

RP300^(a)**Nickel-59 and Nickel-63 Determination in Aqueous Samples****1.0 Scope and Application**

This method is used to determine the activity of ^{59}Ni and ^{63}Ni in a solution. It describes the separation and purification of Ni using column precipitation chromatography. It also describes the subsequent determination of ^{59}Ni and ^{63}Ni by low energy photon spectroscopy (LEPS) and liquid scintillation counting (LSC) methods, respectively. Stable Ni is used as a carrier, and yield is determined by inductively coupled plasma optical emission spectroscopy (ICP-OES) (or equivalent) analysis of the Ni.

The method was developed for use with highly radioactive Hanford tank samples. It has also been applied to activated stainless steel samples. Variations in the radioactive isotopes present and the radioactive Ni activity will be factors used to determine aliquot size and amount of carrier added. Specific sample matrices {other than single-shell tanks (SST), double-shell tanks (DST), and activated stainless steel} may need to be tested with this procedure to ensure its applicability.

2.0 Summary of Method

A sample is equilibrated with stable Ni carrier and prepared for loading onto a dimethylglyoxime (DMG) precipitation column. The sample is loaded on the DMG column where the Ni forms a Ni-DMG complex. Other cations and anions are effectively eluted with the load and wash solutions. Nickel is stripped the column with 3 M HNO_3 . It is again precipitated as the Ni-DMG complex, filtered onto a 1-in. filter paper, and washed. The filter paper Ni-DMG complex is mounted for LEPS counting where ^{59}Ni activity is determined. The Ni-DMG complex is then oxidized in a muffle furnace leaving NiO, which is then dissolved. An aliquot of the Ni solution is then counted by LSC for ^{63}Ni activity, and another aliquot is analyzed for total Ni to determine yield.

3.0 Interferences

3.1 This method has been tested on Hanford waste tank matrices including drainable liquids and acid dissolved sludges. Validation testing of other matrices will need to be performed by the analytical laboratory.

(a) This method was supplied by Robert Strebin, Robert Orr, James Kaye, and Sandra Fadeff (Pacific Northwest Laboratory, Richland, Washington).

- 3.2 Careful attention needs to be paid to the separations of activated cobalt. The separation efficiency can be monitored with use of gamma energy analysis. If further separations are required, the Ni-DMG precipitate may be dissolved, re-precipitated, and washed.
- 3.3 Because stable Ni is used as a yield monitor, the amount of Ni in the original sample may need to be determined to calculate Ni recovery.
- 3.4 The method has not been tested for ratios of $^{59}\text{Ni}/^{63}\text{Ni}$ greater than 1/40. The ^{59}Ni activity is counted by liquid scintillation, and its peak energy is not resolvable from that of ^{63}Ni . A correction of the net LSC count rate may need to be performed for analyzing materials with greater relative ^{59}Ni activities.
- 3.5 The mass of Ni accommodated by this procedure is limited. The DMG precipitation column can only accommodate up to 3 mg Ni. Large amounts of Ni (e.g., >5 mg) may cause absorption and quenching problems with counting techniques. The absorption and quenching problems may be corrected with appropriate calibration curves.
- 3.6 This procedure uses ethanol, which will result in mixed waste; careful separation of the waste needs to be maintained such that the ethanol is not mixed with the HNO_3 .

4.0 Safety

This procedure uses ethanol in conjunction with an organic compound, dimethylglyoxime. Appropriate safety precautions need to be followed when storing and disposing of an organic with HNO_3 . Otherwise, no significant safety problems are presented by this procedure other than the normal precautions for handling radioactive materials, acids, and bases.

5.0 Apparatus and Materials

- Disposable column (Empty Quick-Snap Columns, Isolab or equivalent)
- Stopcock to fit disposable column
- Vortex stirrer
- Centrifuge
- Centrifuge tubes, plastic, 50 mL
- Transfer pipets, plastic and glass
- Plastic beakers, disposable, 5 mL

RP300^(a)

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- Scintillation vial, low potassium glass, 20 mL
- Plastic screw cap bottles, 20 mL
- Filter holder, 50 mL polysulfone filter funnel with stainless steel support screen (Gelman 4203/4204) or equivalent
- Suction flask
- Tuffryn membrane filter, HT 200, 25 mm diameter or equivalent
- Petri dish

6.0 Reagents

All reagents are prepared from analytical reagent grade chemicals. Deionized water, with an electrical resistance of >16 megohms-cm, is used throughout this method. Except for the stable Ni carrier and radioisotope standards, concentrations are not critical and need only be within $\pm 10\%$ of stated values.

Holdback carrier: a suite of carriers may be used as deemed necessary to accommodate the nature of the sample. Suggested carriers for SST and DST samples include Sr, Co, La, Cs, and Y. The chemical form is not important as long as the compound is readily soluble. Carriers are usually prepared to make individual solutions of 1 to 10 mg/mL. A combined carrier mix can be made, which can then be added to each sample.

- Standardized Ni carrier solution of approximately 10 mg/mL in 1 to 2 M HNO_3 or HCl
- ^{63}Ni , certified ^{63}Ni standard from NIST or other certified vendor
- Sodium hydroxide, 5 M and 0.5 M
- Hydrochloric acid, 12 M (concentrated), 9 M and 0.1 M
- Ammonium hydroxide, 14.5 M (concentrated), and 0.7 M
- Ammonium citrate $\{(\text{NH}_4)_2\text{HC}_6\text{H}_5\text{O}_7\}$, 1 M
- Ammonium citrate wash solution (15 mL 1 M ammonium citrate diluted to 100 mL adjusted to pH 8.5 to 9.0 with NH_4OH)

- Nitric acid, 16 M (concentrated), 3 M and 2 volume %
- Ultima-Gold™ LSC cocktail, liquid scintillation cocktail sold by Packard Instrument Company (or equivalent)
- Ethanol, absolute
- Basic buffer solution (water adjusted to pH 8.5 to 9.0 with NH_4OH ; one drop of concentrated NH_4OH in 100 mL of water will buffer to pH 8.5 to 9.0)
- Dimethylglyoxime (DMG)
- Dimethylglyoxime - Saturated Ethanol {Sufficient DMG (2 to 3 g) is added to 50 mL ethanol with mixing and allowed to stand overnight. The solution is saturated when solid DMG is still present. }
- Tartaric acid, 0.7 g/mL of water
- Microthene™, 50 mesh polyethylene powder

7.0 Sample Collection, Preservation, and Handling

Aqueous samples should be acidified to pH < 2 with HCl or HNO_3 . No other preservation or storage requirements are indicated.

8.0 Procedure

8.1 DMG Column Preparation

- 8.1.1 To a beaker, 0.5 g DMG is added, followed by 50 mL ethanol. The mixture is stirred and heated as needed to aid dissolution. (This will be called the DMG-ETOH mix.)

This preparation is enough to fill 10 columns. If more than 10 columns are required, the reagent volumes will need to be proportionately increased.

- 8.1.2 To a beaker containing 4 g Microthene™, 5 to 10 mL of DMG-ETOH mix is added and evaporated with heating and stirring until the mix is a slurry. The addition of the DMG-ETOH mix and evaporation is repeated until all the DMG-ETOH mix from step 8.1.1 has been added, and the mix is a thick slurry.

- 8.1.3 Ten milliliters of basic buffer solution are added dropwise to the slurry with vigorous stirring. (This will be called DMG-MIC mix.)

If more buffer is added, the slurry will separate into phases.

- 8.1.4 A small disposable column is prepared by removing the bottom frit (if present), replacing it with a glass wool plug, and attaching a stopcock to the bottom of the column. The DMG-MIC is transferred to the column until the column has been filled to 3 mL. Excess liquid is drained off as the DMG-MIC is added.

When loading the column, a head of liquid should be kept over the microthene bed.

- 8.1.5 When the bed is filled to 3 mL, the liquid is carefully drained to just over the top of the bed (~1 mm). The frit that was removed from the bottom of the column is placed on the top of the bed, leaving a small gap between the frit bottom and the bed.

The headspace in the column is essential to allow for bed expansion as the Ni-DMG precipitates. Air pockets in the column should be avoided.

- 8.1.6 The prepared column is washed with 5 to 10 mL of basic buffering solution. The column is ready for use and can be allowed to stand wetted with the basic buffering solution. Just before use, the column is washed with 2 to 5 mL of basic buffering solution.

8.2 Separation Procedure

- 8.2.1 A known amount (e.g., 2 mg) of Ni carrier is added to each of the following:

- centrifuge tube (or appropriate size beaker) for each sample
- plastic LSC vial for a yield monitor (to be used to calibrate the stable Ni concentration)
- two glass LSC vials (for LSC blanks)
- two glass LSC vials (for LSC ^{63}Ni standards).

The yield monitor vial is capped and saved for step 8.3.8.

8.2.2 A known amount of ^{63}Ni (sufficient to give good counting statistics in a reasonable count time) is added to each of the following:

- blank spike
- matrix spike (if one is to be processed)
- two glass LSC vials identified above for LSC ^{63}Ni standards.

The LSC ^{63}Ni standards and blanks are saved for step 8.3.6; step 8.3.8 is unnecessary for the the LSC ^{63}Ni standards and blanks.

8.2.3 An appropriate sample aliquot is measured and placed in the 50-mL plastic centrifuge tube (or beaker) containing the Ni carrier. Holdback carriers may be added at this point.

8.2.4 Basic samples (such as tank drainable fluids) are acidified with conc HCl.

When basic tank fluids are acidified, a precipitate may form. The precipitate is centrifuged and washed twice with 6 M HCl, and the washes are combined with the sample. The washed precipitate is discarded.

8.2.5 Large samples (e.g., >5 mL) may be concentrated by evaporation or iron hydroxide precipitation. If concentration is not needed, the method is continued at step 8.2.6.

Iron Hydroxide Precipitation

8.2.5.1 A total of 0.1 to 0.2 mg Fe carrier is added to the sample.

8.2.5.2 Then 5 M NaOH is added dropwise, with stirring after each addition. The pH is tested when the first sign of precipitation occurs. Then 0.5 M NaOH and various concentrations of HCl are added to adjust the pH to 7 to 8.

Wide range pH paper (pH 2 to 10) and narrow range pH paper (pH 6 to 8) are used as appropriate to measure the pH. If the pH is exceeded, $\text{Ni}(\text{OH})_2$ will start to dissolve.

8.2.5.3 The sample is centrifuged 10 min on high setting, and the supernate is discarded.

- 8.2.5.4 The precipitate is washed with 5 to 10 mL of pH 7 water. The supernate is discarded.
- 8.2.5.5 The precipitate is dissolved in approximately 4 to 6 drops (or minimum amount needed) of 6 M HCl with swirling. The sample is diluted to 1 to 5 mL with water.
- 8.2.6 One milliliter of 1 M ammonium citrate is added to the sample, which is then stirred well. Concentrated NH_4OH is added to the sample dropwise to pH 8 to 9.
- The pH is not critical as long as it is at least pH 8 and < pH 10. The sample will turn from a green color to a blue color; the solution pH is checked with pH paper as the matrix color may mask the solution color change.*
- 8.2.7 The sample is loaded onto the DMG column in 1-mL aliquots. The column flow rate should be adjusted to approximately 1 drop per 2 sec.
- The Ni reaction with the DMG gives a red Ni-DMG complex precipitate band on the column.*
- The effluent is discarded.
- 8.2.8 The column is washed with 20 mL of ammonium citrate wash solution added in 1-mL aliquots for the first 5 mL, then in 1- to 3-mL aliquots. The wash volume may be increased. The effluent is discarded.
- A portion of the effluent may be counted to assess the separation.*
- 8.2.9 The Ni is stripped from the column with 15 mL of 3 M HNO_3 added in 1- to 5-mL aliquots.
- The red Ni-DMG dissolves, giving a colorless column and solution.*
- 8.2.10 A filter apparatus with a Tuffryn membrane filter is set up.
- 8.2.11 To the eluent from step 8.2.9, 0.1 mL of 0.7 g/mL tartaric acid is added and the solution stirred. Then 1 mL DMG saturated ethanol is added. Concentrated ammonium hydroxide is added dropwise with stirring to pH 8 to 9. The red Ni-DMG complex will precipitate. The solution is allowed to stand for about 1 h for complete precipitation.

8.3 Preparation for Counting

8.3.1 The solution is filtered (the filter will be removed for subsequent LEPS counting). The Ni-DMG complex precipitate is washed with 10 to 20 mL of hot water. The filter is removed, placed on a petri dish, and dried at 100 to 120° C for approximately 1 h.

8.3.2 The Ni-DMG complex filter is mounted for LEPS counting.

The mount may be performed as follows: Mylar film is sealed to the back of a cardboard planchet holder with a 1 in. diameter hole. The filter containing the Ni-DMG complex precipitate is placed in the opening, covered with mylar, and sealed.

8.3.3 The sample is counted on a LEP detector for ⁵⁹Ni activity.

If better separation is needed as determined from the LEP spectrum, then Ni-DMG may be dissolved in 3 M HNO₃, reprecipitated, and re-washed as indicated in step 8.2.11 and 8.3.1.

8.3.4 After LEPS counting, the Ni-DMG complex/filter is placed in a glass LSC vial and muffled overnight (or until the oxidation is complete) at 500° C.

The Ni-DMG complex is converted to NiO.

8.3.5 The NiO is dissolved with a minimum of aqua regia (three parts conc HCl to one part conc HNO₃). The acid matrix is converted to the chloride form by evaporating the dissolved Ni to near dryness and adding one mL of concentrated HCl. The evaporation and HCl addition are repeated one to two times.

The Ni(NO₃)₂•6H₂O has a boiling point of 137°C and can be lost if a nitrate solution goes to dryness; NiCl₂ is not volatile.

8.3.6 One mL of concentrated HCl is added, and the solution is evaporated to dryness under a heat lamp.

8.3.7 After cooling, exactly 1 mL of 0.1 M HCl is added to the LSC vial. The solution is allowed to stand about 30 min with occasional swirling to ensure dissolution and mixing of the Ni.

8.3.8 An aliquot (e.g., 0.05 or 0.1 mL) of the sample solution is transferred to an LSC vial and an exact volume (e.g., 10 mL of 2 volume % HNO₃) is added.

In the same manner, exactly 10 mL of 2 volume % HNO₃ are added to the yield monitor solution. The samples and yield monitor are ready for Ni analysis by ICP-OES (or equivalent) for yield determination.

- 8.3.9 Ultima-Gold™ LSC cocktail (e.g., 10 mL) is added to the LSC vial (from step 8.3.7), the vial is capped, and the solution is mixed well. The exterior of the LSC vial is wiped with ethanol or other suitable solvent to eliminate surface residues (e.g., fingerprints). The sample is now ready for scintillation counting.

8.4 LEPS Counter Calibration

The low-energy photon detector counting efficiency may be calibrated relative to ⁵⁹Ni or, if ⁵⁹Ni is not available, the counting efficiency may be interpolated relative to other X-ray emitters such as ⁵⁵Fe and ⁶⁵Zn. The K X-ray energies of ⁵⁵Fe (5.90 keV) and ⁶⁵Zn (8.05 keV) are close to that of ⁵⁹Ni (6.93 keV). The X-ray counting efficiencies can be determined after correction for the percentage of K X-rays produced per disintegration. The ⁵⁹Ni X-ray counting efficiency can then be interpolated, and after correction for the K X-ray abundance, the counting efficiency determined. The calibration source can be prepared by pipetting a known amount of ⁵⁹Ni uniformly on the filter paper geometry. Otherwise, two isotopes, ⁵⁵Fe and ⁶⁵Zn, are pipetted uniformly on separate filter papers. Geometry of the standards relative to the samples must be constant.

A calibrated ⁵⁹Ni standard has been prepared in the Ni-DMG complex geometry, and its counting efficiency was well within the error of the efficiency determined previously relative to an ⁵⁵Fe source (work performed at the Analytical Chemistry Laboratory, Pacific Northwest Laboratory, 1993).

8.5 LSC Counter Calibration

The LSC is calibrated relative to the ⁶³Ni standards and blanks prepared with the analytical batch (steps 8.2.1 and 8.2.2). The detector efficiency is determined based on the obtained count rate of the standard and the known ⁶³Ni activity. The calibration blank average containing added stable Ni is used for blank subtraction.

9.0 Calculations

9.1 Determination of ⁵⁹Ni Counting Efficiency

The 6.9 keV Co X-ray peak (from the electron capture decay of ⁵⁹Ni) counted on the LEP diode is used to calculate ⁵⁹Ni in the sample. The counting efficiency can be

determined directly if ^{59}Ni is used as a calibration source. The inverse of the counting efficiency (d/c) is determined for each LEP detector as follows:

$$\frac{d}{c} (^{59}\text{Ni}) = \frac{\text{DR}_{^{59}\text{Ni}}}{\text{net CR}_{^{59}\text{Ni}}}$$

where

DR	=	disintegration rate
CR	=	count rate

The ^{59}Ni X-ray counting efficiency (E) may need to be interpolated between other X-ray emitters, ^{55}Fe and ^{65}Zn for example. The counting efficiencies of the X-rays produced as a result of the electron capture decay of ^{55}Fe and ^{65}Zn on the LEP diode are determined as follows:

$$E_{^{55}\text{Fe}} = \frac{\text{net CR}_{^{55}\text{Fe}}}{\text{DR}_{^{55}\text{Fe}} \cdot \frac{\text{X-ray}_{^{55}\text{Fe}}}{d_{^{55}\text{Fe}}}}$$

$$E_{^{65}\text{Zn}} = \frac{\text{net CR}_{^{65}\text{Zn}}}{\text{DR}_{^{65}\text{Zn}} \cdot \frac{\text{X-ray}_{^{65}\text{Zn}}}{d_{^{65}\text{Zn}}}}$$

where (from Browne and Firestone 1986)

$$\frac{\text{X-ray}_{^{65}\text{Zn}}}{d_{^{65}\text{Zn}}} = 0.3828$$

$$\frac{\text{X-ray}_{^{55}\text{Fe}}}{d_{^{55}\text{Fe}}} = 0.2726$$

The X-ray counting efficiency for ^{59}Ni is then the average of the X-ray counting efficiencies for ^{55}Fe and ^{65}Zn . The inverse of the counting efficiency (d/c) for ^{59}Ni is then determined as follows:

HNO₃, organics were then oxidized with 30% H₂O₂, and digestion was continued with 50% HCl. The sample was brought to 100 mL final volume. Triplicate 20-mL samples were taken and spiked with 0.77 Bq (46 dpm) ⁵⁹Ni and 31.7 Bq (1903 dpm) ⁶³Ni.

A second set of samples was prepared from activated stainless steel samples (Fadeff et al. 1994). These samples were sectioned from non-fuel bearing components of nuclear reactors. The samples were cleaned extensively, then separated according to this method. Two sets of cold stainless steel matrix spikes and blank spikes were run as well as three sets of duplicate samples (CRB6, BPRA5, and RCCA8).

A summary of the analytical bias is given in Table 1, and a summary of the analytical precision is given in Table 2. Typical chemical recoveries are equal to, or greater than, 90%. A summary of chemical recoveries obtained from a batch of 11 samples is given in Table 3.

Table 1 Bias and Precision of ⁵⁹ Ni and ⁶³ Ni						
Sample ID	Bq ⁵⁹ Ni Added	Bq ⁵⁹ Ni Recovered	⁵⁹ Ni % Bias	Bq ⁶³ Ni Added	Bq ⁶³ Ni Recovered	⁶³ Ni % Bias
Tank Waste Matrix						
A	0.767 ± 0.093	0.730 ± 0.087	- 5	31.72 ± 0.38	29.83 ± 1.83	- 5.9
B	0.767 ± 0.093	0.802 ± 0.095	+ 5	31.72 ± 0.38	30.67 ± 2.00	- 3.3
C	0.767 ± 0.093	0.777 ± 0.095	+ 1	31.72 ± 0.38	28.83 ± 1.83	- 9.1
Activated Metals Blank Spike and Matrix Spike						
Matrix Blank Spike 1	1.52 ± 0.18	1.48 ± 0.17	-2.2	127.7 ± 1.6	127.7 ± 10.5	- 0.04
Matrix Blank Spike 2	na			126.9 ± 1.6	128.8 ± 11.5	+ 1.5
Blank Spike 1	1.52 ± 0.18	1.50 ± 0.20	- 1.1	127.7 ± 1.6	128.7 ± 10.8	+ 0.7
Blank Spike 2	na			126.9 ± 1.6	129.9 ± 11.5	+ 2.4

Table 2. Summary of Method Precision

Sample ID	⁵⁹ Ni (Bq)	RPD/RSD ^(a) Tank Waste Matrix	⁶³ Ni (Bq)	RPD/RSD
A	0.730 ± 0.087	4.8%	29.83 ± 1.83	3.1%
B	0.802 ± 0.095		30.67 ± 2.00	
C	0.777 ± 0.095		28.83 ± 1.83	
	(M Bq/g)	Activated Metals Matrix	(M Bq/g)	
CRB6	0.299 ± 0.037	4.2%	36.1 ± 3.0	0.20%
CRB6 duplicate	0.287 ± 0.037		36.0 ± 3.0	
BPRA3	0.077 ± 0.010	3.3%	8.21 ± 0.70	1.4%
BPRA3 duplicate	0.080 ± 0.010		8.10 ± 0.70	
RCCA8	3.05 ± 0.41	3.6%	377 ± 34	4.4%
RCCA8 duplicate	2.94 ± 0.37		361 ± 33	

(a) RPD - relative percent difference
RSD - relative standard deviation

Table 3. Chemical Recoveries

Sample No.	% Recovered
1	94
2	96
3	91
4	97
5	101
6	96
7	64
8	91
9	100
10	96
11	98
Average	93 ± 10

$$\frac{d}{c}({}^{59}\text{Ni}) = \frac{\left(\frac{\text{X-ray } {}^{59}\text{Ni}}{d_{{}^{59}\text{Ni}}}\right)^{-1}}{\left[\left(E_{{}^{55}\text{Fe}}\right) + \left(E_{{}^{65}\text{Zn}}\right)\right] \cdot \frac{1}{2}}$$

where

$$\frac{\text{X-ray } {}^{59}\text{Ni}}{d_{{}^{59}\text{Ni}}} = 0.3306$$

The values of (X-ray/d) are the sum of $K_{\alpha 1}$, $K_{\alpha 2}$, and $K_{\beta 1}$ X-ray absolute abundances per disintegration. Adding the value for $K_{\beta 1}$ to the ratio will depend on the users' LEP detector resolution, as the average energy difference between the $K_{\alpha 2}$ and $K_{\beta 1}$ is approximately 0.7 keV. Typically the resolution {full width half maximum (FWHM)} of a Ge detector is ~2 keV; therefore, a peak of 0.7 keV difference in energy would not be easily resolvable.

9.2 Determination of ${}^{59}\text{Ni}$ and ${}^{63}\text{Ni}$ Activity

The ${}^{59}\text{Ni}$ activity is calculated in the sample using the following equation:

$$A_{{}^{59}\text{Ni}} = \frac{(\text{net CR})(d/c_{{}^{59}\text{Ni}})(k)}{(Y)(Q)}$$

The ${}^{63}\text{Ni}$ activity is determined from the LSC spectra using the following equation:

$$A_{{}^{63}\text{Ni}} = \frac{(\text{net CR})(d/c_{{}^{63}\text{Ni}})(k)(v_{\text{corr}})}{(Y)(Q)}$$

where

A = analyte activity

net CR = the gross count rate minus the background count rate

k = factor to account for sample dilutions, and any other factors needed to produce the appropriate reporting units

Y = yield determined by the amount of stable Ni recovered relative to the amount added

- v_{corr} = sample volume correction at step 8.3.8 where a fraction of sample is taken for ICP analysis (volume total/volume for LSC analysis)
- d/c = disintegrations per count, inverse of efficiency (for LSC, this will include the quenching effects)
- Q = quantity (volume or mass) of sample analyzed.

The error (σ) in the activity is determined as follows:

$$\sigma_{59\text{Ni}} = A_{59\text{Ni}} \cdot \sqrt{\left(\frac{\sigma_{59\text{Ni}} \text{ CR}_{59\text{Ni}}}{\text{net CR}_{59\text{Ni}}}\right)^2 + \left(\frac{\sigma_{\text{eff}_{59\text{Ni}}}}{\text{eff}_{59\text{Ni}}}\right)^2 + \left(\frac{\sigma_{\text{yield}}}{\text{yield}}\right)^2 + \left(\frac{\sigma_Q}{Q}\right)^2}$$

$$\sigma_{63\text{Ni}} = A_{63\text{Ni}} \cdot \sqrt{\left(\frac{\sigma \text{ CR}_{63\text{Ni}}}{\text{net CR}_{63\text{Ni}}}\right)^2 + \left(\frac{\sigma_{\text{eff}_{63\text{Ni}}}}{\text{eff}_{63\text{Ni}}}\right)^2 + \left(\frac{\sigma_{v_{\text{corr}}}}{v_{\text{corr}}}\right)^2 + \left(\frac{\sigma_{\text{yield}}}{\text{yield}}\right)^2 + \left(\frac{\sigma_Q}{Q}\right)^2}$$

where

$$\sigma = \text{error in the specified value}$$

The error tends to be driven by the errors in counting efficiency and yield. For low-level samples, the error in count rate (i.e., net cpm) also becomes significant.

10.0 Quality Control

- 10.1** It is recommended that one reagent blank, one sample duplicate, and one blank spike or matrix spike be run to provide an estimate of the analytical batch bias and precision.
- 10.2** Chemical yields should be over 50% and should not exceed 110%.
- 10.3** Chemical quench factors for liquid scintillation counting should be similar between the samples and the standards.

11.0 Method Performance

This method was performed by a single laboratory on single-shelled tank sludge waste matrix from Hanford samples A, B, and C. Previous studies indicated the waste contained little ^{59}Ni and ^{63}Ni ; therefore, triplicate samples could be spiked with an appropriate amount of ^{59}Ni and ^{63}Ni for a study of precision and bias. A 0.48-g sludge sample was digested in a hot cell with 50%

12.0 References

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13.0 Further Reading

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