FISA EURADWASTE 2025
SNETP Forum

Rapid separation and determination of ^{55}Fe and ^{63}Ni for decommissioning and nuclear forensics

Ewa Topyła[1], Ewelina Chajduk[1], Zbigniew Samczyński[1]
[1]Institute of Nuclear Chemistry and Technology, Dorodna 16, 03-195 Warsaw

Introduction

^{55}Fe and ^{63}Ni are hard-to-measure pure beta emitters and are widely present in activated parts of nuclear reactors: steel, alloys, and water purification systems. Their determination can be helpful in establishing the nuclear reactor origin of an intercepted material in nuclear forensic investigation. Moreover, along with ^{60}Co, they are reported to be vital in early-stage monitoring of a shut-down nuclear reactors due to their relatively short half-lives.

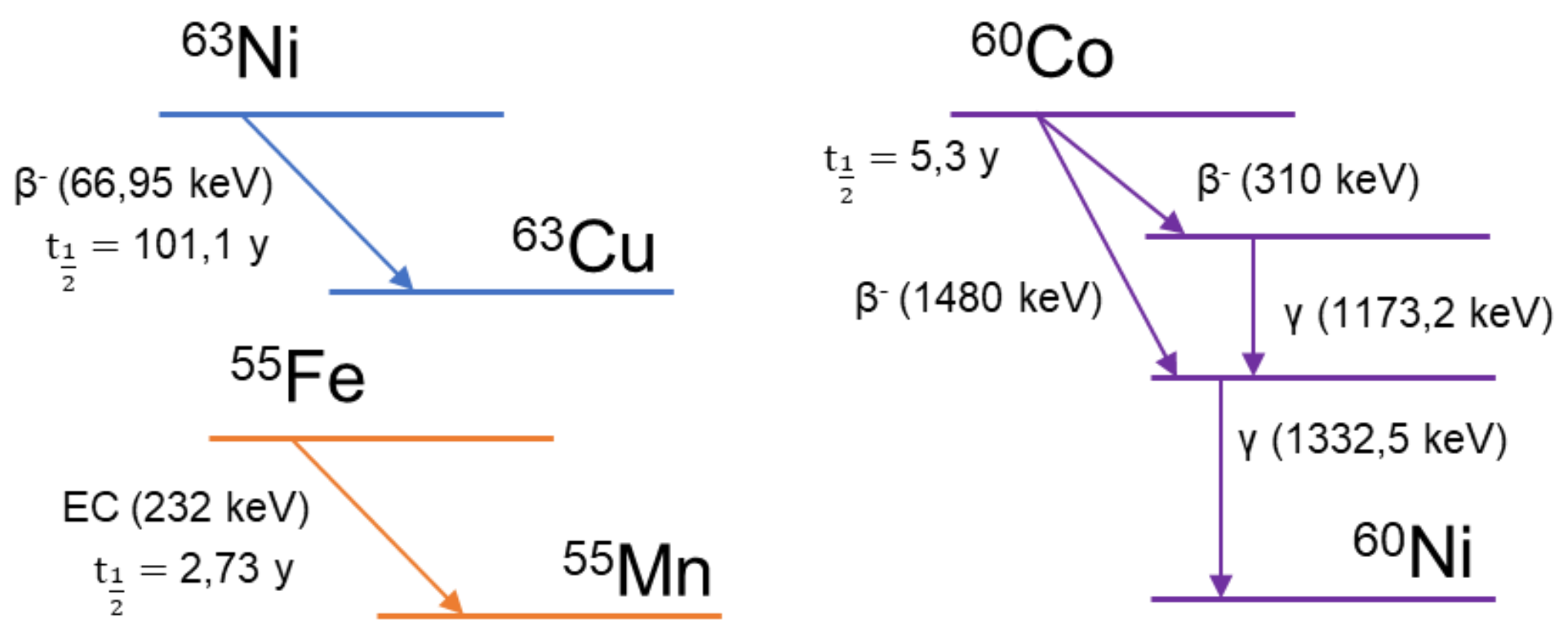


Figure 1. Decay schemes of ^{63}Ni, ^{55}Fe and ^{60}Co

Description of the research problem

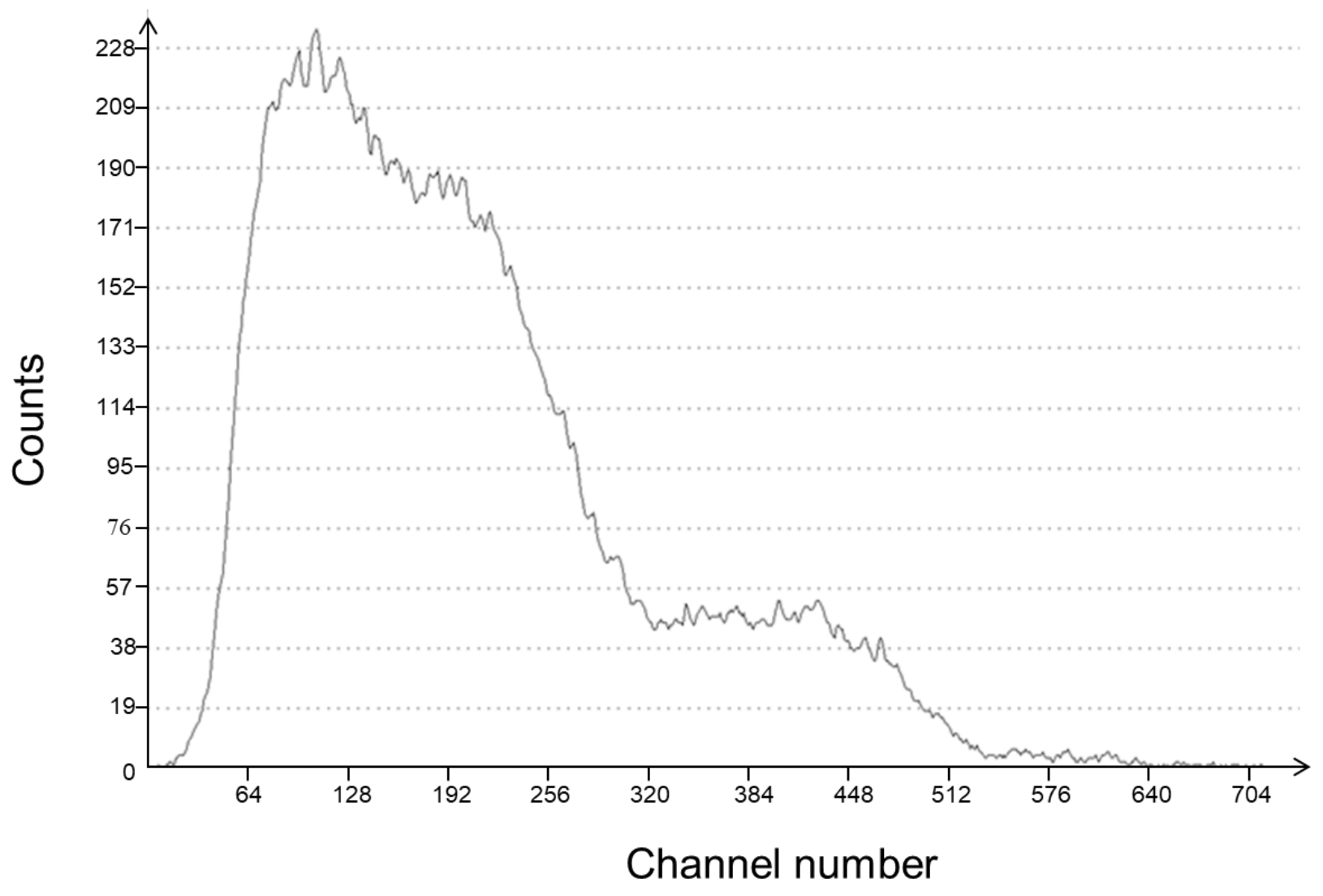


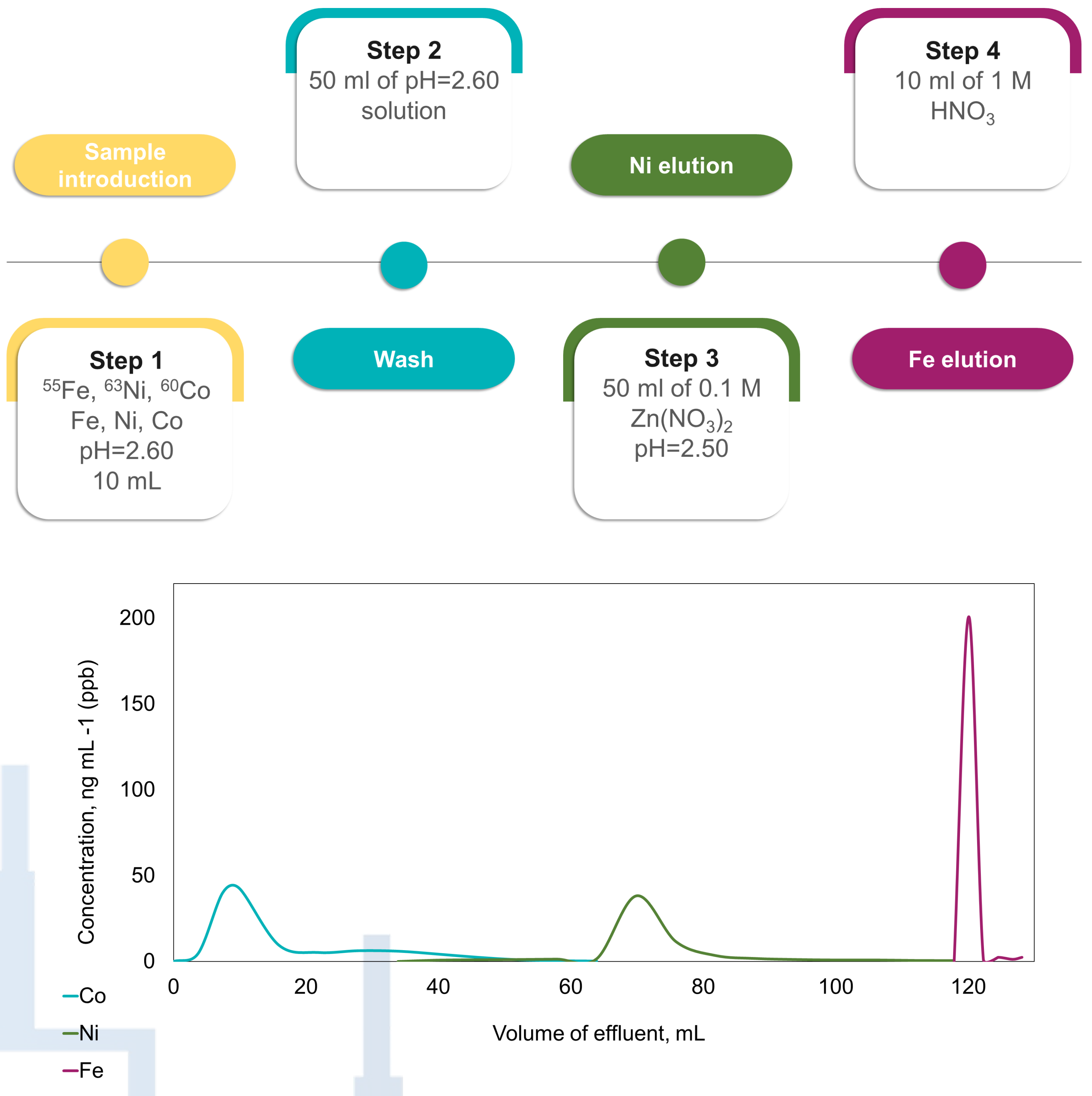
Figure 2. LSC spectrum of mixed solutions of ^{63}Ni, ^{55}Fe and ^{60}Co

In such complicated matrix it is crucial to acknowledge all of the possible interferences that can contribute to the measured signal. Additionally, the procedure developed for nuclear forensic and decommissioning should be as little time and labor consuming as possible. Currently applied methods do not achieve that goal sufficiently.

The aim of this study was to develop a simple and rapid ^{55}Fe and ^{63}Ni separation and determination procedure with LSC detection.

Results

Among tested interferences, only Co(II) constituted an important factor due to the β⁻ activity of ^{60}Co. The method successfully removed ^{60}Co with nitric acid (pH 2.60) before eluting ^{63}Ni with 0.1 M $Zn(NO_3)_2$ and ^{55}Fe with 1 M HNO_3.



Sample introduction

Step 1
^{55}Fe, ^{63}Ni, ^{60}Co
Fe, Ni, Co
pH=2.60
10 mL

Step 2
50 ml of pH=2.60 solution

Wash

Ni elution

Step 3
50 ml of 0.1 M $Zn(NO_3)_2$
pH=2.50

Step 4
10 ml of 1 M HNO_3

Fe elution

Figure 3. Scheme of the elaborated separation procedure and elution curves

Validation with simulated aqueous and stainless steel samples demonstrated an approx. **80% chemical recovery** for both active and non-active Ni and Fe. The detection limits were 0.7 Bq (0.09 Bq/mL) for ^{63}Ni and 1.5 Bq (0.19 Bq/mL) for ^{55}Fe, with an expanded uncertainty of 15%. Despite slightly higher detection limits than alternative methods, this procedure significantly reduces analysis time to ~8 hours, making it suitable for nuclear forensics and emergency response.

Methodology

The procedure employing Chelex 100, well-known weakly acidic chelating cation-exchange resin with a strong selectivity for divalent and trivalent cations was developed and optimized. **Optimization** consisted of **choosing the most efficient conditions of elution (eluents types and volumes, pH), incorporating interferences into the process**, **selecting an ideal scintillation cocktail and LSC measurement conditions**.

- The ^{55}Fe and ^{63}Ni measurements were performed by low-level background Liquid Scintillation Counter (LSC) HIDEX 300 SL
- Gamma measurements were performed with the HPGe (high-purity germanium) well-type gamma detector (Canberra Industries)
- The ELAN DRC II ICP-MS (Perkin-Elmer) equipped with a cross-flow nebulizer, a Scott double-pass, spray chamber and Ni cones was employed for ICP-MS measurements

Conclusions

1) A new separation method for ^{63}Ni and ^{55}Fe was developed using the chelating ion-exchanger Chelex 100.
2) The method was validated with aqueous and stainless steel samples, achieving an 80% chemical recovery rate, as confirmed by LSC and ICP-MS measurements.
3) The procedure effectively removes crucial matrix interferences before elution of ^{63}Ni and ^{55}Fe, preventing disruptions in LSC analysis.
4) Compared to other reactor matrix separation techniques, this method is faster and less labor-intensive, completing analysis within approximately 8 hours. Additionally, its use of a single chromatographic column simplifies the process and makes automation feasible.

Acknowledgments

Partial financial support was received from International Atomic Energy Agency (CRP J02013: Applying Nuclear Forensic Science to Respond to a Nuclear Security Event)