

John White - Ni-63

From: "Adler, Joseph J." <jadler@entergy.com>
To: "John White" <JRW1@nrc.gov>, "James Kottan" <JJK@nrc.gov>
Date: 02/08/2007 12:05 PM
Subject: Ni-63
CC: "Croulet, Donald K" <dcroule@entergy.com>

John and Jim,

Attached is Areva's report concerning the Ni-63 split-sample results for MW-42.

Jay Adler

<<CR 07-05.pdf>>

B/JS

ENVIRONMENTAL LABORATORY



CONDITION REPORT

Condition Level (Circle One)
(Complete at Review Committee Meeting)

1 (2) 3

Condition Report No.: 07-05

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PART 1: IDENTIFICATION OF CONDITION (Originator)

(Fill in all known data or attach Client Corrective Action documents.)

- A) Initiated By (Print): Christopher Shelton Org.: E-LAB Ext.: 36663
- B) Initiation Date: 1/31/07
- C) Reference Document (Manual, Procedure, Audit/Surveillance No., etc.): QA Manual 100
- D) Condition Description (Describe the Condition and Suspected Cause, if Known):
A total of 4 Entergy-IP water samples (L11703-09, -10, -11, -12) indicated non-positive activity for Ni-63. The client noted that a second lab (NRC - ORISE) identified positive activity for Ni-63. Small fractions (unacidified) held for H-3 backup were used to RP the samples. The RP results were verified with Entergy-IP personnel to compare favorably with the ORISE results on 1/31/07.

E) Proposed and Approved Corrective Actions (Originator and LQARC):

| | RM | Action Section | Due Date |
|--|-----|----------------|----------|
| 1) Describe the processing problems that caused the first analyses to be non-positive. | ADB | RC | 2/9/07 |
| 2) Issue RP results with note on use of unacidified backup fractions. | EMM | SC&M | 2/9/07 |
| 3) Verify that existing processes contain sufficient controls to prevent recurrence. | ADB | RC | 2/9/07 |
| | | | |
| | | | |

PART 2: REVIEW OF CONDITION/REPORTABILITY (E-LAB Manager)

1. Immediate Data Quality Concern: NO YES
2. Personnel Injury/Accident - Accident Type: NO YES
3. Recommended Condition Level: 2
4. Client Notifications Required? NO YES (Notify Client Representative) Pete Hollenbeck - Entergy IP
- Reference Client Corrective Action Document (e.g., ER No., IR No., etc.): _____ Plant: Entergy-Indian Point
- E-LAB Manager Signature: [Signature], 2/2/07 (Date)

PART 3: LQARC EVALUATION

- A) Initial Notifications and Reviews Appropriate: NO YES Needs Further Review
- B) Potentially Reportable: NO YES Review Assigned To: _____ / _____ (Due Date)
- C) Condition Level Determined (Annotate on Top of Page 1)
- D) Additional Corrective Action Necessary: NO YES (Add to Part 1.E)
- E) Apparent Cause Code(s): 19A
 Primary Secondary Secondary
- F) LQARC Approval: [Signature], 2/2/07 (Date)
 LQARC Chairperson or Designee

PART 4: CONDITION INVESTIGATION/CORRECTIVE ACTIONS

A) Investigation/Corrective Action Narrative Summary See Attached

- 1) See attached investigation and root cause determination.
- 2) Initial results for Ni-63 for L11703-09, -10, -11 and -12 were issued 12/13/06 as non-positive activity.
 The samples were reprocessed using the unacidified backup H3 fraction and reported on 1/31/07. All 4 results were positive for Ni-63 and per telecon with I.P. personnel the results match the independent lab results.
 Additionally, a Lab Control Spike (LCS) L11959-01, was processed with the RL batch. The bias of this LCS was +4.6% from the known value (QC data package attached).
- 3) Per attached investigation and root cause report, Procedure 201 "Sample Receipt and Chain of Custody Using LIMS" was revised using an Interim Change to include a note for processing of non-routine samples.

B) RM Completing Corrective Actions:

As Amavel, 2/7/07 (Date)

C) Level I CRs Only - VP Review:

N/A, _____ (Date)

PART 5: CLOSEOUT/CANCELLATION

A) Corrective Actions Completed: *J. Dain*, 2/7/07 (Date)
 Laboratory Manager of Designee

B) QA Review (CR Database Updated, Copies Distributed)

E-LAB QA Officer:

C. Shott, 2/7/07 (Date)

BACKGROUND INFORMATION

Condition Report (CR) 07-05 was generated because client samples with Laboratory Sample numbers (LSNs) L11709-09, L11703-10, L11703-11 and L11703-12 showed statistically non positive activity concentrations for Ni-63 analysis. Upon reanalysis, based on client request, all samples showed statistically positive data.

RESULTS & DISCUSSION

A total of 15 water samples were received on November 27, 2006. These samples were assigned Laboratory Sample Numbers L11703-01 through L11703-15. Eleven of these samples (L11703-01 through -08 and L11703-13 through -15) were 1 gallon in size. The remaining four samples (L11703-09 through -12) were only 1 liter.

The sample submission forms provided with the samples indicated that the samples were to be analyzed for gamma emitting radionuclides, tritium, and Sr-90. The client requested that all 15 samples be analyzed for Ni-63 as well via telecom and the additional analysis was added to the Laboratory Information Management System.

The Sample Receipt chemist removed the small H-3 fraction and prepared a 1-liter fraction for gamma spectroscopy analysis. The eleven 1-gallon samples contained sufficient volume to permit the independent analysis of Sr-90 and Ni-63 using ~1800 grams and 550 grams, respectively. The four 1-liter samples were required to be processed sequentially for gamma, Sr-90, and Ni-63 due to the small volume and requested detection limits.

Generally, if Sr-90 and Ni-63 analyses were to be done sequentially on the sample, the Sr-85 tracer and calibrated Ni carrier would have been added to the samples followed by the addition of 1.5M sodium carbonate solution and ammonium hydroxide. The solution containing Sr in the precipitate and Ni in the supernate would be centrifuged. The precipitate of Sr would then be further purified for Sr-89,90 analysis while the supernate containing Ni would have gone through further Ni purification for Ni-63 analysis.

Due to a failure of the Radiochemistry Supervisor and processing chemist to clearly communicate the need for sequential processing, the four 1-liter samples (L11703-09 through L11703-12) were processed using the routine Sr-90 analysis technique. When only Sr-90 analysis is requested, solid Na_2CO_3 is added to precipitate SrCO_3 .

Based upon literature reviews, it is evident that the Ni-63 in the original aliquot processed had co-precipitated as the carbonate along with Sr-carbonate. Carbonate solutions in the presence of ammonium ions complex Ni and this was the premise used in trying to recover Ni-63 from the sample supernate. The Sr Chemist did not add any ammonium hydroxide because the Sr procedure requires the addition of ONLY solid sodium carbonate to precipitate Sr-carbonate.

Entergy-Indian Point requested that the four samples be reprocessed for Ni-63. The Ni-63 re-analysis for samples with LSN L11703-09RP, L11703-10RP, L11703-11RP, and L11703-12RP was performed from the unacidified H-3 fractions. A Laboratory Control Standard (LCS) was processed along with the four reprocess samples. The reported Ni-

63 analytical result for the LCS of 220.2pCi/L had a bias of +4.6% % from the known value of 210.6pCi/L. The client indicated that the reprocessed Ni-63 results for the samples with LSN L11703-09RP, L11703-10RP, L11703-11RP, and L11703-12RP agreed with the independent laboratory results..

CONCLUSION

The Ni-63 results for all other Entergy-Indian Point waters are not affected by this incident. All samples, except the four in question, were analyzed using separate fractions (sequential analysis of Sr-90 and Ni-63 was not required) since sufficient sample volume was provided for gamma spectrometry, Sr-90, and Ni-63 analyses.

When smaller than normal sample volumes are received, it is routine for the Radiochemistry Supervisor, Measurements Supervisor and Laboratory Manager to discuss the situation and determine if requested minimum detection limits can be achieved. This information is then transmitted to the appropriate staff members. For this set of samples, the need to process four of the fifteen samples, L11703-09 through -12, sequentially for Sr-90 and Ni-63 was not communicated effectively or in a timely fashion.

In order to prevent future recurrence of this problem, E-Lab Procedure 201, "Sample Receipt and Chain of Custody using LIMS", has been modified to require that management reviews the receipt of **ALL** non-REMP environmental samples prior to acceptance for analysis. If a sample requires that more than one analysis be performed sequentially, the sample will be clearly labeled to inform the chemist of this requirement. This will ensure that all samples are processed appropriately.



Environmental Laboratory Analysis Report

29 Research Drive
Westboro, MA 01581
508-573-6650

Customer

Entergy Operations Inc
Indian Point Energy Center
450 Broadway, Suite 1, PO Box 249
Buchanan, NY 10511-0249
Attn: Pat Donahue

Product NI-63

Report Date 12/12/06
Receipt Date 11/27/06

| LSN | Client ID & Description | Reference Date | Analysis Date | Nuclide | Activity Concentration +/- 1-Sigma (pCi/L) | TPU 1 Sigma (pCi/L) | Measured MDC (pCi/L) | Required MDC (pCi/L) | Reporting Flags | Level Ratio |
|--------------|-------------------------|----------------|---------------|---------|--|---------------------|----------------------|----------------------|-----------------|-------------|
| <u>Water</u> | | | | | | | | | | |
| L11703-01 | MW-39-200-(005) | 11/17/2006 | 12/10/2006 | Ni-63 | -2.1E+00 +/- 2.1E+00 | 2.1E+00 | 7.2E+00 | 3.0E+01 | | |
| L11703-03 | MW-49-42-(008) | 11/15/2006 | 12/10/2006 | Ni-63 | 2.6E+00 +/- 1.9E+00 | 1.9E+00 | 6.2E+00 | 3.0E+01 | | |
| L11703-04 | MW-49-65-(008) | 11/15/2006 | 12/10/2006 | Ni-63 | -1.8E+00 +/- 2.3E+00 | 2.3E+00 | 7.7E+00 | 3.0E+01 | | |
| L11703-05 | MW-50-42-(008) | 11/15/2006 | 12/10/2006 | Ni-63 | -1.2E+00 +/- 2.0E+00 | 2.0E+00 | 6.7E+00 | 3.0E+01 | | |
| L11703-06 | MW-50-67-(008) | 11/15/2006 | 12/10/2006 | Ni-63 | 1.5E+00 +/- 1.9E+00 | 1.9E+00 | 6.3E+00 | 3.0E+01 | | |
| L11703-07 | MW-58-28-(001) | 11/16/2006 | 12/10/2006 | Ni-63 | -3.2E+00 +/- 1.9E+00 | 1.9E+00 | 6.4E+00 | 3.0E+01 | | |
| L11703-08 | MW-58-65-(001) | 11/16/2006 | 12/10/2006 | Ni-63 | -3.4E+00 +/- 2.3E+00 | 2.3E+00 | 7.8E+00 | 3.0E+01 | | |
| L11703-13 | MW-59-31-(001) | 11/16/2006 | 12/10/2006 | Ni-63 | -2.8E+00 +/- 2.2E+00 | 2.2E+00 | 7.5E+00 | 3.0E+01 | | |
| L11703-14 | MW-59-45-(001) | 11/16/2006 | 12/10/2006 | Ni-63 | -1.6E+00 +/- 1.9E+00 | 1.9E+00 | 6.6E+00 | 3.0E+01 | | |
| L11703-15 | MW-59-68-(001) | 11/16/2006 | 12/10/2006 | Ni-63 | -2.3E+00 +/- 1.9E+00 | 1.9E+00 | 6.4E+00 | 3.0E+01 | | |

- Flags:
- a The measured MDC is greater than the required MDC.
 - b The activity concentration is greater than three times its one sigma counting uncertainty.

Ni-63 analysis was requested by client on November 30, 2006

- c: Barrie Gorman
- Dave Baker
- Michele DuBois

Approved by
E. M. Moreno 12/12/06
E. M. Moreno
Sample Control & Measurements Supervisor

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Environmental Laboratory Analysis Report

29 Research Drive
Westboro, MA 01581
508-573-6650

Customer

Entergy Operations Inc
Indian Point Energy Center
450 Broadway, Suite 1, PO Box 249
Buchanan, NY 10511-0249
Attn: Pat Donahue

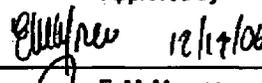
Product NI-63

Report Date 12/14/06
Receipt Date 11/27/06

| LSN | Client ID & Description | Reference | Analysis | Nuclide | Activity Concentration | | TPU | Measured | Required | Reporting Flags Level Ratio | |
|--------------|-------------------------|------------|------------|---------|------------------------|-------------|---------|----------|----------|--------------------------------|--|
| | | Date | Date | | +/- | 1-Sigma | 1 Sigma | MDC | MDC | | |
| | | | | | (pCi/L) | (pCi/L) | (pCi/L) | (pCi/L) | (pCi/L) | | |
| <u>Water</u> | | | | | | | | | | | |
| L11703-02 | MW-49-25-(008) | 11/15/2006 | 12/13/2006 | NI-63 | 8.4E+00 | +/- 6.8E+00 | 6.8E+00 | 2.2E+01 | 3.0E+01 | | |

- Flags:
- a The measured MDC is greater than the required MDC.
 - b The activity concentration is greater than three times its one sigma counting uncertainty.

c: Barie Gorman
Dave Baker
Michele DuBois

Approved by

E. M. Moreno
Sample Control & Measurements Supervisor

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Environmental Laboratory Analysis Report

29 Research Drive
Westboro, MA 01581
508-573-6650

Customer

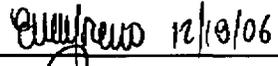
Entergy Operations Inc
Indian Point Energy Center
450 Broadway, Suite 1, PO Box 249
Buchanan, NY 10511-0249
Attn: Pat Donahue

Product NI-63**Report Date** 12/15/06
Receipt Date 11/27/06

| LSN | Client ID & Description | Reference Date | Analysis Date | Nuclide | Activity Concentration | | TPU 1 Sigma (pCi/L) | Measured MDC (pCi/L) | Required MDC (pCi/L) | Reporting Flags Level Ratio |
|--------------|-------------------------|----------------|---------------|---------|------------------------|-------------|---------------------------|----------------------------|----------------------------|--------------------------------|
| | | | | | +/- | 1-Sigma | | | | |
| Water | | | | | | | | | | |
| L11703-09 | MW-42-41-(004) | 11/17/2006 | 12/13/2006 | Ni-63 | 1.9E+00 | +/- 1.9E+00 | 1.9E+00 | 6.4E+00 | 3.0E+01 | |
| L11703-10 | MW-42-43-(004) | 11/16/2006 | 12/13/2006 | Ni-63 | 2.9E+00 | +/- 1.7E+00 | 1.7E+00 | 5.5E+00 | 3.0E+01 | |
| L11703-11 | MW-42-46-(004) | 11/16/2006 | 12/13/2006 | Ni-63 | 2.9E+00 | +/- 1.8E+00 | 1.9E+00 | 6.1E+00 | 3.0E+01 | |
| L11703-12 | MW-42-48-(004) | 11/16/2006 | 12/13/2006 | Ni-63 | 2E-01 | +/- 1.4E+00 | 1.4E+00 | 4.7E+00 | 3.0E+01 | |

- Flags:**
- a The measured MDC is greater than the required MDC.
 - b The activity concentration is greater than three times its one sigma counting uncertainty.

- c: Barrie Gorman
Dave Baker
Michele DuBois

Approved by

E. M. Moreno
Sample Control & Measurements Supervisor

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Environmental Laboratory Analysis Report

29 Research Drive
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508-573-6650

Customer

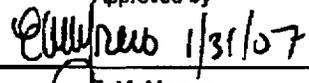
Entergy Operations Inc
Indian Point Energy Center
450 Broadway, Suite 1, PO Box 249
Buchanan, NY 10511-0249
Attn: Pat Donahue

Product NI-63

Report Date 01/31/07
Receipt Date 11/27/06

| LSN | Client ID & Description | Reference Date | Analysis Date | Nuclide | Activity Concentration | | TPU 1 Sigma (pCi/L) | Measured MDC (pCi/L) | Required MDC (pCi/L) | Reporting Flags | Level Ratio |
|--------------|-------------------------|----------------|---------------|---------|------------------------|-------------|---------------------------|----------------------------|----------------------------|--------------------|-------------|
| | | | | | +/- | 1-Sigma | | | | | |
| Water | | | | | | | | | | | |
| L11703-09 | MW-42-41-(004) | 11/17/2006 | 01/27/2007 | Ni-63 | 1.31E+02 | +/- 2.0E+01 | 2.3E+01 | 6.6E+01 | 3.0E+01 | ab | # |
| L11703-10 | MW-42-43-(004) | 11/16/2006 | 01/28/2007 | Ni-63 | 2.28E+02 | +/- 2.1E+01 | 2.8E+01 | 6.6E+01 | 3.0E+01 | ab | # |
| L11703-11 | MW-42-46-(004) | 11/16/2006 | 01/28/2007 | Ni-63 | 2.49E+02 | +/- 1.9E+01 | 2.7E+01 | 5.9E+01 | 3.0E+01 | ab | # |
| L11703-12 | MW-42-48-(004) | 11/16/2006 | 01/29/2007 | Ni-63 | 2.07E+02 | +/- 1.7E+01 | 2.4E+01 | 5.4E+01 | 3.0E+01 | ab | # |

Flags:
 a The measured MDC is greater than the required MDC.
 b The activity concentration is greater than three times its one sigma counting uncertainty.

Approved by

 E. M. Moreno
 Sample Control & Measurements Supervisor

c: Crystal Boucher
 Dave Baker
 Michele DuBois
 Pete Hollenbeck

NI-63 was re-processed using a small aliquot remaining from the unacidified H-3 fraction. Entire acidified sample was previously consumed to perform requested gamma and Ni-63/Sr-90 sequential analyses (limited sample size of 1000 mL was provided by client). An internal Condition Report will be issued to address discrepancy between previous NI-63 sequential analysis result with re-processed "stand-alone" Ni-63 analysis result.

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AREVA NP ENVIRONMENTAL LABORATORY SAMPLE SUBMISSION FORM
(ENVIRONMENTAL/BIOASSAY SAMPLES)

T24.16

Client Name: ELAB
 Client Purchase Order/Contract: _____
 Date of Shipment: _____
 Program: REMP Non-REMP
 Requested Turnaround Time: Standard Rush (COST MULTIPLIER MAY APPLY)
 Working Days (SPECIFY NUMBER) _____

Name of Client Representative: Chris Shelton
 (Person(s) who should receive the results)
 E-Mail: _____
 Phone: () _____ Fax: () _____

| WEEK NO | YEAR | LSN | Sample Type | Station and Description | Collection Period (All Dates/Times Must Reflect EST or EDT) Please check box below to indicate the date to decay results | | | | | | | | | | | | Total Sample Amount Shipped (Specify Units) | Radiological Analyses (Check All That Apply) | | | | | | | | | | | | | Quarterly Composite | | | | | | | | | | | | |
|---------|------|-----------|-------------|-------------------------|--|-----|------|------|---|-----|------|-------|---|-----|------|------|--|--|------------|-------------------|-------|-------|-------|-----|---------------------|------|-------|-----------------------|--------|----------------|---------------------|------------------|------------------|----------|-------|-----------------|-------------|-----------------|-------------------------------------|-------|------------|---------|-----------------|
| | | | | | Start Date (If Applicable) <input type="checkbox"/> | | | | Stop Date (Required) <input type="checkbox"/> | | | | Mid-point Date (If Applicable) <input type="checkbox"/> | | | | | Gross Alpha | Gross Beta | I-131 in Charcoal | Ne-63 | Fe-55 | Gamma | H-3 | I-13 (LL) (Specify) | P-32 | Ct-14 | Am-241 Cm-242 243/244 | Np-237 | Pu-238 239/240 | Pu-241 | Re-226 (Specify) | Rs-228 (Specify) | Sr-89 90 | Tc-99 | U-234, 235, 238 | Th-230, 232 | Other (Specify) | Sample belongs to Quarter (1,2,3,4) | O H-3 | Q Sr-89 90 | Q Gamma | Other (Specify) |
| | | | | | Month | Day | Year | Hour | Month | Day | Year | Hour | Month | Day | Year | Hour | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 04 | 07 | L11959-01 | Water | LCS (IP RP) | | | | | 01 | 26 | 07 | 07:00 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

| CHAIN OF CUSTODY | | FIELD TREATMENTS/COMMENTS | ELAB ACCEPTANCE STAMP |
|-------------------------------------|----------------------------|---------------------------------|-----------------------|
| Collected By: | Phone Number: () | * 500.18g total solution weight | |
| Relinquished By: | Date/Time: | | |
| Received By: <u>GA</u> | Date/Time: <u>11/26/07</u> | | |
| ELAB Comments: <u>L11959</u> | | | |

CR07-05
log 9.5.34

CERTIFICATE OF CONTENT (PAGE 1/2)

Nuclide: Ni-63
 Nuclide ID: S43081-A
 Licensed/Exempt: Exempt
 Reference date of Secondary @T0: 09/01/1994 *07:00 ET*
 Reference Concentration (C0): (1.208)E-3 ±2.40% uCi/gm
 Date of Preparation of Sample (T1): 01/26/2007 *07:00 ET*
 Elapsed Time (Delta T): 12.40 YEARS
 Half Life (t1/2): 100.1000 Year(s)
 Decay Correction (DC): 0.9177
 Concentration @T1 (Ct): (1109 ± 27)E-06 uCi/gm
 Source Supplier/Media: Amersham/DI H2O
 Last Date Of Verification: 05/22/2006
 Final Matrix Media Type: IP Lab ctrl spk prep
 Preparer's Name: KRM

Weight of Secondary Reference Standard

Mettler AE163 Analytical
balance S.N.: F33394

a) Container+reference standard weight: 6.82962 gm
 b) Container weight: 6.73459 gm
 c) Net reference standard weight (Wr): 0.09503 gm

Preservatives added:

HCl (12M) 10.0 mls
 NAHSO3 13.02 mg

Ni-63 LCS 1/26/07
NAHSO3
6.82962 g
6.73459 g
597081-A

Total Weight of Source Matrix

Sartorius U5000D
balance S.N.: 3608009

a) Container + source matrix + preservative +
Reference Standard Weight: 0.54413 kg
 b) Container weight: 0.04395 kg
 c) Net source matrix weight (Ws): 0.50018 kg

Estimated Uncertainty (in %) of Radioactivity Source Matrix (@ 1-Sigma)

Source Standard: 2.40
 Pipetting: 0.00
 Weighing Error: 1.41
 Internal Lab Standardization: 0.00
 Other: 0.00
 Total: 3.81 %
 Uncertainty Conversion: 0.0278

CERTIFICATE OF CONTENT (PAGE 2/2)

Total Activity of Secondary Reference Standard Added

At0 = Wr * CO = (1148 ± 32)E-01 pCi Act @ Ref. Date (t0)
At1 = Wr * CO * DC = (1053 ± 29)E-01 pCi Act @ Prep. Date (t1)

=====
Final Source Matrix Concentration

Cm = At0/(Ws) = (2295 ± 64)E-01 pCi/kg Conc. @ (t0)
Cm = At1/(Ws) = (2106 ± 59)E-01 pCi/kg Conc. @ (t1)

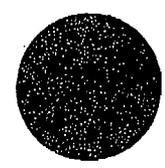
=====
Special Notes/Directions

A lab control spike was requested by Entergy IP to accompany a reprocess for a Ni-63 analysis in water. Approximately 500 mls of DI water preserved with HCl and sodium bisulfite was spiked with Ni-63 from an exempt secondary standard. Source solution was delivered to the chemist in a polypropylene bottle.

=====
ID of Source Just Prepared: E012607


PREPARER'S SIGNATURE


DATE



SECONDARY SOURCE WORKSHEET

Secondary Source Number: S43081-A Prepared by: KRM

Reference Date of Secondary: 09/01/1994 07:00 EST On: July 22, 1999

Primary Standard Supplier: AMERSHAM

Primary Standard Certificate Number: S4/30/81

Reference Date of Primary Standard: 09/01/1994 07:00 EST

| | | |
|--------------------------------------|-------------------------------|------------------|
| Weight of Primary Standard Utilized: | Final Weight of 1° Standard | <u>4.24704</u> g |
| | Initial Weight of 1° Standard | <u>4.34730</u> g |
| | Total Weight of 1° Added | <u>0.10026</u> g |

| | | |
|--------------------------------------|-----------------------|-----------------|
| Weight of Secondary Standard (S.S.): | Diluent and container | <u>135.63</u> g |
| | Weight of container | <u>35.76</u> g |
| | Total weight of S.S. | <u>99.87</u> g |

Diluent used: 0.1 M HCl Precision Pipet: N/A Balance No.: Mettler AE163 SN F33394

Quantitative Transfer Verified
Yes No

| Nuclide | Half Life | Original Activity | Decay Correction | Prepared Activity per Gram of S.S. | Remarks |
|---------|-----------|---------------------|------------------|------------------------------------|--|
| Ni-63 | 100.1 y | 1.245 µCi/g ± 1.94% | 1.00 | 1.208E-03 µCi/g ± 2.4% | Verified to w/in -0.77% on LKB#1 on 04/17/2003 |
| | | | | | 3/10/04 verified via measurements a LKB#5 to w/in ±1.77% ? |
| | | | | | 3/24/05 verified via measurements LKB#2 to w/in ±1.99% 3.1706AB |
| | | | | | 5-22-05 verified via measurements 3.1706AB LKB#2 to w/in ±1.49% |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

RADIOACTIVE SOURCE MATRIX REQUEST

Nuclide: Ni-63
Nuclide ID: S43081-A
Licensed/Exempt: Exempt
Reference date of 2° @T0: 09/01/1994 07:00 EST
Reference Concentration (CO): (1.208)E-3 ±2.40% uCi/gm
Date of Preparation of Sample (T1): 01/26/2007 07:00 EST
Elapsed Time (Delta T): 12.40 YEARS
Half Life (t1/2): 100.1000 Year(s)
Decay Correction (DC): 0.9177
Concentration @T1 (Ct): (1109 ± 27)E-06 uCi/gm
Source Type: LCS IP
Last Date Of Verification: 05/22/2006
Media Type: Pres DI H2O
Preparer's Name: KRM

=====

Desired Values

Desired Activity/Concentration @T1: (2.0000)E 2 pCi/kg
Desired End-Product Weight/Volume: 1 kg

=====

Estimated Values

Final Source Matrix Required: 0.0902 gm Required in 1 kg


PREPARER'S SIGNATURE

1/26/2007
DATE



Environmental Laboratory Analysis Report

29 Research Drive
Westboro, MA 01581
508-573-6650

Customer
AREVA NP
29 Research Drive

DESEL

Product: NI-63

Report Date 02/07/07

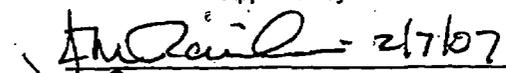
Receipt Date 01/26/07

Westborough, MA 01581
Attn: Chris Shelton

| LSN | Client ID & Description | Reference Date | Analysis Date | Nuclide | Units | Activity Concentration | | TPU 1 Sigma | Measured MDC | Required MDC | Reporting Flags | Level Ratio |
|----------------------|-------------------------|----------------|---------------|---------|---------|------------------------|-------------|----------------|-----------------|-----------------|--------------------|-------------|
| | | | | | | +/- | 1-Sigma | | | | | |
| Control Spike | | | | | | | | | | | | |
| L11959-01 | ID#LCS (IPRP) | 01/26/2007 | 01/30/2007 | NI-63 | (pCi/L) | 2.202E+02 | +/- 2.4E+00 | 1.8E+01 | 4.0E+00 | 6.0E+01 | b | |

- Flags:
- a The measured MDC is greater than the required MDC.
 - b The activity concentration is greater than three times its one sigma counting uncertainty.

Approved by


for E. M. Moreno
Sample Control & Measurements Supervisor

CE 07-05
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INTRALABORATORY CROSS CHECK ANALYSIS SHEET

SAMPLE TYPE: WATER

ISSUANCE DATE: 02/07/2007

REF. DATE: 01/26/2007

LAB SAMPLE NO: 1195901

ANAL DATE: 01/30/2007

UNITS: pCi/L

| NUCLIDE | RESULT 1 | RESULT 2 | RESULT 3 | MEAN | KNOWN VALUE | % DIFF.1 | % DIFF.2 | % DIFF.3 |
|---------|------------------|----------|----------|------|-------------|----------|----------|----------|
| Ni-63 | (2202 ± 24)E-01 | | | | 21.06E 01 | 4.60 | | |

ALL RESULTS PASSED QA PERFORMANCE CRITERIA EXCEPT THOSE NOTED WITH AN ASTERISK

CR 07-05
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AREVA NP
INTERIM MANUAL AND PROCEDURE CHANGE FORM

| | | | |
|------------------|------------|------------------|----------------|
| Prepared By | <u>CO</u> | Procedure No. | <u>201</u> |
| LIMS Coordinator | <u>N/A</u> | Revision No. | <u>10</u> |
| Reviewed By | <u>JMR</u> | Approval Date | <u>2/07/07</u> |
| QA Officer | <u>CO</u> | Effective Date | <u>2/07/07</u> |
| E-LAB Manager | <u>JMR</u> | Next Review Date | <u>5/06/07</u> |

Revised Manual/Procedure Due Date 05/06/07
Cognizant Staff Person CAS

MANUAL/PROCEDURE SECTION

Change:

Add the following Note after step PROCEDURE.C.5:

NOTE: Samples received that are not part of routine REMP processes require management attention upon receipt to ensure that sufficient volume is available to achieve the contractual MDCs. Laboratory management shall ensure discrepancies are resolved with the client and that Laboratory staff are informed of any special processing requirements (e.g., sequential analyses or split volumes).

| | | | |
|------------------|------------|------------------|---------------|
| Prepared By | <u>CJS</u> | Procedure No. | <u>201</u> |
| LIMS Coordinator | <u>DM</u> | Revision No. | <u>10</u> |
| Reviewed By | <u>EMM</u> | Approval Date | <u>7/8/06</u> |
| QA Officer | <u>CB</u> | Effective Date | <u>7/8/06</u> |
| E-LAB Manager | <u>JWR</u> | Next Review Date | <u>7/8/11</u> |

SAMPLE RECEIPT AND CHAIN OF CUSTODY USING LIMS

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PRINCIPLE OF METHOD

Samples received by the AREVA NP Environmental Laboratory (E-LAB) generally fall into two major categories:

1. Environmental (Routine Radiological Environmental Monitoring Program [REMP] and non-routine/site remediation) and Bioassay samples
2. 10 CFR Part 50/61

A sample can only be processed by the Laboratory after the Sample Receipt Staff verifies the sample's compliance with laboratory standards:

1. sample integrity
2. exposure rates
3. completeness and correctness of the sample submission form (SSF)

Client contract requirements should be entered into the Laboratory Information Management System (LIMS) before any sample data is entered. This ensures that work is not performed without an approved client contract. Exceptions to this rule are permitted with management approval provided that final reports are not issued prior to E-Lab review of the contract.

All samples, except for Part 50/61 samples, accepted for processing are entered into LIMS in batch mode. Each sample is assigned a Laboratory Sample Number (LSN) sequentially (NOTE 1). The initial LSN is L2001-01. Each LIMS LSN corresponds to a unique sample on the SSF (Reference 1). The LSN is automatically assigned by LIMS through the login process. The LSN is used to track samples throughout the designated Laboratory processing areas (NOTE 1). Process chains and reports are maintained by LIMS to document sample chain-of-custody (COC).

This procedure outlines the methods by which Sample Receipt Staff receive, inspect, track, and label all samples accepted for analysis. All personnel required to perform the function of sample receipt must be trained to monitor for sample radioactivity and/or contamination. The general sample COC requirements for the Laboratory processing staff are also described in this

procedure.

REFERENCES

1. E-LAB Procedure 605, "Sample Receipt and Reporting: Sample Submission Form Records"
2. E-LAB Safety Manual 160
3. E-LAB Procedure 850, "Receipt and Storage of Radioactive Samples"
4. E-LAB Procedure 200, "Sample Receipt and General Chain of Custody"
5. "Radiological Assessment Protocol For Internal Radionuclide Deposition," M. J. Scannell and A. D. Banavali, EL 605/94, dated December 16, 1994.
6. E-LAB Procedure 110, "Suggested Sample Collection Procedures for Environmental Media"
7. E-LAB Procedure 120, "Sample Storage and Accountability"
8. E-LAB Procedure 692, "Report Generation Using LIMS"
9. E-LAB Procedure 685, "Data File Transfer Between LIMS and Analytical Software, Using Outer-LIMS"
10. E-LAB Procedure 688, "Environmental and Bioassay Data Entry Using LIMS"
11. E-LAB Manual 100, "Laboratory Quality Assurance Plan"

APPARATUS

1. E-LAB Sample Submission Forms, FORM 605.1 through 605.3 (Reference 1).

PRECAUTIONS

1. Care must be exercised when entering sample data into LIMS. Sample log-in is the only point in the chain where certain information is entered. Any error at this point will be transferred along the entire processing chain.
2. In order to enter a sample into LIMS, a template must exist. If a template does not exist, contact the LIMS database manager immediately. Only the LIMS Database Manager has the authority to add/modify/delete templates.
3. Upon entering the sample into LIMS, ensure that a price is listed for the sample. If no price is specified, contact the LIMS database manager immediately.
4. If any errors occur during sample data entry, contact the LIMS database manager immediately.
5. Care must be exercised not to mislabel or cross contaminate samples during handling. Adhere to all safety/PPE precautions outlined in Reference 2.

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6. Ensure that the required radiation survey instrumentation is within calibration. A one-point response check must be performed on a daily or prior-to-use basis. When performing the one-point response check, ensure that radioactive material or potentially contaminated samples are not in the vicinity (Reference 3).
7. Acids/Bases used as preservatives are corrosive and constitute a health and safety hazard. If preservatives are added during sample receipt, wear gloves, safety glasses and laboratory coats to minimize skin contact.
8. If damaged shipping containers known to contain radioactive materials are received, contact the Radiation Safety Officer or his/her designated alternate immediately.
9. Samples that are determined to be unacceptable for processing by management for any reason shall have the deficiency documented on FORM 201.1. This includes samples listed on shipping papers but not found in the shipment. These samples shall be assigned an LSN. The final disposition of the sample shall also be documented in LIMS. Name of individual contacted, shipping details, etc. shall be included in the documentation.
10. Any sample placed "ON HOLD" which is labeled "radioactive" or known to be spiked must be stored in a locked cabinet. In addition, any radioactive or spiked sample remaining in Sample Receipt overnight must be stored in a locked cabinet.
11. If the client is on the posted NELAP list, each individual sample container **MUST** be assigned a separate LSN. Consult with the Sample Control and Measurements Supervisor if multiple containers with identical IDs are received.

PROCEDURE

A. Initial Receipt

1. Carriers delivering samples to the Laboratory should be directed to the rear entrance of the Laboratory.
2. Notify the Radiation Safety staff upon receipt of any package containing radioactive material.
3. Inspect the package for any damage incurred during shipment.
4. Ensure that the daily one-point response check of the radiation survey instrument(s) used for survey/monitoring of samples has been completed.
5. PPE shall be utilized when receiving all samples (see precautions above).
6. Initiate FORM 201.1 for each shipment.

B. Surveying/Monitoring of Samples

1. For each environmental and bioassay sample not marked as "radioactive", scan the package/sample at 2-5 cm per second using a beta/gamma survey instrument. If a repeatable and sustained count rate equal to or greater than the Minimum Detectable Count Rate (MDCR) value listed on the portable instrument is obtained above background (e.g., 50 cpm in a 70 cpm background), contact

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the radiation safety staff. If the positive count rate is not due to radon progeny, the package/sample and all subsequent aliquots shall be labeled as radioactive. The RSO and laboratory management shall determine whether the sample is processed in the environmental area and whether notification is necessary to the cognizant client submitting the sample(s). For any sample found to be >MDCR, note the count rate above background for documentation as per Section C.

2. For each 10 CFR Part 50/61 sample or any sample shipped as limited quantity or DOT labeled (white-I, yellow-II, or yellow-III) refer to Reference 3. 10 CFR Part 50/61 samples and samples marked as "radioactive" are routinely processed in the 10 CFR Part 50/61 area. The RSO and laboratory management shall determine whether a Part 50/61 sample or a sample marked as "radioactive" may be processed in the environmental area.
3. All samples processed in the 10 CFR Part 50/61 processing area are considered radioactive and do not need to be labeled as such. If any Part 50/61 sample or sample fraction is brought into a environmental/bioassay processing area, it must be transferred in a container labeled "radioactive".

C. Acceptance and Completeness of a Sample Submission Form

1. A Sample Submission Form (SSF) (Reference 1) should accompany each sample received by the Laboratory for analysis (NOTE 1). All quality control (QC) samples (e.g., "spiked" internal samples prepared by the Standards Laboratory staff, samples received as part of an external QC program, internal analytical blanks, detector backgrounds, etc.) must also be submitted with a completed SSF.
2. If a client does not utilize a SSF, the information provided with their sample shipment (i.e., shipping paperwork, sample lists, "Vendor Service Request" list, etc.) must be cross-referenced with their samples prior to completing an SSF. This will ensure that a Laboratory Sample Number (LSN) is not assigned to a sample before the sample shipment is receipt inspected. If any discrepancies are noted, such as missing sample information (i.e., incorrect or the lack of a sample reference date, etc.), the cognizant client shall be contacted immediately.

NOTE: If a chain-of-custody (COC) form accompanies a shipment of samples, note the following requirements

- a. Do not sign the COC form for a customer unless you are familiar with the specific requirements of the customer's procedure.
 - b. Do not sign a COC form for a customer until an inventory of all of the materials has been performed.
 - c. If sample receipt and inventory cannot be performed at the time of delivery, the COC form will not be signed until the inventory has been completed.
- (1) If a courier wants an immediate acknowledgment that E-LAB has received the shipment, sign on the COC form the date and time

the shipment was received. Also note that the contents were not verified.

- (2) The COC form can be signed once the shipment contents are verified to contain all samples listed on the COC form.
 - (3) If a client utilizes a SSF, the information provided in the SSF shall be cross-referenced with the information provided on the sample containers in order to ensure proper sample chain-of-custody. If any disagreements are noted, such as missing sample information (i.e., incorrect or lack of a sample reference date, etc.), the cognizant client shall be contacted immediately.
3. If a sample cannot be located within a shipment, a supervisory review of the sample shipment will be initiated. After laboratory supervision confirms a "missing" sample, the client will be contacted immediately and the entire shipment may be put on "hold" (e.g., no processing will take place). Processing may be suspended until the client has provided clear instructions to continue processing. Management will determine if processing may start prior to receiving all sample data. Once cleared for processing, the SSF should be generated for those clients who did not submit a SSF. The turnaround time clock starts at this point, not the receipt date.

NOTE: The paperwork received with samples that are placed "On Hold" or cannot be passed into chemistry or counting within 24 hours of receipt must be placed in the "On Hold" bin. These samples must be placed in the appropriate "On Hold" area of Sample Receipt.

4. Sample documentation must include a reference date and time for each sample. If the client is on the posted NELAP list, collection date/time and collector's name MUST be included in the documentation. Clients may pre-arrange reference times for certain samples (ONLY for non-NELAP clients); however, contract files must contain documentation of the client's approval of this agreement.
5. The minimum sample weights/volumes necessary to achieve the required Minimum Detectable Concentrations (MDCs) for environmental and bioassay samples are described for Laboratory clients in References 5 and 6. Laboratory staff shall contact the cognizant client to resolve any discrepancies.
6. The recommended preservation methods for environmental, bioassay and 10 CFR Part 50/61 samples are described for Laboratory clients in References 5 and 6. Client personnel should note all preservatives added to samples on the SSF.
7. All bioassay and environmental milk samples not preserved by client personnel should be refrigerated immediately upon acceptance (NOTE 2). If any milk sample appears to be curdled, notify Laboratory supervision and note accordingly on the SSF.

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8. All water samples not preserved by client personnel should be preserved according to Reference 5 or 6 upon acceptance.
9. Samples shall be verified to be delivered in appropriate types of containers and have met required EPA holding times (NOTE 3). Laboratory management shall be notified of any deviations and the sample(s) is to be identified as unacceptable with noted deficiencies.
10. Attach all client-supplied documentation (including the air bill if delivered by courier) to the SSF.
11. Once the SSF information is deemed accurate and complete, the SSF shall be stamped for "acceptance" with the date/time by the Sample Receipt Staff. Record the LSN and the count rate above background for any sample that measured >MDCR in Section B. Internal QC samples do not require a stamped SSF.
12. Initial the "Received by" portion of the SSF.
13. Complete FORM 201.1 and attach to the SSF.

D. Assignment of a Laboratory Sample Number for 10 CFR Part 50\61 Samples
Please see Reference 4.

E. Environmental Sample Log-In

1. Load and log in to LV Environmental (SeedPak)
2. Select the **Sample Management/Login** pull-down menu from the main menu.
3. Select Copy/Copy Template.
4. Select the Template ID using the pick list on the left-hand tool bar.

NOTE: In order to enter a sample into LIMS, a template must exist. Do not try to enter a sample without a template. Contact the LIMS database manager immediately.

5. Verify that the Target Type radio button is set to "Login" and the pricing radio button is set to "Fixed". If not, set these buttons to these parameters.
6. Enter the receipt date in the following format: DD-MON-YY.
7. Enter the Collection Stop Date/Time in the following format: DD-MON-YY HH-MM (use 24-hour clock). If only a reference date is required, it should be entered here.
8. Select "OK"
9. Record the Login-Id on the SSF
10. Select "OK"

11. Select LOGIN INFO on the top of the form.
 - a. For REMP clients, enter the week number in the week number data field.
 - b. Select **SAVE** then **CANCEL**.
12. Ensure that you have selected the first sample in the batch
13. Enter the collection start and stop date/time in the following format: DD-MON-YY HH:MM for each sample.

NOTE: If the midpoint checkbox (M) is checked, you must enter both the start and stop collection date/times to allow LIMS to calculate the midpoint of collection. These checkboxes are set up based upon client requirements at the time the project is set up in LIMS. Do not edit these fields without management approval.

If the midpoint checkbox (M) is not checked, do NOT enter the collection start date/time for a sample unless the sample matrix is air particulate or charcoal filter. Do NOT enter the collection start date/time for any other media even if the client provides one on the SSF.

14. Edit the TAT field to match the client's request if an expedited turnaround time (TAT) is requested.

NOTE: If no TAT time is specified, a default TAT of 30 days will be applied to the sample.

15. Check the matrix/product/price template data.

NOTE: Ensure that a price is listed for the sample. If any changes are required or if no price is specified, contact the LIMS database manager immediately.

16. Select **SAVE**.
17. Charcoal filter (CF) and air particulate (AP) samples require volumes to be entered during the initial Login by Sample Receipt. Separate work groups must be created for each media, APs and CFs.
 - a. Select the **Sample Management/Workstat** pull-down menu from the LIMS Main Menu.
 - b. To create a work group for APs and CFs, select the "Status Update Only" radio button.
 - c. In the *Query Criteria* section, you must enter information into the following fields: **DEPARTMENT** (1), **NEW STATUS** (NEED) and the **LOGIN** (applicable L#).

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NOTE: The pick list on the left-hand toolbar can be used to guide the selection of the proper parameters for each field.

- d. Select **OK**.
- e. Enter an "X" next to the applicable samples.
- f. **SAVE** then **CANCEL**.
- g. Select the "Schedule Work" radio button.
- h. Enter the **WORK PROFILE** (AP or CF) and select **NEW**. The *Work Group Definition* window will appear.
- i. Select **SAVE** and the Work Group number will appear. Be sure to make note of this number (WG_ _ _ _) on the Sample Submission Form.
- j. Enter the **LOGIN** in the *Query Criteria* section.
- k. Select **OK**.
- l. Enter an "X" next to the applicable samples.
- m. Select **SAVE** then **CANCEL**. Close any remaining open windows.
- n. Select the **Sample Data/Data/Enter Data** pull-down menu from the LIMS Main Menu.
- o. Select **OK** on the *Data Entry Context* screen.
- p. Select **MODES** from the Seedpak Main Menu and click on **ALL SAMPLES** to enter the data for all samples at once.
- q. Click on the **CLOSE** button.
- r. The *Sample Set* screen will appear and should display the samples you intend to add volumes to.
- s. Select **OK**.
- t. In the **NUMVALUE** field, enter the volumes for each sample in the units as they appear on the Sample Submission Form.
- u. Select **SAVE** then **CANCEL** then **CLOSE**. All remaining windows should be closed and the LIMS Main Menu will appear alone.

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NOTE: When the volumes are entered in ft³, be sure to wait a moment before proceeding to the next step. LIMS requires some time to convert the volumes into m³. Failure to allow enough time will result in volumes being left out on the Login COC form and the sample worksheets. If this occurs, simply cancel out of any LIMS windows until the Main Menu stands alone. Allow further time and then proceed. Repeat, if necessary, until all volumes appear.

- v. Repeat Steps E.17.b through E.17.u for charcoals.
18. Generate a Log-in COC Report for each batch of samples entered into LIMS.
- a. Select the Reports pull-down menu from the LIMS main menu.
 - b. Select Log-in, then Log-in COC Report.
 - c. Enter the log-in number and print a copy of the log-in COC report.
19. Verify all data entry on the Log-in COC Report against the SSF.
- a. If all the information is correct, sign and date the Log-In COC Report and proceed to Section F.
 - b. If an error in the date or time is identified, correct the mistake.
 - (1) Select the **Sample Management/Log in** pull-down menu from the SeedPak main menu.
 - (2) Click on the **Log In** radio button and enter the Log in you wish to correct.
 - (3) Make the necessary changes.
 - (4) Select **SAVE**.
 - (5) Reprocess the sample.
 - (a) Select the **Sample Data/Process Queue** pull-down menu from the Seedpak Main Menu.
 - (b) Enter the Process ID(e.g., LOGINREP or appropriate process from the "pick List")
 - (c) Enter the first sample in the LOGIN (e.g., L2134-01).
 - (d) Select the "Query" button and put an "X" next to the sample to be reprocessed.
 - (e) Click on the "Queue" button to submit this process.
 - (f) Close all windows.
 - c. If an error in the sample volume is identified, correct the mistake.

- (1) Rollback the sample and edit the data.
- (2) Run the Process Queue for the batch of samples including this sample.

NOTE: See the DATABASE ADMINISTRATOR if you are unfamiliar with this process.

F. Sample Analysis Coding and Worksheet Preparation

1. Based upon the analyses requested by the SSF, the Sample Receipt staff will gather the appropriate analysis worksheets (NOTE 5).
2. Select the **Reports/Run Reports** pull-down menu from the LIMS main menu.
3. Select the appropriate worksheet.
4. Click **Run Reports** from the Select Reports screen.
5. On the Runtime Parameter Form, set the Destination Type to **Preview** and enter the log-in number.
6. Select **Run Report** and the report **Previewer** screen will appear.
7. Verify that the analysis worksheets have the correct sample information.
8. Click the **Print** button and the specified worksheet will be printed for all samples in the log in for which it applies.
9. Repeat Steps F.2 through F.8 for all other product worksheets to be printed.
10. Select the **Reports** pull-down menu from the SeedPak main menu to print all pressure-sensitive sample labels.

NOTE: The pressure sensitive stickers are required only for gamma spectrometry measurements of environmental, bioassay, air particulate, and charcoal filter samples. Two stickers per sample may be necessary in order to allow for easy labeling of the calibrated counting container that is used for the gamma spectrometry measurement. Only one sticker is required for air particulate and charcoal samples.

11. Select **Run Reports** from the Report pull-down menu and select the appropriate label report.
12. On the Runtime Parameter Form, set the Destination Type to **Preview** and enter the log-in number.
13. Select **Run Report** and the report **preview** screen will appear.
14. Click the **Print** button and the specified labels will be printed for all samples in the log in for which they apply.

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15. Repeat Steps F.10 through F.14 for all other labels to be printed.
16. Staple the Gamma stickers to the appropriate sample analysis worksheets.

NOTE: When a composite analysis of a media type is requested, the sample must be stored until all other samples from the same station within the same composite period are available. All sample containers, with the exception of Air Particulate filter samples, must have a note to indicate to the processing staff that the sample should be retained for a composite analysis. A new LSN with a new corresponding SSF will be completed for the composited samples at the of each quarter.

G. Special Worksheet/Sample Labeling Instructions

1. Place the sample identification label on each sample.
2. Compare the sample label to the SSF to ensure that the sample has been correctly labeled.
3. For those clients which have a Ba-140 gamma-isotopic MDC requirement, a "DELAY DATE" box shall be filled out on the corresponding sample analysis worksheets by the Sample Receipt staff. The date printed on the note corresponds to eight days after the sample reference date (NOTE 4). The eight-day delay is not required for QC samples.
4. If a client requires that the requested analyses be completed prior to the standard turnaround times offered by the Laboratory, a green-colored circular sticker should be added to the analysis worksheet. A "Count by" date and/or "Due Date" will be printed on the analysis worksheets.
5. Certain designated clients' REMP samples require a special sensitivity analysis. The client and matrix specific MDC requirements will be printed at the bottom of the appropriate analysis worksheets. The Sample Control and Measurements Supervisor shall approve any changes to the required MDCs data in LIMS. If the samples require special attention, (as determined by the Supervisor), a red-colored circular sticker should be added to the analysis worksheet.
6. Other instructions or notes which may aid in the processing of samples may be added to the sample analysis worksheets by the Sample Receipt staff (e.g., reporting units, client name, client PO number/work order number, etc.).

NOTE: If any Sample Submission Form block for Quarterly Composites has been checked, attach a sticker to the sample container(s) stating:

"Save for Quarterly Composite – DO NOT DISPOSE"

Air Particulate filter samples, going for Quarterly Gamma Spectrometry, DO NOT require a sticker.

7. Special instructions must be applied for milk and water samples when less than eight liters are received. If the sample is to be processed for I-131(LL), Sr-89,90 or gross beta in addition to gamma-isotopic analysis, the sample must be split. Send one liter into sample prep for the gamma-isotopic analysis. Send the remaining sample into chemistry for I-131(LL), Sr-89,90 and/or gross beta analyses.

H. Sample Receipt Chain-of Custody

1. Environmental or Bioassay Samples

- a. Sample chain-of-custody (COC) is maintained in LIMS through the use of process chains. Reports can be prepared to verify the COC for any sample (Reference 8).

- (1) Select the **Reports** pull-down menu from the SeedPak main menu.
- (2) Select **Status** from the Reports pull-down menu, then **Sample Status**.
- (3) Click **OK** and print the report.

- b. The location of a sample and/or a particular fraction going for a singular analysis can be determined using LIMS.

- (1) Select the **Sample Management** pull-down menu from the main menu.
- (2) Select **Status**.
- (3) On the Status Query Criteria screen, enter the Log in and click **OK**.
- (4) Select the sample to be checked and click **OK**.
- (5) The status for each analysis will be displayed.

2. 10 CFR Part 50/61 Samples

Please see Reference 4.

I. Generate Sample Acknowledgment Letters and/or Sample Acknowledgment PDF Files

A Sample Acknowledgment Letter is generated for each batch of samples received. This report can be provided as a hard-copy report or a PDF file. This report serves as confirmation of the receipt of client samples. To generate a hard-copy of the report, the following steps are followed:

1. Select the **Reports** pull-down menu from the LIMS main menu.
2. Select **Run Reports** from the Report main menu.

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3. Select the **ACK LETTER** and click **Run Report**.
4. Set the Destination Type to **Preview** on the Run Time Parameter Form.
5. Enter the Log in number on the Run Time Parameter Form and click **Run Report**.
6. Review the Ack Letter as displayed on the Previewer screen and click **Print** to print the report.
7. Scan the original SSF and all supporting documents.
8. To generate a PDF file of the Acknowledgement Letter, which is then emailed to the client, the following steps are followed:
 - a. Select the **Reports** pull-down menu from the LIMS main menu.
 - b. Select **Run Reports** from the Report main menu.
 - c. Select the **ACK PDF** and click **Run Report**.
 - d. Enter the Log in number and the ".pdf" file extension type on the Run Time Parameter Form and click **Run Report**, ex. L4256.pdf.

NOTE: This generates a file which is then emailed to the client. The location of the file, which shows on the parameter is: f:/lims/ACK PDFs/. The file is named as in Step 4 (e.g., L4256.pdf).

- J. Quarterly Composites (many REMP clients require that E-LAB perform analyses on a composite of all samples from a given location on a quarterly basis)
1. Once all samples have been received for a given client, prepare a sample submission form (SSF) for these samples which lists the individual sample LSNs as well as the composite LSN using the "Quarterly Report" or the "Quarterly-AP" report.
 2. Determine if all samples for each location requiring quarterly compositing have been received within ten business days after the end of each calendar quarter.
 3. Select the **Reports** pull-down menu from the SeedPak main menu.
 4. Select **Run Reports** from the Select Report screen.
 5. Select the **Quarterly Report** for all samples other than APs. Select the **Quarterly APs Report** for APs.
 6. Click **Run Report**.
 7. Set the following on the Runtime Parameter Form:

- a. Enter the **Project/Acct** for the client for which the composite is to be prepared for.
 - b. Enter the Matrix for the composite.
 - c. Click **Run Report** and **PRINT**.
8. Prepare a submission form (SSF).
 9. Submit the SSF and quarterly composite reports to the Sample Control and Measurements Supervisor for review.
 10. Assign LSNs to each quarterly composite in accordance with Section E.
 11. Print analysis worksheets and labels in accordance with Section F.
 12. Send the SSFs, quarterly composite reports, analysis worksheets, and labels into Sample Prep to allow the chemistry staff to prepare the composite samples.
- K. Sample Processing Chain-of-Custody (Environmental/Bioassay Samples Only)**
1. All samples and corresponding sample analysis worksheets, which require sample preparation and/or radiochemistry, are sent into the designated chemistry processing areas by the Sample Receipt Staff. Any sample which does not require any sample preparation and/or radiochemistry (i.e., charcoal cartridge samples) is sent directly into the Environmental Counting Room. See Attachment A for a listing of the LIMS status indicators.
 2. The sample preparation and/or radiochemistry staff prepares the sample for analysis using the appropriate Laboratory radiochemical technique. PPE shall be utilized during the processing of all samples in the designated chemistry processing areas. After the samples are prepared for analysis, all data should be entered into LIMS. Once data entry has been completed, the LIMS Sample Prep Department Status (SP) and/or LIMS Chemistry Department Status will be updated to "DONE", and the workgroup number recorded.
 3. The sample and corresponding analysis worksheets are sent into the Environmental Counting Room. Samples and paperwork are placed on the "To be Counted" shelves in the Sample Transfer Room.
 4. The Measurements staff shall analyze the sample or sample fraction using the appropriate calibrated nuclear instrumentation detection system.
 5. Once samples have been counted, the samples are placed on the "Counted" shelves. Once the analysis results are approved using Outer-LIMS (Reference 9), the LIMS Instrumentation Department Status will be updated to "DONE" and the LIMS Report Department Status updated to "HERE".
 6. Analysis worksheets for the counted samples are forwarded to the Recordkeeping area for report generation. The Recordkeeping staff in accordance with Reference 8 generates reports. Once the analysis report is generated, the report status is updated to "RPTD".

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7. The analysis reports are forwarded to senior staff for review and approval.
 8. A fraction of analysis packages are forwarded to the QA Office for Data Integrity Review as defined in Reference 11.
 9. Once signed, the report approver must manually approve each result. Once approved in LIMS, the LIMS Report Department Status will be updated to "DONE".
 10. Next, the report is mailed to the client(s). The LIMS Mail Department Status must be manually updated to "DONE".
 11. Once results are approved and mailed, they become available for inclusion in an electronic data file (EDF). Once the result is sent to an electronic data file, the LIMS Report Department Status is updated to "EDD".
 12. Once a result has been approved and mailed, an invoice for the work can be prepared. Once the invoice is created, the LIMS Invoice Department Status is updated to "DONE".
- L. **Sample Processing Chain-Of-Custody (10 CFR Part 50/61 Samples Only)**
Please see Reference 4.
- M. **Long-Term Storage Chain-of-Custody**
The designated storage areas and required storage periods for the various types of samples received by the E-LAB are described in Reference 7.
- N. **Sample Analysis Report Generation/Sample Billing**
1. All SSF, Chain of Custody, FORM 201.1, and other shipping/packaging documents are forwarded to the E-LAB Project Administrator area for electronic document scanning.
 2. All analysis worksheets and reports are forwarded to the E-Lab Project Administrator area for electronic document scanning.
 3. The worksheets and reports are subsequently filed until the records are archived or returned to the client per contract specification.
 4. Only those reports, which have been approved and signed, will be submitted to the client individual(s).
 5. Samples are billed using mutually agreed upon prices specified in each client's contract.

FINAL CONDITIONS

All samples shipped to the Laboratory are received, processed, analyzed and reported to the client individual(s) in accordance with the general chain-of-custody requirements described by this procedure.

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NOTES

1. Laboratory contamination control samples (e.g., swipes, "tacky" mats, mop waters, hood filters) may be analyzed by Laboratory staff without the assignment of a Laboratory Sample Number (LSN). These analyses are reported internally to the RSO by the nuclear instrumentation staff.
2. Laboratory staff may elect to treat milk samples with formaldehyde and methimazole in accordance with Reference 5 upon sample receipt.
3. For the EPA methods currently used, there are no specific requirements/limitations on containers or holding times.
4. The eight day delay allows for the decay of unsupported La-140 in the sample.
5. Certain routine REMP milk samples require a gamma spectrometry and a Sr-89,90 or I-131(LL) analysis performed on the same sample since a limited amount of milk was available. In these cases, the Sr-89,90 or I-131(LL) analysis worksheets may be held in the Chemistry III or Sample Receipt area until the sample analyzed for gamma spectrometry is returned to the Chemistry III or Sample Receipt area by the instrumentation staff. The Chemistry III or Sample Receipt Staff will forward the sample into the Environmental Chemistry Area.

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ATTACHMENT A

LIMS Status Indicators

| Processing Area or Function | Status Indicator | Description of Status |
|--|------------------|--|
| Sample Receipt (SR) | NEED | Sample Receipt Not Completed |
| | DONE | Sample Receipt Completed |
| Sample Preparation (SP) and Chemistry (CHEM) | NEED | Sample Prep Not Completed |
| | DONE | Sample Prep Completed |
| Instrumentation (INS) | NEED | Instrumentation Not Completed |
| | DONE | Instrumentation Completed |
| Reporting (RPT) | NEED | Report Not Generated/Results Not Available |
| | HERE | Report Ready to be Generated |
| | RPTD | Report Generated but Not Approved |
| | DONE | Report Approved |
| | EDD | EDD Created |
| Mail | NEED | Report Not Mailed |
| | DONE | Report Mailed |
| Invoicing (INV) | NEED | Invoice Not Created |
| | DONE | Invoicing Completed |

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Login Number:

Account:

Project:

- Samples surveyed by beta-gamma "frisker" (* > MDCR noted on SSF or below):
- If client on NELAC list, each bottle assigned unique LSN
- If client on NELAC list, Sample location, date, time, collector, preservation type are provided by client
- Shipping/sample container integrity OK
- SSF/Shipping documents/container ID's identical
- Original Chain-of-Custody returned to client (if applicable)
- Copy of Chain of Custody kept (if applicable)
- Sample date/time and ID provided
- Milk received on "ice"
- Milk-evidence of curdling
- Minimum sample volume per Procedure 110 received:

LSN's

Initial pH

Preservation:

Samples pH > 2, acid addec Y / N

Water: Bisulfite (I131LL or I131 by gamma, Y / N
if applicable)

Milk: Client Preserved Y / N

Methimazole added Y / N

Formaldehyde added Y / N

Refrigerated (if not preserved) Y / N

Samples put on HOLD ?
Released for processing based on: _____

Released by: _____ Management _____ Date _____

Sample(s) Unable to be Processed: LSN(s): _____

Reason / Disposition _____ (incl. shipment tracking #)