

Analytical Chemistry Laboratory Department

ANALYTICAL CHEMISTRY
LABORATORY (ACL)
PROCEDURE COMPENDIUM

Volume 1: Administrative

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A. G. King
Department Manager

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A. Document Number: <u>PNL-ALO-010.3</u> Revision Number: <u>2</u>		Effective Date of ICN: <u>5/26/92</u>
Document Title: <u>ANALYTICAL LABORATORY OPERATIONS (ALO) SAMPLE RECEIPT AND CONTROL</u>		Change Requested by: <u>DR SAUER</u>
Document's Original Author: <u>TM LONGAKER</u>		
B. Action: Replace exhibit 2 with new exhibit 2.		
C. Effect of Change: Allows for change of COC form depending upon current need, or for effeciency.		
D. Reason for Change/Description of Change: To allow for changes based upon need.		
E. Approval Signatures (Please sign and Date)		Type of Change (Check (✓) one) (X) Minor Change () Major Change
QS&R Department Concurrence: <u>TL Ehlert</u> <u>T. L. Ehlert</u>		Date: <u>5/26/92</u>
Approval Authority: <u>AG King</u> <u>A. A. King</u>		Date: <u>5/26/92</u>
Other Approvals: <u>BM Gillespie</u> <u>B. M. Gillespie</u>		Date: <u>5-26-92</u>
: _____		Date: _____

INTERIM CHANGE NOTICE
(ICN)

ICN-PNL-ALO-010.2-R2
Page 1 of 2

A. Document Number: <u>PNL-ALO-010</u> Revision Number: <u>2</u> Document Title: <u>Analytical Laboratory Operations (ALO) Sample</u> <u>Receipt and Control</u> Document's Original Author: <u>T. M. Longaker</u>		Effective Date of ICN: <u>9/25/91</u>
		Change Requested By: <u>B. M. Gillespie</u>
B. Action 3+ Replace page 4 with the attached page.		
C. Effect of Change Clarify that the Single Shell Tank (SST) project has a specific computerized sample tracking system and other projects may have a similar or comparable system.		
D. Reason for Change/Description of Change Reason for Change: Clarify that the SST sample tracking computerized method may be different from other project computerized tracking systems. Description of Change: 4.1 - See attached.		
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: <u>G.K. Gerke</u> <u>[Signature]</u> Date: <u>10/14/91</u> Approval Authority: <u>A.G. King</u> <u>[Signature]</u> Date: <u>10/11/91</u> Other Approvals: <u>B.M. Gillespie</u> <u>[Signature]</u> Date: <u>10/10/91</u> : _____ Date: ____/____/____		

4.1 Sample Tracking System

Sample tracking systems are project specific. Each system may track Chain of Custody documentation, Test Instructions, sample numbers/sub-sample numbers, requested analyses, and sample analysis status, depending on client Statement of Work requirements. For example, the Analytical Laboratory Operations Project Support Office maintains a computerized sample tracking system supported by IBM-PC DBase III Plus which tracks Single Shell Tank project activities.

INTERIM CHANGE NOTICE
(ICN)

ICN- PNL-ALO-010.1
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A. Document Number: <u>PNL-ALO-010</u> Revision Number: <u>2</u> Document Title: <u>Analytical Laboratory Operations (ALO) Sample Receipt</u> Document's and Control Original Author: <u>Tanya Longaker</u>		Effective Date of ICN: <u>4/26/91</u> Change Requested By: <u>B. M. Gillespie</u>
B. Action: Replace page 4 with the attached page.		
C. Effect of Change: Clarify the need to compare COC sample numbers and ALO samples with the sample vial number.		
D. Reason for Change/Description of Change Reason for change: Clarify the need to compare COC sample numbers with the sample vial number. Description of change: 3.3 - See attached 3.4 - See attached		
E. Approval Signatures (Please Sign and Date)	Type of Change: {Check (<input checked="" type="checkbox"/>) one} (<input checked="" type="checkbox"/>) Minor Change (<input type="checkbox"/>) Major Change	
Process Quality OS&R-Department Concurrence: <u>St Gerber, DE</u> Date: <u>4/3/91</u> Approval <input checked="" type="checkbox"/> Authority: <u>[Signature]</u> Date: <u>4/4/91</u> Other Approvals: <u>B.M. Gillespie</u> Date: <u>4/3/91</u> : _____ Date: <u>1 / 1</u>		

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-010, ANALYTICAL LABORATORY OPERATIONS (ALO) SAMPLE RECEIPT AND CONTROL

APPLICABILITY

This procedure describes the approach used for receipt and control of samples associated with Tri-Party Agreement related work coming into the greater 325 Laboratory Complex including 325-A and 325-B Hot Cells.

DEFINITIONS

325 Laboratory Complex - All analytical labs within the 325, 329, 3720, 314 and 3708 buildings

Chemical Analysis Group - Organic, Inorganic, Radiochemical, or special analytical chemistry groups

RESPONSIBLE STAFF

- Project Manager
- Task Leader
- Technical Staff
- ALO Project Support Clerk

PROCEDURE

This procedure describes receipt and control of samples coming into the 325-A Hot Cell, 325-B Hot Cell, or into any of the chemical analysis groups within the greater 325 Laboratory Complex. Guidance is provided below to cover these alternatives. See Figure 1 (attached).

Additional documentation and sample storage requirements may be identified in the project SOW, QAPP, and/or in Test Instructions.

Author	Date	Project Mgr.	Date	QAD Representative	Date
Tanya Hongaker	9/10/90	28 Jones	9/10/90	CK Gerber	9/11/90
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
28 Jones	9/10/90	H. S. [Signature]	9-12-90		
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PNL TECHNICAL PROCEDURE

1.0 325-A Hot Cell

1.1 Sample Receipt

Samples are received into the 325-A Hot Cells accompanied by client chain-of-custody (COC) documentation. The PNL 325-A Hot Cell Task Leader (or his designee) receives samples, examines shipping containers and documentation, and notifies the ALO Project Support Office that samples have arrived. Copies of all client COC documentation are provided to the ALO Project Support Office. The Task Leader (or his designee) is responsible for documenting sample receipt into the appropriate 325-A Hot Cell records. (Any deviation from protocol is reported to the appropriate ALO project manager for resolution).

1.2 Sample Identification

The 325-A Hot Cell Task Leader (or his designee) prepares Test Instructions (TIs) which assign 325-A Hot Cell sample numbers. The 325-A Hot Cell TIs are prepared using guidance obtained from Technical Program Plans (TPP), Letters of Instruction (LOI), Analytical Request Forms (ARF) or TIs issued by respective clients. 325-A Hot Cell sample numbers are developed according to client requirements.

1.3 Transfer of Samples from 325-A to 325-B Hot Cells or Other Facilities

The Hot Cell Chain-of-Custody Form (Exhibit 1) is used to document transfer of samples from the 325-A Hot Cells to the 325-B Hot Cells or to other support facilities. This form provides the appropriate ALO sample number(s) and a brief description of the sample(s). This form is issued by the ALO Project Support Office. The signed, original forms are maintained in the project records in the ALO Project Support Office.

2.0 325-B Hot Cell

2.1 Sample Receipt

Samples received into the 325-B Hot Cell are accompanied by client or internal laboratory COC. The PNL 325-B Hot Cell Task Leader (or his designee) receives samples, examines shipping containers and documentation, and if received directly from an outside client, notifies the ALO Project Support Office that samples have arrived within the time requirements specified in the project SOW. Copies

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PNL TECHNICAL PROCEDURE

of all client COC documentation are provided to the ALO Project Support Office. The Task Leader (or his designee) is responsible for documenting sample receipt into the appropriate 325-B Hot Cell records.

2.2 Sample Identification

All samples received into the 325-B Hot Cell will have associated with them, or will be assigned, a unique ALO sample number. (A sample may have been assigned an ALO sample number by laboratory staff prior to transfer.) ALO sample numbers are assigned according to PNL-MA-597 7-30.11. (Only sections within 7-30.11 which apply to the assignment of unique sample numbers [section 4.0 #1.0] are utilized.)

Samples are prepared for analysis in the 325-B Hot Cell facilities. In addition, some analyses are performed in the B-Hot cell facilities. Instructions for sample preparation and/or analysis are provided to the 325-B Hot Cell Task Leader by TIs or other written guidance. Sub-samples generated during analytical sample preparation are numbered by the addition of trailer alpha-numeric units to the ALO sample number, as per TI or other written guidance from the client or the ALO Project Support Office.

2.3 Transfer of Samples or Sub-Samples to Analytical Laboratories

A COC record (Exhibit 2) is associated with each transfer of sample/sub-sample to the laboratories for analysis. Chain-of-Custody records are prepared in the ALO Project Support Office. Signed, original COC records are maintained in the ALO Project Support Office.

3.0 Analytical Laboratories

3.1 Sample Receipt

Samples transferred from the 325-A or 325-B Hot cell facilities will be accompanied by COC documentation. Some samples are received directly by the chemical analysis group rather than into one of the hot cell facilities. Samples received directly by the chemical analysis groups are handled according to PNL-ALO-051 and are accompanied by client COC documentation. Copies of all client COC documents are provided to the ALO Project Support Office for inclusion into project records.

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PNL TECHNICAL PROCEDURE

3.2 Sample Identification

Samples received by the 325 chemical analysis groups will have associated with them a unique ALO sample number. If a sample is received without an ALO sample number then it is the responsibility of the laboratory staff to obtain the number. ALO sample numbers are assigned according to PNL-MA-597 7-30.11. (Only sections within 7-30.11 which apply to the assignment of unique sample numbers are utilized.)

3.3 Transfer of Samples Between Chemical Analysis Groups

All samples transferred between chemical analysis groups will be accompanied by COC documentation (Exhibit 2) prepared by the ALO Project Support Office as per TIs or other written guidance. The receiver will verify that the COC sample numbers correspond to the sample vial number. Signed original COC documents are maintained in the ALO Project Support Office for inclusion in project records.

3.4 Transfer of Samples Between Buildings

All samples transferred between buildings within the 325 Laboratory Complex will be accompanied by COC documentation (Exhibit 2) prepared by the ALO Project Support Office as per TIs or other written guidance. The receiver will verify that the COC sample numbers correspond to the sample vial number. Signed original COC documents are maintained in the ALO Project Support Office for inclusion in project records.

3.5 Transfer of Samples within a Chemical Analysis Group

COC forms are not required for sample transfers between staff within a Chemical Analysis Group within the same building. The 325 Laboratory is in a secure area and access is controlled. (In addition, unescorted access to the Laboratory is limited by Radiation Work Procedures (RWPs) etc.)

4.0 Sample Tracking/Data Management

4.1 Sample Tracking System

Sample tracking systems are project specific. Each system may track Chain of Custody (COC) documentation, Test instructions, sample numbers/sub-sample numbers, requested analysis, and sample analysis status, depending on client Statement of Work requirements. For example, the Analytical Laboratory Operations Project Support Office maintains a computerized sample tracking system supported by IBM-PC DBase III Plus which tracks Single Shell Tank project activities.

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PNL TECHNICAL PROCEDURE

4.2 Primary Data Record System

All primary analytical data records must be maintained for analyses supporting work covered under Tri-Party Agreement requirements. These requirements include transfer of primary analytical records to the client. The ALO Project Support Office provides administrative support to laboratory staff in meeting these requirements. The ALO Data Management System is described in PNL-ALO-011.

5.0 Sample Return/Disposal

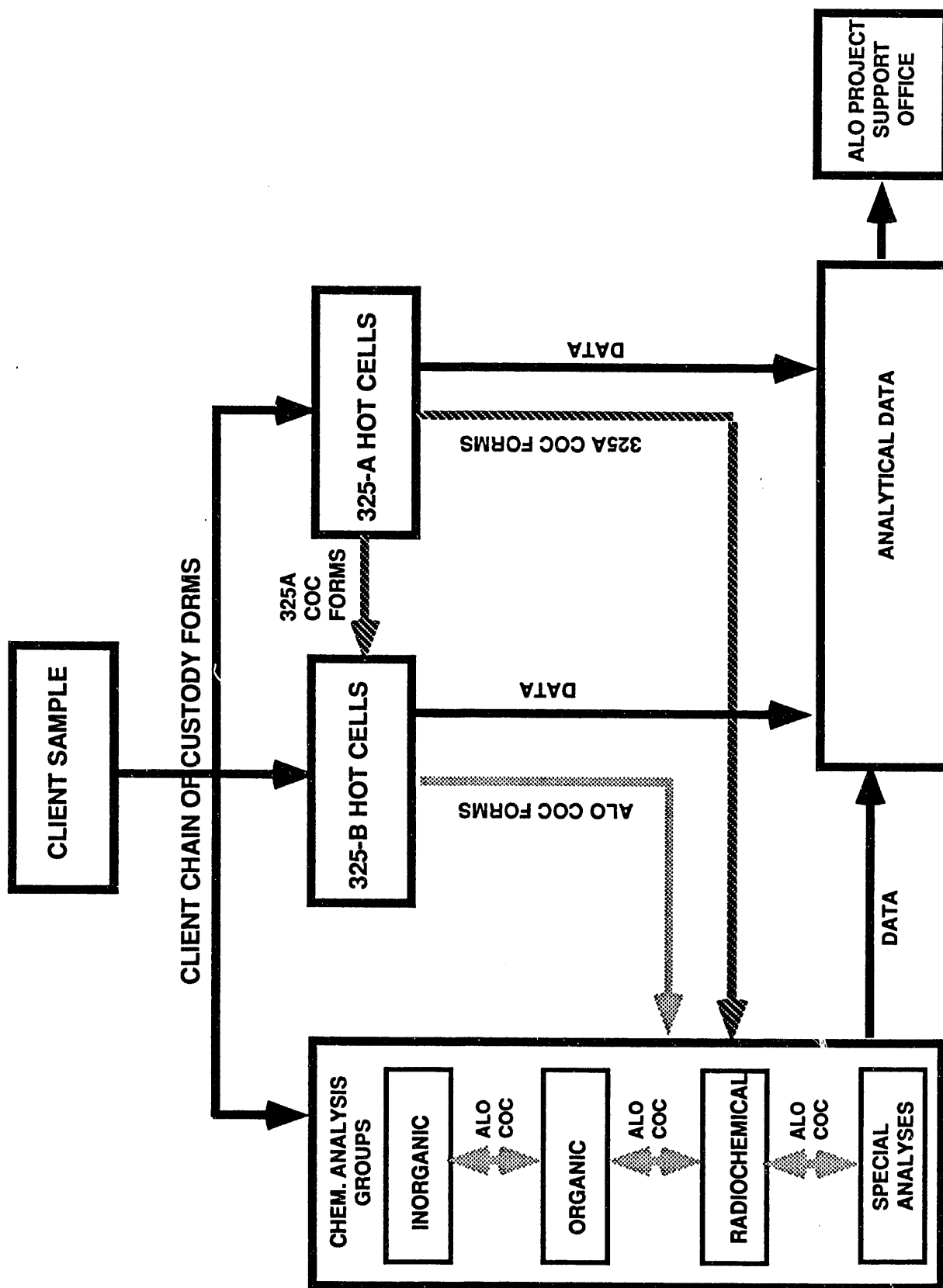
Samples are returned or disposed of as per requirements identified in the project SOW, TPP, TI, or ARFs.

6.0 ALO Test Instructions

6.1 Test Instructions are prepared by ALO Project Management Staff when the complexity of the sample preparation and/or analysis preclude the use of Analytical Request Forms (ARFs). An example of a TI is included as Exhibit 3. Test Instructions shall reference the SOW or test plan, requested analyses, sample numbering system, and shall identify appropriate procedures. Test Instructions are approved by the ALO Project Manager.

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FIGURE 1: SAMPLE CHAIN OF CUSTODY & DATA FLOW PATHS



Chain of Custody Number _____

325-A HOT CELL CHAIN-OF-CUSTODY

DATE OF TRANSFER _____

SENDER _____

RECEIVER _____

WHC SAMPLE IDENTIFICATION NUMBER _____

ALO Sample Number

325-A Hot Cell Sample Identification

90-

Original - ALO Project Support Office
Copy - 325-A Hot Cell
Copy - 325-B Hot Cell

Page 1 of 1

Chain of Custody Number _____

ALO CHAIN OF CUSTODY

[illegible]

ANALYSIS REQUESTED OR DEPARTMENT _____

[illegible]

Original - Project Management
Copy - Sender
Copy - Receiver

PLEASE NOTE: USE OF THIS SPECIFIC FORM IS NOT REQUIRED, BUT THE INFORMATION REQUESTED MUST APPEAR ON ANY FORM USED AS AN ALTERNATIVE.

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 COMPOSITE ANALYSES**

DATE PREPARED : May 30, 1990 PREPARED BY : Tom Jones

SAMPLE NUMBERS : 90-4180

APPROVED BY : TE Jones Date May 30, 1990
Project Manager

CONTROLLING DOCUMENTS :

TEST PLAN: WHC-EP-0210 Project SOW: 16021

Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on Sample 90-4180. This sample comes from the **Core 16 Composite**.

This TI is based on the test plan identified above. Any deviation on the instructions contained in this TI will require that the Project Manager be notified of these changes. The analyses will be done at a QA Level 2.

All analyses are to be completed following the identified procedures. Any deviations for the procedure must be documented and this documentation must accompany the analytical data. All analytical data are returned to the Project Management Office.

REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
ICP Metals (Acid & Water Leach)	PNL-SP-7	Urie
Hg by CVAA	CLP	Urie
pH (Water Leach)	WHC-053-1	Steele

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 COMPOSITE ANALYSES**

TI-SST-105

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Exhibit 3

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<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
IC Anions (Water Leach)	7-40.8	Urie
TOC/TIC/TC (Water Leach)	7-40.7	Urie
Cr(VI) (Water Leach)		Urie
Ammonia (Water Leach)		Urie
Cyanide		Urie
Se & As by AA (Acid Leach)		Urie
Total Alpha (Water Leach)	HTA-4-22, HTA-4-6	Kaye
Total Beta (Water Leach)	HTA-4-23, HTA-4-8	Kaye
GEA (Water Leach)	HTA-4-5	Kaye
Uranium (Water Leach)	HTA-4-16	Kaye
Tc-99 (Water Leach)	HTA-4-12	Kaye
I-129 (Water Leach)	7.40.17, 7-40.26	Kaye
Ni-59/63 (Water Leach)	PNL-SP-38	Kaye
Sr-90 (Water Leach)	HTA-4-8, HTA-11	Kaye
C-14 (Water Leach)	HTA-4-17	Kaye
Tritium (Water Leach)	PNL-SP-30, PNL-SP-33	Kaye
Pu, U Mass Spec (Water Leach)	2.30.6, HTA-4-40	Kaye
Pu, Am, Np, Alpha (Water Leach)	HTA-4-15, HTA-4-5, HTA-4-6	Kaye

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 COMPOSITE ANALYSES**

Semi-Volatile Organics	PNL-ALO-345	Stromatt
TOX/EOX	PNL-ALO-320	Stromatt
Wt.% Solids	PNL-ALO-504	Steele

B-HOT CELL SAMPLE PREPARATION INSTRUCTIONS

Approximately 75 grams of sample were transferred. The following sample preparation steps are required:

<u>Method</u>	<u>Procedure</u>
- Acid Leach for ICP QC Requirements : Duplicate, Spike, Spike Control, and Blank	PNL-ALO-101
- Acid Leach for AA QC Requirements : Duplicate, Spike, Spike Control, and Blank	PNL-ALO-101
- Water Leach QC Requirements - Duplicate, Spike, Spike Control, and Blank	PNL-ALO-103
- Hg Prep. QC Requirements - Duplicate, Spike, Spike Control, and Blank	CLP
- CN Prep in Duplicate QC Requirements - Duplicate, Spike, Spike Control, and Blank	CLP

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 COMPOSITE ANALYSES**

TI-SST-105

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Exhibit 3

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If radiation levels permit the following aliquots of Sample 90-4180 should be dispensed:

- Semi-Volatile Organics - four 1-gram samples in 40 ml vials
- TOX/EOX - Four 1-grams samples in 20 ml vials

Otherwise, the organic analysis prep will have to be completed in the Hot Cell.

SAMPLE IDENTIFICATION SYSTEM

The following sample identification system will be followed. All samples will be identified as 90-4180-X-Number-Analysis. The "X" letter will identify a sample preparation method. The "Number" identify replicate analyses using the sample sample preparation method (i.e., duplicates, spikes, blanks, etc.). The Sample Preparation Method code is:

- | | |
|---|-----------------------|
| A | ICP Acid Leach |
| B | AA Acid Leach |
| C | Water Leach |
| D | Mercury Analysis Prep |
| E | SemiVolatile Organics |
| F | TOX/EOX |
| G | Cyanide Sample Prep |
| H | Wt% Solids |

The ICP Acid Leach Numbering System will be:

- | | |
|-------------|--------------------------|
| 90-4180-A-1 | ICP Acid Leach |
| 90-4180-A-2 | Duplicate ICP Acid Leach |
| 90-4180-A-3 | Spiked ICP Acid Leach |
| 90-4180-A-4 | ICP Spike Control Sample |
| 90-4180-A-5 | Methods Blank |

SST SAMPLE TEST INSTRUCTION FOR CORE 16 COMPOSITE ANALYSES

The AA Acid Leach Numbering System will be:

90-4180-B-1	AA Acid Leach of Sample 90-4180
90-4180-B-2	Duplicate AA Acid Leach of 90-4180
90-4180-B-3	Spiked Sample AA Acid Leach
90-4180-B-4	AA Spike Control Sample
90-4180-B-5	Methods Blank

The Water Leach Sample Preparation aliquots numbering system will be:

90-4180-C-1	Water Leach of Sample 90-4180
90-4180-C-2	Duplicate Water Leach for 90-4180
90-4180-C-3	Spiked Sample Water Leach (IC only)
90-4180-C-4	Spike Control Sample (IC only)
90-4180-C-5	Methods Blank for Water Leach (IC, GEA, Total Beta, Total Alpha, and othe Rad Chem Analyses)

The following aliquots will be despenced from the Water Leach sample:

<u>Analyte</u>	<u>Volume</u>
pH	10 ml
ICP	25 ml
IC, TOC, TIC, TC	25 ml
Cr(VI)	25 ml
NH ₃	25 ml
Radiochemistry	50 ml
Complexant (to WHC)	50 ml

The Mercury Preparation aliquots numbering system will be:

90-4180-D-1	Mercury Sample Prep. for 90-4180
90-4180-D-2	Duplicate mercury Prep.
90-4180-D-3	Mercury Spike Sample
90-4180-D-4	Mercury Standard
90-4180-D-5	Methods Blank

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 COMPOSITE ANALYSES**

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Exhibit 3
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The SemiVolatile Organics numbering system will be:

90-4180-E-1	Semivolatile analysis of 90-4180
90-4180-E-2	Duplicate semivolatile analysis of 90-4180
90-4180-E-3	Matrix Spike
90-4180-E-4	Matrix Spike Duplicate

The TOX/EOX numbering system will be:

90-4180-F-1	TOX/EOX Analysis for 90-4180
90-4180-F-2	Duplicate TOX/EOX Analysis
90-4180-F-3	Spike sample TOX/EOX
90-4180-F-4	Methods Blank

The Cyanide numbering system will be:

90-4180-G-1	Cyanide Analysis for 90-4180
90-4180-G-2	Duplicate Cyanide Analysis
90-4180-G-3	Spike sample
90-4180-G-4	Cyanide Standard
90-4180-G-5	Methods Blank

INTERIM CHANGE NOTICE
ICN

ICN- PNL-ALO-012.1 RO
PAGE 1 OF 1

<p>A. Document Number: <u>PNL-ALO-012.1 RO</u> Revision Number: <u>0</u> Document Title: <u>ALO DATA REPORT/PACKAGE PREPARATION, REVIEW AND CONTROL</u> Document's Original Author: <u>TE JONES</u></p>		<p>Effective Date of ICN: <u>03/30/92</u></p> <p>Change Requested by: <u>TE JONES</u></p>										
<p>B. Action:</p> <p>Replace pages 1-2 with new pages 1-3.</p>												
<p>C. Effect of Change:</p> <p>ADDITION OF SECTION 4.0 (Transfer of Data Documents to Westinghouse Hanford Company Office of Sample Management (WHC-OSM))</p>												
<p>D. Reason for Change/Description of Change:</p> <p>To obtain receipt acknowledging receipt of data package/report</p>												
<table style="width: 100%; border: none;"><tr><td style="width: 50%; vertical-align: top;">E. Approval Signatures (Please sign and Date)</td><td style="width: 50%; vertical-align: top;">289 Type of Change (Check (✓) one) <u>3/31/92</u> (*) Minor Change (X) Major Change</td></tr><tr><td colspan="2" style="padding-top: 10px;">QS&R Department Concurrence: <u>TL Ehlert</u> <i>TL Ehlert</i> Date: <u>3/31/92</u></td></tr><tr><td colspan="2" style="padding-top: 10px;">Approval Authority: <u>AG King</u> <i>AG King</i> Date: <u>3/31/92</u></td></tr><tr><td colspan="2" style="padding-top: 10px;">Other Approvals: <u>BM Gillespie</u> <i>BM Gillespie</i> Date: <u>3-31-92</u></td></tr><tr><td colspan="2" style="padding-top: 10px;">: <u>TE Jones</u> <i>TE Jones</i> Date: <u>3-31-92</u></td></tr></table>			E. Approval Signatures (Please sign and Date)	289 Type of Change (Check (✓) one) <u>3/31/92</u> (*) Minor Change (X) Major Change	QS&R Department Concurrence: <u>TL Ehlert</u> <i>TL Ehlert</i> Date: <u>3/31/92</u>		Approval Authority: <u>AG King</u> <i>AG King</i> Date: <u>3/31/92</u>		Other Approvals: <u>BM Gillespie</u> <i>BM Gillespie</i> Date: <u>3-31-92</u>		: <u>TE Jones</u> <i>TE Jones</i> Date: <u>3-31-92</u>	
E. Approval Signatures (Please sign and Date)	289 Type of Change (Check (✓) one) <u>3/31/92</u> (*) Minor Change (X) Major Change											
QS&R Department Concurrence: <u>TL Ehlert</u> <i>TL Ehlert</i> Date: <u>3/31/92</u>												
Approval Authority: <u>AG King</u> <i>AG King</i> Date: <u>3/31/92</u>												
Other Approvals: <u>BM Gillespie</u> <i>BM Gillespie</i> Date: <u>3-31-92</u>												
: <u>TE Jones</u> <i>TE Jones</i> Date: <u>3-31-92</u>												

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-012, ALO DATA REPORT/PACKAGE PREPARATION, REVIEW, AND CONTROL

APPLICABILITY

This procedure describes the preparation, review, and control of data reporting documents generated by the PNL Analytical Laboratory Operations (ALO) Project Support Office (PSO). Such documents are prepared in support of projects managed under the ALO.

DEFINITIONS

- Data Report - Data Reports are compilations of analytical data, primarily in tabular format. Sample-specific qualify control data are generally included in the Data Reports. Use of Data Reports allow a rapid transmission of resolved analytical data.
- Data Package - Data Packages are complete compilations of resolved data (Data Report) and primary (i.e., raw) analytical data that support information included in the Data Report.
- Letter Report - Letter Reports are a mechanism for addressing specific topics with a limited data set.

RESPONSIBLE STAFF

ALO Project Manager
ALO Project Support Office Staff
Project Task Leaders

PROCEDURE

1.0 Document Preparation

- 1.1 Data Reports: The format for Data Reports is project-specific. However, the Data Reports are structured according to project tasks.

All resolved analytical data are provided to ALO-PSO by the project Task Leaders. Such data have been reviewed as per PNL-MA-70, PAP-70-1101. Data are reviewed for completeness by the ALO-PSO,

Author	Date	Project Mgr.	Date	QAD Representative	Date
TE Jones	08/17/90	TE Jones	08/17/90	GK Gerke	08/27/90
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
BM Gillespie	08/17/90	P Salter	08/20/90	Original signatures on file	
Procedure No.	Revision No.	Effective Date	Page		
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PNL TECHNICAL PROCEDURE

however, no technical changes are made. In some cases data are reformatted for inclusion in the data report.

1.2 Data Packages: Data Packages are a complete compilation of resolved analytical and supporting primary data. The ALO-PSO collects and compiles primary analytical data that were used to develop the resolved data. The format for Data Packages are project-specific. Data Packages are reviewed by the ALO Project Management staff for completeness.

1.3 Letter Reports: Letter reports are prepared as per client requirements. No specific outline is required.

2.0 Document Review

2.1 Data Reports: A Task Leader review is required to assure completeness and accuracy of analytical data prior to distribution to the client. Draft copies of Data Reports are given to each Task Leader who contributed analytical data. A Data Report Technical Review form (see Exhibit 1) accompanies the draft Report. Task Leaders are requested to review and comment on the document as to completeness and accuracy of the data, per requirements identified in the SOW and/or ARF. Comments are resolved by the Project Manager or his/her designee. Signed Technical Review forms are maintained in the project files.

2.2 Data Packages: Data Packages are reviewed by the ALO-PSO for completeness per requirements in the client SOWs or ARFs.

2.3 Letter Reports are handled like Data Reports.

3.0 Document Control

Data Reports, Data Packages, and Letter Reports are transmitted to the client as per project SOW requirements. All Data Reports are dated and include a reference to the SOW/TPP# and a current revision number. Distribution of Data Reports is restricted. Copies are provided to the client point-of-contact only as designated in the SOW. The ALO-PSO does not provide information to any other PNL or WHC group without written direction from the client.

The ALO-PSO maintains all primary analytical records supporting data reports. These records are maintained and controlled in accordance with project SOW and PNL-MA-70 requirements.

4.0 Transfer of Data Documents to Westinghouse Hanford Company Office of Sample Management (WHC-OSM)

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PNL TECHNICAL PROCEDURE

The client (WHC-OSM) shall acknowledge receipt of data reports and/or data packages. This acknowledgement is accomplished by having the Manager, WHC-OSM, or his/her designee, sign the Data Report/Package Acknowledgement form which accompanies the report/package and returning the form to the PNL Project Support Office. The signed form shall become a part of the project records.

The PNL project manager shall be notified in writing by WHC-OSM at the point the data package has been validated. This notification becomes part of the project records.

The Data Report/Package Acknowledgement form shall include the following information, at a minimum:

- Project Title (SST, 200BP-1, etc)
- Date Report/Package issued
- Signature of PNL Project Manager
- Data Report/Package Title
- Signature line to be signed by the WHC-OSM individual receiving the Report/Package

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PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-015, SINGLE SHELL TANK (SST) PROJECT SAMPLE TRACKING SYSTEM

APPLICABILITY

This procedure describes the D-Base III computer program that is used to track SST sample information.

DEFINITIONS

None

RESPONSIBLE STAFF

Project Manager
Project Administrative Staff

PROCEDURE

1.0 Scope and Application

The DBase program is used to track administrative data associated with sample receipt and analysis. Information is coded into the data base by SST administrative staff in the Analytical Laboratory Operations Project Support Office (ALO-PSO). Reports are generated, as required, for the project manager.

2.0 Data Input

Input data are taken from the Westinghouse (WHC) chain of custody (COC), internal PNL COCs, and sample status information received from SST Task Leaders. Input data are entered into the appropriate data base (See Attachment 1) by SST Project administrative staff. The original documents are retained in the ALO-PSO for inclusion into data packages.

The data base program is protected by a password so that no unauthorized entry or revisions can be made.

Author	Date	Project Mgr.	Date	QAD Representative	Date
Tanya Longaker	11/6/90	2 E Gorn	11/6/90	CK Gerbe	11/7/90
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
2 E Gorn	11/6/90	J. Baker	11/6/90		
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PNL-ALO-015	0	NOV 07 1990	1	2	

PNL TECHNICAL PROCEDURE

Bi-Monthly back-up will be made of the tracking system.

3.0 Data Reports

Data reports are generated as required to support the project management requirements. (See Attachment 1)

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SST SAMPLE TRACKING DATA BASES

1. SSTSEG

This database records information found on WHC COC that accompanies each segment. Items are: Date segment received, Core number, Segment number, WHC sample number, WHC shipping number, WHC tank number, WHC riser number, and WHC cask number.

2. SSTASAM

This database is the record of information contained on the 325A Hot Cell Transfer Form. Items included are: 325A sample ID number, ALO sample number, Core number, and Segment number, and COC number.

3. SSTSAMP

This database is the record sample information. Some of this information is contained on the ALO COC form. The status information is obtained from the Task Leaders. Items included are: ALO sample number, Test instruction number, COC number, Analysis required (i.e., organic, inorganic, or radiochemical), Requested analyte, Yes/no response whether data report has been received, Date data report received (if yes), and current status (if no).

4. SSTTI

This database is a record of all Test Instructions issued. It includes: TI number, Origin (325A or 325B), Issue date, Title, Person issued controlled copy, and Work place copy recipients.

5. SSTCOC

This database is a record of all COCs issued. It includes: COC number, origin (325A or 325B), date issued, sender, and receiver.

SST SAMPLE TRACKING REPORTS

Reports available in this program are described below.

1. Core Status

This report is designed to give a status for each SST core. Information reported is: SST sample number, Analysis requested (inorganic, organic, radchem), Specific analyte requested, and yes/no response whether data has been received (if yes, date data received).

You can request it to print out all cores with all segments, specific cores with all segments, specific segments, or specific samples.

2. Sample Status

This report is sorted by sample number (numerical order). Information reported is: Core number, ALO sample number, Analyte requested, date transmitted to labs, date data report received, and current status.

You can request entire report (all cores), specific date ranges, specific analysis tube or specific analyte.

3. TI/COC/Sample Number Report

This report provides an overview of all pertinent numbers associated with a particular core/segment. Information included: 325A ID #, Test Instruction number, all ALO sample numbers associated with TI, all COC numbers associated with sample numbers.

You can ask for specific core with all segments, all cores with all segments, or a specific segment within a core.

4. Test Instruction Log

A controlled document list. Includes TI number, Title, date issued, who it was issued to (controlled copy and work place copies).

Prints a list in TI number order.

5. Chain of Custody Log

A controlled document list. Includes COC number, date transmitted, sender, receiver, and all associated sample numbers.

Prints a list in COC number order.

INTERIM CHANGE NOTICE
(ICN)

ICN- PNL-ALO-051.1

Page 1 of 1

A. Document Number: <u>PNL-ALO-051</u> Revision Number: <u>0</u>		Effective Date of ICN: <u>10/14/91</u>
Document Title: <u>Sample Receiving</u>		Change Requested By: <u>B. M. Gillespie</u>
Document's Original Author: <u>B. M. Gillespie</u>		
B. Action: Replace Exhibit 1 with attached.		
C. Effect of Change: Clarify for which project samples are received.		
D. Reason for Change/Description of Change Reason for change: Clarify for which project samples are received. Description: Add under Received by: _____ "Customer Name or Project: _____"		
E. Approval Signatures (Please Sign and Date)	Type of Change: (Check (<input checked="" type="checkbox"/>) one) <input checked="" type="checkbox"/> Minor Change <input type="checkbox"/> Major Change	
Process Quality OS&R Department Concurrence: <u>GT Gerbe, Quality Engineer</u> Date: <u>5/20/91</u> Approval Authority: <u>[Signature]</u> Date: <u>5/20/91</u> Other Approvals: <u>B.M. Gillespie</u> Date: <u>5/20/91</u> : _____ Date: <u>1/1</u>		

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-051, SAMPLE RECEIVING

APPLICABILITY

This procedure describes the protocol that shall be used to receive Hanford TPA related samples submitted to the ACL. This procedure may be used for other samples if elected by the client or the analyst.

DEFINITIONS

Greater 325 Laboratory - All analytical labs within the 325, 329, 3720, 314 and 3708 buildings.

RESPONSIBLE STAFF

Technical Group Leader
Sample Custodian
Project Manager

PROCEDURE

- 1.0 Samples are delivered by the client to the greater 325 Laboratory. Specific sample receipt sites are identified in the project-specific Quality Assurance Project Plan (QAPP).
- 2.0 Upon receipt, the sample shipping container is moved to the appropriate room for opening by the sample custodian or his/her designee. After the shipping container is opened, the enclosed documentation is removed and the Sample Receipt Form (Exhibit 1) is completed by the sample custodian or his/her designee.
- 3.0 The following information is recorded on the Sample Receipt form (Exhibit 1):
 - Presence/absence of chain-of-custody record

Author <i>B.M. Dillinger</i>	Date <i>9-10-90</i>	Project Mgr. <i>B.M. Dillinger</i>	Date <i>9-10-90</i>	QAD Representative <i>GT Gerte</i>	Date <i>9/11/90</i>
Technical Reviewer <i>2 E Jones</i>	Date <i>9/10/90</i>	Line Mgr. <i>J. L. L. L.</i>	Date <i>9-12-90</i>	Other	Date
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PNL TECHNICAL PROCEDURE

- Condition of shipping container (i.e, broken container, dented, breached plastic bag, temperature of sample container, etc.)
 - Verify temperature of sample container by placing a thermometer between the outer barrier of the/a sample container and the ice in the ice chest. Wait a few minutes (3-5 minutes) and record temperature on the Sample Receipt Form (Exhibit 1).
 - Condition of Seals
 - Condition of sample vial(s)
 - Verification that sample identification agrees with chain-of-custody document
- 4.0 The Sample Custodian shall obtain a laboratory sample log-in number in accordance with PNL-MA-597 70-30.11 for all incoming samples and record them on the Sample Receipt Form (Exhibit 1) for cross-reference.
- 5.0 The Sample Custodian shall orally notify the Project Manager or her/his designee, that samples have arrived within 1-2 hours of receipt.
- 6.0 The Sample Custodian shall document all discrepancies on the Sample Receipt form and chain-of-custody record. The Sample Custodian shall orally notify the Project Manager of any discrepancies within 1-2 hours of receipt. The Project Manager shall take whatever actions required by the project-specific QAPP to resolve the discrepancies. Resolution shall be documented on the Sample Receipt Form.

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SAMPLE RECEIPT FORM

Delivered by: _____ Date/Time: _____

Received by: _____

Customer Name or Project: _____

Customer Sample Number(s): _____

ALO Sample Number(s): _____

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: _____
4. Sample Tag(s) ID Numbers if not Recorded on the Chain-of-Custody Record
or on Sample Vial.
Notes:
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.)
6. Condition of Sample Vials.
7. Verification of Agreement or Nonagreement of Information on Receiving
Documents.
8. Resolution of Problems or Discrepancies.

RETURN COMPLETED FORM TO PROJECT MANAGER

**INTERIM CHANGE NOTICE
(ICN)**

ICN - PNL-ALO-052.3-RO

Page 1 of 1

A.

Document Number: PNL-ALO-052 Revision Number: 0

Document Title: Analytical Balances

Document's Original Author: BM Gillespie

Effective Date
of ICN: 8 / 31 / 92

Change Requested by:
BM Gillespie

B. Action: Replace pages 1 through 2.

C. Effect of Change: Allows analyst to perform balance checks with a weight other than a class "S" weight.

D. Reason for Change/Description of Change:

The use of class "S" weights for balance checks is unnecessary. There are no EPA or PNL requirements that state "S" weights are to be used for balance calibration checks.

Delete the reference to use of Class "S" weights in section 3.0.

E. Approval Signatures:
(Please sign and date)

Type of Change: (Check one):

X Minor Major

Process

Quality Department: TL Ehlert *TL Ehlert* Date: 8 / 27 / 92

Approval Authority: AG King *AG King* Date: 8 / 27 / 92

Other Approvals: _____ Date: / /

: _____ Date: / /

INTERIM CHANGE NOTICE
(ICN)

ICN- PNL-ALO-052.2
Page 1 of 1

A. Document Number: <u>PNL-ALO-052.2</u> Revision Number: <u>0</u> Document Title: <u>ANALYTICAL BALANCES</u> Document's Original Author: <u>BM Gillespie</u>		Effective Date of ICN: <u>11-22-91</u>
		Change Requested By: <u>BM Gillespie</u>
B. Action Replace pages 1 & 2		
C. Effect of Change Delete the requirement that calls for keeping balance instructions with or near the balance.		
D. Reason for Change/Description of Change Reason: Not necessary to have balance instructions by analytical balance. This is a PNL maintenance shop responsibility. Description: Delete "these instructions shall be located with or near the balance" from step 1.0		
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>GK Gerke</u>	Date: <u>11/18/91</u>	
Approval Authority: AG KING <u>A. King</u>	Date: <u>11/18/91</u>	
Other Approvals: BM GILLESPIE <u>BM Gillespie</u>	Date: <u>11/22/91</u>	
:	Date: <u> </u> / <u> </u> / <u> </u>	

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-052, ANALYTICAL BALANCES

APPLICABILITY

This procedure describes the general operation of analytical balances. This procedure applies to operations performed in conjunction with the analysis of samples associated with Tri-Party Agreement activities or any other project/program as elected.

DEFINITIONS

None

RESPONSIBLE STAFF

Analysts

PROCEDURE

- 1.0 All balances in the laboratory shall be operated in accordance with the manufacturer's instructions. ~~These instructions shall be located with or near the balance.~~
- 2.0 Check the balance's calibration sticker to ensure the calibration has not expired. If the calibration has expired, refer to PAP-70-1201.
- 3.0 On a daily basis or when used if the balance is not used on a daily basis, perform a performance check of the balance ~~using a class "S" weight.~~ Use "S" a check weight that is near the range of expected sample weight. If the value is outside of specific tolerance of specification, do not use the equipment.
 - 3.1 Log date, check weight value and observed weight on a daily log sheet or notebook.
- 4.0 Weigh the samples as required.

Author	Date	Project Mgr.	Date	QAD Representative	Date
BM Gillespie		BM Gillespie		TL Ehlert	
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
TE Jones		PF Salter			
Procedure No.	Revision No.	Effective Date	Page		
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PNL TECHNICAL PROCEDURE

5.0 If the equipment calibration has expired or the performance check is out of tolerance, refer to PAP-70-1201. A Calibration Discrepancy Tag shall be written, initiating corrective action.

6.0 Factors affecting analytical balance use include the following:

6.1 A heavy, shock proof table (to minimize vibration effects) may be required for analytical balances capable of weighing to the nearest .0001g or lower.

6.2 The balance temperature, room temperature, and temperature of the material being weighed shall be equilibrated prior to making a measurement.

6.3 When not in use, the balance beam shall be locked and weights released from the beam on mechanical balances. Electronic balances shall be turned off when not in use.

6.4 The interior and exterior of the balance must be kept scrupulously clean. Chemicals must never be placed directly on the balance pan.

6.5 Anti-static cloths shall be used to wipe down nuisance dust from the balance interior.

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PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-053, DUTIES AND RESPONSIBILITIES OF SAMPLE CUSTODIAN

APPLICABILITY

The procedure shall be used to support work performed in compliance with Tri-Party Agreement requirements or any other project/program as elected.

DEFINITIONS

None

RESPONSIBLE STAFF

Sample Custodian
Project Manager

PROCEDURE

Duties and responsibilities of the sample custodian shall include but not be limited to:

- 1.0 Receiving samples
- 2.0 Inspecting sample shipping containers for presence/absence and condition of:
 - custody seals, "evidence tape," etc.
 - container leaks, breakage and/or container integrity
- 3.0 Recording condition of shipping containers and sample containers (bottles, jars, cans, etc.) in appropriate logbooks or on appropriate forms. Verify that samples that must be refrigerated arrive at temperature 4°C or less; i.e., ice still in existence.
- 4.0 Signing appropriate documents shipped with samples (i.e. air bills, chain-of-custody record(s), Traffic Reports, etc.)

Author <i>B.M. Dillman</i>	Date 9-19-90	Project Mgr. <i>B.M. Dillman</i>	Date 9-19-90	QAD Representative <i>GK Gerte</i>	Date 9/19/90
Technical Reviewer <i>E. Jones</i>	Date 9/20/90	Line Mgr. <i>[Signature]</i>	Date 9-19-90	Other	Date
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PNL TECHNICAL PROCEDURE

- 5.0 Verifying and recording agreement or non-agreement of information on sample documents (i.e., sample tags, chain-of custody records, traffic reports, air bills, etc.) in appropriate logbooks or on appropriate forms in accordance with PNL-ALO-051. If there is non-agreement, recording the problems, and notifying appropriate laboratory personnel or project manager PNL-ALO-051 (corrective action directions shall be documented in the case file by the Project Manager).
- 6.0 Assuring that sample tags that must be removed from the sample containers for storage are included in the appropriate sample file. Marking the sample containers as necessary after removal of sample tags to assure sample identity is maintained. Accounting for missing tags in a memo to the project file or documenting that the sample tags are actually labels attached to sample containers or were disposed of due to suspected contamination.
- 7.0 Logging the samples into the laboratory system and initiating the paper work requesting sample analyses on appropriate laboratory documents as required for analysis or according to laboratory standard operating procedures.
- 8.0 Marking or labeling samples with laboratory sample numbers and cross referencing laboratory numbers to client numbers and sample tag numbers.
- 9.0 Placing samples, sample extracts, and spent samples into appropriate storage and/or secure areas as required by Analytical Request Form (ARF), Test Instruction (TI) or Statement of Work (SOW).
- 10.0 Controlling access to samples in storage and assuring that chain-of-custody and laboratory standard operating procedures are followed when samples are removed from and returned to storage.
- 11.0 Assuring that chain-of custody documentation is completed in the laboratory and returned to the ALO-PSO, in accordance with PNL-ALO-010.
- 12.0 Monitoring storage conditions for proper sample preservation such as refrigeration temperature and prevention of cross-contamination in accordance with PNL-ALO-054. Any incidents of improper storage conditions shall be brought to the attention of the Laboratory Manager and Project Manager immediately. Deviations from standard storage protocols shall be documented in accordance with PAP-70-1502.
- 13.0 Disposing of, storage, or returning shipping containers, as appropriate, and in accordance with SOW or ARF requirements.

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PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-054, SAMPLE REFRIGERATOR TEMPERATURE MONITORING

APPLICABILITY

This procedure shall be used to support work performed in compliance with Tri-Party Agreement requirements or any other project/program as elected.

DEFINITIONS

None

RESPONSIBLE STAFF

Sample Custodian

PROCEDURE

- 1.0 The temperature inside all refrigerators containing samples and standards shall be monitored.
- 2.0 Minimum frequency for refrigerator temperature monitoring is once each work day. The temperature shall be checked and recorded along with date and signature (or initials) of the sample custodian or his/her designee at the beginning of every working day, before the refrigerator has been opened for extended periods of time. An alternative is to have a temperature monitor on the refrigerator which is checked each working day.
- 3.0 All temperature monitoring data shall be recorded in a logbook, data sheet, or on the temperature monitor recorder paper. Each refrigerator will utilize one of these recording mechanisms.
- 4.0 All refrigerators shall be at 4 ± 2 degrees centigrade. Any deviation from this range shall immediately be reported to the laboratory manager, project manager and the QA coordinator in a Deficiency Report, in accordance with PAP-70-1502. In addition, the temperature setting shall be re-set, if possible, to achieve the required temperature.

Author <i>B.M. Gillespie</i> 9-19-90	Date 9-19-90	Project Mgr. <i>B.M. Gillespie</i> 9-19-90	Date 9-19-90	QAD Representative <i>C. K. Korte</i> 9/19/90	Date 9/19/90
Technical Reviewer <i>22 Jones</i> 9/20/90	Date 9/20/90	Line Mgr. <i>T. J. [Signature]</i> 9-20-90	Date 9-20-90	Other	Date
Procedure No. PNL-ALO-054	Revision No. 0	Effective Date SEP 26 1990	Page 1	of 1	

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-055, SECURITY

APPLICABILITY

This procedure describes the security associated with the greater 325 Laboratory complex.

DEFINITIONS

Greater 325 Laboratory Complex - All analytical labs within the 325, 329, 3720, 314, and 3708 buildings

RESPONSIBLE STAFF

Laboratory Manager
Analyst

PROCEDURE

1.0 Security - General

The Battelle Pacific Northwest Laboratory in which the analyses are conducted is located in the 300 Area of the Hanford Reservation. The 300 Area perimeter is secured by a chain link fence patrolled by Westinghouse Hanford Company (the primary contractor for the Hanford site). Entry into the secured area must be obtained by passing through a secured guardhouse. All personnel must have a Department of Energy security clearance to obtain unescorted entry into the 300 area.

If samples are being processed, the laboratories which perform the analyses are locked when samples are in the lab during non-working hours. Staff members who have a need to enter the laboratory area have keys.

Author	Date	Project Mgr.	Date	QAD Representative	Date
B.M. Williams	9-10-90	B.M. Williams	9-10-90	C. K. Gierbe	9/11/90
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
J.E. Jones	9/10/90	J. H. H. H.	9-10-90		
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PNL TECHNICAL PROCEDURE

2.0 Security for Visiting Personnel

Non-cleared personnel must obtain proper clearance through DOE or PNL Security to visit the laboratory. Non-cleared personnel are escorted at all times.

3.0 Security for Samples

Samples shall be stored in a locked cabinet or area. Only designated personnel shall have access. Samples shall be returned to storage at the end of the working day.

A sample is checked out to technical staff for analysis. It is the analysts responsibility to assure the sample remains in custody. A sample is under custody if:

- It is in their actual possession
- It is in their view after being in their physical possession
- It was in their possession and then it was locked or sealed up to prevent tampering
- It is in a secure area (locked room with limited access)

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INTERIM CHANGE NOTICE
(ICN)

ICN - PNL-ALO-056.1 R0

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A.

Document Number: PNL-ALO-056 Revision Number: 0

Document Title: Assignment of Staff Responsibilities

Document's Original Author: BM Gillespie

Effective Date
of ICN: 5/12/92

Change Requested by:
TE Jones

B. Action:

Deleting ACT 89.1 and replacing with established records management practices.
Replace all pages due to new format.

C. Effect of Change:

Brings procedure into compliance.

D. Reason for Change/Description of Change:

ACT NOW Directive 89.1 no longer in existence.

E. Approval Signatures:
(Please sign and date)

Type of Change: (Check one):

☒ Minor ☐ Major

Process

Quality Department: TL Ehlert TL Ehlert

Date: 5/7/92

Approval Authority: AG King A. King

Date: 5/17/92

Other Approvals: TE Jones TE Jones

Date: 5/12/92

: _____ Date: 1/1

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-056, ASSIGNMENT OF STAFF RESPONSIBILITIES

APPLICABILITY

This SOP delineates the responsibilities of key personnel in the laboratory performing Tri-Party Agreement support analysis.

DEFINITION

None

RESPONSIBLE STAFF

Technical Group Leader/Task Leader (TGL/TL)
Project Manager
Analysts/Technicians
Sample Custodian
QA Representative
QC Coordinator

PROCEDURE

1.0 The Project Manager shall:

- Review and approve all data prior to release in accordance with PNL-MA-70, PAP-70-1101 and PNL-ALO-012.
- Approve Project QA plans.
- Provide input to line management on project needs, progress and problems.
- Work directly with project personnel to plan and monitor project progress.

Author	Date	Project Mgr.	Date	QAD Representative	Date
BM Gillespie		BM Gillespie		GK Gerke	
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
TE Jones		PF Salter		ALL ORIGINAL SIGNATURES ON FILE	
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PNL TECHNICAL PROCEDURE

- Assure project requirements are accurately and completely transmitted to the TGL/TL through Test Instruction (TI) in accordance with PNL-ALO-010 or Analytical Request Forms (ARFs) as indicated in the project Statement of Work (SOW).
- Compile, review, approve, and transmit data reports and data packages to the client in accordance with PNL-ALO-012.
- Provide the primary point of contact for the client.

2.0 Technical Group Leaders/Task Leaders for Organic analysis, Inorganic analysis and Radiochemistry shall:

- Provide technical direction to the analyst.
- Provide routine supervision of analytical activities in their group.
- Coordinate group/task activities to meet programmatic requirements.
- Review all technical and quality control data then group in accordance with PAP-70-1101 and PNL-ALO-012, and ~~ACT-NOW Directive 89-1~~ established records management practices.
- Report reviewed analytical results and QC data to the Project Manager.
- Assure the analysts under their supervision are technically trained and qualified to do assigned work in accordance with PAP-70-201.
- Assure that analytical work is conducted in accordance with project TIs or ARFs as defined in SOWs or Technical Program Plans (TPPs).

3.0 QA Representative shall:

- Implement QA activities as defined in SOW, QAPP, etc.
- Evaluate laboratory performance to assure that it is within SOW requirements.
- Provide for QA training.
- Provide direct input to line management on laboratory QA concerns and reviews.
- Approve QA project plans.

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PNL TECHNICAL PROCEDURE

4.0 QC Representative/Coordinator

- Evaluate laboratory QC performance.
- Review all data reports for completeness of QC data.
- Validate QC data for correctness.

5.0 Sample Custodian shall:

- Receive samples in accordance with PNL-ALO-051.
- Log in samples on receipt in accordance with PNL-MA-597 7-30.11.
- Maintain storage and inventory of samples in accordance with PNL-ALO-053.
- Return or dispose of sample in accordance with PNL-ALO-053, SOW or ARF requirements.

6.0 Analysts/Technicians shall:

- Be primarily responsible for adherence to QC requirements defined in the procedure and/or TI or ARF.
- Conduct analyses in accordance with approved procedures and TIs or ARF requirements.
- Document analytical results in accordance with the approved procedure, TI and/or ~~ACT NOW Directive 89-1~~ in accordance with established records management practices.
- Document all deviations from approved procedures in a Laboratory Record Book (LRB) and in accordance with PNL-MA-70, PAP-70-1502.
- Review data prior to reporting to the TGL/TL for accuracy, completeness and instrument calibration performance.
- Report data to the TGL/TL.

7.0 Cognizant Scientist shall:

- Provide technical direction to the analyst.
- Provide routine supervision of analytical activities in their assigned analytical area.
- Coordinate group/task activities to meet programmatic requirements.

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PNL TECHNICAL PROCEDURE

- Assure that analytical work is conducted in accordance with project TIs or ARFs as defined in SOWs or TPPs.
- Conduct analyses in accordance with approved procedures and TIs or ARF requirements.
- Document analytical results in accordance with the approved procedure, TI and/or ~~ACT NOW Directive 89-1~~ in accordance with established records management practices.
- Document all deviations from approved procedures in a LRB and in accordance with PNL-MA-70, PAP-70-1502.
- Review data prior to reporting to the TGL/TL for accuracy, completeness and instrument calibration performance.
- Report data to the TGL/TL.

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PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-057, SAMPLE STORAGE

APPLICABILITY

This procedure applies to samples of work to be performed in compliance with the Tri-Party Agreement requirements or other projects/programs as elected.

DEFINITIONS

None

RESPONSIBLE STAFF

Sample Custodian
Analyst

PROCEDURE

- 1.0 After receipt (see PNL-ALO-051) the samples shall be logged by sample custodian or his/her designee onto the Sample Inventory Log (Exhibit 1) and stored in a locked refrigerator or room.
- 2.0 The samples shall be removed from the shipping container and stored in their original sample containers unless damaged. Samples shall be stored at $4 \pm 2^{\circ}\text{C}$, radioactivity levels permitting, or, in accordance with requirements defined in the Statement of Work (SOW) or Analytical Request Form (ARF).
- 3.0 If samples are damaged, the Project Manager shall be notified within 24 hours of discrepancy and the Project Manager shall determine the appropriate manner of disposal in accordance with SOW or ARF requirements. Damaged samples shall then be disposed of in the designated manner and this disposal shall be documented on the Sample Inventory Log (Exhibit 1).

Author <i>B. M. Gilligan</i>	Date <i>11-15-90</i>	Project Mgr. <i>B. M. Gilligan</i>	Date <i>11-15-90</i>	QAD Representative <i>St Gerbe</i>	Date <i>11/19/90</i>
Technical Reviewer <i>2 E. Girono</i>	Date <i>11/15/90</i>	Line Mgr. <i>[Signature]</i>	Date <i>11/16/90</i>	Other	Date
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PNL TECHNICAL PROCEDURE

- 4.0 The storage area shall be kept locked at all times when samples are in storage. The sample custodian or designee shall control access to the storage area. (Duplicate keys for locked storage areas shall be maintained only by the assigned personnel). The locked laboratories 301A, 313, 400 and 417/419 of the 325 Building are designated as the storage facilities at this time. These laboratories are within a controlled area. The identified personnel with access to the storage area are designated by line management manager. A list of authorized personnel that have keys to sample storage areas will be maintained in the section office.
- 5.0 That the laboratory may satisfy sample chain-of-custody requirements, the following standard operating procedures for laboratory/sample security shall be implemented:
- 5.1 Samples shall be stored in a locked area.
 - 5.2 Access to the laboratory shall be limited.
 - 5.3 Uncleared visitors shall be escorted while in the laboratory.
 - 5.4 Refrigerators, freezers, and other sample storage areas shall be locked.
 - 5.5 Only the designated sample custodian and the supervisory personnel shall have keys to locked sample storage area(s).
 - 5.6 Samples shall remain in locked sample storage until removed for sample preparation or analysis.
 - 5.7 After a sample aliquot has been removed from storage and transferred to the analyst, the analyst is responsible for the custody of that aliquot. The remaining sample, after an aliquot is transferred to an analyst, shall be returned to the storage area before the end of the working day or prior to the end of his/her shift.
- 6.0 Standards shall not be stored with samples.
- 7.0 If a sample is totally consumed in the test, this shall be noted on the Sample Inventory Log (Exhibit 1).

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EXHIBIT 1. · SAMPLE INVENTORY LOG

[illegible]

**INTERIM CHANGE NOTICE
(ICN)**

ICN - PNL-ALO-058.1 R0
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A.

Document Number: PNL-ALO-058 Revision Number: 0

Document Title: Data Reporting

Document's Original Author: BM Gillespie

Effective Date
of ICN: 5/12/92

Change Requested by:
TE Jones

B. Action:

Deleting ACT 89.1 and replacing with established records management practices.
Replace all pages due to new format.

C. Effect of Change:

Brings procedure into compliance.

D. Reason for Change/Description of Change:

ACT NOW Directive 89.1 no longer in existence.

E. Approval Signatures:
(Please sign and date)

Type of Change: (Check one):

☒ Minor ☐ Major

Process

Quality Department: TL Ehlert *TL Ehlert*

Date: 5/7/92

Approval Authority: AG King *AG King*

Date: 5/7/92

Other Approvals: TE Jones *TE Jones*

Date: 5/12/92

: _____ Date: 1/1

PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-058, DATA REPORTING

APPLICABILITY

This procedure describes the requirements and authorities for reporting data supporting work performed in compliance with Tri-Party Agreement requirements or any other project/program as elected.

DEFINITIONS

None

RESPONSIBLE STAFF

Analyst/Technician
Technical Group Leader/Task Leader (TGL/TL)
Project Manager

PROCEDURE

1.0 The analyst is responsible for accurately recording in a legible manner what was done, how it was done, and what results were obtained for all steps of the analysis. This includes such information as:

- Preparation of standards
- Sample preparation
- Calculations
- Sample #
- M&TE used by serial #
- Reference to procedure by #, etc.

All work shall be signed and dated by the analyst when performed (as per PNL-MA-70, PAP-70-1101).

2.0 Data that is not recorded on data sheets specified by the procedure shall be recorded in Laboratory Record Books or on machine generated printouts in accordance with ACT NOW Directive 89-1 established records management practices.

Author	Date	Project Mgr.	Date	QAD Representative	Date
BM Gillespie		BM Gillespie		GK Gerke	
Technical Reviewer	Date	Line Mgr.	Date	Other	Date
TE Jones		PF Salter		ALL ORIGINAL SIGNATURES ON FILE	
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PNL TECHNICAL PROCEDURE

- 3.0 When the analysis is completed, the results shall be forwarded to the Technical Group Leader/Task Leader. The Technical Group Leader/Task Leader shall review all results and supporting data for technical reasonableness, compliance with QC limits, legibility, completeness, etc. (as per PAP-70-1101 and ACT NOW Directive 89-1). The TGL/TL shall flag suspect data and corrective action shall be taken and documented. The TGL/TL shall sign and date the analyte result documentation as evidence of review, and shall indicate what data were reviewed.
- 4.0 The Technical Group Leader/Task Leader shall report the data to the Project Manager.
- 5.0 The Project Manager, or his/her designee, shall review the data in the data package for compliance with requirements identified in the SOW and shall indicate review by signature/date documentation.
- 6.0 Only the Project Manager or his/her designee, shall release data to the client.
 - 6.1 Written and signed data reports will be accomplished in accordance with ALO Data Report/Package Review & Control, PNL-ALO-012.
 - 6.2 A written report shall follow the release of data by telecommunications (verbal or FAX) within 3 working days.

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PNL TECHNICAL PROCEDURE

TITLE: PNL-ALO-059, GENERAL REQUIREMENTS FOR GLASSWARE

APPLICABILITY

This procedure is applicable to the chemical analyses of samples for Tri-Party Agreement related programs/projects.

DEFINITIONS

None

RESPONSIBLE STAFF

Analyst
Technical Group Leader (TGL)/Task Leader

PROCEDURE

1.0 General Use of Glassware

An excellent general discussion of glassware can be found in Chapter 4 of the USEPA Handbook of Analytical Quality Control. This chapter is included as Attachment 1.

2.0 Glassware Storage

2.1 Organic Analysis Glassware Storage

I-CHEM series 300, precleaned, certified glassware is utilized. The glassware is stored in its shipping container until ready to use.

Reused glassware is minimal. It is cleaned as per EPA requirements (see Attachment 1, Section 4.6).

Author <i>B.M. Gillespie</i>	Date <i>9-19-90</i>	Project Mgr. <i>B.M. Gillespie</i>	Date <i>9-19-90</i>	QAD Representative <i>G. George</i>	Date <i>9/19/90</i>
Technical Reviewer <i>2 E. Jones</i>	Date <i>9/20/90</i>	Line Mgr. <i>T. Falt</i>	Date <i>9-20-90</i>	Other	Date
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PNL TECHNICAL PROCEDURE

2.2 Inorganic and Radiochemical Analysis Glassware Storage

Chemical reused glassware is stored in designated cabinets in each laboratory sealed with glass stoppers/lids or upside down until ready for use.

3.0 Glassware Cleaning

Glassware cleaning methodology is described, in detail, in Attachment 1. The TGL shall assure that glassware is adequately cleaned. At a minimum this shall be accomplished by washing in hot or cold water, rinsing twice with deionized water, and allowing the glassware to air dry. The acceptability of this method is established through the evaluation of methods blank data. If the concentration of analyte of interest is greater than the detection limit as determined by methods blanks requested by the client, one of the more stringent glass cleaner solutions defined in Attachment 1 shall be used. The exact cleaning solution method to be used is dependent in the nature of the contaminating substance.

It is not general practice to leach all new and used glassware due to the current PNL waste minimization policy. Past analyses indicate no need for leaching glassware (except for Hg analysis). The laboratories typically do not reuse glassware.

4.0 Reference

Chapter 4 from U.S. Environmental Protection Agency, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Office of Research and Development, Cincinnati, Ohio, March 1979.

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Chapter 4

GLASSWARE

4.1 General

The measurement of trace constituents in water demands methods capable of maximum sensitivity. This is especially true for metals and trace organics such as pesticides, as well as for the determination of ammonia and phosphorus. In addition to sensitive methods, however, there are other areas that require special consideration. One such area is that of the cleanliness of laboratory glassware. Obviously, the very sensitive analytical systems are more sensitive to errors resulting from the improper use or choice of apparatus, as well as to contamination effects due to an improper method of cleaning the apparatus. The purpose of this chapter is to discuss the kinds of glassware available, the use of volumetric ware, and various cleaning requirements.

4.2 Types of Glassware

Laboratory vessels serve three functions: storage of reagents, measurement of solution volumes, and confinement of reactions. For special purposes, vessels made from materials such as porcelain, nickel, iron, aluminum, platinum, stainless steel, and plastic may be employed to advantage. Glass, however, is the most widely used material of construction. There are many grades and types of glassware from which to choose, ranging from student grade to others possessing specific properties such as super strength, low boron content, and resistance to thermal shock or alkali. Soft glass containers are not recommended for general use, especially for storage of reagents because of the possibility of dissolving of the glass (or of some of the constituents of the glass). The mainstay of the modern analytical laboratory is a highly resistant borosilicate glass, such as that manufactured by Corning Glass Works under the name "Pyrex" or by Kimble Glass Co. as "Kimax." This glassware is satisfactory for all analyses included in reference 1.

Depending on the particular manufacturer, various trade names are used for specific brands possessing special properties such as resistance to heat, shock, and alkalis. Examples of some of these special brands follow:

- a. Kimax- or Pyrex-brand glass is a relatively inert all-purpose borosilicate glass.
- b. Vycor-brand glass is a silica glass (96 percent) made to withstand continuous temperatures up to 900°C and can be down-shocked in ice water without breakage.
- c. Corning-brand glass is claimed to be 50 times more resistant to alkalis than conventional ware and practically boron-free (maximum 0.2 percent).
- d. Ray-Sorb- or Low-Actinic-brand glass is used when the reagents or materials are light sensitive.
- e. Corex-brand labware is harder than conventional borosilicates and therefore better able to resist clouding and scratching.

The use of plastic vessels, containers, and other apparatus made of Teflon, polyethylene, polystyrene, and polypropylene has increased markedly over recent years. Some of these

materials, such as Teflon, are quite expensive; however, Teflon stopcock plugs have practically replaced glass plugs in burets, separatory funnels, etc., because lubrication to avoid sticking or "freezing" is not required. Polypropylene, a methylpentene polymer, is available as laboratory bottles, graduates, beakers, and even volumetric flasks. It is crystal clear, shatterproof, autoclavable, and chemically resistant.

The following are some points to consider in choosing glassware or plasticware:

- a. The special types of glass listed above, other than Pyrex or Kimax, generally are not required to perform the analyses given in "Methods for Chemical Analysis of Water and Wastes" (1).
- b. Unless instructed otherwise, borosilicate or polyethylene bottles may be used for the storage of reagents and standard solutions.
- c. Dilute metal solutions are prone to plate out on container walls over long periods of storage. Thus, dilute metal standard solutions must be prepared fresh at the time of analysis.
- d. For some operations, disposable glassware is entirely satisfactory. One example is the use of disposable test tubes as sample containers for use with the Technicon automatic sampler.
- e. Plastic bottles of polyethylene and Teflon have been found satisfactory for the shipment of water samples. Strong mineral acids (such as sulfuric acid) and organic solvents will readily attack polyethylene and are to be avoided.
- f. Borosilicate glassware is not completely inert, particularly to alkalis; therefore, standard solutions of silica, boron, and the alkali metals are usually stored in polyethylene bottles.

For additional information the reader is referred to the catalogs of the various glass and plastic manufacturers. These catalogs contain a wealth of information such as specific properties, uses, and sizes.

4.3 Volumetric Analyses

By common usage, accurately calibrated glassware for precise measurements of volume has become known as volumetric glassware. This group includes volumetric flasks, volumetric pipets, and accurately calibrated burets. Less accurate types of glassware including graduated cylinders and serological and measuring pipets also have specific uses in the analytical laboratory when exact volumes are unnecessary.

The precision of volumetric work depends in part upon the accuracy with which volumes of solutions can be measured. There are certain sources of error that must be carefully considered. The volumetric apparatus must be read correctly; that is, the bottom of the meniscus should be tangent to the calibration mark. There are other sources of error, however, such as changes in temperature, which result in changes in the actual capacity of glass apparatus and in the volume of the solutions. The capacity of an ordinary glass flask of 1000-ml volume increases 0.025 ml/deg with rise in temperature, but if the flask is made of borosilicate glass, the increase is much less. One thousand milliliters of water or of most 0.1N solutions increases in volume by approximately 0.20 ml/deg increase at room

temperature. Thus solutions must be measured at the temperature at which the apparatus was calibrated. This temperature (usually 20°C) will be indicated on all volumetric ware. There may also be errors of calibration of the apparatus; that is, the volume marked on the apparatus may not be the true volume. Such errors can be eliminated only by recalibrating the apparatus or by replacing it.

Volumetric apparatus is calibrated to contain or to deliver a definite volume of liquid. This will be indicated on the apparatus with the letters "TC" (to contain) or "TD" (to deliver). Volumetric flasks are calibrated to contain a given volume and are available in various shapes and sizes.

Volumetric pipets are calibrated to deliver a fixed volume. The usual capacities are 1 through 100 ml although micropipets are also available. Micropipets are most useful in furnace work and are available in sizes ranging from 1 to 100 μ l.

In emptying volumetric pipets, they should be held in a vertical position and the outflow should be unrestricted. The tip of the pipet is kept in contact with the wall of the receiving vessel for a second or two after the free flow has stopped. *The liquid remaining in the tip is not removed*; this is most important.

Measuring and serological pipets should also be held in a vertical position for dispensing liquids; however, the tip of the pipet is only touched to the wet surface of the receiving vessel after the outflow has ceased. For those pipets where the small amount of liquid remaining in the tip is to be blown out and added, indication is made by a frosted band near the top.

Burets are used to deliver definite volumes. The more common types are usually of 25- or 50-ml capacity, graduated to tenths of a milliliter, and are provided with stopcocks. For precise analytical methods in microchemistry, microburets are also used. Microburets generally are of 5- or 10-ml capacity, graduated in divisions of hundredths of a milliliter. Automatic burets with reservoirs are also available ranging in capacity from 10 to 100 ml. Reservoir capacity ranges from 100 to 4,000 ml.

General rules in regard to the manipulation of a buret are as follows: Do not attempt to dry a buret that has been cleaned for use, but rinse it two or three times with a small volume of the solution with which it is to be filled. Do not allow alkaline solutions to stand in a buret because the glass will be attacked, and the stopcock, unless made of Teflon, will tend to freeze. A 50-ml buret should not be emptied faster than 0.7 ml/s, otherwise too much liquid will adhere to the walls and as the solution drains down, the meniscus will gradually rise, giving a high false reading. It should be emphasized that improper use or reading of burets can result in serious calculation errors.

In the case of all apparatus for delivering liquids, the glass must be absolutely clean so that the film of liquid never breaks at any point. Careful attention must be paid to this fact or the required amount of solution will not be delivered. The various cleaning agents and their use are described later.

4.4 Federal Specifications for Volumetric Glassware

Reference 2 contains a description of Federal specifications for volumetric glassware. The National Bureau of Standards no longer accepts stock quantities of volumetric apparatus

from manufacturers or dealers for certification and return for future sale to consumers. This certification service is still available, but apparatus will be tested only when submitted by the ultimate user, and then only after an agreement has been reached with the Bureau concerning the work to be done.

Consequently, the various glass manufacturers have discontinued the listing of NBS-certified ware. In its place catalog listings of volumetric glass apparatus that meet the Federal specifications are designated as class A and all such glassware is permanently marked with a large "A." These NBS specifications are listed in table 4-1. The glassware in question includes the usual burets, volumetric flasks, and volumetric pipets.

In addition to the "A" marking found on calibrated glassware and the temperature at which the calibration was made, other markings also appear. These include the type of glass, such

Table 4-1
TOLERANCES FOR VOLUMETRIC
GLASSWARE¹

Type of Glassware	Capacity ² (ml)	Limit of Error (ml)
Graduated flask	25	0.03
	50	0.05
	100	0.08
	200	0.10
	250	0.11
	300	0.12
	500	0.15
	1,000	0.30
	2,000	0.50
Transfer pipet	2	0.006
	5	0.01
	10	0.02
	25	0.025
	30	0.03
	50	0.05
	100	0.08
	200	0.10
Buret ³	5	0.01
	10	0.02
	30	0.03
	50	0.05
	100	0.10

¹ Abridged from reference 3.

² Less than and including.

³ Limits of error are of total or partial capacity. Customary practice is to test the capacity at five intervals.

as Pyrex, Corex, or Kimax; the stock number of the particular item; and the capacity of the vessel. If the vessel contains a ground-glass connection, this will also be included along with the TD or TC symbol. An example of the markings usually found on volumetric ware is shown in figure 4-1. Class A glassware need not be recalibrated before use. However, if it should become necessary to calibrate a particular piece of glassware, directions may be found in texts (4) on quantitative analysis.

4.5 Cleaning of Glass and Porcelain

The method of cleaning should be adapted to both the substances that are to be removed, and the determination to be performed. Water-soluble substances are simply washed out with hot or cold water, and the vessel is finally rinsed with successive small amounts of distilled water. Other substances more difficult to remove may require the use of a detergent, organic solvent, dichromate cleaning solution, nitric acid, or aqua regia (25 percent by volume concentrated HNO_3 in concentrated HCl). In all cases it is good practice to rinse a vessel with tap water as soon as possible after use. Material allowed to dry on glassware is much more difficult to remove.

Volumetric glassware, especially burets, may be thoroughly cleaned by a mixture containing the following: 30 g of sodium hydroxide, 4 g of sodium hexametaphosphate (trade name, Calgon), 8 g of trisodium phosphate, and 1 l of water. A gram or two of sodium lauryl sulfate or other surfactant will improve its action in some cases. This solution should be used with a buret brush.

Dichromate cleaning solution (chromic acid) is a powerful cleaning agent; however, because of its destructive nature upon clothing and upon laboratory furniture, extreme care must be taken when using this mixture. If any of the solution is spilled, it must be cleaned up immediately. Chromic acid solution may be prepared in the laboratory by adding 1 l of concentrated sulfuric acid slowly, with stirring, to a 35-ml saturated sodium dichromate solution. This mixture must be allowed to stand for approximately 15 min in the vessel that is being cleaned and may then be returned to a storage bottle. Following the chromic acid wash, the vessels are rinsed thoroughly with tap water, then with small successive portions of distilled water. The analyst should be cautioned that when chromium is included in the

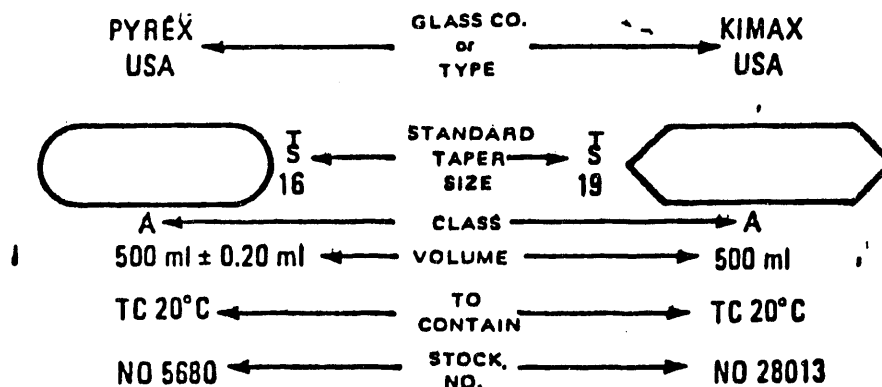


Figure 4-1. Example of markings on glassware.

scheme of analysis, it is imperative that the last traces of dichromate be removed from the apparatus. To this end, a substitute for dichromate cleaning solution, called Nochromix,* is available and may be used to advantage. Fuming nitric acid is another powerful cleaning agent, but is disagreeable to handle. As with dichromate, when the acid becomes dilute, the cleaning mixture is no longer effective. A mixture of concentrated sulfuric and fuming nitric acids is even more efficient but is also hazardous to use. A persistent greasy layer or spot may be removed by acetone or by allowing a warm solution of sodium hydroxide, about 1 g per 50 ml of water, to stand in the vessel for 10 to 15 min; after rinsing with water, dilute hydrochloric acid, and water again, the vessel is usually clean. Alcoholic potassium hydroxide is also effective in removing grease. To dry glass apparatus, rinse with acetone and blow or draw air through it.

4.6 Special Cleaning Requirements

Absorption cells, used in spectrophotometers, should be kept scrupulously clean, free of scratches, fingerprints, smudges, and evaporated film residues. The cells may be cleaned with detergent solutions for removal of organic residues, but should not be soaked for prolonged periods in caustic solutions because of the possibility of etching. Organic solvents may be used to rinse cells in which organic materials have been used. Nitric acid rinses are permissible, but dichromate solutions are not recommended because of the adsorptive properties of dichromate on glass. Rinsing and drying of cells with alcohol or acetone before storage is a preferred practice. Matched cells should be checked to see that they are equivalent by placing portions of the same solution in both cells and taking several readings of the transmittance (T , percent) or optical density (OD) values.

For certain determinations, especially trace metals, the glassware should also be rinsed with a 1:1 nitric acid-water mixture. This operation is followed by thoroughly rinsing with tap water and successive portions of distilled water. This may require as many as 12 to 15 rinses, especially if chromium is being determined. The nitric acid rinse is also especially important if lead is being determined.

Glassware to be used for phosphate determinations should not be washed with detergents containing phosphates. This glassware must be thoroughly rinsed with tap water and distilled water. For ammonia and Kjeldahl nitrogen, the glassware must be rinsed with ammonia-free water. (See ch. 2.)

Glassware to be used in the determination of trace organic constituents in water, such as chlorinated pesticides, should be as free as possible of organic contaminants. A chromic acid wash of at least 15 min is necessary to destroy these organic residues. Rinse thoroughly with tap water and, finally, with distilled water. Glassware may be dried for immediate use by rinsing with redistilled acetone. Otherwise glassware may be oven dried or drip dried. Glassware should be stored immediately after drying to prevent any accumulation of dust and stored inverted or with mouth of glassware covered with foil.

Bottles to be used for the collection of samples for organic analyses should be rinsed successively with chromic acid cleaning solution, tap water, distilled water, and, finally, several times with a redistilled solvent such as acetone, hexane, petroleum ether, or chloroform. Caps are washed with detergent, rinsed with tap water, distilled water, and

*Available from Kodak Laboratories, 6 Varick Street, New York, N.Y. 10013.

solvent. Liners are treated in the same way as the bottles and are stored in a sealed container.

4.7 Disposable Glassware

When the risk of washing a pipet for reuse becomes too great, as in the case of use with toxic materials, or when the cost of washing glassware becomes prohibitive, disposable vessels may be the answer, provided they meet the necessary specification. Various types are available, including bacteriological, serological, and microdilution pipets. Disposable glassware generally is made of soft glass although plastic vessels and pipets are also available.

4.8 Specialized Glassware

The use of vessels and glassware fitted with standard-taper, ground-glass, and ball-and-socket joints has increased because of certain advantages such as less leakage and fewer freezeups. Standard-taper, interchangeable ground joints save time and trouble in assembling apparatus. They are precision-ground with tested abrasives to insure an accurate fit and freedom from leakage. Ball and socket joints increase flexibility of operation and eliminate the need for exact alignments of apparatus. Symbols and their meaning as applied to standard joints, stoppers, and stopcocks are shown below.

4.8.1 Standard Taper (T)

The symbol T is used to designate interchangeable joints, stoppers, and stopcocks that comply with the requirements of reference 5. All mating parts are finished to a 1:10 taper.

The size of a particular piece appears after the appropriate symbol. Primarily because of greater variety of apparatus equipped with T fittings, a number of different types of identifications are used:

- a. For joints—a two-part number as T 24/40, with 24 being the approximate diameter in millimeters at the large end of the taper and 40 the axial length of taper, also in millimeters
- b. For stopcocks—a single number, as T 2, with 2 mm being the approximate diameter of the hole or holes through the plug
- c. For bottles—a single number, as T 19, with 19 mm being the appropriate diameter at top of neck. However, there are differences in dimensions between the bottle and flask stoppers
- d. For flasks and similar containers—a single number, as T 19, with 19 mm being the appropriate diameter of the opening at top of neck

4.8.2 Spherical Joints (S)

The designation S is for spherical (semiball) joints complying with reference 5. The complete designation of a spherical joint also consists of a two-part number, as S 12/2, with 12 being the approximate diameter of the ball and 2 the bore of the ball and the socket, also in millimeters.

4.8.3 Product Standard (§)

The symbol § is used for stopcocks with Teflon plugs, the mating surfaces being finished to a 1:5 taper. As with § stopcocks, a single number is used. Thus, § 2 means a Teflon stopcock with a hole of approximately 2-mm diameter in the plug.

4.9 Fritted Ware

For certain laboratory operations the use of fritted ware for filtration, gas dispersion, absorption, or extractions may be advantageous.

There are six different porosities of fritted ware available, depending on its intended use. Porosity is controlled in manufacture, and disks are individually tested and graded into these classifications. The extra-coarse and coarse porosities are held toward the maximum pore diameter as listed. The medium, fine, very fine, and ultrafine are held toward the minimum pore diameter as listed in table 4-2.

Pore sizes are determined by the method specified in reference 6.

4.9.1 Recommended Procedures for Maximum Filter Life

- a. New Filters. Wash new filters by suction with hot hydrochloric acid, followed by a water rinse.
- b. Pressure Limits. The maximum, safe, differential pressure on a disk is 15 lb/in².
- c. Thermal Shock. Fritted ware has less resistance to thermal shock than nonporous glassware. Hence, excessive, rapid temperature changes and direct exposure to a flame should be avoided. Heating in a furnace to 500°C may be done safely, provided the heating and cooling are gradual. Dry ware may be brought to constant weight by heating at 105°C to 110°C.

Never subject a damp filter of ultrafine porosity to a sudden temperature change. Steam produced in the interior may cause cracking.

Table 4-2
FRITTED-WARE POROSITY

Porosity Grade	Designation	Pore Size (μm)	Principal Uses
Extra Coarse	EC	170-220	Coarse filtration; gas dispersion, washing, and absorption
Coarse	C	40-60	Coarse filtration; gas dispersion, washing, and absorption
Medium	M	10-15	Filtration and extraction
Fine	F	4-5.5	Filtration and extraction
Very Fine	VF	2-2.5	General bacterial filtration
Ultrafine	UF	0.9-1.4	General bacterial filtration

4.9.2 Cleaning of Used Filters

In many cases, precipitates can be removed by rinsing with water, passed through from the underside, with the pressure not exceeding 15 lb/in². The suggestions that follow will be helpful in dealing with material that will not be removed by such a reverse water-wash:

<i>Material</i>	<i>Removal Agent</i>
Albumen	Hot ammonia or hydrochloric acid
Aluminous and siliceous residues. . . .	Hydrofluoric acid (2 percent) followed by concentrated sulfuric acid; rinse immediately with water until no trace of acid can be detected.
Copper or iron oxides	Hot hydrochloric acid plus potassium chlorate
Fatty materials	Carbon tetrachloride
Mercuric sulfide.	Hot aqua regia
Organic matter	Hot, concentrated cleaning solution, or hot, concentrated sulfuric acid with a few drops of sodium nitrite
Silver chloride	Ammonium or sodium hyposulfite

4.10 References

1. Methods for Chemical Analysis of Water and Wastes, U.S. EPA, Office of Research and Development, EMSL (1978).
2. Hughes, J. C., Testing of Glass Volumetric Apparatus, NBS Circular 602, National Bureau of Standards (1959).
3. Peffer, E. L., and Mulligan, G. C., Testing of Glass Volumetric Apparatus, NBS Circular 434, National Bureau of Standards (1941).
4. Willard, H. H., and Furman, N. H., Elementary Quantitative Analysis—Theory and Practice, D. Van Nostrand Co., Inc., New York (1947).
5. Interchangeable Ground Glass Joints, Commercial Standard CS-21-30, National Bureau of Standards (Aug. 25, 1930).
6. Maximum Pore Diameter and Permeability of Rigid Porous Filters for Laboratory Use, E-128, American Society for Testing and Materials, Philadelphia (1968).

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