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Nickel and Cobalt Separation using Methyl Fluoride for Ni-59 Determination by ICP-QQQ

Evaluation of the Agilent 8900 ICP-QQQ for novel isobar separation affecting radionuclides



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Introduction

Decommissioning of nuclear sites represents a significant global challenge given the range of sample types and radionuclides that must be characterized. In the UK alone, the estimated final decommissioning cost for existing sites is predicted to range from £95 to 218 billion. Accurate measurement of radionuclides in decommissioning wastes improves measurement confidence and can contribute to safer, more cost-effective decommissioning.[1,2]

Nickel-59 is a radionuclide that is primarily formed through long-term neutron bombardment of stable ^{58}Ni in steel in nuclear reactors. It has a long half-life of 76,000 ± 5000 years.[3] Accurate characterization of ^{59}Ni is therefore important for appropriate waste sentencing and long-term monitoring.[4]

Mass spectrometry, including triple quadrupole ICP-MS (ICP-QQQ), is increasingly used as a rapid technique for measuring long-lived radionuclides in decommissioning wastes. For this approach to be effective, isobaric, polyatomic, and tailing interferences must first be removed. In the case of ^{59}Ni, monoisotopic ^{59}Co, polyatomic ^{58}Ni^{1}H, and peak tailing from the adjacent ^{58}Ni peak (68.08% abundance)[5] must all be fully separated before measurement. Previous mass spectrometric measurements of ^{59}Ni have been performed using accelerator mass spectrometry (AMS).[6,7] However, there is no routine analytical technique in place for ^{59}Ni determination. So far, significant interferences have prevented measurement of ^{59}Ni by ICP-MS. Offline separation of Ni and Co using anion exchange or extraction chromatography is effective.[8,9] However, because ^{59}Co is monoisotopic, some level of interference at *m/z* 59 is still expected.

The in-instrument interference removal capability of ICP-QQQ has been shown to benefit multiple medium- and long-lived radionuclides, including ^{90}Sr, ^{93}Zr, ^{99}Tc, ^{129}I, and $^{135/137}$Cs.[10–12] A recent study using methyl fluoride (CH_3F) as a cell gas for the determination of a range of radionuclides by ICP-QQQ suggested that this method may be close to being considered an analytical technique for ^{59}Ni.[13] This study builds on those results through a detailed characterization of CH_3F as a cell gas for Ni/Co separation using an Agilent 8900 ICP-QQQ.

Experimental

Sample preparation

Single element standards of Ni and Co (1,000 µg/mL, Romil) were diluted to a concentration range of 0.1–100 ng/mL in 1% HNO_3. The 1% HNO_3 diluent was prepared from concentrated HNO_3 (Romil, SPA grade) diluted in deionized water. Additional single Ni standards were prepared up to a concentration of 10,000 ng/mL to give an initial assessment of ^{58}Ni tailing and hydride formation. Stable ^{60}Ni was used as a stable analog to evaluate instrument conditions relevant to ^{59}Ni measurement, given that a ^{59}Ni standard is not currently available.

Instrumentation

All measurements were performed using an Agilent 8900 ICP-QQQ (#100 Advanced Applications model) located at the Agilent Center of Excellence in Cheadle, UK. The instrument was equipped with a MicroMist nebulizer, a Scott double-pass spray chamber, a 2.5 mm internal-diameter quartz torch, Ni sample and skimmer cones, and x-lens. Before use, the instrument was tuned using a mixed stable standard solution to ensure that sensitivity, measurement uncertainty, oxide and doubly charged ion formation rates, and mass axis settings were within operational norms.

The instrument is equipped with four cell gas lines. The CH_3F cylinder (Air Products) was pre-diluted to 10% CH_3F, with the balance in helium (He). It was introduced through the hydrogen cell line, which allows flow rates up to 10 mL/min.

The instrument was operated in MS/MS (or QQQ) mode throughout using appropriate settings for Q1 and Q2, as shown in Figure 1, to separate Co and Ni. Based on initial results from Russell *et al.* 2025,[13] ramp-cell gas tests were conducted for ^{60}Ni and ^{59}Co to identify the best cell gas flow rate for the formation of the respective metal (M) product ions. The Ni ions were detected at *m/z* + 54 ($MFH(CH_3F)$) and *m/z* +102 ($M(CH_3F)_3$). The addition of He from the dedicated He cell line was also investigated for its impact on cell product ion formation. All other cell parameters were optimized (Table 1). Calibration standards were then run under the optimal instrument setup to assess Ni sensitivity and the achievable Ni/Co separation factor.

Table 1. Agilent 8900 ICP-QQQ operating conditions.

Parameter	Setting		
	Ramp Cell Gas Test	Adding Fixed He	Adding Varied He
Scan Mode	MS/MS Ni: Q1 = 60, Q2 = 114, 162 Co: Q1 = 59, Q2 = 113, 161		MS/MS Ni: Q1 = 60, Q2 = 114, 162 Co: Q1 = 59, Q2 = 113, 161
RF Power (W)	1550		
Integration Time (s)	1		
CH_3F Flow Rate (mL/min)	0.0 – 10.0	0.0 – 10.0	5.0, 7.0, and 10.0
Helium Flow Flow (L/min)	0.0	3.0	0.0 – 10.0
Octopole Bias (V)	–5		
Energy Discrimination (V)	–10		
Octopole RF (V)	140		
Axial Acceleration (V)	1.0		

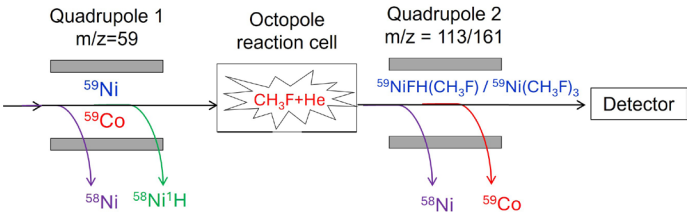


Figure 1. Proposed ICP-QQQ setup for the removal of ^{59}Co interference on ^{59}Ni using CH_3F reaction gas, based on results from stable ^{60}Ni.

Results and discussion

The ramp cell gas tests showed similar behavior for Co and Ni across all flow rates, with both showing peak sensitivity at a flow rate of 2.0 mL/min. At +54 u, Ni was found to shift more than Co, reaching a peak sensitivity at 8.0 mL/min, with similar sensitivity over the range of 6 to 9 mL/min (Figure 2). The optimal separation factor of 55 was achieved at a CH_3F flow rate of 10.0 mL/min. At +102 u, Ni was found to shift more than Co, increasing up to a peak sensitivity at 4.0 mL/min, but with a lower Ni/Co separation factor of 7.9 at 5.5 mL/min (Figure 2). The peak sensitivity of Ni at +102 u was 7.7 times higher than at +54 u. Other cell parameters (Energy Discrimination, Octopole Bias, Octopole RF and Axial Acceleration) had limited impact throughout this study.

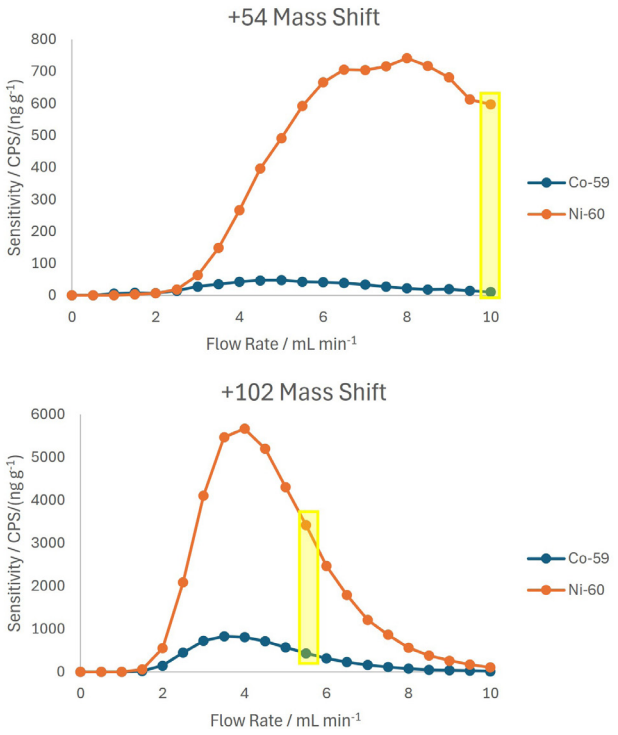


Figure 2. Ni and Co cell product ion formation at different CH_3F flow rates: $MFH(CH_3F)$ product ion (m/z + 54) (top) and $M(CH_3F)_3$ product ion (m/z + 102) (bottom).

To determine whether adding He gas would improve the Ni/Co separation, the ramp cell gas was repeated with He fixed at 3.0 mL/min. At +54 u, the optimal Ni sensitivity was at 5.0 mL/min, lower than the 8 mL/min using CH_3F alone (Figure 3). The optimal Ni/Co separation of 94 was achieved at a CH_3F flow rate of 10 mL/min, with Ni sensitivity improved by a factor of 8.0 compared to using CH_3F alone.

At +102 u, the addition of 3.0 mL/min He resulted in Co shifting significantly more than Ni, meaning no ideal Ni/Co separation could be achieved, a considerable change in behavior compared to CH_3F alone (Figure 3).

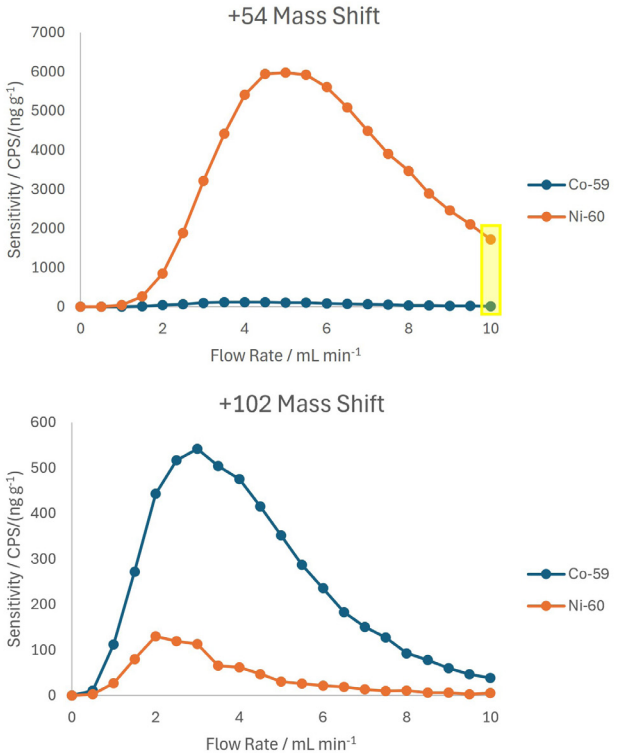


Figure 3. Ni and Co formation of the $MFH(CH_3F)$ product ion (m/z + 54) (top) and $M(CH_3F)_3$ product ion (m/z + 102) (bottom) with different flow rates of CH_3F cell gas and a fixed He cell gas flow rate of 3.0 mL/min.

To determine if varying He gas would improve the Ni/Co separation at +102 u, a ramp cell gas for He was performed with fixed CH_3F flow rates of 5.0, 7.0, and 10.0 mL/min. No significant Ni/Co separation was measured at 5.0 and 7.0 mL/min CH_3F. However, at a CH_3F flow rate of 10.0 mL/min, Ni was found to shift significantly more than Co. A He flow rate of 1.50 mL/min yielded a Ni/Co separation factor of 142 (Figure 4) compared to a maximum of 7.9 using CH_3F alone. The Ni sensitivity was reduced by a factor of 1.7 using a combination of CH_3F and He.

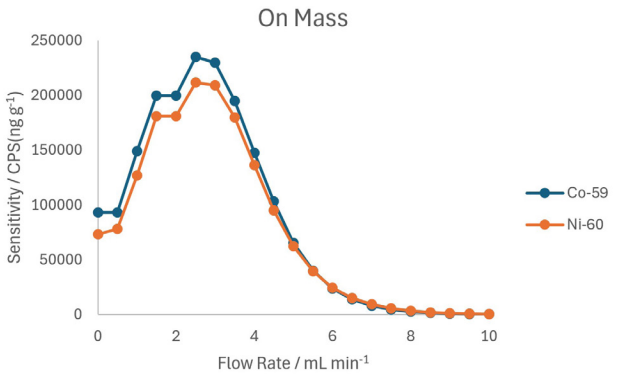


Figure 4. Variation of sensitivity for the formation of the $M(CH_3F)_3$ product ion (*m/z* + 102) for Co and Ni with different flow rates of He cell gas and a fixed CH_3F cell gas flow rate of 10.0 mL/min.

While chemical separation before measurement remains a critical procedural stage to minimize the isobaric ^{59}Co interference, this work demonstrates that residual ^{59}Co present post-chemical separation can be removed using CH_3F cell gas, yielding two potential Ni cell product ions (Table 2). This approach is particularly beneficial in an application where the isobaric interference is monoisotopic. The product ion scan suggested that ^{59}Co in the sample could be monitored at +73 u ($MF_2H(CH_3F)$), where the Ni shift is minimal (Figure 5).

Further testing is needed as stable Ni concentrations in samples containing ^{59}Ni may be very high. However, initial measurements made during this study showed there was no increase in background from polyatomic $^{58}Ni^1H$ or ^{58}Ni tailing up to a concentration of 10 µg/g.

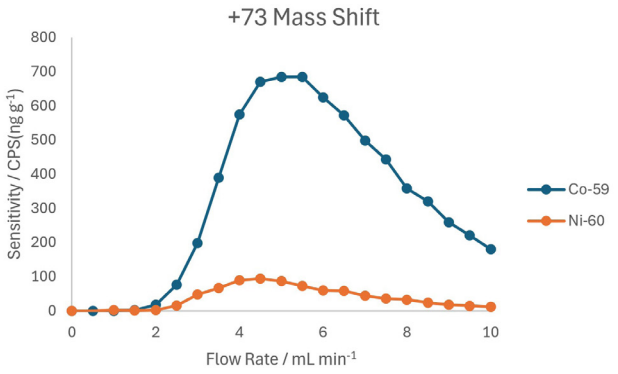


Figure 5. Impact of CH_3F flow rate on formation of $CoF_2H(CH_3F)$ for monitoring the concentration of isobaric ^{59}Co interference free from ^{59}Ni.

Table 2. Summary of optimal cell gas flow rates and Ni/Co separation factors achieved for product ions at +54 and +102 u.

Mass Shift	CH_3F Flow Rate (mL/min)	He Flow Rate (mL/min)	^{60}Ni* (CPS/ ng/g)	^{59}Co (CPS/ ng/g)	Ni*/ Co	BEC (ng/g / Bq/g)
$MFH(CH_3F)$	10.0	0.0	600	11	55	1.7×10^{-3} / 5.0×10^{-3}
	10.0	**3.0**	**1700**	**18**	**94**	5.9×10^{-4} / 1.7×10^{-3}
$M(CH_3F)_3$	5.50	0.0	3415	434	7.9	**2.9×10^{-4} / 8.6×10^{-4}**
	10.0	**1.50**	**1990**	**14**	**142**	5.9×10^{-4} / 1.5×10^{-3}

*Counts per second (CPS) for ^{60}Ni scaled to 100% abundance.

Conclusion

The results demonstrate a significant step towards enabling radioactive ^{59}Ni measurement by ICP-QQQ using optimized cell gas conditions. The stable isotope ^{60}Ni was used as a stable analog of ^{59}Ni as there is no available radioactive standard to evaluate ^{59}Ni directly. Operating the Agilent 8900 ICP-QQQ with a fixed CH_3F cell gas flow rate of 10.0 mL/min, and added helium, would provide the most effective separation of ^{59}Co and ^{59}Ni product ions. This approach has the potential to allow the concentration of the radioactive Ni isotope to be reliably assessed.

The method can next be tested on dissolved steel samples, which are the main sample matrix of interest within the nuclear industry. A broader issue is the lack of a suitable ^{59}Ni tracer for method development and validation, which will need to be addressed as interest in this radionuclide increases.

References

1. Bärenbold, R. *et al.*, Decommissioning of commercial nuclear power plants: Insights from a multiple-case study, *Renewable and Sustainable Energy Reviews*, v 201, p. 114621, Sep. **2024**, doi: 10.1016/j.rser.2024.114621
2. Office for Budget Responsibility, Fiscal risks report – Nuclear decommissioning, London, UK, 2017. Accessed: June 19, 2025. [Online]. Available: https://obr.uk/docs/dlm_uploads/NDA.pdf
3. Mougeot, X. Decay Data Evaluation Project, Laboratoire National Henri Becquerel
4. Persson, P. *et al.*, Detection of ^{59}Ni at the Lund AMS facility, *Nucl Instrum Methods Phys Res B*, 172, 1–4, pp. 188–192, Oct. **2000**, doi: 10.1016/S0168-583X(00)00356-6
5. Meija, J. *et al.*, Isotopic compositions of the elements 2013 (IUPAC Technical Report), *Pure and Appl Chem*, 88, 3, pp. 293–306, Mar. **2016**, doi: 10.1515/pac-2015-0503
6. Gresits, I., Tölgyesi, S. Determination of soft X-ray emitting isotopes in radioactive liquid wastes of nuclear power plants, *J. Radioanal. Nucl. Chem.*, 258, 1, pp. 107–112, Sep. **2003**, doi: 10.1023/A:1026214310645
7. Persson, P. *et al.*, Determination of the detection limit of ^{59}Ni at the Lund AMS facility by using characteristic projectile X-rays, *Nucl Instrum Methods Phys Res B*, 160, 4, pp. 510–514, Apr. **2000**, doi: 10.1016/S0168-583X(99)00622-9
8. Hou, X., Østergaard, L.F., Nielsen, S. P. Determination of ^{63}Ni and ^{55}Fe in nuclear waste samples using radiochemical separation and liquid scintillation counting, *Anal. Chim. Acta*, 535, 1–2, pp. 297–307, Apr. **2005**, doi: 10.1016/j.aca.2004.12.022
9. 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*, 567, 2, pp. 277–285, May **2006**, doi: 10.1016/j.aca.2006.03.043
10. Russell, B. C. *et al.*, Development of a single method for direct measurement of multiple radionuclides using ICP-QQQ, *J Anal At Spectrom*, 38, 1, pp. 97–110, **2023**, doi: 10.1039/D2JA00174H
11. Bu, W., Zheng, J., Liu, X., Long, K., Hu, S., Uchida, S. Mass spectrometry for the determination of fission products ^{135}Cs, ^{137}Cs and ^{90}Sr: A review of methodology and applications, *Spectrochim Acta Part B At Spectrosc*, 119, pp. 65–75, May **2016**, doi: 10.1016/j.sab.2016.03.008
12. Zhou X. Z., Liu, H. W. Accurate determination of calcium and chlorine in food with inductively coupled plasma tandem mass spectrometry, *Spectrosc. Spect. Anal.*, 38, pp. 3567–3571, **2018**
13. Russell, B., Cross, S., Lancaster, S. T., Falksohn, F., North, S. Investigating the potential of methyl fluoride cell gas for radionuclide interference removal and measurement using ICP-QQQ, *J. Anal. At. Spectrom.*, **2025,** 41 (2): 556–567

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DE-018003

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Published in the USA, October 15, 2026
5995-0022EN

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