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Abstract

An intercomparison exercise was carried out for volatile Difficult-To-Measure (DTM) radionuclides in high activity steel samples. The exercise was a follow up project for MoNi intercomparison exercise. 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. Additionally, comparison with MoNi project results are shown.

Key words

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

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Final Report from the NKS-B MoNi II activity (Contract: AFT/B(25)4)

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1. Introduction

Intercomparison exercises (IE) on radiochemical analysis of Difficult-To-Measure (DTM) radionuclides in real radioactive waste has been organised within the NKS community for several years i.e., DTM Decom I-III, RESINA, and MoNi projects (Leskinen et al., 2020b, 2021b, 2022, 2025). Additionally, each year, Easy-To-Measure (ETM) radionuclides have been studied to some extent. Both Nordic and non-Nordic partners have been involved. In addition to the NKS-reports, all the IE results have been published in peer reviewed articles (Leskinen et al., 2020a, 2021a, 2023a). The focus in the DTM Decom I-III projects was on beta emitters whereas RESINA project focused on alpha emitters. MoNi project in 2024 focused on ^{59}Ni and ^{93}Mo analyses, which had not been given attention in previous exercises. In MoNi, a high activity steel sample was first homogenised by the organiser and each partner received an aliquot of the acid digested sample. With this way the possible heterogeneities were counteracted, but analysis of volatile DTMs (e.g., ^{14}C) were hindered. Therefore, a decision was made to focus on non-volatile DTMs in MoNi project whereas volatile DTMs were in focus in MoNi II project. Additionally, MoNi II project studied the effects of possible heterogeneity in the ETM and DTM analyses. Similarly to the previous years, the IE was carried out and statistically analysed according to ISO 13528 standard (International Standard, 2022).

The main volatile DTMs of interest in MoNi II were originally:

- ^{14}C – Analysis generally requires volatilisation and trapping of the radionuclide in a trapping solution. ^{14}C has been studied in some of the previous years, but more emphasis was needed for its analysis as it is a long-lived radionuclide with significance in final disposal conditions. Detection of ^{14}C was foreseen to be carried out with liquid scintillation counting (LSC).
- ^{36}Cl – Analysis requires careful control of chlorine volatility either by hindering or encouragement of volatilisation. This beta emitter was studied in one of the previous exercises (Leskinen et al., 2021a, 2021b). Detection of ^{36}Cl is possible with LSC depending on the activity level. However, mass spectrometric detection may be needed in very low activity samples.
- ^{79}Se – Analysis requires careful control of ^{79}Se volatility. Selenium-79 had not been studied in the previous IEs. It is an important long-lived radionuclide in the final disposal conditions.

The concept of small-scale heterogeneity was studied in MoNi II due to the fact that:

- Each partner was given a number of sample pieces.
- The samples were studied for ETMs in the original sample vial and small aliquot pieces and the results were compared with the MoNi results.
- Some DTMs analysed in the MoNi project were re-analysed and the results were compared.

The MoNi II project content followed the same methodology as in the previous IEs:

- Kick-off meeting was organised to discuss the analysis capabilities, to select the radionuclides of interest, and discuss the material to be studied in March.
- Sending of the material to partners by May.
- Analysis time until September.
- Discussion of preliminary results in a preliminary meeting in October.
- Revision of results until final meeting.
- Final meeting to discuss statistically analysed results in November.

- Reporting of results in a NKS report by the end of the year.

Several young scientists were involved in the MoNi II project. At VTT, a young researcher was responsible for non-volatile DTM radiochemistry and elemental analyses. The research team at DTU also included young researchers; a Postdoctoral Fellow, who was actively responsible for the Cl-36 analysis; a PhD student, who supported with the LSC measurement, and an academic technical staff chemist, who helped with sample pre-treatment.

2. Survey of capabilities in the beginning of the MoNi II project

A partner survey of the DTM analysis capabilities was carried out in the beginning of the project. The results are shown in Table 1. The affiliation codes (A to H) and the sample numbers (1-8) in the following sections do not correlate. 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 MoNi II project.

Affiliation code	Readiness	Overview of planned methods	Equipment
A	Method ready for ^{14}C analysis in different matrices (e.g. steel). ^{36}Cl method ready only in graphite, but can be modified for steel. ^{79}Se method development needed. QQQ-ICP-MS detection in development for ^{93}Mo. ^{93}Mo and ^{93}Zr would be interesting to be studied again.	Oxidative acid digestion, ^{14}C oxidised and trapped in NaOH solution. Yield determination with ^{14}C standard. Detection of ^{14}C using LSC. Oxidative acid digestion, ^{36}Cl volatilised and trapped in H_2SO_4 solution. Purification of ^{36}Cl using Cl-resin. Yield determination with stable Cl analysis (MS) and detection using LSC. ^{79}Se radiochemistry unclear. Detection using LSC or QQQ-ICP-MS.	QQQ-ICP-MS (Agilent), LSC (Hidex).
B	Routine analysis for ^{3}H, ^{14}C, ^{55}Fe, ^{63}Ni, ^{59}Ni, ^{93}Mo, ^{93}Zr, ^{94}Nb, ^{93m}Nb, ^{36}Cl and ^{41}Ca in radioactive samples at low and intermediate level activity (liquids, effluents, steels, concretes and resins...). Method available for ^{79}Se but not used for more than 10 years. We did not have time to measure ^{93}Zr, ^{93m}Nb and ^{94}Nb last year so it would be interesting to study them again.	For ^{14}C, a pyrolyser decomposition is used. For ^{55}Fe, ^{63}Ni, ^{59}Ni, ^{93}Mo, ^{93}Zr, ^{94}Nb, ^{41}Ca, acid digestion or alkaline fusion is used for destruction of matrix. For ^{36}Cl in steel, oxidative acid digestion, ^{36}Cl oxidised and trapped. 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) or 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 ^{93m}Nb, gamma spectrometry for ^{94}Nb. ICP-MS Q for ^{93}Zr. We can use AMS to measure ^{36}Cl and ^{41}Ca at low level if needed.
C	Method available for ^{14}C and ^{36}Cl but not tried on steel samples. Method for ^{79}Se under development. Other methods same as MoNi. ^{63}Ni and ^{93m}Nb to be optimized.		LSC, ICP-MS
D	Methods ready for ^{14}C, ^{55}Fe, ^{63}Ni, ^{59}Ni, ^{93}Mo, ^{93}Zr, ^{93m}Nb, ^{94}Nb, and ^{36}Cl. No method developed for ^{79}Se yet.	^{14}C separated by wet oxidation-acid stripping. Use of Dowex1x8 resin and DMG in ^{55}Fe and $^{59,63}Ni$ 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 precipatation) detection. ICP-OES for Fe and Ni yield determinations. ^{93}Mo, ^{93}Zr, and ^{94}Nb	ICP-MS (Agilent 8900), ICP-OES (Varian 710-ES), LSC (PerkinElmer 4900), Mirion HPGe/LEGe(Low Energy Germanium detector)

		purification tested using TEVA column. Detection of ^{94}Nb using HPGe, Mo-93 using LEGe and ^{93}Zr using LSC.	
E	Method ready for ^{14}C, ^{36}Cl and ^{79}Se will require development. LS determination of ^{55}Fe and ^{63}Ni achieved. Development needed for ^{59}Ni by LS or ICPMS. ICPMS determination of ^{93}Mo has been achieved. More study needed for ^{93}Zr, ^{93m}Nb and ^{94}Nb.	Oxidative digestion of steel in acid with capture of off-gas in alkali - ^{14}C recovered and determined by LSC. Both ^{36}Cl and ^{79}Se required further development	QQQ-ICP-MS (Agilent), LSC (Hidex).
F	Methods available for gamma spectrometry, ^{14}C, ^{36}Cl, ^{55}Fe, ^{63}Ni and chemical impurities. Previous experience with ^{94}Nb and ^{59}Ni	^{14}C: sample is combusted in a two stage catalytic pyrolyser at 900°C under a steady flow of carrier gas (air) and oxygen until the release of carbon-14 is complete. The catalyst is intended to oxidise carbon-14 in the vapour and the $^{14}CO_2$ is trapped in a bubbler of carbon dioxide absorber (Carbosorb). Determination by LSC with sample recovery assessed via a separate standard run(per tube). ^{36}Cl: sample is leached in dilute nitric acid then chlorine separated by cation exchange with precipitation as silver chloride and measurement via LSC. Counting efficiency is determined from counting a ^{36}Cl standard source and the overall method yield is determined from a combination of ^{36}Cl dosed runs and chemical recovery based on calibrated chloride carrier addition. ^{55}Fe and ^{63}Ni determined by chemical separation techniques and LSC finish. An initial separation is carried out using solvent extraction, then the ^{63}Ni fraction is purified further using extraction chromatography and the activity determined by liquid scintillation counting. The ^{55}Fe activity is determined using Liquid Scintillation Counting of sources prepared from the precipitated iron hydroxide. Method yields are obtained from chemical recovery	Raddec pyrolyser, LSC (Hidex, Perkin Elmer), ICP-OES (Agilent)

		of added iron and added nickel as determined by Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES). ^{94}Nb by gamma-spectrometry of dissolution solution. Elemental analysis by ICP-OES/MS of a diluted portion of dissolution solution.	
G	Method valid for ^{14}C, ^{36}Cl, ^{93}Mo. Method development in process for ^{79}Se and ^{93}Zr	Acid digestion in closed system, where ^{14}C is trapped in Carbosorb solution and measured by LSC. ^{36}Cl is trapped in HNO3 and purified by AgCl precipitation and anion exchange resin. Yield determination with stable Cl analysis (ICP-MS) and detection using LSC. ^{93}Mo is purified with anion exchange resin and measured by LSC	ICP-MS (Agilent 8800), ICP-OES (Aglient 5800), LSC (Quantulus 1220), Mirion HPGe
H	Read only methods for ^{14}C and ^{36}Cl. Further method development needed for ^{93}Mo. Method development needed for ICP-MS/MS (new instrument).	Oxidative acid digestion of the steel sample. ^{79}Se and ^{14}C methods unclear. ^{36}Cl method: digestion combined bubbling with nitrogen gas and released gases trapped in NaOH, followed by AgCl precipitation, and Cl removed by anion exchange. Measurement by LSC.	ICP-MS/MS (Thermo Fisher), LSC (Hidex)

Table 2 Abbreviations in MoNi II report.

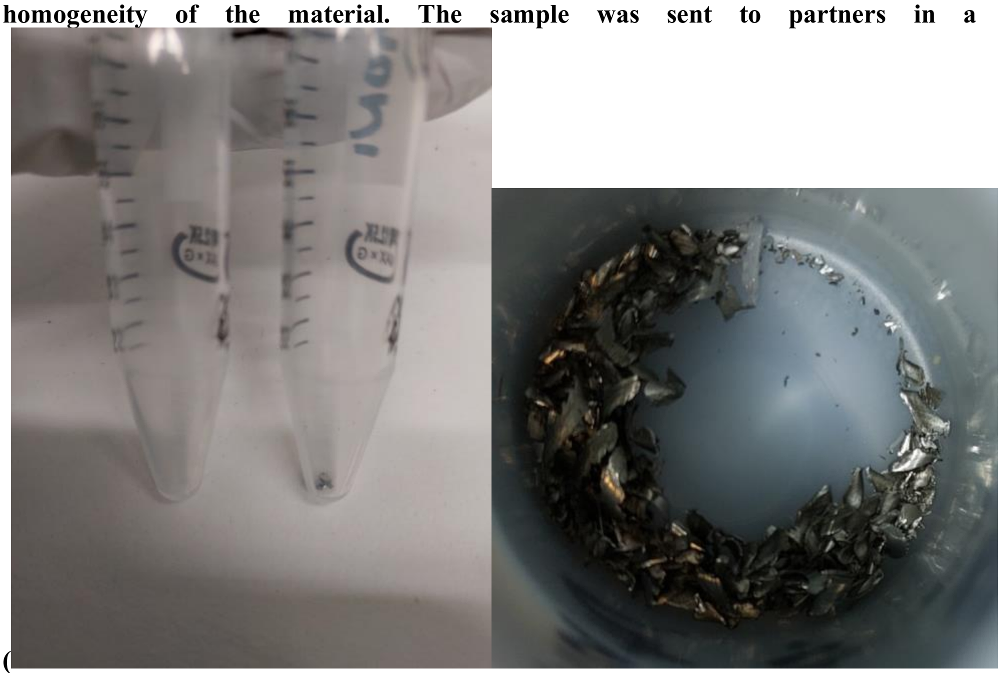
Abbreviation	Abbreviation written open
AMS	Accelerator Mass Spectrometry
BEGe	Broad Energy Germanium
DMG	Dimethylglyoxime
DTM	Difficult-To-Measure
ETM	Easy-To-Measure
HPGe	High Purity Germanium
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
IE	Intercomparison Exercise
ISOCS	In-situ Object Counting System
LEGe	Low Energy Germanium
LOQ, LOD	Limit Of Quantification, Limit Of Detection. These may be used synonymously.
LSC	Liquid Scintillation Counting
RSD	Relative Standard Deviation
SEGe	Standard Electrode Germanium
TDCR	Triple-to-Double Coincidence Ratio
QQQ-ICP-MS	Triple Quadrupole Inductively Coupled Plasma Mass Spectrometry

3. Sample and analysis selection

The studied material was activated steel, which was studied also in MoNi IE in 2024 (Leskinen et al., 2025) and in a trilateral intercomparison exercise in 2023 (Leskinen et al. 2023b). As the heterogeneity studies in the trilateral IE showed an RSD of ~10 %, homogenisation of the material via acid digestion was carried out in MoNi IE prior to distribution to the partners. The homogenisation hindered analysis of volatile DTMs. In MoNi II IE, the sample was distributed in solid form enabling volatile DTM analysis and heterogeneity studies via gamma spectrometric analyses.

The selected analyses were:

Cobalt-60 was selected as a main radionuclide of interest in order to study homogeneity of the material. The sample was sent to partners in a conical tip vial



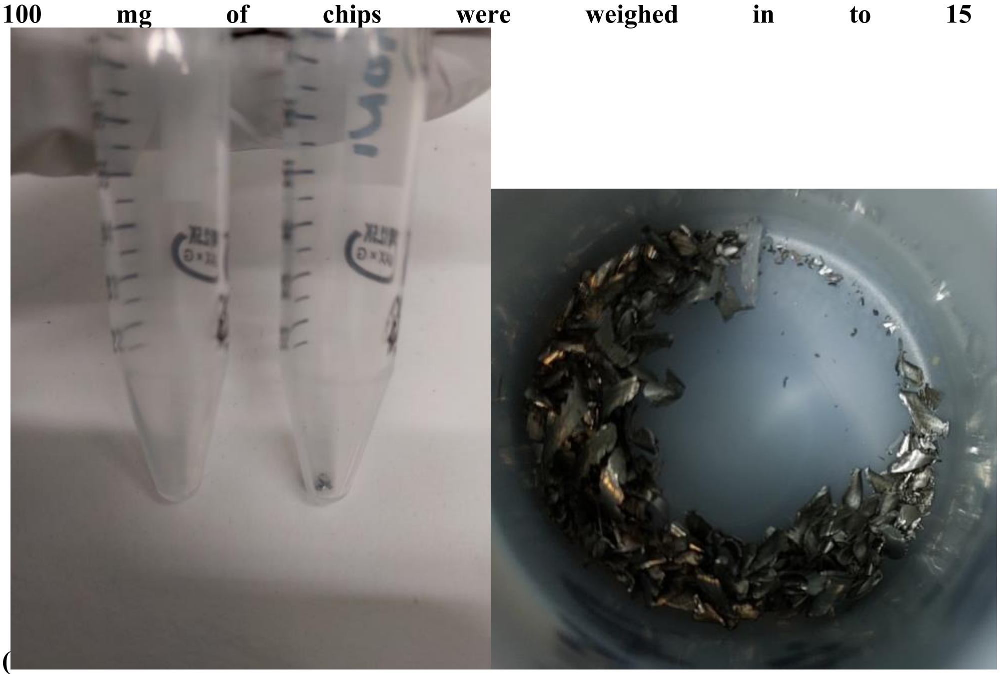
(

- Figure 1) with an empty vial, which could be used for geometry calibrations. The plan was to analyse the sample as it arrived and aliquots in solid and acid digested form.
- Volatile DTM ^{14}C was selected as a main radionuclide of interest as there were enough interest and expertise to analyse it. Additionally, previous studies had shown that the material contains measurable amounts of ^{14}C.
- Volatile DTM ^{36}Cl was discussed to be an interesting candidate for the IE. However, there was no analytical or modelling information available to confirm that the radiochemical analyses would result in quantitative results. The partner experience suggested that an Accelerator Mass Spectrometry (AMS) may be needed in detection of very low activities. Therefore, ^{36}Cl was selected as optional.
- Volatile DTM ^{79}Se analysis was discussed to be interesting but method developments were on-going. Additionally, the modelling results had shown that the activity level could be as low as 10^{-4} Bq g^{-1}, which would be very difficult to achieve quantitatively. Therefore, ^{79}Se was selected as optional.
- Non-volatile ^{55}Fe, ^{63}Ni and ^{59}Ni analyses were selected to be optional.
- Non-volatile ^{93}Mo and ^{93}Zr were selected to be of main interest based on the MoNi results.
- Non-volatile ^{94}Nb and ^{93m}Nb analyses were selected to be optional.

- Elemental analysis was prioritised based on partners’ interest. The main elements of interest were Fe, Ni, Nb, Mo, Co, and Zr. Optional elements were Se, Cl, Mn, Ho, Ag, Ce, Sm, Eu, Li, and Nd.

4. Sample preparation

The activated steel chips (Leskinen et al., 2023b, 2025) were decontaminated by emersion in 1 M HNO_3 for 30 minutes, washing with deionised water, and let to air dry. Approximately 100 mg of chips were weighed in to 15 ml tubes



(Figure 1), which were all measured using gamma spectrometry. Homogeneity study of the samples was carried out based on ^{60}Co activity and as the ^{60}Co relative standard deviation (RSD) was 2.7 %, the samples were considered homogenous.

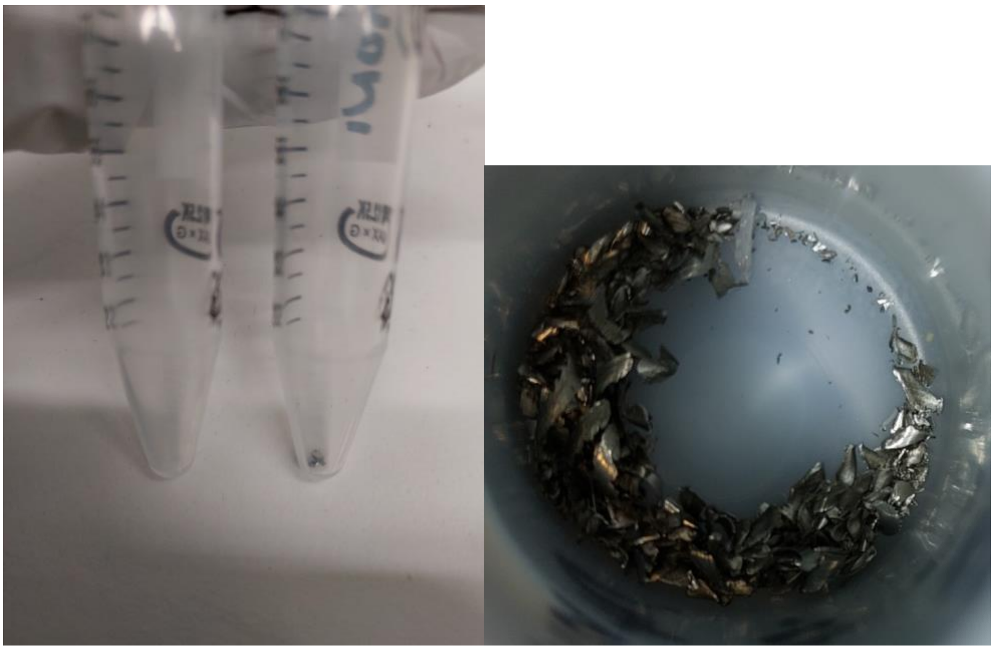


Figure 1. Examples of 15 ml conical tip vials (left). Zoomed image of activated steel chips prepared for MoNi and MoNi II projects (right). An example chip size can be seen in the upper conical tip vial.

5. Preliminary meeting results

The analysis methods, yields, and analysis result trends were discussed in 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. The reported radiochemical analysis yield results prior to the preliminary meeting are shown in Table 3. The ^{14}C yields varied from 49-95 %, ^{59,63}Ni from 36-81 %, ^{36}Cl from 8-80 %, ^{93}Mo from 20-94 %, ^{93}Zr from 57-99 %, ^{94}Nb from 69-100 %, ^{93m}Nb from 69-100 %, and ^{55}Fe 49 %.

ETM, DTM and elemental results are discussed in following sub-sections. Only one set of results were submitted for ^{55}Fe, ^{59}Ni, ^{3}H, Se, Ho, Ce, Sm, Eu, Li, and Nd prior to the preliminary meeting. No results were submitted for ^{79}Se or Cl.

Table 3. Radiochemical yields and uncertainties (*k* = *2*) submitted prior to the preliminary meeting.

Sample number	Yield ± uncertainty [%] (*k* =*2*)							
	^{14}C	^{59,63}Ni	^{36}Cl	^{93}Mo	^{93}Zr	^{94}Nb	^{93m}Nb	^{55}Fe
3	95±21	-	-	-	-	-	-	-
4	49±20	-	-	87±11	98±14	100±18	100±18	-
5	90±10	36±4	8±1	20	57	75±8	-	-
6	93±9	-	56±15	-	-	69±5	69±5	-
7	80±1	81±8	80±1	94±3	99±0	98±0	98±0	49±9

5.1 Preliminary analysis results of ETMs

The sample specific ETM analysis methodologies are reported in Table 4. The samples were measured three ways i.e., in solid form as received, aliquot in solid form, and aliquot in acid digested form. The measurement geometries were in 15 ml, 50 ml or 100 ml vials/bottles. The measurement distance from the sample to the detector were 16 mm, 4 cm or 10 cm. Dead times varied from 0.06-1.4 % and measurement times from 2500 s to 60 hours. All measurements were carried out using Germanium detectors i.e., high purity (HPGe), broad energy (BEGe), or standard electrode (SEGe). Efficiency calibrations were carried out using ISOCS, ^{152}Eu standard source, or mixed/multi standards. The uncertainty calculations were reported to be combined uncertainties. Reported references were either internal instructions, in-house protocols, or NKS reports.

The preliminary ^{60}Co analysis replicate results are shown in Figure 2. Original sample results show that Samples 4 and 6 align well whereas Sample 7 is a little higher than Sample 6. Aliquot solid sample results show that Samples 4 and 6 align well. However, Sample 6 has a significantly higher uncertainty. Aliquot liquid (acid digested solution) results show a good alignment. However, their uncertainties do not cross each other. Additionally, Sample 8 analysis information was not available due to late submission of the results.

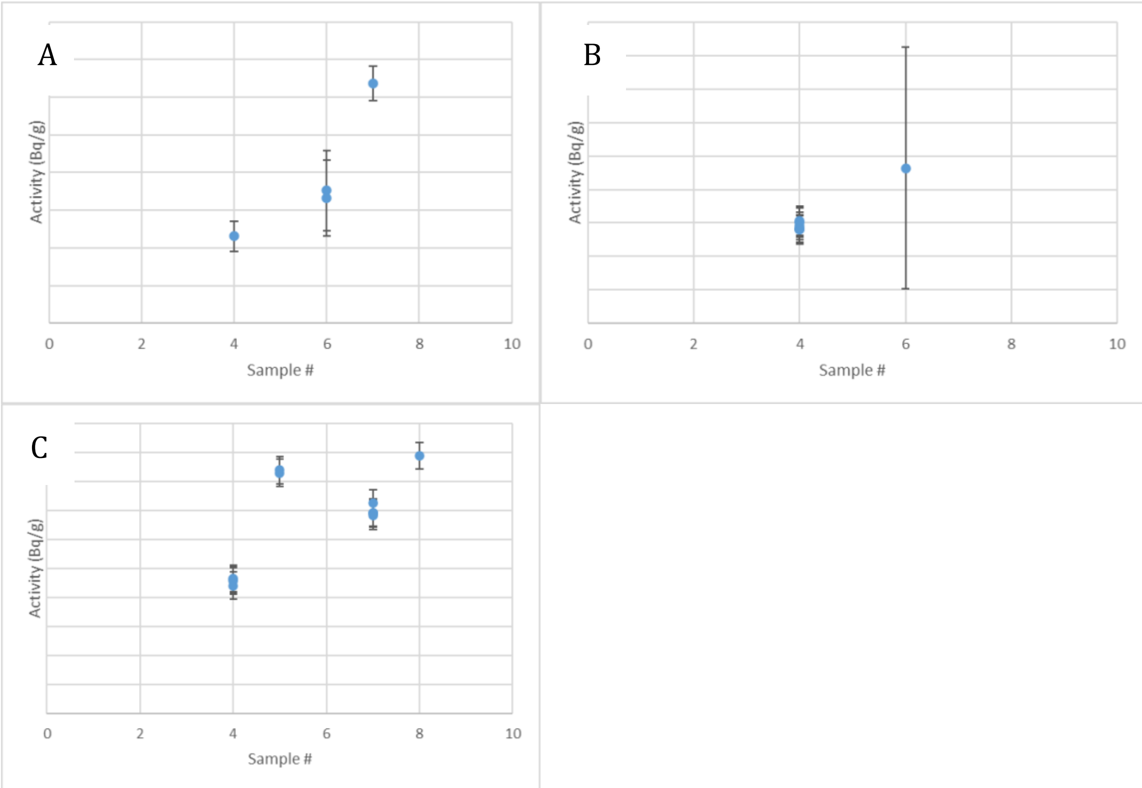


Figure 2. Preliminary ^{60}Co results in original high activity steel sample measured in the original vial (A), solid aliquot (B), and liquid aliquot (C). The uncertainties are stated with a coverage factor of $k = 2$. Analysis methodologies expressed in Table 4.

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

	1	2	3	4	5	6	7	8
Sample prepa-ration	-	-	-	The received sample 4 was directly measured at 10 cm distance for 2500 s. Aliquots A and B were measured in both solid and acid digested forms.	Sample measured after dissolution	20 mg of sample was digested in microwave oven with HNO_3 and HF. A 1 ml aliquot of the digested solution was analyzed	1.The received sample was heated first in a furnace at 500 °C. 2. Acid digestion (sample with HCl + HNO_3 + H_2O_2 +HF on a hot plate) resulting in destruction of the matrix in couple of hours.	-
Measure-ment geometry	-	-	-	Solids were measured in 15 ml plastic vials with conical tips. Acids were measured in 100 ml cylidrical vials. Original sample/aliquot A and B solid/ aliquot B acid 10 cm source to detector distance. Aliquot A acid 4 cm source to detector distance.	Distance to the detector 10 cm in glass vial	Digested solution with a 1 ml aliquot was measured in a 50 mL vial at 16 mm distance on top of the detector.	100 mL plastic bottle / 10 cm distance on top of the detector	-
Dead time	-	-	-	Solid sample orig 1.4 %, aliquots 0.5 %. Aliquot acids 1.2-0.5 %	0.77% and 0.95%	0.06%	1.42%	-
Measure-ment time	-	-	-	Original sample 2500 s; Aliquot A and B in solid form 14400 s; Aliquot A in acid 3600 s; Aliquot B 216000 s.	7200 s	10800 s, one replicate	2 hours, three replicate measurements	-
Detector type and efficiency	-	-	-	Mirion HPGe, 15% S/N-B18067	BeGe detector (Canberra)	Canberra SEGe GC3018 (Al window); 30% relative efficiency	Canberra HPGe 4020	-

Efficiency calibration	-	-	-	ISOCS	Mixed gamma standard	Same geometry efficiency calibration with multi-gamma source	Efficiency calibration using ^{152}Eu standard source	-
Descrip-tion of uncertainty calculations	-	-	-	Sources of uncertainty: masses and spectral.	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.	-

5.2 Preliminary radiochemical ^{14}C results

The sample specific ^{14}C analysis methodologies are reported in Table 5. The overview of the radiochemical methods includes oxidative acid digestion and trapping of volatilised ^{14}C in different trapping solutions (e.g., Carbosorb or NaOH) or pyrolysis followed by a trapping solution (i.e., Carbosorb). The activities were measured using LSC. Stabilisation time for the LSC measurements varied from 15 min to over 12 hours and counting times varied from 600 s to 4 hours. The efficiency calibrations were carried out using triple to double coincidence ratio (TDCR), internal ^{14}C or a quench curve. The uncertainty calculations were reported to be mainly combined uncertainties. The reported references were internal instructions and publications (Brennetot et al., 2017; Hou, 2002; Leskinen et al., 2020c)

The preliminary ^{14}C results in Figure 3 show that two analysis results (Sample 6 and 8) are below limit of quantification (LOQ) and additionally below the four quantitative results. Sample size may have affected the below LOQ results. Those results were, however, produced using a pyrolyser (e.g., raise of temperature in 900 °C and trapping in trapping solution) in which some residue was reported to remain in the sample boat. This may indicate that also some ^{14}C remained in the sample boat. Sample 4 result is significantly higher compared to the other results and it was suggested to suffer from contamination. However, the source of contamination remained unknown as the methodology contained three absorption bottles; first bottle with H_2SO_4, which traps e.g., ^{36}Cl, and second and third bottles with 0.4 M NaOH which trap ^{14}C. Calculations were also re-checked not to include a calculation error.

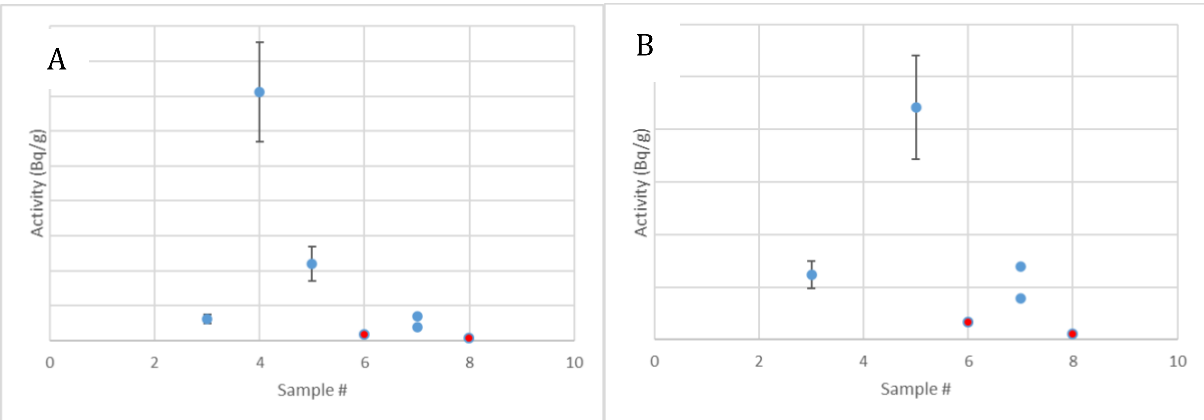


Figure 3. Preliminary ^{14}C results in high activity steel sample. All results (A) and Sample 4 excluded (B). The uncertainties are stated with a coverage factor of $k = 2$. Red dot indicates a result below LOQ. Analysis methodologies expressed in **Table 5.**

Table 5. Carbon-14 analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview of the radiochemical method	-	-	Decomposition of steel by acid digestion to release C, Carbosorb and Perma Flurer to collect the ^{14}C	Oxidative acid digestion and trapment of ^{14}C in 0.4 M NaOH. Measurement of ^{14}C using LSC. Yeald determined with ^{14}C standard solution.	Volatile radionuclides ware absorped during dissolution in washing bottles containg 8 M NaOH	Pyrolysis at 900 °C, recovery in bubbler with Carbosorb (basic medium)	The sample is treated with K2S2O8、4% AgNO3 and 16% H2SO4, temperature was raised to 96 °C, refluxing for 3 hours. CO2 will be absorbed by 10 mL Carbo-sorb E.	-
Measurement information	-	-	Measurement ^{14}C by liquid scintillation counting, the sample and blank were counted for 5 cycles of 60 min each.	Mixture of 5 ml NaOH into 10 ml Hidex Aqua Light+. Stabilisation time > 12 h. Measured 3 times 14400 s and 3600 s. Utilisation of guard.	^{14}C measured with LSC in the 8 M NaOH solution	5 ml of purified ^{14}C fraction was mixed with 10 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 luminescense. Sample was measured once 600 s. Yield was assumed to be 93%, the same as for liquid samples (assumption from NKS DTM 2019).	10 mL Carbo-sorb E and 10 mL Permafluor E(liquid scintillation counting cocktail) in a 20 ml plastic vial. Samples were measured 3 times 1200 s. Yield estimated using standard source.	-
Equipment	-	-	Wallac 1220 Quantulus liquid scintillation counter	Hidex 300 SL	Tri-Carb 3110 TR	Eraly pyrolysis oven, AccuFLEX LSC-8000 Hitachi for LSC.	Perkin Elmer Tri-Carb 4910TR liquid scintillation counting.	-

Efficiency calibration	-	-	~40% derived from the counting result and spiked standard	CoreF corrected TDCR	Internal ^{14}C standard	Quench curve with ^{14}C source	^{14}C efficiency quench curve.	**-**
Description of uncertainty calculations	-	-	Combined uncertainty with the sample preparation and measurement uncertainties	Sources of uncertainty: masses and LSC measurements.	Combined uncertainty	Combined uncertainty with the sample preparation, recovery yield and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties.	-

5.3 Preliminary radiochemical ^{93}Mo results

The sample specific ^{93}Mo analysis methodologies are reported in Table 6. The overview of the radiochemical methods included several methods i.e., resin treatments and evaporations. The sample measurements were carried out in liquid or solid (evaporated on a filter) form. The measurements were carried out using different types of techniques i.e., inductively coupled plasma mass spectrometry (ICP-MS), LSC, and LEGe. The mass spectrometric measurement calibration was carried out using Mo isotopes in natural Mo standard or stable Mo with mass bias interpolation. The LSC efficiency calibrations were carried out using TDCR and LEGe using ^{93m}Nb standard source. 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 & Kameo, 2016).

The preliminary ^{93}Mo analysis replicate results are shown in Figure 4. The results show that three out of four data entries were below LOQ. However, all the results below limit of quantification were correctly above the only quantitative result (Sample 2).

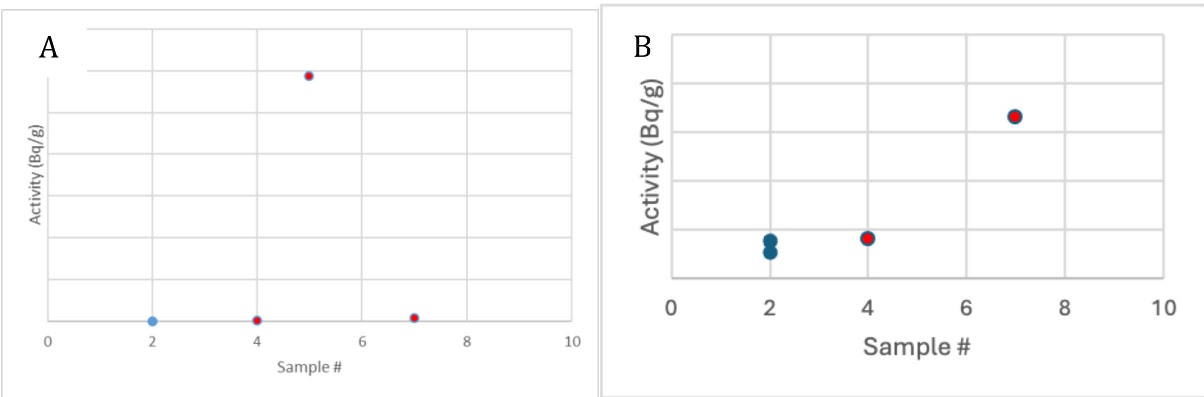


Figure 4. Preliminary ^{93}Mo results in high activity steel sample. All results (A) and Sample 5 excluded (B). The uncertainties are stated with a coverage factor of k = *2*. Red dot denotes a result below LOQ. Analysis methodologies expressed in **Table 6**.

Table 6. Molybdenum-93 analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview of the radiochemical method	-	Iron-55 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 10 M HCl (Co and Ni), 6 M HCl (Zr), 3 M HCl (Mo) and 0.1 M HNO_3 (Fe). Stable iron content measured on a fourth diluted aliquot by ICP-MS.	-	Acid digestion with aqua regia and HF. Stable carrier added for yield determination. Cations removed with cation exchange resin. Mo separated from Zr and Nb using TEVA resin. Final fraction in 3 M HNO3 containing some HF. Yields determined with ICP-OES and activity measurements were performed with LSC.	Mo separation using DOWEX + TEVA resin. Sample was diluted in 2% v/v HNO3 and 0.05% v/v HF and analyzed using TQ-ICP-MS.	-	0.2 mg of Nb, Zr, Mo were added as tracer in each aliquot sample. The aliquot samples were first evaporated to near dryness and dissolved in 0.1 M HF. ^{93m}Nb, ^{94}Nb, ^{93}Zr-93, ^{93}Mo were separated using TEVA resin.	-
Measurement information	-	Purified molybdenum fraction evaporated and dissolved in 5% HNO_3	-	1 ml of sample mixed with 10 ml Hidex Aqua Light+. Stabilisation time 12 h. Measured 3 times 3600 s and 2 times 14400 s using template for H3 with a wider region of interest. Utilisation of guard.	ICP-MS measurement on separated Mo fraction.	-	1 mL of purified Mo fraction were dropped onto 1-inch filter paper and were evaporated to dryness. Samples were measured for 2 hours. Yield was determined by measuring the Nb content with ICP-OES.	-

Equipment	-	Agilent ICP-QQQ 8900	-	Hidex 300 SL	iCAP TQ-ICP-MS (Thermofisher scientific)	-	Canberra LEGe GL0210	-
Efficiency calibration	-	Calibration with stable molybdenum and mass bias interpolated	-	CoreF corrected TDCR	Sensitivity of Mo isotopes in natural Mo standard.	-	^{93m}Nb standard source.	-
Description of uncertainty calculations	-	Complex, but based on measurement uncertainty (varies), yield (varies) and half-life (20%). Means calculated as power moderated mean (see note).	-	Sources of uncertainty: masses and LSC measurements.	Combined uncertainty including sample preparation and measurement uncertainty	-	Combined uncertainty with the sample preparation and measurement uncertainties	-

5.4 Preliminary radiochemical ^{93}Zr results

The sample specific ^{93}Zr analysis methodologies are reported in Table 7. The overview of the radiochemical methods included resin treatments and evaporations. The sample measurements were carried out in liquid form using LSC and ICP-MS. The mass spectrometric measurement calibrations were carried out using Zr isotopes in natural Zr standard. The LSC efficiency calibrations were carried out using ^{63}Ni surrogate efficiency curve or TDCR measurement. The uncertainty calculations were reported to be mainly combined uncertainties. The references were either internal instructions or a publication (Shimada & Kameo, 2016).

The preliminary ^{93}Zr analysis replicate results are shown in Figure 5. The results show that two results were quantitative (Sample 4 and 7) and one, which is between the quantitative results, below LOQ (Sample 5). Additionally, there is a significant difference between Sample 4 and 7 results.

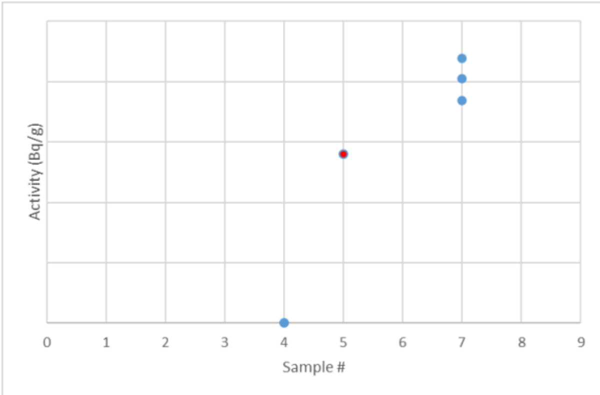


Figure 5. Preliminary ^{93}Zr results in high activity steel sample. The uncertainties are stated with a coverage factor of $k = 2$. Red dot denotes a result below LOQ. Analysis methodologies expressed in **Table 7**.

Table 7. Zirconium-93 analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview of the radiochemical method	-	-	-	Acid digestion with aqua regia and HF. Stable carrier added for yield determination. Cations removed with cation exchange resin. Zr separated from Mo and Nb using TEVA resin. Final fraction in 3 M HNO_3 containing some HF. Yields determined with ICP-OES and activity measurements were performed with LSC.	Zr separation using DOWEX + TEVA resin. Sample was diluted in 2 % v/v HNO3 and 0.05 % v/v HF and analyzed using TQ-ICP-MS.	-	0.2 mg of Nb, Zr, Mo were added as tracer 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.	-
Measurement information	-	-	-	1 ml of sample mixed with 10 ml Hidex Aqua Light+. Stabilisation time 12 h. Measured 5 times 3600 s using template for ^{63}Ni. Utilisation of guard.	ICP-MS measurement on separated Zr fraction.	-	1 mL of purified Zr fraction was mixed with 15 mL Pico-Flour LLT and 4 mL n-hexane. Samples were measured for 30 minutes. Yield was determined by measuring the Zr content with ICP-OES.	-
Equipment	-	-	-	Hidex 300 SL	iCAP TQ-ICP-MS (Thermofisher scientific)	-	PerkinElmer Tri-Carb 4910TR	-
Efficiency calibration	-	-	-	CoreF corrected TDCR	Sensitivity of Zr isotopes in natural Zr standard.	-	^{63}Ni surrogate efficiency curve	-
Description of uncertainty calculations	-	-	-	Sources of uncertainty: masses and LSC measurements.	Combined uncertainty including sample preparation and measurement uncertainty	-	Combined uncertainty with the sample preparation and measurement uncertainties	-

5.5 Preliminary elemental analysis results

The elemental analysis methodologies included either acid digestion of an aliquot or original sample. The elemental analyses were carried out using inductively coupled plasma optical emission spectrometry (ICP-OES) and ICP-MS with and without reaction gases. Quality control measures included multielement standards, controls, spiked samples, and subtraction of background (acids). Multielement and monoelement standards were used in calibrations. The results in Figure 6 show good alignment of results for Fe and Co. Sample 4 and 5 Ni results aligned well whereas Sample 7 result was significantly lower and most likely originating from a calculation error. Nb results align relatively well. Sample 4 and 5 Mo results align well whereas Sample 7 result is higher than the others. Sample 7 analyses were carried out using ICP-OES and difference may originate from result being close to limit of quantification. Zr results show that the submitted results are significantly different. Zr analysis is difficult due to its chemistry, and more results would have been needed for better conclusions. In general, precise acid conditions would have been interesting to know as HF is needed for Zr stability. Sample 7 acids were not disclosed whereas Sample 4 utilised HF. Sample 4 and 5 Mn results aligned well whereas Sample 7 result was a little lower. Ag results showed one result above limit of quantification (Sample 4) and one result below limit of quantification (Sample 7). The difference in the Ag results originate most likely due to the different detection limits of mass and optical emission spectrometers. Only one result was submitted for Se, Ho, Ce, Sm, Eu, Li, and Nd (not shown). No results were submitted for Cl.

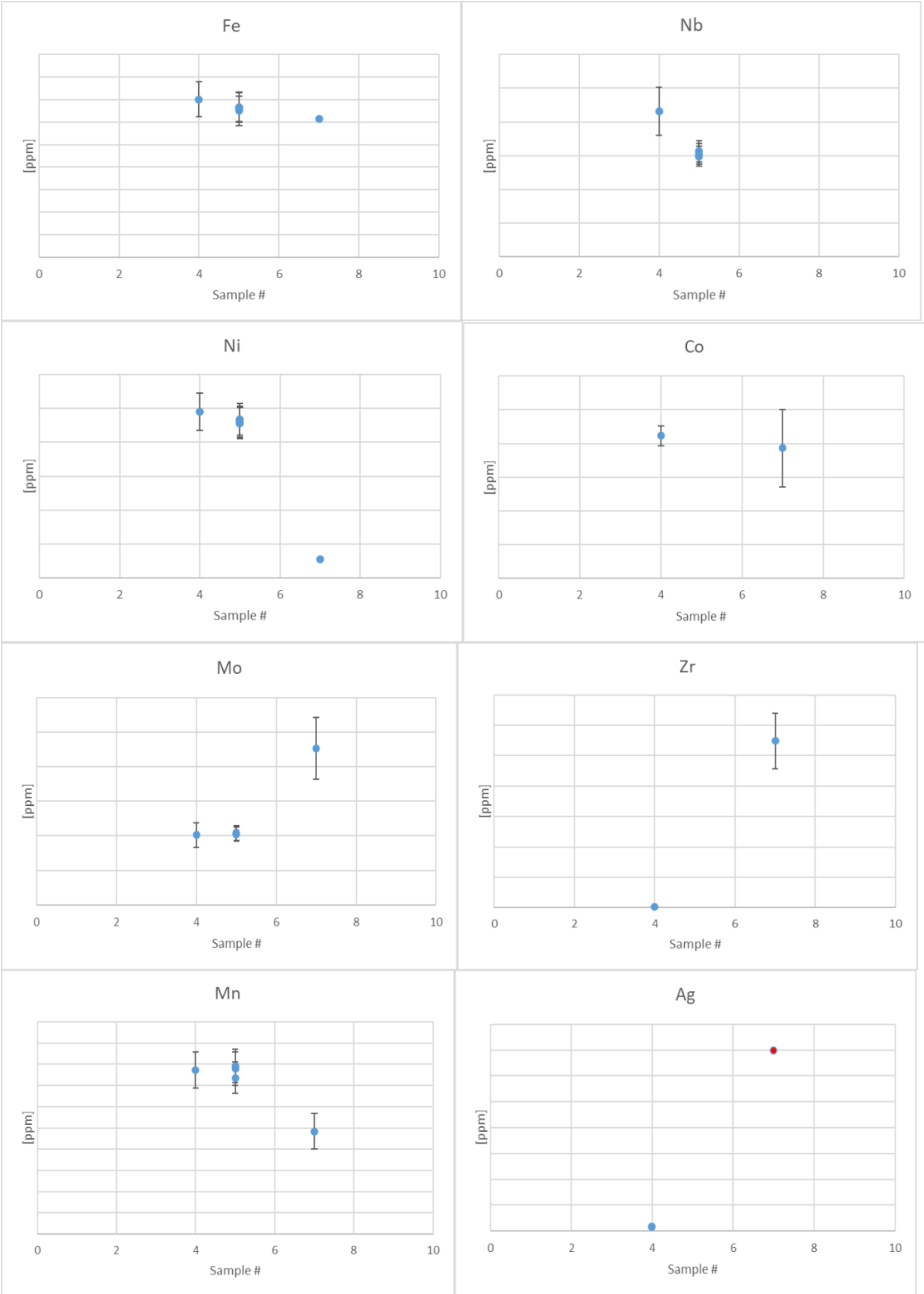


Figure 6. Preliminary elemental analysis results in high activity steel sample. The uncertainties are stated with a coverage factor of $k = 2$. Red dot denotes a result below LOQ. Analysis methodologies expressed in Table 8 and Table 9.

Table 8. Optical emission spectrometric elemental analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview description of the analyses	-	-	-	33 mg of NKS MoNi II 4 activated steel was digested in 8 ml aqua regia and 0.1 ml conc. HF in a PFA beaker on a hotplate. High purity acids were used in digestion. Digested solution was transferred to a plastic vial with some MilliQ water. The elemental analysis was carried out with ICP-OES and QQQ-ICP-MS.	Elemental analysis was carried out on an aliquot of the mother solution where the solid sample was dissolved.	-	The acid digestion solution was analyzed by Agilent 5100 ICP-OES.	-
Overview of the analysis method	-	-	-	Two different dilutions (1:10 and 1:100) were prepared from the original sample solution. Samples and calibration solutions were diluted with u.p. 1% HNO3. Calibration was performed using multielemental standards. Several wavelenghts were measured for each analyte. Results were calculated by choosing one wavelenght that seemed to give reliable results based on control samples and measurement RSD%. The control samples used were multielemental standards (different from the calibration solution) and samples spiked with a known amount of the analyte. Background signal from the 1% HNO3 was substracted from the results.	-	-	The acid digestion solution was analyzed by ICP-OES	-
Measurement information	-	-	-	Measured elements: Ag, Ce, Co, Eu, Fe, Ho, Li, Mn, Mo, Nb, Nd, Ni, Se, Sm, Zr		-	Fe, Ni	-
Equipment	-	-	-	Agilent 5100 ICP-OES		-	Agilent 5100 ICP-OES	-
Calibration	-	-	-	Before measurement, detector calibration and wavelenght calibration were performed. In the measurement, multielemental calibration solutions in the range 0.5-50 ppm were used.	-	-	Establish calibration curve with MERCK standard solution.	-

Description of uncertainty calculations	-	-	-	Uncertainties were calculated as combined uncertainty, taking account uncertainties associated with weighings, dilutions, measurement RSD%, measurement calibration and measurements accuracy. Uncertainties were not determined for results under method detection limit.	-	-	Combined uncertainty with the sample preparation and measurement uncertainties.	-

Table 9. Mass spectrometric analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview description of the analyses	-	-	-	33 mg of NKS MoNi II 4 activated steel was digested in 8 ml aqua regia and 0.1 ml conc. HF in a PFA beaker on a hotplate. High purity acids were used in digestion. Digested solution was transferred to a plastic vial with some MilliQ water. The elemental analysis was carried out with ICP-OES and QQQ-ICP-MS.	Elemental analysis was carried out on an aliquot of the mother solution where the solid sample was dissolved.	-	The acid digestion solution was analyzed by Agilent 5100 ICP-OES.	-
Overview of the analysis method	-	-	-	Two different dilutions (1:50 and 1:100) were prepared from the original sample solution. Samples and calibration solutions were diluted with u.p. 1% HNO3. Internal standards (10 ppb Y and Tb) were added to each measured solution. Calibration was performed using multielemental standards. Mo, Nb and Zr were measured in O2 mass-shift mode and Ag, Ce, Co, Eu, Ho, Nd, Se and Sm in He mode. Control samples were measured to evaluate the accuracy of the results. The control samples used were multielemental standards (different from the calibration solution) and samples spiked with a known amount of the analyte. Background signal from the 1% HNO3 was subtracted from the results.		-	-	-
Measurement information	-	-	-	Measured elements: Ag, Ce, Co, Eu, Ho, Mo, Nb, Nd, Se, Sm, Zr	Fe, Ni, Nb, Mo, Co, Zr, Mn, Ho, Ce, Sm, Eu, Li, Nd	-	-	-
Equipment	-	-	-	Agilent 8900 QQQ-ICPMS	Agilent 7800 ICP-MS	-	-	-

Calibration	-	-	-	Multielemental calibration solutions in the range 0.01-50 ppb were used.	External calibration using corresponding mono-elemental standard	-	-		-
Description of uncertainty calculations	-	-	-	Uncertainties were calculated as combined uncertainty, taking account uncertainties associated with weighings, dilutions, measurement RSD%, measurement calibration and measurements accuracy. Uncertainties were not determined for results under method detection limit.	Combined uncertainty	-	-		-

5.6 Preliminary radiochemical ^{36}Cl results (optional)

The sample specific ^{36}Cl analysis methodologies are reported in Table 10. The overview of the radiochemical methods included resin treatments, precipitations, fixation, and liquid-liquid extractions. The sample measurements were carried out in liquid form using LSC and alpha/beta counting. The efficiency calibrations were carried out using ^{36}Cl standards. The uncertainty calculations were reported to be mainly combined uncertainties. One reference on radiochemistry was reported (Fréchou & Degros, 2005).

The preliminary ^{36}Cl analysis replicate results are shown in Figure 7. The results show that two results were below limit of quantification whereas one quantitative result (Sample 5) was significantly higher. However, Sample 5 result was submitted with a notion that the result probably is an overestimation due to some interference in the LSC spectrum. As the LSC spectrum was not available, no concrete suggestions were made for the origin of the contamination. Additionally, difficulties in ^{36}Cl analyses originate from volatility and yield determinations, which may be difficult depending on the measurement technique.

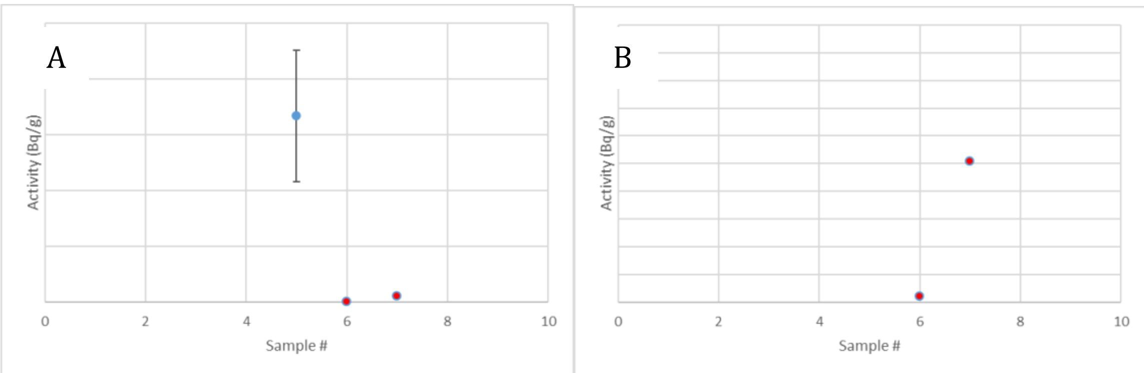


Figure 7. Preliminary ^{36}Cl results in high activity steel sample. All results (A) and Sample 5 excluded (B). The uncertainties are stated with a coverage factor of $k = 2$. Red dot denotes a result below LOQ. Analysis methodologies expressed in Table 10.

Table 10. Chlorine-36 analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview of the radiochemical method	-	-	-	-	Separation of Cl by precipitation as AgCl. Dissolution of AgCl in 25% NH_3 and analysis with LSC. Yield determination by measurement of natural Cl with IC.	Microwave digestion with addition of $AgNO_3$ and NaCl. Precipitation of AgCl, elimination of Ag by precipitation with sulphate, fixation of chlorine on octadecene, liquid-liquid extraction for final purification	The sample is treated with $AgNO_3$ to form AgCl(s), and NH3 to dissolve the precipitation. Cl- was separated by AG1x4 ion exchange resin. Collect the eluant, and add $AgNO_3$ to form precipitation of AgCl(s).	-
Measurement information	-	-	-	-	LSC	5 ml of purified ^{36}Cl 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 luminescense. Sample was measured once 10800s. Yield was determined by measuring stable Cl after separation ion chromatography.	AgCl(s) precipitation on the filter paper. Yield was determined by measuring the stable Cl content.	-
Equipment	-	-	-	-	Tri-Carb 3110 TR	AccuFLEX LSC-8000 Hitachi for LSC, Ion chromatography (ICS2100 Thermo).	Series 5 XLB Automatic Low Background Alpha/Beta Counting System.	-
Efficiency calibration	-	-	-	-	Internal ^{36}Cl standard	Quench curve with ^{36}Cl source	^{36}Cl efficiency curve.	-
Description of uncertainty calculations	-	-	-	-	Combined uncertainty	Combined uncertainty with the sample preparation, recovery yield and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties.	-

5.7 Preliminary radiochemical ^{94}Nb results (optional)

The sample specific ^{94}Nb analysis methodologies are reported in Table 11. The overview of the radiochemical methods included resin treatments, liquid-liquid extractions, precipitations, and evaporations. The sample measurements were carried out in liquid form or solid form (precipitate) using gamma spectrometry. The efficiency calibrations were carried out using ISOCS, LabSOCS, mixed gamma standard, or ^{152}Eu/mixed source. The uncertainty calculations were reported to be mainly combined uncertainties. The references were internal instructions or in-house methods (not published).

The preliminary ^{94}Nb analysis replicate results are shown in Figure 8. The overall results were assessed to be good especially considering the analysis difficulties, due to Nb instability in solution.

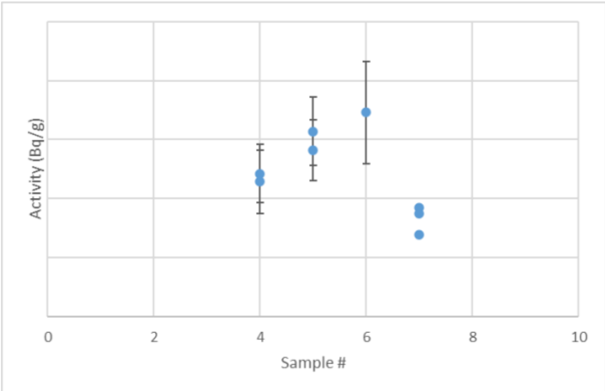


Figure 8. Preliminary ^{94}Nb results in high activity steel sample. The uncertainties are stated with a coverage factor of $k = 2$. Analysis methodologies expressed in Table 4.

Table 11. Niobium-94 analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview of the radiochemical method	-	-	-	Acid digestion with aqua regia and HF. Stable carrier added for yield determination. Cations removed with cation exchange resin. Nb separated from Mo and Zr using TEVA resin. Final fraction in 1 M HNO_3 containing some HF. Yields determined with ICP-OES and activity measurements were performed with ISOCS.	Nb separation using Dowex resin; Yield determination by analysis of Nb before and after the separation with ICP-MS	Microwave digestion with stable Nb, HNO_3 and HF. Liquid-liquid extraction with Diisopropyl ketone (DIPK) and then liqui-liquid extraction with N-benzoyl-phenyl hydroxylamine (BPHA) in chloroform. Final precipitation of Nb with Pyrrolidine dithiocarbamate (PDTC)	0.2 mg of Nb, Zr, Mo were added as tracer in each aliquot sample. The aliquot samples were first evaporated to near dryness and dissolved in 0.1 M HF. ^{93m}Nb, ^{94}Nb, ^{93}Zr, ^{93}Mo were separated using TEVA resin.	-
Measurement information	-	-	-	Measured on top of the detector. Measurement times 90600 s and 152961 s. Coinsidence correction not carried out.	Gamma measuremant on the separated Nb-94 fraction	Precipitate was measured at 6 mm distance on top of the detector. Sample was measured 240000 s.	Samples were measured by HPGe for 2 hours. Yield was determined by measuring the Nb content with ICP-OES.	-
Equipment	-	-	-	Mirion HPGe, 15% S/N-B18067	BeGe gamma detector Canberra	Canberra BEGe BE3830 (carbon epoxy window) ; 34% relative efficiency Mettler Toledo XP 504 balance for final Nb precipitate	Canberra HPGe 4020	-
Efficiency calibration	-	-	-	ISOCS	mixed gamma standard	LabSOCS efficiency calibration	^{152}Eu standard source.	-

Description of uncertainty calculations	-	-	-	Sources of uncertainty: masses and spectral.	Combined uncertainty	Combined uncertainty with the sample preparation, recovery yield and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties	-

5.8 Preliminary radiochemical ^{93m}Nb results (optional)

The sample specific ^{93m}Nb analysis methodologies are reported in Table 12. The overview of the radiochemical methods included evaporations, resin treatments, liquid-liquid extractions, and precipitations. The sample measurements were carried out in liquid and solid forms (evaporated on a filter or precipitate). The detections were carried out using gamma spectrometry (LEGe) and LSC. The LSC efficiency calibration was carried out using ^{93m}Nb standard solution, the gamma spectrometric LEGe efficiency calibration was carried out using either a ^{93m}Nb standard source or multi-nuclide standard. The uncertainty calculations were reported to be combined uncertainties. The methodologies were reported to be not published internal instructions and in-house methods.

The preliminary ^{93m}Nb analysis replicate results are shown in Figure 9. The results show a good alignment. The overall results were assessed to be good especially considering the analysis difficulties.

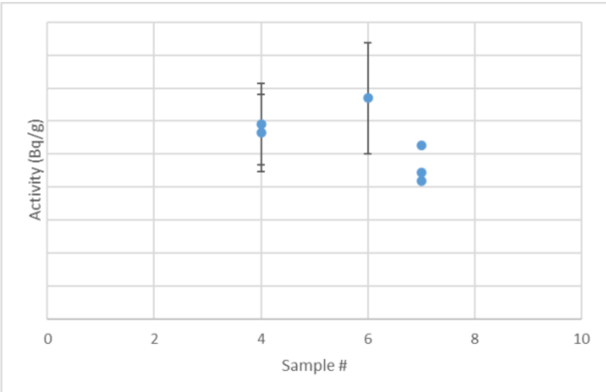


Figure 9. Preliminary ^{93m}Nb results in high activity steel sample. The uncertainties are stated with a coverage factor of $k = 2$. Analysis methodologies expressed in Table 12.

Table 12. Niobium-93m analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview of the radiochemical method	-	-	-	Acid digestion with aqua regia and HF. Stable carrier added for yield determination. Cations removed with cation exchange resin. Nb separated from Mo and Zr using TEVA resin. Final fraction in 1 M HNO_3 containing some HF. Yields determined with ICP-OES and activity measurements were performed with LSC.	-	Microwave digestion with stable Nb, HNO_3 and HF. Liquid-liquid extraction with Diisopropyl ketone (DIPK) and then liqui-liquid extraction with N-benzoyl-phenyl hydroxylamine (BPHA) in chloroform. Final precipitation of Nb with Pyrrolidine dithiocarbamate (PDTC)	0.2 mg of Nb, Zr, Mo were added as tracer 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.	-
Measurement information	-	-	-	2 ml of sample mixed with 10 ml Hidex Aqua Light+. Stabilisation time 12 h. Measured 3 x 14 400 s using template for ^{93m}Nb. Utilisation of guard and external standard.	-	Precipitate was measured at 8 mm distance on top of the detector. Sample was measured 400000s.	1 mL of purified Nb fraction were dropped onto 1-inch filter paper and were evaporated to dryness. Samples were measured for 2 hours. Yield was determined by measuring the Nb content with ICP-OES.	-
Equipment	-	-	-	Hidex 300 SL	-	Canberra LEGe GL0210 (Be window). Mettler Toledo XP 504 balance for final Nb precipitate	Canberra LEGe GL0210	-

Efficiency calibration	-	-	-	^{93m}Nb quenching curve using QPE (external standard)	-	Equivalent geometry multi-nuclide efficiency calibration & sample/standard attenuation correction	^{93m}Nb standard source.	-
Description of uncertainty calculations	-	-	-	Sources of uncertainty: masses and LSC measurements.	-	Combined uncertainty with the sample preparation, recovery yield and measurement uncertainties	Combined uncertainty with the sample preparation and measurement uncertainties	-

5.9 Preliminary radiochemical ^{55}Fe results (optional)

The sample specific ^{55}Fe analysis methodologies are reported in Table 13. These results were not discussed in the preliminary meeting as there was only one data entry at this point.

Table 13. Iron-55 analysis methodologies submitted prior to the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview of the radiochemical method	-	-	-	-	-	-	An aliquot was taken for the original chemical composition studies using ICP-OES first. ^{55}Fe was purified in conjuction with $^{59/63}$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 inpurities.	-
Measurement information	-	-	-	-	-	-	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.	-
Equipment	-	-	-	-	-	-	PerkinElmer Tri-Carb 4910TR and Agilent 5100 ICP-OES	-
Efficiency calibration	-	-	-	-	-	-	Using standard ^{55}Fe sources with the same activities but different tSIEs to fit an efficiency regression line on LSC.	-
Description of uncertainty calculations	-	-	-	-	-	-	Combined uncertainty with the sample preparation and measurement uncertainties, including the efficiency, counts, weight, recovery rates.	-

5.10 Preliminary radiochemical ^{63}Ni results (optional)

The sample specific ^{63}Ni analysis methodologies are reported in Table 14. The overview of the radiochemical methods included different resin or DMG treatments and precipitation. The activities were measured using LSC with either internal ^{63}Ni standard or ^{63}Ni quenching curve. The uncertainty calculations were reported to be combined uncertainties.

The preliminary ^{63}Ni analysis replicate results are shown in Figure 10. The results show a good alignment between the submitted results.

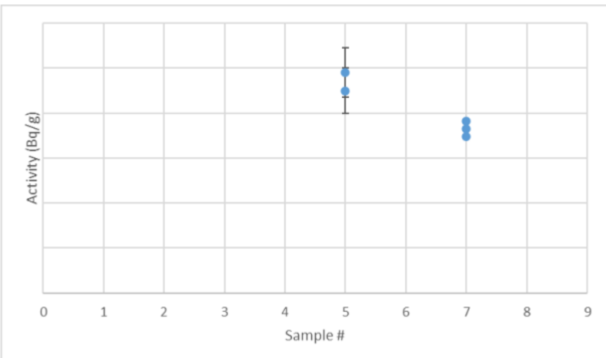


Figure 10. Preliminary ^{63}Ni results in high activity steel sample. The uncertainties are stated with a coverage factor of $k = 2$. Analysis methodologies expressed in Table 14.

Table 14. Nickel-63 analysis methodologies discussed in the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview of the radio- chemical method	-	-	-	-	Ni was separated using an Fe/Co separation on a Dowex resin followed by a Ni separation on Ni resin from Triskem. Separation yield was determined by the analysis of the Ni concentration before and after the separation.	-	An aliquot was taken for the original chemical composition studies using ICP-OES first. $^{59,63}Ni$ was 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.	-
Measurement information	-	-	-	-	0,5 g of the purified ^{63}Ni fraction was mixed with HiSafe cocktail and measured 3 times for 10 minutes.	-	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.	-
Equipment	-	-	-	-	Tri-Carb 3110 TR	-	PerkinElmer Tri-Carb 4910TR and Agilent 5100 ICP-OES	-
Efficiency calibration	-	-	-	-	Internal ^{63}Ni standard	-	Using standard ^{63}Ni sources with the same activities but different tSIEs to fit an efficiency regression line on LSC.	-
Description of uncertainty calculations	-	-	-	-	Combined uncertainty	-	Combined uncertainty with the sample preparation and measurement uncertainties, including the efficiency, counts, weight, recovery rates.	-

5.11 Preliminary radiochemical ^{59}Ni results (optional)

The sample specific ^{59}Ni analysis methodologies are reported in Table 15. These results were not discussed in the preliminary meeting as only one set of results were submitted.

Table 15. Nickel-59 analysis methodologies submitted prior to the preliminary meeting.

	1	2	3	4	5	6	7	8
Overview of the radiochemical method	-	-	-	-	-	-	An aliquot was taken for the original chemical composition studies using ICP-OES first. $^{59/63}$Ni was purified in conjuction 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.	-
Measurement information	-	-	-	-	-	-	Purified $^{59/63}$Ni fraction was let to dry on a membrane filter. Samples were measured 3 times 20 hrs. Yield was determined by measuring the stable Ni content in the purified fraction.	-
Equipment	-	-	-	-	-	-	Mirion(Canberra) LEGe GL0210 and Agilent 5100 ICP-OES	-
Efficiency calibration	-	-	-	-	-	-	Calibrated by ^{152}Eu standard source.	-
Description of uncertainty calculations	-	-	-	-	-	-	Combined uncertainty with the sample preparation and measurement uncertainties, including the efficiency, counts, weight, recovery rates.	-

5.12 Preliminary meeting conclusions

The general preliminary meeting conclusions were:

- Lack of time or lack of sample mass hindered submission of more ^{60}Co results.
- More elemental analysis results would have been appreciated. However, the sample size limited the number of analyses.
- ^{79}Se would have been an interesting DTM, but referenced activities were too low for such sample size. Similar problem also with ^{36}Cl.
- Several ^{14}C results gave the opportunity to assess a volatile DTM results.
- The results were good for ^{94}Nb and ^{93m}Nb. The analysis is difficult and now there were consistent results with couple of detection techniques.
- Improvement in ^{93}Zr results compared to MoNi.

6. Methodology for statistical analysis of the reported results

The statistical analysis of the reported results was carried out according to the ISO 13528 (International Standard, 2022) standard on Statistical methods for use in proficiency testing by interlaboratory comparison. Calculation of assigned values were based on the participants' results and carried out using a robust statistical method (International Standard, 2022). Algorithm A was used in calculation of robust mean and robust standard deviation. Algorithm A is robust for outliers, when the expected proportion of outliers is less than 20% (International Standard, 2022). Performance assessments were carried out using z scores (Eq. 1) (International Standard, 2022). The analysis results with z score were (International Standard, 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 was large ($1\sigma > 20\%$), the uncertainty of the assigned value (Eq. 2) was used as σ_{pt} in order to give an action signal for results which were not fit for purpose (International Standard, 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 to revise their results between the preliminary and final meetings. The revisions included:

- New set of results for Sample 1 i.e., ^{60}Co, ^{93}Mo, ^{14}C, and elemental.
- New set of results for Sample 3 i.e., ^{36}Cl.
- Replicate ^{14}C analysis for Sample 4.
- Updated elemental results for Sample 7 i.e., Ni and Li.
- Updated results for Sample 8 i.e., ^{60}Co, ^{55}Fe, ^{63}Ni, ^{14}C, ^{36}Cl, ^{3}H and elemental.

The new and updated analysis yields are shown in Table 16 and methodologies in Table 17 and

Table 20. References were reported for in-house methods and publications (Hou, 2005; Hou et al., 2007).

Statistical analyses were carried out for results which had at least five data entries. Indicative statistical analyses were carried out for results which had four data entries and illustrative analyses were carried out for results with 3 data entries. Other results were reported as trend graphs. The discussed results in the final meeting were yet again updated in this report to include all sets of results:

- Sample 6 ^{60}Co result in aliquot acid (previously allocated as original sample vial).
- Sample 1 and Sample 5 elemental results.

When available, the MoNi II results were compared with MoNi results.

Table 16. New and updated radiochemical yields and uncertainties (k = 2) for the final report. Yields discussed in the preliminary meeting are in **Table 3**.

	Yield ± uncertainty [%] (k =2)				
	^{14}C	$^{59,63}Ni$	^{36}Cl	^{3}H	^{55}Fe
1	40±4	-	-	-	-
3	95±24		27-45	-	-
4	49±20	-	-	-	-
8	85±3	87	3-95	98±3	75

Table 17. New and updated radiochemical methods of Samples 1 and 3. The methodologies discussed in the preliminary meeting are in Table 5 - Table 15.

	1	1	3	3
	^{93}Mo	^{14}C	^{14}C	^{36}Cl
Overview of the radiochemical method	Steel sample was digested using a mixture of 4 M H_2SO_4 and 1 M H_3PO_4, evaporated to dryness and dissolved in 9 M HCl. Mo was separated using AG1x4 anion exchange column. Interfering elements were first removed using (1) 9 M HCl (Ni), 5 M HCl (Zr, Co, Nb), (3) 0.05 M HNO_3 + 2% H_2O_2 (Fe, Nb), and then Mo was eluted with 8 M HNO_3. The eluted solution was measured by ICP-MS/MS both for stable Mo and ^{93}Mo. Yield was measured using stable Mo sample taken prior to separation.	Oxidative acid digestion was used to determinate ^{14}C from the steel sample. Sample was dissolved using 4 M H_2SO_5 and 1 M H_3PO_4 mixture and ^{14}C was oxidized (potassium per sulphate) and trapped in 0.4 M NaOH solution. ^{14}C was measured with LSC and yield was determined with ^{14}C standard.	Decomposition of steel by acid digestion to release C, Carbosorb and Perma Flurer to collect the ^{14}C	Acid digestion to decompose the matrix and release Cl. Separation of Cl by precipitation as AgCl, repeat 2-3 times. Purification of ^{36}Cl with AG1×4 ion exchange resin. Collect the eluant and heat to near dryness, dissolve the residue with 5mL MilliQ water.
Measurement information	Purified Mo fraction was diluted 10 and 100 times with 2 % HNO_3 and measured using ICP-MS/MS.	5 m L of the trap solution (NaOH) was mixed with 15 mL of HiSafe 2 (Perkin Elmer) liquid scintillation couting cocktail in 20 mL plastic vial. Samples were left to stabilize overnight to reduce luminescense and they were measured 3 times for 600 min. Yield was determined with C-14 standard.	Measurement ^{14}C by liquid scintillation counting, the sample and blank were counted for 5 cycles of 60 min each.	5mL purified ^{36}Cl fraction was mixed with 15mL of HiSafe 3 cocktail in a 20mL glass vail. Samples were measured for 5 cycles of 60 min each. Yield was determined by measuring stable Cl by ICP-MS/MS.

Equipment	Thermo Fisher iCAP TQ ICP-MS	Quantulus 1220 LSC counter (Wallac)	Wallac 1220 Quantulus liquid scintillation counter	Wallac 1220 Quantulus liquid scintillation counter/Agilent 8800 ICP-MS/MS
Efficiency calibration	ICP-MS/MS: Calibration with stable Mo standard solution and mass bias interpolation.	Efficiency was determined using a solid-standard method.	~40% derived from the counting result and spiked standard	~90% derived from the counting result and spiked standard
Description of uncertainty calculations	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,recovery yield and measurement uncertainties

Table 18. New and updated radiochemical methods of Sample 8. The methodologies discussed in the preliminary meeting are in Table 5 - Table 15.

	8	8	8	8	8
	^{55}Fe	^{63}Ni	^{14}C	^{36}Cl	^{3}H
Overview of the radiochemical method	An aliquot of the sample is spiked with stable Fe and Ni and a sub-sample is removed for stable determination. Fe and Ni are separated from one another using di-isopropyl ether solvent extraction. The Ni-63 is then purified further using ion-exchange (AG1-X8) and extraction chromatography (TRU and Ni resin) and the activity determined by liquid scintillation counting. Iron is back-washed from the solvent phase and preciptated as the hydroxide before redissolution and LSC source preparation before liquid scintillation counting. Chemical recovery is determined gravimetrically for Fe and by ICP-OES analysis of stable Ni and Fe in the initial fractions and stable Ni in the purified fractions.	See ^{55}Fe	A known mass of sample material is combusted in a two-stage catalytic pyrolyser. The sample, in a sample boat, is placed into the sample zone furnace and and gradually combusted under a steady flow of carrier gas (air) and usually oxygen until the release of tritium and carbon-14 is complete at 900°C. The $^{14}CO_2$ is trapped in a bubbler of carbon dioxide absorber (Carbosorb) and the tritium as tritiated water (HTO) in a bubbler of 0.1M nitric acid.	Sample was leached/dissolved in a round-bottom flask with Leibig condenser using 0.1 M nitric acid with hydrochloric acid. An aliquot of the digest is taken and a hydroxide scavenge performed then further purification using ion exchange (Eichrom 50Wx8) carried out. Silver nitrate is added to the eluate to precipitate silver chloride prior to LSC source preparation.	Measured from the same portion used for ^{14}C.

Measure-ment information	Dissolve the FeOH precipitate using the minimum 3M nitric acid then evaporate to dryness. Dissolve the residue in 1mL 2M phosphoric acid then dispense 15 ± 0.5mL Ultima Gold AB liquid scintillation cocktail into the vial. Wipe the outside of the vials with a tissue dampened with acetone, to remove any marks. Allow the vials to dark adapt for at least 1 hour before counting	Nickel is eluted from the Ni resin column with 4mL of 3M nitric acid then evaporated to dryness. The residue is dissolved with 1mL of 0.1M nitric acid and dispense 15 ± 0.5mL Ultima Gold AB liquid scintillation cocktail. Wipe the outside of the vials with a tissue dampened with acetone, to remove any marks. Allow the vials to dark adapt for at least 1 hour before counting.	6 ml of the Carbosorb E from the ^{14}C bubbler is added to a glass LSC vial and 14ml of Permafluor added.	Precipitate dissolved in 2 ml concentrated ammonia with heat, then 3 mL of water added to the vial, mixed thoroughly, then 15 mL Ultima Gold AB liquid scintillant added.	A portion of the bubbler solution is added to Ultima Gold LLT LS cocktail
Equipment	Hidex 600 Sle. Agilent 725 ICP-OES	Perkin Elmer Tri-Carb 2910TR. Agilent 725 ICP-OES	Raddec Pyrolyser. Hidex 600 SLe liquid scintillation counter	Perkin Elmer Tri-Carb 2910TR liquid scintillation counter	
Efficiency calibration	Count standard containing a known activity of ^{55}Fe prepared in the same way as for the samples as described in measurement information above.	Count standard containing a known activity of ^{63}Ni prepared in the same way as for the samples as described in measurement information above.	Quench curve (tSIE/QPE vs efficiency) produced per LSC for a series of ^{14}C standard vials of known activity and increasing quench.	Counting efficiency was determined from counting a ^{36}Cl standard source and the overall method yield determined from a combination of ^{36}Cl dosed runs and chemical recovery based on calibrated chloride carrier addition	Quench curve (tSIE/QPE vs efficiency) produced per LSC for a series of ^{3}H standard vials of known activity and

					increasing quench.
Description of uncertainty calculations	Combined measurement uncertainty	Combined measurement uncertainty	Combined measurement uncertainty	Counting statistics only	Combined measurement uncertainty

Table 19. Updated elemental analysis methodologies. The elemental analysis methodologies discussed in the preliminary meeting are in Table 8 and Table 9.

	1	8	8
Overview of the analysis method	Stable elements were analyzed from the dissolved steel sample by ICP-MS/MS.	ICP-OES 750 series Agilent spectrometer results used for Fe, Ni, Mn	NEXION 5000 mass spectrometer results used for other elements:
Measurement information		Sample dissolution liquor diluted x10 and x100 in duplicate with an additional aliquot spiked with mixed elemental spiking solution	Sample dissolution liquor diluted x10 and x100 in duplicate with an additional aliquot spiked with mixed elemental spiking solution.
Equipment	Thermo Fisher iCAP TQ ICP-MS	ICP-OES 750 series Agilent spectrometer	Perkin Elmer Nexion 5000 ICP-MS
Calibration	ICP-MS/MS: Calibration with stable standard solutions.	5ppm Yttrium as internal standard added to samples to check/correct for matrix effects or sensitivity drift over batch analysis, 0.5, 1, 2, 5, 10ppm (µg/mL) calibration range	10 ppb Indium as internal standard autodiluted into samples as they are pumped into spray chamber of instrument, to check/correct for matrix effects or sensitivity drift over batch analysis. 1, 5, 10, 20, 50, 100 (ng/mL) calibration standards, top standards removed down to 20ppb where necessary
Description of uncertainty calculations	5 % instrument uncertainty	Combined measurement uncertainty	Combined measurement uncertainty

Table 20. Updated ETM analysis methodologies. The ETM analysis methodologies discussed in the preliminary meeting are in Table 4.

	1	8
Sample preparation	Sample was measured as received using the provided mass information.	As-received sample (4 x pieces) placed in a glass v-vial and measured using a HPGE detector. A calibration geometry was prepared by adding mixed gamma reference solution onto a ~ 30mg aliquot of blank steel in a similar v-vial, allowed to dry and the calibration performed (note that some dissolution of the steel was observed). Note that we would consider the measurements of undissolved steel to be semi-quant at best. Four separate measurements of single piece sub-samples in a v-vial were conducted in the same v-vials and measured using the calibration. A single sub-sample was selected and dissolved using nitric and hydrochloric acids, then the solution filtered into a 100 ml volume in a volumetric flask. The solution and filter paper were transferred to a calibrated geometry and gamma spectrometry performed.
Measurement geometry	Sample was measured at 10 mm distance on top of the detector.	As-received sample and individual single piece sub-samples: glass v-vial on detector end-cap. The 100 ml dissolution solution was measured in a calibrated geometry at ~ 5 cm distance from the detector end-cap.
Dead time	2.20 %	Solid sub-samples: 6- 7 %; Solution: 3 %
Measurement time	5000 s, three replicate measurements	Solid sub-samples: 3600 s; Solution: 50000 s
Detector type and efficiency	Canberra HPGe detector, 31 %	Solution measured using a p-type HPGe detector
Efficiency calibration	Efficiency calibration was performed using a mixed standard solution.	Calibrated using mixed radionuclide solution of metrological traceability
Description of uncertainty calculations	Combined uncertainty with the sample preparation and measurement uncertainties.	For the as-received sample and each sub-sample, the uncertainty is reported as the combined measurement uncertainty. The uncertainty on the average of the sub-samples is reported as twice the standard deviation of the four replicate measurements. The reported uncertainty on the solution activity is the combined measurement uncertainty for the technique.

7.1 Gamma spectrometry results

Indicative statistical analysis of ^{60}Co results in original sample vial were carried out with four data entries. The assigned value with the robust standard deviation (k = 1) in the original sample vial was 447 ± 170 kBq g^{-1}. Illustrative statistical analysis was carried out for aliquots in solid form as only three data entries were submitted. In this case, the assigned value with the robust standard deviation (k = 1) was 501 ± 55 kBq g^{-1}. Five data entries were submitted for analyses carried out in aliquot liquid samples (acid digested sample) and in this case, the assigned value with the robust standard deviation (k = 1) was 478 ± 45 kBq g^{-1}. All the ^{60}Co data entries with the assigned value and uncertainties (k = 2) are shown in Figure 11. The z scores were calculated using the robust standard deviation in aliquot solid and liquid (11 % and 5 %, respectively) whereas standard uncertainty of assigned value (24 %) was used in original sample because the robust standard deviation was large (38 %). As seen in Table 21, all ^{60}Co z scores were in acceptable range. Comparison of MoNi II ^{60}Co results with MoNi results (Leskinen et al., 2025) in Figure 12 show that the assigned value uncertainty in original sample vial is larger compared to the other analyses. However, all the results are comparable.

Homogeneity study was reported for four individual pieces of Sample 8. The results in Figure 13 show that at least in this exercise, the pieces show comparable results. Additionally, the RSD was 8.8 %. The overall results in ^{60}Co analysis show that there does not seem to be a clear heterogeneity in the studied samples.

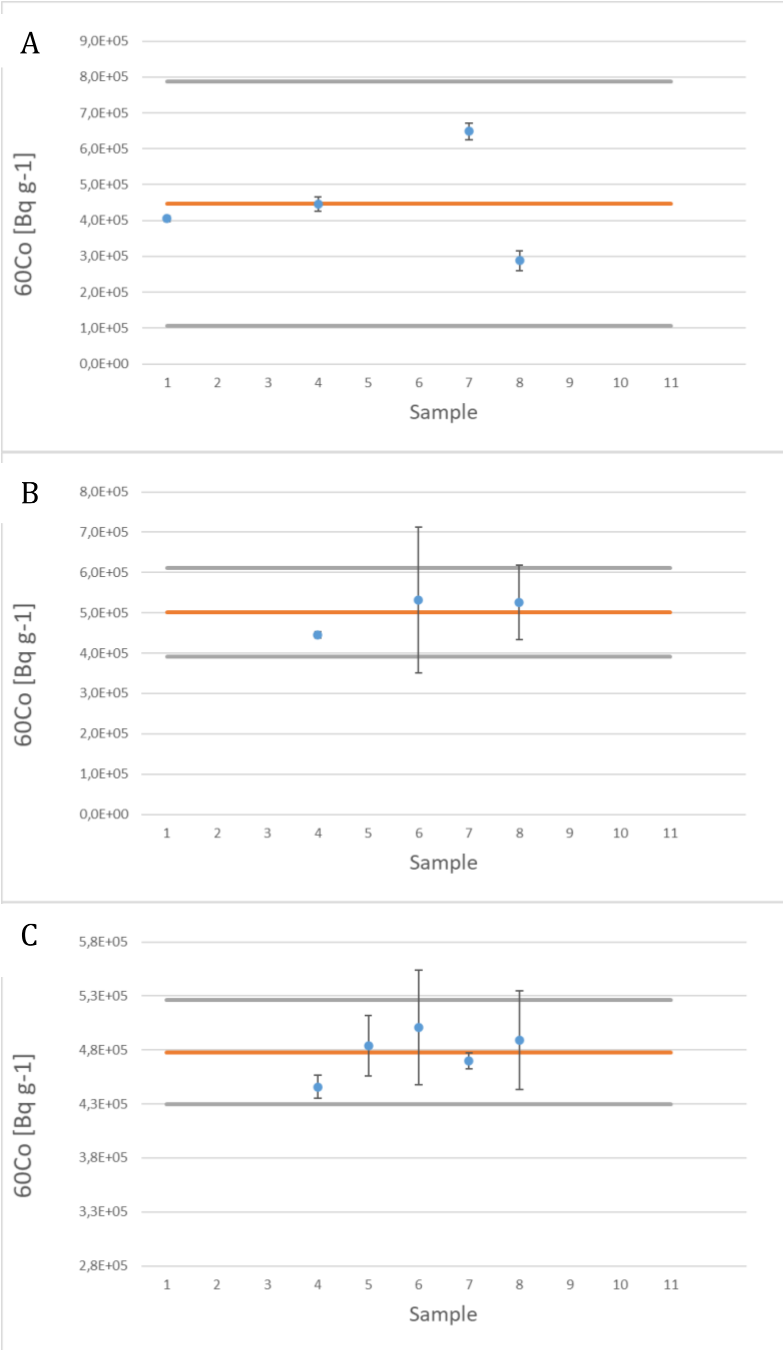


Figure 11. Final ^{60}Co results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Original sample vial results (A), solid aliquot (B), and liquid aliquot (C).

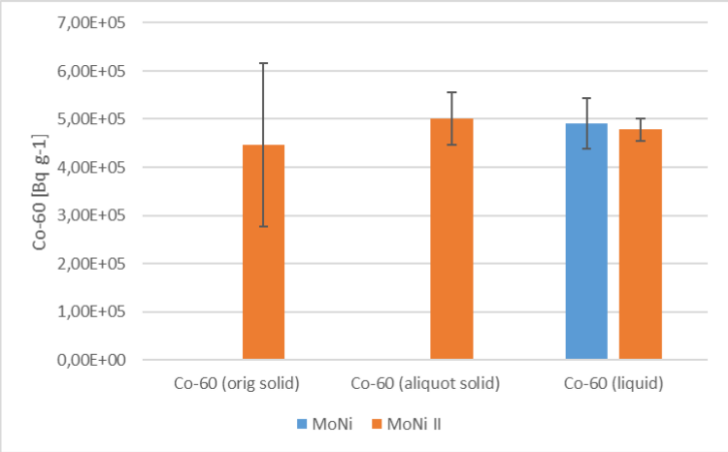


Figure 12. Comparison of final MoNi and MoNi II ^{60}Co assigned values (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

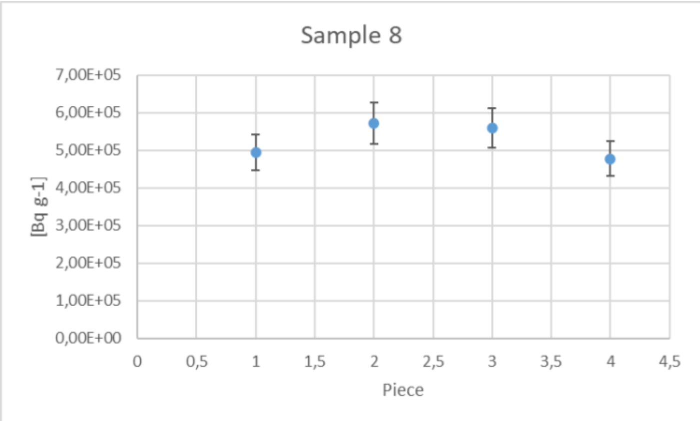


Figure 13. Cobalt-60 measurement results in four pieces of high activity steel with uncertainties stated with a coverage factor of $k = 2$.

Table 21. ETM z scores in MoNi II intercomparison exercise. Results are acceptable when $z \leq 2.0$, warning signal is given when $2.0 < z < 3.0$, and unacceptable when $z \geq 3.0$.

Sample	z score		
	^{60}Co (orig. sample)	^{60}Co (aliquot solid)	^{60}Co (aliquot liquid)
1	0.4	-	-
2	-	-	-
3	-	-	-
4	0.0	1.0	1.3
5	-	-	0.3
6	-	0.6	1.0
7	1.9	-	0.3
8	1.5	0.5	0.5

7.2 Radiochemical analysis results

Indicative statistical analysis of ^{14}C results were carried out with four data entries as two of the submitted results were below limit of detection (Sample 6 and 8) and one result an outlier (Sample 4). The assigned value with the robust standard deviation ($k = 1$) was 115 ± 88 Bq 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 (48 %) since the robust standard deviation was large (77 %). As seen in Table 22, all ^{14}C z scores (Sample 4 outlier) are in acceptable range. Even though Sample 4 was considered an outlier, the analysis had been repeated producing comparable results.

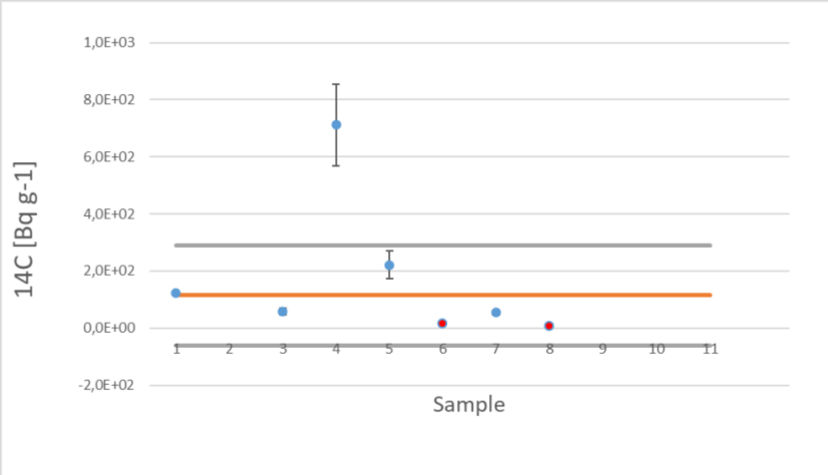


Figure 14. Final ^{14}C results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Red dot indicates a result below limit of quantification.

Statistical analysis of ^{93}Mo results was not carried out even though five data entries were submitted (Figure 15), because three data entries were reported to be below limit of detection and Sample 1 and 2 results were two orders of magnitude different. However, Sample 1 reporting included a remark, that the ^{93}Mo purification had failed as there was another m/z 93 present in the analysis. Therefore, the results were submitted only for information purposes. Comparison of MoNi and MoNi II results (not shown) indicate that the reference ^{93}Mo is most likely somewhere around 10-20 Bq g^{-1}.

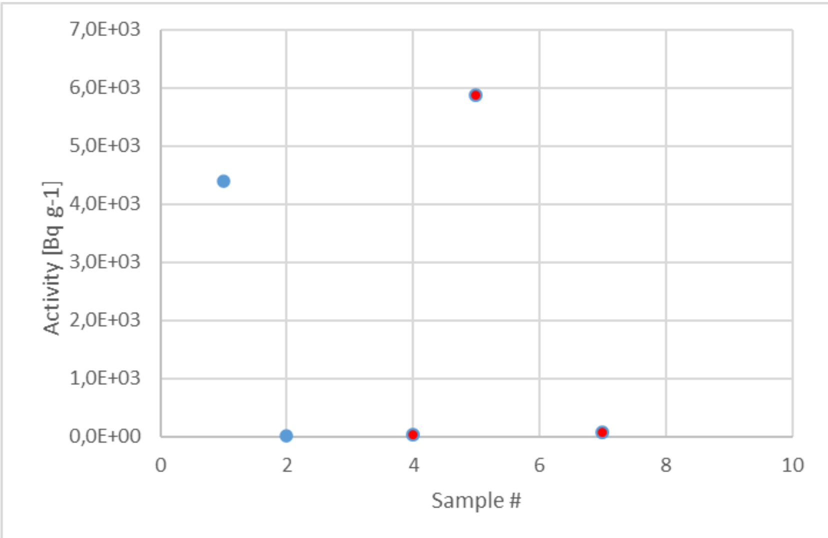


Figure 15. Final ^{93}Mo results in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Red dot indicates a result below limit of quantification.

Statistical analysis of ^{93}Zr results was not carried out, because one out of three data entries were reported to be below LOQ (Figure 16). Additionally, this Sample 5 result was between the quantitative Sample 4 and 7 results. Comparison of MoNi (Figure 17) and MoNi II results show that the inconsistencies originate most likely from the instability of Zr in solution (HF needed in stabilisation) and interferences in detection.

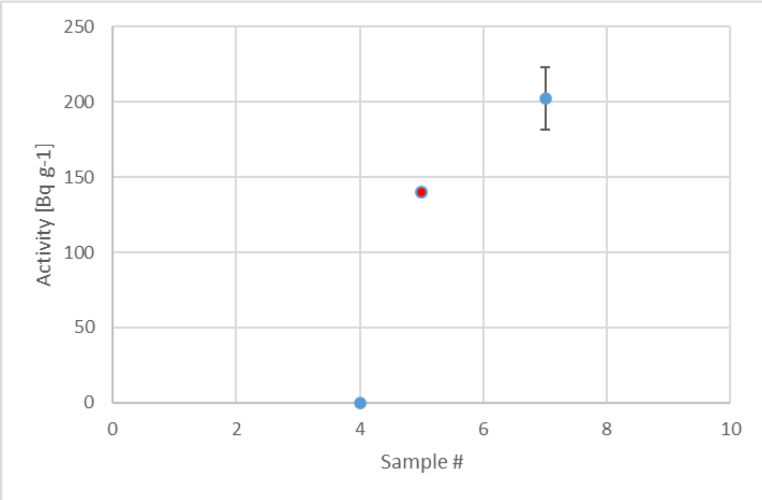


Figure 16. Final ^{93}Zr results in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Red dot indicates a result below LOQ.

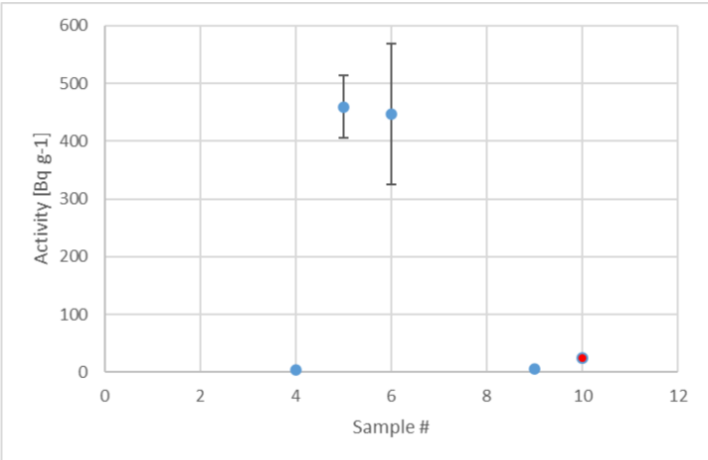
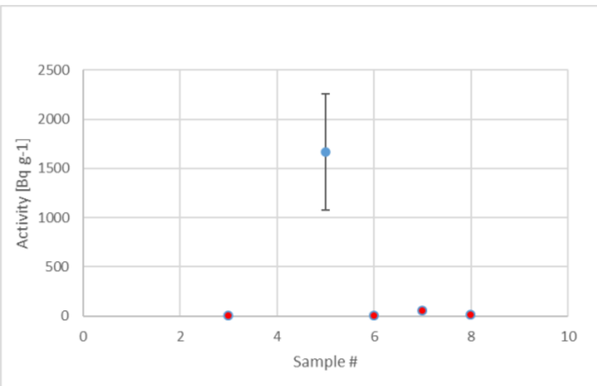


Figure 17. Final MoNi project ^{93}Zr results in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Red dot indicates a result below LOQ.

Statistical analysis of ^{36}Cl results was not carried out even though five data entries were submitted (Figure 18), because four data entries were reported to be below limit of detection and Sample 5 results most likely contained some contamination as an interference. The origin of the interference is not known. It can be concluded, however, that in case the sample contained ^{36}Cl, the LSC sensitivity was not low enough in this IE to determine ^{36}Cl.



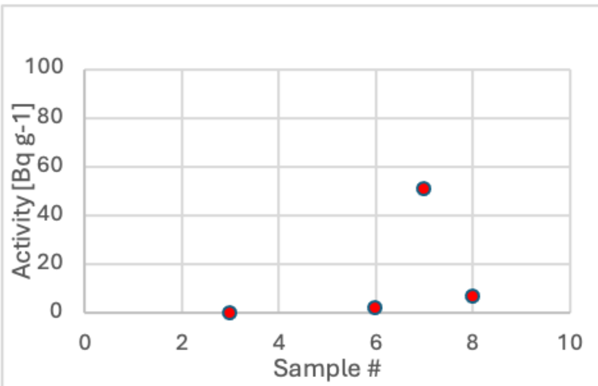


Figure 18. Final ^{36}Cl results in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Red dot indicates a result below LOQ.

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 131 ± 44 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 (21 %) since the robust standard deviation was large (34 %). As seen in Table 22, all ^{94}Nb z scores are in acceptable range. Comparison of MoNi and MoNi II results (Figure 20) show that the results are comparable.

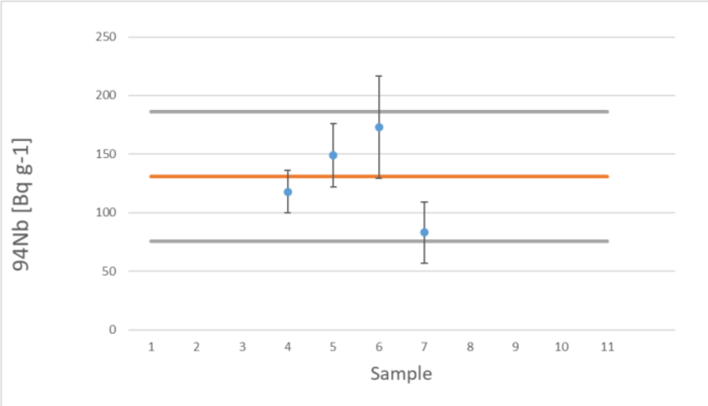


Figure 19. Final ^{94}Nb results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

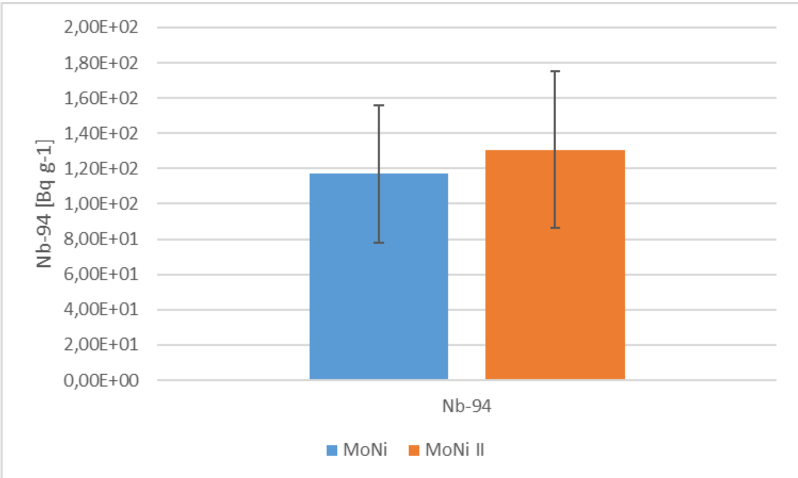


Figure 20. Comparison of final MoNi and MoNi II ^{94}Nb assigned values in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

Illustrative statistical analysis of ^{93m}Nb results were carried out with three data entries. The assigned value with the robust standard deviation ($k = 1$) was 1140 ± 236 Bq g^{-1}. The data entries with the assigned value and uncertainties ($k = 2$) are shown in Figure 21. The z score was calculated using the standard uncertainty of assigned value (15 %) since the robust standard deviation was large (21 %). As seen in Table 22, all ^{93m}Nb z scores are in acceptable range. Comparison of MoNi and MoNi II results (Figure 22) show that the results are comparable. The results are especially good considering the Nb stability in solution difficulties and ^{93m}Nb detection.

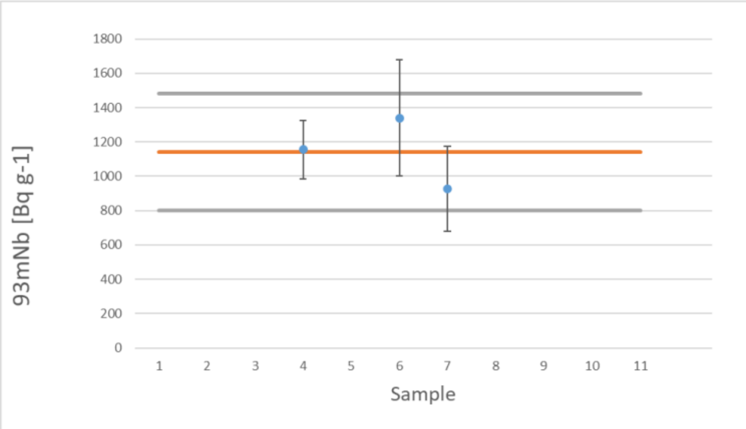


Figure 21. Final ^{93m}Nb results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of *k = 2*.

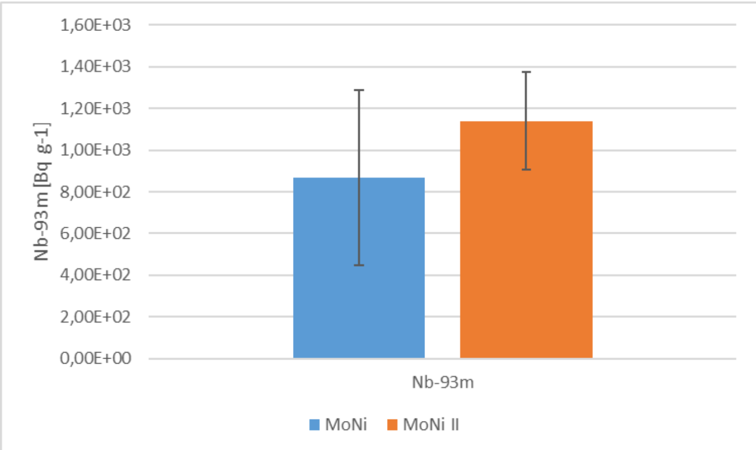


Figure 22. Comparison of final MoNi and MoNi II ^{93m}Nb assigned values in high activity steel with uncertainties stated with a coverage factor of *k = 1*.

Statistical analysis of ^{55}Fe results was not carried out as only 2 data entries were submitted (Figure 23). However, these results are comparable with each other and MoNi results (Figure 24).

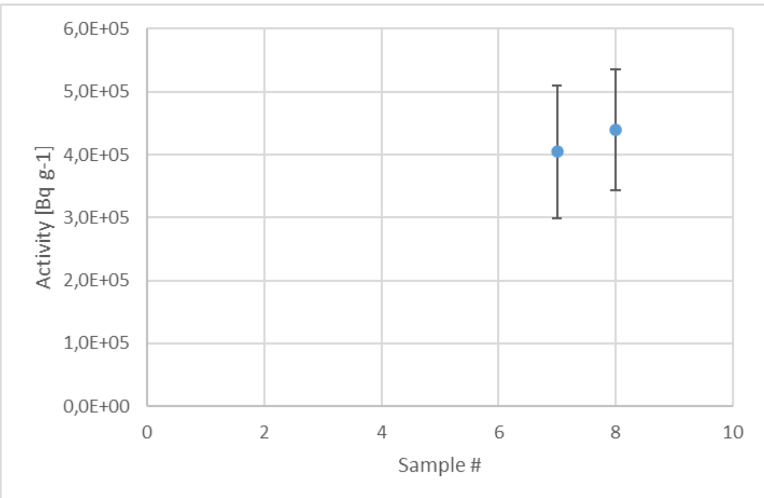


Figure 23. Final ^{55}Fe results in high activity steel with uncertainties stated with a coverage factor of *k = 2*.

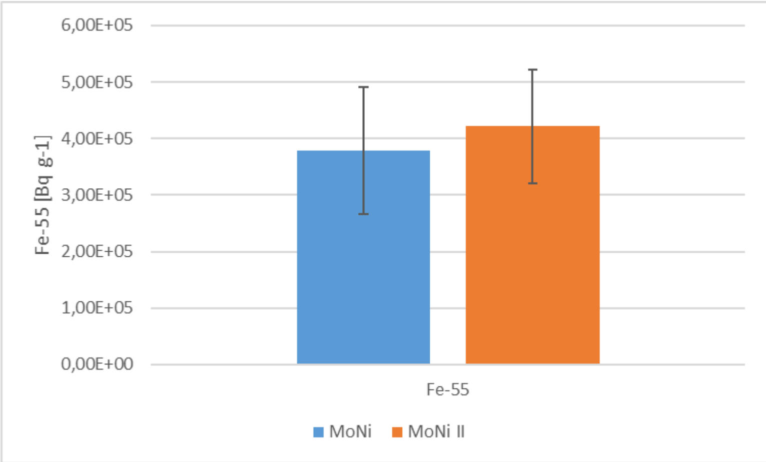


Figure 24. Comparison of final MoNi assigned values and MoNi II average ^{55}Fe results in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

Illustrative statistical analysis of ^{63}Ni results were carried out with three data entries. The assigned value with the robust standard deviation ($k = 1$) was 840 ± 120 kBq g^{-1}. The data entries with the assigned value and uncertainties ($k = 2$) are shown in Figure 25. The z score was calculated using robust standard deviation (14 %). As seen in Table 22, all ^{63}Ni z scores are in acceptable range. Comparison of MoNi and MoNi II results (Figure 26) show that the results are comparable.

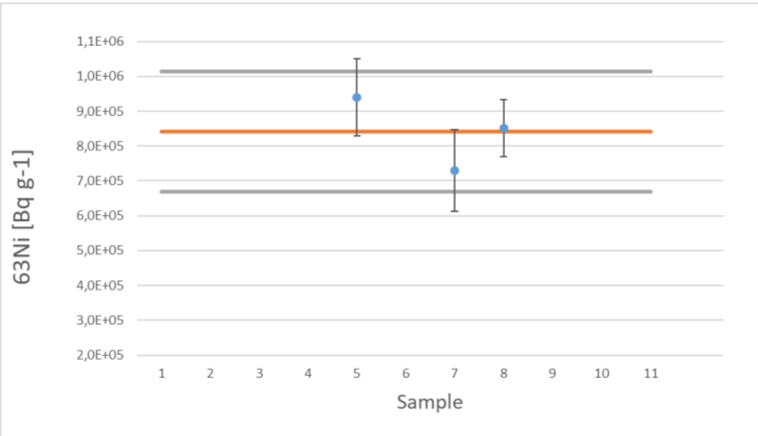


Figure 25. Final ^{63}Ni results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

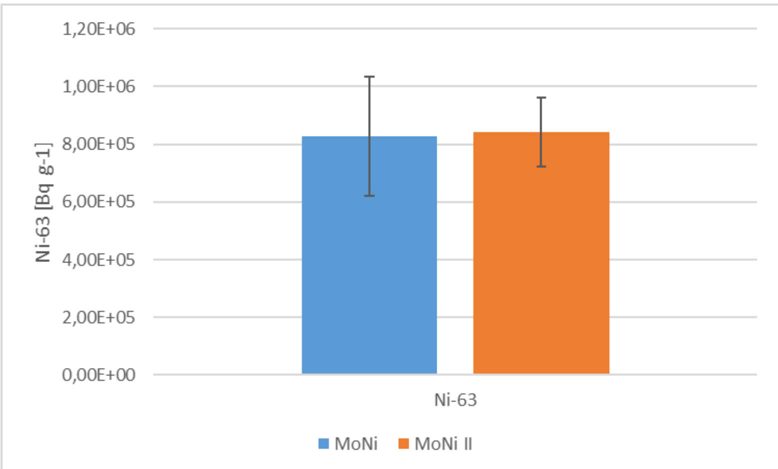


Figure 26. Comparison of final MoNi and MoNi II ^{63}Ni assigned values in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

Statistical analysis of ^{59}Ni results was not carried out, because only one data entry was reported (Figure 27). Comparison of the MoNi and MoNi II results (Figure 28) show that the results are good, especially considering the analysis difficulty level.

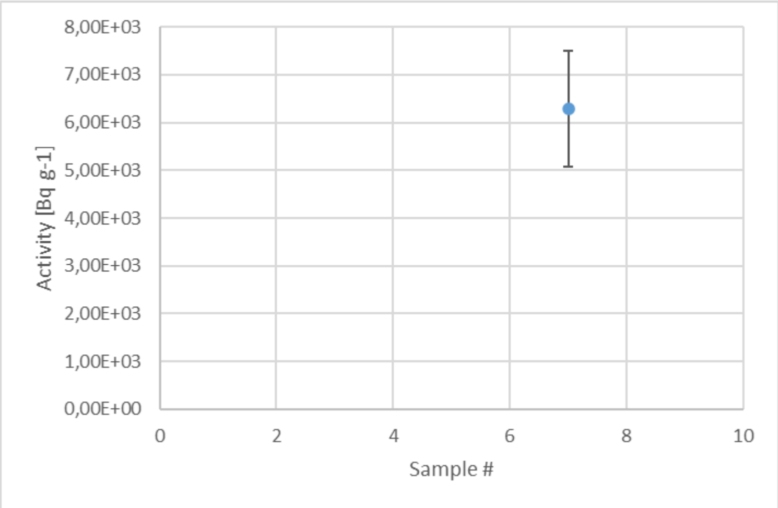


Figure 27. Final ^{59}Ni results in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

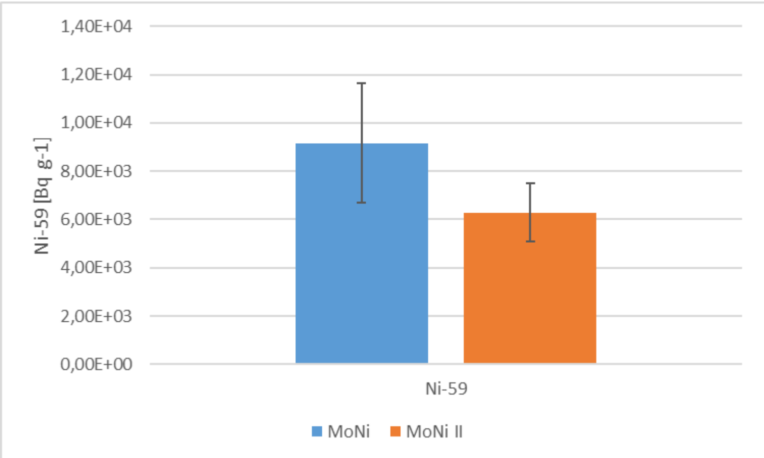


Figure 28. Comparison of final MoNi assigned value and MoNi II ^{59}Ni result in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

Statistical analysis of ^{3}H results was not carried out, because the only one data entry was below LOQ (Figure 30).

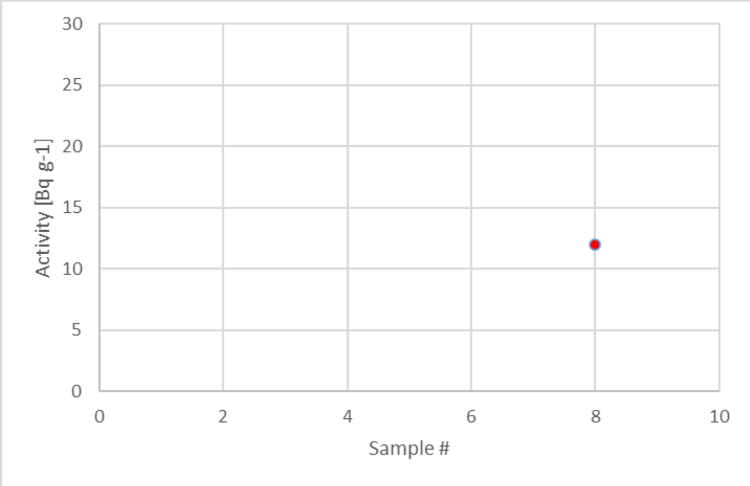


Figure 29. Final ^{3}H results in high activity steel with uncertainties stated with a coverage factor of $k = 2$. Red dot indicates a result below limit of quantification.

Table 22. DTM z scores in MoNi II 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			
	^{14}C (indicative)	^{94}Nb (indicative)	^{93m}Nb (illustrative)	^{63}Ni (illustrative)
1	0.1	-	-	-
2	-	-	-	-
3	0.6	-	-	-
4	-	0.5	0.1	-
5	1.2	0.7	-	0.8
6	-	1.5	1.2	-
7	0.7	1.7	1.3	0.9
8	-	-	-	0.1

7.3 Elemental analysis results

Statistical analysis of Fe results was carried out with five data entries. The assigned value with the robust standard deviation ($k = 1$) was 664598 ± 64687 mg kg^{-1}. The data entries with the assigned value and uncertainties ($k = 2$) are shown in Figure 30. The z score was calculated using the robust standard deviation (10 %). As seen in Table 23, all Fe z scores are in acceptable range. Comparison of the MoNi and MoNi II results (Figure 31) show that the results are comparable.

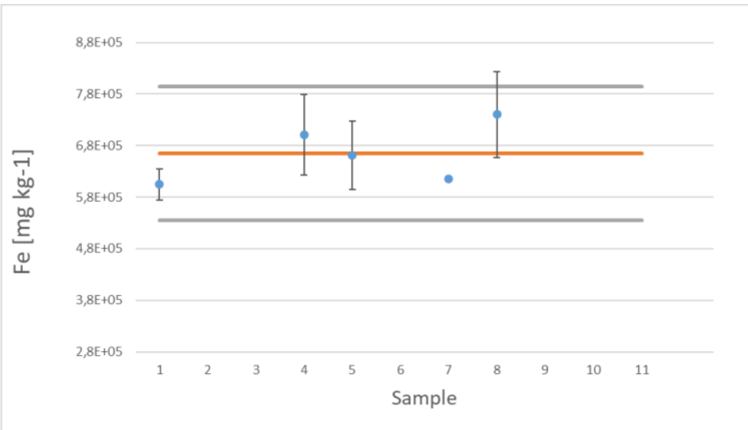


Figure 30. Final Fe results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

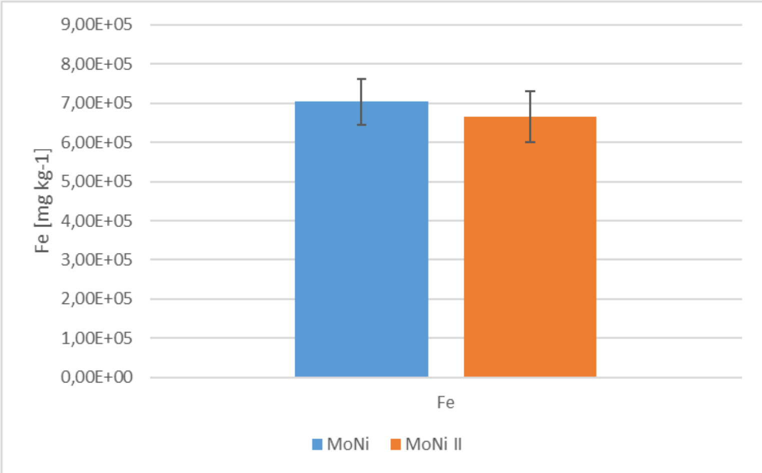


Figure 31. Comparison of final MoNi and MoNi II Fe assigned values in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

Statistical analysis of Ni results was carried out with five data entries. The assigned value with the robust standard deviation ($k = 1$) was 98408 ± 7723 mg kg^{-1}. The data entries with the assigned value and uncertainties ($k = 2$) are shown in Figure 32. The z score was calculated using the robust standard deviation (8 %). As seen in Table 23, all Ni z scores are in acceptable range. Comparison of the MoNi and MoNi II results (Figure 33) show that the results are comparable.

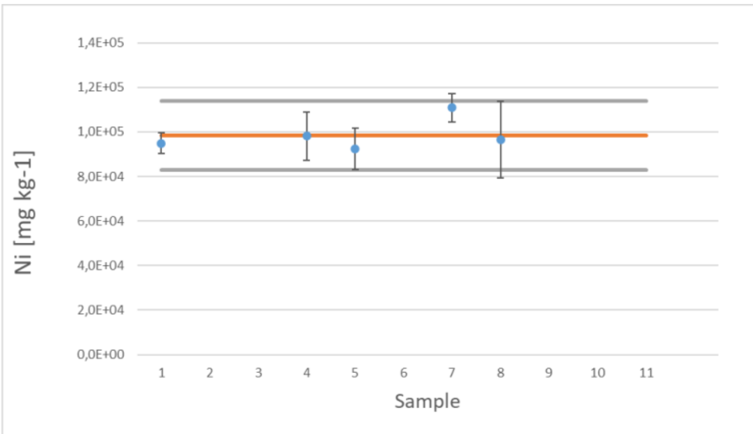


Figure 32. Final Ni results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

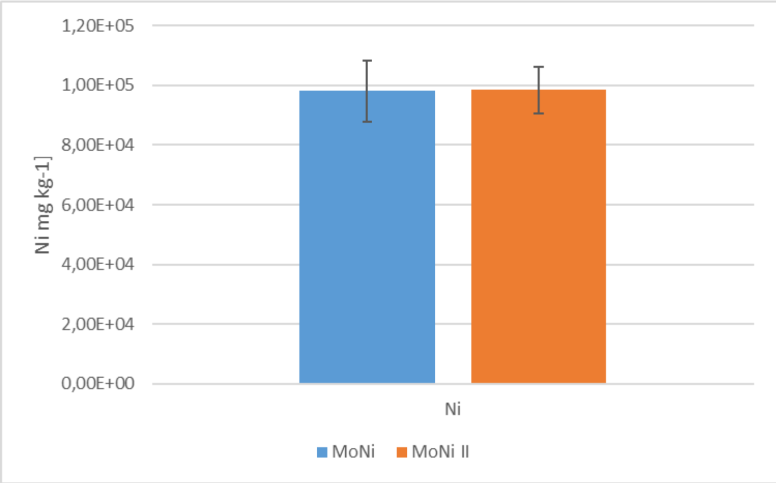


Figure 33. Comparison of final MoNi and MoNi II Ni assigned values in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

Indicative statistical analysis of Nb results was carried out with four data entries. The assigned value with the robust standard deviation ($k = 1$) was 2104 ± 1436 mg kg^{-1}. The data entries with the assigned value and uncertainties ($k = 2$) are shown in Figure 34. The z score was calculated using standard uncertainty of assigned value (43 %) as the robust standard deviation was large (68 %). As seen in Table 23, all Nb z scores are in acceptable range. However, the assessment is very indicative as there was a low number of data entries and large variation in the results as seen in the large robust standard deviation and standard uncertainty of assigned value. Comparison of MoNi and MoNi II results (Figure 35) show that the results are comparable even though the MoNi II result include a significantly large uncertainty (68 %).

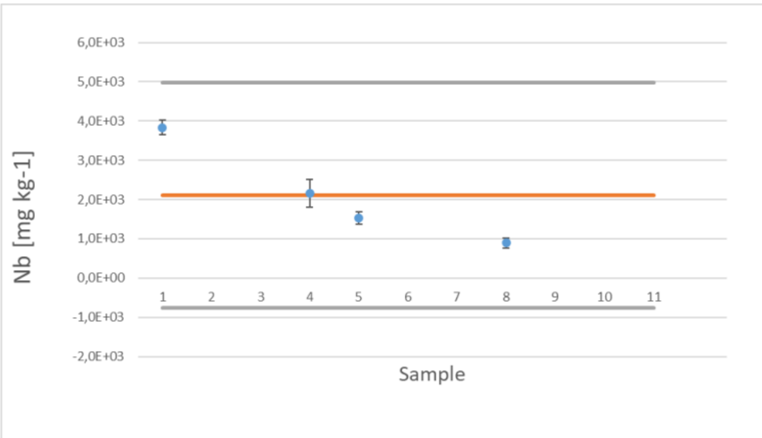


Figure 34. Final Nb results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

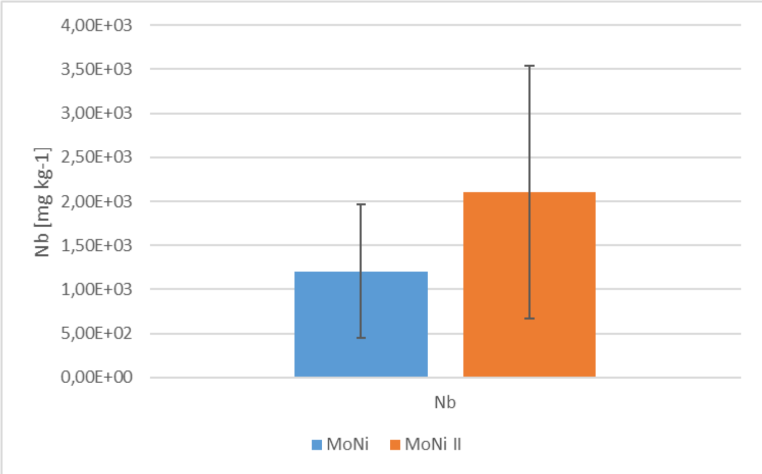


Figure 35. Comparison of final MoNi and MoNi II Nb assigned values in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

Indicative statistical analysis of Mo results was carried out with four data entries since one of the results (Sample 7) was an outlier. The assigned value with the robust standard deviation ($k = 1$) was 1845 ± 281 mg kg^{-1}. The data entries with the assigned value and uncertainties ($k = 2$) are shown in Figure 36. The z score was calculated using the robust standard deviation (15 %). As seen in Table 23, all Mo z scores are in acceptable range (Sample 7 excluded). Comparison of the MoNi and MoNi II results (Figure 37) show that the results are comparable.

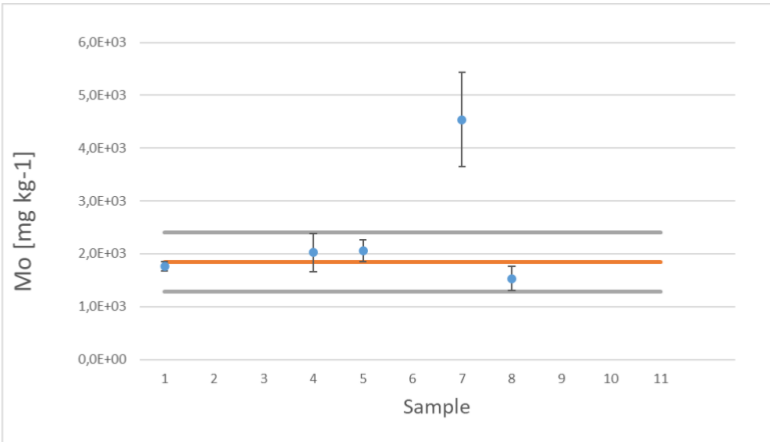


Figure 36. Final Mo results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

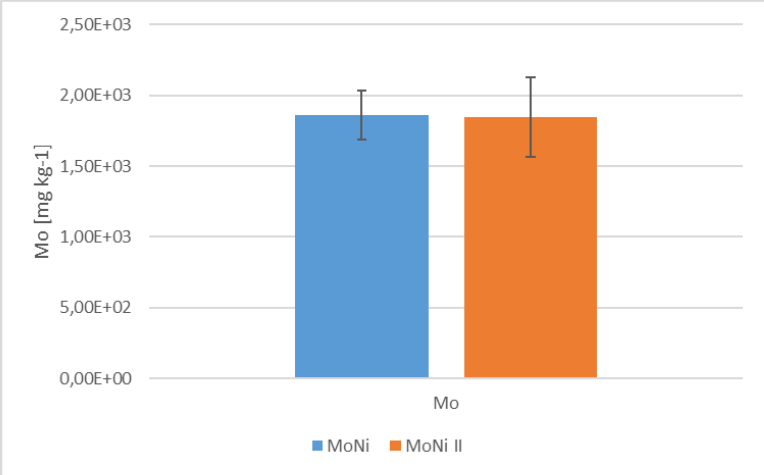


Figure 37. Comparison of final MoNi and MoNi II Mo assigned values in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

Indicative statistical analysis of Co results was carried out with four data entries. The assigned value with the robust standard deviation ($k = 1$) was 804 ± 35 mg kg^{-1}. The data entries with the assigned value and uncertainties ($k = 2$) are shown in Figure 38. The z score was calculated using the robust standard deviation (4 %). As seen in Table 23, all Co z scores are in acceptable range. Comparison of the MoNi and MoNi II results (Figure 39) show that the results are comparable.

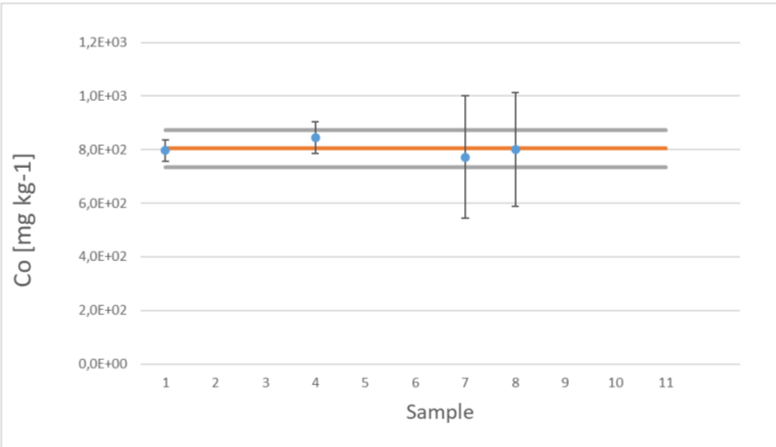


Figure 38. Final Co results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

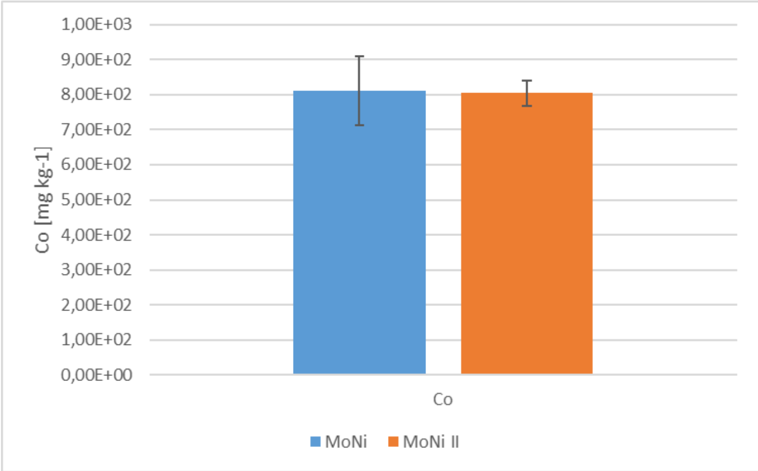


Figure 39. Comparison of final MoNi and MoNi II Co assigned values in high activity steel with uncertainties stated with a coverage factor of *k = 1*.

Statistical analysis was not carried out for Zr results since the four submitted results varied significantly (Figure 40) from 19-2742 mg kg^{-1}. As MoNi project included only one result (13 ± 1 mg kg^{-1}), it can be only concluded that Zr analysis is difficult. Additionally, the difference in ^{93}Zr results may originate also from the elemental analysis.

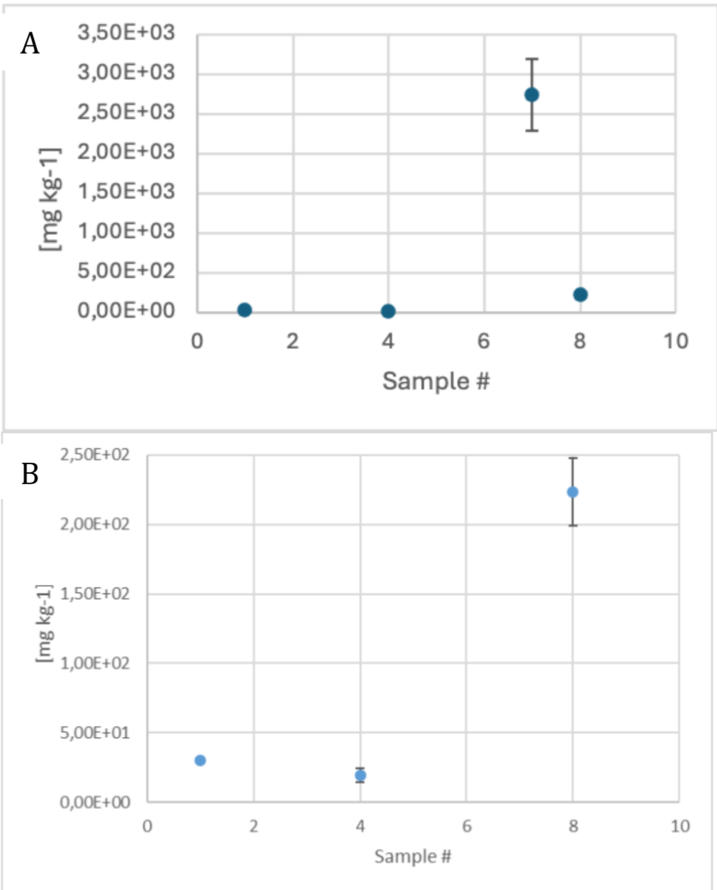


Figure 40. Final Zr results in high activity steel with uncertainties stated with a coverage factor of *k = 2*. All results (A) and Sample 7 excluded (B).

Statistical analysis of Mn results was carried out with five data entries. The assigned value with the robust standard deviation (*k = 1*) was 15058 ± 1433 mg kg^{-1}. The data entries with the assigned value and uncertainties (*k = 2*) are shown in Figure 41. The z score was calculated using the robust standard deviation (4 %). As seen in Table 23, all Mn z scores except Sample 7 (unacceptable) are in acceptable range. Comparison of the MoNi (average) and MoNi II results (Figure 42) show that the results are comparable.

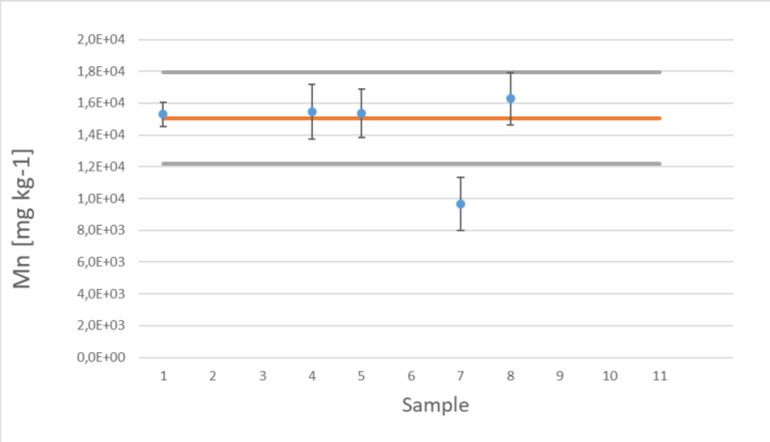


Figure 41. Final Mn results and assigned value (orange line) in high activity steel with uncertainties stated with a coverage factor of $k = 2$.

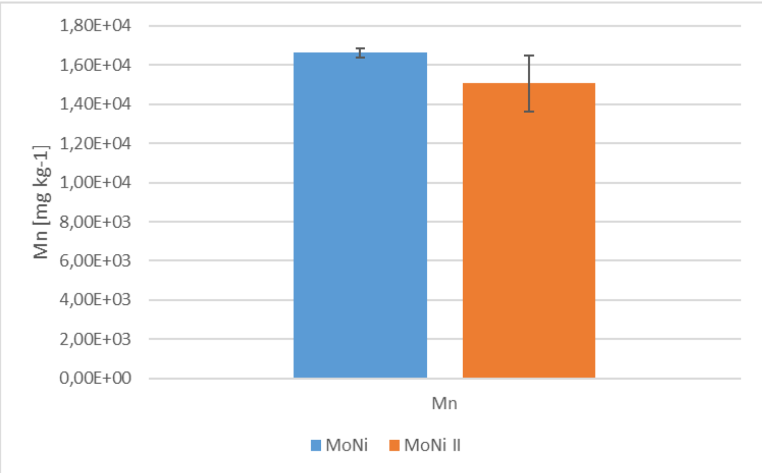


Figure 42. Comparison of final MoNi average and MoNi II Mn assigned value in high activity steel with uncertainties stated with a coverage factor of $k = 1$.

Statistical analysis of Li results was not carried out as only two quantitative results were submitted whereas two data entries were below LOQ (Figure 43). Sample 7 data entry is between the quantitative results (Sample 1 and 8) whereas Sample 4 data entry is correctly above the quantitative results. Li is a challenging element to analyse and even though the quantitative results differ, they are in the same order of magnitude.

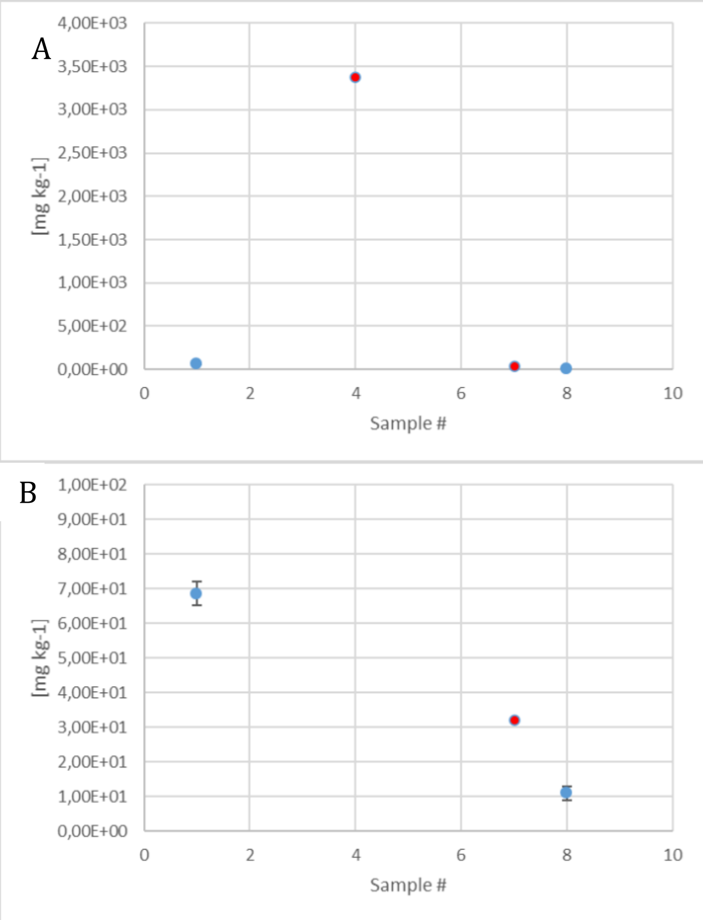


Figure 43. Final Li results in high activity steel with uncertainties stated with a coverage factor of *k = 2*. All results (A) and Sample 4 excluded (B). Red dot indicates a results below limit of quantification.

Elemental analysis results with only one data entry above limit of quantification or data entries only below LOQ are summarized in Figure 44.

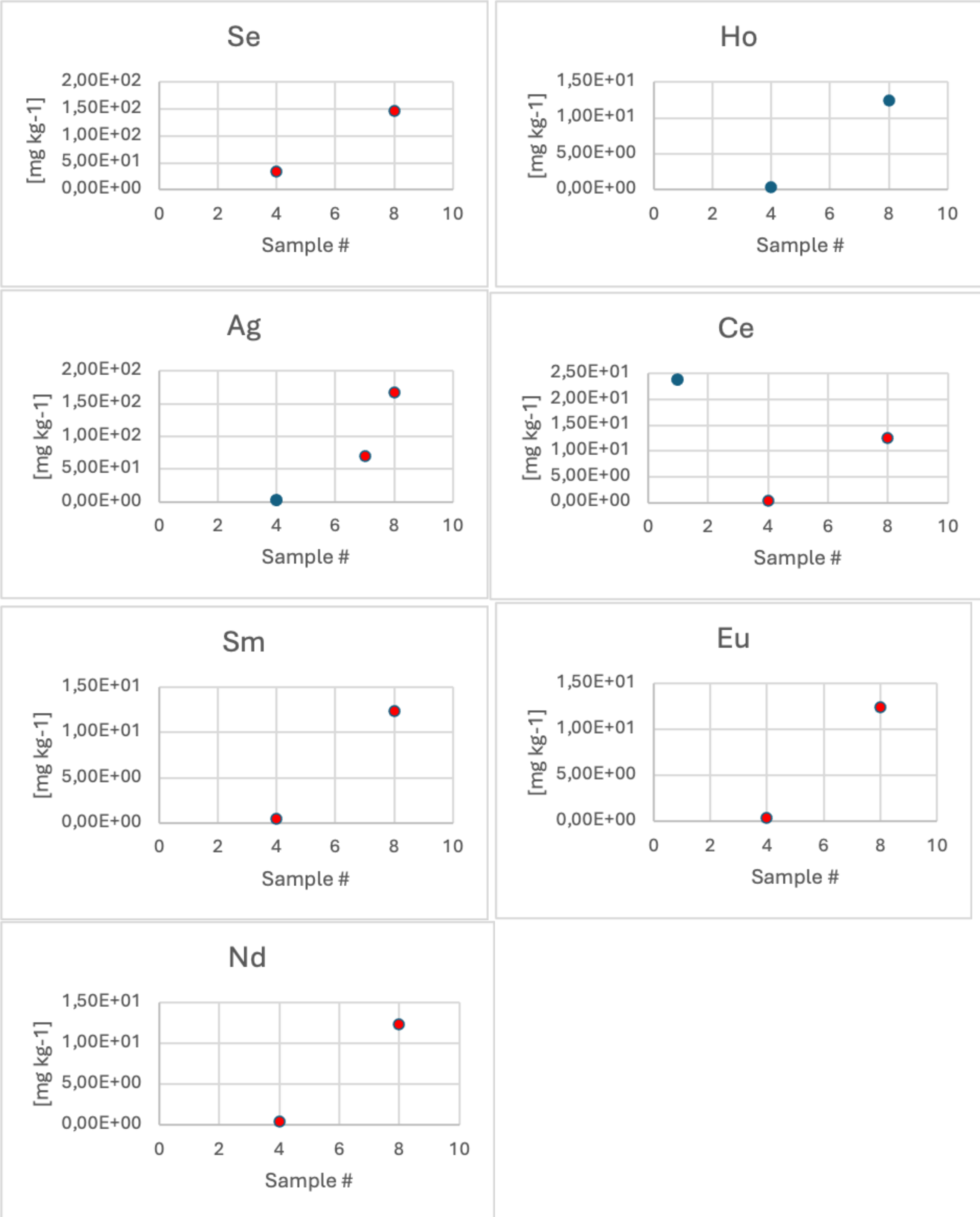


Figure 44. Se, Ho, Ag, Ce, Sm, Eu, and Nd elemental analysis results in high activity steel with uncertainties stated with a coverage factor of $k = 1$. Red dot indicates a result below limit of quantification.

Table 23. Elemental analysis z scores in MoNi II 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**	**Nb (indicative)**	**Mo (indicative)**	**Co (indicative)**	**Mn**
1	0.9	0.5	1.9	0.3	0.2	0.2
2	-	-	-	-	-	-
3	-	-	-	-	-	-
4	0.6	0.0	0.1	0.6	1.2	0.3
5	0.1	0.8	0.6	0.8	-	0.2
6	-	-	-	-	-	.
7	0.8	1.6	-	-	0.9	3.8
8	1.2	0.2	1.4	1.1	0.1	0.9

8. Conclusions

MoNi II was a follow-up project for MoNi, in which elemental, ETM and DTM analyses were carried out in homogenised high activity steel samples. MoNi II, on the other hand, studied un-homogenised steel samples, for which volatile DTM analyses were carried out. Additionally, studies were carried out for homogeneity. The main analyses of interest were:

- Cobalt-60 was selected as a main radionuclide of interest in order to study homogeneity of the material.
- Volatile DTM ^{14}C was selected as a main radionuclide of interest as there were enough interest and expertise to analyse it.
- Non-volatile ^{93}Mo and ^{93}Zr were selected to be of main interest based on the MoNi results.
- The main elements of interest were Fe, Ni, Nb, Mo, Co, and Zr.

The ETM analyses were carried out in the original sample vial, solid aliquot, and liquid aliquot. The results showed that analysis results in original sample vial were not as well aligned as in aliquot measurements. The best alignment (i.e., lowest robust standard deviation 5 %) were seen for liquid sample measurements. This shows that the capabilities to analyse ETMs in liquid samples are most advanced. The efficiency calibration modelling for solid samples is not so easy due to the vial and sample geometry. Additionally, signs of heterogeneity of the sample pieces could not be detected. The z scores (Figure 45) were all 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, and liquid-liquid extractions. The DTM detection included a variety of detection techniques, namely LSC, mass spectrometry, gamma spectrometry, and X-ray spectrometry. The ^{14}C results showed that the sample results, which were analysed using a pyrolyser, were below LOQ whereas all the other methodologies resulted in quantitative results. One set of results was an outlier resulting in significantly higher activity results compared to the other quantitative results. The indicative statistical analysis of ^{14}C results showed significant scattering (i.e., robust standard deviation 77 %) and therefore, further studies are needed for ^{14}C analyses. The ^{94}Nb and ^{93m}Nb results showed good results even though the robust standard deviations were large (i.e., 34 % and 21 %), which can be explained by considering the Nb stability difficulties in solution. Additionally, the ^{94}Nb and ^{93m}Nb results were comparable with MoNi results. Illustrative statistical analysis of ^{63}Ni results were well aligned (i.e., robust standard deviation 14 %) and comparable with MoNi results. A summary of statistically analysed z scores show (Figure 45) that all DTM analyses were in the acceptable range. However, some outliers were not included in the summary figure.

The ^{93}Mo results showed that all but one result was below LOQ or the result included an interference. Comparison of the quantitative results with MoNi results showed that the reference ^{93}Mo activity is most likely somewhere around 10-20 Bq g^{-1}. The ^{93}Zr results showed that the ^{93}Zr analysis remains difficult as inconsistent results were obtained both in MoNi and MoNi II. The ^{36}Cl results showed that the utilised analysis and detection techniques could not quantitatively detect it. The ^{55}Fe and ^{59}Ni results were consistent with MoNi results even though two and only one, respectively, data entries were submitted. The only submitted ^{3}H result was below LOQ.

Elemental analyses were carried out using ICP-OES and ICP-MS techniques. The statistical analysis of the submitted results showed that all Fe, Ni, Nb, Mo, and Co results were in acceptable range (Figure 46). One Mn result was in unacceptable range. The assigned values were comparable with MoNi assigned values. However, Nb results showed significant scattering most likely due to instability of Nb in solution. Zr and Li results also suffered from inconsistent results. Below LOQ or only one data entry were reported for Se, Ho, Ag, Ce, Sm, Eu, and Nd.

As a conclusion, the MoNi II project studied a wide range of radionuclide and elemental analyses. The results show that further studies are needed for ^{14}C, Nb isotopes (stable and unstable), ^{93}Mo, Zr isotopes (stable and unstable), and ^{36}Cl analyses. 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.

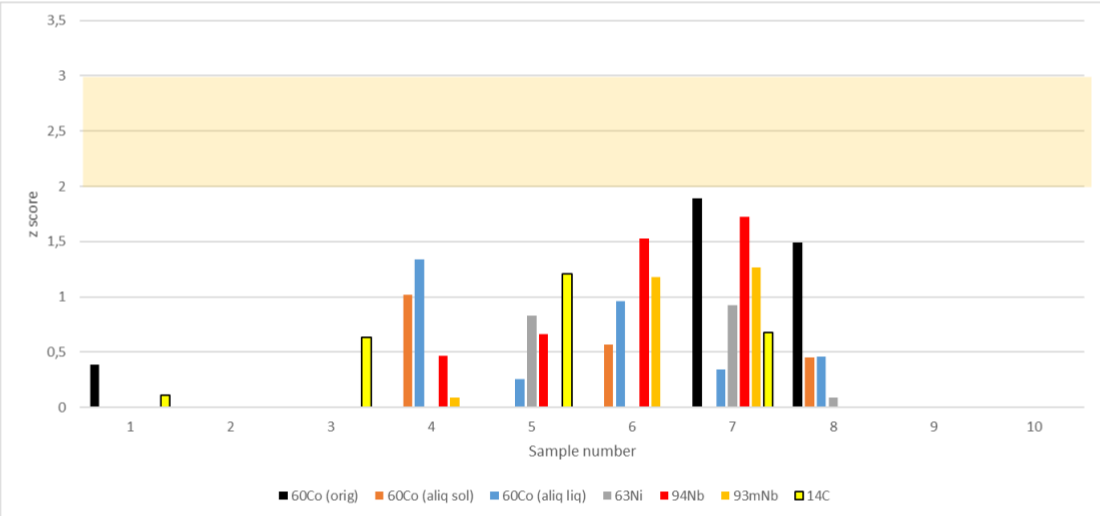


Figure 45. Summary of ETM and DTM z scores in MoNi II. Results are acceptable when z≤2.0, warning signal is given when 2.0<z<3.0, and unacceptable when z≥3.0.

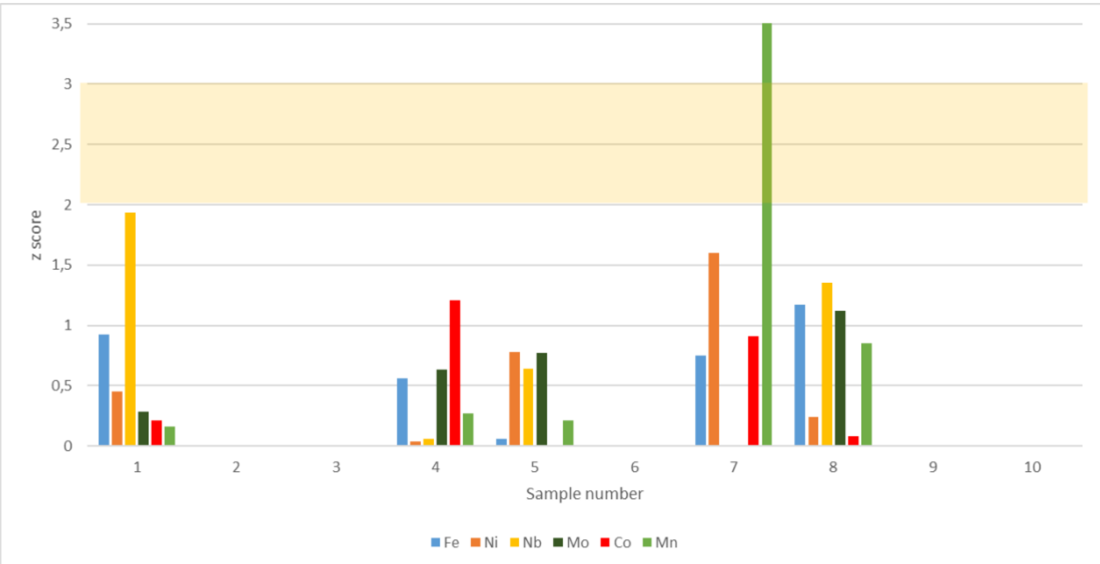


Figure 46. Summary of elemental analysis z scores in MoNi II. Results are acceptable when z≤2.0, warning signal is given when 2.0<z<3.0, and unacceptable when z≥3.0.

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Abstract max. 2000 characters	An intercomparison exercise was carried out for volatile Difficult-To-Measure (DTM) radionuclides in high activity steel samples. The exercise was a follow up project for MoNi intercomparison exercise. 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. Additionally, comparison with MoNi project results

are shown.

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