

Article

Determination of Trace ^{55}Fe and ^{63}Ni in Steel Samples via Liquid Scintillation Counting

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Abstract

In the decommissioning of nuclear facilities, activated steel often contains radionuclides such as ^{55}Fe and ^{63}Ni , which are categorized as hard-to-measure due to their emission of only low-energy beta particles or X-rays. In samples exhibiting very low radioactivity, close to background levels, a large quantity of steel must undergo extensive physical and chemical processing to achieve the Minimum Detectable Activity Concentration (MDC) necessary for clearance, recycling, or reuse. Italian regulations set particularly stringent clearance levels for these radionuclides (1 Bq/g for both ^{55}Fe and ^{63}Ni), significantly lower than those specified in the EU Directive 2013/59 (1000 Bq/g for ^{55}Fe and 100 Bq/g for ^{63}Ni). Additionally, Italian authorities may enforce even stricter limits depending on specific circumstances. The analytical challenge is compounded by the presence of large amounts of non-radioactive Fe and Ni, which can cause color quenching, further extending analysis times. This study presents a reliable and optimized method for the quantitative determination of ^{55}Fe and ^{63}Ni in steel samples with activity levels approaching regulatory thresholds. The methodology was specifically developed and applied to steel from the Frascati Tokamak Upgrade (FTU) facility, under decommissioning by ENEA. The optimization process demonstrated that achieving the required MDCs necessitates acquisition times of approximately 5 days for ^{55}Fe and 6 h for ^{63}Ni , ensuring compliance with stringent regulatory requirements and supporting efficient laboratory workflows.



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1. Introduction

The ENEA Research Center in Frascati housed the Frascati Tokamak Upgrade (FTU), a high-magnetic-field tokamak dedicated to research on radio-frequency plasma heating [1,2]. This facility is currently undergoing decommissioning to make space for the new Divertor Tokamak Test (DTT), which will investigate alternative divertor configurations relevant to DEMO, the demonstration fusion power plant.

As part of the Radiological Characterization Plan, analyses of steel components have been conducted to identify and quantify radionuclides resulting from neutron activation processes. Gamma-emitting radionuclides are readily identified using conventional gamma spectrometry. In contrast, beta-emitting radionuclides such as ^{55}Fe and ^{63}Ni require complete separation from the sample matrix and other radionuclides due to over-lapping beta energy spectra, which complicate direct measurement.

Both ^{55}Fe and ^{63}Ni are products of neutron activation:

^{55}Fe ($t_{1/2} = 2.7$ years) is primarily formed through the reactions $^{54}\text{Fe}(n,\gamma)^{55}\text{Fe}$ and $^{56}\text{Fe}(n,2n)^{55}\text{Fe}$. It decays via electron capture to stable ^{55}Mn , emitting Auger electrons and low-energy X-rays (notably 5.89 keV, with 16.9% intensity). Due to the low energy of these emissions, detection using low-energy gamma/X-ray detectors or gas flow proportional counters is possible, but these methods typically yield low counting efficiencies (<1%) compared to gamma spectroscopy [3].

^{63}Ni is a pure beta emitter with a maximum beta energy of 66.95 keV and a long half-life of 100.1 years. It is generated via $^{62}\text{Ni}(n,\gamma)^{63}\text{Ni}$ and $^{63}\text{Cu}(n,p)^{63}\text{Ni}$ reactions. In steels, the presence of Ni in significant concentrations results in a corresponding presence of ^{63}Ni . This radionuclide is crucial for clearance and waste acceptance due to its prevalence in reactor components. Trace amounts of Ni and Cu, and thus ^{63}Ni , may also be found in other reactor materials such as graphite, concrete, lead, and aluminum alloys [4,5].

The measurement of ^{63}Ni is also challenging due to its low beta energy, with detection methods such as windowless gas flow proportional counters or ion-implanted silicon detectors yielding limited efficiencies (ranging from 2.6% to 20%) [6].

While Inductively Coupled Plasma Mass Spectrometry (ICP-MS) has been considered for radionuclide determination, it is not suitable in this context for either ^{55}Fe or ^{63}Ni . Their high specific activities (8.7×10^{13} Bq/g for ^{55}Fe and 2.1×10^{12} Bq/g for ^{63}Ni) result in detection limits well above those required for clearance. Moreover, the presence of the decay product ^{55}Mn significantly interferes with ^{55}Fe analysis, further limiting the applicability of mass spectrometry.

Liquid Scintillation Counting (LSC) is therefore the preferred technique due to its high counting efficiency—up to 95% for low-energy beta emitters. Accurate LSC measurement of ^{55}Fe and ^{63}Ni requires prior mineralization of the sample using strong acid mixtures, followed by chemical separation steps. Several separation techniques have been documented in the literature, including chelating agents, anion exchange chromatography, solvent extraction, hydroxide precipitation, and, for Ni, complexation with dimethylglyoxime (DMG), followed by precipitation or organic extraction of the Ni-DMG complex [7,8]. Chelation chromatography and hydroxide precipitation can be effective but often require combination with additional methods for adequate separation [6].

In the case of FTU steel samples, the activity due to neutron activation is extremely low (e.g., <2 mBq/g of ^{55}Fe in blanket components) [3]. As a result, substantial quantities of steel must be processed to reach the Minimum Detectable Activity Concentration (MDC) required for clearance. This generates highly colored solutions that still contain both radioactive and non-radioactive Ni and Fe in appreciable amounts. Color quenching, a known issue in LSC, reduces measurement sensitivity by impairing the optical transmission of scintillation light to the detector [9,10]. While reducing sample volume can help minimize quenching, it simultaneously increases acquisition time, which is a concern when aiming for low MDCs that are compliant with clearance regulations.

According to the Italian Legislative Decree 101/2020, clearance levels for ^{55}Fe and ^{63}Ni are set at 1 Bq/g, significantly more stringent than those established in the EU Directive 2013/59 (1000 Bq/g for ^{55}Fe and 100 Bq/g for ^{63}Ni). Furthermore, the Italian regulatory authority may impose stricter requirements: in the FTU case, to approve a material for recycling or reuse the measured activity must not only fall below clearance levels, but the MDC must also be $\leq 30\%$ of the clearance level, corresponding to 0.3 Bq/g.

The decommissioning of nuclear fusion facilities remains relatively rare today, but this will likely change as more research installations reach the end of their operational lifespans. The resulting activated steel will require precise and efficient characterization. This paper presents a robust and optimized methodology for the quantitative determination of ^{55}Fe and ^{63}Ni in low-activity steel samples, capable of meeting regulatory MDCs down to

fractions of a Bq/g. Although developed for FTU decommissioning, the method holds potential applicability for other facilities operating under similar neutron flux conditions.

2. Materials and Methods

2.1. Chemicals and Equipment

All reagents used in this study were of analytical grade. The following chemicals were supplied by Sigma-Aldrich: Hydrochloric acid (HCl), Phosphoric acid (H₃PO₄), Nitric acid (HNO₃), Phosphonic acid (H₃PO₃), and Hydrofluoric acid (HF). Ion exchange resins were obtained from DOWEX: AG 1x8 (200–400 mesh, anion exchange) and AG 50Wx8 (200–400 mesh, cation exchange).

Radioactive standard solutions of ⁵⁵Fe and ⁶³Ni were provided by Eckert & Ziegler, Berlin, Germany.

LSC was performed using a Hidex 300 SL triple-to-double coincidence ratio (TDCR) system (Hidex, Turku, Finland). This system is equipped with three photomultiplier tubes, providing significantly higher detection efficiency than conventional LSC systems [11]. The scintillation cocktail used for all measurements was Ultima Gold AB (PerkinElmer, Shelton, CT, USA), specifically designed for alpha–beta discrimination, with an excellent sample holding capacity, making it ideal for a variety of acid sample types.

Sample mineralization was performed using a Microwave Digestion System (Milestone Ethos One, Sorisole, Italy), equipped with sealed TFM vessels. This system is designed to reduce the risk of acid fume exposure to both the operator and the environment. The HPR-ME-02 protocol [12] was followed, with the conditions detailed in Table 1.

Table 1. Microwave program from application notes HPR-ME-02.

Step	Time (min)	T1 (°C)	T2 (°C)	P (bar)	Power (W)
1	20	220	120	45	1500
2	15	220	120	45	1500

Each steel sample was digested in a mixture of 4 mL of concentrated HNO₃, 4 mL of concentrated HCl, and 2 mL of concentrated HF.

2.2. Methods

The objective of this method was to establish a robust and reproducible protocol for the quantitative determination of trace levels of ⁵⁵Fe and ⁶³Ni in steel samples via LSC. A preliminary validation study was conducted using spiked, non-irradiated AISI 316L steel to evaluate chemical yield, overall detection efficiency, and the resulting MDC.

This methodology was then applied to four steel samples (0.4–0.5 g each) from the decommissioned ENEA FTU (Frascati Tokamak Upgrade) facility. These samples were potentially activated due to neutron irradiation during the facility's operation. Based on theoretical activation modeling, the expected activity concentrations were below the clearance MDC of 0.3 Bq/g established by the Italian regulatory authorities. The radiological characterization aimed to confirm these theoretical estimations.

As LSC measurements are highly sensitive to color quenching, which affects the photon transmission from scintillation events to the photomultiplier, steel samples must undergo a rigorous chemical separation prior to counting [13,14]. Following microwave digestion, the resulting solutions were intensely colored due to high concentrations of non-radioactive Fe and Ni isotopes, making them unsuitable for direct measurement (Figure 1).

To purify the target radionuclides, the digested solution was evaporated to dryness and redissolved in 6 mL of 6 M HCl. The solution was passed through a column containing 10 mL of AG 1x8—200–400 mesh (anion exchange resin), previously pre-cleaned with 10 mL

of 3 M HNO_3 , rinsed with 15 mL of H_2O , and conditioned with 15 mL of 6 M HCl . Fe was retained and then eluted with 0.5 M HCl . The Ni-containing fraction was collected for further purification. Ni purification was carried out on a second column packed with AG 50Wx8—200–400 mesh (cation exchange resin), pre-conditioned with 15 mL of 6 M HCl , rinsed with 32 mL of H_2O , and eluted with 15 mL of 1.8 M HCl (Figure 2).

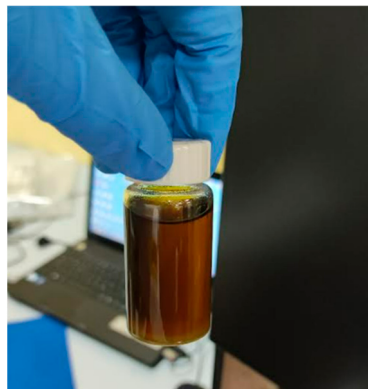


Figure 1. Visual evidence of color quenching in mineralized steel solutions.

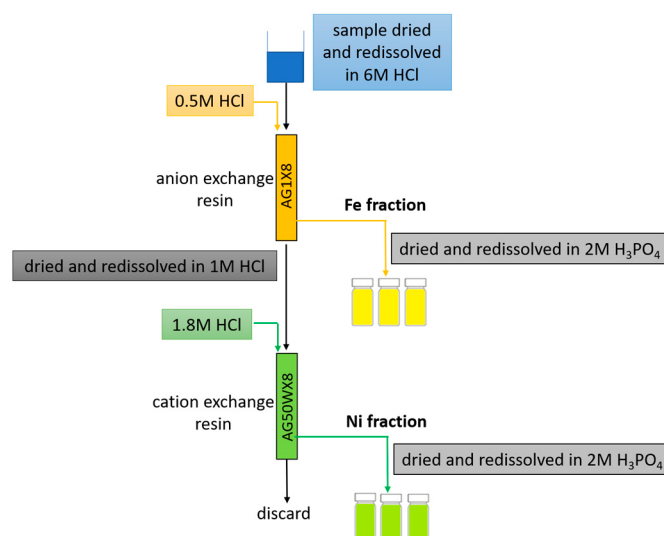
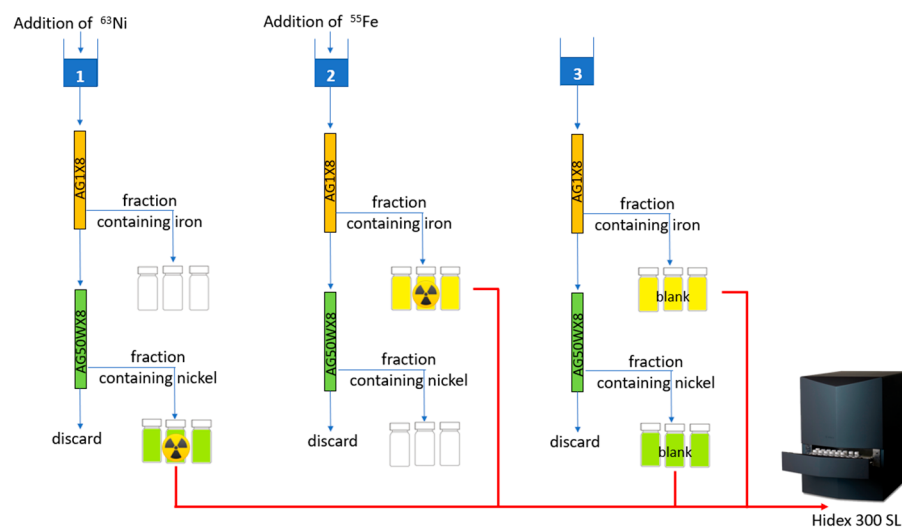


Figure 2. Flow diagram of Fe and Ni isolation procedure.

To assess overall efficiency, three non-irradiated AISI 316L samples were processed: two were respectively spiked with ^{55}Fe (20 ± 1 Bq) and ^{63}Ni (70 ± 1 Bq), and the third served as a blank for background subtraction (sample weights are listed in Table 2). The blank sample contained all the components of a regular sample except for the radioactive material being measured. It served to subtract background noise or contributions from other sources that were not attributable to the analyte of interest (e.g., solvent and/or resin residues). The overall workflow applied to the three samples is shown in Figure 3. The radioactive standard for ^{55}Fe is a calibrated solution of 0.1 M HNO_3 containing 100 micrograms of iron per mL, while for ^{63}Ni it is a 0.1 M HCl solution containing 100 micrograms of nickel per mL. The volume of calibrated solution added to the vial to prepare the spiked samples corresponds to only fractions of a microgram of iron and nickel. The impact of these small amounts on the overall quench was considered negligible compared to the quenching effect caused by the iron and nickel already present as steel components, which were present in quantities more than four orders of magnitude higher.

Table 2. Sample weights for optimization study.

Sample	Weight (g)
1	0.5070 ± 0.0200
2	0.5006 ± 0.0300
3	0.5070 ± 0.0180

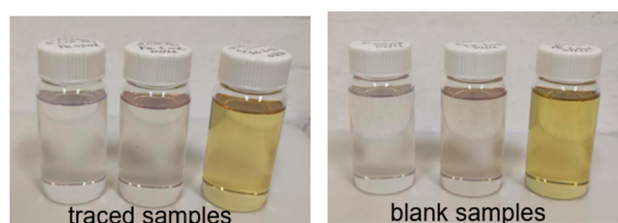
**Figure 3.** Workflow of the optimization process.

Each Fe and Ni fraction was dried and redissolved in 4 mL of 2 M H_3PO_4 , which was found to significantly reduce color quenching [15].

As previously mentioned, the solutions eluted from the chromatographic columns remain visibly colored. Therefore, it is essential to evaluate the impact of color quenching on the LSC measurements. This effect varies significantly depending on the acid used to dissolve the samples and has direct consequences on both detection efficiency and acquisition time—two critical parameters, particularly when analyzing numerous low-activity samples.

The chemical interaction of various acids with Fe and Ni—at concentrations representative of their presence in AISI 316L steel ($\text{Fe} = 69.06\% w/w$, $\text{Ni} = 9.25\% w/w$)—has been evaluated in previous studies [15]. These investigations demonstrated that phosphoric acid (H_3PO_4) and phosphorous acid (H_3PO_3) were the most effective choices for minimizing color quenching effects associated with both Fe and Ni. The use of these acids enables the attainment of the required Minimum Detectable Concentration (MDC) of 0.3 Bq/g within a reasonable measurement time, thereby making the analytical process more efficient and reliable.

LSC samples were prepared in triplicate for each radionuclide using aliquots of 0.5 mL, 1.0 mL, and 2.0 mL of the final solution. Each was brought to a volume of 4 mL with deionized water and mixed with 16 mL of Ultima Gold AB (Figures 4 and 5).

**Figure 4.** LSC vials showing Fe fractions at increasing concentrations.

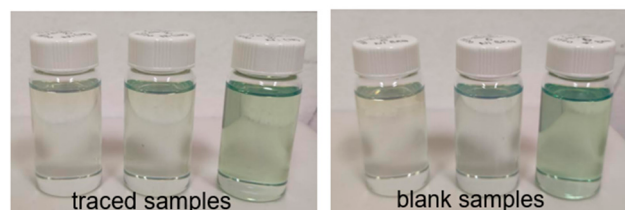


Figure 5. LSC vials showing Ni fractions at increasing concentrations.

For each radionuclide, the overall efficiency was calculated as:

$$\varepsilon = \frac{R - R_b}{A}$$

where

- R: sum of double coincidences (light photons detected in coincidence by two photo-multipliers) per unit of time in the appropriate range of spectrum channels for the traced sample.
- R_b : sum of double coincidences per unit of time in the appropriate range of spectrum channels for the blank sample.
- A: known activity in the vial (Bq).

Since activity A is added before performing the chemical procedure, the overall efficiency considers both the chemical yield of the process and the LSC efficiency.

Once the efficiency had been calculated, to determine the acquisition time (t) required to reach a target MDC, the following equation was used [16]:

$$\text{MDC} \left[\frac{\text{Bq}}{\text{g}} \right] = \frac{2.71 + 4.65 \cdot \sqrt{R_b \cdot t}}{\varepsilon \cdot m \cdot t}$$

where m is the mass of steel in the vial (g) and t is the acquisition time (s).

The optimum sample concentration corresponding to the minimum acquisition time was evaluated and chosen for ^{55}Fe and ^{63}Ni quantifications (Tables 3 and 4).

Table 3. Efficiency and MDC evaluation for ^{55}Fe .

Vial	Sample Mass (g)	ε (%)	Acq. Time to Reach MDC (Days)
#1	0.063 ± 0.002	28.3 ± 2.0	12
#2	0.125 ± 0.002	21.2 ± 1.0	5
#3	0.250 ± 0.004	2.0 ± 0.1	157

Table 4. Efficiency and MDC evaluation for ^{63}Ni .

Vial	Sample Mass (g)	ε (%)	Acq. Time to Reach MDC (Hours)
#1	0.063 ± 0.002	63.0 ± 4.0	59
#2	0.127 ± 0.002	54.0 ± 3.0	19
#3	0.254 ± 0.004	47.0 ± 3.0	6

To achieve the required MDC (0.3 Bq/g), a sample mass of 0.125 g of steel (corresponding to the 1 mL aliquot, vial #2) is the best compromise for ^{55}Fe quantification. In this case, an acquisition time of about 5 days is needed. The optimal result for ^{63}Ni is obtained with 0.25 g of steel (corresponding to the 2 mL aliquot, vial #3), requiring only about 6 h of measurement.

The trends shown in Tables 3 and 4 reveal the different relationships between sample mass, detection efficiency, and acquisition time for ^{55}Fe and ^{63}Ni . For ^{55}Fe , increasing the sample mass leads to lower efficiency and significantly longer acquisition times. In contrast, for

^{63}Ni , while efficiency also decreases with greater mass, the acquisition time actually becomes shorter. These opposing behaviors may be due to isotope-specific properties, such as beta energy, self-absorption, or quenching effects. Further details are provided in the Discussion section.

3. Results

Beta energy spectra were acquired for each FTU steel sample using LSC. The Region of Interest (ROI) was set from channel 0 to 200 for ^{55}Fe and channel 0 to 400 for ^{63}Ni . Efficiency was evaluated using a blank sample and a traced standard, both treated using the same chemical procedure (refer to Tables 3 and 4 for details; the traced standard corresponds to vial #2 in Table 3 for ^{55}Fe and to vial #3 in Table 4 for ^{63}Ni).

The measured spectra of the FTU samples showed no significant signal above the background in the defined ROIs for either radionuclide. This indicates that no detectable activity was observed above the critical level (L_C) during the selected acquisition periods.

For ^{55}Fe , in all spectra except the one corresponding to the traced standard (named spiked sample), the observed values were consistent within the measurement uncertainty in the channel range 0–200 (Figure 6). The total number of counts in the 5-day blank measurement was 327,000, corresponding to a detection limit (L_D) of 2700 counts, according to the methodology defined in [16]. This equates to a Minimum Detectable Concentration (MDC) of 0.2 Bq/g. All four analyzed samples (FTU#1 to FTU#4) had count values below L_C , confirming the non-detectability of ^{55}Fe at or above 0.2 Bq/g.

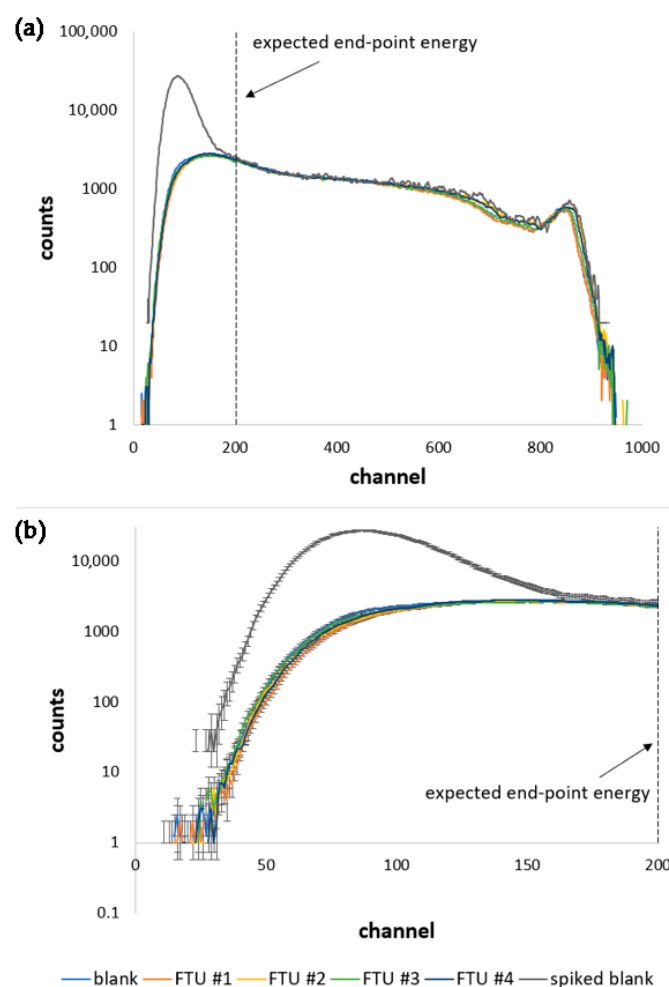


Figure 6. Beta energy distribution of the Fe fraction, including blank, FTU samples, and traced sample: (a) shows the distribution in the whole channel range (0–1024) while (b) shows the distribution with error bars in the range 0–200, corresponding to the beta expected end-point energy for ^{55}Fe .

Similarly, for ^{63}Ni , in all spectra except the one corresponding to the traced standard (named spiked sample), the observed values were consistent within the measurement uncertainty in the channel range 0–400 (Figure 7). The 6 h blank measurement yielded 33,300 counts, corresponding to an L_D of 850 counts, equating to an MDC of 0.3 Bq/g. Again, all FTU samples exhibited count values below L_C , and thus, below the required MDC threshold.

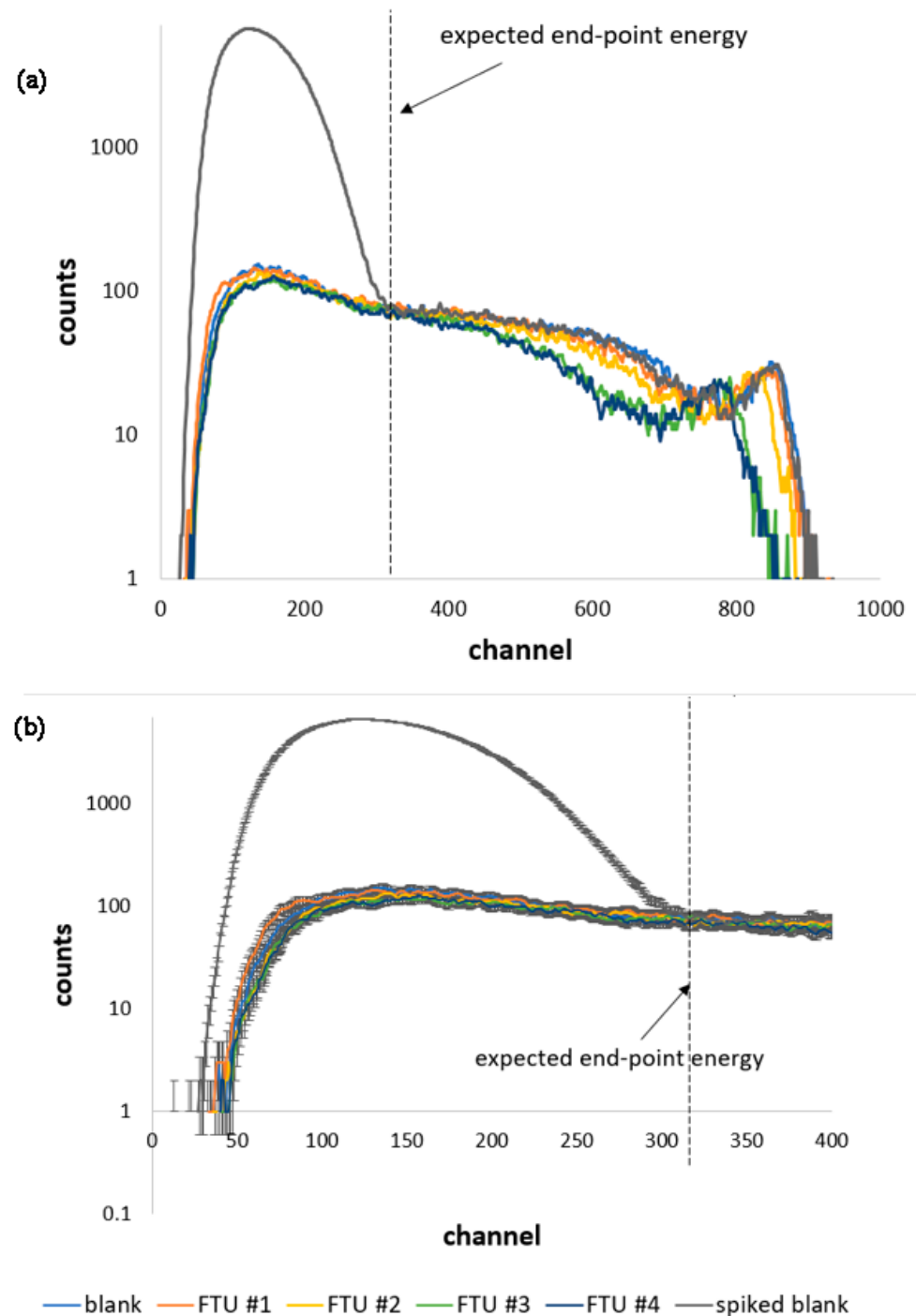


Figure 7. Beta energy distribution of the Ni fraction, including blank, FTU samples, and traced sample: (a) shows the distribution in the whole channel range (0–1024) while (b) shows the distribution with error bars in the range 0–400, considering that 320 corresponds to the beta expected end-point energy for ^{63}Ni .

These results confirm that the activity concentrations of ^{55}Fe and ^{63}Ni in the investigated steel samples are below 0.3 Bq/g, meeting the regulatory requirements for clearance established by the Italian regulatory authority.

4. Discussion

Numerous methods for the isolation of ^{55}Fe and ^{63}Ni have been reported in the literature, including precipitation, solvent extraction, and the use of novel resins such as di-isobutyl ketone-based chromatographic media [13,14]. However, many of these are complex or require highly specific and costly materials. For example:

- TRU resins, while commercially available, have limited capacities (only ~3 mg of Fe).
- Chelex-100 has been used as a pre-concentrator for Fe but lacks selectivity and often requires additional purification steps such as di-isopropyl ether extraction [7].
- Silica-immobilized formyl salicylic acid has a high Fe loading capacity but, again, lacks Fe specificity [17].

In contrast, the method described in this study is cost-effective, scalable, and can be applied to multiple low-activity samples simultaneously—a critical requirement for nuclear facility decommissioning workflows.

After ion-exchange separation, Fe and Ni were collected in HCl fractions, which could not be directly analyzed by LSC due to intense coloration, as shown in Figure 8.



Figure 8. HCl fractions containing Fe (left) and Ni (right) after chemical separation.

The coloration stems from the chemical state of the ions: Fe^{3+} forms yellow to brown hydrolyzed species, contributing to color quenching; Ni^{2+} forms green, often hydrated, complexes such as $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$.

The experimental results confirmed the strong influence of color quenching on detection efficiency.

For ^{55}Fe , doubling the sample mass from 0.125 g to 0.25 g reduced efficiency from ~21% to ~2%, a tenfold drop, due to the increased concentration of the yellow Fe^{3+} complex.

This increase in color quenching significantly extends the acquisition time required to reach the regulatory MDC, making the choice of dissolution acid a crucial factor.

To mitigate this effect, samples were redissolved in H_3PO_4 , which forms colorless complexes with Fe^{3+} and minimizes quenching [15]. While some earlier studies proposed the use of solvent extraction or reducing agents for decolorizing Fe-containing solutions [6,7], our approach of using H_3PO_4 provided a practical and efficient alternative. Although Hou [18] noted the potential of H_3PO_4 for decolorizing Fe^{3+} , its application for optimizing ^{55}Fe quantification in low-activity steel samples remains underexplored.

For ^{63}Ni , LSC performance was less affected by color quenching.

Increasing the sample mass from 0.125 g to 0.25 g only reduced the detection efficiency from 54% to 47%.

This minor decrease implies that most acids can provide acceptable performances for ^{63}Ni analysis, as previously indicated in [15].

5. Conclusions

The chemical procedure presented in this study offers significant advantages over the traditional approaches for the analysis of ^{55}Fe and ^{63}Ni in low-activity steel samples, such as those subjected to limited neutron activation. Compared to methods involving multiple steps—such as organic solvent extraction, precipitation, and chromatography with high-cost stationary phases [6]—this procedure is notably cost-effective, simpler, and results in the reduced generation of radioactive laboratory waste.

Building upon prior experience in the optimization of acid-based sample preparation for LSC, this work has successfully developed a reliable and reproducible methodology for the quantification of trace amounts of ^{55}Fe and ^{63}Ni , even in the presence of large quantities of their non-radioactive isotopes. The method was effectively applied to steel samples from the decommissioning of the ENEA FTU facility, confirming that both radionuclides were below the Minimum Detectable Concentration (MDC) of 0.3 Bq/g, as required by regulatory authorities. These findings validate previous theoretical assessments and activation calculations.

Looking ahead, there is a promising potential for automating this chemical procedure to support high-throughput analysis during nuclear facility decommissioning campaigns. However, strict control of cross-contamination risks is essential, especially considering the extremely low detection thresholds involved. Future developments should focus on automation systems utilizing disposable components and standardized protocols to ensure analytical integrity and compliance with the clearance criteria.

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References

1. Andreani, R. *The FTU Frascati Tokamak Upgrade*; Fusion Technology, Pergamon Books Inc.: Elmsford, NY, USA, 1987; pp. 149–160.
2. Pucella, G.; Alessi, E.; Almagiva, S.; Angelini, B.; Apicella, M.L.; Apruzzese, G.; Aquilini, M.; Artaserse, G.; Baiocchi, B.; Baruzzo, M.; et al. Overview of the FTU results. *Nucl. Fusion* **2022**, *62*, 042004. [\[CrossRef\]](#)
3. Marzo, G.A.; Lepore, L.; Levizzari, R.; Di Pace, L.; Cherubini, N. Non-destructive radiological characterization applied to fusion waste management. *Fus. Eng. Des.* **2021**, *173*, 112805.
4. Griffiths, M. Ni-based alloys for Reactor Internals and Steam Generator Applications. In *Structural Alloys for Nuclear Energy Applications*; Odette, G.R., Zinkle, S.J., Eds.; Elsevier: Amsterdam, The Netherlands, 2019; Volume 9, pp. 349–409. [\[CrossRef\]](#)
5. Wu, Q.; Wu, Y.; Tong, W.; Ma, H. Utilization of nickel slag as raw material in the production of Portland cement for road construction. *Constr. Build. Mater.* **2018**, *193*, 426–434. [\[CrossRef\]](#)
6. Hou, X.; Østergaard, L.R.F.; Nielsen, S.V. Determination of ^{63}Ni and ^{55}Fe in nuclear waste samples using radiochemical separation and liquid scintillation counting. *Anal. Chim. Acta* **2005**, *535*, 297–307. [\[CrossRef\]](#)
7. Konig, W.; Schupfer, R.; Schüttelkopf, H. A fast and sensitive LSC procedure to determine Fe55 in steel and concrete. *J. Radioanal. Nucl. Chem.* **1995**, *193*, 119. [\[CrossRef\]](#)
8. Warwick, P.E.; Cundy, A.B.; Croudace, I.W.; Bains, M.E.D.; Dale, A.A. The Uptake of Iron-55 by Marine Sediment, Macroalgae, and Biota Following Discharge from a Nuclear Power Station. *Environ. Sci. Technol.* **2001**, *35*, 2171. [\[CrossRef\]](#) [\[PubMed\]](#)
9. Hou, X.; Dai, X. Environmental liquid scintillation analysis. In *Handbook of Radioactivity Analysis*; L'Annunziata, M.F., Ed.; Elsevier: Amsterdam, The Netherlands, 2020; Volume 2, pp. 41–136.

10. Peng, C.T. Quenching Correction in Liquid Scintillation Counting. In *Advances in Tracer Methodology*; Rothchild, S., Ed.; Springer: New York, NY, USA, 1966. [CrossRef]
11. Priya, S.; Gopalakrishnan, R.K.; Goswami, A. TDCR measurements of ^3H , ^{63}Ni and ^{55}Fe using Hidex 300SL LSC device. *J. Radioanal. Nucl. Chem.* **2014**, *302*, 353–359. [CrossRef]
12. *SK-10 High Pressure Rotor Application Book*; Milestone: Sorisole, Italy, 2019. Available online: <https://subitam.sinop.edu.tr/wp-content/uploads/sites/93/2019/12/Mikroalga.pdf> (accessed on 13 July 2025).
13. Warwick, P.E.; Croudace, I.W. Isolation and quantification of ^{55}Fe and ^{63}Ni in reactor effluents using extraction chromatography and liquid scintillation analysis. *Anal. Chim. Acta* **2006**, *567*, 277–285. [CrossRef]
14. Corcho-Alvarado, J.A.; Sahli, H.; Röllin, S.; von Gunten, C.; Ossola, J. Validation of a radiochemical method for the determination of ^{55}Fe and ^{63}Ni in water and steel samples from decommissioning activities. *J. Radioanal. Nucl. Chem.* **2020**, *3*, 455–463. [CrossRef]
15. Cozzella, M.L.; Gandolfo, G.; La Notte, G.; Marzo, G.A. A comparative study on the influence of the type of acid used for ^{55}Fe and ^{63}Ni determination in steel samples ANIMMA Lucca (Italy). *EPJ Web Conf.* **2023**, *288*, 07002. [CrossRef]
16. Lloyd, A. Currie, Limits for Qualitative Detection and Quantitative Determination: Application to Radiochemistry. *Anal. Chem.* **1968**, *40*, 586–593.
17. Mahmoud, M.E.; Soliman, E.M. Silica-Immobilized Formylsalicylic Acid As A Selective Phase For The Extraction Of Iron (III). *Talanta* **1997**, *44*, 15–22. [CrossRef] [PubMed]
18. Hou, X. Liquid scintillation counting for determination of radionuclides in environmental and nuclear application. *J. Radioanal. Nucl. Chem.* **2018**, *318*, 1597–1628. [CrossRef]

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