

## AR TARGET SHEET

The following document was too large to scan as one unit, therefore, it has been broken down into section.

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SECTION: 1 of 5

1100-EM-1 GROUNDWATER CHARACTERIZATION;  
PHASE 1

DATA PACKAGE/REPORT No. 1, REVISION 0

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- S. K. Fadeff
- J. H. Kaye
- E. A. Lepel
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April 1992

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Pacific Northwest Laboratory  
Richland, Washington 99352

CONTENTS

CERTIFICATION.....	1
INTRODUCTION.....	2
SUMMARY.....	3
KEY CONTRIBUTORS.....	7
SAMPLE NUMBERS.....	8
APPENDIX A - ADMINISTRATIVE DOCUMENTS.....	A00-001
APPENDIX B - RADIOCHEMISTRY STANDARDS AND CONTROLS.....	B00-001
APPENDIX C - ICP-MS FOR TC-99.....	C00-001
APPENDIX D - GAMMA ENERGY ANALYSIS.....	D00-001
APPENDIX E - TOTAL BETA ANALYSIS.....	E00-001
APPENDIX F - STRONTIUM-90 ANALYSIS.....	F00-001
APPENDIX G - TRITIUM ANALYSIS.....	G00-001
APPENDIX H - LIQUID SCINTILLATION COUNTING.....	H00-001
APPENDIX J - RADIOCHEMICAL ANALYSIS FOR TC-99.....	J00-001

9 2 1 3 3 0 1 1 3 3

CERTIFICATION

To the best of my knowledge, the work reported in this document was accomplished in accordance with the requirements specified in the U.S. Department of Energy (DOE) Request for Services (RFS) Number TD3204 Statement of Work (SOW) and all referenced lower-tier documents.

Bruce A. Prentice 4/17/92  
Bruce A. Prentice Date  
Project Manager  
Analytical Chemistry Laboratory

CONCURRENCE:

J. Leland Daniel 4/16/92  
J. Leland Daniel Date  
Data Quality Officer  
Analytical Chemistry Laboratory

## INTRODUCTION

Previous analyses of groundwater samples from the 1100-EM-1 Operable Unit have indicated that the samples exceeded drinking water standards for gross beta activity. It has been suggested that technetium-99 (Tc-99) may account for the bulk of that activity. The goal of the work described in this report was to confirm the total beta activity of groundwater samples from the 1100-EM-1 Operable Unit and determine to what extent Tc-99 contributes to that activity. Analyses were performed to determine the presence of several other radiochemical species, should Tc-99 be found not to be the predominant contributor to the total beta activity.

Technetium-99 concentration was determined by Inductively-Coupled Plasma Mass Spectrometry (ICP-MS) according procedure PNL-ALO-281. Total beta analysis was performed according to procedures PNL-ALO-106, PNL-ALO-462, and PNL-ALO-463. Technetium-99 was also determined by chemical separation and counting according to procedure 7-40.39. The supplemental analyses performed included: tritium (H-3) by procedures PNL-ALO-441 and PNL-ALO-443, strontium-90 (Sr-90) by PNL-ALO-465, liquid scintillation analysis of the beta energy spectrum, and gamma energy analysis (GEA) by PNL-ALO-465.

Samples were selected by the client for each method of analysis. The final data packages for analyses of these samples are presented in the appendixes of this document, organized by analytical method and each including summary results and raw data for that method. A summary of the findings follows.

The complete records for this project can be obtained from Pacific Northwest Laboratory (PNL) by referencing PNL Project Number 19432.

## SUMMARY

For each sample, the client technical contact identified its source well number; these are cross-referenced to client sample identification and Analytical Chemistry Laboratory (ACL) sample number in the "SAMPLE NUMBERS" section of this document. He identified the following wells as representative of the plume containing high total beta concentration: MW-10, MW-11, MW-12, MW-13, MW-14, and MW-15. He also identified the following wells as representative of up-gradient conditions: MW-8 and MW-9.

The results of the ICP-MS Tc-99 analyses and the total beta analyses are summarized in Table 1, expressed in picocuries per liter (pCi/L) of sample. Also presented are the differences between the total beta and the Tc-99 results (Total Beta - Tc-99).

For samples identified as being in the plume, the agreement between the total beta and Tc-99 analyses is good. For these analyses, total beta concentration ranged from 75.3 to 138 pCi/L, with a mean of 105 pCi/L, while Tc-99 concentration ranged from 84 to 149 pCi/L, with a mean of 116 pCi/L. The mean difference between total beta and Tc-99 concentration for analysis of plume samples was -11.6 pCi/L. This mean difference, and the preponderance of negative differences suggests a small bias in one or both of the methods. We have not determined the exact nature of the apparent bias; suggested explanations include matrix sensitivity of the total beta procedure or effects related to preservation of the samples, the bias is not apparent if samples from the November sampling, which were unpreserved, are considered separately. While the differences ranged from -42.7 to 36.0 pCi/L, this variability is not substantially different from that seen between repeat samples of the same well and duplicate sample analyses, and it is substantially larger than the apparent bias between the methods; it is likely that statistical variability associated with these analyses accounts for much, if not most, of the differences seen between the total beta and Tc-99 analyses, beyond the apparent method bias.

The samples identified as being representative of up-gradient conditions exhibited detectable, but relatively low, concentrations of total beta and Tc-99; these analyses are also in good agreement.

Samples from the following wells exhibited total beta and Tc-99 concentrations similar to those found in the plume samples: MW-20, FF5-8A, SNP-9, and SNP-15. The following wells exhibited low total beta and Tc-99 results, similar to up-gradient wells: MW-19, MW-21, MW-22, FF5-7A, and SNP-24. The sample from well S27-E14 does not resemble either of these profiles, having total beta and Tc-99 results of 33.9 and 20 pCi/L, respectively.

Liquid scintillation results confirm the agreement between the total beta and Tc-99 results for plume samples. The beta energy spectrum analysis of plume samples shows only one identifiable peak, coincident with the theoretical location of a Tc-99 peak. Samples from wells SNP-9 and SNP-15 show similar peaks, but appear to have one or two additional peaks. The sample from well S27-E14 exhibited a very broad beta spectrum with no definitive peaks, though the signal was above background in the Tc-99 region.

No peaks were detected above detection limits in the gamma energy analysis (GEA) of any of the samples and no samples exhibited strontium-90 (Sr-90) results above detection limits.

There were no tritium results above detection limits except for one sample from well MW-13, which showed concentrations of 5100 and 4800 pCi/L for replicate analyses of the sample (Client ID B01BW5; ACL Number 92-2747); however, the result of tritium analysis of another sample from this well (Client ID B015T5; ACL Number 92-2731) was below the detection limit. Tritium analysis of one sample from well MW-10 (Client ID B01C38; ACL Number 92-2750) gave a result at the detection limit of 450 pCi/L; however, other samples from this well showed no detectable tritium.

Technetium-99 determinations by procedure 7-40.39 could not be completed due to difficulties associated with the Tc-95m standard. The standard is used

for spiking all samples, to correct for differences in recovery during the separation step for each sample.

In summary, for samples taken from wells identified as being within the high beta plume (MW-10, MW-11, MW-12, MW-13, MW-14, and MW-15), total beta concentration appears to be approximately 105 pCi/L (+/- 20 pCi/L) when determined as Tc-99. Technetium-99 appears to account for most, if not all, of this beta activity. There is no data in the results obtained from this study to suggest that there is any other significant contributor to the total beta activity found in these samples.

TABLE 1. Summary of Tc-99 and Total Beta Analyses

ACL No.	Client ID	Well Number	Total Beta (pCi/L)	Tc-99 (pCi/L)	Difference (pCi/L)
92-2724	B01572	MW-8	10.8	17	-6.2
92-2725	B01573	MW-9	5.72	15	-9.28
92-2726	B01574	FF5-7A	11.4	24	-12.6
92-2727	B01575	FF5-8A	85.2	121	-35.8
92-2728	B015S9	MW-10	90.1	125	-34.9
92-2729	B015T1	MW-11	123	128	-5
92-2730	B015T3	MW-12	107	149	-42
92-2731	B015T5	MW-13	75.3	118	-42.7
92-2732	B015T7	MW-14	100	139	-39
92-2733	B015T9	MW-15	78.2	84	-5.8
92-2734	B015V1	SNP-9	105	115	-10
92-2735	B015V3	SNP-15	153	141	12
92-2736	B015V5	SNP-24	4	<DL	
92-2737	B015V7	MW-19	11.7	<DL	
92-2738	B015V8	MW-20	139	155.5	-16.5
92-2739	B015V9	MW-21	8.87	<DL	
92-2740	B015W0	MW-21D	10.2	<DL	
92-2741	B015W1	MW-21S	8.65	<DL	
92-2742	B015W2	MW-22	17.8	<DL	
92-2743	B015W3	T BLK	<DL	<DL	
92-2744	B01BV6	MW-10	108	135	-27
92-2745	B01BV9	MW-11	115	140	-25
92-2746	B01BW2	MW-12	138	121	17
92-2747	B01BW5	MW-13	102	104	-2
92-2748	B01BW8	MW-14	128	99	29
92-2749	B01BX1	MW-15	76.1	86	-9.9
92-2750	B01C38	MW-10DUP	122	86	36
92-2751	B01C11	MW-19	14.1	17	-2.9
92-2752	B01C28	S27-E14	33.9	20	13.9

**Summary for Plume Samples:**  
(MW-10, MW-11, MW-12, MW-13, MW-14, MW-15)

Average	104.82	116.46	-11.6
Standard Deviation	20.48	22.48	26.6
N	13	13	13
SEM	5.7	6.2	7.4
CV	0.20	0.19	-2.29
Maximum	138	149	36.0
Minimum	75.3	84	-42.7

<DL -- Less than Detection Limit; total beta - 3 pCi/L, Tc-99 - 10 pCi/L

## KEY CONTRIBUTORS

### Client Representatives

W Greenwald, USACE      Technical Contact  
RK Stewart, DOE-RL      Contract/Funding Contact

### Project Management

BA Prentice      Project Manager  
SK Fadeff      Deputy Project Manager

### ACL Quality Control

JL Daniel      Data Quality Officer  
KJ Kuhl-Klinger

### PNL Quality Programs

BC King      Group Leader  
RR LaBarge      Group Leader  
TL Ehlert      Quality Engineer  
VC Thompson

### Project Support

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SR Butler  
JL Reilly

### Records Management

PM Lindsay      Task Leader  
LH Pennington

### Radiochemical Analysis

LR Greenwood      Group Leader  
GG Brodaczynski  
SK Fadeff  
JH Kaye  
EA Lepel  
DL McMullin  
RS Strebin  
NL Wynhoff

### Advanced Inorganic Analysis

DW Koppenaal      Group Leader  
NL Abbey  
JP Bramson  
EJ Wyse

1100-EM-1 SAMPLE NUMBERS

<u>Client ID</u>	<u>ACL Sample Number</u>	<u>Well Number</u>
B01572	92-002724	MW-8
B01573	92-002725	MW-9
B01574	92-002726	FF5-7A
B01575	92-002727	FF5-8A
B015S9	92-002728	MW-10
B015T1	92-002729	MW-11
B015T3	92-002730	MW-12
B015T5	92-002731	MW-13
B015T7	92-002732	MW-14
B015T9	92-002733	MW-15
B015V1	92-002734	SNP-9
B015V3	92-002735	SNP-15
B015V5	92-002736	SNP-24
B015V7	92-002737	MW-19
B015V8	92-002738	MW-20
B015V9	92-002739	MW-21
B015W0	92-002740	MW-21D
B015W1	92-002741	MW-21S
B015W2	92-002742	MW-22
B015W3	92-002743	T-BLK
B01BV6	92-002744	MW-10
B01BV9	92-002745	MW-11
B01BW2	92-002746	MW-12
B01BW5	92-002747	MW-13
B01BW8	92-002748	MW-14
B01BX1	92-002749	MW-15
B01C38	92-002750	MW-10
B01C11	92-002751	MW-19
B01C28	92-002752	S27-E14

1100-EM-1 GROUNDWATER CHARACTERIZATION  
PHASE I

DATA PACKAGE/REPORT No. 1

Revision 0

Appendix A

ADMINISTRATIVE DOCUMENTS

TABLE OF CONTENTS

- A01 - Westinghouse Chain of Custody and Sample Analysis Request Forms  
A02 - PNL Chain of Custody Forms  
A03 - Technical Procedures
- PNL-ALO-105 Procedure for Preparation of Samples to be counted by  
Gamma-Ray Spectroscopy
- PNL-ALO-106 Acid Digestion for Preparation of Samples for  
Radiochemical Analysis
- PNL-ALO-280 Inductively Coupled Plasma-Mass Spectrometric (ICP-MS)  
Analysis
- PNL-ALO-281 ICP/MS Determination of <sup>99</sup>Tc
- PNL-ALO-441 Radionuclide Separation and Analysis Procedure for Tritium
- PNL-ALO-443 Liquid Scintillation Counting Procedure for Tritium
- PNL-ALO-462 Source Preparation for Gross Beta Analysis
- PNL-ALO-463 Beta Counting Procedure
- PNL-ALO-464 Procedure for Gamma Counting and Data Reduction in the  
Low-Level Counting Room, 329 Building
- PNL-ALO-465 Strontium-90 Analysis (Oxalate-Nitric Acid Method)
- PNL-ALO-470 Procedure for Maintaining Control of Germanium  
Spectrometers Used for Gamma-Ray Spectroscopy

A00-001

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/19/91 Time 1045
Sample Locations	1100-EM-1		
Ice Chest No.	Delta-2	Field Logbook and Page No.	W4C-W-370, 105
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> <sub>12-20-91</sub>		

**SAMPLE IDENTIFICATION**

BO 1522 1, 3L, P, WATER, Tc-99
-----------------------------------

**CHAIN OF POSSESSION**

Relinquished by: <i>Kathy Lee</i>	Received by: <i>Matthew Walker</i>	Date/Time: <i>9/19/91 1315</i>
Relinquished by: <i>Matthew Walker</i>	Received by: <i>J.H. Kaye</i>	Date/Time: <i>12/20/91 - 1335</i>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/17/91 Time 1215
Sample Locations	1100-EM-1		
Ice Chest No.	Delta 2	Field Logbook and Page No.	W4C-N-370.p.105
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL <sup>325</sup> LAB <sup>PHB</sup> 12-20-91		

**SAMPLE IDENTIFICATION**

BO 1523 1, 3L, P, WATER, Tc-99
-----------------------------------

**CHAIN OF POSSESSION**

Relinquished by: <i>L.D. Walker</i>	Received by: <i>Robert P. Baker</i>	Date/Time: 9-19-91 1315
Relinquished by: <i>Robert P. Baker</i>	Received by: <i>J.H. Hayes</i>	Date/Time: 12/20/91 1335
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/19/91 Time 0925
Sample Locations	1100-EM-1		
Ice Chest No.	Delta 2	Field Logbook and Page No.	LVHC-N-370, 105
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> <sup>PAB</sup> 9-20-91		

**SAMPLE IDENTIFICATION**

B01574 1, 3L, P, WATER, Tc-99
----------------------------------

**CHAIN OF POSSESSION**

Relinquished by: <i>Robert L. K. 22</i>	Received by: <i>Robert L. K. 22</i>	Date/Time: 9/19/91 1315
Relinquished by: <i>Robert L. K. 22</i>	Received by: <i>J. M. Hayes</i>	Date/Time: 12/20/91 1335
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/19/91
Sample Locations	1100-EM-1	Time	0747
Ice Chest No.	Weta 2	Field Logbook and Page No.	WHC-N-370, 104
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> <sup>WAC</sup> <sub>12-20-91</sub>		

**SAMPLE IDENTIFICATION**

BO 1575  
 1, 3L, P, WATER, Tc-99

**CHAIN OF POSSESSION**

Relinquished by: <i>[Signature]</i>	Received by: <i>[Signature]</i>	Date/Time: 9/19/91 1315
Relinquished by: <i>[Signature]</i>	Received by: <i>[Signature]</i>	Date/Time: 12/20/91 1335
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/23/91 Time 0753
Sample Locations	1100-EM-1		
Ice Chest No.	Delta 2	Field Logbook and Page No.	WHL-N-370, 107
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>328</sup> <sup>P.B.</sup> 9-30-91		

**SAMPLE IDENTIFICATION**

<p>B01559 1, 3L, P, WATER, Tc-99</p> <p><del>B01575 B, 3, 40 ml, G, water - C.L. - W.D. - P.D.C. 9/23/91</del></p>
--

**CHAIN OF POSSESSION**

Relinquished by: <i>K. J. Lee K.J.C.</i>	Received by: <i>Robert Walker</i>	Date/Time: 9/23/91 1206
Relinquished by: <i>Robert Walker</i>	Received by: <i>J. H. Gage</i>	Date/Time: 12/20/91 1335
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

92-02729

14432151

CHAIN OF CUSTODY				
Company Contact	B.H. FORD	Telephone	509-376-6465	
Sample Collected by	L. WALKER	Date	9/23/91	Time 0832
Sample Locations	1100-EM-1			
Ice Chest No.	Delta 2	Field Logbook and Page No.	WHC-N-370 p. 107	
Remarks	N/A			
Bill of Lading No.	N/A	Offsite Property No.	N/A	
Method of Shipment	HAND DELIVER			
Shipped to	PNL/325 LAB <sup>329</sup> <sub>13-20-91</sub> PAB			

SAMPLE IDENTIFICATION

BO 1571 1, 3L, P, WATER, Tc-99
-----------------------------------

CHAIN OF POSSESSION

Relinquished by: <i>Robert P. Lee P.D.C.</i>	Received by: <i>Robert P. Lee</i>	Date/Time: 9/23/91 1206
Relinquished by: <i>Robert P. Lee</i>	Received by: <i>J. St. Rayer</i>	Date/Time: 12/20/91 1335
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/23/91 Time 0925
Sample Locations	1100-EM-1		
Ice Chest No.	#10	Field Logbook and Page No.	WHC-N-370 2107
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>327</sup> P110 12-20-91		

SAMPLE IDENTIFICATION
BO157J 1, 3L, P, WATER, Tc-99

CHAIN OF POSSESSION		
Relinquished by: <i>Karby P. Lee &amp; Co.</i>	Received by: <i>P. H. Walker</i>	Date/Time: 9/23/91 1206
Relinquished by: <i>P. H. Walker</i>	Received by: <i>J. H. Hayes</i>	Date/Time: 12/20/91 - 1345
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/23/91 Time 11045
Sample Locations	1100-EM-1		
Ice Chest No.	#10	Field Logbook and Page No.	WHC-N-370 p. 107
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> <sub>12-20-91</sub> PAB		

SAMPLE IDENTIFICATION
BO 1575 1, 3L, P, WATER, Tc-99

CHAIN OF POSSESSION		
Relinquished by: <i>Kerry A Lee K.O.L.</i>	Received by: <i>Pittman Prother</i>	Date/Time: 9/23/91 1206
Relinquished by: <i>Pittman Prother</i>	Received by: <i>J.H. Kaye</i>	Date/Time: 12/20/91 1345
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/23/91
		Time	1016
Sample Locations	1100-EM-1		
Ice Chest No.	#10	Field Logbook and Page No.	WHC-N-370, 107
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>329</sup> <sub>12-20-91</sub>		

**SAMPLE IDENTIFICATION**

BO 1577 1, 3L, P, WATER, Tc-99
-----------------------------------

**CHAIN OF POSSESSION**

Relinquished by: <i>Ruby P Lee KDC</i>	Received by: <i>Robert L. Walker</i>	Date/Time: 9/23/91 1200
Relinquished by: <i>Robert L. Walker</i>	Received by: <i>J. H. Kaye</i>	Date/Time: 12/20/91 1345
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9-20-91 Time 0850
Sample Locations	1100-EM-1		
Ice Chest No.	#10	Field Logbook and Page No.	WMC-N-370/pg. 106
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> PAB 12-21-91		

SAMPLE IDENTIFICATION

BO 1579 1, 3L, P, WATER, Tc-99
-----------------------------------

CHAIN OF POSSESSION

Relinquished by: <i>L.D. Walker</i> L.D. Walker	Received by: <i>P. Butler</i> P. Butler	Date/Time: 9-20-91 0900
Relinquished by: <i>P. Butler</i> P. Butler	Received by: <i>J.H. King</i> J.H. King	Date/Time: 12/20/91 1345
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9-30-91 Time 1105
Sample Locations	1100-EM-1		
Ice Chest No.	#10	Field Logbook and Page No.	WTC-N-370 / pg. 110
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325-LAB <sup>327</sup> <sup>PHS</sup> 12-20-91		

**SAMPLE IDENTIFICATION**

BO 15 V1 1, 3L, P, WATER, Tc-99
------------------------------------

**CHAIN OF POSSESSION**

Relinquished by: <i>AD Walker</i> <i>L.D. Walker</i>	Received by: <i>Robert the Archiver</i>	Date/Time: <sup>10-1-91</sup> 9-30-91 0830
Relinquished by: <i>PHS Walker</i> <i>PHS Walker</i>	Received by: <i>J. H. King</i>	Date/Time: 12/20/91 1345
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY					
Company Contact	B.H. FORD	Telephone	509-376-6465		
Sample Collected by	L. WALKER	Date	9-30-91	Time	1240
Sample Locations	1100-EM-1				
Ice Chest No.	#10	Field Logbook and Page No.	WHC-N-370 / pg. 111		
Remarks	N/A				
Bill of Lading No.	N/A	Offsite Property No.	N/A		
Method of Shipment	HAND DELIVER				
Shipped to	PNL/325 LAB <sup>325</sup> <sub>12-20-91</sub>				

SAMPLE IDENTIFICATION

BO 1503 1, 3L, P, WATER, TC-99
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CHAIN OF POSSESSION

Relinquished by: <i>L.D. Walker</i>	Received by: <i>P.H. Butler</i>	Date/Time: <i>10-1-91 0830</i>
Relinquished by: <i>P.H. Butler</i>	Received by: <i>P.H. Butler</i>	Date/Time: <i>9-30-91</i>
Relinquished by: <i>P.H. Butler</i>	Received by: <i>J.H. Hayes</i>	Date/Time: <i>12/20/91 1345</i>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9-30-91
		Time	1000
Sample Locations	1100-EM-1		
Ice Chest No.	#10	Field Logbook and Page No.	WHC-N-370 / pg. 110
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> <sup>PAB</sup> 12-20-91		

**SAMPLE IDENTIFICATION**

BO 15 V5 1, 3L, P, WATER, Tc-99
------------------------------------

**CHAIN OF POSSESSION**

Relinquished by: <i>L. Walker</i> <i>L. Walker</i>	Received by: <i>Pitts</i> <i>Pitts</i>	Date/Time: <del>70-3</del> 10-1-91 9-30-91 0830
Relinquished by: <i>Pitts</i> <i>Pitts</i>	Received by: <i>J. H. Keyser</i>	Date/Time: 12/20/91 1345
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	09/20/91 Time 0755
Sample Locations	1100-EM-1		
Ice Chest No.	#10	Field Logbook and Page No.	WHC-N-370 / pg. 106
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> <sup>PWB</sup> 12-20-91		

**SAMPLE IDENTIFICATION**

<p>BO 1547 1, 3L, P, WATER, Tc-99</p>
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**CHAIN OF POSSESSION**

Relinquished by: <i>L.D. Walker</i>	Received by: <i>P. Butcher</i>	Date/Time: <i>9/20/91 0900</i>
Relinquished by: <i>P. Butcher</i>	Received by: <i>J.H. Hays</i>	Date/Time: <i>12/20/91 1345</i>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9-20-91 Time 0930
Sample Locations	1100-EM-1		
Ice Chest No.	Freezy 1	Field Logbook and Page No.	WMC-N-370 / pg. 106
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> <i>PNL</i> 12-20-91		

SAMPLE IDENTIFICATION

BO 1508 1, 3L, P, WATER, Tc-99
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CHAIN OF POSSESSION

Relinquished by: <i>L.D. Walker</i> L.D. Walker	Received by: <i>PNL/325</i> PNL/325	Date/Time: 9/23/91 0900 9-20-91
Relinquished by: <i>PNL/325</i> PNL/325	Received by: <i>J.M. Key</i> J.M. Key	Date/Time: 12/20/91 1332
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY				
Company Contact	B.H. FORD	Telephone	509-376-6465	
Sample Collected by	L. WALKER	Date	9/23/91	Time 1137
Sample Locations	1100-EM-1			
Ice Chest No.	Freezer-1	Field Logbook and Page No.	WHL-N-370 p. 187	
Remarks	N/A			
Bill of Lading No.	N/A	Offsite Property No.	N/A	
Method of Shipment	HAND DELIVER			
Shipped to	PNL/325 LAB <sup>329</sup> <sup>PHB</sup> 2-20-91			

**SAMPLE IDENTIFICATION**

BO 15 V9  
 1, 3L, P, WATER, Tc-99

**CHAIN OF POSSESSION**

Relinquished by: <i>Kirby Baker</i>	Received by: <i>P. H. Butcher P. H. Butcher</i>	Date/Time: <i>9/23/91 1206</i>
Relinquished by: <i>P. H. Butcher P. H. Butcher</i>	Received by: <i>J. M. Hayes</i>	Date/Time: <i>J.M.H. 12/20/91 1332</i>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B. H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/23/91 Time 1137
Sample Locations	1100-EM-1		
Ice Chest No.	540027-1	Field Logbook and Page No.	WHC-N-370 p 107
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>329</sup> P40 12-20-91		

**SAMPLE IDENTIFICATION**

B01520 1, 3L, P, WATER, Tc-99
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**CHAIN OF POSSESSION**

Relinquished by: <i>K. J. Ke...</i>	Received by: <i>P. H. ...</i>	Date/Time: 9/23/91 1206
Relinquished by: <i>P. H. ...</i>	Received by: <i>J. M. ...</i>	Date/Time: 12/20/91 1332
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9/23/91 Time 1137
Sample Locations	1100-EM-1		
Ice Chest No.	Shelby -1	Field Logbook and Page No.	Wisc N - 370 p 107
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> P40 12-20-91		

**SAMPLE IDENTIFICATION**

BO 15W1 1, 3L, P, WATER, Ic-99
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**CHAIN OF POSSESSION**

Relinquished by: <i>Anthony A. Lee K.D.C.</i>	Received by: <i>P. Butcher P. Butcher</i>	Date/Time: <i>9/23/91 1206</i>
Relinquished by: <i>David L. Walker</i>	Received by: <i>J. H. Kaye</i>	Date/Time: <i>12/20/91 1332</i>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.H. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9-20-91 Time 1025
Sample Locations	1100-EM-1		
Ice Chest No.	510027-1	Field Logbook and Page No.	WHL-N-370/ pg 106
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>371</sup> <sup>PNL</sup> 12-20-91		

SAMPLE IDENTIFICATION

BO 15W2  
 1, 3L, P, WATER, Tc-99

CHAIN OF POSSESSION

Relinquished by: <i>L.D. Walker</i> L.D. Walker	Received by: <i>Robert P. Walker</i> P. Walker	Date/Time: 9-20-91 0700
Relinquished by: <i>Robert P. Walker</i> P. Walker	Received by: <i>J. H. Ray</i> J. H. Ray	Date/Time: 12/20/91 1332
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

CHAIN OF CUSTODY			
Company Contact	B.N. FORD	Telephone	509-376-6465
Sample Collected by	L. WALKER	Date	9-30-91
		Time	0630
Sample Locations	1100-EM-1		
Ice Chest No.	Sneezy-1	Field Logbook and Page No.	WHL-N-370/pg. 110
Remarks	N/A		
Bill of Lading No.	N/A	Offsite Property No.	N/A
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 LAB <sup>325</sup> <sub>12-20-91</sub>		

SAMPLE IDENTIFICATION

BO 15W3 1, 3L, P, WATER, Tc-99
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CHAIN OF POSSESSION

Relinquished by: <i>L. D. Walker</i> L. D. WALKER	Received by: <i>Robert L. Attkin</i>	Date/Time: <i>10-1-91</i> 9-30-91 0830
Relinquished by: <i>P. H. ...</i>	Received by: <i>J. H. ...</i>	Date/Time: <i>12/20/91</i> 1332
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

<b>Westinghouse Hanford Company</b>	<b>CHAIN OF CUSTODY</b>		
Custody Form Initiator	PH BUTCHER		
Company Contact	PH BUTCHER		Telephone (509)376-5045
Project Designation/Sampling Locations	1100-EM-1		Collection Date 11-13-91
Ice Chest No.	See Note		Field Logbook No. WHC-N-370
Bill of Lading/Airbill No.	NA		Offsite Property No. NA
Method of Shipment	HAND DELIVER		
Shipped to	PNL/325 BLDG <sup>324</sup> <sub>2190141</sub> <sup>PMB</sup>		
Possible Sample Hazards/Remarks	N/A		

Sample Identification

BO: BVC  
~~3~~, 11, P, WATER, Tc-99, HNO3  
 1, 4L  
 LW 11-13-91

<input type="checkbox"/> Field Transfer of Custody	Chain of Possession	(Sign and Print Names)
Relinquished by: <i>L.D. Walker</i> L.D. Walker	Received by: <i>PH Butcher</i> PH Butcher	Date/Time: 11/14/91 0700
Relinquished by: <i>PH Butcher</i> PH Butcher	Received by: <i>J. H. Kaye</i>	Date/Time: 12/20/91 1322
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition		
Disposal Method:	Disposed by:	Date/Time:
Comments:		

Note: Sample was double bagged

<b>Westinghouse Hanford Company</b>	<b>CHAIN OF CUSTODY</b>	
Custody Form Initiator <b>PH BUTCHER</b>	Telephone <b>(509)376-5045</b>	
Company Contact <b>PH BUTCHER</b>	Collection Date <b>11-13-91</b>	
Project Designation/Sampling Locations <b>1100-EM-1</b>	Field Logbook No. <b>WHC-N-370</b>	
Ice Chest No. <b>See Note</b>	Offsite Property No. <b>NA</b>	
Bill of Lading/Airbill No. <b>NA</b>		
Method of Shipment <b>HAND DELIVER</b>		
Shipped to <b>PNL/325 BLDG <sup>327</sup> 12-20-91</b>		
Possible Sample Hazards/Remarks <b>N/A</b>		

Sample Identification

BO18V9  
~~3~~, IL, P, WATER, Tc-99, HNO3  
 1,4L  
 LW  
 11-13-91

<input type="checkbox"/> Field Transfer of Custody	Chain of Possession	(Sign and Print Names)
Relinquished by: <i>L.D. Walker</i> L.D. Walker	Received by: <i>PH Butcher</i> PH Butcher	Date/Time: 11/14/91 0100
Relinquished by: <i>PH Butcher</i> PH Butcher	Received by: <i>J. H. Kaye</i> J. H. Kaye	Date/Time: 12/20/91 1322
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:
Comments:		

Note: Sample was double bagged

<b>Westinghouse Hanford Company</b>	<b>CHAIN OF CUSTODY</b>	
Custody Form Initiator <b>PH BUTCHER</b>	Telephone <b>(509)376-5045</b>	
Company Contact <b>PH BUTCHER</b>	Collection Date <b>11/12/91</b>	
Project Designation/Sampling Locations <b>1100-EM-1</b>	Field Logbook No. <b>WHL-V-370 p 111</b>	
Ice Chest No. <b>See Note</b>	Offsite Property No. <b>NA</b>	
Bill of Lading/Airbill No. <b>NA</b>		
Method of Shipment <b>HAND DELIVER</b>		
Shipped to <b>PNL 325 BLDG</b> <i>325 PNB 12/20/91</i>		
Possible Sample Hazards/Remarks <b>N/A</b>		

**Sample Identification**

BO/BW2  
~~3~~ <sup>10L</sup> P, WATER, Tc-99, HNO3  
 1, 4L

<input type="checkbox"/> Field Transfer of Custody	Chain of Possession	(Sign and Print Names)
Relinquished by: <i>Philip Lee W.D. Lee</i>	Received by: <i>PH Butcher PH Butcher</i>	Date/Time: <i>11/14/91</i> <i>11/12/91</i> 0100
Relinquished by: <i>PH Butcher PH Butcher</i>	Received by: <i>J. H. Hayes</i>	Date/Time: <i>12/20/91</i> 1322
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition		
Disposal Method:	Disposed by:	Date/Time:
Comments:		

*Note: Sample was double bagged*

Westinghouse  
Hanford Company

CHAIN OF CUSTODY

Custody Form Initiator PH BUTCHER

Company Contact PH BUTCHER

Telephone (509)376-5045

Project Designation/Sampling Locations 1100-EM-1

Collection Date 11/12/91

Ice Chest No. See Note

Field Logbook No. WDC-N-32-P.112

Bill of Lading/Airbill No. NA

Offsite Property No. NA

Method of Shipment HAND DELIVER

Shipped to PNL/325 BLDG <sup>729</sup> <sup>476</sup> 12/20/91

Possible Sample Hazards/Remarks N/A

Sample Identification

BO <sup>1 Bulb</sup>  
3, 1L, P, WATER, Tc-99, HNO3

<input type="checkbox"/> Field Transfer of Custody		Chain of Possession		(Sign and Print Names)	
Relinquished by: <i>L.D. Walker</i> L.D. Walker	Received by: <i>PH Butcher</i> PH Butcher	Date/Time: 11/14/91			0700
Relinquished by: <i>PH Butcher</i> PH Butcher	Received by: <i>G. H. Hayes</i> G. H. Hayes	Date/Time: 12/20/91			1322
Relinquished by:	Received by:	Date/Time:			
Relinquished by:	Received by:	Date/Time:			

Final Sample Disposition

Disposal Method:	Disposed by:	Date/Time:
Comments:		

Note: Sample was double bagged

<b>Westinghouse Hanford Company</b>	<b>CHAIN OF CUSTODY</b>
Custody Form Initiator PH BUTCHER	
Company Contact PH BUTCHER	Telephone (509)376-5045
Project Designation/Sampling Locations 1100-EM-1	Collection Date 11-12-91
Ice Chest No. <i>see Note</i>	Field Logbook No. <i>WHC-N-370 / pg. 112</i>
Bill of Lading/Airbill No. NA	Offsite Property No. NA
Method of Shipment <b>HAND DELIVER</b>	
Shipped to PNL/ <sup>329</sup> 325 BLDG <sup>12/20/91</sup>	
Possible Sample Hazards/Remarks N/A	

Sample Identification

BO 1308  
~~3~~, IL, P, WATER, Tc-99, HNO3  
 1, 4L  
 LW 11-12-91

<input type="checkbox"/> Field Transfer of Custody	Chain of Possession	(Sign and Print Names)
Relinquished by: <i>L.D. Walker</i> L.D. Walker	Received by: <i>PH Butcher</i> - PH Butcher	Date/Time: <i>11/14/91</i> 0700
Relinquished by: <i>PH Butcher</i> - PH Butcher	Received by: <i>J. H. Hayes</i>	Date/Time: <i>12/20/91</i> 1322
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition		
Disposal Method:	Disposed by:	Date/Time:
Comments:		

*Note: Sample was double bagged*

<b>Westinghouse Hanford Company</b>	<b>CHAIN OF CUSTODY</b>	
Custody Form Initiator <b>PH BUTCHER</b>	Telephone <b>(509)376-5045</b>	
Company Contact <b>PH BUTCHER</b>	Collection Date <b>11-12-91</b>	
Project Designation/Sampling Locations <b>1100-EM-1</b>	Field Logbook No. <b>WHC-N-370/Pg.112</b>	
Ice Chest No. <i>See Note</i>	Offsite Property No. <b>NA</b>	
Bill of Lading/Airbill No. <b>NA</b>		
Method of Shipment <b>HAND DELIVER</b>		
Shipped to <b>PNL/325-BLDG</b> <sup>325</sup> <sup>PHB</sup> <sub>12/20/91</sub>		
Possible Sample Hazards/Remarks <b>N/A</b>		

Sample Identification

BO/BX1  
~~3~~, IL, P, WATER, Tc-99, HNO3  
 1, 4L  
 LW 11-12-91

[ ] Field Transfer of Custody	Chain of Possession	(Sign and Print Names)
Relinquished by: <i>L.D. Walker</i>	Received by: <i>PH Butcher</i>	Date/Time: <i>11/14/91 0700</i>
Relinquished by: <i>PH Butcher</i>	Received by: <i>J. H. Kaye</i>	Date/Time: <i>12/20/91 121322</i>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition		
Disposal Method:	Disposed by:	Date/Time:
Comments:		

*Note: Sample was double bagged*

Westinghouse  
Hanford Company

CHAIN OF CUSTODY

Custody Form Initiator PH BUTCHER

Company Contact PH BUTCHER

Telephone (509)376-5045

Project Designation/Sampling Locations 1100-EM-1

Collection Date 11-13-91

Ice Chest No. See Note

Field Logbook No. WHC-N-370

Bill of Lading/Airbill No. NA

Offsite Property No. NA

Method of Shipment HAND DELIVER

Shipped to PNL/325 BLDG <sup>329</sup> <sup>P118</sup> 12/12/91

Possible Sample Hazards/Remarks N/A

Sample Identification

BO/C38

1, 4L, P, WATER, Tc-99, HN03

[ ] Field Transfer of Custody		Chain of Possession	(Sign and Print Names)	
Relinquished by: <i>L.D. Walker</i>	<i>L.D. Walker</i>	Received by: <i>PH Butcher</i>	Date/Time: 11/14/91	0700
Relinquished by: <i>PH Butcher</i>	<i>PH Butcher</i>	Received by: <i>J. H. Hayes</i>	Date/Time: 12/20/91	1322
Relinquished by:		Received by:	Date/Time:	
Relinquished by:		Received by:	Date/Time:	

Final Sample Disposition		
Disposal Method:	Disposed by:	Date/Time:
Comments:		

Note: Sample was double bagged

<b>Westinghouse Hanford Company</b>	<b>CHAIN OF CUSTODY</b>	
Custody Form Initiator	PH BUTCHER	
Company Contact	PH BUTCHER	Telephone (509)376-5045
Project Designation/Sampling Locations	1100-EM-1	Collection Date 11-12-91
Ice Chest No.	See Note	Field Logbook No. WHC-N-370
Bill of Lading/Airbill No.	NA	Offsite Property No. NA
Method of Shipment	HAND DELIVER	
Shipped to	PNL <sup>329</sup> 325 BLDG <sup>2006</sup> 12/20/91	
Possible Sample Hazards/Remarks	N/A	

Sample Identification

BO 1C11  
~~3~~, 1t, P, WATER, Tc-99, HNO3  
 1, 4L  
 LW 11-12-91

[ ] Field Transfer of Custody	Chain of Possession	(Sign and Print Names)
Relinquished by: <i>L. D. Walker</i>	Received by: <i>PH Butcher PH Butcher</i>	Date/Time: 11/14/91 0700
Relinquished by: <i>PH Butcher PH Butcher</i>	Received by: <i>J. H. Lane</i>	Date/Time: 12/20/91 1322
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

Final Sample Disposition		
Disposal Method:	Disposed by:	Date/Time:
Comments:		

*Note Sample was double bagged*

<b>Westinghouse Hanford Company</b>	<b>CHAIN OF CUSTODY</b>
Custody Form Initiator PH BUTCHER	
Company Contact PH BUTCHER	Telephone (509)376-5045
Project Designation/Sampling Locations 1100-EM-1	Collection Date 11-15-91
Ice Chest No. See Note	Field Logbook No. WHC-11-370
Bill of Lading/Airbill No. NA	Offsite Property No. NA
Method of Shipment HAND DELIVER	
Shipped to PNL/325 BLDG <sup>2008</sup> 12/20/91	
Possible Sample Hazards/Remarks N/A	

Sample Identification

BO 1228  
~~3~~, IT, P, WATER, Tc-99, HNO3  
 1,4L  
 LW 11-15-91

<input type="checkbox"/> Field Transfer of Custody		Chain of Possession		(Sign and Print Names)	
Relinquished by: <i>L.D. Walker</i> L.D. Walker	Received by: <i>PH Butcher</i> PH Butcher	Date/Time: 11/16/91			0700
Relinquished by: <i>PH Butcher</i> PH Butcher	Received by: <i>G. H. King</i> G. H. King	Date/Time: 12/20/91			1322
Relinquished by:	Received by:	Date/Time:			
Relinquished by:	Received by:	Date/Time:			

Final Sample Disposition		
Disposal Method:	Disposed by:	Date/Time:
Comments:		

Note: Sample was double bagged

19432/T1

Date Sample(s) Received: \_\_\_\_\_ Date Analysis Results Required: \_\_\_\_\_  
 Sample Log-In Number(s): \_\_\_\_\_  
 Client Sample Number(s): \_\_\_\_\_ Client: USACE  
 In-House Sample Number: 91161-1129  
 Responsible Technical Group Leader(s): \_\_\_\_\_ Received by: J.R. Fung  
 PHL Project Number: 19432 - OR - ED Work Order Number: \_\_\_\_\_  
 QA Plan: MCS-033 ASR/ARF/SOW/11#/LO1: \_\_\_\_\_  
 HA-70 Impact Level: (Circle One) I II III Other QA Criteria: N/A  
 Sample Archive Requirements:  Yes  No Storage Requirements:  Yes  No  
 Sample Type: (Circle One) Solid Gas Solution Slurry Soil Water Sludge Waste Other \_\_\_\_\_

SAMPLE PREP METHOD		ANALYSIS		
(Circle One) Lab	Shielded	Radiochemical	Inorganic	Organic
<input type="checkbox"/> Acid Digestion		<input checked="" type="checkbox"/> GEA	<input type="checkbox"/> ICP Cations	<input type="checkbox"/> GC/MS VOA
<input type="checkbox"/> ICP		<input type="checkbox"/> Tot Alpha	<input checked="" type="checkbox"/> ICP/MS	<input type="checkbox"/> GC/MS SVOA
<input type="checkbox"/> GFAA		<input checked="" type="checkbox"/> Tot Beta	<input type="checkbox"/> GFAA	<input type="checkbox"/> GC Organic
<input type="checkbox"/> Fusion		<input type="checkbox"/> U by laser fluor.	<input type="checkbox"/> Flame AA	<input type="checkbox"/> PCB's
<input type="checkbox"/> KOH		<input type="checkbox"/> I-129	<input type="checkbox"/> Hg (CVAA)	<input type="checkbox"/> Pesticides
<input type="checkbox"/> Na <sub>2</sub> O <sub>2</sub>		<input type="checkbox"/> C-14	<input type="checkbox"/> IC Anions	<input type="checkbox"/> TOX
<input type="checkbox"/> Other		<input checked="" type="checkbox"/> Sr-90 <i>cont. 2/11/92</i>	<input type="checkbox"/> TC/TOC/TIC (Soln)	<input type="checkbox"/> EOX
<input type="checkbox"/> Distillation		<input checked="" type="checkbox"/> H-3	<input type="checkbox"/> EP Toxic Metals	<input type="checkbox"/> POX
<input type="checkbox"/> Hg		<input type="checkbox"/> Am, Cm, AEA	<input type="checkbox"/> CN	<input type="checkbox"/> TOC
<input type="checkbox"/> CN		<input type="checkbox"/> Np, AEA	<input type="checkbox"/> Free CN	<input type="checkbox"/> GC Screen
<input type="checkbox"/> I		<input type="checkbox"/> Pu, AEA	<input type="checkbox"/> Cr (IV)	<input type="checkbox"/> Other
<input type="checkbox"/> Water Leach		<input type="checkbox"/> Pu, MS Isotopic	<input type="checkbox"/> Fe(II)/Fe(III)	
<input type="checkbox"/> TLCP		<input type="checkbox"/> U, MS Isotopic	<input type="checkbox"/> pH	
<input type="checkbox"/> Ion Exchange Separation		<input type="checkbox"/> Se-75/79	<input type="checkbox"/> Conductivity	
<input type="checkbox"/> Solvent Extract Sep.		<input type="checkbox"/> Sn-126	<input type="checkbox"/> OH	<input type="checkbox"/> Shielded Lc
<input type="checkbox"/> VOA		<input type="checkbox"/> Ra-226	<input type="checkbox"/> Density-S.G.	<input type="checkbox"/> WLX Solids
<input type="checkbox"/> Methanol Dilution		<input type="checkbox"/> Nb-93m/94	<input type="checkbox"/> WLX Solids	<input type="checkbox"/> Miller Numbr
<input type="checkbox"/> Hexadecane Extract.		<input type="checkbox"/> Ni-59/63	<input type="checkbox"/> Particle Size	<input type="checkbox"/> Dosimetry Sc
<input type="checkbox"/> Semi-VOA		<input checked="" type="checkbox"/> Tc-99 <i>cont. 2/11/92</i>	<input type="checkbox"/> Surface Area	<input type="checkbox"/> Retain Fissl
<input type="checkbox"/> Hydrogen Analysis		<input type="checkbox"/> Zr-93	<input type="checkbox"/> Fuels Assay (Pu/U)	<input type="checkbox"/> pH
<input type="checkbox"/> Tritium Analysis		<input type="checkbox"/> Burnup	<input type="checkbox"/> O/H	<input type="checkbox"/> Carbon Analy
<input type="checkbox"/> Archive		<input type="checkbox"/> pH	<input type="checkbox"/> H/N/O/S/C (Metals)	<input type="checkbox"/> TC
<input type="checkbox"/> Burnup Dissolution		<input type="checkbox"/> Other	<input type="checkbox"/> Gas Chromatography	<input type="checkbox"/> TIC
<input type="checkbox"/> Other		<u>Liquid scintillation</u>		<input type="checkbox"/> TOC
		<u>cont. 2/11/92</u>		<input type="checkbox"/> C-14
<input type="checkbox"/> None				1/23/91

\* Superseded by login form rec'd by  
 BA Prentice 1/17/92 - BAP

Sample Log-In Number	Client Sample Number	In-House Sample Number	Exception	
			Analysis	Sample Type
92-02724	BO1572	1101		
2725	BO1573	1102		
2726	BO1574	1103		
2727	BO1575	1104		
2728	BO1579	1105		
2729	BO15T1	1106		
2730	BO15T3	1107		
2731	BO15T5	1108		
2732	BO15T7	1109		
2733	BO15T9	1110		
2734	BO15V1	1111		
2735	BO15V2	1112		
2736	BO15V5	1113		
2737	BO15V7	1114		
2738	BO15V8	1115		
2739	BO15V9	1116		
2740	BO15W0	1117		
2741	BO15W1	1118		
2742	BO15W2	1119		
2743	BO15W3	1120		
2744	BO18V6	1121		
2745	BO18V9	1122		
2746	BO18W2	1123		
2747	BO18W5	1124		
2748	BO18W8	1125		
2749	BO18X1	1126		
2750	BO1C38	1127		
2751	BO1C11	1128		
2752	BO1C23	1129		

LEGEND  
+ Adds specified analysis to others entered on front  
- Includes analysis on front minus the one specified  
0 Only this analysis for the sample number specified

12/20/91

142-02733- (12-02743)

14434/T1

SAMPLE RECEIPT FORM

Delivered by: P H BUTCHER Date/Time: 12/20/91 - 13<sup>15</sup>

Received by: J. H. KAYE J. H. Kaye 12/20/91

Customer Sample Number(s): B015W1, B015W2, B015W3, B015V9, B015VS

B015WD  
ALO Sample Number(s): 92-02741, 92-02742, 92-02743, 92-02745, 92-02738  
92-02740

1. Customer Chain-of-Custody Form: Present  Absent

2. Additional Shipping Forms (list):

3. Custody Seals on Shipping and/or Sample Containers and their Conditions.

Present  Absent

If Present, Condition: in tact

4. Sample Tag(s) ID Numbers if not Recorded on the Chain-of-Custody Record or on Sample Vial.

Notes: N/A

5. Condition of Shipping Container (i.e., broken container, dented, breached plastic bag, temperature of sample container as defined in Section 3.0 in PNL-ALO-051, etc.)

OK

6. Condition of Sample Vials.

in tact

7. Verification of Agreement or Monagreement of Information on Receiving Documents.

agreement

8. Resolution of Problems or Discrepancies.

RETURN COMPLETED FORM TO PROJECT MANAGER

A01-032

SAMPLE RECEIPT FORM

Delivered by: P H BUTCHER Date/Time: 12/20/91 - 1315

Received by: J. H. KAYE J. H. Kaye 12/20/91

Customer Sample Number(s): BO1BW5, BO1C25, BO1BW2, BO1BW5,  
BO1BX1, BO1BV6, BO1C11, BO1BV9, BO1C35  
ALO Sample Number(s): 92-02749, 92-02752, 92-02746, 92-02747,  
92-02749, 92-02744, 92-02751, 92-02745, 92-02750

1. Customer Chain-of-Custody Form: Present  Absent \_\_\_\_\_

2. Additional Shipping Forms (list): ALL Request Form  
ARF # 19432-2 BAP into 1/16/92

3. Custody Seals on Shipping and/or Sample Containers and their Conditions.  
Present  Absent \_\_\_\_\_

If Present, Condition: OK

4. Sample Tag(s) ID Numbers if not Recorded on the Chain-of-Custody Record or on Sample Vial.

Notes: N/A

5. Condition of Shipping Container (i.e., broken container, dented, breached plastic bag, temperature of sample container as defined in Section 3.0 in PNL-ALO-051, etc.) OK

6. Condition of Sample Vials. OK

7. Verification of Agreement or Nonagreement of Information on Receiving Documents. OK

8. Resolution of Problems or Discrepancies. N/A

RETURN COMPLETED FORM TO PROJECT MANAGER

SAMPLE RECEIPT FORM

Delivered by: P. H. Butcher Date/Time: 12/20/91 - 1335

Received by: J. H. Kaye J. H. Kaye 12/20/91

Customer Sample Number(s): 301571, 301573, 301575, 301574

301559, 301572  
ALO Sample Number(s): 92-02729, 92-02725, 92-02726,  
92-02728, 92-02724

- 1. Customer Chain-of-Custody Form: Present  Absent \_\_\_\_\_
- 2. Additional Shipping Forms (list):
- 3. Custody Seals on Shipping and/or Sample Containers and their Conditions.  
Present  Absent \_\_\_\_\_  
If Present, Condition: OK
- 4. Sample Tag(s) ID Numbers if not Recorded on the Chain-of-Custody Record or on Sample Vial.  
Notes: N/A
- 5. Condition of Shipping Container (i.e., broken container, dented, breached plastic bag, temperature of sample container as defined in Section 3.0 in PNL-ALO-051, etc.)  
OK
- 6. Condition of Sample Vials.  
OK
- 7. Verification of Agreement or Nonagreement of Information on Receiving Documents.  
OK
- 8. Resolution of Problems or Discrepancies.

RETURN COMPLETED FORM TO PROJECT MANAGER

SAMPLE RECEIPT FORM

Delivered by: PH BUTCHER Date/Time: 12/20/91 - 1345

Received by: J. H. KAYE

Customer Sample Number(s): B015T3, B015T5, B015T9, B015V3, B015V5  
B015V7, B015V1, B015T7

ALO Sample Number(s): 92-02730, 92-02731, 92-02733, 92-02735, 92-02736  
92-02737, 92-02734, 92-02732

1. Customer Chain-of-Custody Form: Present  Absent

2. Additional Shipping Forms (list): ACL Request Form

ARF#19432-1 - BAPhoto 1/16/92

3. Custody Seals on Shipping and/or Sample Containers and their Conditions.

Present  Absent

If Present, Condition: OK

4. Sample Tag(s) ID Numbers if not Recorded on the Chain-of-Custody Record or on Sample Vial.

Notes: N/A

5. Condition of Shipping Container (i.e., broken container, dented, breached plastic bag, temperature of sample container as defined in Section 3.0 in PNL-ALO-051, etc.)

OK

6. Condition of Sample Vials.

in tact

7. Verification of Agreement or Nonagreement of Information on Receiving Documents.

agreement

8. Resolution of Problems or Discrepancies.

RETURN COMPLETED FORM TO PROJECT MANAGER

J. H. Kaye 12/20/91

ALO CHAIN OF CUSTODY

<u>92-02724</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01572</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Stuber Jr</u>		<u>1-15-92</u> DATE
RECEIVER <u>EJ Wipe</u>		<u>1/15/92</u> DATE

<u>92-02725</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01573</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Stuber Jr</u>		<u>1-15-92</u> DATE
RECEIVER <u>EJ Wipe</u>		<u>1/15/92</u> DATE

<u>92-02726</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01574</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Stuber Jr</u>		<u>1-15-92</u> DATE
RECEIVER <u>EJ Wipe</u>		<u>1/15/92</u> DATE

<u>92-02727</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01575</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Stuber Jr</u>		<u>1-15-92</u> DATE
RECEIVER <u>EJ Wipe</u>		<u>1/15/92</u> DATE

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 Copy - Sender  
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Applicable Test Instruction

ALO CHAIN OF CUSTODY

1914 2/17/1

*S Robert H. Williams 1-15-92 4-2 Robert H. Williams 1-15-92*

<u>92-02728</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01579</u> SAMPLE DESCRIPTION
SENDER <u><i>Robert H. Williams</i></u>		<u>1-15-92</u> DATE
RECEIVER <u><i>E. J. Wynn</i></u>		<u>1/15/92</u> DATE

<u>92-02729</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01571</u> SAMPLE DESCRIPTION
SENDER <u><i>Robert H. Williams</i></u>		<u>1-15-92</u> DATE
RECEIVER <u><i>E. J. Wynn</i></u>		<u>1/15/92</u> DATE

*T Robert H. Williams 1-15-92 9-2 Robert H. Williams 1-15-92*

<u>92-02730</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015T3</u> SAMPLE DESCRIPTION
SENDER <u><i>Robert H. Williams</i></u>		<u>1-15-92</u> DATE
RECEIVER <u><i>E. J. Wynn</i></u>		<u>1/15/92</u> DATE

<u>92-02731</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015T5</u> SAMPLE DESCRIPTION
SENDER <u><i>Robert H. Williams</i></u>		<u>1-15-92</u> DATE
RECEIVER <u><i>E. J. Wynn</i></u>		<u>1/15/92</u> DATE

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Applicable Test Instruction

ALO CHAIN OF CUSTODY

<u>92-02732</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015T7</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Sullivan</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. H. Wipe</u>		<u>1/15/92</u> DATE

<u>92-02733</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015T9</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Sullivan</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. H. Wipe</u>		<u>1/15/92</u> DATE

<u>92-02734</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015V1</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Sullivan</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. H. Wipe</u>		<u>1/15/92</u> DATE

<u>92-02735</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015V3</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Sullivan</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. H. Wipe</u>		<u>1/15/92</u> DATE

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Applicable Test Instruction

ALO CHAIN OF CUSTODY

<u>92-02736</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015V5</u> SAMPLE DESCRIPTION
SENDER <u>Robert M. Stulen</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. J. Wipe</u>		<u>1/15/92</u> DATE

<u>92-02737</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015V7</u> SAMPLE DESCRIPTION
SENDER <u>Robert M. Stulen</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. J. Wipe</u>		<u>1/15/92</u> DATE

<u>92-02738</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015V8</u> SAMPLE DESCRIPTION
SENDER <u>Robert M. Stulen</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. J. Wipe</u>		<u>1/15/92</u> DATE

<u>92-02739</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015V9</u> SAMPLE DESCRIPTION
SENDER <u>Robert M. Stulen</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. J. Wipe</u>		<u>1/15/92</u> DATE

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Applicable Test Instruction

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ALO CHAIN OF CUSTODY

<u>92-02740</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015W0</u> SAMPLE DESCRIPTION
SENDER <u>Robert J. Stulen J</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. N. Nye</u>		<u>1/15/92</u> DATE

<u>92-02741</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015W1</u> SAMPLE DESCRIPTION
SENDER <u>Robert J. Stulen J</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. N. Nye</u>		<u>1/15/92</u> DATE

<u>92-02742</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015W2</u> SAMPLE DESCRIPTION
SENDER <u>Robert J. Stulen J</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. N. Nye</u>		<u>1/15/92</u> DATE

<u>92-02743</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B015W3</u> SAMPLE DESCRIPTION
SENDER <u>Robert J. Stulen J</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. N. Nye</u>		<u>1/15/92</u> DATE

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Applicable Test Instruction

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ALO CHAIN OF CUSTODY

<u>92-02744</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01BV6</u> SAMPLE DESCRIPTION
SENDER <u>Robert D. Strickland</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. Wayne</u>		<u>1/15/92</u> DATE

<u>92-02745</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01BV9</u> SAMPLE DESCRIPTION
SENDER <u>Robert D. Strickland</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. Wayne</u>		<u>1/15/92</u> DATE

<u>92-02746</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01BW2</u> SAMPLE DESCRIPTION
SENDER <u>Robert D. Strickland</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. Wayne</u>		<u>1/15/92</u> DATE

<u>92-02747</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01BW5</u> SAMPLE DESCRIPTION
SENDER <u>Robert D. Strickland</u>		<u>1-15-92</u> DATE
RECEIVER <u>E. Wayne</u>		<u>1/15/92</u> DATE

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Applicable Test Instruction

ALO CHAIN OF CUSTODY

<u>92-02748</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01BW8</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Stuber</u>		<u>5 Robert A. Stuber 11-15-92</u> <u>1-18-92</u> DATE
RECEIVER <u>EJ Noye</u>		<u>1/15/92</u> DATE

<u>92-02749</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01BX1</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Stuber</u>		<u>5 Robert A. Stuber 11-15-92</u> <u>1-18-92</u> DATE
RECEIVER <u>EJ Noye</u>		<u>1/15/92</u> DATE

<u>92-02750</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01C38</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Stuber</u>		<u>5 Robert A. Stuber 11-15-92</u> <u>1-18-92</u> DATE
RECEIVER <u>EJ Noye</u>		<u>1/15/92</u> DATE

<u>92-02751</u> ALO SAMPLE NUMBER	<u>ICP MS</u> ANALYSIS REQUESTED	<u>B01C11</u> SAMPLE DESCRIPTION
SENDER <u>Robert A. Stuber</u>		<u>5 Robert A. Stuber 11-15-92</u> <u>1-18-92</u> DATE
RECEIVER <u>EJ Noye</u>		<u>1/15/92</u> DATE

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Applicable Test Instruction



ALO CHAIN OF CUSTODY

<u>92-02724</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B01572</u> SAMPLE DESCRIPTION
<u>Robert J. Miller</u> SENDER		<u>2-11-92 Robert J. Miller</u> 2-11-92 <del>1-15-92</del> DATE
<u>Elwood Lopez</u> RECEIVER		<u>2/11/92</u> DATE

<u>92-02725</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B01573</u> SAMPLE DESCRIPTION
<u>Robert J. Miller</u> SENDER		<u>2-11-92 Robert J. Miller</u> 2-11-92 <del>1-15-92</del> DATE
<u>Elwood Lopez</u> RECEIVER		<u>2/11/92</u> DATE

<u>92-02726</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B01574</u> SAMPLE DESCRIPTION
<u>Robert J. Miller</u> SENDER		<u>2-11-92 Robert J. Miller</u> 2-11-92 <del>1-15-92</del> DATE
<u>Elwood Lopez</u> RECEIVER		<u>2/11/92</u> DATE

<u>92-02727</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B01575</u> SAMPLE DESCRIPTION
<u>Robert J. Miller</u> SENDER		<u>2-11-92 Robert J. Miller</u> 2-11-92 <del>1-15-92</del> DATE
<u>Elwood Lopez</u> RECEIVER		<u>2/11/92</u> DATE

Original - Project Management Office  
 Copy - Sender  
 Copy - Receiver

Applicable Test Instruction

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ALO CHAIN OF CUSTODY

<u>92-02728</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B01579</u> SAMPLE DESCRIPTION
SENDER <u>Sandra Faddy</u>		<u>1/20/92</u> DATE
RECEIVER <u>Elwood Kessel</u>		<u>1/28/92</u> DATE

500ml skt 1/28/92

S Robert J. ... 1-15-92

<u>92-02729</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015X1</u> SAMPLE DESCRIPTION
SENDER <u>Sandra Faddy</u>		<u>1/28/92</u> DATE
RECEIVER <u>Elwood Kessel</u>		<u>1/28/92</u> DATE

500ml skt 1/28/92

T 161-1-11/15-92

<u>92-02730</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015T3</u> SAMPLE DESCRIPTION
SENDER <u>Sandra Faddy</u>		<u>1/28/92</u> DATE
RECEIVER <u>Elwood Kessel</u>		<u>1/28/92</u> DATE

500ml skt 1/28/92

<u>92-02731</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015T5</u> SAMPLE DESCRIPTION
SENDER <u>Sandra Faddy</u>		<u>1/28/92</u> DATE
RECEIVER <u>Elwood Kessel</u>		<u>1/28/92</u> DATE

500ml skt 1/28/92

Original - Project Management Office  
 Copy - Sender  
 Copy - Receiver

Applicable Test Instruction

1/28/92

19432171

ALO CHAIN OF CUSTODY

<u>92-02732</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015T7</u> SAMPLE DESCRIPTION
SENDER <u>Sandra Fadoff</u>		<u>1/20/92</u> DATE
RECEIVER <u>Elwood Kessel</u>		<u>1/28/92</u> DATE

500 ml sk 7 1/28/92

<u>92-02733</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015T9</u> SAMPLE DESCRIPTION
SENDER <u>Sandra Fadoff</u>		<u>1/20/92</u> DATE
RECEIVER <u>Elwood Kessel</u>		<u>1/28/92</u> DATE

500 ml sk 7 1/28/92

<u>92-02734</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015V1</u> SAMPLE DESCRIPTION
SENDER <u>Sandra Fadoff</u>		<u>1/20/92</u> DATE
RECEIVER <u>Elwood Kessel</u>		<u>1/28/92</u> DATE

500 ml sk 7 1/28/92

<u>92-02735</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015V3</u> SAMPLE DESCRIPTION
SENDER <u>Sandra Fadoff</u>		<u>1/28/92</u> DATE
RECEIVER <u>Elwood Kessel</u>		<u>1/28/92</u> DATE

2 x 500ml sk 7 1/28/92

Original - Project Management Office  
 Copy - Sender  
 Copy - Receiver

Applicable Test Instruction

<u>92-02736</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015V5</u> SAMPLE DESCRIPTION
SENDER <u>[Signature]</u>		<u>2-11-92</u> DATE
RECEIVER <u>[Signature]</u>		<u>2/11/92</u> DATE

<u>92-02737</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015V7</u> SAMPLE DESCRIPTION
SENDER <u>[Signature]</u>		<u>2-11-92</u> DATE
RECEIVER <u>[Signature]</u>		<u>2/11/92</u> DATE

<u>92-02738</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015V8</u> SAMPLE DESCRIPTION
SENDER <u>[Signature]</u>		<u>2-11-92</u> DATE
RECEIVER <u>[Signature]</u>		<u>2/11/92</u> DATE

<u>92-02739</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015V9</u> SAMPLE DESCRIPTION
SENDER <u>[Signature]</u>		<u>2-11-92</u> DATE
RECEIVER <u>[Signature]</u>		<u>2/11/92</u> DATE

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Applicable Test Instruction

ALO CHAIN OF CUSTODY

<u>92-02740</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015W0</u> SAMPLE DESCRIPTION
SENDER <u>GG Brodaczynski</u>		<u>2-14-92</u> DATE
RECEIVER <u>SA Lepel</u>		<u>2/14/92</u> DATE

<u>92-02741</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015W1</u> SAMPLE DESCRIPTION
SENDER <u>GG Brodaczynski</u>		<u>2-14-92</u> DATE
RECEIVER <u>SA Lepel</u>		<u>2/14/92</u> DATE

<u>92-02742</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015W2</u> SAMPLE DESCRIPTION
SENDER <u>GG Brodaczynski</u>		<u>2-14-92</u> DATE
RECEIVER <u>SA Lepel</u>		<u>2/14/92</u> DATE

<u>92-02743</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>B015W3</u> SAMPLE DESCRIPTION
SENDER <u>GG Brodaczynski</u>		<u>2-14-92</u> DATE
RECEIVER <u>SA Lepel</u>		<u>2/14/92</u> DATE

Original - Project Management Office  
Copy - Sender  
Copy - Receiver

Applicable Test Instruction

ALO CHAIN OF CUSTODY

<u>92-02744</u>	<u>GEA</u>	<u>B01BV6</u>
ALO SAMPLE NUMBER	ANALYSIS REQUESTED	SAMPLE DESCRIPTION
SENDER <u>GH Brodaczynski</u>		<u>2-14-92</u>
		DATE
RECEIVER <u>SA Lopez</u>	<u>88</u>	<u>2-14-92</u>
		DATE
	<u>2-14-92</u>	

<u>92-02745</u>	<u>GEA</u>	<u>B01BV9</u>
ALO SAMPLE NUMBER	ANALYSIS REQUESTED	SAMPLE DESCRIPTION
SENDER <u>GH Brodaczynski</u>		<u>2-14-92</u>
		DATE
RECEIVER <u>SA Lopez</u>		<u>2-14-92</u>
		DATE

<u>92-02746</u>	<u>GEA</u>	<u>B01BW2</u>
ALO SAMPLE NUMBER	ANALYSIS REQUESTED	SAMPLE DESCRIPTION
SENDER <u>GH Brodaczynski</u>		<u>2-14-92</u>
		DATE
RECEIVER <u>SA Lopez</u>		<u>2-14-92</u>
		DATE

<u>92-02747</u>	<u>GEA</u>	<u>B01BW5</u>
ALO SAMPLE NUMBER	ANALYSIS REQUESTED	SAMPLE DESCRIPTION
SENDER <u>GH Brodaczynski</u>		<u>2-14-92</u>
		DATE
RECEIVER <u>SA Lopez</u>		<u>2-14-92</u>
		DATE

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 Copy - Receiver

Applicable Test Instruction

<u>92-02748</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>BO1BW8</u> SAMPLE DESCRIPTION
SENDER <u>GH Brodaszynski</u>		<u>2-14-92</u> DATE
RECEIVER <u>SA Lopez</u>		<u>2-14-92</u> DATE

<u>92-02749</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>BO1BX1</u> SAMPLE DESCRIPTION
SENDER <u>GH Brodaszynski</u>		<u>2-14-92</u> DATE
RECEIVER <u>SA Lopez</u>		<u>2/14/92</u> DATE

<u>92-02750</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>BO1C38</u> SAMPLE DESCRIPTION
SENDER <u>GH Brodaszynski</u>		<u>2-14-92</u> DATE
RECEIVER <u>SA Lopez</u>		<u>2/14/92</u> DATE

<u>92-02751</u> ALO SAMPLE NUMBER	<u>GEA</u> ANALYSIS REQUESTED	<u>BO1C11</u> SAMPLE DESCRIPTION
SENDER <u>GH Brodaszynski</u>		<u>2-14-92</u> DATE
RECEIVER <u>SA Lopez</u>		<u>2-14-92</u> DATE

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Copy - Sender  
Copy - Receiver

Applicable Test Instruction



INTERIM CHANGE NOTICE  
(ICN)

~~CONTROLLED DOCUMENT~~

~~COPY NO. 000003~~

ICN- PNL-ALO-105.1

Page 1 of 1

<p>A. Document Number: <u>PNL-ALO-105</u> Revision Number: <u>0</u>                  Document Title: <u>Procedure for Preparation of Samples to be counted Document's by Gamma-Ray Spectroscopy</u>                  Original Author: <u>E. A. Lepel</u></p>	<p>Effective Date of ICN: <u>4/26/91</u>                  Change Requested By: <u>E. A. Lepel</u></p>
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B. Action:

Please destroy old procedure and replace with the procedure attached.

C. Effect of Change:

To allow for more efficient use of manpower when handling radioactive samples. Additionally, reduced personnel exposure and reduced likelihood of workplace contamination would result.

D. Reason for Change/Description of Change

To allow for more efficient use of manpower when handling radioactive samples. Additionally, reduced personnel exposure and reduced likelihood of workplace contamination would result.

Description of Change: See attached

<p>E. Approval Signatures (Please Sign and Date)</p> <p>Process Quality OSER Department</p> <p>Concurrence: <u>[Signature]</u> <u>DF</u></p> <p>Approval Authority: <u>[Signature]</u></p> <p>Other Approvals: <u>[Signature]</u></p> <p><u>B. M. Dilligan</u></p>	<p>Type of Change: (Check ( / ) one)</p> <p>( ) Minor Change      <input checked="" type="checkbox"/> Major Change</p> <p>Date: <u>4/10/91</u></p> <p>Date: <u>4/12/91</u></p> <p>Date: <u>4/12/91</u></p> <p>Date: <u>4/15/91</u></p> <p><b>A03-001</b></p>
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PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-105, PROCEDURE FOR PREPARATION OF SAMPLES TO BE COUNTED BY GAMMA-RAY SPECTROSCOPY

SCOPE

This procedure shall be used to prepare samples for gamma energy counting on germanium spectrometers (See PNL-ALO-464). The samples may be liquids, solids (soils, sediments, etc.), air filters, vegetation, or other miscellaneous samples. This procedure was developed by PNL Scientists for the preparation of samples to be counted by gamma-ray spectroscopy.

APPLICABILITY

This procedure covers the preparation of samples to be counted in the 329 Laboratory Gamma-Ray Spectroscopy facilities.

DEFINITIONS/ACRONYMS

- ARF - Analytical Request Form
LRB - Laboratory Record Book
LTC - Large Tuna Can
STC - Small Tuna Can
TI - Test Instruction

RESPONSIBLE STAFF

Counting Room Manager
Cognizant Scientist/Analyst

PROCEDURE

1.0 Tolerances

Tolerances for all measurements made during an analysis shall be specified in the following manner: 1) A tolerance limit can be stated

Table with 4 rows and 5 columns containing metadata: Author (EA Lepel), Project Mgr. (BM Gillespie), QAD Representative (GK Gerke), Technical Reviewer (KH Abel), Line Mgr. (JM Latkovich), Procedure No. (PNL-ALO-105), Revision No. (0 A03-002), Effective Date (SEP 26 1990), Page (1 of 7).

## PNL TECHNICAL PROCEDURE

with a measurement value given in a method, or 2) if a tolerance limit is not stated with a measurement value, then the following system of tolerances shall be in effect:

- a. When two or more significant figures are specified, the tolerance limit is  $\pm 5$  in the next digit beyond the last one stated. For example, 5.0 mL means  $5.0 \pm 0.05$  mL; 450 g means  $450 \pm 5$  g; 369 mL means  $369.0 \pm 0.5$  mL.
- b. If a single significant figure is specified, the actual measurement shall be within  $\pm 5\%$  of the stated value. For example, 20 mL means a volume between 19 and 21 mL.

### 2.0 Equipment and Materials

Balances - Mettler PC4400 electronic balance. Dual range balance of 0.01-400.00 g and 1.0-4000.0 g

Large tuna can sealer

Small tuna can sealer

Drying oven

Hot plate

Mortar and pestle

Assorted polyethylene bottles

White plastic tape

Large tuna can (LTC) made of aluminum, 5.9-cm dia x 2.8-cm height, 220 g usable capacity

Small tuna can (STC) made of aluminum, 3.4-cm dia x 2.4-cm height, 80 g usable capacity

Shatterbox to pulverize rock chips into a fine powder. Made by SPEX Industries Inc., Mutuchen, NJ

Assorted volumetric glassware and beakers.

Dessicator and dessicant

### 3.0 Sample Preparation Procedures

The large variety of samples that have been processed in the Low-Level Counting Rooms have resulted in a number of "standard" sample geometries. The number of sample geometries have been minimized because each geometry requires calibration of that detector system. Typical sample geometries are:

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-105	0	SEP 26 1990	2	7

A03-003

PNL TECHNICAL PROCEDURE

TABLE 1: Typical Geometries Used in Gamma Counting

Geometry	Containment
1) Sealed large tuna can (LTC) $\leq 220$ g (solid)	sealed LTC, sealed in a poly bag
2) Sealed small tuna can (STC) $\leq 80$ g (solid)	sealed STC, sealed in a poly bag
3) 125-ml poly bottle - 10,20,50,100 ml (liquid)	screw cap, tape, sealed in poly bag
4) 500-ml poly bottle - 500 ml (liquid)	screw cap, tape, sealed in poly bag
5) 9-oz jar - 50,100,150,200 ml (liquid or solid)	screw cap, tape, sealed in poly bag
6) 1-in. x 6-in. petri dish (solid)	tape, sealed in poly bag
7) Point source on card	tape on both sides, (plastic bag)
8) Scintillation vial - 1,5,10,20 ml (liquid)	screw cap, tape, sealed in poly bag
9) 2-dram poly vial (solid or liquid)	heat seal vial and poly bag
10) 2/5,2/27 and 2-dram poly vial (solid or liquid)	snap cap heat sealed, in poly bag
11) Marinelli beaker - 1 L, 2 L (liquid or solid)	tape, poly bag
12) Filter - 47 mm (solid)	heat-sealed in poly bag
13) 3-in. dia puck, 1/2-in. thick (1/2" x 3") (solid)	PVC ring, tape, mylar or poly bag

The choice of a specific geometry is based on the type of sample (whether solid or liquid), the amount of sample available, whether that geometry has been calibrated for that type of sample, and the amount of sample preparation required to get the sample into a specific geometry. The sample geometry is determined for each sample by the cognizant staff member/analyst. For some kinds of samples, little or no sample preparation is required before counting a sample.

### 3.1 Liquid Samples

Liquid samples may be counted directly by putting the appropriate amount of liquid into the correct container. In addition, liquid samples may result from other chemical preparation or concentration procedures. In that case, it is assumed that a procedure is in effect to cover those additional steps.

Procedure No. PNL-ALO-105	Revision No. 0	Effective Date SEP 26 1990	Page 3	of 7
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A03-004

## PNL TECHNICAL PROCEDURE

- 3.1.1 Choose the appropriate sample geometry (container) from Table 1 based on the volume and type of liquid available and accuracy required.
- 3.1.2 Using clean containers, transfer the appropriate volume by weighing into the chosen container. The sample volume shall be one of the volumes listed in Table 1 associated with the selected container geometry unless otherwise specified in a Test Instruction (TI), Analytical Request Form (ARF), or other documentation from the client. Seal the container with the lid, wrap with plastic tape, and label with sample identification (ID).
- 3.1.3 Seal the sample and container into a plastic bag to prevent cross contamination and contamination of the counting cave.
- 3.1.4 Record in the LRB the sample ID, sample geometry chosen, weight and volumes taken, and balance number.
- 3.1.5 Sample is ready for gamma counting (see PNL-MA-599, PNL-ALO-464). Transfer the sample to the appropriate storage cabinet in the Counting Rooms (Rooms 13C, 14C, 4D, 329 Building) to await counting.

### 3.2 Solid Samples

Solid samples, such as soils, sediments, rocks, gravel, resins, etc., may be counted without any sample preparation. Depending on the degree of accuracy of the analysis and the concern with respect to homogeneity of the sample, the samples may be reduced to a fine powder using the SPEX shatterbox. In both cases, a geometry is chosen from Table 1.

After the geometry has been chosen, the non-radioactive sample must be dried before taking an aliquot for counting. Equivalently, a small aliquot (2-5 g) may be used to determine the moisture content while an undried aliquot is counted. If this is done, the data obtained must be corrected to reflect "Dry Weight." Generally, it is easier to dry the total amount of material first and then take an aliquot for counting.

However, for radioactive samples (especially hotter samples), it is best to take an aliquot and measure the sample as received. An additional small aliquot may be used to determine the moisture content. From the standpoint of personnel exposure and ALARA, this would be preferred method for radioactive samples.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-105	0	SEP 26 1990	4	7

A03-005

PNL TECHNICAL PROCEDURE

3.2.1 Choose the appropriate counting geometry (container) based on the type and size of the sample, and the accuracy required. Record choice in LRB.

3.2.2 Determine the moisture content of the sample. If the dried sample is nonradioactive, the whole sample may be dried or equivalently a small aliquot (2-5 g) may be dried. If the sample is radioactive, a small aliquot (2-5 g) may be used for the moisture determination.

Put an aliquot of the sample into a tared container and determine the WET weight. Record weight and balance number in LRB. Place the container in the drying oven for about 16 hr at about 110°C. If the sample is dry, remove it and let it cool to room temperature in a desiccator. Determine the DRY weight and record in LRB. An unstable balance display is indicative of: a) a container that hasn't cooled to ambient temperature, or b) hydrostatic changes in the sample. If this is the case, place the sample back into the drying oven and dry for at least 1 hour (maximum of 24 hours). Let the sample cool to room temperature in a desiccator, and then reweigh. If the difference in sample weights is  $\leq 0.01$  g between the first and second weighings, constant weight has been achieved. The final dry weight obtained is the dry weight of the sample. If not, repeat the drying process until constant weight is achieved. Transfer the sample to a container for storage and label with sample ID if not doing the next step.

3.2.3 If requested by TI, ARF, or documentation from the client, and for non-radioactive samples only, pulverize the sample to a fine powder using the SPEX Shatterbox. Take an aliquot of sample and place into the sample chamber. Run the Shatterbox for 2-10 min to produce a fine powder. Transfer the powder to a container for storage and label.

3.2.4 Transfer a known amount of dried solid to the container in which the analysis will be made. The container shall be chosen from Table 1. Typically, this will be the large tuna can geometry which can hold up to 220 g of dry solid material. Record in the LRB the weight of the sample and the balance used, and label the sample with the sample ID.

3.2.5 Sample is ready for gamma counting (See PNL-MA-599, PNL-ALO-464). Transfer the sample to the appropriate

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-105	0	SEP 26 1990	5	7

A03-006

## PNL TECHNICAL PROCEDURE

storage cabinet in the Counting Rooms (Rooms 13C, 14C, 4D, 329 Building) to await counting.

### 3.3 Air Filters

- 3.3.1 Choose the appropriate geometry (container) for the air filter to be counted. The 329 Roof Filters have been counted in the 1 x 6 geometry, where the 47 mm filters have been counted individually.
- 3.3.2 Prepare the sample according to the geometry chosen, and record in LRB. Seal in a plastic bag. Record sample ID on the plastic bag or sample card that accompanies the sample.
- 3.3.3 Sample is ready for gamma counting (See PNL-MA-599, PNL-ALO-464). Transfer the sample to the appropriate storage cabinet in the Counting Rooms (Rooms 13C, 14C, 4D, 329 Building) to await counting.

### 3.4 Vegetation

Vegetation samples may have minimal or extensive sample preparation prior to gamma counting. If the preparation steps are extensive, they shall be covered under an appropriate procedure supplied by the requester.

The amount of sample dictates the geometry (container) to be used. If there is plenty of sample, it can be pressed into a "Marinelli" geometry (container); otherwise it may be counted in a LTC.

- 3.4.1 Based on the amount of sample, choose the sample geometry (container) in which the samples are to be counted.
- 3.4.2 To the appropriate geometry container, add the vegetation material. Determine the net weight of the material by weighing.
- 3.4.3 Record in the LRB the geometry chosen, sample weight, and the balance identification.
- 3.4.4 Sample is ready for gamma counting (See PNL-MA-599, PNL-ALO-464). Transfer the sample to the appropriate storage cabinet in the Counting Rooms (Rooms 13C, 14C, 4D, 329 Building) to await counting.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-105	0	SEP 26 1990	6	7

A03-007

PNL TECHNICAL PROCEDURE

4.0 Records

Records will be maintained and controlled so as to conform to requirements of PAP-70-1701. Laboratory Record Books (LRBs) and Data Sheets provide a mechanism for control of most records. Laboratory Record Books will be used in accordance with the Act Now Directive 89.1.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-105	0	SEP 26 1990	7	7

A03-008

INTERIM CHANGE NOTICE  
(ICN)

ICN-PNL-ALO-106.2  
Page 1 of 1

A. Document Number: <u>PNL-ALO-106</u> Revision Number: <u>0</u> Document Title: <u>Acid Digestion for Preparation of Samples for Radiochemical Analysis</u> Document's Original Author: <u>N. L. Wynhoff</u>	Effective Date of ICN: <u>7/30/91</u> Change Requested By: <u>NL Wynhoff</u>
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B. Action:  
Replace pages 3 and 4.

C. Effect of Change:  
Allow analyst to address operational needs during the leaching procedure that the original procedure did not allow for.

D. Reason for Change/Description of Change If sample volume is reduced by evaporation, additional solids may precipitate. It may then be necessary to repeat the filtering as in previous steps. Also, it may be useful to reduce leachate volume before dilution to a known volume.  
Add the following before the last sentence in Section 4.5, "If a ppt forms, repeat Step 4.4."  
To the end of Sec. 5.7, add, "It may be useful to evaporate the leach solution to a lower volume before dilution to a known volume. If a ppt forms, filter and discard ppt"  
Add the following before the last sentence in Section 5.8, "If a ppt forms, filter and discard the ppt."

E. Approval Signatures (Please Sign and Date)	Type of Change: (Check ( / ) one) <input type="radio"/> Minor Change <input checked="" type="radio"/> Major Change
Process Quality <del>Control</del> Department Concurrence: <u>GT Gerbe RE</u> Date: <u>3/15/91</u>	
Approval Authority: <u>[Signature]</u> Date: <u>3/18/91</u>	
Other Approvals: <u>B.M. Gubzin</u> Date: <u>3/1/89</u>	
	Date: <u>1/1</u>

"TOP SECRET - FROTH ONLY"

~~CONTROLLED DOCUMENT~~  
COPY NO. \_\_\_\_\_

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-106, ACID DIGESTION FOR PREPARATION OF SAMPLES FOR RADIOCHEMICAL ANALYSIS

APPLICABILITY

The procedure provides methods for acid digestion of radionuclides in water and soil/sediment samples for analysis by alpha, beta or gamma counting. For soil/sediment analysis an acid leach is employed. This method was developed by PNL radioanalytical scientists, and is based on over 10 years experience in analyzing a variety of samples for radionuclides by alpha, beta, or gamma counting techniques.

DEFINITIONS/ACRONYMS

ml - milliliter  
um - micrometer  
ppt - precipitate  
g - grams

RESPONSIBLE STAFF

Cognizant Scientist  
Analyst/Technician

PROCEDURE

1.0 Tolerances

Tolerances for all measurements made during an analysis shall be specified in the following manner: 1) State with a measurement value given in a method or 2) as specified below if not stated with a measurement value.

- (a) Unless otherwise specified, all values for measurements stated in the methods (volume, weight, time, etc.) are approximate values.

Author	Date	Project Mgr.	Date	QAD Representative	Date
<i>W. J. ...</i>	9-21-90	<i>B. N. ...</i>	9-24-90	<i>C. ...</i>	9/24/90
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
<i>J. ...</i>	9-21-90	<i>J. ...</i>	9-21-90		
Procedure No.	Revision No.	Effective Date		Page	of
PNL-ALO-106	0	A03-01 SEP 26 1990		1	4

## PNL TECHNICAL PROCEDURE

The actual measurements used, however shall be within  $\pm 10\%$  of the stated value.

- (b) When one or more significant figures are given to the right of the decimal point, the tolerance limit is  $\pm 5$  in the next digit located beyond the last one stated.

### 2.0 Reagents, Equipment and Materials

Nitric acid - 8 M. 50 mls Type II water and 50 mls reagent grade acid or multiples thereof.

Hydrochloric acid - 6 M. 50 mls Type II water and 50 mls reagent grade acid or multiples thereof.

ASTM Type II water (ASTM D1193).

Hot plate:

Pyrex beakers, 50 ml, 250 ml (or other appropriate vessel).

Watch glasses, to fit above beakers.

Filter apparatus - appropriately sized for volume of sample filtered.

Filters - glass microfiber or membrane, 1.5  $\mu\text{m}$  effective retention or better, sized to fit above filter apparatus (or equivalent).

### 3.0 Quality Control

Quality control requirements are defined for each specific radiochemical analysis in the specific radiochemical analysis procedure. The analyst shall refer to the appropriate analysis procedure for guidance on QC requirements in that analysis. Analysis procedures are specified for a sample or group of samples by a TI or ARF. If an analysis procedure is not specified in a TI or ARF, then the cognizant scientist shall determine the appropriate procedure to be used for the requested analysis for the sample(s).

### 4.0 Water Sample Preparation

4.1 Shake sample and transfer at least 100 ml of well-mixed sample to a 250 ml glass beaker. (If more than 100 ml sample is needed to reach detectable levels, evaporate, at below boiling temperatures, the sample to about 100 ml.) Record initial sample volume.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-106	0	SEP 26 1990	2	4

A03-011

PNL TECHNICAL PROCEDURE

4.2 If directed by a cognizant scientist, add radionuclide tracer/spike as required by the requested analysis. The spike/tracer amount shall be specified by the cognizant scientist. It should be close to the expected amount of nuclide present or at the minimum, a detectable amount (3x5 times the instrument detection limit). Place the spike/tracer for blanks or standards into 100 ml of Type II water.

Note: Record exact volume, isotope and calibrated activity of tracer solution in the LRB or Data Sheet as specified in the analysis procedure(s).

4.3 Add 10 ml of 8M  $\text{HNO}_3$  to the sample. Cover with a watch glass or similar cover. Heat on a hot plate for 2-3 hours at  $100 \pm 20^\circ\text{C}$  or until sample volume is reduced to between 25 and 50 ml. Make certain sample does not boil. Cool sample to room temperature.

4.4 If visible solids are present, filter to remove insoluble material except do not filter samples if preparing samples for Sr analysis only. Samples may not be filtered for any analyses upon client request in an ARF, SOW or TI.

Note: In place of filtering, the sample, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

4.5 Adjust sample volume to meet the needs of the radionuclide separation procedure requested. If a ppt forms, repeat Step 4.4. The sample is now ready for analysis.

5.0 Soil/Sediment Sample Preparation

5.1 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 10.0 g portion of sample and transfer to a beaker. Record weight in the LRB or on the data sheet.

5.2 If directed by a cognizant scientist, add radionuclide tracer/spike as required by the requested analysis. The spike/tracer amount shall be specified by the cognizant scientist. It should be close to the expected amount of nuclide present or at the minimum, a detectable amount (3x5 times the instrument detection limit). Place the spike/tracer for blanks or standards into the 10 ml 8 M nitric acid of the next step.

Procedure No. PNL-ALO-106	Revision No. 0	Effective Date SEP 26 1990	Page 3	of 4
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A03 012

## PNL TECHNICAL PROCEDURE

Note: Record exact volume, isotope and calibrated activity of tracer solution in the LRB or on laboratory data sheets as specified in the analysis procedure.

- 5.3 Add enough 8 M  $\text{HNO}_3$  to cover the sample (about 10 ml), mix the slurry, and cover with a watch glass. Heat the sample to to  $100 \pm 20^\circ\text{C}$ ) and reflux for 10 - 30 minutes without boiling. Add 10 ml of concentrated  $\text{HNO}_3$ , replace the watch glass, and reflux for 10 - 30 minutes. Do not allow the volume to be reduced to less than 5 ml while maintaining a covering of solution over the bottom of the beaker.
- 5.4 Allow the sample to cool to room temperature. Filter. Save filtrate in a labelled beaker.
- 5.5 Return the ppt and filter to the beaker. Repeat steps 5.3 and 5.4 one time, adding filtrate to the previously saved filtrate.
- 5.6 Return the ppt and filter to the beaker. Add enough water to cover the sample, mix the slurry and cover with a watch glass. Heat sample to  $100 \pm 20^\circ\text{C}$ ) and reflux 10 - 30 minutes without boiling. Repeat step 5.4, adding filtrate to the previously saved filtrate.
- 5.7 If the entire sample leach solution will not be used for a single analysis, dilute sample to a known volume and record the volume. It may be useful to evaporate the leach solution to a lower volume before dilution to a known volume. If a ppt forms, filter and discard ppt.
- 5.8 Take aliquots for analysis and adjust the volume of these aliquots to meet the needs of the radionuclide separation requested. Aliquot volume will be determined by the cognizant scientist. Use Type II water for dilution and evaporation for volume reduction. If a ppt forms, filter and discard the ppt. The sample is now ready for analysis.

### 6.0 Records

Records will be maintained and controlled so as to conform to requirements of PNL-MA-70, PAP-70-1701. All record of weights, volumes, and spike/tracer conc./vol. shall be on Alpha or Beta Counting Data Sheets, LRBs or Sample Record log data sheets (depending on analysis requested). Laboratory Record Books will be used in accordance with the ACT NOW Directive 89-1.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-106	0	SEP 26 1980	4	4

A03-013

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-280, INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRIC (ICP-MS) ANALYSIS

APPLICABILITY

This procedure is applicable to the receiving and handling of samples, the operations of the ICP-MS VG Plasmaquad (PQ), and the reporting of analytical results. Controlling software for this instrument is VG supplied (latest version). Although some specific and general information regarding the analytical operations of the VG Plasmaquad are described below, this procedure is not written to replace VG supplied operations and PlasmaQuad instruction manuals. Specific analytical procedures are described which include sufficient information to allow a skilled operator to repeat the analyses performed using this instrument.

When this procedure is approved in accordance with PNL-MA-70, this procedure is applicable for the analysis by ICP-MS.

DEFINITIONS/ACRONYMS

MATRIX MATCH - Add to the calibrating standards any major matrix components present in the samples.

LRB - laboratory record book

AMU - atomic mass unit

RSD - Relative Standard Deviation

TUNE SOLUTION - A solution containing 100 ppb Mg, In, Pb for the purpose of tuning the instrument and establishing a mass response curve. Any solution containing 50 - 500 ppb of a low, medium, and high mass element can be used alternatively as a TUNE SOLUTION.

Author <i>Eric J. Wipe</i>	Date 9/17/90	Project Mgr. <i>B.M. Williams</i>	Date 9-19-90	QAD Representative <i>G. Coerte</i>	Date 9/19/90
Technical Reviewer <i>D.W. Koppelaar</i>	Date 9/17/90	Line Mgr. <i>A. Salder</i>	Date 9-19-90	Other	Date
Procedure No. PNL-ALO-280	Revision No. 0	Effective Date A03-014 SEP 26 1990		Page 1	of 13

PNL TECHNICAL PROCEDURE

RESPONSIBILITIES

Analyst

Cognizant Scientist

PROCEDURE

1.0 Receipt and Handling of Samples

1.1 Receiving Samples

Upon receipt, samples are logged into a Laboratory Record Book designated for that purpose. Information logged includes the number of samples, sample identification, date received and from whom, blanks and standards included (if any), and sample and/or standard preparation, if necessary. Additional information logged may include specific instructions for analysis (e.g., a memo, DSI, ARF, TI, or SOW), and the work order number, if available at the time of log-in.

1.2 Preparation

1.2.1 Dissolution

If the samples to be analyzed were received as a solid, they must be dissolved for conventional ICP-MS analysis. (This is not necessary for laser ablation ICP-MS.) Dissolution procedures vary greatly between sample types, but the chosen procedure (or designated in a TI, ARF or SOW) is always described in its entirety in the sample log-in LRB.

1.2.2 Dilution

If the elements of concern are in sufficiently high concentration (>500ppb), dilution is usually necessary. If the elements of concern are below this concentration, or are in a matrix that is expected to dramatically diminish the elements' response signal, dilution may not be necessary. Dilutions are usually made by volume (rather than by weight) with calibrated pipets; preferred ultimate matrix is 1.0% HNO<sub>3</sub>. The dilutions are described in detail in the designated LRB.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-280	0	SEP 26 1990	2	13

A03-015

PNL TECHNICAL PROCEDURE

Example: 10X dilution: 8.5 mLs 5% HNO<sub>3</sub> + 1.0 ml sample + 0.5 ml lppm In

1.2.3 Internal Standard

An appropriate internal standard (e.g., In) is added to both samples and standards for semi-quantitative as well as quantitative analyses. For quantitative analysis, it is used to correct for instrument drift and matrix effects. For semi-quantitative analysis, it is used as a reference standard. The internal standard concentration is determined by the operator based on that specific analysis, but is typically 10-200ppb. The internal standard chosen depends on the elements of concern and the sample matrix. It is crucial that the samples do not contain the internal standard selected, and that there are no interferences from other element isotopes, molecular ion species, or matrix effects. For example, In-115 would not be appropriate as an internal standard for Sn analysis, because Sn has an isotope at 115.

1.2.4 Standard Preparation

Standards chosen depend on the elements of concern, and are typically prepared with element concentrations in the same approximate range as that of the samples. For best quantitative results, at least two different concentrations of the same element shall be prepared and run to generate a calibration curve (rather than a simple ratio) for the calculation of results. Standards prepared for analysis are dilutions of NBS or other recognized primary standards, or dilutions of prepared stock solution standards traceable to said primary standards, and are listed and described in a separate Laboratory Record Book designated for that purpose. This "Standards LRB" shows the traceability of all of the standards used in analysis from how they were made (pipet calibration and stock solution preparation information) back through information regarding all of the primary standards used (Lot Number, concentration, matrix, etc.).

1.3 Procedure File and Sample Analysis Identification

When it is time for the samples to be analyzed, a procedure file is set up with a unique identification number for that analysis run. The number is four digits long, followed by a letter, and each digit has a specific meaning:

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-280	0	SEP 26 1990	3	13

A03-016

PNL TECHNICAL PROCEDURE

Example: Procedure File #8c15a

1st digit: Designates the year of analysis (1988)

2nd digit/letter: Designates the month of analysis  
(1-9 = Jan-Sep; a-c = Oct-Dec)

3rd & 4th digits: Designates the date of analysis (01-31)

5th letter: Designates the analysis procedure number (set of samples) of that date (a-g = first-seventh analysis)

6th digit: Designates the sample in that procedure (set).

For the example listed above, it is the first analysis run on December 15, 1988. Individual samples are numbered as well, meaning the sixth sample run in Procedure File #8c15a is designated 8c15a6.

The sample ALO number is cross referenced in the ICP-MS log-in LRB with the analysis procedure number (set of samples analyzed that day) and the sample in that procedure (samples in set are numbered consecutively).

2.0 Instrument Operation

The following procedures are to be used in conjunction with the VG Plasmaquad Operating and User's Manuals. The procedures below assume that the instrument is operating and functioning normally.

2.1 Instrument Maintenance

The ICP/MS shall be maintained on a daily basis. The ANALYST must ascertain that the individual instrument components are clean before beginning an analysis procedure. The following is a general description of the expected periodic maintenance (see VG Plasmaquad User's Manual):

Clean sample, skimmer cone; Daily  
torch, elbow

Clean spray chamber Weekly or biweekly (depends on sample history)

Clean ion lens stack Semi-annually/  
as needed

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-280	0	SEP 26 1990	4	13

A03-017

## PNL TECHNICAL PROCEDURE

Clean quadrupole	ONLY when necessary (See SCIENTIST)
Change roughing pump oil	Semi-annually
Change channeltron	As needed

### 2.2 Tuning the Instrument

The ICP/MS is tuned by aspirating the TUNE SOLUTION and manually adjusting the lens voltages for optimal sensitivity. The element in the TUNE SOLUTION having the middle mass (e.g., indium at 115 amu) is typically tuned first, with the low and high masses being tuned subsequently to obtain approximately equivalent signal response for each analyte mass.

#### 2.2.1 Instrument Response and Precision Check: TUNEIR

The instrument response and precision are evaluated by running the TUNE SOLUTION five consecutive times using the isotope ratio procedure TUNEIR in IR SCAN found under SCAN ACQUIRE. TUNEIR hard copies must be signed and dated by the ANALYST, and logged in a notebook designated for that purpose. (NOTE: The TUNEIR is not a calibration, but rather an indication of the instrument's sensitivity, stability, and overall performance. The maintenance of these records are helpful in reviewing trends in performance over a period of time.)

### 2.3 Program Descriptions

Numerous programs and their subroutines are used for analysis, depending on the type of results the operator or sponsor wishes to obtain. A brief description of each program and its uses are described below.

#### 2.3.1 Element Menu Definition Program

This program is used to define the elements (isotopes) to be analyzed, either for isotope ratio or multi-element work. This program also defines the run conditions for acquiring the data, either range scanning or peak jumping. Follow VG PlasmaQuad Software Manual to select the elements of interest.

Procedure No. PNL-ALO-280	Revision No. 0	Effective Date A03-018 SEP 26 1990	Page 5	of 13
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## PNL TECHNICAL PROCEDURE

The Element Menu Program also sets scan parameters such as the number of channels, sweeps, length of dwell time, and mass regions which will be skipped.

### 2.3.2 Multi Element Analysis Procedure Definition Program

This Program defines the order and type of samples to be run. Constructing and upgrading procedures are clearly described in the VG PlasmaQuad Software Manual. Standards, their preparation and their certification shall be documented in the LRB.

### 2.3.3 Isotope Ratio Analysis Procedure Definition Program

This program is identical to the multi-element analysis program with the exception that the number of ratios to be performed is requested instead of the Conc File name.

The measurement of isotopic ratios on the VG PlasmaQuad shall be carried out in a similar manner to quantitative analysis, i.e., the sample shall be compared to a well defined isotopic standard. The standards, their preparation and their certification shall be documented in the LRB. Specific details of the isotopic ratio program procedures are described in the VG PlasmaQuad Software Manual.

### 2.3.4 Acquire Programs

Three acquisition programs are addressed through Survey Acquire, Scan Acquire and Peakjump Acquire programs.

#### 2.3.4.1 Survey Acquire

Survey Acquire is typically used for short qualitative or semi-quantitative analyses. Scan parameters can be altered, but neither an element menu nor an element procedure is necessary for survey acquire.

Semi-quantitative calculations are also performed under survey acquire. For semi-quantitative analysis, an internal standard is added to each sample; calculations are performed based on the concentration of the internal standard.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-280	0	SEP 26 1990	6	13

A03 019

## PNL TECHNICAL PROCEDURE

Mass calibration is also performed under survey acquire. See 2.3.7 for details.

### 2.3.4.2 Scan Acquire

Scan Acquire is used when a common element menu and element procedure is desired for running a consecutive series of samples. An element procedure is necessary for quantitative analysis; scan acquire is therefore the most efficient acquire program to use for this capability in that an element procedure is requisite for acquisition.

### 2.3.4.3 Peakjump Acquire

Peakjumping Acquire does not scan, but as the name implies, it jumps from pre-selected isotopes and spends a pre-selected dwell on each individual isotope mass. This method of acquiring is useful when isotopes of interest are spaced across a wide mass range. Longer dwell per amu and fast acquisition times make peakjumping ideal for small sample volumes and for minimizing detection limits.

### 2.3.5 Calculations Program

With the exception of semi-quantitative calculations (see 2.3.4.1), the calculations program includes all calculation types whether they be multi-element, isotope ratio, standard addition, or isotope dilution.

#### 2.3.5.1 Multi-Element Calculation Program

When performing multi-element calculations, appropriate response files must be used. For very precise and accurate data output, calibration curves which incorporate standard concentrations in the range of the sample concentrations are necessary. In addition, when samples have significant amounts of dissolved solids (e.g., brines), standards which are in similar matrices must be used to account for ionization factors, oxide interferences, and sample introduction (nebulization) efficiencies.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-280	0	SEP 26 1990	7	13

A03-020

PNL TECHNICAL PROCEDURE

2.3.5.2 Isotope Ratio Calculation Program

Isotope ratio calculations must include standards which have certifiable isotopic abundance ratios. Interference corrections must be considered when sample matrices are highly variable. All sample isotopic concentrations must also be in the range of the standard concentrations used in order to account for the dead time differences between predominant versus less predominant isotopes. When considering low mass isotopes, it is very important to consider the quadrupole sensitivity. In general, the response curves near the low and high masses can be checked using standards.

2.3.5.3 Standard Addition Program

The standard addition program enables sample concentrations from single isotopes to be calculated using the standard addition technique. If quantitative analysis cannot be achieved directly, then using the qualitative values, process samples by standard addition.

2.3.5.4 Isotope Dilution Program

In this calculation, a specific isotope of an element is added to the sample. Knowing the concentration of this isotope and assuming normal isotopic abundances of the element(s) of concern, the concentrations are calculated. This is a very accurate method for determining elemental concentrations because quenching effects, interference corrections, and unforeseen matrix effects can be accounted for. In the event samples do not have compositions with normal isotopic ratios, isotopic abundances of each analyte of interest must first be determined.

2.3.6 Utility Program

The system utilities are designed to simplify the use of the data system, to provide flexibility in data manipulation, and to enable the operator to define this system configuration. Spectral interference corrections are input through the utilities program via Edit Database. Data

Procedure No. PNL-ALO-280	Revision No. 0	Effective Date SEP 26 1990	Page 8	of 13
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A03-021

## PNL TECHNICAL PROCEDURE

manipulation and system configuration procedures are detailed in the VG PlasmaQuad Software Manual. Because spectral interferences other than isobaric interferences can be incorporated by using the Edit Database option, it is important that the ANALYST qualifies these "special" interferences in the final report (Section 3).

### 2.3.7 Mass Calibration

Before the results are calculated, the ANALYST must first review a sample spectrum to ensure that the peaks observed correspond to their respective masses, i.e., that the instrument is properly mass calibrated. Mass calibration is performed under Survey Acquire, and should be done if the peaks observed appear to be shifted from center mass. If the instrument is operating properly, mass calibration should not have to be necessary on a regular basis, but rather only periodically; it should be done if more than 0.5 amu off peak center in accordance with the VG PlasmaQuad Software Manual.

Mass calibration is usually performed only when the instrument has been cleaned or repaired. Documentation of instrument maintenance is in the instrument maintenance log. The new mass calibration parameters are written to file and also are in each sample analysis file and printed as a part of the header for each sample file.

### 3.0 ANALYSIS PROCEDURES

- 3.1 Tune machine as described in 2.2. An instrument response of less than 1 million counts/second/ppm is not acceptable for analysis. The instrument must be shutdown, cleaned and/or adjusted for better sensitivity.
- 3.2 In the Procedure Definition Program, call Element Menu and define elements and their isotopes for analysis. If qualitative scan is desired, call survey acquire and proceed directly to Section 3.5.
- 3.3 In the Procedure Definition Program, call Multi-Element Procedure Definition (ME PROC DEF) for elemental analysis; call Isotope Ratio Procedure Definition (IR PROC DEF) if isotopic ratio analysis is to be performed.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-280	0	SEP 26 1990	9	13

A03-022

PNL TECHNICAL PROCEDURE

3.3.1 Multi-Element Procedure

Define Blanks, Standards, and Samples with uniquely identifiable names, as described in 1.3. Define the element file (defined in Element Menu Program). For standards, enter the appropriate concentrations for the standard's elements and create the Response File. If an internal standard is to be used, input the element symbol and mass of the internal standard. The internal standard, if used, must be present in all solutions at the same concentration.

3.3.2 Isotope Ratio Procedure

Define Blanks, Standards, and Samples with uniquely identifiable names, as described in 1.3. Define the isotopic ratio to be investigated.

3.4 Quit Procedure Definition Files and call Acquire Programs

3.5 Call Scan Acquire subroutine for analyses with pre-defined procedure definitions; otherwise call Survey Acquire and input element menu and define run parameters. Aspirate the sample; once it is ascertained that the sample has entered the plasma, commence scanning.

3.6 After acquiring data, each of the mass spectra will be automatically stored on a magnetic device under files defined in the procedure definition file. The files can be retrieved as raw data or integrated data (counts/sec/mass unit).

3.7 Perform Calculations

3.7.1 Under Survey Acquire: For semi-quantitative analyses, the default response curve is used to calculate the results based on the internal standard concentration. Input the internal standard concentration, the analysis procedure or the sample file identification, and commence calculations. For better semi-quantitative accuracy, a response curve can be generated using a standard solution containing low, medium, and high mass elements; see VG PlasmaQuad Software Manual for details.

3.7.2 Under Calculations: For multi-element analyses, reference material is used to calibrate the mass responses of the samples. For each of the elements, a mass response curve is generated from the responses of the reference materials; at

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-280	0	SEP 26 1990	10	13

A03-025

921191036

PNL TECHNICAL PROCEDURE

least two different concentrations of each element that are similar in concentration to that of the samples should be analyzed to generate a proper calibration curve. An  $R^2$  value for each curve is reported; an  $R^2$  value of less than 0.9 is unacceptable, and the results for that element shall either be omitted in the final report, or reported as suspect.

4.0 Interferences and Matrix Effects

4.1 Spectral Interferences

Spectral interferences are usually few in number and, in most cases, small in relative magnitude. Interference factors for normal day-to-day operations are accessed by analyzing various concentrations of single element solutions for each of the elements to be addressed. Only those interferences  $>0.1\%$  for elements common at higher concentrations and  $>1\%$  for less common elements shall be reported in the final results summary. For more details on interpreting interferences, refer to the VG PlasmaQuad Software Manual.

4.2 Acid Concentration

Acid concentration can have effects on the accuracy of the data by causing quenching of the analytical signal. For standard "ICP/MS" analyses, in which the calibration standards are in dilute acid, the samples should be diluted to acid strengths of similar concentration. In cases where the acid concentration is greater than 5% and dilution is not an acceptable solution due to loss of signal, then calibration standards can be made with acid concentrations which approximate the samples ( $\pm 10$  relative percent); see 4.3. However, if the internal standard used is similar in chemical properties to the element(s) of interest, the quenching effect on the internal standard will be representative of the effect on the samples, and matrix matching will then be unnecessary.

4.3 Matrix Matching

Solutions with high levels of dissolved solids should be analyzed against "matrix matched" calibrating standards, because increasing dissolved solids tend to depress (or quench) the analytical signal. The simplest way to avoid matrix matching is by dilution. However, if this is not acceptable, then the matrix effects must be accessed by spiking the matrix with an appropriate standard and observing the effect on the elements of that standard. If the effects are not

Procedure No. PNL-ALO-280	Revision No. 0	Effective Date SEP 26 1990	Page 11	of 13
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A03-024

PNL TECHNICAL PROCEDURE

acceptable, as determined by the qualified ANALYST and the customer, then matrix matched standards (e.g. digested geologic material) must be employed.

5.0 Reporting and Archiving Results

The precision and accuracy are assessed for each data set.

5.1 Quantitative Analysis

If the check standard results are accurate, and the RSD of the results are acceptable (<10%), the results are tabulated and reported as requested. Results having RSD's of 10-50% are reported preceded by the RSD value; results having RSD's greater than 50% shall be reported as semiquantitative values. Elements by which the check standard results differ by 10-50% are either normalized, or reported as being suspect; elements by which the check standard results differ by >50% may be normalized, but the results should be reported as suspect.

5.2 Semi-quantitative Analysis

Semi-quantitative analysis is reported in the same format as quantitative analysis, but without the constraints on accuracy; values should be designated as semi-quantitative, with associated errors as being -50% to +100%. Precision between runs should be the same as for quantitative analysis.

5.3 Reporting Results

The tabulated results, along with any documentation requested in the statement of work, are arranged in report format with a cover letter to the customer. The final report is signed by the ANALYST and reviewed by SCIENTIST on the basis of accuracy and technical adequacy.

Copies of the report, as well as the output of the results summary, the procedure listing, the element menu, the raw data, and all other computer output are assimilated and filed in a folder and cabinet designated for that purpose in 3708 Bldg., Room 108.

5.4 Archiving

At least once every two months, the raw data (found in directory C:\RAWDATA on the computer) is backed up onto a mass storage device (MSD) for every sample run since the last backup took place. The

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-280	0	SEP 26 1990	12	13

A03-025



PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-281 - ICP/MS DETERMINATION OF <sup>99</sup>Tc

APPLICABILITY

This procedure is applicable to the determination of <sup>99</sup>Tc in aqueous, acidified samples, using ICP/MS techniques. Samples in other physical forms or matrices should be digested, fused, leached or otherwise prepared to result in an aqueous matrix, preferably acidified with HNO<sub>3</sub> to an acid content not exceeding 5% vol/vol. This procedure is applicable to the determination of <sup>99</sup>Tc at concentrations ≥10 ppt (170 pCi/L), in solution.

This procedure shall be applied in accordance with PNL-ALO-280 (Inductively Coupled Plasma Mass Spectrometric (ICP/MS) Analysis) and PNL-MA-70.

DEFINITIONS/ACRONYMS

amu - atomic mass unit

acps - area counts per second (units of integrated ICP/MS signal intensity)

ARF - analytical request form

CERCLA - Comprehensive Environmental Response, Compensation, and Liability Act

cps - counts per second (units of ICP/MS signal intensity)

ICP/MS - inductively coupled plasma mass spectrometry

IP - ionization potential, in units of electron volts (eV)

m/z - mass (in amu) divided by charge

NIST - National Institute of Standards Technology

PNL - Pacific Northwest Laboratory

Author <i>Eric J. W. [Signature]</i>	Date 9/17/90	Project Mgr. <i>B.M. [Signature]</i>	Date 9-19-90	QAD Representative <i>G. [Signature]</i>	Date 9/19/90
Technical Reviewer <i>D.W. [Signature]</i>	Date 9/17/90	Line Mgr. <i>A. [Signature]</i>	Date 9-20-90	Other	Date
Procedure No. PNL-ALO-281	Revision No. 0	Effective Date SEP 26 1990	Page 1	of 4	

A03-037

PNL TECHNICAL PROCEDURE

ppb - parts per billion ( $\mu\text{g/L}$ )

ppt - parts per trillion (ng/L)

RSD - Relative Standard Deviation

SOW - statement of work

TI - test instruction

TP - Test Procedure

TUNE SOLUTION - A solution containing 100 ppb Mg, In, Pb for the purpose of tuning the ICP/MS instrument. Any solution containing 50 - 500 ppb of at least one low, medium, and high mass element can be used alternatively as a TUNE SOLUTION.

PROCEDURE

- 1.0 Define ELEMENT MENU in instrument operations software to include  $^{99}\text{Tc}$  (see PNL-ALO-280 2.3.1 and 3.2). Stipulate data acquisition in either scanning, peak jumping, or single ion monitoring mode depending on anticipated  $^{99}\text{Tc}$  concentration, other analytes to be determined, and possible interferences that may require monitoring and correction. Name, save and record ELEMENT MENU with all operational parameters.
- 2.0 Define ANALYSIS PROCEDURE as described in PNL-ALO-280 3.3 and 1.3. Run duplicate sample acquisitions in all cases (sample volume permitting). Number and frequency of standards, blind standards, spiked samples, and blanks shall be in accordance with the default QUALITY CONTROL procedures given below (8.0), or as specified in governing TI, SOW, or ARF instructions. Name, save and record ANALYSIS PROCEDURE.
- 3.0 Start up ICP/MS instrument, allow 30 minute warm-up period, and perform instrument response and precision check using TUNEIR analysis procedure as specified in PNL-ALO-280 2.2 and 3.1. Verify adequate instrument response and stability (criterion: response  $\geq 1 \times 10^6$  acps/ppm for tune elements In or Pb; stability  $\leq 7\%$  RSD concentration/isotope ratio precision for tune elements Mg, In, and Pb). Save and archive TUNEIR data and results.
- 4.0 Prepare samples by adding appropriate level and type of INTERNAL STANDARD as specified in PNL-ALO-280 1.2.3. Internal standard elements/isotopes should be judiciously chosen based on proximity in mass range and ionization potential to Tc (m/z 99 and 7.28 eV, respectively). Candidate

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-281	0	SEP 26 1990	2	4

A03-028

PNL TECHNICAL PROCEDURE

internal standards for this determination include Rh (m/z 103; IP 7.46 eV) and In (m/z 113,115; IP 5.79 eV).

- 5.0 Standards solutions shall be prepared, using dilutions of previously prepared stock calibration standards, to yield standard concentrations that bracket the anticipated sample concentrations. During analysis, if a sample concentration exceeds two times that of the maximum concentration standard, a dilution shall be made of the sample so that it is within the calibration standard range; if it is below that of the minimum concentration standard and above 100 ppt, a standard containing an appropriate lower concentration of <sup>99</sup>Tc (e.g., 500 ppt) shall be prepared and analyzed. Internal standards shall be added to the standard solutions in exact concentration and form as the samples, in accordance with 4.0 above. Stock calibration standards that are available for use in this determination are the following:

52433-43: 12.56 ppb <sup>99</sup>Tc (BNW LRB 52433, p 43)  
52433-47: 98.74 ppb <sup>99</sup>Tc (BNW LRB 52433, p 47)  
52433-48D: NIST SRM 3.759E4 kBq/g soln (59.27 ppm <sup>99</sup>Tc [BNW LRB 52433, p 48]).

Working standards shall be prepared fresh weekly, at minimum.

- 6.0 Prepared samples and standards shall be analyzed by ICP/MS according to ICP/MS Procedure PNL-ALO-280 2.3.4 and 3.5, the instrument operating manuals, and SOPs for this instrument.
- 7.0 Calculation of the acquired data shall be performed via instrument software in accordance with PNL-ALO-280 3.7. Calibration curves shall be constructed using standards intensity and concentration data; linear/quadratic regression fitting shall be performed using this data. Identity and number of standards, type of fit, and regression coefficients shall be stored in CALCULATION PROCEDURE RESPONSE files and output with data results. Reporting of the results shall be in accordance with PNL-ALO-280 5.3, and shall include the uncertainty of the results, which are based on deviations between values obtained from analytical runs as well as those obtained from different elemental isotopes.

8.0 Quality Control

All analysis and quality control data shall be maintained and available for easy reference or inspection.

Two quality control options are defined. Option A shall be used unless a SOW written by WHC defines CERCLA protocols. Option B shall then be

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-281	0	SEP 26 1990	3	4.

A03-029

PNL TECHNICAL PROCEDURE

followed. The analyst will recognize the need to use Option B when a Chain-of-Custody (COC) defines a Test Instruction (TI). Additional QC samples may be requested by a client in an Analytical Request Form (ARF) or a Statement of Work (SOW), and these will be conveyed to the analyst through a TI.

- A. Employ a minimum of one blank per sample batch (20 samples or less) to determine if contamination is occurring. Run duplicate analysis upon client request. A spiked sample or standard (NIST traceable) shall be periodically employed to ensure that correct procedure is followed and that all equipment is operating properly.
- B. For all SOW's written by WHC for CERCLA protocol requests for analysis, employ a minimum of one blank per sample batch (20 samples or less) to determine if contamination is occurring. Run one duplicate sample for every 20 samples or for each set, whichever is smaller. A duplicate sample is a sample brought through the whole sample preparation and analytical process. A spike sample or standard (NIST traceable) shall be run for every 20 samples analyzed or per every set of samples, whichever is smaller.

9.0 Results

ICP/MS results are normally reported in weight/volume concentration units, typically as ppb or ppt. Radiometric concentrations can alternatively be computed/reported, using the calculation below for results in pCi/L units:

$$[^{99}\text{Tc}], \text{ pCi/L} = ([^{99}\text{Tc}], \text{ ppt} * 1 \times 10^{-9} * A * \ln 2) / AW * t_{1/2} * C$$

where:

- AW = 99 g/mole;
- $t_{1/2} = 6.72 \times 10^{12}$  sec;
- A =  $6.023 \times 10^{23}$  atoms/mole; and
- C = 0.037 dps/pCi

which reduces to:

$$[^{99}\text{Tc}], \text{ pCi/L} = [^{99}\text{Tc}], \text{ ppt} * 16.95$$

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-281	0	SEP 26 1990	4	4

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-441, RADIONUCLIDE SEPARATION AND ANALYSIS PROCEDURE FOR TRITIUM

APPLICABILITY

This procedure describes methods used to determine the concentration of <sup>3</sup>H in sample matrices. Primarily, the procedure is designed for dissolved species, although solid sample analysis can be performed as well. This procedure describes initial radionuclide separation and radionuclide purification methods. This procedure was developed by PNL radioanalytical scientists, and is based on over 10 years experience in analyzing a variety of samples for tritium by liquid scintillation counting.

DEFINITIONS/ACRONYMS

- dpm - Disintegration per minute (number of atoms of a radioactive sample which decay in a one minute interval)
- cpm - Counts per minute (counting rate measured with a radiation detection instrument)
- d/c - Disintegrations per count (i.e., efficiency factor)
- LRB - Laboratory Record Book
- LSC - Liquid Scintillation Cocktail

RESPONSIBLE STAFF

Cognizant Scientist  
Analyst/Technician

PROCEDURES

Prerequisites: Personnel trained in this procedure may perform the work. Personnel training records are kept in the Section office.

Author <i>B.M. Dilligian</i>	Date 11-26-90	Project Mgr. <i>B.M. Dilligian</i>	Date 11-26-90	QA0 Representative <i>GK George</i>	Date 11/27/90
Technical Reviewer <i>M.J. Wynn</i>	Date 11-27-90	Line Mgr. <i>[Signature]</i>	Date 11-27-90	Other	Date
Procedure No. PNL-ALO-441	Revision No. 0	Effective Date NOV 27 1990	Page 1	of 10	

## PNL TECHNICAL PROCEDURE

### 1.0 Tolerances

Tolerances for all measurements made during an analysis shall be specified in the following manner: 1) a tolerance limit can be stated with a measurement value given in a method; or 2) if a tolerance limit is not stated with a measurement value, then the following system of tolerances shall be in effect:

- (a) Unless otherwise specified, all values for measurements stated in the methods (volume, weight, time, etc.) are approximate values. The actual measurements used, however shall be within  $\pm 10\%$  of the stated value.
- (b) When one or more significant figures are given to the right of the decimal point, the tolerance limit is  $\pm 5$  in the next digit located beyond the last one stated.

### 2.0 Reagents

- 2.1 Low-tritium background water - water that contains less than required detection limits of tritium as demonstrated by the methods blank.
- 2.2 6 M HCl - 50 ml low-tritium water (2.1) and 50 ml conc. HCl
- 2.3 Sodium Carbonate,  $\text{Na}_2\text{CO}_3$  - Reagent grade
- 2.4 Hold Back Carrier - 10 g. cupric sulfide, 10 g. sodium carbonate, 10 g. strontium nitrate, 10 g. potassium iodide and 40 g. silver nitrate, mixed thoroughly until it appears homogeneous (i.e. even distribution of color and particle size).
- 2.5 Packard Opti Fluor liquid scintillation cocktail. (Or equivalent)

### 3.0 Equipment

- 3.1 Distillation Apparatus: For aqueous distillation: 125 ml or 250 ml short neck, flat bottom boiling flask, connecting side arm adapter, condenser and heating mantle or hot plate.
- 3.2 Polyethylene or glass liquid scintillation vials - 25 ml
- 3.3 Coincidence-type liquid scintillation spectrometer. (See appropriate liquid scintillation counting procedure for LSC system to be used such as PNL-ALO-443.)

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-441	0	NOV 27 1990	2	10

A03-032

PNL TECHNICAL PROCEDURE

- 3.4 Stirrer/Hot plate
- 3.5 Filter paper - Whatman 934-AH glass microfiber, or membrane filter, 1.5 mm effective retention or better (or comparable).
- 3.6 Glass vials - 25 ml.
- 3.7 Volumetric flasks, various sizes
- 3.8 Balance capable of accurately weighing to the nearest 0.01 g.
- 3.9 Graduated cylinders - 20 ml, 50 ml or 100 ml. (or other size to meet volume requirements)

4.0 Sample Preparation

The procedure is written for 125 ml flat bottom boiling flasks but may be scaled up appropriately to 250 ml or larger flat bottom boiling flasks as necessary for environmental samples or leachate volumes.

The aliquot sizes specified are for low level test-well samples. For other samples, the cognizant scientist may specify smaller aliquots due to the radioactivity levels and waste minimization issues. In the analysis of radioactive samples, the distillate receiving vessel is different. The receiving vessel is attached such that it is not easily removed from the distillation condenser and the first 5-10 ml distilled aliquot is not discarded.

All data for sample preparation shall be recorded on a Tritium Analysis Data Sheet (Exhibit 1). This attached Data Sheet is an example of the Tritium Analysis Data Sheet which may be expanded to include more samples per batch or modified to delete the solid samples section if only liquid samples are analyzed.

4.1 Quality Control

All quality control data shall be maintained as hard copy and available for easy reference or inspection (QC samples are recorded on a Tritium Analysis Log sheet, Exhibit 1 and on the data forms associated with the appropriate liquid scintillation counting procedure).

Two quality control options (A and B) are defined below. Option A shall be used unless a Statement of Work (SOW) written by a client defines CERCLA requirements. Option B shall then be followed. The analyst will recognize the need to use option B when a Chain-of-

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-441	0	NOV 27 1990	3	10

A03-033

PNL TECHNICAL PROCEDURE

Custody (COC) defines a Test Instruction. Additional QC samples may be requested by a client in an Analytical Request Form (ARF) or a Statement of Work (SOW), and these will be conveyed to the analyst through a Test Instruction (TI).

- A. Employ a minimum of one methods blank per sample batch (20 samples or less) to determine if contamination is occurring. Run duplicate analysis upon client request. Except when client requires Impact Level III analysis, a spiked sample or standard (NIST traceable) shall be analyzed per batch to ensure that the correct procedure is followed and that all equipment is operating properly.
- B. For all SOWs written by a client to meet CERCLA requirements that request analysis, employ a minimum of one methods blank per sample batch/set (20 samples or less) to determine if contamination is occurring. Run one duplicate sample for every 20 samples or for each set, whichever is smaller. A duplicate sample is a sample brought through the whole sample-preparation and analytical process. A spike sample or standard (NIST traceable) shall be employed for every 20 samples analyzed or per every set of samples, whichever is smaller. A matrix spike duplicate and/or laboratory control sample may be analyzed with a set/batch of samples if so directed by a Test Instruction (TI).

A flow diagram of samples and QC requirements is attached (Exhibit 2).

4.2 Liquid Samples

- 4.2.1 Put an aliquot of solution into a 125 ml flat bottom boiling flask. Use a 50 ml aliquot of solution if possible. Proceed to 4.4 for blank and standard make-up and on to 5.0 for distillation and counting. The cognizant scientist may specify a second distillation by repeating Step 5.1 - 5.3 again.

4.3 Soil/Sediment/Sludge/Solid Samples

- 4.3.1 Weigh a 1-50 g. aliquot of sample, as received, into an appropriate size beaker. Record weight on the Tritium Analysis Log sheet (Exhibit 1).
- 4.3.2 Add enough 6 M HCl to cover the sample aliquot plus about 3-5 ml in excess. Add a stirring bar and watch glass cover.

Procedure No. PNL-ALO-441	Revision No. 0	Effective Date NOV 27 1990	Page 4	of 10
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A03-034

PNL TECHNICAL PROCEDURE

- 4.3.3 Heat on a stirring hot plate at 50-70°C (hot plate setting of 4-5) for at least 15 min. Do not let the sample go dry. Add additional 6 M HCl as necessary.
- 4.3.4 Cool and filter through a Whatman 934-AH glass microfiber filter (or comparable).
- 4.3.5 Return filtered solids aliquot and filter paper to the leaching beaker. Repeat steps 4.3.2 through 4.3.4 on the same filtered solids aliquot; combine filtered leachates.
- 4.3.6 Return filtered solids aliquot and filter papers to the leaching beaker. Repeat steps 4.3.2 through 4.3.4 using low-tritium water in place of HCl.
- 4.3.7 Combine filtered water leachate with the other 2 combined filtered acid leachates. Bring to a known volume with low-tritium water using an appropriate size graduated cylinder. Record volume on the tritium Analysis Log sheet (Exhibit 1).
- 4.3.8 Put up to 80 ml (if possible) of the combined acid leachates and water leachate from step 4.3.7 into a 125 ml distillation flask. Add a spatula tip (~0.5 g) of holdback carrier.
- 4.3.9 Connect the side arm adapter and condenser to the outlet of the flask, and insert a beaker under the outlet of the condenser. Heat the sample to boiling to distill. Distill 5-10 ml into the beaker and discard the distillate. This cleans the condenser.
- 4.3.10 Place a clean beaker under the condenser to collect the next 50 ml (if possible) of distillate.
- 4.3.11 Transfer distillate to another 125 ml flat bottom distillation flask. Proceed to 4.4.2 for preparation of blanks, standards and spikes and then on to 5.0 for the second distillation.
- 4.3.12 To prepare a quality control matrix spike or standard recovery correction factor, if requested by an SOW, ARF or TI, use the filtered solid from step 4.3.4, and spike with a known amount of tritium standard as directed by the cognizant scientist. The spike should be similar in concentration to the concentration anticipated in the

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-441	0	NOV 27 1990 A03-035	5	10

PNL TECHNICAL PROCEDURE

samples being analyzed or at a minimum a detectable amount (3 to 5 times background). Record the spike aliquot on the Tritium Analysis Log sheet (Exhibit 1). Follow procedure in steps 4.3.2 through 4.3.11. The filtered solid is used as it provides a tritium free sample to spike.

4.4 Blanks/Raw Water Standard/Distilled Water Standard

4.4.1 If liquid samples are being analyzed and if requested by a SOW, ARF or TI, prepare raw water standard distillate by placing 50 ml low tritium water in a 125 ml distillation flask. Prepare in duplicate. Add (NIST traceable) tritium standard spikes to each as determined by the cognizant scientist (activity added to achieve about 1000 dpm/ml). Record spike aliquots on the Tritium Analysis Log sheet (Exhibit 1). Distill raw water standards as per section 5.0.

4.4.2 To prepare blanks and distilled water standards, place 60 ml of low tritium water in a 125 ml distillation flask. Add a spatula tip (~0.5 g) of holdback carrier.

4.4.3 Connect the side arm adapter and condenser to the outlet of the flask, and insert a beaker under the outlet of the condenser. Heat the sample to boiling to distill. Distill 5 to 10 ml of solution into a flask and discard distillate. This cleans the condenser.

4.4.4 Place a clean beaker under the outlet of the condenser. Collect 20-35 ml of distillate for use in the following steps.

Note: A large volume (e.g. 1 liter) of low tritium water may be distilled and used for several sets of sample analyses. Therefore, step 4.4.2 may not be necessary.

4.4.5 Prepare methods blank (background) solutions from 4 ml aliquot of low tritium background water distillate from step 4.4.4 thoroughly mixed with 19 ml of scintillator solution in liquid scintillation counting vials. Prepare in duplicate. Record volume on Tritium Analysis Log Sheet (Exhibit 1).

4.4.6 Prepare two distilled water standards by adding spike to each of two liquid scintillation counting vials containing 19 ml of scintillation solution. Each vial shall be spiked with a

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-441	0	A03-038 NOV 27 1990	6	10

PNL TECHNICAL PROCEDURE

different activity as specified by the cognizant scientist. The spike activities are normally ~50,000 dpm and ~100,000 dpm or less, respectively, but may be increased if higher tritium activity is expected in the samples. Record spike aliquots on the Tritium Analysis Log Sheet (Exhibit 1).

- 4.4.7 Add low tritium background water distillate from step 4.4.4 to each standard to make 4 ml total aqueous volume in each vial. Mix each vial thoroughly.

5.0 Tritium Analysis

- 5.1 To a sample aliquot from the sample preparation (step 4.2.1, 4.3.11 or 4.3.12) add a spatula tip (~0.5 g) of holdback carrier. If solution is not already basic (pH>7) add Na<sub>2</sub>CO<sub>3</sub> until basic (check with pH paper).
- 5.2 Connect the side arm adapter and condenser to the outlet of the flask, and insert a beaker under the outlet of the condenser. Heat the sample to boiling to distill. Distill 5 to 10 ml of solution into a flask and discard distillate. This cleanses the condenser.
- 5.3 Place a clean beaker under the outlet of the condenser. Distill until about 10-25 ml of distillate is collected into a 25 ml glass vial.
- 5.4 To prepare sample aliquot for counting, thoroughly mix a 4 ml aliquot of distillate from 5.3 with 19 ml of scintillator solution in a liquid scintillation counting vial. Prepare in duplicate. Record the sample aliquot volume on the Tritium Analysis Log sheet (Exhibit 1). Save remaining sample distillate pending analysis of sample counting data.

Note: Less than 4 ml of sample may be used as the aliquot size depending on the concentration of tritium anticipated in the samples. Low tritium background water distillate from step 4.4.4 shall be added to each vial to bring to 4 ml total aqueous volume.

- 5.5 Count all samples in the liquid scintillation spectrometer for 100 minutes or 2σ counting statistics, whichever comes first. Refer to the Liquid Scintillation (LSC) Counting Procedure appropriate for the LSC system being used.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-441	0	A03-037 NOV 27 1990	7	10

PNL TECHNICAL PROCEDURE

- 5.6 Using the background-corrected count rates of the standards, determine the efficiency of the scintillator solution. Use this efficiency to determine the tritium concentrations of the background-corrected sample solutions.
- 5.7 If a sample count rate exceeds 60,000 cpm, the cognizant scientist shall determine a smaller appropriate aliquot size. This sample aliquot shall be prepared and counted according to steps 5.4 and 5.5.

6.0 Calculations

6.1 Efficiency factor, E, d/c

$$E = \frac{G}{D - B}$$

where:

D = distilled Water standard (from 4.4.7) count rate, cpm,  
B = background count rate (from 4.4.5), cpm, and  
G = activity of distilled water standard (dpm)

6.2 Recovery correction factor for raw water standard,  $F_w$

$$F_w = \frac{(L - B) \times E}{M}$$

where:

L = raw water standard distillate (from 4.4.1) count rate, cpm,  
B = background count rate (from 4.4.5), cpm,  
E = counting efficiency, as determined in 6.1, and  
M = activity of raw water standard (before distillation), dpm

6.3 Recovery correction Factor for solids/soils/sludges,  $F_s$ ,

$$F_s = \frac{(L - B) \times E}{M}$$

L = soil matrix spike distillate (from 4.3.12) count rate, cpm,  
B = background count rate (from 4.4.5), cpm,  
E = counting efficiency, as determined in 6.1, and  
M = activity of spike in soil matrix (before distillation), dpm

PNL TECHNICAL PROCEDURE

6.4 Activity of Tritium in aqueous sample, A

$$\bar{A} = \frac{(C - B) \times E \times Q}{V \times F_w}$$

where:

- C = sample count rate, cpm,
- B = background count rate, cpm,
- E = counting efficiency, as determined in 6.1,
- V = volume of the distilled sample aliquot mixed with LSC in ml (example: 4 ml), (from 5.4 or 4.4.7)
- F<sub>w</sub> = recovery factor, as determined in 6.2,
- Q = factor or factors to account for sample dilutions or needed to produce the appropriate reporting units. These factors shall be documented as part of the project record.

6.5 Activity of Tritium in soils/solids/sludge sample, A

$$A = \frac{(C - B) \times VL \times E \times Q}{V \times F_s \times W}$$

where:

- C = sample count rate, cpm,
- B = background count rate, cpm,
- E = counting efficiency, as determined in 6.1,
- V = volume of the distilled sample aliquot mixed with LSC in ml (example: 4 ml), (from 5.4 or 4.4.7)
- VL = volume of filtered leachate, ml,
- F<sub>s</sub> = recovery factor, as determined in 6.3,
- W = weight of sample leached, g,
- Q = factor or factors to account for sample dilutions or needed to produce the appropriate reporting units. These factors shall be documented as part of the project record.

7.0 Records

Records will be maintained and controlled so as to conform to requirements of PNL-MA-70, PAP-70-1701. Laboratory Record Books (LRB) and Analytical Report Cards/Data Sheets provide a mechanism for control of most records. Laboratory Record Books will be used in accordance with the ACT NOW Directive 89.1.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-441	0	NOV 27 1990	9	10

PNL TECHNICAL PROCEDURE

8.0 Specific Qualification

This procedure describes the calibration of instrumentation using radionuclide standards in analysis process control and, as such, is considered self-qualifying as defined in PNL-MA-70, PAP-70-901.

9.0 References:

"Tritium in Drinking Water, Method 906.0", Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August 1980.

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Procedure No. PNL-ALO-441	Revision No. 0	Effective Date NOV 27 1990	Page 10	of 10
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~~A03-010~~

TRITIUM ANALYSIS LOG SHEET

Pipet No. \_\_\_\_\_ Nominal Vol. \_\_\_\_\_ Calibrated Vol. + Error \_\_\_\_\_ Date Pipetted \_\_\_\_\_  
\_\_\_\_\_ 50 lambda \_\_\_\_\_  
\_\_\_\_\_ 100 lambda \_\_\_\_\_  
\_\_\_\_\_ 4 ml \_\_\_\_\_

IF SOLID SAMPLES: Balance No. \_\_\_\_\_

Sample No: \_\_\_\_\_ Matrix Spike Sample No: \_\_\_\_\_

beaker + sample \_\_\_\_\_ Spike i.d. \_\_\_\_\_  
beaker wt. \_\_\_\_\_ nominal vol. \_\_\_\_\_  
sample wt. \_\_\_\_\_ leachate vol. \_\_\_\_\_  
leachate vol. \_\_\_\_\_

Sample No: \_\_\_\_\_ Sample No: \_\_\_\_\_

beaker + sample \_\_\_\_\_ beaker + sample \_\_\_\_\_  
beaker wt. \_\_\_\_\_ beaker wt. \_\_\_\_\_  
sample wt. \_\_\_\_\_ sample wt. \_\_\_\_\_  
leachate vol. \_\_\_\_\_ leachate vol. \_\_\_\_\_

Backgrounds: Raw Water Stds. #1 and #2:

Blank #1 Vol: \_\_\_\_\_ Spike i.d. \_\_\_\_\_

Blank #2 Vol: \_\_\_\_\_ nominal vol. \_\_\_\_\_

Distilled Water Stds: Spike i.d. \_\_\_\_\_

Std #1 nominal vol. \_\_\_\_\_ Std #2 nominal vol. \_\_\_\_\_

COUNTING ALIQUOTS FOR ALL SAMPLES:

Sample No: \_\_\_\_\_ Matrix Sample No: \_\_\_\_\_

Nominal Vol: \_\_\_\_\_ Nominal Vol: \_\_\_\_\_

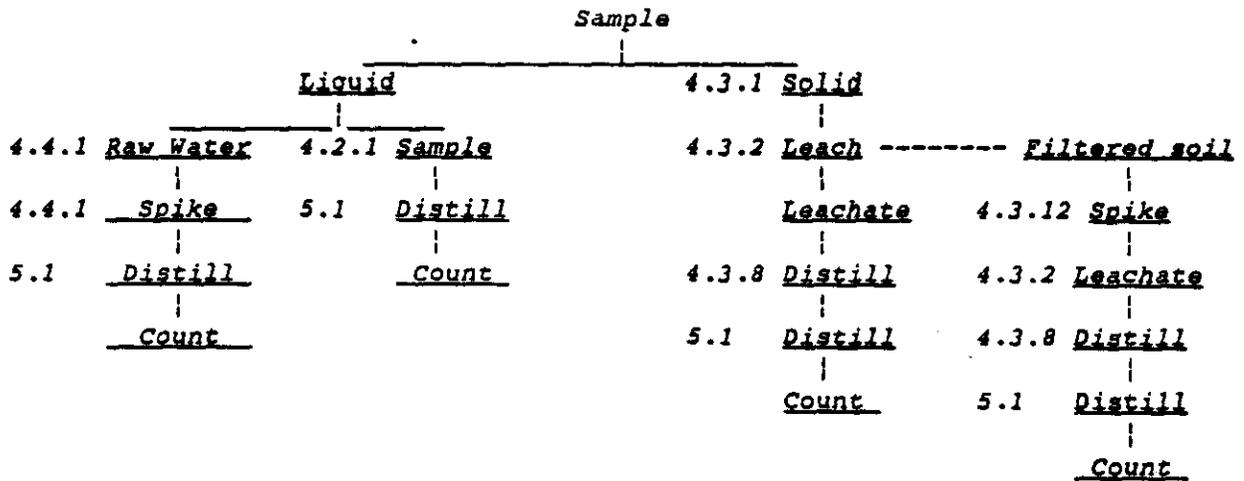
Sample No: \_\_\_\_\_ Sample No: \_\_\_\_\_

Nominal Vol: \_\_\_\_\_ Nominal Vol: \_\_\_\_\_

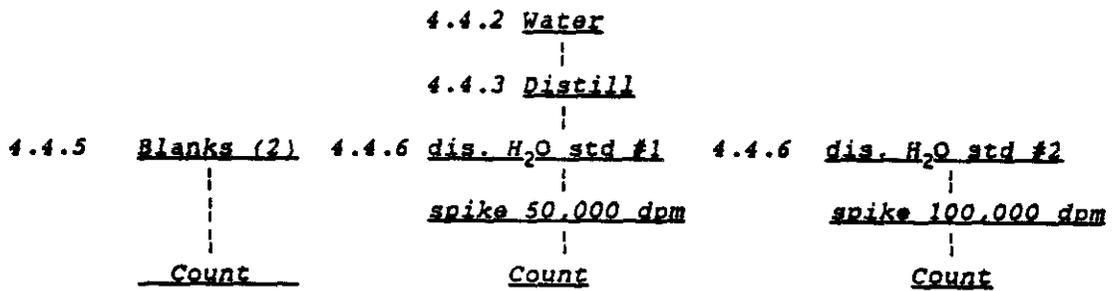
Analyst's Signature \_\_\_\_\_ Date \_\_\_\_\_

Exhibit 2

FLOW DIAGRAM FOR TRITIUM ANALYSIS



Water Standards and Blanks



INTERIM CHANGE NOTICE  
(ICN)

ICN- PNL-ALO-443.1  
Page 1 of 1

A. Document Number: <u>PNL-ALO-443</u> Revision Number: <u>0</u> Document <u>Liquid Scintillation Counting Procedure</u> Title: <u>for Tritium</u> Document's Original Author: <u>B. M. Gillespie</u>	Effective Date of ICN: <u>4/26/91</u>
	Change Requested By: <u>NL Wynhoff</u>

B. Action:

Replace pages 3 and 4.

C. Effect of Change:

Discontinue counting of a C-14 standard which is irrelevant to tritium counting.

D. Reason for Change/Description of Change

- Delete references to a C-14 standard.
- Delete reference to a C-14 standard and to a raw water standard since soil samples do not include a raw water std. and the recovery factor spike for either waters or soils may be counted in any order with the samples.

DESCRIPTION OF CHANGE - Edit Sec. 1.3.1, First sentence to read: "Sealed <sup>3</sup>H and blank standards (Beckman Nos. H277092 and B11080, respectively, or equivalent) shall be..."  
 Edit Sec. 2.1, First sentence to read: "... tower #1, the sealed blank, the sealed <sup>3</sup>H standard, blanks (backgrounds), distilled water standards (for d/c), samples and then a stop tower."

E. Approval Signatures (Please Sign and Date)	Type of Change: (Check ( / ) one)
	<input type="checkbox"/> Minor Change <input checked="" type="checkbox"/> Major Change
Process Quality <del>QA</del> Department Concurrence: <u>GK Gerbe, DE</u> Date: <u>3/15/91</u>	
Approval Authority: <u>[Signature]</u> Date: <u>3/18/91</u>	
Other Approvals: <u>B M Gillespie</u> Date: <u>3/18/91</u>	
: _____ Date: <u>1 1</u>	



## PNL TECHNICAL PROCEDURE

The LSC system is intended for use by operators experienced with the methodology of in vitro diagnostic, radioassay, and nuclear chemistry procedures which require nuclear counting of certain reagents with particular energy bands by liquid scintillation techniques.

### 1.1 Components in the LSC System

The LSC system is a Beckman LS-8000, serial number 7804114. The system consists of a 300-sample conveyor sample changer, photomultiplier tubes, circuit boards and operator/program selection towers.

### 1.2 Counter Quench Correction

The system is equipped with an external standardization method in which a high-energy gamma source ( $^{137}\text{Cs}$ ) can be automatically positioned near the sample vial to produce a continuum of Compton recoil electrons which can excite solvent molecules that will eventually transfer the energy of excitation to solute molecules to produce photons. If quenching is present in the sample, the resulting Compton pulse height distribution is affected in the same manner as the light distribution of the radioactive sample. The true quench level of the sample is expressed as a shift in the Compton edge of the sample relative to an unquenched sample and the difference in the channel settings represents the extent quenching has affected the light output.

The microprocessors in the LS 8000 system analyze the Compton distribution to determine the location of the Compton edge of the sample. This determination is based on measuring the area of the Compton edge in special, discrete counting channels to determine the inflection point of the edge. When compared to the channel setting of the inflection point of an unquenched sample, the H-Number or difference in channel settings is determined.

Samples counted with an H-Number can be subjected to quench correction in order to determine actual sample activity-disintegrations per minute (dpm) - regardless of quenching in the sample. Quenched standards, which contain the same type of isotope as in the sample but whose activity is known, are prepared and counted to determine count rates-counts per minute (cpm). H-Numbers are measured for each standard and a quench curve generated which relates the counting efficiency of each standard to its corresponding H-Number. This becomes part of the permanent record in the software program used for the specific analysis.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-443	0	NOV 27 1990 A03-045	2	5

## PNL TECHNICAL PROCEDURE

### 1.3 Counter Performance Tests

The geometry of the LSC system is fixed and depends on the smooth operation of the sample changer and the pedestal of which counting vials are lowered in front of the photomultiplier tube. Any malfunction of this portion of the LSC system will be recognized by the control circuits and printed as an appropriate note on the readout tape.

1.3.1 Sealed  $^3\text{H}$  and blank standards (Beckman Nos. H277092 and B11080, respectively, or equivalent) shall be counted before each set of samples and the count rates compared with previously recorded counts to verify consistent system operation. The count rates of these standards shall be recorded in the Beckman LSC LS8000 notebook. The isotope standard count rates shall be within  $3\sigma$  of the mean as established previously, after decay correction, and background count rates shall be within  $3\sigma$  of the previously recorded backgrounds.

1.3.1.1 Control charts shall be set up and maintained as per PNL-MA-597, Vol 1, Section 7.

1.3.2 Discontinue counting the sample set if the sealed standards exceed the control limits specified in 1.3.1. After cleaning the vials, recount the sealed standards two times.

1.3.3 If both recounts are in control, counting may continue on any remaining samples in the set. If both recounts of each standard are not within the control limits, any data collected is disqualified. The samples shall be recounted after the equipment is repaired or the samples may be prepared again and reanalyzed. A sign shall be posted on the LSC system indicating the instrument is out of control and shall not be used until repaired and recalibrated. A DR is needed if samples have been counted.

1.3.4 The record of these count rates also assists the Beckman service representative to analyze specific system operating parameters during preventive maintenance and troubleshooting activities. The LSC system performance is also monitored with each set of samples inserted for analysis. At least two spiked samples and two blanks, matching the specific characteristics of the sample set with respect to scintillator and dissolved sample aliquot, are counted

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-443	0	NOV 27 1990	3	5

~~A03-046~~

## PNL TECHNICAL PROCEDURE

scintillator and dissolved sample aliquot, are counted (corrected for  $^3\text{H}$  decay) and compared with previously recorded  $^3\text{H}$  count rates.

### 2.0 Sample Counting

2.1 When setting up blanks, standards and samples in the conveyor sample changer for counting in the liquid scintillation system, place them in the following order: tower #1, the sealed blank, the sealed  $^3\text{H}$  standard, blanks (backgrounds), distilled water standards (for d/c), samples and then a stop tower. Log positions on the 3000 Tri Carb data sheet (Exhibit 1. It is only used to log in the blanks, standards and sample positions in the cassette. The control settings section of the form shall not be filled in for this instrument).

2.2 Print out the program logged into the system by pressing the following key on the key pad of the LSC system:

Print Program Summary

If the current operator number is not 1 and the program is not 1 then proceed. If the operator number is 1 and the program is one and the sample count time is 100 min. then proceed to step 2.7.

2.3 Recall the operator's number using the key pad on the LSC system by pressing the following keys in the following order:

User Number - #1 - Enter - Proceed.

2.4 Input the correct program by pressing the following keys in the following order on the key pad:

Library Program - 1 - Enter - Proceed

2.5 Input the correct count time for samples by pressing the following keys in the following order on the key pad:

Common Parameters - 1 - Enter - 100 (min) - Enter - Proceed

2.6 Print out the program to make sure the parameters are correct by pressing the following key on the key pad:

Print Program Summary

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-443	0	NOV 27 1990	4	5

A03-047

## PNL TECHNICAL PROCEDURE

2.7 If all is correct on print-out as above, start counting the sample set by pressing the following key on the key pad:

Auto Count

2.8 When all counting is complete for the sample batch/set, evaluate the instrument printed data for the sealed blank and standards per section 1.3.

2.9 Calculate activities of samples, raw standards, distilled standards and blanks/ backgrounds in accordance with instructions from the cognizant scientist or as required by the analyte analysis procedure (e.g. PNL-ALO-441).

### 3.0 Records

Records will be maintained and controlled so as to conform to requirements of PNL-MA-70, PAP-70-1701. Laboratory Record Books (LRB) and Analytical Report Cards/Data Sheets provide a mechanism for control of most records. Laboratory Record Books will be used in accordance with the ACT NOW Directive 89-1.

### 4.0 Specific Qualifications

This instrument is calibrated using radiochemical standards and as such is self qualifying as per definitions in PNL-MA-70, PAP-70-901.

### 5.0 References

Beckman LS 8000 Series Liquid Scintillation System Manual

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-443	0	NOV 27 1990	5	5

~~A03-048~~



PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-462, SOURCE PREPARATION FOR GROSS BETA ANALYSIS

APPLICABILITY

This procedure is applicable to the samples received for gross beta analysis. The methodology in this procedure is consistent with that outlined in procedure 9310 of the SW 846 methods manual.

DEFINITIONS/ACRONYMS

cpm - counts per minute  
conc. - concentrated

RESPONSIBILITIES

Cognizant Scientist  
Analyst/Technician

PROCEDURE

Prerequisite: Personnel trained in this procedure may perform the work. Training Records are kept on file in the Section Office.

1.0 Tolerances for all measurements made during an analysis shall be specified in some manner: 1) State with a measurement value given in a method or 2) as specified below if not stated with a measurement value.

- (a) Unless otherwise specified, all values for measurements stated in the methods (volume, weight, time, etc.) are approximate values. The actual measurements used, however shall be within  $\pm 10\%$  of the stated value.
- (b) When one or more significant figures are given to the right of the decimal point, the tolerance limit is  $\pm 5$  in the next digit located beyond the last one stated.

Author	Date	Project Mgr.	Date	QAD Representative	Date
<i>N. J. Williams</i>	9-21-90	<i>B. M. Dillipin</i>	9-24-90	<i>C. K.erbe</i>	9/24/90
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
<i>B. M. Dillipin</i>	9-24-90	<i>J. Falk</i>	9-24-90		
Procedure No.	Revision No.	Effective Date	Page	of	
PNL-ALO-462	0	A03-050SEP 26 1990	1	3	

PNL TECHNICAL PROCEDURE

2.0 Quality Control

Except where responsibility is specifically assigned to a cognizant scientist, the analyst may perform all work. All pertinent data, observations and calculations, e.g., aliquot volumes, shall be recorded on a Beta Counting Data Sheet (Exhibit 1) or on an Analytical Data Sheet.

All quality control data shall be maintained and available for easy reference or inspection.

Two quality control options (A and B) are defined. Option A shall be used unless a Statement of Work (SOW) written by a client defines CERCLA requirements. Option B shall then be followed. The analyst will recognize the need to use Option B when a Chain-of-Custody (COC) defines a Test Instruction. Additional QC samples may be requested by a client in an Analytical Request Form (ARF) or a Statement of Work (SOW), and these will be conveyed to the analyst through a Test Instruction (TI).

- A. A minimum of one blank per sample batch (20 samples or less) shall be employed to determine if contamination is occurring. Run duplicate analysis upon client request. Except when QA Level III is specified, one spike sample or standard (NIST traceable or commercially certified) per batch shall also be analyzed to ensure that correct procedure is followed and that all equipment is operating properly.
- B. For all SOWs written by WHC for CERCLA protocol requests for analysis, a minimum of one blank per sample batch (20 samples or less) shall be analyzed to determine if contamination is occurring. One duplicate sample shall be analyzed for every 20 samples or for each set, whichever is smaller. A duplicate sample is a sample brought through the whole sample-preparation and analytical process. A spike sample or standard (NIST traceable or commercially certified) shall be employed for every 20 samples or per every set of samples, whichever is smaller.

3.0 Reagents and Apparatus

Nitric acid, 8N - dilute 500 ml reagent grade conc.  $\text{HNO}_3$  with deionized or distilled water to 1000 ml

Hot plate

Heat Lamp

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-462	0 A03-051	SEP 26 1990	2	3

## PNL TECHNICAL PROCEDURE

Stainless steel dish, 1-inch diameter, flamed - heat on high setting on a porcelain top hot plate until color changes to dark brown/black (3-5 minutes). Remove to cool.

### 4.0 Sample Preparation and Analysis

- 4.1 Evaporate the sample or sample aliquot to a few ml (1-5) on a hot plate. Do not evaporate to dryness.
- 4.2 Quantitatively transfer the sample to a flamed 1" counting dish using 8N HNO<sub>3</sub>. Evaporate the solution under a heat lamp to dryness.
- 4.3 Count the beta activity (refer to procedure PNL-ALO-463). Record all pertinent data including sample identification, sample volume, and all counting data on a Beta Counting Data form (Exhibit 1).
- 4.4 Calculate total beta activity:

$$A = \frac{\text{cpm} \times \text{d/c}}{B}$$

where:

A = net beta activity of the sample

cpm = gross counts per minute beta less background cpm (from the most recent background count which must have been taken within the last week)

d/c = disintegrations per count, an efficiency factor (see PNL-ALO-463)

B = factor to account for sample volumes, dilutions, weights, leach solution volumes and any other factors needed to produce the appropriate reporting units. These factors shall be documented as part of the project records on the Beta Counting Data Sheet (Exhibit 1) or on an Analytical Data Sheet.

### 5.0 Reference

SW-846, Method 9310, 3rd Edition.

Procedure No. PNL-ALO-462	Revision No. 0 A03-052	Effective Date SEP 26 1990	Page 3	of 3
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PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-463, BETA COUNTING PROCEDURE

APPLICABILITY

This procedure is applicable to the operations of the Beta Counting Systems located in the 329 Building as they are applied to the analyses of samples for beta-emitting radionuclides. The objective of this procedure is to communicate both specific and general information regarding the analytical operations of a beta counting system. The information shall be complete enough to allow someone skilled in the art to be able to reproduce any work performed with this type of a counter. This procedure was developed by PNL analytical radiochemists and is based on over 10 years of experience in analyzing samples for beta activity.

DEFINITIONS/ACRONYMS

cpm - counts per minute  
 bkg - background  
 LRB - Laboratory Record Book

RESPONSIBLE STAFF

Cognizant Scientist  
 Analyst/Technician

PROCEDURE

1.0 Tolerances for all measurements made during an analysis shall be specified in some manner: 1) State with a measurement value given in a method or 2) as specified below if not stated with a measurement value.

(a) Unless otherwise specified, all values for measurements stated in the methods (volume, weight, time, etc.) are approximate values. The actual measurements used, however shall be within  $\pm 10\%$  of the stated value.

Author <i>M. J. W. Hill</i>	Date 9-21-90	Project Mgr. <i>B. M. Gelspi</i>	Date 9-24-90	QAD Representative <i>C. Gerbe</i>	Date 9/24/90
Technical Reviewer <i>B. M. Gelspi</i>	Date 9-24-90	Line Mgr. <i>J. H. H. H.</i>	Date 9-21-90	Other	Date
Procedure No. PNL-ALO-463	Revision No. 0	Effective Date SEP 26 1990		Page 1	of 6

## PNL TECHNICAL PROCEDURE

- (b) When one or more significant figures are given to the right of the decimal point, the tolerance limit is  $\pm 5$  in the next digit located beyond the last one stated.

Except where responsibility is specifically assigned to a cognizant scientist, the analyst may perform all work. All pertinent data, observations and calculations, e.g., aliquot volumes, shall be recorded in a project record in a manner traceable to the sample (i.e., Beta Counting Data Sheet or an Analytical Data Sheet). All data pertaining to counter performance tests (control source counts), counter background counts, and instrument repairs shall be documented in the "Beta Counter Calibration" notebook.

### 2.0 Beta Counting Description

Both regular and low-background beta counting systems are located in the 329 Radiochemistry Lab. The background on the low-background systems is typically  $\leq 1$  cpm while the background on the regular systems is typically 10-20 cpm. Low-background beta counters are used to measure low-level concentrations of beta emitting radionuclides in samples.

The beta counting systems consist of end-window gas-flow proportional counters, pre-amplifiers, amplifiers, timers and scalers. The low background systems have an additional array of gas-flow proportional counters which surround the detector and provide pulses which serve to reduce the registration of counts from cosmic ray and other background sources by means of anticoincidence gate circuitry. The detectors operate with P-10 counting gas (90% argon + 10% methane). The counter windows are aluminized 1/4 mil mylar.

### 3.0 Geometry Measurement

The efficiency due to geometry of each instrument shall be measured using the  $^{90}\text{Sr} + ^{90}\text{Y}$  standard source whenever a detector has been repaired. The standard source currently in use contained 28312 dpm beta  $\pm 2\%$  on June 14, 1963, traceable to NIST, as referenced in LRB 53293, page 8. The source is evaporated on a 1-inch stainless steel dish. Using this source, counting efficiency of all shop-made counters in 329 Building has been maintained within  $\pm 3\%$  of the efficiency established in 1963. The history of these counters is documented in chronological Beta Counter Logbooks.

Another NIST-traceable beta source may be used to maintain the system as long as the efficiency determined with that source is first established in comparison to the historically-used standard source.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-463	0	A03-055 SEP 26 1990	2	6

PNL TECHNICAL PROCEDURE

- 3.1. To establish the geometry, place the beta standard source on the 1" dish holder assigned to the instrument being tested. Slide the dish holder into shelf 1 of the counter.
- 3.2 On the low background counters, press the "Noise Rejection Disabled" button. Set the counter for a counting interval sufficient to accumulate a minimum of 10,000 counts and start the count. Record the date, voltage setting of the power supply and the count time in minutes in the Beta Counter Calibration notebook on a Counter Control Data form (Exhibit 1). When the count interval is completed, record the number of counts.
- 3.3 Repeat step 3.2 for a total of five counts.
- 3.4 Obtain the most recent background cpm (within the last week) and record it. Compute the net cpm for each count as follows:

$$\text{net cpm} = \frac{\text{counts}}{\text{count time}} - \text{bkg cpm}$$

- 3.5 Determine the average net cpm for the five counts and record it. Determine the geometry as follows:

$$\text{efficiency due to geometry} = \frac{\text{average net cpm}}{\text{dpm in std}}$$

A d/c factor is often used in sample calculations instead of an efficiency factor (c/d). It is defined as:

$$\text{d/c} = \frac{\text{dpm in std}}{\text{net cpm}}$$

- 3.6 Record the geometry. The geometry of the system shall be within  $0.549 \pm 2\%$  (i.e., 0.538 - 0.560). If geometry is not within limits, a cognizant scientist shall evaluate the counter and shall perform repairs or adjustments, if necessary, and/or repeat steps in section 3.0. This evaluation and any actions taken shall be documented.
- 4.0 Counter Performance Tests and Quality Control

Control of each beta detector and counting system performance shall be established by measuring NIST-traceable control sources. These sources

Procedure No. PNL-ALO-463	Revision No. 0403-056	Effective Date SEP 26 1990	Page 3	of 6
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## PNL TECHNICAL PROCEDURE

consist of  $^{90}\text{Sr} + ^{90}\text{Y}$  solution evaporated on standard 1" stainless steel dishes.

Control of system performance shall be reestablished according to Section 3.0 and steps 4.1 - 4.5 when a new detector is put in operation, when control source analysis warrants it (see steps 4.4 - 4.6) or when a cognizant scientist determines that the replacement of an electronic component makes it necessary.

When instruments are in nearly continuous use, a periodic check of performance shall be made (at least once a week). If the instrument is not used for an extended period (2-3 weeks or more), these checks may be discontinued until the operations are again started. Control checks shall be made as follows:

- 4.1 Count the assigned control source ( $^{90}\text{Sr} + ^{90}\text{Y}$ ) on each counter for a time sufficient to accumulate a minimum of 10,000 counts (usually 10 minutes). On the low background counters be sure to press the "Noise Rejection Disabled" button. Record the date, high voltage setting, total counts, length of count and the most recently measured background cpm (within the last week) in the Beta Counter Calibration notebook. In addition, all instrument settings shall be recorded.
- 4.2 Calculate the net cpm for each count as follows:  
$$\text{net cpm} = \frac{\text{counts}}{\text{count time}} - \text{bkg cpm}$$
- 4.3 After a total of 10 control counts have been recorded, compute the mean and standard deviation and record on the Counter Control Data form.
- 4.4 When each subsequent control count is made, all instrument settings shall be examined and verification made that none of the settings established in step 4.1 have been changed (except for the count time). This verification shall be documented in the Beta Counter Calibration notebook with the control count data. If any settings have been changed, this shall be recorded. The system shall be posted "Out of Control" until a cognizant scientist evaluates the changes, establishes and documents the effect of any past data, and determines if any instrument repairs or adjustments are required.

Procedure No. PNL-ALO-463	Revision No. 0 A03-057	Effective Date SEP 26 1990	Page 4	of 6
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PNL TECHNICAL PROCEDURE

- 4.5 For control purposes, any subsequent control count shall be within 2% of the mean value or within  $\pm 2$  standard deviations of the mean, whichever is the larger limit. Suspect data is rerun.
- 4.6 If a count is not within the control limits, it shall be labelled "out" on the Counter Control Data form and the control source shall be recounted twice and recorded. If either recount is also outside the control limits, a sign shall be posted on the instrument: "Out of Control. Do Not Use" and a DR per PNL-MA-70, PNL-70-1502 shall be prepared. A cognizant scientist shall evaluate the system and determine if instrument adjustments and/or repairs are required and shall reestablish control of the counter. This evaluation and repair shall be documented in the Beta Counter Calibration notebook and on the DR. All samples counted on this detector since last acceptable control check shall be reanalyzed or recounted on detector that is in control unless the cognizant scientist has determined that the problem is clearly with the control count only. An example of this would be an error in the control count calculations.

5.0 Background Measurements

5.1 When a counter is in nearly continuous use, a background of the counter shall be taken at least once each week. Determine the background by counting a new, unused, ethanol-rinsed standard 1" dish. Count for 1000 minutes. Record the date, length of count and total counts in the Beta Counter Calibration notebook. Backgrounds are normally recorded on Counter Background forms (Exhibit 2).

5.2 Calculate the bkg cpm as follows:

$$\text{bkg cpm} = \frac{\text{counts}}{\text{count time}}$$

This bkg cpm is used in calculating control count and sample counting results.

5.3 Before using a detector for sample analysis, a Cognizant Scientist compares the current background with sample detection limit requirements and determines if the detector is suitable for the measurement. If the background is too high to achieve the requested detection limit, a different, lower-background detector shall be used, and/or the high-background detector shall be replaced with a new detector.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-463	0 A03-058	SEP 26 1990	5	6

## PNL TECHNICAL PROCEDURE

### 6.0 Counting Samples

Specific sample preparation and count times vary greatly depending on the matrices involved and the analyses desired. Specific step by step radioanalytical preparation procedures have been employed to cover the samples analyzed.

#### 6.1 Sample Counting Procedures

- 6.1.1 Place the sample on a counting dish and then place the dish onto the dish holder. Slide the dish holder into the counter.

Check to make sure voltage setting is the same as the current efficiency factor was based on (Exhibit 1).

- 6.1.2 Initiate the count and record the sample identification, the date/time of counting, and the length of count. When the count has completed, record the number of counts and record the most recent background cpm (within the last week) from the background data in the beta counter control notebook. All data shall be recorded in the sample LRB or on the Beta Counting Data Sheet for the sample.

### 7.0 Specific Qualifications

This procedure utilizes chemical standards for procedure control and as such meets the definition to be self-qualifying as per PNL-MA-70, PAP-70-901.

### 8.0 Records

All data pertaining to counter performance tests (control source counts), counter background counts, and instrument repairs shall be documented in the "Beta Counter Calibration" notebook in Room 16A of 329 Building.

Records will be maintained and controlled so as to conform to requirements of PNL-MA-70, PAP-70-1701. Laboratory Record Books (LRB) and Analytical Report Cards/Data Sheets provide a mechanism for control of most records. Laboratory Record Books will be used in accordance with the ACT NOW Directive 89.1.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-AO-463	0	A03-05 SEP 26 1990	6	6





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ICN- PNL-ALO-464.1  
Page 1 of 1

A. Document Number: <u>PNL-ALO-464</u> Revision Number: <u>0</u> Document Title: <u>Procedure for Gamma Counting and Data Reduction in the</u> <u>Document's Low-Level Counting Room, 329 Building</u> Original Author: <u>E. A. Lepel</u>	Effective Date of ICN: <u>4/26/91</u> Change Requested By: <u>E. A. Lepel</u>
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B. Action:  
Please replace pages 1, 6, 7, 9, 10, and 11 with the attached pages.

C. Effect of Change:  
To clarify requirements for use with the Low Energy Photon Detectors located in Rooms 13C and 16A of the 329 building.

D. Reason for Change/Description of Change  
Reason for Change:  
To clarify requirements for use with the Low Energy Photon Detectors located in Rooms 13C and 16A of the 329 building.  
Description of change: See attached

E. Approval Signatures (Please Sign and Date)	Type of Change: (Check ( / ) one) ( ) Minor Change <input checked="" type="checkbox"/> Major Change
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*Address Quality Dept  
QSR Department*  
Concurrence: [Signature] DE Date: 4/10/91  
Approval Authority: [Signature] Date: 4/12/91  
Other Approvals: [Signature] Date: 4/12/91  
: B M Dillipin Date: 4/15/91  
A03-062

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-464, PROCEDURE FOR GAMMA COUNTING AND DATA REDUCTION IN THE LOW-LEVEL COUNTING ROOM, 329 BUILDING

SCOPE

This procedure describes the generic methods of Gamma Spectroscopy used to obtain elemental and radionuclide concentrations in test materials (samples). It is applicable to samples that contain radionuclides emitting gamma photons with energies ranging from about 50 to 3000 keV and from about 4 to 250 keV for low energy photon detectors (LEPD).

This procedure is used to ensure that the gamma-ray spectrometry results obtained for various biological, geological, and other samples are accurate and reproducible. Although gamma spectrometry is a relatively straightforward method used to determine gamma-ray emitting radionuclide concentrations, it is crucial that all of the counting system components be operating properly on a day-to-day and year-to-year basis.

This procedure provides an overview of gamma spectroscopy, the spectrometer systems, and the data analysis programs used in the reduction of data. This procedure was developed by PNL Scientists, and reflects over 10 years of experience in gamma-ray spectroscopy of a wide variety of samples.

APPLICABILITY

This procedure is implemented to assure quality and is approved and written to conform with PNL-MA-70.

This procedure is applicable to the Counting Room Manager and trained and qualified personnel within the Analytical Chemistry Laboratory and Nuclear Chemistry Department of PNL.

This procedure is primarily intended to assure that the samples are counted on the gamma spectrometry equipment in the proper manner and that the data reduction is performed appropriately.

Author	Date	Project Mgr.	Date	QA0 Representative	Date
EA Lepel		BM Gillespie		GK Gerke	
Technical Reviewer	Date	Line Mgr.	Date	Other ALL SIGNATURES ON Date	
KH Abel		JM Latkovich		FILE WITH DOCUMENT CONTROL	
Procedure No.	Revision No.	Effective Date		Page	of
PNL-ALO-464	0	A03-063 SEP 26 1990		1	14

PNL TECHNICAL PROCEDURE

DEFINITIONS/ACRONYMS

- dpm - disintegrations per minute (concentration term)  
ng - nanograms ( $10^{-9}$  grams)  
ug - micrograms ( $10^{-6}$  grams)  
 $t_{1/2}$  - half life  
LRB - Laboratory Record Book  
CTL - Control  
Count - to acquire data via digitized pulses from radioactive sources in gamma spectrometer systems  
Control Source - aliquot of known mixed radioactive source contained in a specific counting geometry  
Diode - Detector - Part of the gamma detector system which includes the actual detector crystal in a metal cylindrical housing and its immediate electronics up to the preamplifier  
Ge(Li) - Lithium Drifted Germanium Detector.

RESPONSIBLE STAFF

Counting Room Manager  
Cognizant Staff Members/Analysts

PROCEDURE

Due to the diverse nature of experimental techniques that employ gamma spectroscopy, it is not feasible to write one all-encompassing procedure in detail to cover all of the possible variables. Rather, a generic procedure follows for a typical gamma spectroscopy system along with the equipment used. A brief discussion of spectrometer stability and reliability will also be included along with its relevance to gamma spectroscopy.

1.0 Tolerances

Tolerances for all measurements made during an analysis shall be specified in the following manner: 1) a tolerance limit can be stated with a measurement value given in a method, or 2) if a tolerance limit is not stated with a measurement value, then the following system of tolerances shall be in effect:

- a. When two or more significant figures are specified, the tolerance limit is  $\pm 5$  in the next digit beyond the last one stated. For example, 5.0 mL means  $5.0 \pm 0.05$  mL; 450 g means  $450 \pm 5$  g; 369 mL means  $369.0 \pm 0.5$  mL.

Procedure No. PNL-ALO-464	Revision No. 0 A03-064	Effective Date SEP 26 1990	Page 2	of 14
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PNL TECHNICAL PROCEDURE

b. If a single significant figure is specified, the actual measurement shall be within  $\pm 5\%$  of the stated value. For example, 20 mL means a volume between 19 and 21 mL.

2.0 Equipment and Materials

Radioactive Standard Sources such as the Amersham Mixed Radioactive Source which contains the following radionuclides:  $^{57}\text{Co}$ ,  $^{60}\text{Co}$ ,  $^{85}\text{Sr}$ ,  $^{88}\text{Y}$ ,  $^{109}\text{Cd}$ ,  $^{113}\text{Sn}$ ,  $^{137}\text{Cs}$ ,  $^{203}\text{Hg}$ , and  $^{241}\text{Am}$ ; and/or other radioactive control sources containing  $^{241}\text{Am}$ ,  $^{137}\text{Cs}$  and  $^{60}\text{Co}$ .

Detectors - NaI(Tl), Ge(Li), intrinsic germanium (IG), low-energy photon detector (LEPD), multidimensional detector.

Associated Electronics - high voltage power supply, preamplifier, amplifier, analog-to-digital converter, pulse pile-up rejector/live-time corrector, and multichannel analyzer.

DEC PDP 11/44, DEC uVAX, or a PC computer for data reduction.

3.0 General Information

A wide variety of gamma counters, each with distinct advantages, has been developed. According to common definition, the detectors are called either scintillation or solid-state detectors. The large sodium iodide scintillation detectors can be used to good advantage where it is necessary to count large samples since the counting geometry is not as critical. If the gamma spectrum is simple and of sufficient activity to overcome the system background, then the NaI(Tl) detector can count more samples per time than the solid-state diode detectors for a given accuracy. In general, the NaI(Tl) detector is more sensitive than the solid-state detector. However, the solid-state detector (diode) will have significantly better peak resolution.

3.1 The Building Blocks

A. Detector

The detector for the gamma counter may be NaI(Tl), Ge(Li), an intrinsic semiconductor detector (e.g., pure Ge) or a complex interconnection of the above detectors to enhance background suppression and permit coincidence/noncoincidence modes of operation.

The NaI(Tl) detectors are scintillators which require a photomultiplier closely coupled optically to the detector to

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-464	0 A03-065	SEP 26 1990	3	14

PNL TECHNICAL PROCEDURE

change the light pulses occurring in the NaI(Tl) to electrical pulses. The semiconductor detectors generate hole-electron pairs, which induce a current proportional to the incident gamma-ray energy.

B. Power Supply

Each detector requires a stable high voltage supply. Generally, about 1250 volts on the photomultiplier is used, or 2500-4000 volts on a semiconductor-type detector is used.

C. Preamplifier

The preamplifier is a very stable, linear-pulse amplifier used mainly for conditioning the detector signals and for driving the amplifier transmission line. Most recent preamplifiers are designed with an FET input transistor which operates at very low noise levels and small capacitances. The preamplifier also is designed as a pulse-pass filter, which effectively reduces noise levels passed on to the amplifier. Most preamplifiers have a gain on the order of 1 to 10.

D. Amplifiers

The modern amplifier has two functions: 1) to provide a gain in signal amplitude of about 1000 with very good stability, and 2) to act as a pulse-shaping network. The gain controls (coarse and fine) are two of the most important controls used by the operator, as these controls are used to adjust the standard peaks to the proper channels for energy peak calibration. It is important to remember that because the different electrical system components are interconnected, an adjustment of one component often affects the others. Thus, when an adjustment is made in one section, the total system shall be checked to see that it is working properly.

E. The Analog-To-Digital Converter (ADC)

The purpose of the ADC is to convert the pulse height of the amplifier pulse into a train of pulses (the train length is proportional to pulse height). Once digitized, the pulses are accepted into the analyzer memory. The zero adjust and the upper and lower discriminator controls are on the ADC. Usually, only the zero adjust is changed after the instrument is set up. Usually, this zero adjust is set to allow the first channel to correspond to 0 keV.

Procedure No.

PNL-ALO-464

Revision No.

0

A03-066 SEP 26 1990

Effective Date

Page

4

of

14

## PNL TECHNICAL PROCEDURE

The discriminator controls are for eliminating excessive noise counts. Typically, the upper level discriminator should be set just below the maximum energy being converted by the ADC. The lower-level discriminator is set to eliminate low-energy noise signals. Normally, these controls are initially set and require no further adjustment.

### F. Pulse Pile-Up Rejector

The pulse pile-up rejector is a special device connected to the amplifier and ADC. It contains special circuitry that detects incoming pulses that are so close together that the regular circuitry cannot determine that they are separate pulses. If these pulses are allowed to pass through the system, they would be recorded as summed pulses; that is, a large pulse of higher energy than the separate pulses. The pile-up rejector detects changes in the initial slope of the pulse to decide whether or not a pulse is to be accepted. If the pulse is rejected, the pile-up rejector turns off the internal timer and the ADC until sufficient time has elapsed for the sum pulse to decay away. Without the pulse pile-up rejector, the summed pulse would likely be counted as either representing the presence of a nonexistent radioisotope or added to the total counts in the spectrum in a channel area, which may or may not correspond to a real radioisotope. The pulse pile-up rejector is not needed for low count rates.

### G. The Analyzer Memory

The peak height of the counted pulse from the ADC is digitized so that it can be stored in the memory as a binary number. This allows a spectrum to be stored in a binary number distribution that is sorted according to precalibrated energy channels. At the end of the counting period, the appropriate channels (2048 or 4096) in the memory will contain the entire integrated spectrum in digitized form. These data may be retrieved in the form of counts per channel or may be transferred to a computer for rapid and complete data analysis.

## 4.0 Counting Systems

Any of these counting systems may be selected for counting a sample. Selection is based on sample type and size, anticipated level of radioactivity, specific radioisotopes of interest, required detection

Procedure No.	Revision No.	Effective Date	Page	of
PNL-AL0-464	0	A03-067 SEP 26 1990	5	14

## PNL TECHNICAL PROCEDURE

limits, etc., and the choice is made by the cognizant staff member/analyst.

### 4.1 Multidimensional Sodium Iodide System

A schematic diagram of a typical detector system with necessary electronics is shown in Figure 1. The electronic components are listed with their manufacturer model numbers. These, equivalent, or superior components are to be used.

A mixed  $^{60}\text{Co}$  and  $^{137}\text{Cs}$  standard is used for control determinations for each of the four multidimensional counting systems. Each standard is housed in a 1" x 6" diameter polyvinyl chloride (PVC) housing and absorbed onto  $\text{Al}_2\text{O}_3$  powder.

### 4.2 Low Energy Photon Detectors (LEPD)

The intrinsic Ge detector systems used as Low Energy Photon Detectors (LEPD) and typical associated electronic components are diagrammed in Figure 2. The electronic components are listed with their manufacturer model numbers. These, equivalent, or superior components are to be used.

Natural uranium standards are used for control standards. Each standard is in a pellet geometry enclosed in plastic to prevent detector contamination.

### 4.3 Conventional Ge(Li) (or Intrinsic Ge) Detector

Diode "L", a typical Ge(Li) detector, is used for measuring the gamma energy spectrum between 50-3000 keV. This diode and its associated electronic components are diagrammed in Figure 3. The electronic component configuration is shown with listed manufacturer model numbers. These components, equivalent, or superior models are to be used with this detector.

A mixed  $^{241}\text{Am}$ ,  $^{60}\text{Co}$ , and  $^{137}\text{Cs}$  standard is used for control determination. This standard closely approximates a point source and is mounted on aluminum plates specially designed to fit the machined plexiglass holder used for diode "L".

### 4.4 Coincidence-Noncoincidence Ge(Li)-NaI(Tl) Detector

The detection capability of these systems is similar to that of a standard Ge(Li) type (i.e., "L"). However, the coincidence-noncoincidence configuration offers better signal-to-noise ratio for single gamma emissions. Diode "I" and its associated

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-464	0	A03-068 SEP 26 1990	6	14

## PNL TECHNICAL PROCEDURE

electronic components are diagrammed in Figure 4. The electronic component configuration is shown with listed manufacturer model numbers. These components, equivalent, or superior models are to be used with this detector.

The control standard of  $^{241}\text{Am}$ ,  $^{60}\text{Co}$ , and  $^{137}\text{Cs}$  is similar to that used with standard Ge(Li) detectors.

### 5.0 General Gamma Spectrometry Procedure

#### 5.1 Calibration

Gamma-ray spectrometers must be calibrated so that the peak energies appear at identifiable channel numbers. Absolute efficiencies can then be calculated. This is accomplished using isotope standards with accurately-known disintegration rates. A typical isotope standard that has been used is a mixed radioisotope Standard Reference Material (SRM) containing  $^{57}\text{Co}$ ,  $^{60}\text{Co}$ ,  $^{85}\text{Sr}$ ,  $^{88}\text{Y}$ ,  $^{109}\text{Cd}$ ,  $^{113}\text{Sn}$ ,  $^{137}\text{Cs}$ , and  $^{203}\text{Hg}$ . Each radioisotope has known branching ratios for radioactive decay and easily recognizable (interference free) gamma-ray peaks. This SRM is ideally suited for determining the energy calibration and efficiency of the spectrometer. Although it is not necessary that the standard contain as many radioisotopes as indicated above to assure proper operation, it should at least have radionuclides with gamma energies across the spectrum being used for measuring unknown gamma energies. Typically, this covers the energy range of 50 to 3000 keV or about 4 to 250 keV for low energy photon detectors.

##### 5.1.1 Energy Calibration

The gamma-ray spectrometer is calibrated for energy by adjusting the analyzer amplifier gain to achieve the desired resolution. Typically, a gain of 0.5 or 1.0 keV per channel (See Section 3.1.D) is chosen by counting a standard radioisotopic source (control source) containing at least two radioisotopes--one with a high-energy gamma ray, and one with a low-energy gamma ray. After the gain is set by the analyst, the analyzer memory is cleared and the control source is counted per Section 5.2 of this procedure to check the gain setting. This energy calibration is usually performed after initial instrument set-up, after a detector is replaced, or after a detector is found to be out of control as determined by PNL-ALO-470.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-464	0 A03-069	SEP 26 1990	7	14

PNL TECHNICAL PROCEDURE

5.1.2 Detector System Counting Efficiencies

In addition to energy calibration, the gamma-ray spectrometer absolute counting efficiencies as a function of gamma energy must be determined for each detector and for each counting geometry in use on that detector. These counting-efficiency versus gamma-energy data are determined by counting a "standard" that is made from a nationally-recognized reference material of known radionuclide concentration and that is in the same physical geometry as the "unknown" or samples which are to be counted. The radionuclide reference material must contain known amounts of various radionuclides that emit gamma photons with energies well spaced over the normal range of analysis. The Amersham Mixed Radioactive Source is an example of a good reference material for use in making "standards" to be used in establishing system counting efficiencies.

The "standard" is counted in accordance with Section 5.2 for the time required to yield specific integrated gamma-ray peaks of  $\leq 5\%$  (preferably  $\leq 1\%$ ) relative error from counting statistics. The detector efficiency for a given integrated gamma-ray peak (radionuclide) is calculated by hand and/or by the computer by dividing the net count rate for the peak by the product of the activity of the radionuclide in the "standard" associated with that peak and the gamma-ray abundance of the radionuclide being measured. Efficiencies for radionuclides not specifically determined by counting the "standard" are interpolated by the computer from the efficiency data obtained from that "standard" for the detector and the specified counting geometry. Once the detector system counting efficiencies are established via this "standard," samples can be counted on the same system and quantitative results obtained as long as the samples are in the sample physical geometry and counted in the exact same position relative to the detector as the "standard," and the detector stability is maintained via the control check procedure (PNL-ALO-470). The currently "calibrated" geometries (i.e., sample geometries) for the gamma spectrometer are listed in the "Packaging and Handling Protocol for the Low-Level Counting Rooms 13C, 14C, and 4D, 329 Building," as well as in Procedure PNL-ALO-105, Sample Preparation for Gamma Counting. Counting efficiency data and/or curves are on file for the currently "calibrated"

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-464	0	SEP 26 1990	8	14

A03-070

## PNL TECHNICAL PROCEDURE

geometries in Room 14C of the 329 Building, and are stored in the counting system computer.

**Note:** The efficiency for a single specific energy may be determined instead of an efficiency curve as a function of energy when using a LEPS. This efficiency may be determined by a hand-calculated interpolation of data from the "standards" instead of by "computer."

Generally, detector/geometry counting efficiencies are determined at initial system set-up, when a new detector is installed, when new counting geometries are established for a given detector, or when a detector system is determined to be out of control per PNL-ALO-470.

### 5.1.3 Calibration Control Checks

The calibration of the detector system is monitored and checked by periodic counting of a system "control check source." This control source is a gamma-ray emitting source (of known gamma-ray energies and efficiencies) mounted on a holder which is placed onto the detector in always the exact same position. Spectral data from the counting of the "control check source" are examined for consistency of photopeak activities (integrated net peak areas). The counting of the control source checks that 1) the system is functioning properly, and 2) the system remains stable. The procedure for monitoring/checking the germanium spectrometers used in gamma-ray counting is PNL-ALO-470. At a minimum, the calibration control check (PNL-ALO-470) is conducted bi-monthly. More frequent calibration control checks may be conducted as specified by a Statement of Work (SOW), Technical Project Plan (TPP) or Project Quality Assurance Plan (QAPJP), or as determined by the cognizant staff member.

### 5.2 Sample and Standards Counting

After verifying that the detector system to be used is in control by checking the "Detector Reliability Chart" (See PNL-ALO-470), the basic sample counting/data acquisition procedure is as follows:

#### 5.2.1 Position

Position the sample in front of the detector using the appropriate sample holder for the required geometry.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-464	0	SEP 26 1990	9	14

A03-071

## PNL TECHNICAL PROCEDURE

### 5.2.2 Analyzer Start-Up

Clear the analyzer memory and store a pre-set live-time value. Start the analyzer counting and record the date and time on a "Counting Sheet," or record directly into memory, if appropriate. A typical counting sheet is shown in Figure 5. Fill in known information on the counting sheet. Initial the sheet with the individual's initials who started the count, and sign the bottom of the sheet. The signature at the bottom is only required once for each Counting Sheet. For non-radioactive and low-level radioactive samples, the sample count time is usually 1000 minutes.

### 5.2.3 Analyzer Shutdown

If the preset time has not elapsed and sufficient counts have been acquired in the regions of interest to meet precision requirements, stop the multichannel analyzer from acquiring data. The multichannel analyzer stops automatically when the preset time has elapsed. In either case, record the elapsed time if available and initials of the Analyst changing the count on the count sheet. If the Analyst is different than in 5.2.2, the Analyst also signs at the bottom of the sheet. This signature at the bottom is only done once on each Counting Sheet. Ready the analyzer to transmit the spectral data to a computer.

### 5.2.4 Data Transfer and Storage

Transfer the spectral data and accompanying sample parameters from the analyzer to the data acquisition computer system, and store the data on a magnetic storage device (hard disk, floppy disk, etc.).

### 5.2.5 Data Identification

The following parameters shall accompany the spectral data, on a hard copy printout from a printer and in the associated file stored on a magnetic storage device:

- a. Identification (sample description).
- b. Computer file name (if on computer) unique to the particular sample.
- c. Start time of the count.
- d. Time zero, i.e.; end of neutron bombardment for INAA and RNAA; or,

Procedure No. PNL-ALO-464	Revision No. 0	Effective Date A03-072 SEP 23 1990	Page 10	of 14
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## PNL TECHNICAL PROCEDURE

Date and time of sample collection for radionuclide direct counting.

- e. Live time of count (actual time the MCA is free to accept pulses). This should be determined by the MCA and transferred within the spectra.
- f. Real time (the actual duration of count in regular clock time).
- g. Sample mass and/or volume.

### 5.2.6 Data Analysis

Use a gamma-ray data reduction program to analyze the individual spectra and calculate the final concentration values. The calculated values may be verified by hand calculations, if necessary. The programs used are: "CANGAS" and/or "RAYGUN" for Instrumental Neutron Activation Analysis and Radiochemical Neutron Activation Analysis; "SUM" and/or "RAYGUN" for Natural Radionuclide counting; and "MDA" for multidimensional analysis. The data reduction employed for the LEPD systems may include the use of a PC spreadsheet or hand calculations.

### 6.0 Gamma Spectroscopy Data Reduction

The gamma-ray data reduction programs are used to generate activity and/or concentration values (d/m/unit, mg/g, %). The following describes a basic method used to calculate the information from results in an activity or concentration value. The same calculation may be performed by hand to verify the information. Pertinent calculations performed determine the net count rate,  $N$ , and net count rate uncertainty,  $\sigma N$ . The RAYGUN data analysis code is significantly more sophisticated.

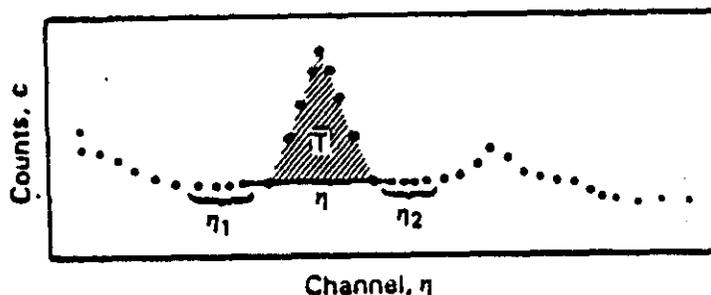
The principle of gamma-peak analysis is outlined below.

Net Count Rate (c/m),  $R$ : The net count rate for any particular gamma-ray is determined by integrating the channels containing the photopeak and subtracting the background Compton. The net counts,  $N$ , divided by the count time (CT) in minutes yields the net count rate (c/m).

This technique can be explained by using a simplified diagram of a gamma photopeak and a Compton continuum (Figure 6).

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-464	0 A03-073	SEP 26 1990	11	14

PNL TECHNICAL PROCEDURE



**FIGURE 6:** An Idealized Diagram of a Gamma Photopeak on a Compton Continuum

$$N \pm 1 \sigma_N \text{ error} = T - C \pm \left[ \frac{n^2}{4} \left( \frac{t_1}{n_1^2} + \frac{t_2}{n_2^2} \right) + T \right]^{1/2}$$

where

N = net counts  
 T = total counts in n channels of photopeak

$$= \sum_{i = n_1}^n C (\text{counts})_i$$

t<sub>1</sub> = total counts in n<sub>1</sub> channels of Compton continuum

t<sub>2</sub> = total counts in n<sub>2</sub> channels of Compton continuum

C = total counts in n Compton channels

$$= \frac{n}{2} \left( \frac{t_1}{n_1} + \frac{t_2}{n_2} \right)$$

PNL TECHNICAL PROCEDURE

Finally:

$$R \pm \sigma_R = \frac{N}{CT \text{ (min)}} \pm \frac{\sigma_R}{CT \text{ (min)}}$$

In some cases, the system's background ( $R_{Bkg}$ ) must be corrected for. In this case:

$$N^* \pm \sigma_{N^*} = N - R_{Bkg} (CT) \pm \left[ \sigma_N^2 + (\sigma_{Bkg} \times CT)^2 \right]^{1/2}$$

where

$R_{Bkg}$  = System's background count rate (c/m)

$N^*$  = System's background corrected net counts

In all cases, this calculation is performed; generally the system's background count rate is insignificant when counting control standards. The system backgrounds and their uncertainties are obtained from long ( $\geq 1000$  min) background counts.

The count rate (R) is then used in the calculation of activity or concentration as calculated by RAYGUN, SUM, or MDA for "direct counting," and RAYGUN or CANGAS for comparative analysis as used in instrumental neutron activation analysis.

Further information on each program is contained in the User's Guide located in the Low-Level Counting Room in the 329 Building, or from references, Laul, et al. and/or Hensley, et al.

### 7.0 Specific Qualifications

This procedure utilizes radiochemical standards for control, and as such meets the definition to be self-qualifying as per PNL-MA-70, PAP-901.

Procedure No.

PNL-ALO-464

Revision No.

0 A03-075

Effective Date

SEP 26 1990

Page

13

of

14

PNL TECHNICAL PROCEDURE

8.0 Records

Records will be maintained and controlled so as to conform to requirements of PAP-70-1701. Laboratory Record Books (LRBs) and Data Sheets provide a mechanism for control of most records. Laboratory Record Books will be used in accordance with the Act Now Directive 89.1.

9.0 References

Laul, J. C., C. L. Wilkerson, and V. L. Crow. 1979. "Computer Methodology and It's Application to Geological and Environmental Matrices". Computers in Activation Analysis and Gamma-Ray Spectroscopy, Proceedings of the American Nuclear Society Topical Conference at Mayaguez, Puerto Rico. April 30 - May 4, 1978. CONF-780421: 840-856.

Hensley, W. H., E. A. Lepel, M. E. Yuly, and K. H. Abel. 1988. "Adaptation and Implementation of the RAYGUN Gamma-Ray Analysis Code on the IBM PC." J. Radioanalytical and Nuclear Chemistry, Articles 124(2): 481-499.

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Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-464	0 A03-076	SEP 26 1990	14	14

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A03-077

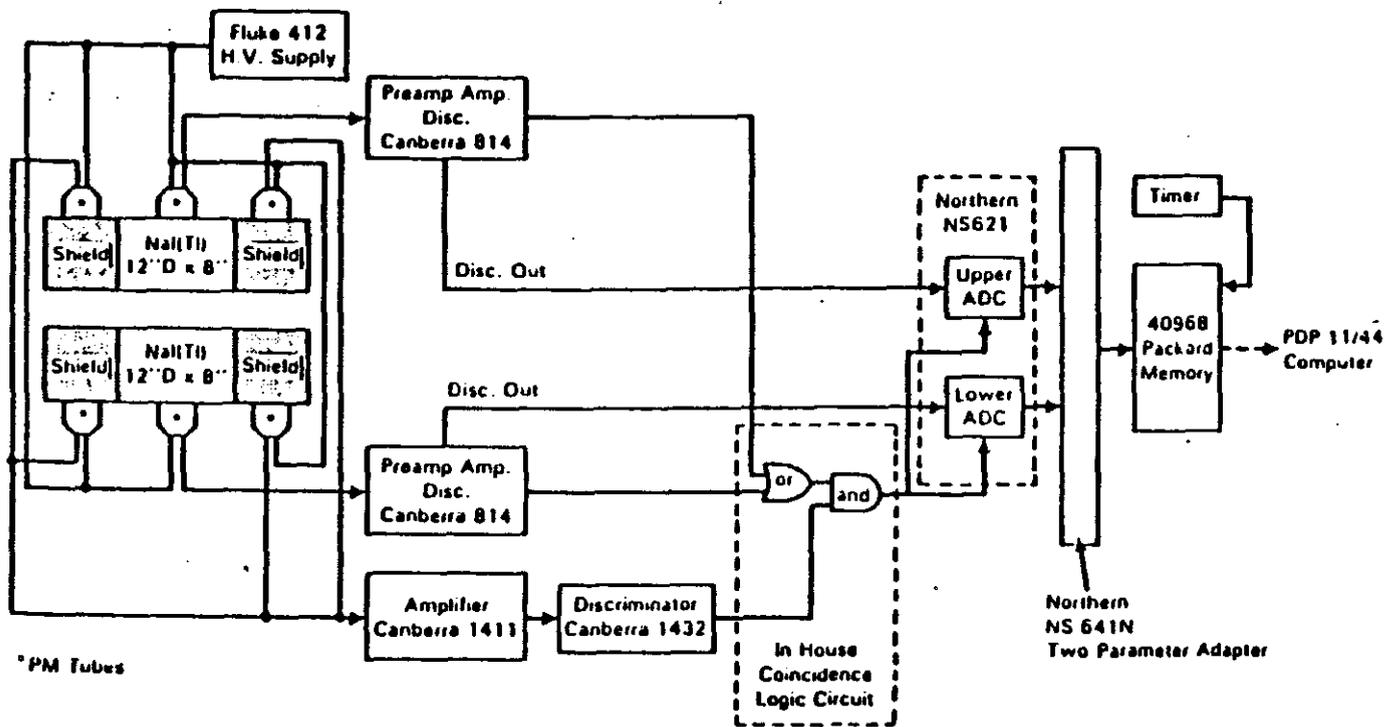


FIGURE 1. Multidimensional System Electronic Block Diagram of a Gamma-Gamma Multidimensional Spectrometer System

A03-078

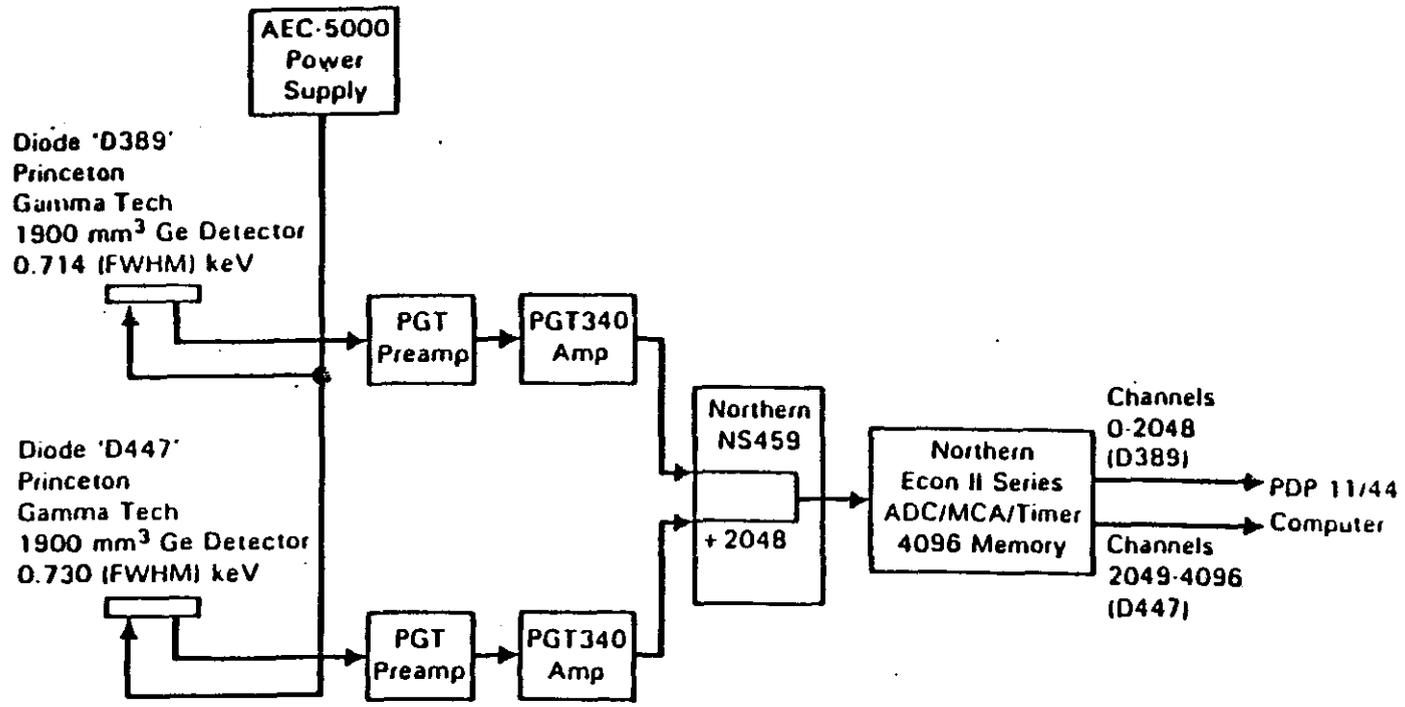


FIGURE 2. Low Energy Photon Detector Electronic Block Diagram for Diodes 'D389' and 'D447'

9 2 1 1 2

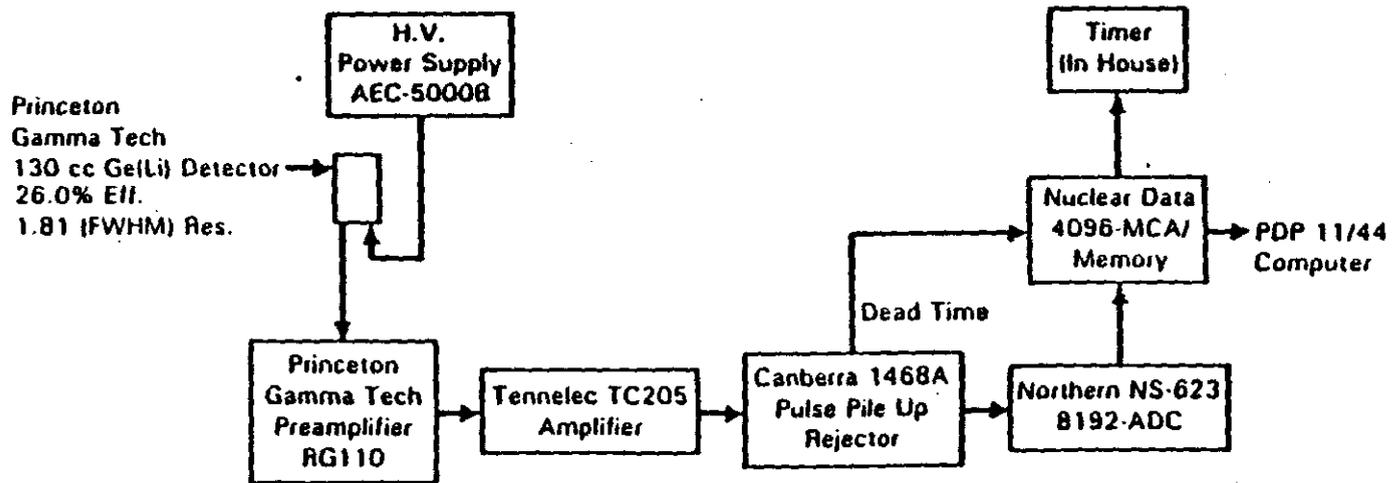


FIGURE 3. Conventional Ge(Li) Detector Electronic Block Diagram for Diode 'L'

A03-079

A03-080

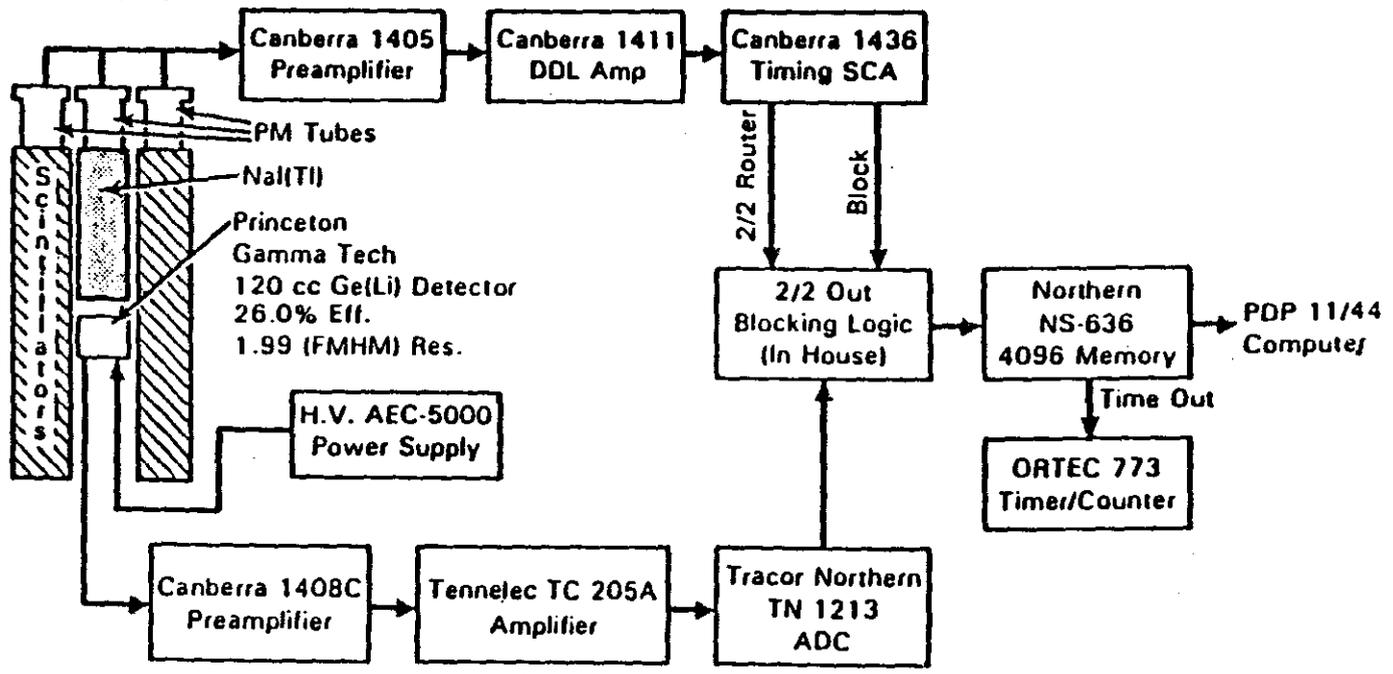


FIGURE 4. Anticoincidence-Coincidence Electronic Block Diagram for Diode 'I'





INTERIM CHANGE NOTICE  
(ICN)

ICN-PNL-ALO-465.3

Page 1 of 1

A. Document Number: <u>PNL-ALO-465</u> Revision Number: <u>0</u>	Effective Date of ICN: <u>11/04/91</u>
Title: <u>Strontium-90 Analysis</u> Document's Original Author: <u>NL Wynhoff</u>	Change Requested By: <u>SK Fadeff</u>

B. Action  
Change step 4.41  
Replace pages 9-11

C. Effect of Change  
Allows for more complete separation of  $\text{Na}_2\text{CO}_3$  from  $\text{SrCO}_3$ . This will provide a more accurate product weight and therefore, more accurate results.

D. Reason for Change/Description of Change  
 $\text{Na}_2\text{CO}_3$  is not always sufficiently washed away from the  $\text{SrCO}_3$  product. This procedural change will allow for complete separation.  
Replace step 4.41 with the following:  
Repeat step 4.40 except pour off the supernate into a clean beaker. Add a drop of  $\text{Ca}^{2+}$  solution and visually check for a cloudy precipitate. Discard the supernate solution. Repeat washing step until an insignificant amount of  $\text{CaCO}_3$  precipitate forms in the supernate solution (as evidenced by visual examination). At this point,  $\text{Na}_2\text{CO}_3$  is negligible in solution.

E. Approval Signatures (Please sign and date)	Type of Change: (Check (✓) one) <input type="checkbox"/> Minor Change <input checked="" type="checkbox"/> Major Change
Process Quality Department: GK Gerke <u>[Signature]</u> <u>DE</u>	Date: <u>11/5/91</u>
Approval Authority: AG King <u>[Signature]</u>	Date: <u>11/8/91</u>
Other Approvals: BM Gillespie <u>[Signature]</u>	Date: <u>11/12/91</u>
:	Date: <u>   /   /   </u>

INTERIM CHANGE NOTICE  
(ICN)

ICN-PNL-ALO-465.2-R0  
Page 1 of 1

A. Document Number: <u>PNL-ALO-465</u> Revision Number: <u>0</u>	Effective Date of ICN: <u>9/26/91</u>
Document Title: <u>Strontium - 90 Analysis</u>	Change Requested By: <u>S. K. Fadeff</u>
Document's Original Author: <u>N. L. Wynhoff</u>	

B. Action

Replace pages 8 through 11 with the attached pages.

C. Effect of Change

To make procedure read like it did before ICN-PNL-ALO-465.1.

D. Reason for Change/Description of Change

Reason for Change: Sections 4.22 and 4.23 were inadvertently left out in last ICN.

Description: Insert sections 4.22 and 4.23 at the top of page 8.

E. Approval Signatures	Type of Change: (Check (✓) one)
(Please sign and date)	<input type="checkbox"/> Minor Change <input checked="" type="checkbox"/> Major Change
Process Quality Department: <u>CK Gerbe, DE</u>	Date: <u>9/30/91</u>
Approval Authority: <u>[Signature]</u>	Date: <u>10/6/91</u>
Other Approvals: <u>[Signature]</u>	Date: <u>10/4/91</u>
	<u>A03-084</u> Date: <u>   /   /   </u>

INTERIM CHANGE NOTICE  
(ICN)

A. Document Number: <u>PNL-AL0-465</u> Revision Number: <u>0</u>	Effective Date of ICN: <u>7/30/91</u>
Document Title: <u>Strontium - 90 Analysis</u>	Change Requested By: <u>S. K. Fadeff</u>
Document's Original Author: <u>N. L. Wynhoff</u>	

B. Action:  
Replace pages numbers 6 and 7.

C. Effect of Change:  
Allows analyst to address operational needs during the strontium separation that the original procedure version did not allow for.

- D. Reason for Change/Description of Change
1. It is difficult to adjust the pH to exactly 5.0, an acceptable pH range is more appropriate for step 4.14.
  2. Plactic centrifuge tubes are less expensive and are perfectly acceptable for use in step 4.16. In some cases, the supernate volume is too large to be handled in one centrifuge tube.
  3. Environmental samples can be different enough from each other and thus require a pH range before precipitate will form (section 4.17) upon addition of NH<sub>4</sub>OH. If pH target is overshoot, the analyst needs to back-adjust with acid.
  4. Soil samples typically need a larger volume of 6M HCl for complete dissolution of precipitate, Section 4.19. A more successful dissolution can be obtained if oxalic acid is added after the 6M HCl.
- CONTINUED ON PAGE 2

E. Approval Signatures  (Please Sign and Date)	Type of Change:	(Check ( / ) one)
	( ) Minor Change	( X ) Major Change
Process Quality <del>Secret</del> Department Concurrence: <u>G. Kerber DE</u>	Date: <u>3/5/91</u>	
Approval Authority: <u>[Signature]</u>	Date: <u>3/18/91</u>	
Other Approvals: <u>B. M. [Signature]</u>	Date: <u>3/11/91</u>	
	Date: <u>1/1</u>	

INTERIM CHANGE NOTICE  
(ICN)

ICN- PNL-ALO-465.1  
Page 2 of 2

DESCRIPTION OF CHANGES

1. Section 4.14 - Delete "5.0"; insert "4.5 to 5.5".
2. Section 4.16 - In second sentence, add "or plastic" right after the word "glass" and before "centrifuge". Add "(if supernate volume is large, additional centrifuge tubes may be used to collect the supernate.)" to the end of the second sentence.
3. Section 4.17 - After the second sentence, add "If a precipitate does not form after reaching pH 5, continue adding  $\text{NH}_4\text{OH}$  until a precipitate does form (up to pH 7.5).

After the first sentence, add "If the solution volume is large or intensely colored such that the color change of brom cresol green indicator is masked, 5 to 12 more drops of indicator may be added."

At the end of the second sentence, add "and  $\text{HNO}_3$ ."

4. Section 4.19, Prior to first sentence, insert "Dissolve sample in 5 mL HCl or the minimum volume needed to obtain complete dissolution."

Delete second and third sentences.

Insert "Stir vigorously and" to the beginning of the last sentence.

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-465, STRONTIUM-90 ANALYSIS (OXALATE-NITRIC ACID METHOD)

APPLICABILITY

This procedure is employed to analyze for Sr-90 in water and liquid leachate samples. If the sample volume is large, an initial volume reduction is made by precipitating strontium and other alkaline earth metals as carbonates. The carbonates are then redissolved with nitric acid. Additional purification is accomplished by precipitation as strontium oxalate metathesizing to strontium nitrate. The strontium is finally precipitated as a carbonate, washed to remove the excess carbonate, and dried on a dish. Strontium activity is determined by beta counting. The yield is determined gravimetrically by comparing the amount of stable strontium carrier added with the weight of the strontium carbonate on the dish and by determining the Sr-90 yield from a spiked sample run with each sample batch. This procedure was developed by PNL Radioanalytical Scientists, and is based on over 10 years experience in Sr radioanalytical chemistry.

DEFINITIONS/ACRONYMS

- dpm - disintegrations per minute
- cpm - counts per minute
- ppt - precipitate
- sat. - saturated
- conc. - concentrated
- method blank - deionized or distilled water (treated the same as a sample in process).
- LRB - Laboratory Record Book

RESPONSIBLE STAFF

Cognizant Scientist  
 Technician/Analyst

(Note: Except where responsibility is specifically assigned to a Cognizant Scientist, the Technician/Analyst may perform all work.)

Author <i>M. W. Zyl</i>	Date 9-21-90	Project Mgr. <i>B. M. Dillipuri</i>	Date 9-24-90	QA Representative <i>C. Kerbe</i>	Date 9/24/90
Technical Reviewer <i>G. O. Harvey</i>	Date 9/24/90	Line Mgr. <i>J. F. Salt</i>	Date 9-27-90	Other	Date
Procedure No. PNL-ALO-465	Revision No. 0	Effective Date SEP 26 1990	Page 1	of 11	

PNL TECHNICAL PROCEDURE

PROCEDURE

1.0 Tolerances for all measurements made during an analysis shall be specified in the following manner: 1) a tolerance limit can be stated with a measurement value given in a method, or 2) if a tolerance limit is not stated with a measurement value, then the following system of tolerances shall be in effect:

- (a) Unless otherwise specified, all values for measurements stated in the method (volume, weight, time, etc.) are approximate values. The actual measurements used, however, shall be within  $\pm 10\%$  of the stated value.
- (b) When one or more significant figures are given to the right of the decimal point, the tolerance limit is  $\pm 5$  in the next digit located beyond the last one stated.

2.0 Quality Control

Two quality control options (A and B) are defined. Option A shall be used unless a Statement of Work (SOW) written by the client defines CERCLA requirements. Option B shall then be used. The cognizant scientist will recognize the need to use option B when a Chain-of-custody (COC) defines a Test Instruction. Additional quality control samples may be requested by the client in an Analytical Request Form (ARF) or a Statement of Work (SOW). The cognizant scientist will convey these additional requirements to the analyst through the Test Instruction (TI).

A. At a minimum a method blank and a Sr-90 spike sample shall be analyzed with every set (8 or less) of samples. This spiked sample is used in calculating the sample yield.

Note: A spike sample for soils/solids/sludges is defined in Section 4.0, QC for Soils/Solids/Sludges.

B. For all SOWs written by a client for CERCLA requirements that request analysis, employ a minimum of one method blank, one duplicate and a Sr-90 spike sample for each set (set size is 8 or less). This spiked sample is used in calculating the sample yield.

Note: A spike sample for soils/solids/sludges is defined in Section 4.0, QC for Soils/Solids/Sludges.

The beta counting instruments shall be calibrated according to the current beta counting procedure (PNL-ALO-463 or equivalent).

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-465	0	SEP 26 1990	2	11

PNL TECHNICAL PROCEDURE

3.0 Reagents and Materials

Reagents - All reagent chemicals will be reagent grade unless specified.

Deionized or Distilled Water - All water used as a solvent in this procedure will be deionized or distilled.

Sr-Y-90 Spike (Sr-90 and Y-90 in secular equilibrium). Use appropriate concentration as designated by a Cognizant Scientist (See Section 4.0). Documentation of spike origin and traceability is in a LRB under control of a Cognizant Scientist.

Strontium Carrier, 20 mg/ml - Dissolve  $48.32 \pm 0.01$  g strontium nitrate ( $\text{Sr}(\text{NO}_3)_2$ ) in water. Dilute to one liter with water in a Class A volumetric flask.

Nitric Acid, Conc.

Nitric Acid, 8 M - dilute 500 ml conc.  $\text{HNO}_3$  with water to 1000 ml.

Nitric Acid, 2 M - dilute 125 ml conc.  $\text{HNO}_3$  with water to 1000 ml.

Fuming Nitric Acid, 90%.

Sodium Hydroxide, 12 N - Dissolve 480 g of NaOH in 800 ml of water, allow to cool to less than  $30^\circ\text{C}$ , then dilute to one liter with water.

Ammonium Hydroxide - Any concentration from 6 M to concentrated.

Ammonium Hydroxide, 4% - 4 ml conc.  $\text{NH}_4\text{OH}$  diluted to 100 ml with water.

Phenolphthalein, 1% - Dissolve 2.5 g phenolphthalein in 12.5 ml ethanol and dilute to 250 ml with water.

Sodium Carbonate, Sat. - Dissolve anhydrous  $\text{Na}_2\text{CO}_3$  in one liter of water until saturated.

Brom Cresol Green, 0.04% - Mix 0.1 g brom cresol green with  $14.3 \pm 0.2$  ml 0.01 M NaOH in a mortar and pestle. Transfer to a flask that will hold a volume of 250 ml. Dilute mixture to 250 ml with water.

Iron Carrier, 10 mg/ml - Dissolve 36.23 g  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  in  $\text{H}_2\text{O}$ . Dilute to 500 ml with water. (Note: Another ferric salt may be

92126

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-465	0	SEP 26 1990	3	11

A08-089

PNL TECHNICAL PROCEDURE

used or another concentration as long as the required mg of Fe<sup>+3</sup> are added.)

Oxalic Acid, Sat. - Dissolve oxalic acid in one liter of water until saturated.

Hydrochloric Acid, 6 M - Dilute 500 ml conc. HCl with water to 1000 ml.

Ethanol.

Materials and Equipment

Beakers - 150 ml to 4000 ml.

Glass or Plastic Centrifuge Tubes - 50 or 100 ml.

Centrifuge - for above tubes. The centrifuge is operated at the highest, safe speed possible (i.e., such that the glass or plastic centrifuge tubes do not break).

Vortex Stirrer.

Hot Plate/Stirrer.

Heat Lamp.

Stir Bar - 1- to 2-inch, Teflon coated, magnetic.

pH Paper - fine range near 1, and fine range centered near 5.0.

Flamed Stainless Steel Counting Dishes - heat on high on porcelain-top hot plate until color turns dark (2-3 min), then cool.

4.0 Chemical Procedure

QC for Solutions

Unless otherwise specified in project QC requirements (See Section 2.0), take two aliquots of deionized distilled water. Label one "Blank" (method blank) and one "Spike" (spike sample). The volume of these aliquots shall be 25 ml if the sample volumes will be less than 50 ml and 100 ml if the sample volume will be 50 ml or more. Add Sr-Y-90 tracer to "Spike" as directed by the Cognizant Scientist. This is usually 2000-3000 dpm of Sr-Y-90 at secular equilibrium. Analyze the "Blank" and "Spike" along with the sample batch.

9 2 1 3 0 9 0 1 1 3

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-465	0	A03-090 SEP 26 1990	4	11

PNL TECHNICAL PROCEDURE

Note: Record in LRB, on Beta Counting Data Sheet or an Analytical Data Sheet the exact volume and calibrated activity of Sr-Y-90 tracer added, since Sr-90 recovery is used in calculating sample yields.

QC for Soils/Solids/Sludges

Unless otherwise specified in project QC requirements (See Section 2.0), take an aliquot of water and label it "Blank" (method blank). The volume of the aliquot shall be 25 ml if the sample volumes will be less than 50 ml and 100 ml if the sample volume will be 50 ml or more. Spike a soil/solid/sludge sample as directed by the Cognizant Scientist before leaching. Label it "Spike" (spike sample). This is usually 2000-3000 dpm of Sr-Y-90 at secular equilibrium. Analyze the "Blank" and "Spike" along with the sample batch.

Note: Record in LRB, on Beta Counting Data Sheet or on an Analytical Data Sheet the exact volume and calibrated activity of Sr-Y-90 tracer added, since Sr-90 recovery is used in calculating sample yields.

4.1 If sample volume to be used for Sr analysis, as specified by a Cognizant Scientist, is less than 50 ml, add 2000 ul of standardized 20 mg/ml Sr carrier to sample and proceed to Step 4.11. If sample volume is  $\geq 50$  ml, proceed to Step 4.2.

Note: Record in the LRB, on Beta Counting Data Sheet, or Analytical Data Sheet the exact volume and standardized concentration of Sr carrier added, since yields are based on carrier recovery.

4.2 If sample is not in acid media, add 5 ml conc.  $\text{HNO}_3$  to sample. Shake or mix sample and acid until thoroughly mixed.

4.3 Transfer an aliquot, as specified by a Cognizant Scientist, of acidified sample to beaker labeled with sample identification. If total sample volume is less than 1 liter, approximately, measure volume of remaining sample. Record volume of aliquot and remaining sample volume, if required, in the LRB, on the Beta Counting Sheet, or Analytical Data Sheet.

4.4 Add 2000 ul of standardized 20 mg/ml Sr carrier to beaker.

Note: Record in LRB, on the Beta Counting Data Sheet, or Analytical Data Sheet the exact volume and standardized

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-465	0	SEP 26 1990	5	11

A03-091

PNL TECHNICAL PROCEDURE

concentration of Sr carrier added, since yields are based on carrier recovery.

- 4.5 Add 3-5 drops phenolphthalein indicator. Add 12 M NaOH drop-wise with stirring until red color persists (pH ~8).
- 4.6 Add sat. sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) solution to sample with stirring until precipitate forms. Add an additional 30 ml ( $\pm 5$ )  $\text{Na}_2\text{CO}_3$  (in excess). Stir vigorously and allow to cool or chill to room temperature or less.

Note: Solution must be basic for ( $\text{SrCO}_3$ ) precipitation. Temperature is also a factor since  $\text{SrCO}_3$  is soluble to the extent of 0.01 mg/ml  $\text{H}_2\text{O}$  at 18°C and 0.65 mg/ml at 100°C.

- 4.7 Allow precipitate to settle for at least one hour (preferably overnight or longer) while cold, to achieve a clear supernatant.

Note: Settling must be complete (supernate must be clear) before decanting supernate.

- 4.8 Carefully slurp (withdraw under vacuum) or decant supernate to waste. Quantitatively transfer precipitate to labeled centrifuge tube with minimum (<10 ml) deionized  $\text{H}_2\text{O}$ .
- 4.9 Centrifuge for 5-10 minutes and discard supernate.
- 4.10 Dissolve precipitate with a minimum of 8 M  $\text{HNO}_3$ . (It is usually convenient to add it drop-wise with constant stirring using vortex stirrer until all ppt is dissolved.)
- 4.11 Checking pH with a fine range pH paper, continue addition of 8N  $\text{HNO}_3$  until pH is less than 1.
- 4.12 Add 6-12 drops brom cresol green indicator.

Note: Indicator is yellow at pH lower than 4.5, green at pH 5, and blue at pH 5.5 and above.

- 4.13 Add 1 ml of 10 mg/ml Fe carrier to acidic sample.
- 4.14 Slowly adjust pH back to 4.5 to 5.5 (green color) with drop-wise addition of ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) (any concentration between 6N and conc. may be used). Use vortex stirrer.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-AL0-465	0	A03-092 SEP 26 1990	6	11

PNL TECHNICAL PROCEDURE

Note: It may be useful to also check pH with a fine range pH paper whose range is centered near pH 5.0.

4.15 Cool to room temperature and allow to sit while precipitate forms. If no precipitate forms, add 5-10 drops of Fe carrier and readjust pH to 5.0.

4.16 Centrifuge for 5-10 minutes. Decant supernate to clean, labeled glass or plastic centrifuge tube (if supernate volume is large, additional centrifuge tubes may be used to collect the supernate.) Wash precipitate twice thoroughly with 5-10 ml H<sub>2</sub>O. Centrifuge after each wash for 5-10 minutes and add supernate to previous supernate in tube. Discard precipitate.

Note: Scavenger step removes insoluble particles, silicates, Fe and other contaminants, leaving a relatively pure solution.

4.17 Add 15 ml sat. oxalic acid to supernate. If the solution volume is large or intensely colored such that the color change of brom cresol green indicator is masked, 5 to 12 more drops of indicator may be added. Stir vigorously, adjusting pH to green point (pH 5.0) with drop-wise addition of NH<sub>4</sub>OH and HNO<sub>3</sub>. If a precipitate does not form after reaching pH 5, continue adding NH<sub>4</sub>OH until a precipitate does form (up to pH 7.5). Cool in ice-water bath to below room temperature.

Note: Vigorous stirring is essential: use vortex or mechanical stirrer.

Note: SrC<sub>2</sub>O<sub>4</sub> (strontium oxalate) has definite temperature-dependent solubility of 0.05 mg/ml H<sub>2</sub>O at 18°C and 50 mg/ml at 100°C.

4.18 Centrifuge 5-10 minutes. Discard supernate.

OK  
(scrip)  
4.19 Dissolve sample in 5 mL HCl or the minimum volume needed to obtain complete dissolution. Add 5 ml sat. oxalic acid. Stir vigorously and dilute with 15 ml (±5) H<sub>2</sub>O.

4.20 Add 8-12 drops of brom cresol green. Increase pH to indicator green point (pH 5.0) with drop-wise addition of conc. NH<sub>4</sub>OH, stirring vigorously. Cool to room temperature.

4.21 Centrifuge solution 5-10 minutes. Discard supernate.

Procedure No. PNL-ALO-465	Revision No. 0 A03-093	Effective Date SEP 26 1990	Page 7	of 11
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PNL TECHNICAL PROCEDURE

Note: Strontium oxalate metathesizes to strontium nitrate.

- 4.24 Centrifuge solution 5-10 minutes. Discard supernate.
- 4.25 Repeat Steps 4.23 and 4.24 one time.
- 4.26 Dissolve precipitate in 1-3 ml H<sub>2</sub>O. Add 5 ml fuming nitric and 20 ml conc. nitric acid, stirring vigorously for 1 minute. Cool to room temperature.

Note: Begin Step 4.30 only when beta counters are in control and when sufficient time is available to complete chemical separation (2 hours or more) and have samples counted within 10 hours of the Sr separation time. It is usually best to allow a full day.

- 4.27 Check with counting room personnel on availability of beta counters. Reserve counters for overnight background counts and for sample counting.
- 4.28 Count backgrounds overnight on beta counters. See beta counting procedure, PNL-ALO-463.
- 4.29 Count control sources and plot on control charts. Confirm that counters are in control per procedure PNL-ALO-463 before continuing with Step 4.30.
- 4.30 Centrifuge precipitate from Step 4.26 5-10 minutes and discard supernate.
- 4.31 Wash precipitate once with 15 ml conc. HNO<sub>3</sub>. Centrifuge precipitate 5-10 minutes and discard supernate.
- 4.32 Dissolve ppt in 10 ml 2N HNO<sub>3</sub> (or more, if necessary). Add 1 ml of 10 mg/ml Fe carrier. Heat in hot water bath to 80°C.
- 4.33 Adjust to pH 8 with conc. NH<sub>4</sub>OH (check with pH paper). Record the time conc. NH<sub>4</sub>OH was added as the "Sr separation time" on Beta Counting Data Sheet or Analytical Data Sheet.
- 4.34 Cool in ice bath. Centrifuge precipitate 5-10 minutes. Decant supernate into a clean centrifuge tube.
- 4.35 Wash ppt with 4% NH<sub>4</sub>OH. Centrifuge precipitate 5-10 minutes. Add supernate to previous supernate in clean tube.

Procedure No. PNL-ALO-465	Revision No. 0	Effective Date A03-094 SEP 26 1990	Page 8	of 11
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PNL TECHNICAL PROCEDURE

- 4.33 Adjust to pH 8 with conc.  $\text{NH}_4\text{OH}$  (check with pH paper). Record the time conc.  $\text{NH}_4\text{OH}$  was added as the "Sr separation time" on Beta Counting Data Sheet or Analytical Data Sheet.
- 4.34 Cool in ice bath. Centrifuge precipitate 5-10 minutes. Decant supernate into a clean centrifuge tube.
- 4.35 Wash ppt with 4%  $\text{NH}_4\text{OH}$ . Centrifuge precipitate 5-10 minutes. Add supernate to previous supernate in clean tube.
- 4.36 Repeat Step 4.35. Discard ppt.
- 4.37 Add 1 drop phenolphthalein indicator.
- 4.38 Add 15 ml sat.  $\text{Na}_2\text{CO}_3$  with vigorous stirring for 1 minute. Cool to room temperature.

Note: Samples must be cool before next step.

- 4.39 Centrifuge precipitate 5-10 minutes. Discard supernate.
- 4.40 Wash with 5-10 ml of  $\text{H}_2\text{O}$ . Centrifuge precipitate and discard supernate.
- 4.41 Repeat Step 4.40, ~~but do not transfer ppt to a new tube, except pour off the supernate into a clean beaker.~~ Add a drop of  $\text{Ca}^{++}$  solution and visually check for a cloudy precipitate. Discard the supernate solution. Repeat washing step until an insignificant amount of  $\text{CaCO}_3$  precipitate forms in the supernate solution (as evidenced by visual examination). At this point,  $\text{Na}_2\text{CO}_3$  is negligible in solution.

- 4.42 Transfer precipitate on flamed, tared 1" stainless steel dish using  $\text{H}_2\text{O}$  or ethanol. Dry to stable weight under heat lamp.

Note: Be careful to maintain sample identity of sample dishes.

- 4.43 Cool and weigh plated sample. Record net weight and balance identification in LRB, on Beta Counting Data Sheet, or Analytical Data Sheet. Count samples immediately and complete the counting within 10 hours of the Sr separation time.
- 4.44 Take initial 1- to 5-minute count on regular beta counters. If <25 cpm, count samples on low-background counters.
- 4.45 Record all pertinent data, including sample identity, sample volume, Sr separation time, precipitate weight, Analyst, and any other data requested by the Cognizant Scientist. A Beta Counting Sheet is normally used to record this and all counting data.

Procedure No. PNL-ALO-465	Revision No. 0 A03-095	Effective Date SEP 26 1990	Page 9 OF 11
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PNL TECHNICAL PROCEDURE

5.0 Calculations

$$YS = \frac{\{cpm - cpm (1 - e^{-\lambda y \Delta t})\} \times TSC \times d/c}{WP \times dpm \text{ Sr-90}}$$

Where:

YS = yield of Sr-90 spike.

cpm = Gross counts per minute less background counts per minute, for "Spike".

Net Counts = Gross counts per minute less background counts per minute.

TSC = theoretical SrCO<sub>3</sub> ppt. weight for 100% yield for amount of Sr carrier added to the sample, in mg.

λy = decay constant of Y-90

Δt = interval between the Sr Separation Time and the mid-point of the sample count

d/c = disintegrations per count, an efficiency factor. This factor is provided by a Cognizant Scientist from documented, NIST-traceable calibration measurements. Documentation of origin and traceability is in a LRB under the control of a Cognizant Scientist.

WP = weight of SrCO<sub>3</sub> ppt. on dish, in mg.

dpm Sr-90 = dpm Sr-90 tracer added to "Spike."

$$A = \frac{\{cpm - cpm (1 - e^{-\lambda y \Delta t})\} \times TSC \times d/c}{WP \times YS \times B}$$

Where:

A = Sr-90 activity of the sample (units depend on requested reportable units)

cpm = Gross counts pr minute less background counts per minute, for the sample.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-465	0 A03-096	SEP 26 1990	10	OF 11

PNL TECHNICAL PROCEDURE

B = factor or factors to account for sample volumes, dilutions, weights, leach solution volumes and any other factors needed to produce the appropriate reporting units. These factors shall be documented as part of the project record.

These calculations include an approximate correction for the ingrowth of Y-90 from the Sr separation, time to the time the sample is counted. It assumes that the counting efficiencies of Sr-90 and Y-90 are approximately equal. Calculations are not corrected for Sr-89 activity.

5.0 Specific Qualification

NIST-traceable Sr isotopes are used to trace/establish yield for this process. Thus, this procedure is self-qualifying as defined in PNL-MA-70, PAP-70-901.

6.0 Records

Records will be maintained and controlled so as to conform to requirements of PNL-MA-70, PAP-70-1701. Laboratory Record Books (LRBs) and Analytical Data Sheets provide a mechanism for control of most records. LRBs will be used in accordance with the Act Now Directive 89.1.

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-465	0 A03-097	SEP 26 1990	11	OF 11

INTERIM CHANGE NOTICE  
(ICN)

~~CONTROLLED DOCUMENT~~

COPY NO. 000003

ICN- PNL-ALO-470.1

Page 1 of 1

A. Document Number: <u>PNL-ALO-470</u> Revision Number: <u>0</u> Document Title: <u>Procedure for Maintaining control of Germanium Spectrometers</u> Document's used for <u>Gamma-Ray Spectroscopy</u> Original Author: <u>E. A. Lepel</u>	Effective Date of ICN: <u>4/10/91</u> Change Requested By: <u>E. A. Lepel</u>
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B. Action:  
Please replace page 1 with the attached page.

C. Effect of Change:  
  
To allow coverage of the multidimensional (MDA) counting systems by this procedure. The MDA systems consist of NaI (TI) detectors.

D. Reason for Change/Description of Change  
Reason for change:  
To allow coverage of the multidimensional (MDA) counting systems by this procedure. The MDA systems consist of NaI (TI) detectors.  
Description of change:  
See attached

E. Approval Signatures (Please Sign and Date)	Type of Change: ( ) Minor Change <input checked="" type="checkbox"/> Major Change
Process Quality OSNR Department Concurrence: <u>CK Genta, DE</u> Date: <u>4/10/91</u>	
Approval Authority: <u>[Signature]</u> Date: <u>4/10/91</u>	
Other Approvals: <u>[Signature]</u> Date: <u>4/12/91</u>	
: <u>B.M. Bellis</u> Date: <u>4/15/91</u>	

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-470, PROCEDURE FOR MAINTAINING CONTROL OF GERMANIUM SPECTROMETERS USED FOR GAMMA-RAY SPECTROSCOPY

SCOPE

This procedure is to be used to assure the reliability and stability control of Germanium and Sodium Iodide Spectrometers used for gamma-ray spectroscopy. This will be assured by the periodic counting of "control check standards" in which specific peak areas are monitored. The reproducibility of the count data is a measure of the instrument stability and reliability. The periodic gamma counting of calibration control check standards, automated transfer of spectra from analyzers to data manipulation computers, the ability to retrieve and display control parameters as a function of time (possibly years), and archival storage of data for each counting system provide not only a measure of assurance of instrument stability but also accuracy and precision of each gamma spectrometer. This procedure was developed by PNL Scientists, and reflects over 10 years of experience in gamma-ray spectroscopy of a wide variety of samples.

APPLICABILITY

This procedure is implemented to assure quality and is approved and written to conform with PNL-MA-70.

This procedure is applicable to the Counting Room Manager and trained and qualified personnel within the Analytical Chemistry Laboratory and Nuclear Chemistry Department of PNL.

This procedure is primarily intended to assure that the gamma spectrometry equipment is maintained in control and in proper working condition.

DEFINITIONS/ACRONYMS

- dpm - disintegrations per minute (activity term)
- ng - nanograms ( $10^{-9}$  grams)
- ug - micrograms ( $10^{-6}$  grams)
- tl/2 - half-life

Author	Date	Project Mgr.	Date	QAD Representative	Date
EA Lepel		BM Gillespie		GK Gerke	
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
KH Abel		JM Latkovich		ALL SIGNATURES ON FILE WITH DOCUMENT CONTROL	
Procedure No.	Revision No.	Effective Date	Page	of	
PNL-ALO-470	0	SEP 26 1990	1	6	

## PNL TECHNICAL PROCEDURE

LRB - Laboratory Record Book

CTL - Control

Count - to acquire data via digitized pulses from radioactive sources in gamma spectrometer systems

Control Source - aliquot of mixed radioactive source contained in counting geometry

Diode - Detector - Part of the gamma detector system which includes the actual detector crystal in a metal cylindrical housing and its immediate electronics up to the preamplifier.

### RESPONSIBLE STAFF

Counting Room Manager  
Analysts

### PROCEDURE

#### 1.0 Equipment and Materials

Detectors - intrinsic germanium (IG) detector, lithium drifted germanium detector [Ge(Li)], low-energy photon detector (LEPD), multidimensional detector

Associated Electronics - preamplifier, amplifier, analog-to-digital converter (ADC), pulse pile-up rejector, multichannel analyzer, CAMAC interface, DEC PDP 11/44 computer, DEC  $\mu$ VAX, or PC

Mixed radioactive control source containing at least two of these radioisotopes:  $^{241}\text{Am}$ ,  $^{137}\text{Cs}$ , or  $^{60}\text{Co}$ .

#### 2.0 Gamma Spectrometry Control Procedure

This procedure defines the means for providing detector reliability and stability information to the system user. The primary method for monitoring gamma spectrometer system performance is the "detector reliability" chart. Each instrument must be checked routinely using radioactive sources to ensure that the stability, efficiency, and resolution of each detector is within a specified calibration range. The frequency of the calibration control check counts are based on the usage of the detector. When the detectors are in use, calibration control check counts are taken at least bi-monthly (two times a month) or as specified in a project-specific Statement of Work (SOW), Technical Project Plan (TPP) or Quality Assurance Project Plan (QAPjP). The proper standards and methods used for the calibration control check must be

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-470	0	SEP 26 1990	2	6

A03-100

## PNL TECHNICAL PROCEDURE

matched to the particular counting system being used. Graphing the control counting data (appropriate decay corrections must be made for the radioactive sources) readily determines that the detector system is operating properly and also provides a permanent record that aids in measuring instrument reliability.

2.1 The control check standard for each gamma-ray detection system is listed in Tables 1 and 2. The control check source standards are identified by LRB reference numbers. Each control check source standard is mounted either on a plastic holder which fits onto the detector or an aluminum card which is placed in a specific shelf of a card holder. Using the control check source and geometry specified for the detector, count the source for the time required to yield specific integrated gamma-ray peaks of <5% relative error ( $\leq 1\%$  relative error, preferably) from counting statistics, per Sections 5.2.1-5.2.4 of PNL-ALO-464. The count times are listed in Table 2.

2.2 The accumulated spectrum is then transferred to the computer, the peaks integrated, resolution checked, and the calibration listed on the user's computer terminal. This is accomplished by using the control program (See Section 3.0).

2.3 The control program (CTL) stores the spectral data by date and the detector identification onto magnetic media. The program will then print and plot all values necessary to determine if the detector system is within control values based on 2 and 3 sigma ranges.

### 3.0 The Control (CTL) Program

The Control (CTL) program is used to track the consistency of a diode. The CTL program is also used to transfer an accumulated spectrum to the computer from a specific multichannel analyzer and performs the required data reduction and interpretation. This is done by typing CTL "X" at the ">" prompt, where X is the identification of the detector. After pressing Enter (or Return on the keyboard terminal), the program will transfer the data to the computer, integrate specific peak areas, check the resolution, and list the energy calibration on the computer terminal. The program then will print and plot the "Detector Reliability" Chart.

#### 3.1 Computer Methodology

The CTL program assumes that for a particular diode its control source standard has been counted. CTL will transfer the spectrum into the computer and integrate the peaks according to the library ANTI.LIB or SING.LIB (depending on how the detector is configured

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-470	0	SEP 26 1990	3	6

A03-101

PNL TECHNICAL PROCEDURE

with respect to coincidence-noncoincidence). The results of the integration (in counts per minute) are compared against a saved average, with a report and plot being generated.

The average activity (in cpm) for an area of interest (as designated by the SUM library) is calculated as follows:

Until 10 control check spectra are recorded, the average is used to determine the "goodness" of the latest control check spectrum. Once 10 control check spectra have been completed and accepted by the analyst, they determine the average for the detector. The average stays fixed from then on to guard against creeping or abnormal changes that could go unnoticed with a running average. The latest 50 control check spectra are kept in memory, and subsequent control check spectra are consecutively written over the top of the oldest. The plot uses the average as the center line, with the dotted lines on either side representing 2 sigma from the average and the next solid lines representing 3 sigma from the average.

3.2 CTL "Detector Reliability" Chart

The "Detector Reliability" chart shows the activities and resolution of the measured control source photopeaks as calculated by the CTL program. The chart is reviewed by the Counting Room Manager or Cognizant Staff to see if the activities and resolution of the latest counts are within 3 sigma of the standard deviation of the mean computed using the first 10 "good" control check spectra obtained for the control check source/standard. If the values are acceptable, the chart is signed and stored beside the detector system in a plastic folder which is readily visible. If the observed values are out of the 3 sigma range, the user will check the control check source/standard and geometry and rerun the control check source (per Section 2) to see if positioning or just a statistical variation was responsible [for 99.7% confidence (3 sigma), three in a thousand should fall outside the limits]. If the observed values are still outside the 3 sigma range, a Deficiency Report (DR) shall be issued per PNL-MA-70, PAP-70-1502, and the user will consult an instrument technician and/or engineer to determine and repair the problem. The response shall be documented on the Control Chart and/or Instrument Log and on the DR. Another CTL count of the control check sources shall then be made per Section 2 to see if all observed data are within the 3 sigma control value. All samples counted on that detector between the last good CTL counted and the bad CTL counted shall be checked for validity by cross calibration to another detector and/or recounted on that detector when the control check standard spectra shows all

Procedure No.	Revision No.	Effective Date	Page	of
PNL-ALO-470	0	SEP 26 1990	4	6

PNL TECHNICAL PROCEDURE

activities again within the 3 sigma range. The Counting Room Manager shall maintain copies of the Detector Reliability Charts which cover the full cycle of 50 entries for each detector system.

3.3 Photopeak Resolution (FWHM) Calculation

The FWHM (full-width, half-maximum) is a measure of the peak sharpness. In gamma spectrometry, clearly resolved peaks are Gaussian in shape, provided the electronic components are adjusted correctly. The simplest method used to calculate the FWHM of a peak is by a graphical method; however, numerical methods are required in order to do this by computer. An illustration of the FWHM is shown in Figure 1.

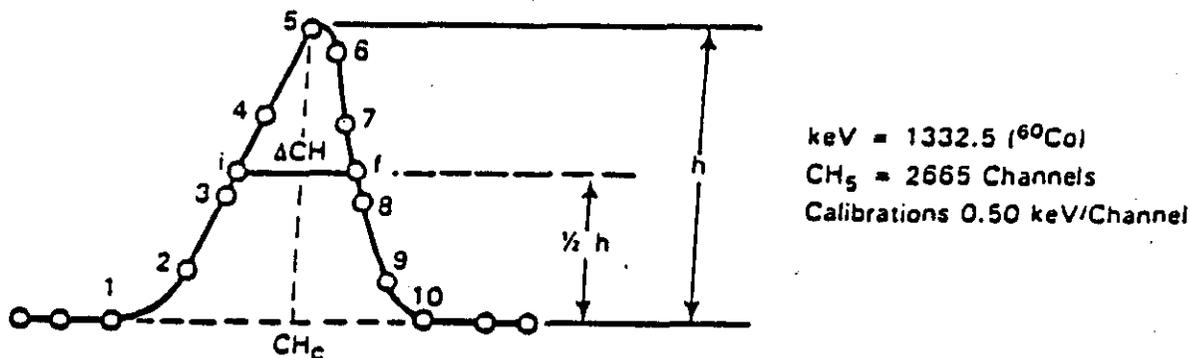


FIGURE 1. An Illustration of the FWHM Valve

The resolution (R) is calculated as follows:

$$R = \frac{\Delta CH(\gamma \text{ peak KeV})}{CH_C}$$

Where:

$\Delta CH$  = channel width at half max (1/2 h) in channels

$CH_C$  = channel number at centroid of peak

Procedure No. PNL-ALO-470	Revision No. 0	Effective Date SEP 26 1990	Page 5	of 6
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A03-103

9 2 1 2 9 1 1 5

PNL TECHNICAL PROCEDURE

and  $\Delta CH$  is calculated by determining  $i$  and  $f$  fractional channel numbers by calculating the slope between channels in which the half max count lies between.

This resolution is checked as described in Section 3.2.

4.0 Records

Records will be maintained and controlled so as to conform to requirements of PAP-70-1701. Laboratory Record Books (LRBs) and Data Sheets provide a mechanism for control of most records. Laboratory Record Books will be used in accordance with the Act Now Directive 89.1.

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Procedure No. PNL-ALO-470	Revision No. 0	Effective Date SEP 26 1990	Page 6	of 6
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9 2 1 3 5 9 3 1 3

Nuclear Constants Gamma- $\lambda$ , Energies, and Activities for  
Control Sources Used with Germanium Spectrometers

TABLE 1. Control Sources for Gamma Spectrometry

PNL Control	Detector(s)	Radioisotopes	Origin (cross ref.)	STD. No.	Ref. date	dpm/ml or gm <sup>t</sup>	dpm/std	
STD. No.	Detector(s)	( $\gamma$ -peak energy)	$t_{1/2}$	NBS SRM No.	PNL No.	Ref. date	dpm/ml or gm <sup>t</sup>	dpm/std
50690-27-1	L	<sup>241</sup> Am (59.5)	432 y	Amersham (AM 2.44)	4491-101-0	1/2/84	6.20 x 10 <sup>5</sup>	3.72 x 10 <sup>5</sup>
50690-27-2	BNW	<sup>137</sup> Cs (661.8)	30.17 y	(SRM No. 4216-0)	1892-136-0	1/2/84	2.09 x 10 <sup>7</sup>	5.23 x 10 <sup>5</sup>
50690-27-3	E	Mixed						
50690-27-4	J	Std <sup>60</sup> Co (1173.2)	5.27 y	(SRM No. 4216-0)	3368-54-10	1/2/84	7.48 x 10 <sup>6</sup>	1.50 x 10 <sup>6</sup>
50690-27-5	K	(1332.5)						
50690-27-6	I							
50690-117-6	N							
50690-117-7	H							
50690-117-8	R							
50690-117-1	EE	<sup>241</sup> Am (59.5)	432 y	Amersham (AM 2.44)	4491-101-0	1/2/84	7.75 x 10 <sup>4</sup>	4.65 x 10 <sup>4</sup>
50690-117-2	II							
50690-117-3	JJ	Mixed						
		Std <sup>60</sup> Co (1173.2)	5.27 y	(SRM No. 4216-0)	3368-54-10	1/2/84	7.48 x 10 <sup>6</sup>	1.50 x 10 <sup>6</sup>
		(1332.5)						
54491-58-1	389	NAT. <sup>238</sup> U (63.2)	4.47 x 10 <sup>9</sup> y				3.313 x 10 <sup>3</sup>	5.014 x 10 <sup>4</sup>
		U <sup>230</sup> Th (67.4)		O.I.E.A.-7	4491-58-1		3.313 x 10 <sup>3</sup>	5.014 x 10 <sup>4</sup>
		STD. <sup>226</sup> Ra (186.2)					3.313 x 10 <sup>3</sup>	5.014 x 10 <sup>4</sup>
		<sup>210</sup> Pb (46.5)					3.313 x 10 <sup>3</sup>	5.014 x 10 <sup>4</sup>
3368-87-10	447	NAT. <sup>238</sup> U (63.2)	4.47 x 10 <sup>9</sup> y				3.313 x 10 <sup>3</sup>	5.014 x 10 <sup>4</sup>
		U <sup>230</sup> Th (67.4)		O.I.E.A.-7	3368-87-10		3.313 x 10 <sup>3</sup>	5.014 x 10 <sup>4</sup>
		STD. <sup>226</sup> Ra (186.2)					3.313 x 10 <sup>3</sup>	5.014 x 10 <sup>4</sup>
		<sup>210</sup> Pb (46.5)					3.313 x 10 <sup>3</sup>	5.014 x 10 <sup>4</sup>
<u>M-D SYSTS.</u>								
6408-27-13P	P-4	<sup>137</sup> Cs (661.8)	30.17 y	(SRM No. 4216-0)				
6408-27-10P	P-5				N-067-500	1/11/77	9.982 x 10 <sup>4</sup>	*4.991 x 10 <sup>4</sup>
6408-27-12P	P-8	<sup>60</sup> Co (1173.2)	5.27 y	(SRM No. 4216-0)	N-066-250	8/7/75	1.028 x 10 <sup>5</sup>	*2.056 x 10 <sup>4</sup>
6408-101-14P	*P-9	(1332.5)						

\*The multi-dimensional system (M-D SYSTS) - P-9 uses a lower activity dpm/std.  
<sup>t</sup>Natural U standard is in dpm/gn, <sup>241</sup>Am-<sup>137</sup>Cs-<sup>60</sup>Co standard is in dpm/ml.

A03-105

TABLE 2: Control Source Parameters for Use With Germanium Spectrometers

DETECTOR IDENTIFICATION	CONTROL SOURCE	LENGTH OF TIME	POSITION-	CTL PROGRAM "SUM" LIBRARY USED
BNW	50690-27-2	600s	w/holder	Sing. Lib
E	50690-27-3	600s	3" setting	Anti. Lib
* EE	50690-117-1	6000s	3" setting	Anti. Lib
G	50690-27-3	600s	w/holder	Sing. Lib
H	50690-117-7	600s	w/holder	Sing. Lib
I	50690-27-6	600s	w/holder ctl setting	Anti. Lib
* II	50690-117-2	6000s	w/holder ctl setting	Anti. Lib
J	50690-27-4	600s	w/holder phosphors closed	Anti. Lib
* JJ	50690-117-3	600s	w/holder phosphors closed	Anti. Lib
K	50690-27-5	600s	w/holder	Sing. Lib
L	50690-27-1	600s	w/holder	Sing. Lib
N	50690-117-6	600s	shelf #5	Sing Lib.
R	50690-117-8	400s	shelf #4	Sing Lib.
389	4491-58-1(pellet)	6000s	w/holder	389. Lib
447	3368-87-10(pellet)	6000s	w/holder	447. Lib

\* Actually the detector is identified by the single numeric character. The two digit character is used when transferring the spectrum to the computer.