample. Additionally, an aliquot was taken for the original chemical composition studies using ICP-OES. The aliquot samples were first evaporated to near dryness and treated with conc. HF several times before dissolving in dilute HF. Nb was separated from cationic species using AG cation exchange resin. Nb fraction was purified using TEVA® resin.
Measurement information	-	See ^{55}Fe	-	-	-	1 mL of purified Nb fraction were dropped onto 1-inch filter paper and were evaporated to dryness. Samples were measured by LEGe for 16 hours. Yield was determined by measuring the Nb content with ICP-OES.	-	-	-	2 ml of purified Nb fraction was mixed with 10 ml of HiSafe 3 liquid scintillation counting cocktail in a 20 ml glass vial. Samples were let to stabilise minimum of 12 hours to reduce luminescence. Samples were measured 3 times 14400 s. Yield was determined by measuring the stable Nb content in the purified fraction.
Equipment	-	See ^{55}Fe	-	-	-	LEGe CANBERRA GL0210	-	-	-	Hidex 300 SL liquid scintillation counting. Agilent ICP-OES 5100 SVDV. Mettler Toledo AX 504 balance.
Efficiency calibration	-	See ^{55}Fe	-	-	-	Calibrated by ^{93m}Nb standard source	-	-	-	^{93m}Nb standard QPE CPM curve (external standard efficiency calibration).
Description of uncertainty calculations	-	See ^{55}Fe	-	-	-	Combined uncertainty with the sample preparation and measurement uncertainties. (including uncertainty of the counting rates, counting efficiency, weights, pipets, and the chemical recovery)	-	-	-	Combined uncertainty with the sample preparation and measurement uncertainties

5.9 Preliminary meeting conclusions

The preliminary meeting discussions regarding ^{93}Mo and ^{93}Zr results concluded that the scattering of the results may originate from different interferences in the measurement phase. Partners were encouraged to share the spectra (both LSC and mass spectrometry) to discuss the topic in the final meeting. Additionally, use of correct half-lives is essential in mass spectrometric analyses. A new ^{93}Mo half-life has been published and should be used in calculations. The uncertainty on the half-lives of ^{59}Ni and ^{93}Zr are both relatively large (6.6% and 3.8% at k = *1* respectively) and therefore increase measurement uncertainly accordingly when converting between mass and activity values. Supplementary information on the half-lives given to partners are listed in Table 12.

Table 12. Half-lives of the DTMs studied in MoNi.

Half-lives				
	Value (y)	**u(Value) (k = *1*)**	**± %**	**Reference**
^{55}Fe	2.747	0.008	0.29 %	http://www.lnhb.fr/nuclides/Fe-55_tables.pdf
^{59}Ni	76000	5000	6.58 %	http://www.lnhb.fr/nuclides/Ni-59_tables.pdf
^{63}Ni	98.7	2.4	2.43 %	http://www.lnhb.fr/nuclides/Ni-63_tables.pdf
^{93}Zr	1610000	60000	3.73 %	http://www.lnhb.fr/nuclides/Zr-93_tables.pdf
^{93m}Nb	16,12	0,15	0.93 %	http://www.lnhb.fr/nuclides/Nb-93m_tables.pdf
^{94}Nb	20300	1600	7.88 %	https://doi.org/10.1016/j.nds.2006.08.001
^{93}Mo	4839	63	1.30 %	https://doi.org/10.1016/j.nds.2011.04.001

Some partners had reported that the indicative elemental analysis results shared in the beginning of the analyses seemed to have an error in the stable Nb results. Therefore, each partner was encouraged to report their elemental analysis results, which would be viewed in the final meeting.

Some partners reported that the amount of sample was not large enough to carry out all the analyses and better results would have been achieved with larger sample amount.

The ^{60}Co and ^{55}Fe results were assumed to be better aligned as these analyses have been carried out in several intercomparisons and the sample was homogenised.

6 Methodology for statistical analysis of the reported results

The statistical analysis of the reported results was carried out according to 3rd version of the ISO 13528 (ISO, 2022) standard on Statistical methods for use in proficiency testing by interlaboratory comparison. The third version did not significantly differ from the second version which had been followed in the previous exercises.

A robust statistical method was utilised for calculation of assigned values based on the participants' results (ISO, 2022). Robust mean and robust standard deviation were calculated using Algorithm A, which is robust for outliers, when the expected proportion of outliers is less than 20% (ISO, 2022). Performance assessment was carried out using z score (Eq. 1) [International Standard 2015]. The analysis results with z score were (ISO, 2022);

- acceptable when $|z| \leq 2.0$
- a warning signal was given for results with $2.0 < |z| < 3.0$
- results were unacceptable when $|z| \geq 3.0$

$$z_i = \frac{(x_i - x_{pt})}{\sigma_{pt}} \qquad (1)$$

Where

x_{pt}: the assigned value

σ_{pt}: standard deviation for the proficiency assessment

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 (ISO, 2022). 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} (ISO, 2022).

$$u(x_{pt}) = \frac{1.25 \cdot s^*}{\sqrt{p}} \qquad (2)$$

Where

s^*: robust standard deviation of the results

p: number of samples

7 Final results

The partners had an opportunity revise their results between the preliminary and final meeting in November. The revisions included:

- Elemental analysis results were submitted for Samples 1, 2, 4, 5, 7, 8, and 10. In couple of cases the units and calculations were checked with the partner.
- New set of results were submitted for Samples 1 and 8 (see Table 13 and Table 14).
- Corrections for Sample 4 ^{59}Ni results were attempted without success. Details of the attempts were not submitted by the finalisation of this report.
- Revision of ^{93}Mo results were carried out for Sample 5. The initial reported value of ^{93}Mo which was presented in the preliminary meeting included other isobaric and polyatomic interferences at mass-to-charge 93 (since it was analysed using ICP-MS). The sample has then been subjected to ion exchange chromatography to remove isobaric interferences. This reduced the result significantly due to the removal of the ^{93}Nb interference, however, polyatomic interferences were still present. Therefore, our reviewed result of ^{93}Mo is still higher than those reported by other laboratories.
- An error in the dilution ratio for Sample 6 ^{55}Fe and ^{63}Ni results were noticed in review of the data after the preliminary meeting. The results were corrected with the correct dilution ratio.
- Revision of ^{59}Ni results and new entry of ^{93m}Nb results were carried out for Sample 9. After the preliminary meeting results, it was suspected that the old efficiency calibration source for ^{59}Ni was deteriorated and therefore, a new surrogate source was prepared. ^{55}Fe source was prepared by evaporating a ^{55}Fe standard solution on a membrane. The source was covered with the plastic foil and placed in the standard geometry close to the detector. Efficiency value was corrected for the differences in the emission probabilities of ^{59}Ni and ^{55}Fe. An aliquot of 0.618 g of Sample 9 was evaporated to dryness, then evaporated with 2 times 2 ml 37 % HCl, and redissolved in 10 ml 6 M HCl. 100 ul aliquot was taken and diluted to 10 mL for total Ni analysis (i.e., yield determination) with MP-AES. Fe was extracted with a MIBK resin. Ni was separated in a DMG resin. The Ni fraction was evaporated, re-dissolved in 25 ml 1 M HCl, and Ni was precipitated with DMG. The $Ni(DMG)_2$ was filtered on a 25 mm 0.45 µm membrane. The precipitate was dried and covered with a plastic foil, and was used as a ^{59}Ni source for LEGe gamma spectrometry. After gamma-spectrometric measurement, the Ni(DMG)2 precipitate was dissolved and evaporated in 3 times 2 ml 65 % HNO_3. The residue was dissolved in 2 ml 1M HNO_3. 200 µl was taken out and diluted to 10 ml with 1 M HNO_3 for total Ni determination (chemical yield) with MP-AES. The ^{93m}Nb results were revised and efficiency calibration source was prepared by evaporating ^{93m}Nb standard solution on a filter membrane. Samples for ^{93m}Nb and ^{94}Nb determination were chemically processed together using a cation exchange column, a DGA column and TEVA column. After measurement of ^{94}Nb by gamma spectrometry and determining the chemical recovery by ICP-MS, the solution was evaporated to dryness and the residue taken up with drops of 1:1 HF was evaporated as a point source.

Statistical analyses were carried out for results which had at least five data entries. In some cases, indicative statistical analyses were carried out for results which had four data entries. Other results were reported in graphs and tables.

Table 13. Updated radiochemical yields and uncertainties (*k = 2*). Yields discussed in the preliminary meeting are in Table 3.

Yield ± uncertainty [%] (*k = 2*)							
	^{55}Fe	**^{63}Ni**	**^{59}Ni**	**^{93}Mo**	**^{93}Zr**	**^{94}Nb**	**^{93m}Nb**
1	95 ± 10	66 ± 7	-	-	-	-	-
8	-	-	102 ± 19	100 ± 10	-	-	-
9	-	-	93 ± 5	-	-	-	~65-85

Table 14. Updated radiochemical methods for the final meeting. The radiochemical methodologies discussed in the preliminary meeting are in Table 5, Table 6, Table 7, Table 8, Table 9, Table 10, and Table 11.

	1	1	5	8	8	8	8	9	9
	^{55}Fe	^{63}Ni	^{93}Mo	^{55}Fe	^{63}Ni	^{59}Ni	^{93}Mo	^{59}Ni	^{93m}Ni
Overview of the radiochemical method	^{55}Fe was purified in conjunction with ^{63}Ni. One aliquot sample of ca. 1.5 g was taken for the radiochemical analysis. Additionally, an aliquot (0.1 g) was taken for the original chemical composition studies and measured using ICP-OES. The aliquot sample was precpitated with NaOH, then dissolved in 9M HCl. ^{55}Fe was separated from ^{63}Ni and purified using AG ion exchange resin.	^{55}Fe was purified in conjunction with ^{63}Ni. One aliquot sample of ca. 1.5 g was taken for the radiochemical analysis. Additionally, an aliquot (0.1 g) was taken for the original chemical composition studies and measured using ICP-OES. The aliquot sample was precpitated with NaOH, then dissolved in 9M HCl. ^{55}Fe was separated from ^{63}Ni and purified using AG ion exchange resin. ^{63}Ni	Mo was separated and purified using an ion exchange resin. Sample was diluted in 2% v/v HNO3 and 0.05% v/v HF and analyzed using ICP-MS.	Fe fraction was separated in conjunction with Ni and Mo fractions. First NaOH was used to precipitate the elements of interest, then the precipitate was kept and dissolved in 9 M HCl. The solution was then loaded to AG1x4 anion exchange column. The column was first washed with 4 M HCl (to remove the interfering elements) and Fe fraction was eluted using 0.5 M HCl. Solution was evaporated to dryness and redissolved to 1 M H_3PO_4	Ni fraction was separated in conjunction with Fe and Mo fractions. First NaOH was used to precipitate the elements of interest, then the precipitate was kept and dissolved in 9 M HCl. The solution was then loaded to AG1x4 anion exchange column (to remove interfering elements) and the elute passing through was collected as Ni fraction. Solution was then evaporated to dryness, dissolved with 1 M HCl, ammonium citrate was added and pH was adjusted to 9 with ammoniumhydroxide. Then the fraction was further purified using Ni columns from which the Ni fraction was eluted using 3 M HNO_3. An aliquot of the eluted solution was then mixed with	Ni fraction was separated in conjunction with Fe and Mo fractions. First NaOH was used to precipitate the elements of interest, then the precipitate was kept and dissolved in 9 M HCl. The solution was then loaded to AG1x4 anion exchange column (to remove interfering elements) and the elute passing through was collected as Ni fraction. Solution was then evaporated to dryness, dissolved with 1 M HCl, ammonium citrate was added and pH was adjusted to 9 with ammoniumhydroxide. Then the fraction was further purified using Ni columns from which the Ni fraction was eluted using 3 M HNO_3. The eluted solution was measured by ICP-MS	Mo fraction was separated in conjunction with Fe and Ni fractions. First NaOH was used to precipitate the elements of interest, then the precpitate was kept and dissolved in 9 M HCl. The solution was then loaded to AG1x4 anion exchange column (to remove interfering elements) and the Mo fraction was eluted using 8 M HNO_3. Solution was then evaporated to approx 0.5	^{59}Ni and ^{63}Ni were chemically processed together as long as the Ni-DMG2 precipitate was formed.	Samples for ^{93m}Nb and ^{94}Nb determination were chemically processed together using a cation exchange column, a DGA column and TEVA column (see at ^{94}Nb). After measurement of ^{94}Nb by gamma spectrometry and determining the chemical recovery by ICP-MS, the solution was evaporated to dryness and the residue taken up with drops of 1:1HF was evaporated as

		fraction was purified using Ni-resin.		and mixed with Ultima Gold LLT. Stable Fe was measured using ICP-MS.	Ultima Gold LLT. Stable Ni was measured using ICP-MS.	both for stable Ni and ^{59}Ni.	mL and transferred to LSC vials, which were measured with HPGe. Afterwards, the solution was evaporated to dryness and the acid composition was adjusted to 6.5 M HCl/0.1M HF. Then the solution was applied to TBP column which was then washed with the same acid mixture. Mo was eluted using MQ-water. The collected solution was again evaporated to dryness and redissolved to 0.1 M HF. The solution was applied to alumina		a point source.

							column which was washed with 0.1 M HF. Then Mo was eluted using 20 % NH_4OH. The eluted solution was measured by ICP-MS both for stable Mo and ^{93}Mo.		
Measurement information	5 ml of purified ^{55}Fe fraction was mixed with 15 ml of Ultima Gold liquid scintillaiton counting cocktail in a 20 ml plastic vial. Samples were let to stabilise minimum of 12 hours to reduce luminescense. Samples were measured 3 times 3600s. Yield was determined by measuring the stable Fe content in the	5 ml of purified ^{63}Ni fraction was mixed with 15 ml of Ultima Gold liquid scintillaiton counting cocktail in a 20 ml plastic vial. Samples were let to stabilise minimum of 12 hours to reduce luminescense. Samples were measured 3 times 3600s. Yield was determined by measuring the stable Ni content in the	m/z 93	Regrettably, the instrument was non-functional at the time the measurements could have been conducted.	Regrettably, the instrument was non-functional at the time the measurements could have been conducted. NB: not possible to measure by ICP-MS due to the interference from ^{63}Cu	Purified Ni fraction in 3 M HNO_3 was evaporated to near dryness and diluted to 10 mL using MilliQ water. Further 10 and 100 times dilutions were done for ICP-MS using 2 % HNO_3. NB: some mass 59 signal in the reference material that should not have any ^{59}Ni but may have traces of ^{59}Co or there may be an overlap from the large mass 58 peak. Might be best to consider this a maximum activity.	Purified Mo fraction was evaporated to dryness and redissolved to 10 mL of 2 % HNO_3. Further 10 and 100 times dilutions were done for ICP-MS using 2 % HNO_3. NB: high RSD for mass 93. Interferences from ^{92}Zr and ^{94}Zr, corrected using for Zr	The Ni-DMG2 precipitate was filtered on a membrane and covered after drying with a plastic foil, that was placed in a standard geometry close (about 2 cm) to the detector. Counting time was 234000 s with dead time of 0.09%	^{93m}Nb was detected by X-ray spectrometry.

	purified fraction by ICP-OES.	purified fraction by ICP-OES.					using ^{90}Zr. Interferences in ICP-MS results and due to that it was not possible to get reliable results.		
Equipment	Quantulus 1220	Quantulus 1220	Agilent 7800 ICP-MS	Hidex Ulla Ultra Low Level Analyzer	Hidex Ulla Ultra Low Level Analyzer	Thermo Fisher Element Series HR-ICP-MS	Thermo Fisher Element Series HR-ICP-MS	LE HPGe detector with 25 µm Be window attached to Canberra 16k MCA with Genie software	LE HPGe detector with 25 µm Be window attached to Canberra 16k MCA with Genie software
Efficiency calibration	Quench curve using ^{55}Fe standards	Standard addition method using ^{63}Ni standard	External calibration using monoelemental standard. A multielemental standard was used for quality control of the calibration.			Calibration with stable Ni standard solution and mass bias interpolation	ICP-MS: Calibration with stable Ni standard solution and mass bias interpolation	Counting efficiency was approximately 0.03. ^{55}Fe source was prepared by evaporating a ^{55}Fe standard solution on a membrane. The source was covered with the plastic foil and was placed in the standard geometry close to the detector.	Efficiency about 0.02, it was determined with a standard - ^{93m}Nb point source

								Efficiency value was corrected for the differences in the emission probabilities of ^{59}Ni and ^{55}Fe.	
Description of uncertainty calculations	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty including sample preparation and measurement uncertainty			Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties

Table 15. Updated ETM analysis methodologies. The ETM methodologies discussed in the preliminary meeting are in Table 4.

	1	**8**
Sample preparation	Subsample fraction (0.1g) was taken and measured after dilution for 80 times.	Sample was measured as received using the provided mass information
Measurement geometry	Sample was measured directly on the endcap of the detector	Sample was measured once at 10 mm distance on top of the detector
Dead time	0.4%	12.69%
Measurement time	152878 seconds	2695 s
Detector type and efficiency	Mirion BE3830 7500SL-RDC-6-ULB (rel. Eff. ~35%)	Canberra HPGe detector, 31 %
Efficiency calibration	Eckert & Ziegler 7503ML solution + EFFTRAN for efficiency transfer and coincidence summing corrections	Efficiency calibration was performed using a mixed standard solution.
Description of uncertainty calculations	Combined uncertainty with the sample preparation and measurement uncertainties	Combined uncertainty with the sample preparation and measurement

7.1 Gamma spectrometry results

Statistical analysis of ^{60}Co results were carried out with nine data entries. The assigned value with the robust standard deviation (k = *1*) was 491 ± 53 kBq g^{-1}. The data entries with the assigned value and uncertainties (k = *2*) are shown in Figure 11. The z score was calculated using the robust standard deviation (10.7 %). As seen in Table 16, all ^{60}Co z scores were in acceptable range.

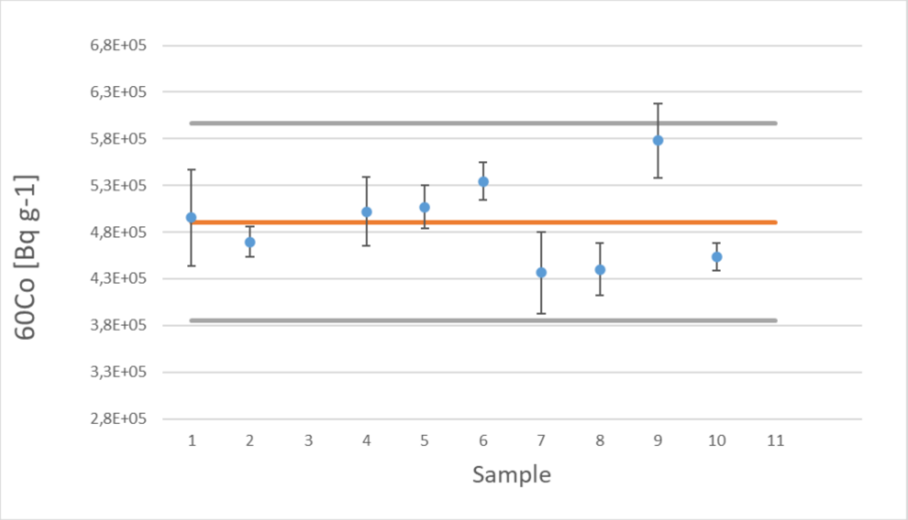

Figure 11. Final ^{60}Co results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of k = *2*.

Table 16. ETM z scores in MoNi intercomparison exercise. Results are acceptable when z≤2.0, warning signal is given when 2.0<z<3.0, and unacceptable when z≥3.0.

Sample	z score
	^{60}Co
1	0.1
2	0.4
3	-
4	0.2
5	0.3
6	0.8
7	1.0
8	1.0
9	1.7
10	0.7

7.2 Radiochemical analysis results

Statistical analysis of ^{55}Fe results were carried out with eight data entries. The assigned value with the robust standard deviation (k = *1*) was 380 ± 120 kBq g^{-1}. The data entries with the assigned value and uncertainties (k = *2*) are shown in Figure 12. The z score was calculated using the standard uncertainty of assigned value (13.2 %) since the robust standard deviation was large (29.8 %). As seen in Table 17, one ^{55}Fe z score (Sample 2) is in unacceptable range, two results (Samples 5 and 9) in warning signal range and all the other entries in acceptable range.

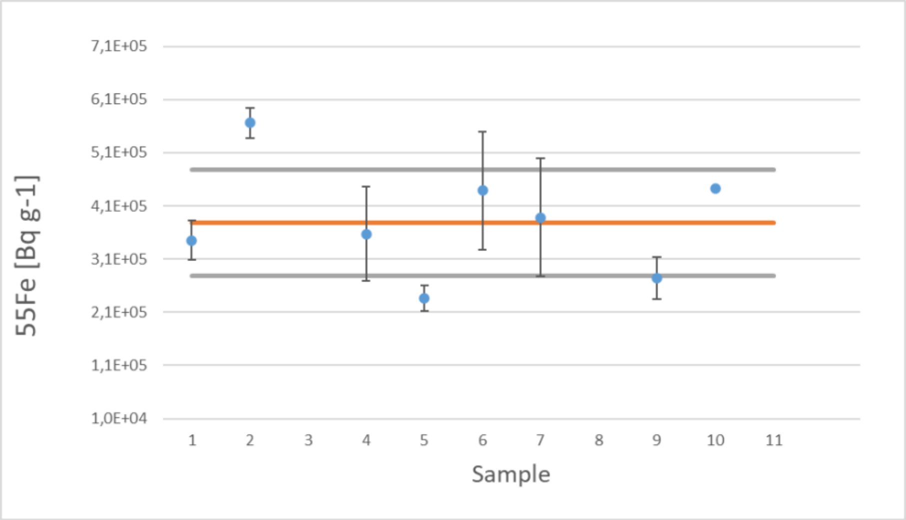

Figure 12. Final ^{55}Fe results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of *k = 2*.

Table 17. DTM z scores in MoNi intercomparison exercise. Results are acceptable when z≤2.0, warning signal is given when 2.0<z<3.0, and unacceptable when z≥3.0.

Sample	z score			
	^{55}Fe	^{63}Ni	^{59}Ni	^{94}Nb
1	0.7	1.9	-	-
2	3.8	-	-	-
3	-	-	-	-
4	0.4	0.4	-	-
5	2.9	2.3	-	0.9
6	1.2	2.5	1.1	1.7
7	0.2	0.7	0.4	
8	-	-	-	
9	2.1	2.3	1.1	0.6
10	1.3	0.3	1.8	1.4

Statistical analysis of ^{63}Ni results were carried out with seven data entries. The assigned value with the robust standard deviation (*k = 1*) was 830 ± 210 kBq g^{-1}. The data entries with the assigned value and uncertainties (*k = 2*) are shown in Figure 13. The z score was calculated using the standard uncertainty of assigned value (11.8 %) since the robust standard deviation was large (25.0 %). As seen in Table 17, three ^{63}Ni z scores (Samples 5, 6 and 9) are in warning signal range and all the other entries in acceptable range.

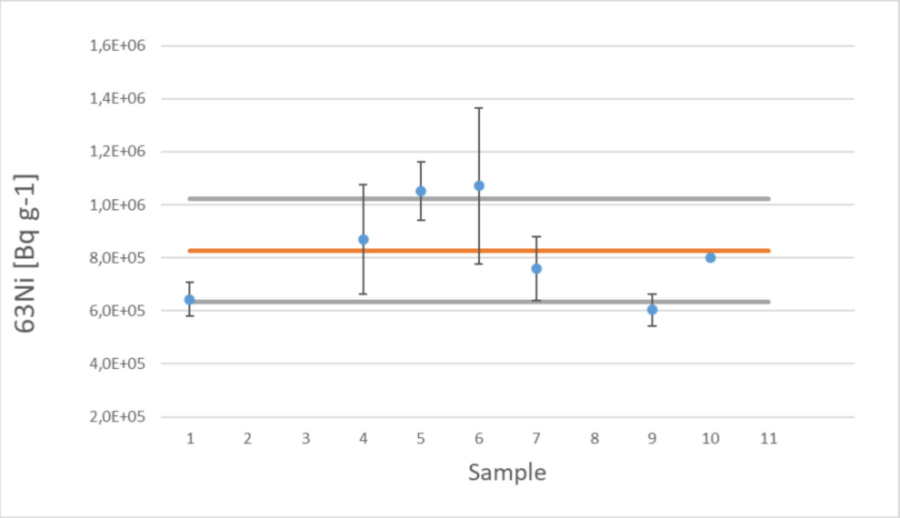

Figure 13. Final ^{63}Ni results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of *k = 2*.

Indicative statistical analysis of ^{59}Ni results were carried out with four data entries. In total, six data entries had been submitted. However, Sample 8 was a clear outlier whereas Sample 4 was below limit of detection. The assigned value with the robust standard deviation (*k = 1*) was 9.2 ± 2.5 kBq g^{-1}. The data entries with the assigned value and uncertainties (*k = 2*) are shown in Figure 14. The z score was calculated using the standard uncertainty of assigned value (16.9 %) since the robust standard deviation was large (27.1 %). As seen in Table 17, all ^{59}Ni z scores are in acceptable range. However, it must be emphasised that the assigned value was calculated from only four data entries.

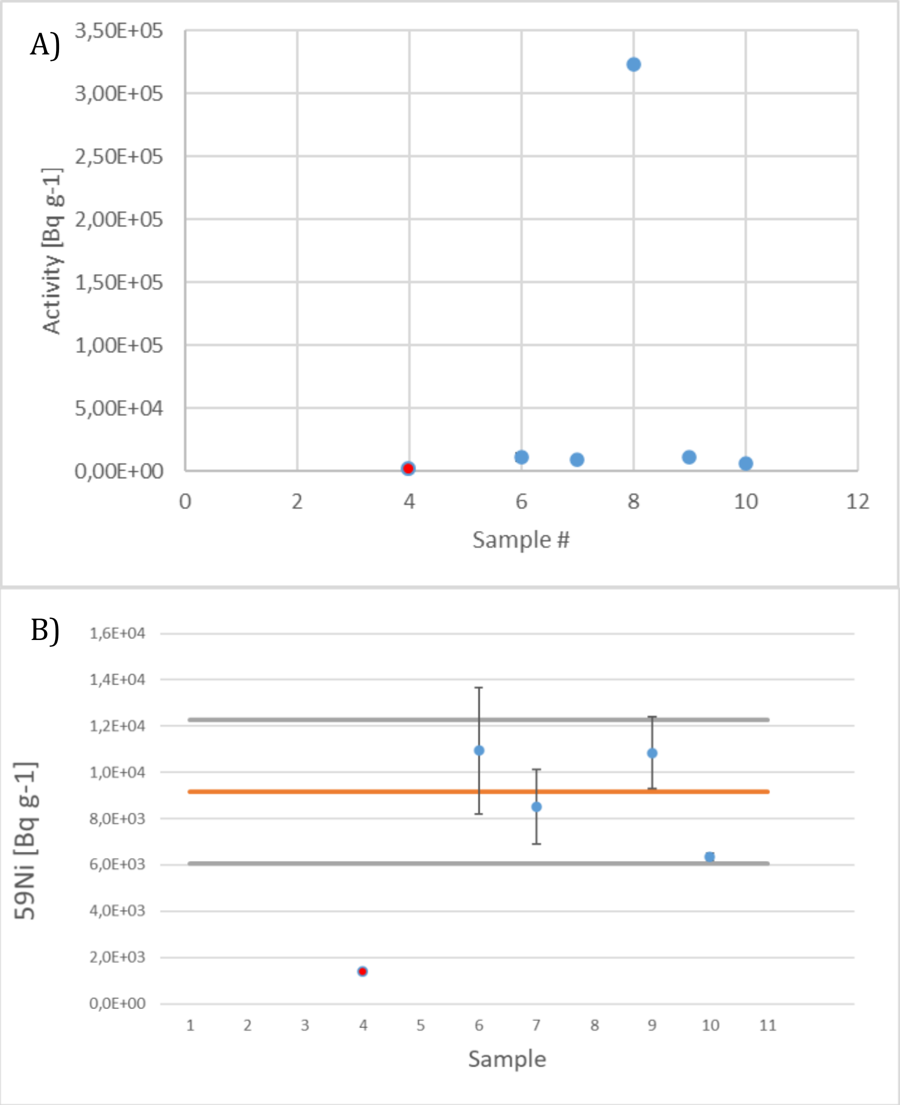

Figure 14. Final ^{59}Ni results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Figure A shows all results and figure B without Sample 8 result.

Statistical analysis of ^{93}Mo results was not carried out due to low number of data entries above limit of detection. In total, six data entries had been submitted. However, Sample 5 was a clear outlier whereas Samples 4 and 6 were below limit of detection (Figure 15). Figures B and C, however, show good results considering difficulties in ^{93}Mo detection, efficiency calibration, and low activity. For example, ^{93}Mo standard solution availability is rare and consequently efficiency calibrations are challenging. However, the data entries in figure C were produced using three different techniques i.e., Sample 2 using mass spectrometry, Sample 7 with LEGe, and Sample 9 with average of LSC and mass spectrometric results, which were consistent with each other. The variety of techniques and relatively consistent results give validity to the methodologies. An attempt to mitigate the issue was carried out via submission of measurement spectra, which are shown in Figure 16. Spectrum A was produced using TDCR, but most likely due to the peak in the beginning of the spectra, limit of detection calculation was not possible, as TDCR did not relay reasonable efficiencies. The sample analyser had notified that there have been instances where a peak at luminescence region has been more persistent than previously. The reason for the persistency is unknown but the peak has been suggested to originate from a new cocktail, which performance is under study. Other partners had not noticed a similar phenomenon in their studies. Spectrum B had been produced using ^{55}Fe calibration and the signal does not seem to contain interferences. Spectrum C is an X-ray spectrum and does not seem to contain interferences. A deeper dive should be carried out to find out the origin(s) of the difference between Sample 5 and Samples 2, 7, and 9.

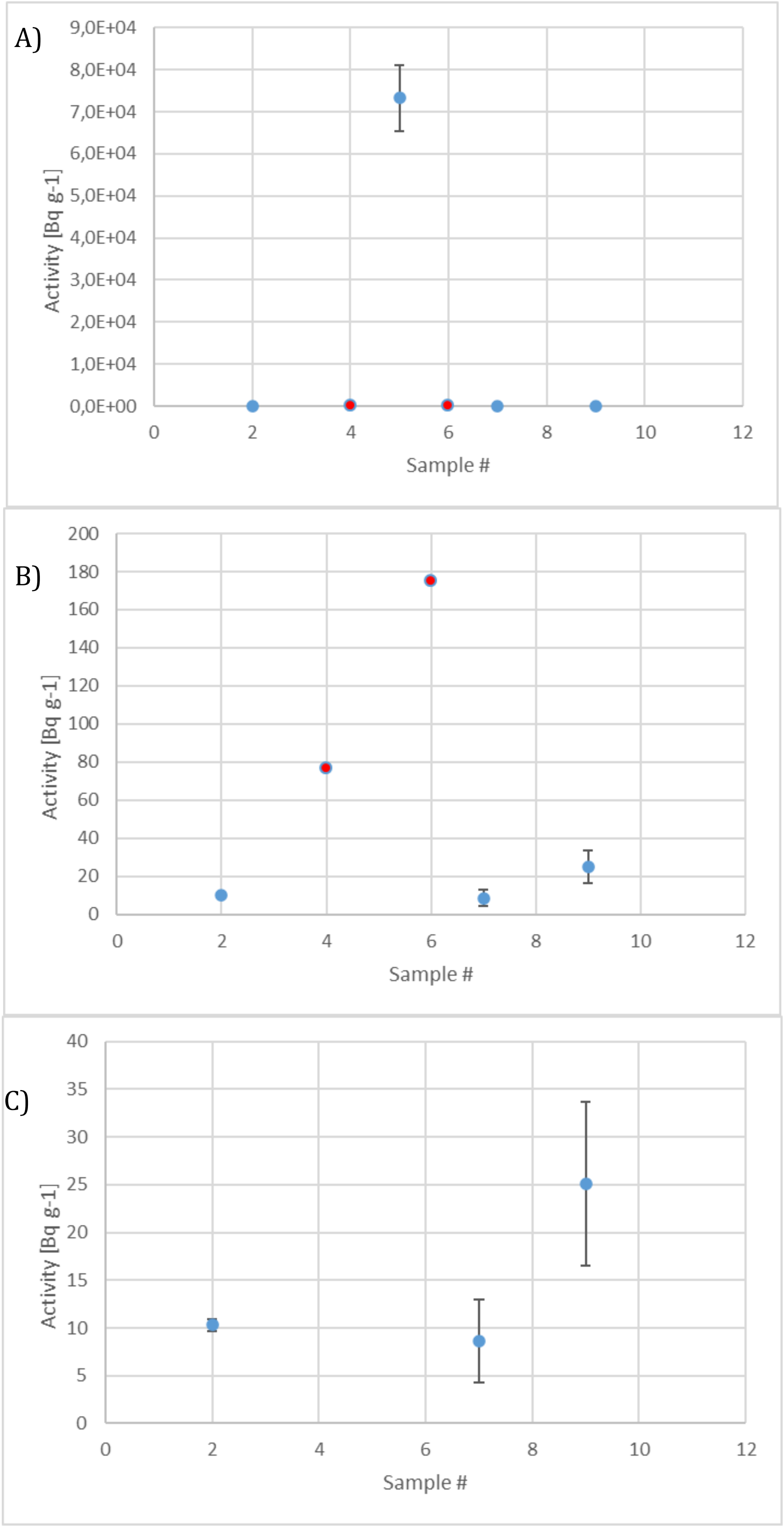

Figure 15. The final ^{93}Mo results in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Red dots indicate results below limit of detection. Figure A shows all results, figure B shows results without Sample 5, and figure C shows Sample 2, 7 and 9 results.

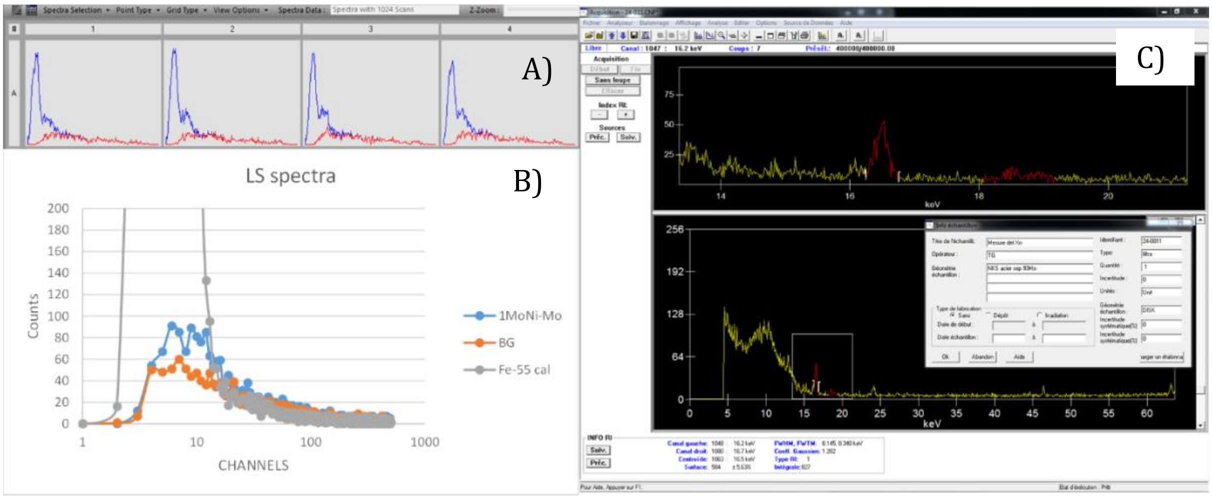

Figure 16. Examples of ^{93}Mo spectra. LSC spectra A show the total counts (blue) and triple counts (red) of a background sample and three aliquots. LSC spectrum B shows the counts for ^{55}Fe calibration standard in grey, background in orange, sample in blue. X-ray spectra C shows the energy discrete counts with region of interest in red.

Statistical analysis of ^{93}Zr results was not carried out due to inconsistent results (Figure 17). Sample 5 and 6 results were well aligned with each other whereas Samples 4 and 9 results were well aligned with each other. Sample 10 result was below limit of detection and little bit above the second set of results. Therefore, the results seem to contain two sets of results with one limit of detection result in between. Samples 5 was measured using mass spectrometry and Sample 6 using LSC. Samples 4 and 10 were measured using LSC. Sample 9 ^{93}Zr results is the average of two ICP-MS and one LSC measurement data and as the three values were in good agreement, the average of them was reported. Therefore, the detection technique cannot be directly attributed to be the reason for the two sets of results. The submitted spectra did not give a reason for the difference (Figure 18). As concluded with ^{93}Mo results, a deeper dive in the ^{93}Zr analyses is needed to find out the origin(s) of the differences.

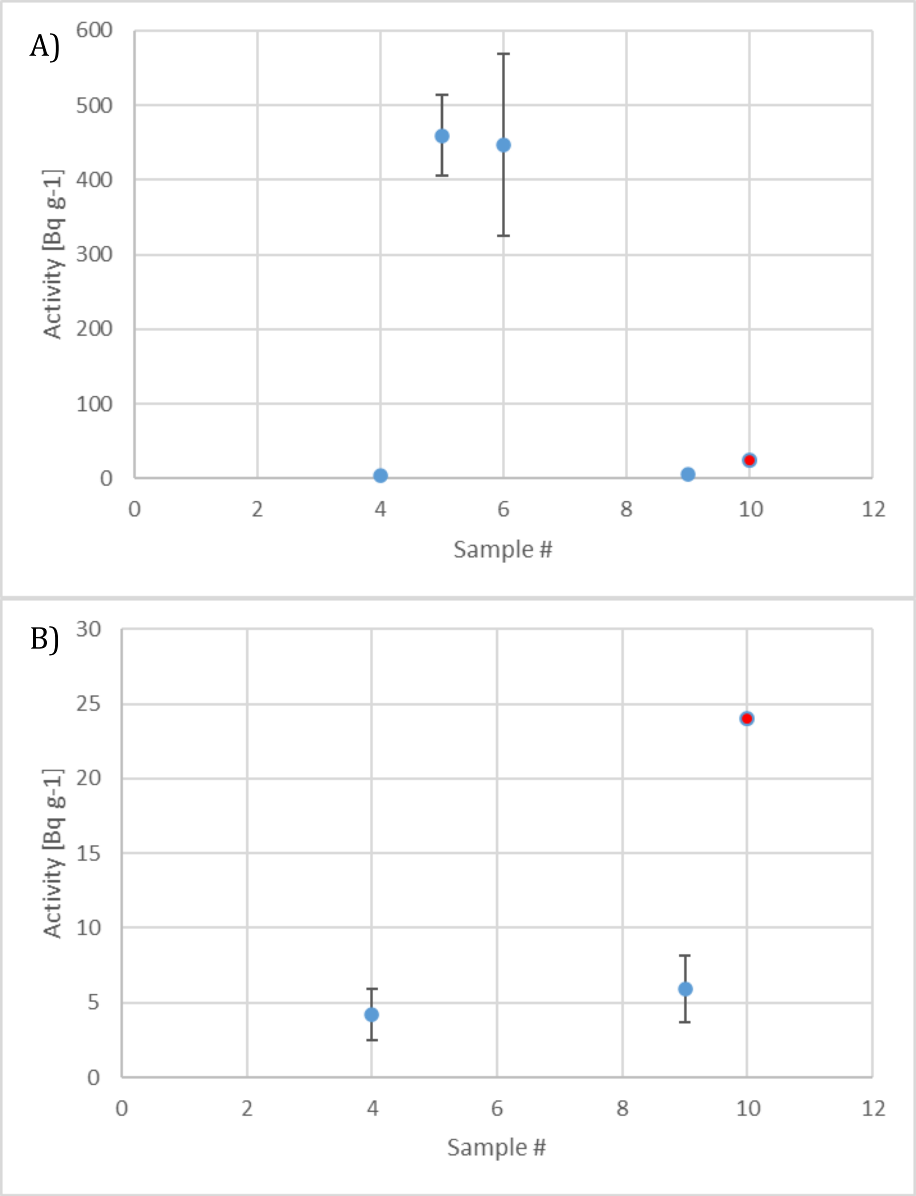

Figure 17. The final ^{93}Zr results in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Red dots indicate results below limit of detection. Figure A shows all results and figure B results of Samples 4, 9, and 10.

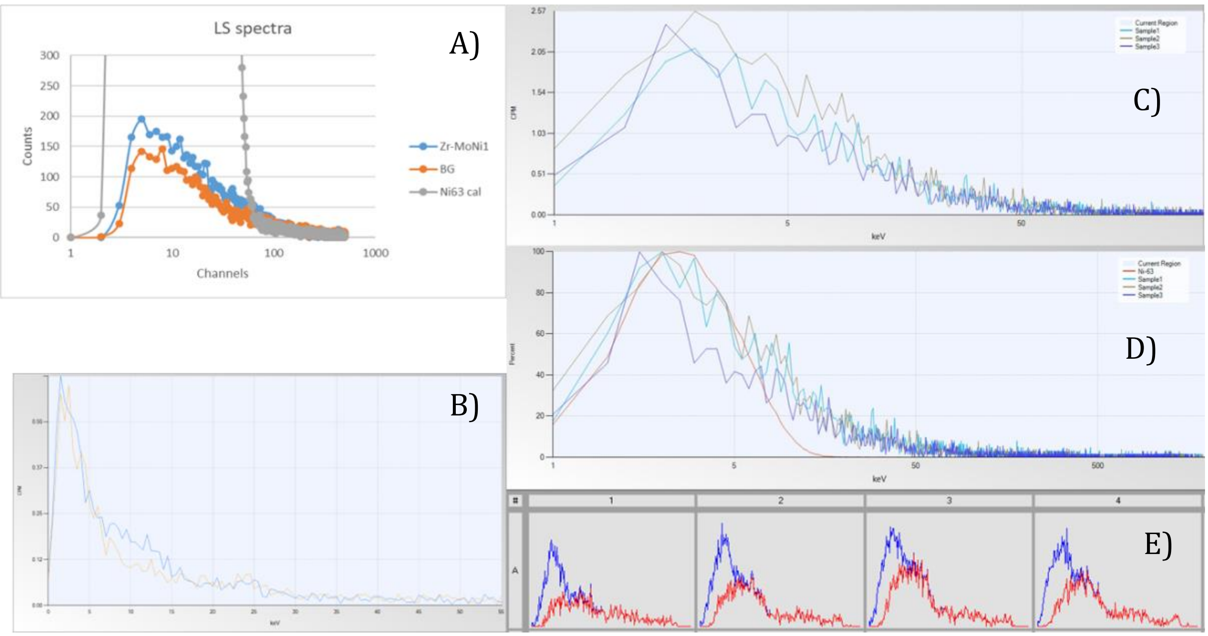

Figure 18. Examples of ^{93}Zr spectra. LSC spectrum A shows ^{63}Ni calibration standard signal in grey, background signal in orange, and sample results in blue. LSC spectrum B shows blank sample signal in orange and sample in blue. LSC spectra C and D show ^{63}Ni standard signal in orange and samples in other color. LSC spectra E show the total counts (blue) and triple counts (red) of a background sample and three aliquots.

Indicative statistical analysis of ^{94}Nb results were carried out with four data entries. The assigned value with the robust standard deviation ($k = 1$) was 117 ± 39 Bq g^{-1}. The data entries with the assigned value and uncertainties ($k = 2$) are shown in Figure 19. The z score was calculated using the standard uncertainty of assigned value (20.9 %) since the robust standard deviation was large (33.4 %). As seen in Table 17, all ^{94}Nb z scores are in acceptable range. However, it must be emphasised that only four data entries were included in the statistical analysis and the robust standard deviation and standard uncertainty of assigned value were large (>20 %).

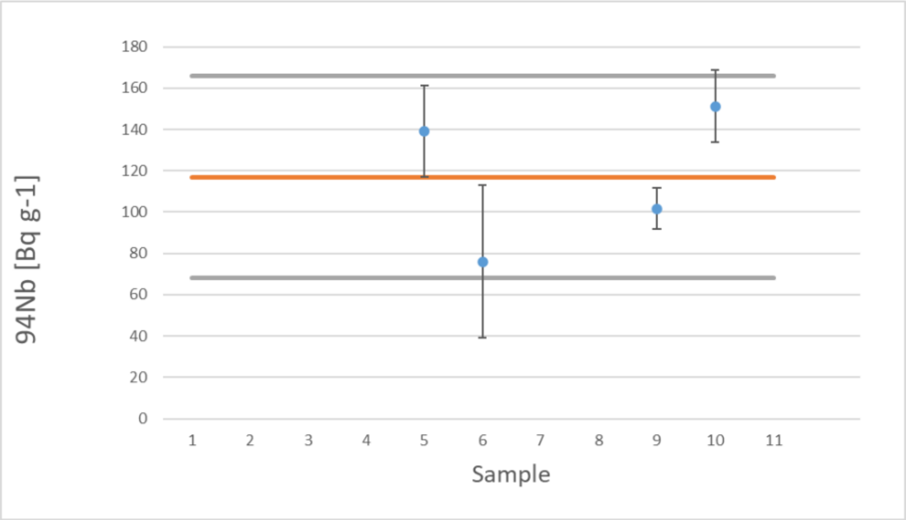

Figure 19. Final ^{94}Nb results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

Statistical analysis of ^{93m}Nb results was not carried out due to low number of data entries (Figure 20). In total, three data entries were submitted. The Sample 9 result is approximately half of the Sample 10 result whereas Sample 6 result is in between them and its uncertainty almost covers the other two results. Samples 6 and 9 were measured in solid form (evaporated on a filter and evaporated as a point source, respectively) using LEGe and Sample 10 in liquid form using LSC (Table 11, Table 14). In all cases the efficiency calibration was carried out using ^{93m}Nb standards. However, more data entries should be included to make conclusions.

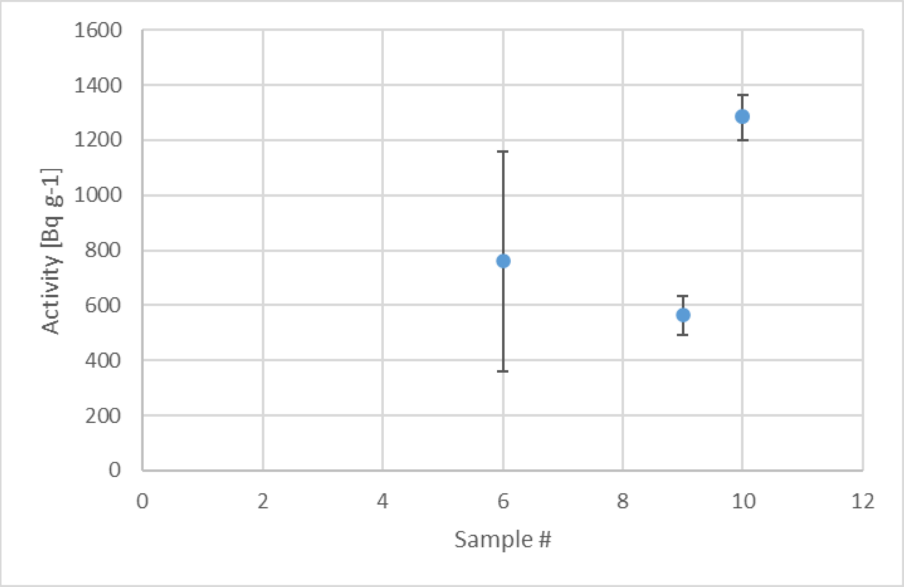

Figure 20. The final ^{93m}Nb results in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

All statistically analysed z scores of ETM and DTM results are summarised in Figure 21. The results show that Sample 1, 4, 7, 8, and 10 are systematically in acceptable range with 1 to 5 data entries. Samples 5, 6, and 9 contain also data entries in warning signal range. Only Sample 2 contain one data entry in unacceptable range.

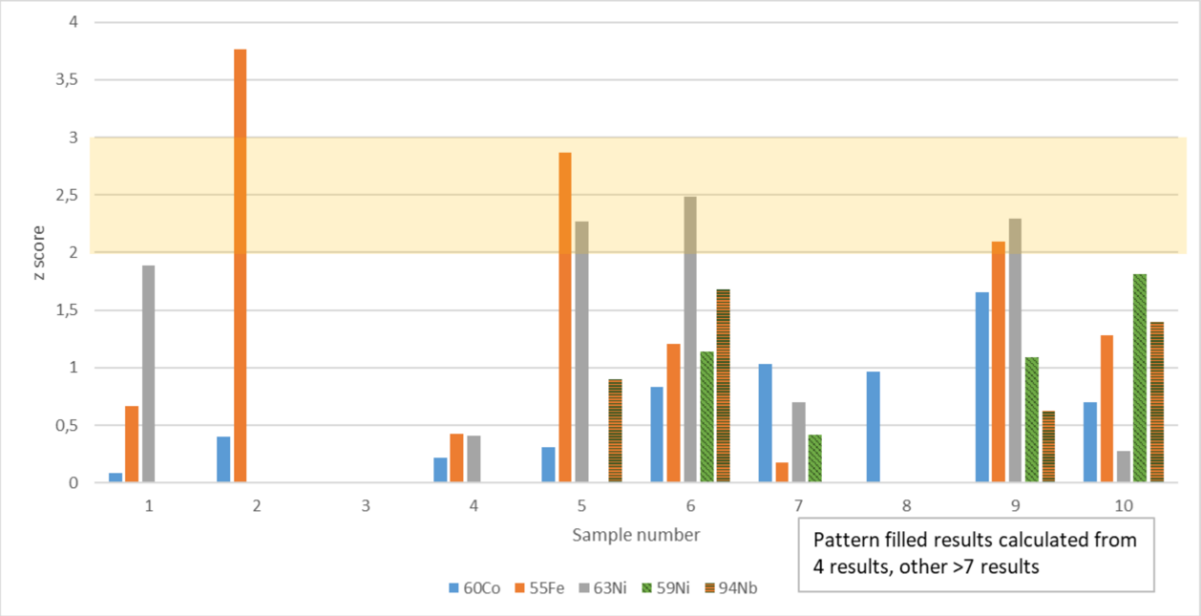

Figure 21. The final ETM and DTM z scores in MoNi intercomparison exercise. Results are acceptable when z≤2.0, warning signal is given when 2.0<z<3.0, and unacceptable when z≥3.0.

7.3 Elemental analysis results

Statistical analysis of Fe results was carried out with six data entries. The assigned value with the robust standard deviation ($k = 1$) was 704000 ± 60000 mg kg^{-1}. The data entries with the assigned value and uncertainties (*k* = *2*) are shown in Figure 22. The z score was calculated using the robust standard deviation (8.4 %). As seen in Table 18, all Fe z scores are in acceptable range.

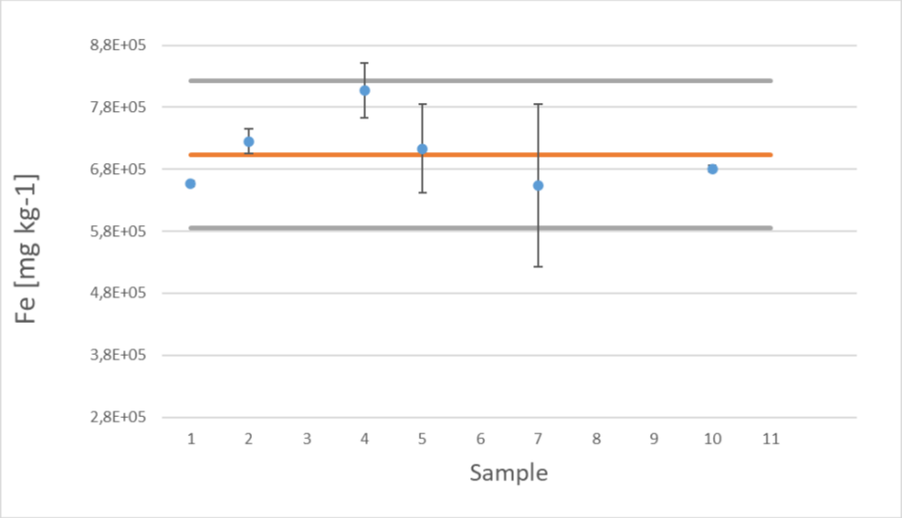

Figure 22. Final Fe results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of *k* = *2*.

Table 18. Elemental z scores (*k* = *2*) in MoNi intercomparison exercise. Results are acceptable when z≤2.0, warning signal is given when 2.0<z<3.0, and unacceptable when z≥3.0.

Sample	z score			
	Fe	Ni	Mo	Co
1	0.8	0.4	0.4	0.4
2	0.4	0.1	0.6	0.3
3	-	-	-	
4	1.7	1.2	1.5	1.3
5	0.2	0.2	-	-
6	-	-	-	-
7	0.8	0.4	0.6	-
8	-	3.1	-	-
9	-	-	-	-
10	0.4	0.8	0.9	0.6

Statistical analysis of Ni results was carried out with seven data entries. The assigned value with the robust standard deviation (*k* = *1*) was 98000 ± 11000 mg kg^{-1}. The data entries with the assigned value and uncertainties (*k* = *2*) are shown in Figure 23. The z score was calculated using the robust standard deviation (10.4 %). As seen in Table 18, one Ni z score is in unacceptable rage (Sample 8) whereas all the others are in acceptable range.

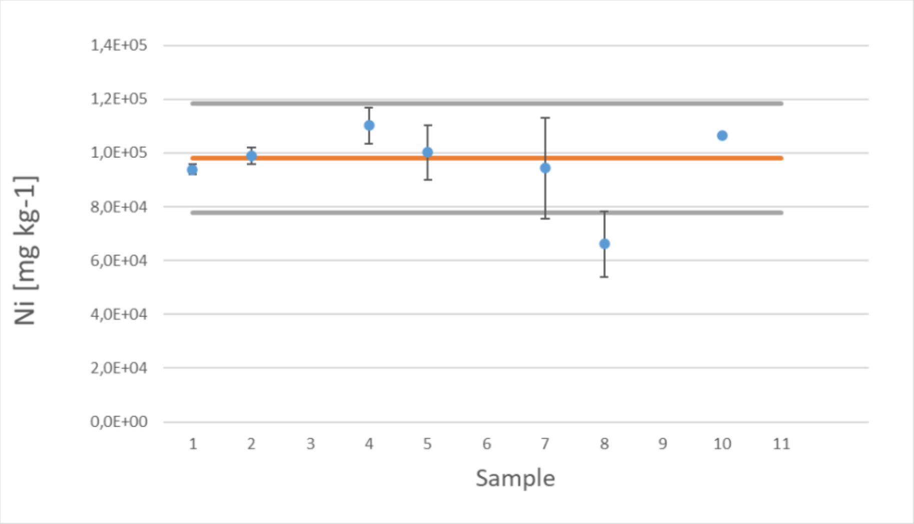

Figure 23. Final Ni results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

Statistical analysis of Nb results was not carried out due to inconsistent results (Figure 24). Sample 1 result was submitted as indicative. Sample 2 (maximum) was approximately 4 times higher than Sample 4 (minimum). Even though only four data entries were submitted, it can be concluded that difficulties in Nb analysis originates most likely to its instability in solution. HF had been added in the acid mixture to aid Nb to remain in the solution. The amount of HF was decided by partners but the concentration in the solution may have been too low due to high amount of Fe, which also consumes HF. Additionally, one partner reported presence of small amounts of solids, which was attributed to Nb. As the stability of Nb was concluded to be problematic, this concept will be further studied in the proposed subsequent project MoNi II.

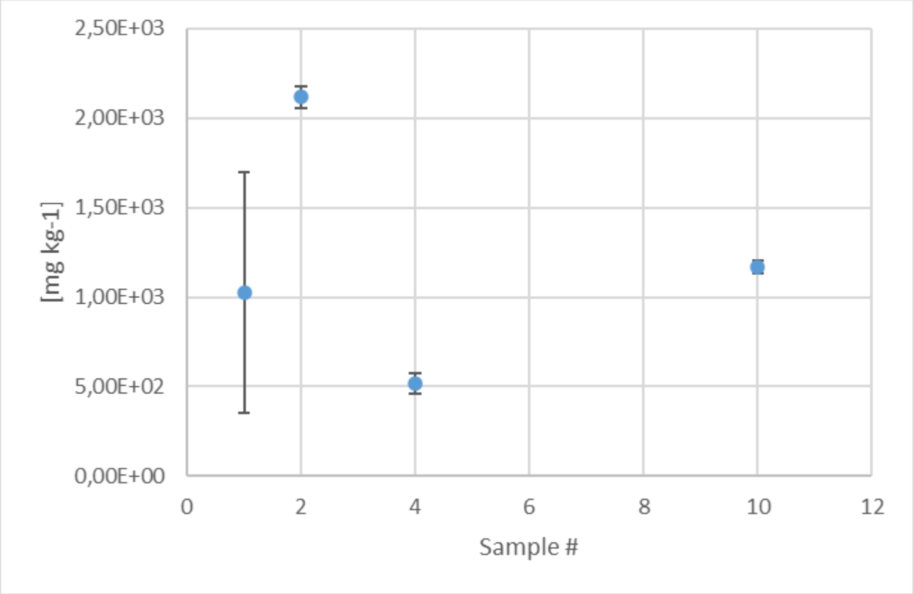

Figure 24. Final Nb results in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

Statistical analysis of Mo results was carried out with five data entries. The assigned value with the robust standard deviation ($k = 1$) was 186 ± 180 mg kg^{-1}. The data entries with the assigned value and uncertainties ($k = 2$) are shown in Figure 25. Sample 8 uncertainty was very high (over two orders of magnitude higher than the analysis result) due to the large uncertainty in ICP-MS measurement when using masses 92, 94, and 95. For clarity, the Sample 8 uncertainty is not included in the result figure. Additionally, Sample 7 uncertainty was also high (60%), because the concentration was close to the quantification limit due to a high dilution factor, but it was included in the result figure. The z score was calculated using the robust standard deviation (9.3 %). As seen in Table 18, all Mo z scores are in acceptable range.

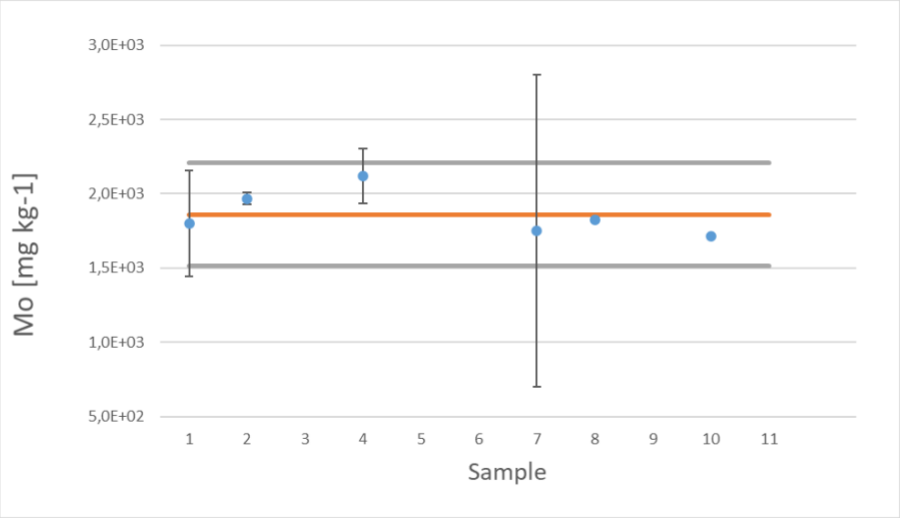

Figure 25. Final Mo results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of *k* = *2*.

Statistical analysis of Co results was carried out with four data entries and consequently the analysis is indicative. Additionally, Sample 1 result was submitted as indicative. The assigned value with the robust standard deviation (*k* = *1*) was 812 ± 99 mg kg^{-1}. The data entries with the assigned value and uncertainties (*k* = *2*) are shown in Figure 26. The z score was calculated using the robust standard deviation (12.1 %). As seen in Table 18, all Co z scores are in acceptable range. Sample 4 uncertainty was attributed to high dilution factor, which was needed due to a restriction that the sample activity introduced into the measurement equipment needed to be below 1 Bq per 10 ml solution.

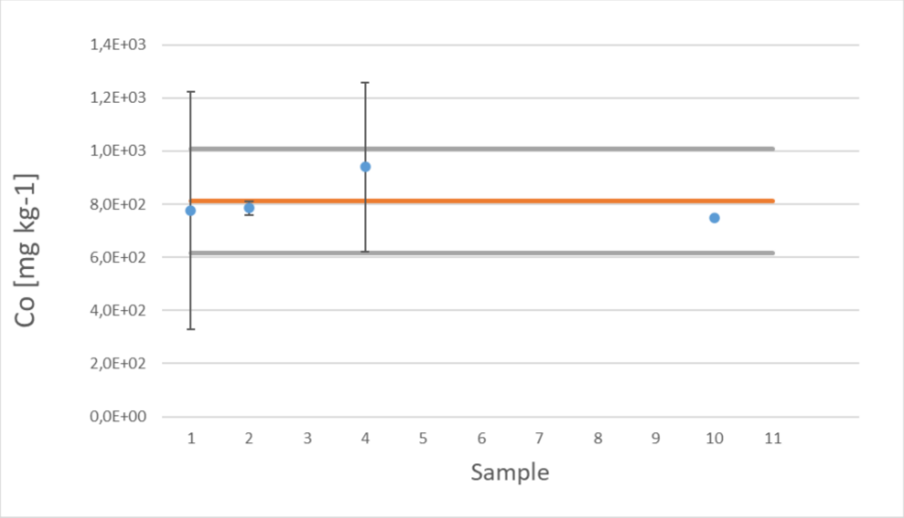

Figure 26. Final Co results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of *k* = *2*.

Statistical analysis of Mn and Cr results was not carried out due to only having two data entries. These results are shown in Figure 27 whereas results with only one data entry are shown in

Table 19. Sample 10 result for Mn is approximately 95 % of Sample 2 result whereas are the difference is a little higher for Cr (i.e, 85 %).

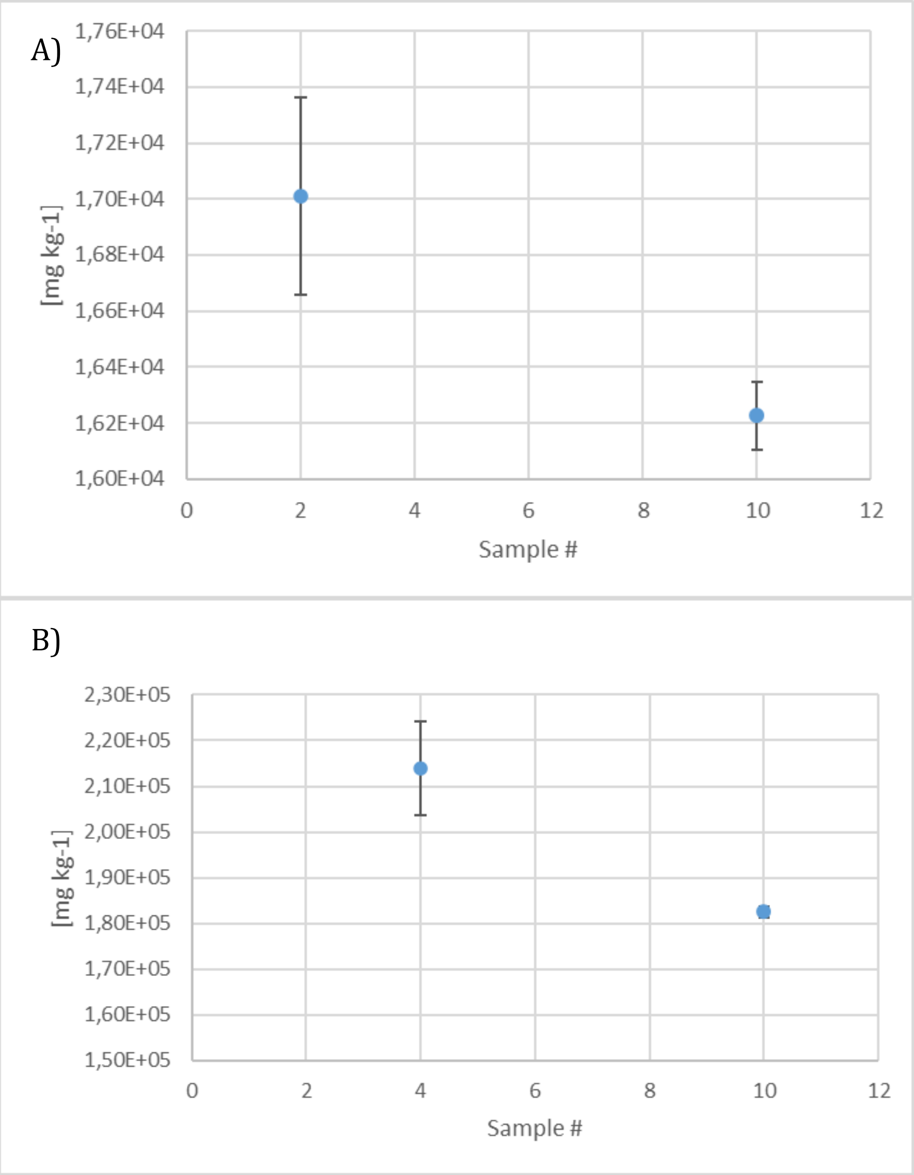

Figure 27. Final Mn (A) and Cr (B) results in high activity steel with uncertainties stated with a coverage factor of *k = 2*.

Table 19. Sample 2 analysed Cu, Zr and Sn results in high activity steel with a coverage factor of *k = 2*.

Sample	Cu [mg kg^{-1}]	Zr [mg kg^{-1}]	Sn [mg kg^{-1}]
2	0.990 ± 0.023	0.013 ± 0.001	0.094 ± 0.003

All statistically analysed z scores of elemental analysis results are summarised in Figure 28. The result show that all data entries for Samples 1, 2, 4, 7, and 10 are in acceptable range. Sample 8 Ni result was in unacceptable range.

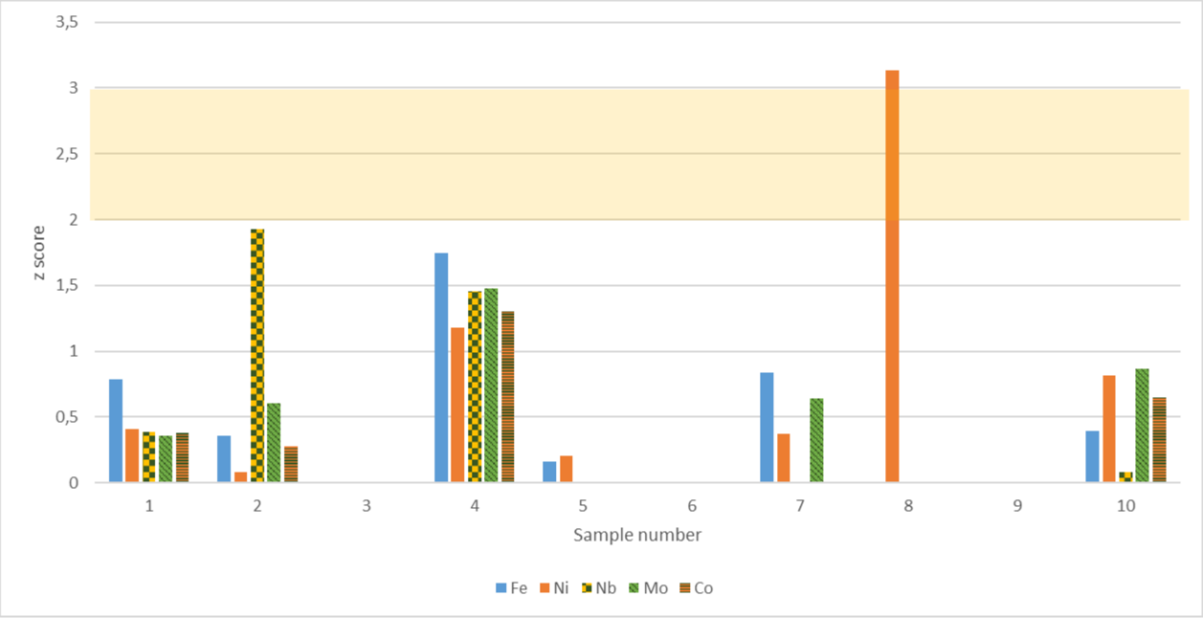

Figure 28. The elemental analysis z scores in MoNi intercomparison exercise. Results are acceptable when z≤2.0, warning signal is given when 2.0<z<3.0, and unacceptable when z≥3.0.

8 Conclusions

ETM and DTM radionuclides were determined in the high activity steel sample, which was homogenised via acid digestion prior to distribution to the partners. The main radionuclides of interest were ETMs (such as ^{60}Co), ^{59}Ni and ^{93}Mo. Optional radionuclides to analyse were ^{55}Fe, ^{63}Ni, ^{93}Zr, ^{94}Nb and ^{93m}Nb. An intercomparison exercise for volatile DTM (i.e., ^{14}C, ^{36}Cl and ^{79}Se) analysis has been suggested for 2025.

The ETM analyses were carried out mainly for aliquots with or without dilutions. Radionuclides were detected using HPGe detectors with different efficiency calibrations (i.e., ^{60}Co, ^{152}Eu or mixed sources and ISOCS/Labsocs), measurement times (from 15 min to 22 h), and measurement distances (from 16 mm to 10 cm). The preliminary meeting discussions concluded that even if the results were well aligned, the expectation was that homogenisation of the sample should have resulted even better trends. However, the statistical analysis of the results according to ISO 13528 standard showed good alignment (10.7 % robust standard deviation) and all z scores were in acceptable range.

The radiochemical separations of DTM radionuclides included a variety of methodologies, mainly using ion exchange and extraction chromatography with different resins combinations, evaporations, dilutions, and liquid-liquid extractions. The DTM detection included a variety of detection techniques, namely LSC, mass spectrometry, gamma spectrometry, and X-ray spectrometry. The preliminary ^{55}Fe results seemed somewhat scattered, which was also seen in final results (29.8 % robust standard deviation). The statistical analysis of final results showed that one ^{55}Fe z score was in unacceptable range, two results in warning signal range and the remaining results in acceptable range. The preliminary ^{63}Ni results were well aligned, but the statistical analysis of final results showed that three ^{63}Ni z scores were in warning signal range and the remaining results in acceptable range. The preliminary ^{59}Ni results did not show a clear trend and indicative (only four data entries) statistical analysis of final results showed that the results were somewhat scattered (27.1 % robust standard deviation), even though all the data entries above limit of detection were in acceptable range. The preliminary ^{93}Mo results showed one overestimated result, two data entries below limit of detection, and three data entries with good alignment. The final ^{93}Mo results were not statistically analysed due to the low number of data entries. However, ^{93}Mo analysis was concluded to be difficult due to low activities and challenging detection techniques. Similar conclusion is appropriate also for ^{93}Zr, which preliminary and final results formed two sets of results. The preliminary ^{94}Nb results showed that direct analysis in original solution using gamma spectrometry resulted in a high limit of detection and quantitative results were possible only after radiochemical purifications and the trend of results was relatively good. Indicative statistical analysis of final ^{94}Nb results showed that the results were scattered (33.4 % robust standard deviation), but all were in acceptable range. Only two preliminary data entries were submitted for ^{93m}Nb and even though three final data entries were available, more data entries should be available to make conclusions.

Elemental analyses were carried out for the sample aliquots using ICP-OES and ICP-MS techniques. The statistical analysis of the submitted results showed that all Fe, Mo, and Co results were in acceptable range. One Ni result was in unacceptable range.

As a conclusion, the MoNi project studied a wide range of radionuclide analyses and additionally elemental analyses resulting in strengthening of analysis capabilities. The results will be further assessed in a peer reviewed article, in which also activation calculation results will be compared with the radionuclide analysis results. The elemental analysis results of MoNi will be utilised as an input in the activation calculations. The project is proposed to be continued in MoNi II, in which volatile radionuclides will be studied in addition to homogeneity of the material and at least elemental analysis of Nb.

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Disclaimer

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Abstract max. 2000 characters	An intercomparison exercise was carried out for Difficult-To-Measure (DTM) radionuclides in high activity steel samples. The steel sample was homogenized via acid digestion prior to distribution

to the partners. Results were collected from radioactive and elemental analyses. The results were analysed according to the ISO 13528 standard. The performance assessment was carried out using z score. The report includes an overview of the radioanalytical procedures, preliminary and final results, and performance assessments.

Key words Decommissioning, Difficult-to-measure radionuclides, intercomparison exercise, high activity steel, elemental analysis