

Los Alamos National Laboratory
Quality Assurance Documents
for the
Nevada Nuclear Waste Storage Investigations

VOLUME I

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LANL-NNWSI-QAPP, R2	Los Alamos National Laboratory Quality Assurance Program Plan for Nevada Nuclear Waste Storage Investigations.

QUALITY ASSURANCE PROCEDURES (QP)

NOTE: Please note that the table of contents now reflects new procedural alpha numeric designators. As each procedure is revised, the new designator will be used and ultimately the old designator will be completely removed.

<u>Present Designations</u>	<u>Title</u>	<u>New Designations</u>
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CHANGE REQUESTS (CR)

CR No. 008	Modifies QP-06, R2 (CR in front of QP)
CR No. 009	Modifies QP-18, R1 (CR in front of QP)
CR No. 012	Modifies QP alpha numeric code (CR in front of Volume I Table of Contents)
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CR No. 017	Modifies QP-07, R2 (CR in front of QP)
CR no. 032	Modifies QP-17, R0 (CR in front of QP)
CR No. 019	Modifies QP-14, R1 (CR in front of QP)
CR No. 024	Modifies QP-16, R1 (CR in front of QP-16, R1)
CR No. 026	Modifies QP-19,R0 (CR in front of QP)

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TWS-INC-DP-26, R0	Preparation of Aqueous Standards for Analysis of Water Samples
TWS-INC-DP-27, R0	Trace Element Determination by Plasma Emission Spectrometry
TWS-INC-DP-30, R0	Partial CO ₂ Atmospheric Control of Groundwater Chemistry
TWS-INC-DP-34, R0	Sulfide Electrode Measurements
TWS-INC-DP-35, R0	pH Measurements (CR006)
TWS-INC-DP-36, R0	Eh (Oxidation-Reduction Potential) Measurements
TWS-INC-DP-37, R0	Anaerobic Field Filtering Apparatus
TWS-INC-DP-38, R0	Determination of Detergent Concentrations, Anionic
TWS-INC-DP-39, R0	Dissolved Oxygen Determinations
TWS-INC-DP-40, R0	Chloride Ion, Dissolved Electrode Method
TWS-INC-DP-41, R0	Carbon Dioxide, Gaseous Electrode Method
TWS-INC-DP-42, R0	Measurement of Conductivity using the YSI Model 31 Conductivity Bridge
TWS-INC-DP-43, R0	Procedure for Titration of Alkalinity by Strong Acid Using an Automatic Titrator
TWS-INC-DP-44, R0	Procedure for Titration of Alkalinity Using the Hach Titration System
TWS-INC-DP-45, R0	Analysis of Strong Acid Anions by Ion Chromatography (Dionex Model 16)
TWS-INC-DP-60, R1	Preparation of NTS Core Samples for NNWSI Solid Core Experiments
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TWS-INC-DP-63, R0	Preparation of NTS Core Samples for NNWSI Crushed Rock Experiments
TWS-INC-DP-65, R0	Procedure for Volcanism Field Studies

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TWS-HSE5-DP-202, R0	Operating Instructions for Amray Model 1000 Scanning Electron Microscope and Kevex Model 7000 Energy Dispersive X-Ray Analyzer for Evaluation of Air Samples Collected on Nuclepore Filters
TWS-HSE5-DP-206	Fiber Counting Procedure
TWS-HSE5-DP-211, R0	Preparation and use of Air Particulate Filter Sampling Devices
TWS-HSE5-DP-212, R0	Preparation, Calibration, and use of Cascade Impactors
TWS-HSE5-DP-213, R0	Procedure For The Calibration and Use of SKC Personal Sampling Pumps
TWS-HSE5-DP-214, R0	Procedures For The Calibration and Use of Alpha-1 Personal Sampling Pumps
TWS-HSE5-DP-215, R0	Procedures For The Calibration of The Singer Dry Gas Meter

WX-Design Engineering Division DPs

TWS-WX-DP-59, R0	NNWSI Exploratory Shaft Facility Design Control Procedure
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CHANGE REQUESTS (CR)

CR No. 006	Modifies DP-35,R0 (CR in front of DP)
CR No. 025	Modifies TWS-INC-DP-35, R0 (CR in front of DP-35, R0)

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TWS-ESS-DP-03, R2	Nevada Test Site Core Petrography Procedure
TWS-ESS-DP-04, R4	Thin Section Preparation Procedure
TWS-ESS-DP-06, R2	Operating Instructions for DV-502 Vacuum Evaporator Used in Carbon Coating Samples
TWS-ESS-DP-07, R2	Microprobe Operating Procedure
TWS-ESS-DP-10, R1	Procedure for Compressive Strength Tests
TWS-ESS-DP-16, R3	Siemens X-Ray Diffraction Procedure
TWS-ESS-DP-20, R1	Preparation of Fused Beads for Electron Microprobe Analysis of Rock Powders
TWS-ESS-DP-24, R0	Procedure: Alignment of the Siemens Diffractometer
TWS-ESS-DP-25, R2	Clay Mineral Separation and Preparation for X-Ray Diffraction Analysis
TWS-ESS-DP-28, R0	Nevada Test Site Fracture Filling Studies Procedure
TWS-ESS-DP-50, R0	Sputter Coater Operating Procedure for Gold Coating Samples
TWS-ESS-DP-51, R0	Mettler H80 Operation Procedure (X-Ray Fluorescence Analysis Sample Weighing Procedure)
TWS-ESS-DP-52, R0	Fusing Using The Junior Orbit Shaker
TWS-ESS-DP-53, R0	Pulverizing Using the Spex 8500 Shatterbox
TWS-ESS-DP-54, R0	Crushing: Operation of 50 Ton Hydraulic Press
TWS-ESS-DP-55, R0	Rock Splitting: Operation of 50 Ton Hydraulic Press
TWS-ESS-DP-56, R1	Brinkman Automated Grinder Procedure
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TWS-ESS-DP-103, R0	Geopetal Orientation Measurement
TWS-ESS-DP-105, R0	Thermal Calibration Procedure
TWS-ESS-DP-106, R0	Philips X-ray Diffraction Procedure
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TWS-ESS-DP-112, R0	Operating Instructions for International Scientific Instruments Model DS-130 Scanning Electron Microscope and Tracor Northern Series II X-Ray Analyzer
TWS-ESS-DP-113, R0	Procedure: Temperature Determinations From Fluid Inclusion Studies
TWS-ESS-DP-114, R0	Sample Collection Procedure for Rock Varnish Studies
TWS-ESS-DP-115, R0	Vaisala HMI-32 Humidity and Temperature Probe Procedure

ENVIRONMENTAL SCIENCE

TWS-HSE12-DP-301, R0	Field Collection of Experimental Materials
TWS-HSE12-DP-307, R0	Sample Identification and Control
TWS-HSE12-DP-310, R0	Calibration and Use of the Phototachometer
TWS-HSE12-DP-311, R0	Sample Preparation
TWS-HSE12-DP-312, R0	Particle Size Reduction of Geologic Media
TWS-HSE12-DP-313, R0	Calibration and Use of Centrifuges
TWS-HSE12-DP-316, R0	Preparation of Standard and Reagent Solutions
TWS-HSE12-DP-317, R0	Calibration and Use of Analytical and TOP-Loading Balances
TWS-HSE12-DP-318, R0	pH Measurement, Acid-Base Solution Standardization, and Total Alkalinity Procedure

CHANGE REQUESTS (CR)

CR No. 022	Modifies TWS-ESS-DP-28, R0 (CR in front of DP-28, R0)
CR No. 028	Modifies TWS-ESS-DP-04, R4 (CR in front of DP-04, R4)
CR No. 029	Modifies TWS-ESS-DP-114, R0 (CR in front of DP-114, R0)
CR No. 030	Modifies TWS-ESS-DP-53, R0 (CR in front of DP-53, R0)

QUALITY ASSURANCE PROGRAM INDEX OF PROCEDURES
FOR LOS ALAMOS NNWSI PROJECT

This index is prepared and maintained in accordance with
TWS-MSTQA-QP-02.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
1.	Organization	
2.	Quality Assurance Program	LANL-NNWSI-QAPP Sections 1 and 2. TWS-QAS-QP-02.1 TWS-MSTQA-QP-18
3.	Design Control	LANL-NNWSI-QAPP Section 3. TWS-WX-DP-59
4.	Procurement Document Control	LANL-NNWSI-QAPP Section 4. TWS-QAS-QP-04.1 TWS-QAS-QP-22
5.	Instructions, Procedures, and Drawings	LANL-NNWSI-QAPP Section 5. TWS-QAS-QP-03 TWS-QAS-QP-05.1 TWS-QAS-QP-05.2 TWS-MSTQA-QP-07 TWS-MSTQA-QP-09 TWS-MSTQA-QP-11 TWS-QAS-QP-12.1 TWS-QAS-QP-13.1 TWS-MSTQA-QP-14 TWS-MSTQA-QP-15 TWS-INC-WP-12 TWS-ESS-DP-01 TWS-INC-DP-02 TWS-ESS-DP-03 TWS-ESS-DP-04 TWS-CNC-DP-05

* Procedures affected by this issue have been underscored.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
5.	Instruction, Procedures, and Drawings (continued)	TWS-ESS-DP-06 TWS-ESS-DP-07 TWS-ESS-DP-09 TWS-ESS-DP-10 TWS-ESS-DP-11 TWS-CNC-DP-14 TWS-CNC-DP-15 TWS-ESS-DP-16 TWS-CNC-DP-17 TWS-CNC-DP-18 TWS-ESS-DP-20 TWS-CNC-DP-22 TWS-CNC-DP-23 TWS-ESS-DP-24 TWS-ESS-DP-25 TWS-INC-DP-26 TWS-INC-DP-27 TWS-ESS-DP-28 TWS-INC-DP-30 TWS-INC-DP-34 TWS-INC-DP-35 TWS-INC-DP-36 TWS-INC-DP-37 TWS-INC-DP-38 TWS-INC-DP-39 TWS-INC-DP-40 TWS-INC-DP-41 TWS-INC-DP-42 TWS-INC-DP-43 TWS-INC-DP-44 TWS-INC-DP-45 TWS-ESS-DP-50 TWS-ESS-DP-51 TWS-ESS-DP-52 <u>TWS-ESS-DP-53</u> <u>TWS-ESS-DP-54</u> TWS-ESS-DP-55 TWS-ESS-DP-56 TWS-WX-DP-59 TWS-INC-DP-60 TWS-INC-DP-61 TWS-INC-DP-62 TWS-INC-DP-63 TWS-INC-DP-65 TWS-INC-DP-101 TWS-ESS-DP-102 TWS-ESS-DP-103 TWS-ESS-DP-105 TWS-ESS-DP-106

* Procedures affected by this issue have been underscored.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
5.	Instruction, Procedures, and Drawings (concluded)	TWS-ESS-DP-107 TWS-ESS-DP-110 TWS-ESS-DP-111 TWS-ESS-DP-112 TWS-ESS-DP-113 TWS-INC-DP-114 TWS-ESS-DP-115 TWS-HSE5-DP-201 TWS-HSE5-DP-202 TWS-HSE-5-DP-206 TWS-HSE5-DP-211 TWS-HSE5-DP-212 TWS-HSE5-DP-213 TWS-HSE5-DP-214 TWS-HSE5-DP-215 TWS-HSE12-DP-301 TWS-HSE12-DP-307 <u>TWS-HSE12-DP-310</u> <u>TWS-HSE12-DP-311</u> <u>TWS-HSE12-DP-312</u> <u>TWS-HSE12-DP-313</u> <u>TWS-HSE12-DP-316</u> <u>TWS-HSE12-DP-317</u> <u>TWS-HSE12-DP-318</u>
6.	Document Control	LANL-NNWSI-QAPP Section 6. TWS-MSTQA-QP-03 <u>TWS-QAS-QP-07</u> TWS-MSTQA-QP-10 TWS-MSTQA-QP-19
7.	Control of Purchased Items and Services	LANL-NNWSI-QAPP Section 7. TWS-QAS-QP-04.1
8.	Identification and Control of Items	LANL-NNWSI-QAPP Section 8.
9.	Control of Processes	LANL-NNWSI-QAPP Section 9. TWS-MSTQA-QP-14

* Procedures affected by this issue have been underscored.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
10. & 11.	Inspection and Test and Control	LANL-NNWSI-QAPP Sections 10 & 11. TWS-MSTQA-QP-11 TWS-MSTQA-QP-14
12.	Control of Measuring and Test Equipment	LANL-NNWSI-QAPP Section 12. TWS-QAS-QP-12.1, R1
13.	Handling, Storage, and Shipping	LANL-NNWSI-QAPP Section 13. TWS-QAS-QP-13.1
14.	Inspection, Test, and Operating Status	LANL-NNWSI-QAPP Section 14.
15.	Control of Nonconforming Items	LANL-NNWSI-QAPP Section 15. TWS-MSTQA-QP-16
16.	Corrective Action	LANL-NNWSI-QAPP Section 16. TWS-QAS-QP-21
17.	Quality Assurance Records	LANL-NNWSI-QAPP Section 17. TWS-MSTQA-QP-03 TWS-QAS-QP-17.1
18.	Audits	LANL-NNWSI-QAPP Section 18. <u>TWS-QAS-QP-17</u>

* Procedures affected by this issue have been underscored.

Change Request No. 017

Rev 0

Date November 3, 1987

LOS ALAMOS NATIONAL LABORATORY
NNWSI
CHANGE REQUEST

Procedure Number QP-07, R2

Change Requested Revise 2.0 Scope to read as follows:

2.0 SCOPE

The process shall apply to documents such as: Scientific Investigation Plans (SIP's), study plans, work plans, and technical reports, articles, papers, and abstracts prior to their publication which are required to fulfill LANL responsibilities to the NNWSI Project. The SIP's, study plans, and work plans will be reviewed annually and revised as necessary.

This QA requirement is not intended to infringe on the rights of individual Los Alamos researchers to submit scientific findings for publication in the open literature. Rather it is intended to ensure that reports of work identified or identifiable as supported by NNWSI have been reviewed for technical content and programmatic (policy) concerns.

Reason for Change QP-07, R2, revised to reflect WMPO Audit 87-01 response to SDR-003, Part 1.

Change Requested By

Henry Paul Hunt

Date

12/14/87

Reviewed By

Patricia M. Tillery

Date

12/14/87

Date

QAPL Approval

John A. Whit

Date

May 12, 1988

TPO Approval

R. J. Hunt

Date

12/15/1988

Effective Date

May 12, 1988

Date

5/12/88

LOS ALAMOS NATIONAL LABORATORY
NNWSI
CHANGE REQUEST

Change Request No. 032
Date May 13, 1988

Procedure No. TWS-QAS-OP-17, R0

Change Requested:

1. 4.1 Start the fourth paragraph with the following sentence: "Random surveys will be performed as needed to augment the audit program."
2. 4.2 Add the following information as the first bulleted phrase: "• All elements of the 18 requirements as listed in the QAPP (however, elements not specifically applicable to the work in progress may be waived for the purpose of the audit, and this decision must be documented in the audit report)."
3. 4.4 Replace the entire section with the following information: "The QAL, in conjunction with the audited organization, shall determine the corrective action, measures taken to prevent recurrence, and the implementation schedule for each corrective action, as a result of any audit findings. (See the audit finding report, CR Attachment 1.) Responses to audit findings must be made and sent to the lead auditor within 30 calendar days of the audit report date. The lead auditor, with the concurrence of the QAPL, must assess the responses for acceptability and shall concur with acceptable responses by signing the appropriate place in the audit finding report and returning it to the management of the audited organization."

A monthly status report will be issued by the QAS and sent to the affected organizations to report the status of open audit findings."

Reason for Change:

1. To bring this portion of this procedure into compliance with LANL QAPP 18.3.
2. To bring this portion of this procedure into compliance with LANL QAPP 18.2.7.
3. To bring this portion of this procedure into compliance with LANL QAPP 18.2.9.

Change Requested By Henry Paul Dunes Date 5/13/88
Reviewed By Patricia M. Tillery Date 5/13/88
QAPL Approval R. J. H. KAW Date 5/17/88
TPO Approval D. J. K. L. Date 5/17/88
Effective Date May 17, 1988 Date 5/17/88

Los Alamos

5, MS J521

Los Alamos National Laboratory
Los Alamos, New Mexico 87545

LOS ALAMOS NNWSI QUALITY ASSURANCE AUDIT FINDING REPORT

FINDING SECTION: (TO BE FILLED OUT BY AUDITOR)

Page 1 of 2

AUDITED ORGANIZATION: _____

AUDIT No.: _____ **FINDING No.:** _____ **AUDIT DATE(S):** _____

LEAD AUDITOR: _____ **REPORT DATE:** _____

AUDITORS: _____

UNUSUAL OCCURRENCE REPORT REQUIRED? ☐ YES ☐ NO (DOE ORDER 5000.3)

REQUIREMENT:

DEFICIENCY:

LEAD AUDITOR

DATE

FINDING CORRECTION HAS BEEN VERIFIED BY:

SURVEY No.: _____ **RE-AUDIT No.:** _____ **LEAD AUDITOR:** _____

COMMENTS: _____ **INITIALS**

LEAD AUDITOR

DATE

Los Alamos

5. MS J521

Los Alamos National Laboratory
Los Alamos, New Mexico 87545

LOS ALAMOS NNWSI QUALITY ASSURANCE
AUDIT FINDING REPORT

RESPONSE SECTION: (TO BE FILLED OUT BY AUDITEE. ATTACH ADDITIONAL NUMBERED PAGES WHEN NECESSARY)

Page 2 of 2

1. THE ROOT CAUSE(S) OF THIS DEFICIENCY IS (ARE):

2. THE STEPS TO BE TAKEN TO CORRECT THIS AUDIT DEFICIENCY ARE:

3. THE DATE BY WHICH THIS CORRECTIVE ACTION WILL BE COMPLETED: _____

4. THE STEPS TO BE TAKEN TO PREVENT RECURRENCE OF THIS DEFICIENCY ARE:

5. THE DATE BY WHICH STEPS TO PREVENT RECURRENCE WILL BE COMPLETED: _____

COMMENTS:

AUDIT RESPONSE ACCEPTANCE:

GROUP LEADER OR PRINCIPAL INVESTIGATOR

LEAD AUDITOR

DATE

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TWS-INC-DP-27, R0	Trace Element Determination by Plasma Emission Spectrometry
TWS-INC-DP-30, R0	Partial CO ₂ Atmospheric Control of Groundwater Chemistry
TWS-INC-DP-34, R0	Sulfide Electrode Measurements
TWS-INC-DP-35, R0	pH Measurements (CR006)
TWS-INC-DP-36, R0	Eh (Oxidation-Reduction Potential) Measurements
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TWS-INC-DP-42, R0	Measurement of Conductivity using the YSI Model 31 Conductivity Bridge
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Health, Safety, and Environmental Division DPs

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WX-Design Engineering Division DPs

TWS-WX-DP-59, R0	NNWSI Exploratory Shaft Facility Design Control Procedure
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CHANGE REQUESTS (CR)

CR No. 006	Modifies DP-35,R0 (CR in front of DP)
CR No. 025	Modifies TWS-INC-DP-35, R0 (CR in front of DP-35, R0)

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TWS-ESS-DP-06, R2	Operating Instructions for DV-502 Vacuum Evaporator Used in Carbon Coating Samples
TWS-ESS-DP-07, R2	Microprobe Operating Procedure
TWS-ESS-DP-10, R1	Procedure for Compressive Strength Tests
TWS-ESS-DP-16, R3	Siemens X-Ray Diffraction Procedure
TWS-ESS-DP-20, R1	Preparation of Fused Beads for Electron Microprobe Analysis of Rock Powders
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TWS-ESS-DP-52, R0	Fusing Using The Junior Orbit Shaker
TWS-ESS-DP-53, R0	Pulverizing Using the Spex 8500 Shatterbox
TWS-ESS-DP-54, R0	Crushing: Operation of 50 Ton Hydraulic Press
TWS-ESS-DP-55, R0	Rock Splitting: Operation of 50 Ton Hydraulic Press
TWS-ESS-DP-56, R1	Brinkman Automated Grinder Procedure
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TWS-ESS-DP-107, R0	Thermogravimetric and Differential Scanning Calorimetry Analyses
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TWS-ESS-DP-114, R0	Sample Collection Procedure for Rock Varnish Studies
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TWS-HSE12-DP-311, R0	Sample Preparation
TWS-HSE12-DP-312, R0	Particle Size Reduction of Geologic Media
TWS-HSE12-DP-313, R0	Calibration and Use of Centrifuges
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TWS-HSE12-DP-317, R0	Calibration and Use of Analytical and TOP-Loading Balances
TWS-HSE12-DP-318, R0	pH Measurement, Acid-Base Solution Standardization, and Total Alkalinity Procedure

CHANGE REQUESTS (CR)

CR No. 022	Modifies TWS-ESS-DP-28, R0 (CR in front of DP-28, R0)
CR No. 028	Modifies TWS-ESS-DP-04, R4 (CR in front of DP-04, R4)
CR No. 029	Modifies TWS-ESS-DP-114, R0 (CR in front of DP-114, R0)
CR No. 030	Modifies TWS-ESS-DP-53, R0 (CR in front of DP-53, R0)

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This index is prepared and maintained in accordance with
TWS-MSTQA-QP-02.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
1.	Organization	
2.	Quality Assurance Program	LANL-NNWSI-QAPP Sections 1 and 2. TWS-QAS-QP-02.1 TWS-MSTQA-QP-18
3.	Design Control	LANL-NNWSI-QAPP Section 3. TWS-WX-DP-59
4.	Procurement Document Control	LANL-NNWSI-QAPP Section 4. TWS-QAS-QP-04.1 TWS-QAS-QP-22
5.	Instructions, Procedures, and Drawings	LANL-NNWSI-QAPP Section 5. TWS-QAS-QP-03 TWS-QAS-QP-05.1 TWS-QAS-QP-05.2 TWS-MSTQA-QP-07 TWS-MSTQA-QP-09 TWS-MSTQA-QP-11 TWS-QAS-QP-12.1 TWS-QAS-QP-13.1 TWS-MSTQA-QP-14 TWS-MSTQA-QP-15 TWS-INC-WP-12 TWS-ESS-DP-01 TWS-INC-DP-02 TWS-ESS-DP-03 TWS-ESS-DP-04 TWS-CNC-DP-05

* Procedures affected by this issue have been underscored.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
5.	Instruction, Procedures, and Drawings (continued)	TWS-ESS-DP-06 TWS-ESS-DP-07 TWS-ESS-DP-09 TWS-ESS-DP-10 TWS-ESS-DP-11 TWS-CNC-DP-14 TWS-CNC-DP-15 TWS-ESS-DP-16 TWS-CNC-DP-17 TWS-CNC-DP-18 TWS-ESS-DP-20 TWS-CNC-DP-22 TWS-CNC-DP-23 TWS-ESS-DP-24 TWS-ESS-DP-25 TWS-INC-DP-26 TWS-INC-DP-27 TWS-ESS-DP-28 TWS-INC-DP-30 TWS-INC-DP-34 TWS-INC-DP-35 TWS-INC-DP-36 TWS-INC-DP-37 TWS-INC-DP-38 TWS-INC-DP-39 TWS-INC-DP-40 TWS-INC-DP-41 TWS-INC-DP-42 TWS-INC-DP-43 TWS-INC-DP-44 TWS-INC-DP-45 TWS-ESS-DP-50 TWS-ESS-DP-51 TWS-ESS-DP-52 <u>TWS-ESS-DP-53</u> TWS-ESS-DP-54 TWS-ESS-DP-55 TWS-ESS-DP-56 TWS-WX-DP-59 TWS-INC-DP-60 TWS-INC-DP-61 TWS-INC-DP-62 TWS-INC-DP-63 TWS-INC-DP-65 TWS-INC-DP-101 TWS-ESS-DP-102 TWS-ESS-DP-103 TWS-ESS-DP-105 TWS-ESS-DP-106

* Procedures affected by this issue have been underscored.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
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6.	Document Control	LANL-NNWSI-QAPP Section 6. TWS-MSTQA-QP-03 <u>TWS-QAS-QP-07</u> TWS-MSTQA-QP-10 TWS-MSTQA-QP-19
7.	Control of Purchased Items and Services	LANL-NNWSI-QAPP Section 7. TWS-QAS-QP-04.1
8.	Identification and Control of Items	LANL-NNWSI-QAPP Section 8.
9.	Control of Processes	LANL-NNWSI-QAPP Section 9. TWS-MSTQA-QP-14

* Procedures affected by this issue have been underscored.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
10. & 11.	Inspection and Test and Control	LANL-NNWSI-QAPP Sections 10 & 11. TWS-MSTQA-QP-11 TWS-MSTQA-QP-14
12.	Control of Measuring and Test Equipment	LANL-NNWSI-QAPP Section 12. TWS-QAS-QP-12.1, R1
13.	Handling, Storage, and Shipping	LANL-NNWSI-QAPP Section 13. TWS-QAS-QP-13.1
14.	Inspection, Test, and Operating Status	LANL-NNWSI-QAPP Section 14.
15.	Control of Nonconforming Items	LANL-NNWSI-QAPP Section 15. TWS-MSTQA-QP-16
16.	Corrective Action	LANL-NNWSI-QAPP Section 16. TWS-QAS-QP-21
17.	Quality Assurance Records	LANL-NNWSI-QAPP Section 17. TWS-MSTQA-QP-03 TWS-QAS-QP-17.1
18.	Audits	LANL-NNWSI-QAPP Section 18. <u>TWS-QAS-QP-17</u>

* Procedures affected by this issue have been underscored.

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NOTE: Please note that the table of contents now reflects new procedural alpha numeric designators. As each procedure is revised, the new designator will be used and ultimately the old designator will be completely removed.

<u>Present Designations</u>	<u>Title</u>	<u>New Designations</u>
TWS-QAS-QP-02.1, R0	NNWSI Personnel Selection, Training, and Certification	
TWS-QAS-QP-03, R7	Document Control Procedures	TWS-QAS-QP-06.X, R0
TWS-QAS-QP-04.1, R0	NNWSI Procurement Procedures	
TWS-QAS-QP-05.1, R1	Preparation of Quality (Administrative) Procedures	
TWS-QAS-QP-05.2, R0	Preparation of a Detailed Technical Procedure	
TWS-QAS-QP-07, R2	Procedure for Technical Review of Publications	TWS-QAS-QP-03.X, R0
TWS-QAS-QP-09, R0	Records Control Procedure	TWS-QAS-QP-17.1, R0
TWS-MSTQA-QP-10, R0	Document Control of the Ex- ploratory Shaft Test Plan	TWS-QAS-QP-03.X, R0
TWS-MSTQA-QP-11, R1	NNWSI Surveillance Procedure	TWS-QAS-QP-18.2, R0

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TWS-MSTQA-QP-14, R1	Research and Development (Experimental) Procedure	TWS-QAS-QP-03.X, R0
TWS-MSTQA-QP-16, R0	NNWSI Control of Nonconfor- mances	TWS-QAS-QP-15.X, R0
TWS-QAS-QP-17, R0 TWS-QAS-QP-17.1, R0	NNWSI QA Audits Records Management Procedure (Draft QAL Manuals)	TWS-QAS-QP-18.1, R0
TWS-MSTQA-QP-18, R1	Assignment of Quality Levels for Los Alamos NNWSI Activ- ities and Items	TWS-QAS-QP-02.X, R0
TWS-MSTQA-QP-19, R0 TWS-QAS-QP-21, R0 TWS-QAS-QP-22, R0	NNWSI Change Requests Corrective Action NNWSI Supplier Qualification	TWS-QAS-QP-06.X, R0 TWS-QAS-QP-16.X, R0 TWS-QAS-QP-07.X, R0

CHANGE REQUESTS (CR)

CR No. 008	Modifies QP-06, R2 (CR in front of QP)
CR No. 009	Modifies QP-18, R1 (CR in front of QP)
CR No. 012	Modifies QP alpha numeric code (CR in front of Volume I Table of Contents)
CR No. 013	Modifies QP-16, R0 (CR in front of QP)
CR No. 017	Modifies QP-07, R2 (CR in front of QP)
CR no. 032	Modifies QP-17, R0 (CR in front of QP)
CR No. 019	Modifies QP-14, R1 (CR in front of QP)
CR No. 024	Modifies QP-16, R1 (CR in front of QP-16, R1)
CR No. 026	Modifies QP-19,R0 (CR in front of QP)

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TWS-INC-WP-12, R0	Volcanic Hazard Investigations
TWS-INC-DP-02, R3	Quality Control in Counting Radioactive Nuclides
TWS-CNC-DP-05, R1	Sorption, Desorption Ratio Determinations of Geologic Materials by a Batch Method
TWS-CNC-DP-14, R1	Permeability Measurement Procedure
TWS-CNC-DP-15, R1	Crushed Rock Column Studies
TWS-CNC-DP-17, R1	Procedures for Samples Required In Their "Natural State"
TWS-CNC-DP-22, R2	Preparation of Microautoradiographs
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TWS-INC-DP-27, R0	Trace Element Determination by Plasma Emission Spectrometry
TWS-INC-DP-30, R0	Partial CO ₂ Atmospheric Control of Groundwater Chemistry
TWS-INC-DP-34, R0	Sulfide Electrode Measurements
TWS-INC-DP-35, R0	pH Measurements (CR006)
TWS-INC-DP-36, R0	Eh (Oxidation-Reduction Potential) Measurements
TWS-INC-DP-37, R0	Anaerobic Field Filtering Apparatus
TWS-INC-DP-38, R0	Determination of Detergent Concentrations, Anionic
TWS-INC-DP-39, R0	Dissolved Oxygen Determinations
TWS-INC-DP-40, R0	Chloride Ion, Dissolved Electrode Method
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TWS-INC-DP-65, R0	Procedure for Volcanism Field Studies

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TWS-HSE5-DP-213, R0	Procedure For The Calibration and Use of SKC Personal Sampling Pumps
TWS-HSE5-DP-214, R0	Procedures For The Calibration and Use of Alpha-1 Personal Sampling Pumps
TWS-HSE5-DP-215, R0	Procedures For The Calibration of The Singer Dry Gas Meter

WX-Design Engineering Division DPs

TWS-WX-DP-59, R0	NNWSI Exploratory Shaft Facility Design Control Procedure
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CHANGE REQUESTS (CR)

CR No. 006	Modifies DP-35,R0 (CR in front of DP)
CR No. 025	Modifies TWS-INC-DP-35, R0 (CR in front of DP-35, R0)

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TWS-ESS-DP-04, R4	Thin Section Preparation Procedure
TWS-ESS-DP-06, R2	Operating Instructions for DV-502 Vacuum Evaporator Used in Carbon Coating Samples
TWS-ESS-DP-07, R2	Microprobe Operating Procedure
TWS-ESS-DP-10, R1	Procedure for Compressive Strength Tests
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TWS-ESS-DP-50, R0	Sputter Coater Operating Procedure for Gold Coating Samples
TWS-ESS-DP-51, R0	Mettler H80 Operation Procedure (X-Ray Fluorescence Analysis Sample Weighing Procedure)
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TWS-ESS-DP-53, R0	Pulverizing Using the Spex 8500 Shatterbox
TWS-ESS-DP-54, R0	Crushing: Operation of 50 Ton Hydraulic Press
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TWS-ESS-DP-105, R0	Thermal Calibration Procedure
TWS-ESS-DP-106, R0	Philips X-ray Diffraction Procedure
TWS-ESS-DP-107, R0	Thermogravimetric and Differential Scanning Calorimetry Analyses
TWS-ESS-DP-110, R0	Zeolite Purification/Separation Procedure
TWS-ESS-DP-111, R0	Procedure for X-ray Fluorescence Analysis

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TWS-ESS-DP-114, R0	Sample Collection Procedure for Rock Varnish Studies
TWS-ESS-DP-115, R0	Vaisala HMI-32 Humidity and Temperature Probe Procedure

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TWS-HSE12-DP-310, R0	Calibration and Use of the Phototachometer
TWS-HSE12-DP-311, R0	Sample Preparation
TWS-HSE12-DP-312, R0	Particle Size Reduction of Geologic Media
TWS-HSE12-DP-313, R0	Calibration and Use of Centrifuges
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TWS-HSE12-DP-317, R0	Calibration and Use of Analytical and TOP-Loading Balances
TWS-HSE12-DP-318, R0	pH Measurement, Acid-Base Solution Standardization, and Total Alkalinity Procedure

CHANGE REQUESTS (CR)

CR No. 022	Modifies TWS-ESS-DP-28, R0 (CR in front of DP-28, R0)
CR No. 028	Modifies TWS-ESS-DP-04, R4 (CR in front of DP-04, R4)
CR No. 029	Modifies TWS-ESS-DP-114, R0 (CR in front of DP-114, R0)
CR No. 030	Modifies TWS-ESS-DP-53, R0 (CR in front of DP-53, R0)

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5.	Instructions, Procedures, and Drawings	LANL-NNWSI-QAPP Section 5. TWS-QAS-QP-03 TWS-QAS-QP-05.1 TWS-QAS-QP-05.2 TWS-MSTQA-QP-07 TWS-MSTQA-QP-09 TWS-MSTQA-QP-11 TWS-QAS-QP-12.1 TWS-QAS-QP-13.1 TWS-MSTQA-QP-14 TWS-MSTQA-QP-15 TWS-INC-WP-12 TWS-ESS-DP-01 TWS-INC-DP-02 TWS-ESS-DP-03 TWS-ESS-DP-04 TWS-CNC-DP-05

* Procedures affected by this issue have been underscored.

Section No.	Title	NNWSI Procedure Reference
5.	Instruction, Procedures, and Drawings (continued)	TWS-ESS-DP-06 TWS-ESS-DP-07 TWS-ESS-DP-09 TWS-ESS-DP-10 TWS-ESS-DP-11 TWS-CNC-DP-14 TWS-CNC-DP-15 TWS-ESS-DP-16 TWS-CNC-DP-17 TWS-CNC-DP-18 TWS-ESS-DP-20 TWS-CNC-DP-22 TWS-CNC-DP-23 TWS-ESS-DP-24 TWS-ESS-DP-25 TWS-INC-DP-26 TWS-INC-DP-27 TWS-ESS-DP-28 TWS-INC-DP-30 TWS-INC-DP-34 TWS-INC-DP-35 TWS-INC-DP-36 TWS-INC-DP-37 TWS-INC-DP-38 TWS-INC-DP-39 TWS-INC-DP-40 TWS-INC-DP-41 TWS-INC-DP-42 TWS-INC-DP-43 TWS-INC-DP-44 TWS-INC-DP-45 TWS-ESS-DP-50 TWS-ESS-DP-51 TWS-ESS-DP-52 <u>TWS-ESS-DP-53</u> TWS-ESS-DP-54 TWS-ESS-DP-55 TWS-ESS-DP-56 TWS-WX-DP-59 TWS-INC-DP-60 TWS-INC-DP-61 TWS-INC-DP-62 TWS-INC-DP-63 TWS-INC-DP-65 TWS-INC-DP-101 TWS-ESS-DP-102 TWS-ESS-DP-103 TWS-ESS-DP-105 TWS-ESS-DP-106

* Procedures affected by this issue have been underscored.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
5.	Instruction, Procedures, and Drawings (concluded)	TWS-ESS-DP-107 TWS-ESS-DP-110 TWS-ESS-DP-111 TWS-ESS-DP-112 TWS-ESS-DP-113 TWS-INC-DP-114 TWS-ESS-DP-115 TWS-HSE5-DP-201 TWS-HSE5-DP-202 TWS-HSE-5-DP-206 TWS-HSE5-DP-211 TWS-HSE5-DP-212 TWS-HSE5-DP-213 TWS-HSE5-DP-214 TWS-HSE5-DP-215 TWS-HSE12-DP-301 <u>TWS-HSE12-DP-307</u> <u>TWS-HSE12-DP-310</u> <u>TWS-HSE12-DP-311</u> <u>TWS-HSE12-DP-312</u> <u>TWS-HSE12-DP-313</u> <u>TWS-HSE12-DP-316</u> <u>TWS-HSE12-DP-317</u> <u>TWS-HSE12-DP-318</u>
6.	Document Control	LANL-NNWSI-QAPP Section 6. TWS-MSTQA-QP-03 <u>TWS-QAS-QP-07</u> TWS-MSTQA-QP-10 TWS-MSTQA-QP-19
7.	Control of Purchased Items and Services	LANL-NNWSI-QAPP Section 7. TWS-QAS-QP-04.1
8.	Identification and Control of Items	LANL-NNWSI-QAPP Section 8.
9.	Control of Processes	LANL-NNWSI-QAPP Section 9. TWS-MSTQA-QP-14

* Procedures affected by this issue have been underscored.

<u>Section No.</u>	<u>Title</u>	<u>NNWSI Procedure Reference</u>
10. & 11.	Inspection and Test and Control	LANL-NNWSI-QAPP Sections 10 & 11. TWS-MSTQA-QP-11 TWS-MSTQA-QP-14
12.	Control of Measuring and Test Equipment	LANL-NNWSI-QAPP Section 12. TWS-QAS-QP-12.1, R1
13.	Handling, Storage, and Shipping	LANL-NNWSI-QAPP Section 13. TWS-QAS-QP-13.1
14.	Inspection, Test, and Operating Status	LANL-NNWSI-QAPP Section 14.
15.	Control of Nonconforming Items	LANL-NNWSI-QAPP Section 15. TWS-MSTQA-QP-16
16.	Corrective Action	LANL-NNWSI-QAPP Section 16. TWS-QAS-QP-21
17.	Quality Assurance Records	LANL-NNWSI-QAPP Section 17. TWS-MSTQA-QP-03 TWS-QAS-QP-17.1
18.	Audits	LANL-NNWSI-QAPP Section 18. <u>TWS-QAS-QP-17</u>

* Procedures affected by this issue have been underscored.

LOS ALAMOS NATIONAL LABORATORY
NNWSI
CHANGE REQUEST

Change Request No. 030
Date 12 May 1988

Procedure No. TWS-ESS-DP-53, R0

Change Requested:

Replace Section 3.6.1 with the following:

For samples being prepared for trace element analysis, a "dry cleaning" run MUST be made using all equipment and containers that will be used to process the final sample. This step alleviates the possibility of contamination as a result of a previously ground rock. The "dry cleaning" run may be omitted when using this procedure for preparation of XRD samples.

Reason for Change:

The dry cleaning run was included primarily for preparation of samples requiring trace element analyses. For x-ray diffraction, however, the thorough scrubbing and washing step is more than adequate to remove any detectable contamination.

Change Requested By Steve J. Chapin Date May 12th 1988
Reviewed By David L. Bush Date 5/12/88
QAPL Approval Kevin A. West Date May 23, 1988
TPO Approval J. T. Alley Date 5-26-88
Effective Date May 26, 1988

SAMPLE IDENTIFICATION AND CONTROL

Effective Date: 4/29/88

Edward H. Essington 3/22/88
Edward H. Essington Date
Preparer

W.L. Polzer 3/23/88
W.L. Polzer Date
Technical Reviewer

K. West 4/29/88
K. West Date
Acting QAPL

D.T. Oakley 4/29/88
D.T. Oakley Date
TPO

SAMPLE IDENTIFICATION AND CONTROL

1.0 PURPOSE

This procedure describes the requirements for identification (labeling) and control (receiving, storing, and transferring) of solid and solution materials used in the C-well Reactive Tracer Study for the Nevada Nuclear Waste Storage Investigation (NNWSI) project.

2.0 SCOPE

This procedure is to be followed in labeling, receiving, storing, and transferring solid or liquid geologic materials used in tracer experiments or in transferring the materials to the control of a group other than project laboratory personnel (Eg. CLS-1) for analysis. These materials will be used in generation of Quality Level I data to be used in site characterization.

3.0 PRINCIPLE (NA)

4.0 DEFINITIONS (NA)

5.0 RESPONSIBILITIES

It is the responsibility of the project Principal Investigator (PI) to assure correct implementation of this procedure. The project PI may designate a Laboratory Supervisor (LS) to conduct this procedure and to certify and supervise other personnel in the use of this procedure; that designation is documented by memorandum to the group resident file. The scientific investigator (SI) may advise or recommend specific activities in the conduct of this procedure (Eg. sample labeling and acceptance).

6.0 PROCEDURE

6.1 Sample Identification

A sample identification number is assigned to each individual sample so that the sample or any of its derivatives can be traced through processing back to its origin. Any cross-reference needed to complete the trail and any information relative to the origin and handling of the sample will be documented in the scientific notebook, sample logbook, or resident file as appropriate. Pertinent data about the samples and information not easily accessed in official documents will also be recorded in the scientific notebook.

6.2 Receiving Samples

Refer to TWS-HSE12-DP-301 for instructions on receiving, accepting, and storing samples that were collected and shipped

to the HSE-12 laboratory by project personnel. Samples received at the laboratory that were not collected by HSE-12 project personnel are inspected by the LS for damage, integrity, labeling, and accountability. A copy of the shipping manifest or a memo verifying receipt of shipped samples shall be placed in the resident file. Sample identification and pertinent information provided with the sample are documented in the sample logbook or scientific notebook, as appropriate. If the shipment is unacceptable or incomplete, inquiries are made to ascertain the source of the problem and any resolution that can be expected. If samples have been adulterated, as determined by the PI, LS or SI, they will not be used in tracer experiments unless they are subsequently certified for such use. All such information and decisions about the samples are documented in the sample logbook or scientific notebook.

6.3 Sample Storage

6.3.1 Well-Water Samples

Well-water samples contained in 55-gal drums are stored in an area protected against unauthorized entry. Subsamples withdrawn from the drums are stored in the same manner or at 4°C in a refrigerator dedicated to NNWSI sample storage. Withdrawals are noted in the sample logbook; entries are initialed and dated by the person making the withdrawal.

6.3.2 Solid Samples

Solid samples are stored dry in appropriate containers (generally polyethylene or polypropylene) labeled with sample identity permanently scribed on the side of the container. To prevent inadvertent loss, destruction or contamination of samples, the containers are stored in a cabinet dedicated to NNWSI sample storage. Withdrawals are noted in the sample logbook; entries are initialed and dated by the person making the withdrawal.

6.3.3 Prepared Samples

Aliquots of solid or liquid samples prepared for characterization and aliquots drawn from the tracer sorption experiments will need a short duration storage before transfer for analysis. Those samples will be prepared and stored in a manner consistent with the analyses needed. Storage of liquid samples or samples that cannot be dried will be at 4°C in a refrigerator dedicated to NNWSI sample storage.

6.4 Labeling of Experimental Samples

Any sample created during the conduct of an experiment will be labeled with a unique identification number; that number is cross-referenced to the parent sample so that the created sample can be traced to its origin. That information will be recorded in the scientific notebook.

6.5 Transfer of Samples

A chain of custody must be maintained for transfer of samples (liquid or solid) to another group for analysis (Eg. CLS-1). This chain of custody shall be maintained by issuing a memo with a copy to the resident file. The memo will identify the originator and recipient of the samples, date of transfer, amount of each sample, and sample identification number(s). If samples are sent outside LANL for analysis, shipping records shall be maintained in the resident file. Details of the transfer will be recorded in the scientific notebook and sample logbook as appropriate. If needed, a form documenting sample identification and specifics relative to the required analyses will be prepared and will accompany the samples sent for analysis (see attachment 1 for an example).

6.6 Sample Preparation

The guiding detailed procedure will specify any required special sample treatment (Eg. acidification). The PI, LS, or SI may also determine that special conditions are required to preserve the sample integrity; these conditions shall be noted on the sample container, in the sample logbook, and in scientific notebook as appropriate. Prior to using any sample, the PI, LS, or SI shall evaluate the condition of the samples and may reject them for a specific analysis.

7.0 QUALITY ASSURANCE

7.1 Documentation

Details of any sample custody transfer shall be documented in the scientific notebook, sample logbook, and by memo; copy of the memo shall be placed in the resident file. Each sample shall be uniquely identified and labeled so that it is traceable through its preparation and analysis and can ultimately be traced to its origin. Certification and training shall be documented according to LANL NNWSI project certification procedures. Documentation generated during conduct of this procedure shall conform to LANL NNWSI project requirements for documentation. This procedure shall be issued.

revised, and controlled in accordance with LANL NNWSI project document control procedures.

7.2 Personnel and Training

Project personnel conducting any task in this procedure must be approved and certified by the LS. Project personnel directed to use this procedure will read the procedure and will demonstrate a working understanding of the operational steps to the satisfaction of the LS.

7.3 Handling, Shipping and Storage Requirements. (NA)

7.4 Acceptance/Rejection Criteria

See Sections 6.2 and 6.6.

8.0 REFERENCES

1. TWS-HSE12-DP-301: Field Collection of Experimental Materials.

9.0 ATTACHMENTS

1. Attachment 1: Standard Analytical Request Form for LANL CLS-1.

ATTACHMENT 1

ANALYSIS REQUEST

INFORMATION SUPPLIED BY SUBMITTER

Is Sample Radioactive? Yes or No _____ If yes, Radioactive Components are
_____ and Contact β - γ Radiation is _____ mR/hr.

Group _____ Sample ID _____ Date _____
20-character-limit, please

Form B Code _____ Submitter _____ Tel. _____

Does Quality Assurance Apply? Yes or No _____

Sample Description and Special Analysis Conditions _____

Sample Handling Safety Precaution, Yes or No _____ If Yes, it is _____

INFORMATION SUPPLIED BY SUBMITTER AND REGARDING COMPONENTS TO BE DETERMINED

Submitter		CLS-1		Submitter		CLS-1	
Component	Expected Level	Section	Sample Quantity	Component	Expected Level	Section	Sample Quantity

INFORMATION SUPPLIED BY CLS-1

Sol No. _____ Acct No. _____ Proj No. _____ SS ID _____

Rct. Value _____ Bulk Wt _____ % Enr _____

Are results given on back, Yes or No _____

CALIBRATION AND USE OF THE PHOTOTACHOMETER

Effective Date: 5/2/88

Edward H. Essington
Edward H. Essington
Preparer

3/22/88
Date

W.L. Polzer
W.L. Polzer
Technical Reviewer

3/23/88
Date

K. West
K. West
Acting QAFL

4/28/88
Date

D.T. Oakley
D.T. Oakley
TPO

5/2/88
Date

CALIBRATION AND USE OF THE PHOTOTACHOMETER

1.0 PURPOSE

This procedure describes the calibration and use of a phototachometer that will be utilized to establish and calibrate rotating shaft speed (rpm) of various types of equipment (Eg. centrifuges, mixers) used in the C-Well Reactive Tracer Study for the Nevada Nuclear Waste Storage Investigation (NNWSI) project.

2.0 SCOPE

This procedure is to be followed in the use and calibration of photo-sensitive tachometers such as the Ametek C-891. Calibrations and measurements made with the phototachometer relate to data used in site characterization; therefore, the activity is Quality Level I.

3.0 PRINCIPLE (NA)

4.0 DEFINITIONS (NA)

5.0 RESPONSIBILITIES

It is the responsibility of the project Principal Investigator (PI) to assure correct implementation of this procedure. The project PI may designate and certify a Laboratory Supervisor (LS) to conduct the procedure and to certify and supervise personnel in the use of this procedure; that designation will be documented by memorandum to the group resident file. Either the PI or LS will direct activities associated with this procedure and delegate to certified project personnel under the supervision of either the PI or LS any of the tasks in the conduct of this procedure.

6.0 PROCEDURE

6.1 Calibration of Phototachometer.

The phototachometer is to be calibrated against a known source before each use and at the discretion of the user or as specified by the LS. To perform a calibration first examine the phototachometer to see that it is in good working order and that the batteries have proper charge as determined by the built-in battery tester. Follow the manufacturer's instructions on calibrating against a fluorescent light source (see Attachment 1). If the value read for the light source is not acceptable (deviates more than 5% from the true value) repeat the calibration procedure from the beginning. If the calibration is still not acceptable, consult the LS for correction of the problem. Calibration of the phototachometer

may be performed by a calibration service group and will be noted in the scientific notebook.

6.2 Use of Phototachometer

Service and calibrate the phototachometer according to the Sec. 6.1. To use the phototachometer follow the instructions on the back of the unit (see Attachment 1).

7.0 QUALITY ASSURANCE

7.1 Documentation

Document all calibrations and measurements as required by the guiding detailed procedure in the scientific notebook. This procedure shall be issued, revised, and controlled in accordance with LANL NNWSI document control procedures.

7.2 Calibration

The phototachometer shall be user calibrated before each use and more often at the discretion of the user or as specified by the LS. A sticker displaying "OPERATOR CALIBRATED" shall be placed on the phototachometer according to the LANL NNWSI implementing procedures.

7.3 Personnel Certification and Training

Project personnel conducting any task in support of this procedure must be approved and certified by the PI or LS. Certifications shall be documented according to LANL NNWSI personnel certification procedures. Project personnel directed to use this procedure will read the procedure and will demonstrate an understanding of the operational steps to the satisfaction of the LS.

7.4 Handling, Shipping, and Storage Requirements (NA)

8.0 ATTACHMENTS

1. Operator's instructions for the Ametek C-981 Phototachometer.

MODEL C-891

OPERATING DIRECTIONS

FIELD CALIBRATION is on 0-10,000

scale. Direct probe near any fluorescent light, press the "Press to read" button and adjust "calibrate" slot so meter reads "7200" (6000 on 50 cycles). This brings other scales into calibration also.

PREPARATION FOR PHOTO SENSING: Rotating shafts and discs or vibrating objects may be prepared to obtain maximum dark-to-light contrast in one of the following ways:

Apply one of the adhesive marking labels to shaft or object of speed reading so light from probe will "see" a good contrast of reflection when shaft rotates. Clean oil from shaft before applying.

Three methods are used:

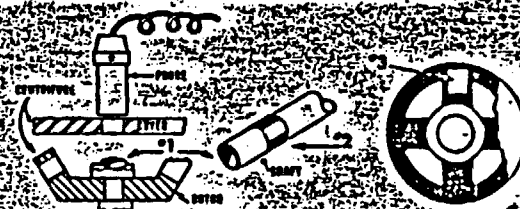
(1) Round bright label (#11-21) half black, is applied to the end of centrifuge spindles or other shafts.

(2) Long narrow label (#11-20) is wrapped around the shaft leaving the bright end exposed. Label may need to be cut shorter, or two put end to end, depending on diameter of shaft.

(3) Providing the shaft is clean, good results may be had by applying a short piece, $\frac{1}{4}$ to 1 inch long, to the shaft or wheel of the Scotchlite furnished. Leave at least $\frac{1}{2}$ of shaft diameter uncovered. The Scotchlite reflects so much better than the metal surface that a good signal is obtained.

Use bone charcoal black paint for permanent markings. In emergency, eye mascara can be used for marking.

TAKING READINGS: Hold the pickup within 12 inches and perpendicular to the mark in the rotating object. Push the "Press to read" button and read the speed.



SLOW SPEEDS: 10-100 RPM may be read by making a special marking with 10 equally separated lines. Speed is read on 0-1000 scale.

MAINTENANCE: Probe lamp is G.E. 222X, rated 2.25 V, but operated at 1.5 V. for long life. To change, remove two Allen screws from large end of probe. Socket can now be removed.

Lamp uses 1.5 V. "D" size cell. Printed circuit uses standard alkaline 9-volt transistor battery. Change batteries by removing four bottom screws of base. Meter should read well below line on Battery Test before changing battery.

Should service become necessary, pack carefully and enclose description or fault. Return prepaid by U.P.S. or express (not mail) to:

AMETEK

MANSFIELD & GREEN DIVISION
8800 SOMERSET DRIVE
LARGO, FLORIDA 33543

Made in U.S.A.

SAMPLE PREPARATION

Effective Date: 5/2/88

Edward H. Essington
E. H. Essington
Preparer

3/17/88
Date

Kimberly M. Thomas
K. Thomas
Reviewer

3/17/88
Date

Janet A. West
K. West
Acting QAPL

4/29/88
Date

D. T. Oakley
D. T. Oakley
TPO

5/2/88
Date

SAMPLE PREPARATION

1.0 PURPOSE

This procedure describes methods of preparing finely divided materials from bulk rock samples or from representative mineral specimens. The procedure also describes the preparation of field-collected well-water samples. The prepared materials are used in tracer investigations for the C-Well Reactive Tracer Study, which is part of the Nevada Nuclear Waste Storage Investigation (NNWSI) project.

2.0 SCOPE

This procedure applies to preparation and purification of solid geologic materials and well-water samples. Samples prepared under the direction of this procedure are used in generation of data to be used in site characterization. The activity is Quality Level I.

3.0 PRINCIPLE (NA)

4.0 DEFINITIONS (NA)

5.0 RESPONSIBILITIES

It is the responsibility of the project Principal Investigator (PI) to assure correct implementation of this procedure. The project PI may designate and certify a Laboratory Supervisor (LS) to conduct this procedure and to certify and supervise project personnel to conduct this procedure; that designation will be documented by memorandum to the group resident file. The project PI may designate, by memo to the resident file, a scientist qualified to examine a mineral specimen and to identify and assess the purity of the mineral specimen. This procedure, or portions thereof, will be conducted according to the recommendations of the scientific investigator (SI).

6.0 PROCEDURE

This procedure is designed to prepare large amounts of homogeneous material and filtered well water (Eg. Kg and gal) for use in batch and column reactive tracer evaluations. The steps described in this procedure will be the basis for preparation of solid material and well-water samples; however, the exact combination of preparation steps used will depend on the nature and purity of the original material or well water and on experimental needs that will be specified by the LS or SI.

6.1 Equipment

6.1.1 Dissecting Scope

Dissecting scope (up to 40X) and external lighting source, and magnifying hand lens (up to 20X).

6.1.2 Sonifier

Branson Model 250 sonifier, with converter and horn, capable of processing suspensions in 100-mL glass beaker or larger.

6.1.3 Drying Oven

Drying oven, capable of maintaining 105°C , $\pm 5^{\circ}\text{C}$. Temperature will be monitored with a commercial grade laboratory thermometer (calibrated by manufacturer).

6.1.4 Sieves

Sieves, stainless steel, 500- μm screen opening, ASTM or equivalent specifications.

6.1.5 Solution Pump

Peristaltic or tubing type pump, capable of generating up to 25 cm (Hg) vacuum and 20 psi for moving water from 55-gal drums through a filter apparatus.

6.1.6 Micropore Filtration Apparatus

Filter holder, stainless steel, for micropore filter membranes capable of operating within pressure limits of peristaltic solution pump.

6.1.7 Vacuum Filter

Porous ceramic or high-fired aluminum oxide vacuum filtration device, capable of holding 1-bar tension.

6.2 Types of Geologic Materials

Bulk rock material includes whole rock samples, core samples, core cuttings, fracture minerals, fracture coatings, and inclusions in host bulk rock. Representative minerals are primarily single mineral crystals or clusters. The identity and purity of the representative mineral is to be certified by the

supplier or by a qualified person after examination or the identity and purity may be determined by X-ray diffraction analysis. Well water (considered as a geologic material for purposes of this procedure) is collected from specific water-bearing formations accessed by a well bore. The choice of procedures for preparation and purification of the materials for experimental use will be defined by the SI or LS.

6.3 Solid Material Treatment

6.3.1 Preliminary Washing

Place pieces of consolidated material or mineral clumps on a sieve of sufficiently small mesh (Eg. 100- μ m mesh opening) to trap those particles that are to remain as part of the sample. Rinse the material with distilled water and use a clean non-metallic bristle brush to scrub off the entire surface of the sample. Clean the sample of all visible traces of drilling mud or other contaminants. Air-dry the samples in a clean area; protect the samples from dust and contamination by covering with a lint-free towel. Store the dry sample (sample storage is specified in Sec. 7.4). Further sample drying will be recommended by the LS or SI or will be described in individual experimental procedures.

6.3.2 Washing by Sonification

Wash friable or poorly consolidated materials by sonification and sedimentation methods to remove finely divided impurities. Loosen adhering contaminant particles from the samples by sonification using the Branson Model 250 sonifier. Place about 200 grams of sample in a 600-mL beaker. Fill the beaker with distilled water so that the water level is about 3 cm over the top of the solids. Set the output level dial to 6, the timer to one minute, the duty cycle to 50%, and then sonify. When machine has shut off stir the sample thoroughly and repeat sonification for another minute.

After sonification transfer sample suspension to a 2000-mL plastic bottle using distilled water. Fill the bottle with distilled water until the water is 15 cm above the top of any settled sample. Mix the solid and liquid by inverting the bottle several times. Let the sample settle undisturbed for two hours or more. Slowly and without agitation of the

solids, siphon off the top 10 cm of water into a clean container of appropriate size. Siphon rate should not exceed 80 mL/min. If necessary, save the decanted solution for retrieval of fines; disposal of the fines will be directed by the LS or SI. Repeat the washing procedure until water is nearly clear. Dewater the suspension by vacuum filtration using a porous ceramic cup and vacuum source (Sec. 6.8). Wash sample at least two more times using bulk solution (Eg. J-13 water) if conditioning is required (See Sec. 6.6.1). Dewater and air dry the sample before storage or use.

6.3.3 Visual Sorting

Carefully separate individual mineral crystals or crystal clusters from the mineral mass using a chisel and hammer. This is best done in a plastic bag to avoid loss of sample material and to protect operator. Examine a small amount of the original or separated material under a magnifying hand lens or dissecting scope. Remove adhering impurities, or pick out clean crystals of desired mineral. If impurities are still present, some may be removed by washing with distilled water (see Sec. 6.3.1), or the impurities can be chipped away. Visually recheck for removal of impurities; acceptable samples are air dried before storage or use. Examples of minerals that may be treated by this method are quartz, albite, biotite, and possible hematite.

6.4 Particle-Size Reduction and Sieving

6.4.1 Particle-Size Reduction

Particle-size reduction is accomplished by crushing, grinding, and sieving the material so that the particle size range required for specific experiments can be obtained. Both bulk rock samples and representative mineral material may be treated in this manner. The method of choice for grinding experimental materials is described in TWS-HSE12-DP-312. As an alternative, the grinding steps in TWS-INC-DP-63 may be used.

6.4.2 Sieving

Dry sieve the ground sample material to a particle diameter not greater than 500 μm ; material not

passing the 500- μ m sieve will be further ground and sieved so that the resultant fine material is representative of the original sample material (refer to TWS-HSE12-DP-312). Certain experiments may require a more finely divided material or the use of wet-sieving; those requirements will be specified by the LS or SI (Eg. for preparation of the sample for X-Ray diffraction see TWS-ESS-DP-01) or will be described as part of the experimental design. Sieves are prepared by brushing, rinsing with distilled water, drying, and visually inspecting for cleanliness and integrity before any sieving is conducted. A good hand lens (Eg. 10X to 20X) is used to inspect the sieve. The sieve must not be used if the screen mesh has tears, any of the mesh holes are deformed, or the sieve is otherwise damaged so that improper sieving can occur. Collect the sieved materials for storage or use.

Wet sieving is conducted in the same manner only using distilled water; the sample is dewatered as described in Sec. 6.9 and then air dried before storage or use.

6.5 Purification

6.5.1 Sieving

Sieving or a combination of particle-size reduction and sieving can be used to purify some representative minerals if the impurity is of sufficiently different size than the desired mineral. Refer to Sec. 6.4.2 for procedural details.

6.5.2 Magnetic Separation

An electromagnet (Eg. Frantz Magnetic Separator) or strong permanent magnet is used to separate minerals or impurities that have magnetic susceptibilities different from the base material; finely divided hematite, magnetite, and goethite can easily be separated from quartz or feldspars by this method. The procedure is described in TWS-HSE12-DP-322).

6.5.3 Sedimentation

Sedimentation is used to purify finely divided materials (Eg. clay and zeolite). Clinoptilolite is

separated from smectite impurities following TWS-ESS-DP-110. The clays are separated from other materials (Eg. quartz and feldspars) following TWS-ESS-DP-25. The sedimentation parameters will be specified by the LS or SI depending on the nature of the material required for study.

6.5.4 Water Filtration

Bulk samples of water (Eg. J-13 well water) are filtered as a standard method of preparation before use in tracer experiments. Carefully assemble the filter holder (previously washed with distilled water) with a 0.45- μ m membrane filter (142 mm diameter) over a 0.05- μ m (142-mm diameter) membrane microfilter. Use a solution pump to transfer bulk water sample from the original container through the filter assembly into an appropriate receiving container (Eg. 2.5-gal cubitainer). Prior to use, wash the tubing and the receiving container with 0.1N HNO_3 followed by a thorough washing with distilled water; drain all excess water from container.

6.6 Material Conditioning

Chemical treatment of the solid materials may be necessary to condition them for use in specific experiments. For example, a commonly used conditioning treatment involves equilibrating the solids with the bulk solution (Eg. J-13 water) so that minimal system changes will occur upon the introduction of the tracer. Another typical treatment is saturating the exchange sites with a single ion, converting the material to a monoionic system.

6.6.1 Equilibration with Bulk Solution

Substitute the desired bulk solution for the distilled water in the steps for washing the solid materials described in Sec. 6.3 or 6.5. Monitor successive reacted solutions for key chemical constituents. Conditioning is complete when changes in those constituents from successive washings is less than 5%.

6.6.2 Exchange Site Saturation

Suspend the solid to be conditioned in a solution of the saturating ion (Eg. 0.1M), in a suitable container. Thoroughly mix for a period of time prescribed by the LS or SI. Remove saturating

solution by decanting after centrifugation (see TWS-HSE12-DP-313 for use and calibration of the centrifuge) or sedimentation, or by vacuum filtration (described in Sec. 6.9). Repeat saturation and solution removal steps at least three times. Wash excess saturating ion from the solids with distilled water or other specified solution using the same procedure. Monitor the washings until the concentration of the saturating ion in the washings is less than 1% of the ion concentration in the initial saturating solution. Dewater sample as above and air dry the treated solid material prior to use or storage.

6.7 Drying of Solids

Solid samples are dried in one of the following ways; air dry, 60°C oven dry, 105°C oven dry or as otherwise specified by the LS, SI, or guiding procedure.

6.7.1 Air Dry Solids

Spread the moist sample in a thin layer in a clean container (Eg. porcelain or glass tray), cover the sample with a lint-free towel, and place the container in a clean area of the laboratory where airborne dust is minimal. Periodically mix the sample until it appears to be dry. For small samples, weigh the entire container, allow for additional air drying time of at least 2 hours, and reweigh the container. Drying is complete when successive weights differ by less than 1% of the estimated sample weight. Drying may be done in the original container if sufficient air exchange can be provided and the container can be weighed to the appropriate tolerance. See TWS-HSE12-DP-317 for instructions on use and calibration of balances.

6.7.2 Drying at 60°C or 105°C

Spread moist sample evenly in a tray, cover the tray with aluminum foil so that the foil does not touch the sample. Poke several holes (Approx. 1/2 cm diameter) in the foil as vents. Place the tray in a drying oven set at the appropriate temperature as monitored by a commercial quality thermometer. Check for completeness of drying in the same manner as in Sec. 6.7.1. Drying can be done in the original container if sufficient air exchange is available, if

the container can stand the drying temperature without failure, and the container can be weighed to the appropriate tolerance.

6.8 Homogenization

Homogenize the dry sample before use. The need for additional homogenization and the specific procedure to use will be specified by the LS or SI and will depend on the combination of steps in the preparation of the sample. For most samples (Eg. samples that can fit entirely into one container), successively inverting the sample container with a rolling action for about 30 sec will mix the contents sufficiently. For large samples that cannot be contained in one container repeated quartering or riffing may be necessary and will be specified by the LS or SI.

6.9 Dewatering

Certain of the above steps generate suspensions that must be dewatered before the sample can be dried or used. Centrifuge (TWS-HSE12-DP-313) the suspension or allow the suspension to settle until particles greater than a specified size have settled or until the supernate is clear; decant as much water as possible without losing solids. Insert a porous cup vacuum filter into the remaining suspension so that the cup is entirely covered with suspension. Attach the cup to a vacuum flask and the flask to a vacuum source. High vacuum is not required for this operation. Apply vacuum to the flask and cup until no free water remains in the sample. Back flush the cup by applying a pressure to the interior of the cup with a squeeze bottle of distilled water to release the solids from the exterior of the cup. Air dry the sample prior to use or storage.

6.10 Determination of Purity

Mineral materials that have not been certified as to purity and identity or that have been altered by treatment may be characterized for their main components by a qualified scientist (Sec. 6.2) or may be analyzed by X-ray diffraction techniques. Samples are prepared for X-ray analysis using TWS-ESS-DP-01. Samples are analyzed using procedures TWS-ESS-DP-16 and -106. The prepared materials are dried to constant weight at 60°C in a drying oven. Store the fines in an appropriate labeled plastic container. Withdraw a representative aliquot of the sample for mineralogical analysis (Eg. X-ray diffraction, see Sec. 6.8).

7.0 QUALITY ASSURANCE

7.1 Documentation

Procedures followed during the preparation and purification of sample materials and results of any measurements made (Eg. weight of material and associated balance checks) shall be documented in the scientific notebook. Any deviation from this procedure shall be documented in the scientific notebook as needed for clarification. Each sample shall be uniquely identified and labeled so that it is traceable through its preparation and can ultimately be traced to its origin. Personnel certification and training shall be documented according to LANL NNWSI project certification procedures. Documentation generated during the conduct of this procedure shall conform to LANL NNWSI project requirements for documentation. This procedure shall be issued, revised, and controlled in accordance with LANL NNWSI project document control procedures.

7.2 Personnel Certification and Training

Project personnel conducting any task described in this procedure must be approved and certified by the PI and/or LS. Project personnel directed to use this procedure will read the procedure and will demonstrate a working understanding of the operational steps to the satisfaction of the LS.

7.3 Acceptance/Rejection Criteria

The LS or SI may, on good scientific and professional judgement, reject any material or sample prior to or after preparation or processing, or after characterization. Once rejected, the material will not be used in reactive tracer sorption experiments unless it is recertified for use.

7.4 Handling, Shipping, and Storage Requirements

Receipt, transfer or custody, and storage of any of the experimental materials shall be documented in the sample logbook and otherwise handled as directed in TWS-HSE12-DP-307. Geologic materials shall be stored in appropriate labeled containers and kept under the control of NNWSI project personnel. Any information relative to the source integrity, and disposition of the materials will be noted in the sample logbook, scientific notebook, or resident file, as appropriate. Liquid samples for analysis should be stored in a refrigerator at 4°C until analysis; and information pertinent to the handling of these samples shall be documented in the sample logbook or the scientific notebook.

8.0 REFERENCES

1. TWS-HSE12-DP-312: Particle Size Reduction of Geologic Media.
2. TWS-INC-DP-63: Preparation of NTS Core Samples for NNWSI Crushed Rock Experiments.
3. TWS-ESS-DP-01: X-Ray Powder Diffraction Analysis.
4. TWS-HSE12-DP-322: Magnetic Separation of Impurities.
5. TWS-ESS-DP-110: Zeolite Purification/Separation Procedure.
6. TWS-ESS-DP-25: Clay Mineral Separation and Preparation for X-Ray Diffraction Analysis.
7. TWS-HSE12-DP-313: Calibration and Use of Centrifuges.
8. TWS-HSE12-DP-317: Calibration and Use of Analytical and Top-Loading Balances.
9. TWS-ESS-DP-16: Siemens X-Ray Diffraction Procedure.
10. TWS-ESS-DP-106: Philips X-Ray Diffraction Procedure.
11. TWS-HSE12-DP-307: Sample Identification and Control.

PARTICLE SIZE REDUCTION OF GEOLOGIC MEDIA

Effective Date: 5/2/88

Edward H. Essington 3/22/88
Edward H. Essington Date
Preparer

W. L. Polzer 3/23/88
W.L. Polzer Date
Technical Reviewer

K. West 4/28/88
K. West Date
Acting QAPL

D. C. Oakley 5/2/88
D.C. Oakley Date
TPO

PARTICLE SIZE REDUCTION OF GEOLOGIC MEDIA

1.0 PURPOSE

This procedure describes the steps for reducing the particle size of geologic materials. The prepared geologic materials are used in laboratory tracer experiments in the C-well Reactive Tracer Study for the Nevada Nuclear Waste Storage Investigation (NNWSI) project.

2.0 SCOPE

This procedure applies to size reduction of large amounts of solid geologic materials using hand crushing, ore crushing, and pulverizing techniques. This procedure is an extension of TWS-HSE12-DP-311. Materials prepared by this procedure are used in generation of data for site characterization; therefore, the activity is Quality Level I.

3.0 PRINCIPLE (NA)

4.0 DEFINITIONS (NA)

5.0 RESPONSIBILITIES

It is the responsibility of the project Principal Investigator (PI) to assure correct implementation of this procedure. The project PI may designate a Laboratory Supervisor (LS) to conduct this procedure and to certify and supervise personnel in the use of this procedure; that designation will be documented by memorandum to the group resident file. This procedure, or portions thereof, will be conducted according to the recommendation of the scientific investigator (SI).

6.0 PROCEDURE

Solid geologic materials are crushed, pulverized, and sieved through a 500- μ m screen or other size screen as specified by the LS or SI. Small-sized samples can be pulverized according to TWS-INC-DP-63. Only one sample at a time shall be processed through any crushing, pulverizing, or sieving activity to avoid cross-contamination. Materials that come in contact with the sample during particle size reduction operations shall be of a composition so as to avoid or minimize introduction of contaminants (Eg. ferruginous matter).

6.1 Equipment

6.1.1 Ore Crusher

Chipmunk jaw crusher type, Braun type VD, capable of reducing rock materials of hardness of least 8 on the "Mohs Scale" (Eg. quartz hardness is 7) to 0.5 cm diameter. See Attachment 1 for catalog description.

6.1.2 Pulverizer

Rotating ceramic plate BICO type UD pulverizer capable of pulverizing material of hardness of at least 8 on the "Mohs Scale". Note that the ceramic plates are hardness 9. See Attachment 2 for catalog description of typical UD pulverizer.

6.1.3 Mortar and Pestle

Ceramic or agate mortar and pestle, capable of grinding material of hardness of at least 8 on the "Mohs Scale".

6.1.4 Blender

Blender with stainless steel blades and glass container, Waring type.

6.1.5 Sieve

Sieves with stainless steel screen, 500- μ m screen opening, ASTM or equivalent specifications.

6.1.6 Vacuum Filter

Porous ceramic or high fired aluminum oxide vacuum filtration device, capable of holding one bar of tension (see Attachment 3).

6.2 Hand Crushing

The intent of this step is to break large pieces of solid material into pieces no larger than about 3 cm or less so that they can be fed into the jaw crusher. Place into a strong plastic bag the solid material to be crushed, a stainless steel bucking plate (about 12 x 12 x 1 cm), and a hammer (preferably stainless steel). The bag is to protect the operator from flying chips and to prevent against loss of sample. Place a large piece of sample on the bucking plate and carefully break it with the hammer into pieces small enough to fit into jaw crusher.

6.3 Jaw Crushing

6.3.1 Preparing Crusher

Clean jaw crusher plates first, by removing scale and rust with a wire brush and carborundum sandpaper, and second, by washing with ethanol using a stiff-bristle brush (Eg. toothbrush). The plates may need to be removed from the

crusher to be properly cleaned. Affix a strong plastic bag to the delivery port of the crusher in order to catch all of the crushed material. The jaw crusher plates should be made of stainless steel; the use of any other plate material in the jaw crusher will depend on the recommendation of the LS or SI.

6.3.2 Operating Crusher

Carefully add pieces of material to the crusher. Keep crusher inlet covered to retain chips that might be expelled during the crushing operation. This crushing step is to prepare the solid materials to be accepted by the pulverizer; therefore, one pass through the crusher to produce particles about 0.5 cm diameter will be sufficient.

6.4 Pulverizing

6.4.1 Preparing Pulverizer

Make sure the white ceramic grinding plates of the pulverizer are in good condition. Metal plates must not be used. Clean plates by passing about 100 g of clean silica sand through pulverizer and then wash plates and interior of pulverizer thoroughly with ethanol using a bristle brush and/or lint-free towel. Adjust plate clearance until the plates just touch and then back off the adjustment screw 1/8 to 3/4 turn depending on the degree of grinding desired. Line the inside of the catch tray with a plastic bag taking care to tape the bag in place; install the tray into the pulverizer. Place tape around the outside of the catch tray door and anywhere else that sample dust may leak out while pulverizing sample.

6.4.2 Operating Pulverizer

Material to be pulverized must be dry. Only put sample material in the pulverizer while the motor is running. Slowly feed sample into the pulverizer so that the sample will gravity feed smoothly. Do not overload pulverizer nor process more sample than will fit into the catch tray. Shut off the pulverizer when an appropriate amount of sample has been processed. Do not open the pulverizer until all parts have stopped moving and the power cord has been removed from the power source.

6.4.3 Retrieving Sample

After opening the pulverizer, gently brush any sample sticking to the inside of the pulverizer chamber into the catch tray. Sieve sample through a 500- μ m screen saving the fines as sample and returning the coarse material to pulverizer with the plate clearance readjusted for finer pulverizing. Repeat this step until all material passes the sieve.

6.5 Pulverizing Micas

For pulverizing mica (Eg. biotite) the following special wet blending procedure is necessary due to the platy nature of the mineral.

6.5.1 Preparing Blender

Assemble blender blades to blender container. Rinse container with 0.1N HNO_3 and thoroughly rinse with distilled water; drain excess water before use. Clean a 600-mL beaker or other suitable container in the same manner. Thoroughly clean and rinse the sieve with distilled water.

6.5.2 Operating Blender

Place the crushed material (Eg. <0.5 cm diameter), into a "Waring"-type blender and suspending in distilled water or reaction solution. Blend suspension until large particles are reduced to a finer size in the blender, pass the suspension through a 500- μ m screen, and collect the fines in a 600-mL beaker or other suitable container. Allow fines to settle for several minutes, then decant the solution back to the blender and return the coarse fraction that did not pass the screen back to the blender. Repeat this step until almost all material passes the sieve.

6.5.3 Preparing Pulverized Sample

Dry the fines by first removing the excess water and then by air drying to constant weight (TWS-HSE12-DP-317). Excess water can be removed by allowing the suspension to settle for about 24 hours and carefully decanting the clear supernate. Vacuum filtration using a porous ceramic cup filter may be used to remove excess water (Sec. 6.9 of TWS-HSE12-DP-311).

6.6 Safety

Persons conducting this procedure must be observant of the hazards associated with crushing, pulverizing and blending operations and of the physical and electrical hazards associated with the operating equipment. Safety glasses must be worn during operation of these devices.

7.0 QUALITY ASSURANCE

7.1 Documentation

Procedures followed during preparation of solid materials shall be documented in the scientific notebook. Any deviation from this procedure shall be documented in the scientific notebook. Each sample shall be uniquely identified and labeled so that it is traceable through its preparation. Documentation generated during the conduct of this procedure shall conform to LANL NNWSI project requirements for documentation. This procedure shall be issued, revised, and controlled in accordance with LANL NNWSI project document control procedures.

7.2 Acceptance/Rejection Criteria.

The LS or SI may, on good scientific and professional judgement, reject any material prior to or after preparation or processing, or after characterization. Once rejected, the material may not be used in reactive tracer experiments unless it is recertified for use.

7.3 Personnel Certification and Training

Project personnel conducting any task described in this procedure must be approved and certified by the PI and/or LS. Certification and training shall be documented according to LANL NNWSI project certification procedures. Project personnel directed to use this procedure will read the procedure and will demonstrate a working understanding of the operational steps to the satisfaction of the LS.

7.4 Handling Shipping and Storage

Receipt, disposition, and storage of any of the experimental materials shall be documented in the sample logbook and will be otherwise handled as directed in TWS-HSE12-DP-307. Transfer of custody of samples for analysis will be according to that same procedure. Original or prepared geologic material shall be stored in appropriately labeled containers and kept in cabinets dedicated to the NNWSI program.

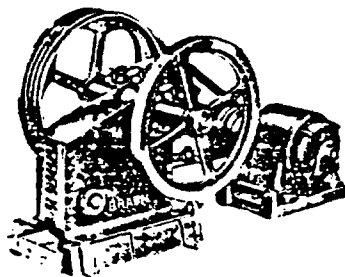
8.0 ATTACHMENTS

1. Attachment 1: Chipmunk Ore Crusher
2. Attachment 2: Type UD Pulverizer
3. Attachment 3: Porous Ceramic Cups

9.0 REFERENCES

1. TWS-HSE12-DP-311: Sample Preparation
2. TWS-INC-DP-63: Preparation of NTS Core Samples for Crushed Rock Experiments
3. TWS-HSE12-DP-317: Calibration and Use of Analytical and Top-Loading Balances
4. TWS-HSE12-DP-307: Sample Handling and Transfer.

Attachment 1



S-60836-10

CRUSHER — Ore, Chipmunk, Improved Type VD, with 2 hp Motor.

Identical with S-60836 except supplied with 2 173 kg.

hp, 220/440 volt, 50/60 Hz, 3 phase motor,

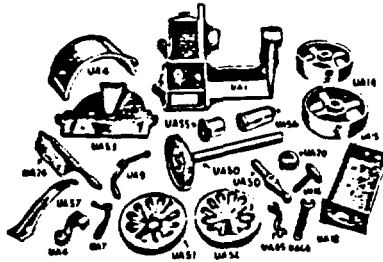
with switch, sample pan, four V-belts, and

adjustable mounting blocks. Shipping weight,

Each 2098.00

Attachment 2

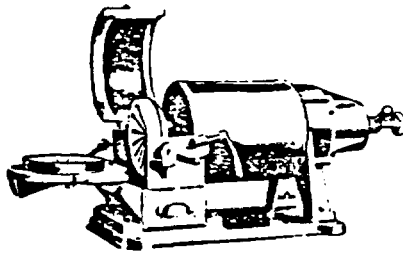
Replacement Parts for UA Pulverizer



	Type No.	Cat. No.
Drop Clutch and Hand screw for clutch	UA 6*/UA 7*	24258-026
Stationary plate, iron alloy	UA 51	24258-048
Revolving plate, iron alloy	UA 52	24258-060
Front bearing bushing (Ser. #s prior to 32260)	UA 55	24258-081
Needle bearing, complete	UA 55N	24258-106
Rear bearing bushing	UA 56	24258-128
Stationary plate, low phosphorus carbon steel	UA 81	24258-140
Revolving plate, low phosphorus carbon steel	UA 82	24258-161
Stationary plate, manganese steel	UA 83	24258-183
Revolving plate, manganese steel	UA 84	24258-208
Stationary plate, hardened	UA 85	24258-220
Revolving plate, hardened	UA 86	24258-241
Stationary plate, ceramic	UA 92XX	24258-263
Revolving plate, ceramic	UA 93XX	24258-285

*Always give serial No. of pulverizer when ordering.

Type UD Direct Drive Pulverizer



Features

Compact; occupies less than 2 x 3' space.
No belts to slip; no gears to strip or wear.
Enclosed motor protected against overload by thermal relay.
High tensile strength cast iron frame; alloy steel shaft.
Permanently lubricated ball bearings.

Specifications

Motor speed: 900 rpm
Dimensions: 33" L x 16" W x 15 1/4" H
Motor: 2-hp., 220/440 v., 50/60 Hz., 3-phase AC, thermal relay switch
Net weight: 375 lbs; shipping weight: 450 lbs.

Supplied with one each 24258-048 and 24258-060 Grinding Plates

Note: Specify voltage when ordering so proper switch motor can be used.

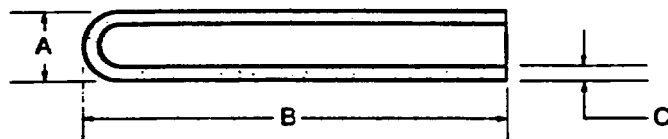
24267

POROUS CERAMIC CUPS, TUBES & PLATES

The following parts are available from stock. Order by new Part No.

Special porous cups, tubes and plates can also be provided. Write for further information giving details of size, material, and quantity required.

ROUND BOTTOM STRAIGHT WALL CUPS



NEW PART NO.	OLD PART NO.	MATERIAL	A	B	C	PRICE
652X1-B1M1	2133	1 BAR, STANDARD	6 mm	6.5 cm	1 mm	3.10
652X2-B1M1	2133-1	1 BAR, STANDARD	6 mm	8.0 cm	1 mm	4.00
652X3-B1M1	2105-1	1 BAR, STANDARD	1/4"	1-1/8"	1/16"	2.50
652X3-B1M3		1 BAR, HIGH FLOW	1/4"	1-1/8"	1/16"	5.20
652X4-B1M1	2134	1 BAR, STANDARD	1 cm	10.0 cm	2 mm	4.00
652X4-B1M3		1 BAR, HIGH FLOW	1 cm	10.0 cm	2 mm	5.20
652X7-B1M1	2550-1	1 BAR, STANDARD	3/8"	1-1/8"	1/16"	3.10
652X7-B1M3		1 BAR, HIGH FLOW	3/8"	1-1/8"	1/16"	4.80
652X7-B2M1		2 BAR, STANDARD	3/8"	1-1/8"	1/16"	4.50
652X7-B3M1		3 BAR, STANDARD	3/8"	1-1/8"	1/16"	3.90
652X7-B5M1		5 BAR, STANDARD	3/8"	1-1/8"	1/16"	3.90
652X8-B1M1	2605-1	1 BAR, STANDARD	7/8"	3-1/8"	5/32"	2.20
652X9-B1M3		1 BAR, HIGH FLOW	7/8"	3-1/2"	1/4"	5.20
652X10-B1M3		1 BAR, HIGH FLOW	7/8"	3-1/2"	1/8"	5.30
652X11-B1M3		1 BAR, HIGH FLOW	1/2"	2-1/2"	1/8"	5.90
652X16-B1M1	2800-1	1 BAR, STANDARD	3/4"	2-5/8"	5/32"	2.20
652X16-B1M3		1 BAR, HIGH FLOW	3/4"	2-5/8"	5/32"	5.20
652X18-B1M3		1 BAR, HIGH FLOW	1.57"	7.5"	.2"	12.00
652X18-B.5M2		1/2 BAR, HIGH FLOW	1.57"	7.5"	.2"	12.00

High Flow refers to high fired Al Oxide

SOILMOISTURE EQUIPMENT CORP.
P.O. Box 30025
Santa Barbara, CA 93105
S.A.

RFS-

1

8- 705-964-3525



CALIBRATION AND USE OF CENTRIFUGES

Effective Date: 5/2/88

Edward H. Essington 3/23/88
Edward H. Essington Date
Preparer

W. L. Polzer 3/23/88
W.L. Polzer Date
Technical Reviewer

R. West 4/28/88
R. West Date
Acting QAPL

D. T. Oakley 5/2/88
D. T. Oakley Date
TPO

CALIBRATION AND USE OF CENTRIFUGES

1.0 PURPOSE

This procedure describes the calibration and use of centrifuges for separating the solid phase, or fraction thereof, from the liquid phase of particle suspensions. These separations are required for clarification of solutions for chemical analysis, preparation of solids for mineral characterization, and for determination of particle size distributions for material characterization in the C-Well Reactive Tracer Study for the Nevada Nuclear Waste Storage Investigation (NNWSI) project.

2.0 SCOPE

This procedure is to be followed in the use of high- and low-speed centrifuges, calibration of high-speed centrifuges, and rpm checks of low-speed centrifuges. Centrifugation is used in the development of data for site characterization; therefore, this activity is Quality Level I.

3.0 PRINCIPLE (NA)

4.0 DEFINITIONS (NA)

5.0 RESPONSIBILITIES

It is the responsibility of the project Principal Investigator (PI) to assure correct implementation of this procedure. The project PI may designate and certify a Laboratory Supervisor (LS) to conduct this procedure or certify and supervise personnel to conduct this procedure; that designation will be documented by memorandum to the group resident file.

6.0 PROCEDURE

6.1 Equipment

6.1.1 High-Speed Centrifuge

A high-speed centrifuge (Eg. SORVAL RC5B) can be run up to about 20,000 rpm depending on rotor head and sample container configuration. Calibration of the internal tachometer will provide the necessary assurance that rotor speed can be set to the prescribed rpm.

6.1.2 Low-Speed Centrifuge

A centrifuge that can be run up to about 2,000 rpm depending on rotor head and sample container

configuration. This type of centrifuge is not expected to require rpm calibration.

6.2 Calibrations

Perform calibrations of the centrifuge's internal tachometer or establish operating rpm using a calibrated phototachometer (TWS-HSE12-DP-310). Follow the tachometer instructions for installing stick-on reflectors on the centrifuge shaft. The need, schedule, and type of calibration is specified by the LS or is specified by the governing detailed procedure.

6.2.1 High-Speed Centrifuge Internal Tachometer

Calibrate internal tachometer with the centrifuge rotor removed taking great care not to allow the drive shaft to rotate faster than recommended by the manufacturer. Adjust the speed to 10,000 ($\pm 5\%$) rpm based on the phototachometer. Adjust the centrifuge tachometer to read 10,000 ($\pm 5\%$) rpm. Check the calibration at another angular velocity (Eg. 4,000 rpm). Recheck the calibration at both rpms. If the calibration cannot be maintained or appears inadequate consult the LS.

6.2.2 Low-Speed Centrifuge

Low-speed centrifuges will not be calibrated but may be checked for approximate rpm using a phototachometer. The check is accomplished in the same manner as in Sec 6.4.1 using 2,000 rpm and 1,000 rpm to check the rotor speed. For models that have no internal tachometer the only method of checking the speed is through use of the phototachometer.

6.3 Operation (General)

Adherence to the following operating procedures is necessary for separations of acceptable quality and for the safety of the operator. Never operate the centrifuge beyond the maximum specified operating speed for a given rotor (consult manufacturer's recommendations). Before each run visually check rotor, tubes, and caps for weak spots and check rotor O-rings (if present) for breakage. Do not use rotor, tubes, or O-rings which appear fatigued. Make sure that rotor and tubes are properly balanced. Weigh each tube and place a tube of the same weight (± 0.1 g) directly across from it in the rotor. Double check that the rotor is balanced and that all tubes are in the proper position before operating the centrifuge. Never open the

centrifuge door while the rotor is still turning except as required for calibration.

6.4 Particle Separations

To accomplish the separation of a pre-determined particle size, a calibrated centrifuge will be used. The rpm and the run time must be determined based on the sample dimensions expected during centrifuge operation.

6.4.1 Calculation of Run Time

Based on Stoke's Law of a particle settling in a liquid, the run time (t) in minutes can be calculated given the angular velocity (w) in rad/sec by:

$$t = \frac{18v[\ln(R1w^2) - \ln(R2w^2)]}{(d_p - d_l)d^2w^2}$$

where R1 and R2 are the upper and lower radii from the center of rotation to the top of the liquid in the centrifuge tube and to the bottom of the tube (cm), respectively, under operating conditions; d_p is the density (g/cc) of the particle, d_l is the density (g/cc) of the liquid, v is the kinematic viscosity (centipoise) of the liquid, and d is the particle diameter (cm).

6.4.2 Computer Program

The computer program CENT.BAS (Attachment 1) can be used to calculate the run time for separation of specific particle diameters in aqueous solutions. The program operates on fixed rotor parameters or can use input values of R1 and R2.

7.0 QUALITY ASSURANCE

7.1 Documentation

Procedures followed during the calibration and use of centrifuges and results of any measurements made (Eg. rpm and time) shall be documented in the scientific notebook. Any deviation from this procedure shall be documented in the scientific notebook as needed for clarification. A sticker displaying "OPERATOR TO CALIBRATE" shall be placed on the centrifuge that has had its internal tachometer calibrated. Certification and training shall be documented according to

LANL NNWSI project certification procedures. Documentation generated during the conduct of this procedure shall conform to LANL NNWSI project requirements for documentation. This procedure shall be issued, revised, and controlled in accordance with LANL NNWSI project document control procedures.

7.2 Personnel Certification and Training

Project personnel conducting any task described in this procedure must be approved and certified by the PI or LS. Training of project personnel directed to use this procedure will consist of reading the procedure and demonstrating a working understanding of the operational steps to the satisfaction of the LS.

7.3 Handling, Shipping, and Storage (NA)

8.0 REFERENCES

1. TWS-HSE12-DP-310: Calibration and Use of the Phototachometer

9.0 ATTACHMENTS

1. Attachment 1: IBM-PC BASIC Program -- CENT.BAS

```

5 ' CENT.BAS V1.0, 6/25/87, E.H. ESSINGTON
10 'PROGRAM CALCULATES TIME NECESSARY TO SEGREGATE PARTICLE SIZE GIVEN
20 'ROTOR/SAMPLE CONFIGURATION AND OPERATING RPM AT SPECIFIED
30 'TEMPERATURE
40 DIM Q(42),V(42)
50 FOR I=1 TO 41:READ Q(I):NEXT I
60 FOR I=1 TO 41:READ V(I):NEXT I
70 CLS
80 PRINT
90 PRINT "*****"
100 PRINT "*"
110 PRINT "* Centrifuge rotor/sample configuration - radius *"
120 PRINT "*"
130 PRINT "* 1 Sorval GSA: R1= 9.5 cm, R2= 13.9 cm. *"
140 PRINT "* 2 Sorval SS-34: R1= 5.2 cm, R2= 10.4 cm. *"
150 PRINT "* 3 IEC Model CL: R1= 4.0 cm, R2= 14.0 cm. *"
160 PRINT "* 4 Other *"
170 PRINT "* 5 Exit *"
180 PRINT "*"
190 PRINT "*****"
200 PRINT
210 INPUT "Enter choice: ",M
220 IF (M<>1) GOTO 260
230 B=9.5
240 C=13.9
250 GOTO 420
260 IF (M<>2) GOTO 300
270 B=5.2
280 C=10.4
290 GOTO 420
300 IF (M<>3)GOTO 340
310 B=4!
320 C=14!
330 GOTO 420
340 IF (M<>4)GOTO 380
350 INPUT "Enter R1 radius in cm: ",B
360 INPUT "Enter R2 radius in cm: ",C
370 GOTO 420
380 IF (M<>5)GOTO 400
390 GOTO 620
400 INPUT "choice not valid, try again ",M
410 GOTO 220
420 INPUT "Enter RPM: ",F
430 F=(F*6.283)/60!:REM Converts RPM to radians
440 INPUT "Enter particle diameter in microns: ",G
450 G=G/10000!
460 INPUT "Enter operating temperature, deg C: ",QQ
470 GOSUB 630
480 A=A*18
490 D=2.5: REM Density of particle
500 E=1!: REM density of water
510 I=A/((D-E)*G^2*F^2)
520 J=(LOG(C*F^2))-(LOG(B*F^2))
530 T=I*J
540 T=T/60
550 F=(F/6.283)*60
560 L=F
570 PRINT:PRINT "Particle diameter (cm)= ",G

```



```
580 PRINT "Angular velocity (RPM)= ",L&
590 PRINT "Run time (min)      = ",T:PRINT
600 INPUT "Enter <CR> to continue",N
610 GOTO 70
620 SYSTEM
630 FOR I=1 TO 41
640 IF (QQ<>Q(I)) THEN NEXT I
650 IF (I<41)GOTO 680
660 PRINT "Temperature choice out of range - ending program"
670 END
680 A=V(I)/100!:RETURN
690 DATA 10,11,12,13,14,15,16,17,18,19,20,21,22,23,24,25,26,27,28,29,30,31,32
700 DATA 33,34,35,36,37,38,39,40,41,42,43,44,45,46,47,48,49,50
710 DATA 1.307,1.271,1.235,1.202,1.169,1.139,1.109,1.081,1.053,1.027,1.002
720 DATA 0.9779
730 DATA 0.9548,0.9325,0.9111,0.8904,0.8700,0.8513,0.8327,0.8148,0.7975,0.7808
740 DATA 0.7647
750 DATA 0.7491,0.7340,0.7194,0.7052,0.6915,0.6783,0.6664,0.6529,0.6408
760 DATA 0.6291,0.6178,0.6067,0.5960
770 DATA 0.5856,0.5755,0.5656,0.5561,0.5468
```

PREPARATION OF STANDARD AND REAGENT SOLUTIONS

Effective Date: 5/2/88

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3/22/88
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PREPARATION OF STANDARD AND REAGENT SOLUTIONS

1.0 PURPOSE

This procedure describes steps for preparing stock solutions (standards and reagents) and for making dilutions. The solutions are used in the C-Well Reactive Tracer Study for the Nevada Nuclear Waste Storage Investigation (NNWSI) project.

2.0 SCOPE

This procedure is to be followed for preparation of standard and reagent solutions and their dilutions if they are to be used in generating results that will be used in site characterization. The activity is Quality Level I.

3.0 PRINCIPLE (NA)

4.0 DEFINITIONS (NA)

5.0 RESPONSIBILITIES

It is the responsibility of the project Principal Investigator (PI) to assure correct implementation of this procedure. The project PI may designate and certify a Laboratory Supervisor (LS) to conduct this procedure and to certify and supervise personnel in the use of this procedure; that designation will be documented by memorandum to the group resident file. This procedure, or portions thereof, will be conducted according to the recommendation of the scientific investigator (SI).

6.0 PROCEDURE

6.1 Specifications

6.1.1 Reagent Specifications

All reagents shall be "reagent grade" or better in quality; if not, appropriate standardization and characterization must be done. The compound or material must be sufficiently soluble in a suitable solvent that is compatible with the needs of the guiding experiment. The compound, material, or stock solution must not be outdated based on a manufacturer's expiration date or a date specified by the LS.

6.1.2 Solvent Specifications

The primary solvent will be distilled water. Distilled water used for preparing stock solutions or dilutions shall have a resistivity of at least 0.5 meg ohms

(conductivity of 2 μ mhos/cm or less, refer to TWS-HSE12-DP-314). Natural solutions (Eg. J-13 well water) used for preparing stock solutions or dilutions must have been autoclaved and/or filtered through a 0.05- μ m pore diameter filter unless specified otherwise by the guiding detailed procedure or by the LS. Preparation and use of solvents other than distilled water will be specified by the guiding procedure or by the LS.

6.1.3 Container Specifications

Class "A" volumetric containers (Ref. 2) shall be used in making stock solutions or dilutions. When in-house calibration of volumetric containers is judged necessary by the LS or SI, elements of a standard procedure (Eg. Ref. 3) will be used. Containers are washed twice with 0.1N HNO_3 and rinsed a minimum of three times with distilled water unless specified otherwise by the guiding procedure or by the LS.

6.1.4 Solution Storage Specifications

Store all prepared solutions in appropriate containers (Eg. polyethylene, polypropylene or glass). Rinse containers twice with 0.1N HNO_3 and at least three times with distilled water unless specified otherwise in the guiding procedure or by the LS. Each solution container shall be labeled with a unique identifier (to allow for traceability through its preparation) and with other pertinent information (Eg. preparation date, contents of solution, concentration, preparer's initials, and expiration date if applicable). Solutions that require refrigeration for storage will be identified by the LS or SI. Those solutions will be stored in a refrigerator dedicated to the NNWSI project.

6.2 Drying of Stock Reagents

Where appropriate, dry a quantity of the standard or reagent material to a constant weight (within $\pm 1\%$ of the total weight for two successive weighings). Drying temperature will normally be 100°C unless specified otherwise in the guiding procedure or by the LS. If dried at elevated temperatures, allow to cool to room temperature in a desiccator over a suitable desiccant before weighing. Containers should be weighed with the cap (closure) unless specific conditions require that caps not be included. Dried stock reagents may be stored in a desiccator. Redry the reagent if the desiccant is spent or at the discretion of the LS.

6.3 Preparation of Stock Solutions from Dry Materials

Weigh the dried material using a calibrated analytical balance of suitable sensitivity (refer to TWS-HSE12-DP-317). Dissolve the material in an appropriate solvent, usually either distilled water or a mineral acid, and dilute to a known volume. Attention must be paid to special precautions that may be required (Eg. use of carbonate-free water or control of violent reaction during mixing).

6.4 Preparation of Stock Solutions from Other Solutions.

For preparation of stock solutions from liquid sources, measure the reagent by weight or by appropriate Class "A" volumetric ware, combine with solvent and dilute to a known volume.

6.5 Preparation of Dilutions

Avoid contaminating the stock solution. This can be accomplished by dispensing a portion of the stock solution into a second clean container and removing the required aliquot from that container. Do not return stock solution to the original container once it is dispensed. Place the desired volume of stock solution into a Class "A" volumetric container and dilute to volume with the appropriate solvent. Store the diluted solution in an appropriate labeled container.

7.0 QUALITY ASSURANCE

7.1 Documentation

Procedures followed during preparation of stock solutions or dilutions and results of measurements made (Eg. weight of materials and associated balance checks) shall be documented in the scientific notebook. Any deviation from this procedure shall be documented in the scientific notebook as needed for clarification. Each solution shall be uniquely identified and labeled so that it is traceable through its preparation. Documentation generated during the conduct of this procedure shall conform to LANL-NNWSI project requirements for documentation. This procedure shall be issued, revised, and controlled in accordance with LANL-NNWSI project document control procedures.

7.2 Handling, Shipping, and Storage

Storage of stock solutions and dilutions is described in Sec. 6.1.4. All stock solutions, dilutions, and original reagent materials are stored in cabinets or in a refrigerator dedicated to the NNWSI project. Distribution of aliquots of either the stock solutions or dilutions will be made according to TWS-HSE12-DP-307.

7.3 Personnel Certification and Training

Project personnel conducting any task described in this procedure must be approved and certified by the PI and/or LS. Certification and training shall be documented according to LANL-NNWSI project certification procedures. Project personnel directed to use this procedure will read the procedure and will demonstrate a working understanding of the operational steps to the satisfaction of the LS.

7.4 Calibration

A record of any volumetric ware calibrations will be maintained in the scientific notebook. Certificates of calibration will be placed in the resident file.

7.5 Acceptance/Rejection Criteria

The LS or SI may, on good scientific and professional judgement, reject any material planned for use in making stock solutions or dilutions, and may reject any prepared solution. Once rejected, the material or solution may not be used in reactive tracer experiments unless it is recertified for use.

8.0 REFERENCE

1. TWS-HSE12-DP-314: Electrical Conductivity Measurement.
2. ASTM Procedure E 694-83, Standard Specifications for Volumetric Ware.
3. ASTM Procedure E 542-85, Standard Practice for Calibration of Volumetric Ware, 1986 ed. of ASTM Procedures.
4. TWS-HSE12-DP-317: Calibration and Use of Analytical and Top-Loading Balances.
5. TWS-HSE12-DP-307: Sample Handling and Transfer.

CALIBRATION AND USE OF ANALYTICAL AND TOP-LOADING BALANCES

Effective Date: 5/2/88

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CALIBRATION AND USE OF ANALYTICAL AND TOP-LOADING BALANCES

1.0 PURPOSE

This procedure describes steps for the calibration and use of weighing instruments.

2.0 SCOPE

This procedure applies to all weight determinations and calibrations that use analytical and top-loading balances for the C-Well Reactive Tracer Studies for the Nevada Nuclear Waste Storage Investigation (NNWSI) project. Materials weighed under the direction of this procedure are used in generating data for site characterization; therefore, the activity is Quality Level I.

3.0 PRINCIPLE (NA)

4.0 DEFINITIONS

4.1 Operator

Person certified to operate balance; a certified calibrator is automatically certified as an operator.

4.2 Calibrator

Person certified to conduct instrument calibrations, adjustments, and minor repairs (Eg. the LS).

4.3 Calibration Group

The Los Alamos National Laboratory (LANL) Standards and Calibration Group, which provides NBS-traceable calibration services.

5.0 RESPONSIBILITIES

It is the responsibility of the project Principal Investigator (PI) to assure correct implementation of this procedure. The project PI may designate and certify a Laboratory Supervisor (LS) to conduct this procedure and to certify and supervise project personnel to conduct this procedure; that designation shall be documented by memorandum to the group resident file. The LS has the responsibility to place a suitable warning notice on any balance that is out of calibration, removing the balance from service.

6.0 PROCEDURE

6.1 Equipment

- 6.1.1 Analytical Balance, capacity: 200g, readability: 0.1mg, standard deviation: 0.05mg, accuracy: 0.1% above 10mg. (Eg. Sartorius model 2462).
- 6.1.2 Top-Loading Balance, capacity: 1200g, readability: 0.01g, standard deviation: 0.005g, accuracy: 1% above 1g. (Eg. Sartorius model 3704).
- 6.1.3 Top-Loading Balance (dual range), capacity: 300g/3000g, readability: 0.01g/0.1g, standard deviation: 0.005g/0.05g, accuracy: 1% above 1g/10g. (Eg. Sartorius model 3713MP).
- 6.1.4 Primary Weights, ASTM-Class 2 (1), calibration traceable to NBS weights.
- 6.1.5 Secondary Weights (check weights), Any stable mass that has its weight determined by comparison to a primary weight.

6.2 Balance Set Up.

Before using a balance make sure the work area around the balance is clean and free of dust and other materials that may interfere with the use of the instrument. Make sure that the balance is located in an area free of unacceptable vibrations and strong air currents. Check the level of the balance and correct if necessary. Check that the weights to be used (primary or secondary) show no signs of damage and are clean. Check overall function of the balance (Eg. lamps or electronic readout). In case of balance malfunction or damaged or unclean weights notify LS who will advise on further use of balance.

6.3 Balance Check

- 1) The balance operator shall conduct a balance check at the beginning of a weighing session, during the weighing session at the discretion of the operator or at the direction of the LS, upon suspicion that the balance has been moved or may be malfunctioning, and at the conclusion of the weighing session.
- 2) Record the instrument identification and the identification number of the weight set.
- 3) Record the response of the balance to at least two weights that bracket the expected weight of the sample; the heavier weight should be only slightly greater than the anticipated sample weight and the zero setting of the

balance may serve as the lower end of the weight check range. 4) If the check weight does not register within the desired tolerance (Eg. 0.5% of the true weight) check the balance level and check work area for interferences, correct the problem, and repeat the check weighing. 5) If the check still fails, notify the LS.

6.4 Balance Use

Always weigh samples on weighing paper or in an appropriate size container (Eg. beaker or weighing boat). Treat all balances gently. Do not bump the balance or weigh objects heavier than those recommended by the manufacturer. Keep weighing pans and platforms clean of any debris or spills. Record the results of all check weighings, the identification of the weight set and the balance, and the results of sample weighings in the scientific notebook.

6.5 Calibration

Balance calibration may be conducted in house by certified personnel (calibrator) or by an approved calibration service (Eg. see 4.3). All calibrations will use primary weights. Statements that follow refer to in-house calibrations.

6.5.1 Calibration of Top-Loading Balances

Conduct balance setup (Sec 6.1). Choose a series of at least three primary weights that span the capability of the balance. Record the weight values and the readings, as well as the balance and weight identifications in the scientific notebook. If the weights do not reproduce within 1% of the stated weight value (weights of 1 g or larger) the balance is out of calibration and must be removed from use until it is serviced and recalibrated.

6.5.2 Calibration of Analytical Balances

Conduct balance setup (see 6.2). Document response of the balance to each of the following weights, 1g (to check response of 0- to 1000-mg scale), 2g, 3g, 4g, 5g, 10g, 20g, 30g, 40g, 50g, and 100g (use the 100-g weight at the 99- and 100-g settings to check the 200-g capacity of the balance). Record the weight set identification number, each weight value and the reading in the scientific notebook. If any of the readings fails to reproduce within 1% of the respective calibration weight, check the balance

interior weights for proper mounting and check other parts of balance for proper operation, then repeat the calibration. If the weights do not reproduce within 1% or the stated weight value the balance is out of calibration and must be removed from use until it is serviced and recalibrated.

6.5.3 Calibration of Secondary Weights

A secondary weight must be calibrated against a primary weight. Prepare the balance as for weighing samples. Prepare and weigh the mass that is to become the secondary weight on an appropriate balance. Document in the scientific notebook the actual weights of the secondary weight and the primary weight used in the calibration. Also document the means for uniquely identifying the secondary weight. The secondary weights may be used as check weights for the purpose of this procedure.

7.0 QUALITY ASSURANCE

7.1 Documentation

Procedures followed in the calibration and use of analytical or top-loading balances and results of any weighings shall be documented in the scientific notebook. Any deviation from this procedure shall be documented in the scientific notebook as needed for clarification. Certification and training shall be documented according to LANL NNWSI project certification procedures. Documentation generated during the conduct of this procedure shall conform to LANL NNWSI project requirements for documentation. This procedure shall be issued, revised, and controlled in accordance with LANL NNWSI project document control procedures.

7.2 Calibration Schedule

Calibrate all balances according to this procedure at least once each year, upon detection and remedy of malfunctions, or at the request of the LS. A sticker displaying "OPERATOR TO CALIBRATE" containing the proper information shall be placed on the balance. Primary weights used in balance calibrations shall be calibrated against NBS standards and certified at least every 2 years as recommended by the LANL calibration group. Secondary weights shall be recertified by the calibrator against primary weights at least annually. If weights appear to have been mishandled or damaged, recertification must be completed before use.

7.3 Personnel and Training

Project personnel conducting any task in support of this procedure must be approved and certified by the PI or LS. Project personnel directed to use this procedure will read the procedure and will demonstrate an understanding of the operational steps to the satisfaction of the LS.

7.4 Acceptance/Rejection

The LS shall have the authority to require that balances be serviced and calibrated at any time. The LS shall have authority to accept or reject any calibration or weight data and to require any weighing to be repeated. Any weighings conducted after a successful check weight but before an unsuccessful check weight will be reweighed (if possible) after the discrepancy in check weights is determined and corrected.

8.0 REFERENCE

- (1) Standard Specification for Laboratory Weights and Precision Mass Standards, ASTM Procedure E 617-81, 1986 ASTM Procedures.

pH MEASUREMENT, ACID-BASE SOLUTION STANDARDIZATION,
AND TOTAL ALKALINITY PROCEDURE

Effective Date: 5/2/88

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**pH MEASUREMENT, ACID-BASE SOLUTION STANDARDIZATION,
AND TOTAL ALKALINITY PROCEDURE**

1.0 PURPOSE

This procedure describes the use and standardization of the pH meter-electrode system for measurement of pH of aqueous solutions, for potentiometric titrations in the standardization of acidic and basic solutions, and in determination of total alkalinity. The preparation of standardization buffers and standard acidic or basic reagents is also described. This procedure is conducted in support of tracer sorption studies performed as part of the C-Well Reactive Tracer Studies for the Nevada Nuclear Waste Storage Investigation (NNWSI) project.

2.0 SCOPE

This procedure describes the use of the pH meter-electrode system to measure solution pH or hydrogen ion activity. The pH meter-electrode system is also used to detect titration endpoints in the potentiometric standardization of acidic and basic solutions and in the determination of total alkalinity. Results generated by performance of this procedure will be used to derive site-characterization data; therefore, this activity is Quality Level I.

3.0 PRINCIPLE (NA)

4.0 DEFINITIONS

4.1 Buffers

Buffers are hydrogen ion concentration standards for pH meter calibration.

5.0 RESPONSIBILITIES

It is the responsibility of the project Principal Investigator (PI) to assure correct implementation of this procedure. The project PI may designate and certify a Laboratory Supervisor (LS) to conduct this procedure and to certify and supervise personnel to conduct this procedure; that designation shall be documented by memorandum to the group resident file. This procedure, or portions thereof, will be conducted according to the recommendations of the scientific investigator (SI).

6.0 PROCEDURE

Refer to TWS-HSE12-DP-316 for details on standard and reagent solution preparation and to TWS-HSE12-DP-317 for instructions on weighing and use of analytical balances.

6.1 Equipment

6.1.1 pH meter-electrode

The pH meter should have capabilities similar to the Corning Model 125 (Eg. pH range: 0-14, resolution: 0.01 pH unit, relative accuracy: ± 0.02 pH unit). The electrode should have capabilities similar to the Orion Model 91-15 (Eg. gel filled, combination, for use in narrow configurations).

6.1.2 Standard Burette

The burette should be of glass and must be Class "A" (ASTM).

6.1.3 Automatic Burette

The automatic burette is an electro-mechanical solution dispensing device designed to accurately track the amount of solution dispensed. A typical unit is the Fisher Burette Model 394 with a 25-mL delivery capacity and delivered-volume accuracy of ± 0.03 mL.

6.2 Buffer Preparation

For the pH range of approximately 4 to 12, standard buffer solutions are used to standardize the pH meter. Beyond this range standard solutions are prepared according to the information in Attachment 1 using reagent grade materials or acidic or basic solutions that are standardized according to the methods described in Sec. 6.5.

6.2.1 pH 4-12

Commercial grade buffer solutions or powders can be used as purchased to prepare working buffer solutions in the range of pH 4-12. The buffer powders or solutions must not be used past the expiration date noted by the manufacturer; the hydrogen ion concentration or the pH of the buffers must be traceable to NBS. Buffer powders are dissolved in distilled water according to the manufacturer's recommendations. Ref. 3 suggests that distilled water greater than 0.5 meg-ohm resistance is acceptable for preparing buffer solutions. Buffer solutions are used as provided or are diluted as recommended by the manufacturer.

6.2.2 pH >12 or <2

Attachment 1 lists mixtures of materials for preparing solutions of known pH. These preparations may be used for routine pH standardization but only at the discretion of the LS or SI. More accurate pH standards are prepared in the same manner but the acidity and basicity is determined by potentiometric titration against known standards (described in Sec. 6.5).

6.2.3 Buffer or pH Standard Solution Acceptability

If the buffer is not completely clear (contains suspendible material), has exceeded its expiration date, or is deemed inappropriate by the LS or SI, the buffer solution is discarded and not used in tracer studies.

6.3 Standardization of pH Meter-Electrode System

Standardize the system to the appropriate pH buffers (± 0.05 pH unit) before, during, and after a series of measurements; a different precision may be specified by the LS or SI. Standardization is performed with at least two standard buffers that bracket the expected pH range of the sample. The general procedure includes the following steps: 1) condition buffer solutions to approximately the same temperature as the electrode; 2) turn on the pH meter and assure that the electrode is properly serviced and functional; 3) standardize according to manufacturer's recommendations with the two buffers that bracket the expected range of sample pH, rinse with distilled water, and gently shake excess water from the electrode before and after use in each solution; 4) alternate between buffers adjusting standardization and slope controls until meter responds properly to each buffer; and 5) rinse electrode with distilled water and store the electrode in the manner recommended by the manufacturer.

6.4 pH Measurements

References (4), (5), (6), and (7) contain relevant information on the measurement of pH. Details of steps used in measurement of pH will depend on the specific experimental conditions and will be specified by the LS or SI. The general procedure includes the following steps: 1) conduct pH meter standardization as described in Sec. 6.3; 2) condition sample to approximately same temperature as the buffers used to standardize the pH meter; 3) place electrode in sample solution

and allow pH meter reading to stabilize; 4) record reading; and 5) rinse electrode for further use or storage.

Recheck standardization at the end of measurements on a series of samples or upon suspicion that the pH meter-electrode system has malfunctioned. If the pH value of either of the buffers has shifted more than 0.1 pH unit, restandardize the pH meter. Repeat pH measurements on the last sample measured progressing backward through the sample series until the new pH reading reproduces the original reading (within ± 0.05 pH unit). Record the new results as final if there are differences. If the pH meter-electrode system does not maintain standardization consult the LS.

6.5 Potentiometric Titrations

Commercial acidic and basic standard solutions or dilutions of those standard solutions (TWS-HSE12-DP-316) are used as standard titrants. If such solutions are not available, a prepared titrant is standardized against reference or reagent grade materials traceable to NBS. Reference 3 contains relevant information on potentiometric (electrometric) titrations.

The general procedure described here uses the electrometric method and includes the following steps: 1) load standard or automatic burette with the appropriate titrant; 2) transfer an appropriate aliquot of standard solution, sample, or blank into a clean container, or accurately weigh on an analytical balance an appropriate amount of solid into a clean container and dissolve the solid in distilled water; 3) titrate the sample to the specified endpoint (Sec. 6.6) and record the results; 4) if a standard is titrated calculate the normality (N) of the titrant applying any corrections for blank; and 5) if an unknown is titrated calculate the acidity of the unknown applying any corrections for blank.

6.6 Total Alkalinity

Additional information on measurement of total alkalinity is given in Ref. 8 and 9.

6.6.1 Measurement

The procedure for measuring total alkalinity includes the following steps: 1) standardize the pH meter-electrode system according to Sec. 6.3; 2) use a standardized acidic titrating solution (Eg. hydrochloric or sulfuric acid at about 0.003 to 0.01M) to titrate the sample; 3) titrate the sample

at room temperature (20° to 30°C) to pH 4.5 and record the volume of acid required to reach this pH; 4) continue the titration to pH of 4.2 and record the volume of acid required to reach this pH; 5) treat a similar aliquot of distilled water or other specified solution as a blank; and 6) calculate the total alkalinity as suggested in Ref. (10) or as in Sec. 6.6.2.

6.6.2 Calculation

Assume:

A = aliquot size (mL or g).

VB5 = volume of titrant to reach pH 4.5 in blank (mL).

VB2 = volume of titrant to reach pH 4.2 in blank (mL).

VS5 = volume of titrant to reach pH 4.5 in unknown (mL).

VS2 = volume of titrant to reach pH 4.2 in unknown (mL).

N = acid strength (meq/mL).

BA = titration response to blank, and

TA = total alkalinity (meq/unit of sample, mL or g).

$$BA = \frac{[VB5 - (VB2 - VB5)]N}{A}$$

$$TA = \frac{[VS5 - (VS2 - VS5)]N}{A} - BA$$

7.0 QUALITY ASSURANCE

7.1 Documentation

Procedures followed in measurements of pH or in titrations and the results of any measurements (Eg. weight of material, associated balance checks, and volume of titrant) shall be documented in the scientific notebook. Any deviation from this procedure shall be documented in the scientific notebook as needed for clarification. Personnel certification and training shall be documented according to LANL NNWSI project certification procedures. Documentation generated during the conduct of this procedure shall conform to LANL NNWSI project requirements for documentation. This procedure shall be issued, revised, and controlled in accordance with LANL NNWSI project document control procedures.

7.2 Handling, Shipping and Storage Requirements

Receipt, disposition, and storage of any of the experimental materials awaiting analysis shall be documented in the sample logbook and handled as directed in TWS-HSE12-DP-307. Transfer of custody for analysis will be according to that same procedure. Samples shall be stored in appropriate labeled containers and kept under the control of NNWSI project personnel. Any information relative to sample disposition and changes in sample integrity as a result of treatment by this procedure shall be noted in the sample logbook, scientific notebook, or resident file, as appropriate. Liquid samples for analysis should be stored at 4 C unless specified otherwise by the LS or SI.

7.3 Materials

The purity of all materials used as standards should be traceable to NBS or be of reagent grade or better; use of any material is at the discretion of the LS or SI.

7.4 Personnel Certification and Training

Project personnel conducting any task described in this procedure must be approved and certified by the PI or LS. Training of project personnel directed to use this procedure will consist of reading the procedure and demonstrating a working understanding of the operational steps to the satisfaction of the LS.

7.5 Acceptance/Rejection Criteria

The LS or SI, on good scientific and professional judgement, may reject any pH measurement, titration, or standardization and may require any measurement to be repeated.

8.0 REFERENCES

1. TWS-HSE12-DP-316: Preparation of Stock Solutions and Reagents.
2. TWS-HSE12-DP-317: Calibration and Use of Analytical and Top-loading Balances.
3. Standard Methods for the Examination of Water and Wastewater, 16th edition., 1985, (Ed. M.A. Franson), "Acidity" pp. 265-269.
4. Standard Methods for the Examination of Water and Wastewater, 16th edition., 1985, (Ed. M.A. Franson), "pH Value" pp. 429-437.

5. ASTM D 1293-84 (1986), Methods for the pH of Water.
6. Methods of Soil Analysis, Pt. 2, 2nd Edition., 1982, (Ed. A.L. Page), Am. Soc. Agronomy Inc., Madison, Wis. pp. 199-209.
7. TWS-INC-DP-35: pH Measurement.
8. Standard Methods for the Examination of Water and Wastewater, 16th edition., 1985, (Ed. M.A. Franson), "Alkalinity" pp. 269-273. ASTM D 1067-82 (1986), Acidity or Alkalinity of Water.
9. ASTM D 1067-82 (1986), Acidity or Alkalinity of Water.
10. Thomas, J.F.J., and J.J. Lynch, 1960, Determinations of Carbonate Alkalinity in Natural Waters. Jour. AWWA, Feb. 1960. pp. 259-268.
11. TWS-HSE12-DP-307: Sample Handling, Storage, and Transfer.

9.0 ATTACHMENTS

1. Attachment 1: Table from Handbook of Chemistry and Physics, 54th Edition, (Ed. R.C. West), CRC Press. 1973-1974. pp. D-112 to D-114.

BUFFER SOLUTIONS OPERATIONAL DEFINITIONS OF pH

Prepared by R. A. Robinson

The operational definition of pH is:

$$\text{pH} = \text{pH}(s) + E/k$$

where E is the e.m.f. of the cell:



the half cell on the left containing the solution whose pH is being measured and that on the right a standard buffer mixture of known pH; $k = 2.303RT/F$, where R is the gas constant, T the temperature in degrees Kelvin and F the value of the faraday.

Alternatively, the cell:



can be used, the glass electrode being calibrated using a standard buffer mixture or, if possible, two standard buffer mixtures whose pH values lie on either side of that of the solution which is being measured. Suitable standard buffer mixtures are:

- 0.05 M potassium hydrogen phthalate (pH = 4.008 at 25°C)
- 0.025 M potassium dihydrogen phosphate
- 0.025 M disodium hydrogen phosphate (pH = 6.865 at 25°C)
- 0.01 M borax (pH = 9.180 at 25°C)

For most purposes pH can be equated to $-\log_{10} \gamma_{\text{H}^+} m_{\text{H}^+}$, i.e., to the negative logarithm of the hydrogen ion activity. There is a small difference between those two quantities if pH > 9.2 or pH < 4.0, given by:

$$\begin{aligned} -\log \gamma_{\text{H}^+} m_{\text{H}^+} &= \text{pH} + 0.014(\text{pH} - 9.2) \text{ for } \text{pH} > 9.2 \\ &= \text{pH} + 0.009(4.0 - \text{pH}) \text{ for } \text{pH} < 4.0 \end{aligned}$$

It should be noted that in the table titled "Solutions giving Round Values of pH at 25°C" it is $-\log \gamma_{\text{H}^+} m_{\text{H}^+}$ and not pH which is quoted when there is a difference between them.

References:

- R. G. Bates, "Electrometric pH Determinations: Theory and Practice" Wiley, New York, 1954.
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PROPERTIES OF STANDARD AQUEOUS BUFFER SOLUTIONS AT 25°C

Solution	Buffer substance	Molality m	Weight of salt in air per liter solution	Density g/ml	Molarity M	Dilution value ΔpH_1	ΔpH_2	Buffer value, equiv. per pH	Temp coeff., dpH_2/dt Units per °C
Tetroxalate	$\text{KH}_2(\text{C}_2\text{O}_4)_2 \cdot 2\text{H}_2\text{O}$	0.05	12.61	1.0032	0.04962	+0.186	-0.0028	0.070	+0.001
Tartrate	$\text{KHC}_4\text{H}_4\text{O}_6$, sat. sol'n. at 25°C	0.0341	1.0036	0.034	+0.049	-0.0003	0.027	-0.0014
Phthalate	$\text{KHC}_8\text{H}_4\text{O}_4$	0.05	10.12	1.0017	0.04958	+0.052	-0.0009	0.016	+0.0012
Phosphate	KH_2PO_4 + Na_2HPO_4	0.025 ^b	3.39 3.63	1.0028	0.0249 ^b	+0.080	-0.0006	0.029	-0.0028
Phosphate	KH_2PO_4 + Na_2HPO_4	0.008695 ^c 0.03043 ^c	1.179 4.30	1.0020	0.008665 ^c 0.03032 ^c	+0.07 ^c	-0.0005	0.016	-0.0028
Borax	$\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	0.01	3.80	0.9996	0.009971	+0.01	-0.0001	0.020	-0.0082
Calcium hydroxide	$\text{Ca}(\text{OH})_2$, sat. sol'n. at 25°C	0.0203	0.9991	0.02025	-0.28	+0.0014	0.09	-0.033

- ^a $\Delta\text{pH}_2 = \text{pH}_2$ (M Molar solution) - pH_1 (m molal solution).
- ^b Concentration of each phosphate salt.
- ^c KH_2PO_4 .
- ^d Na_2HPO_4 .
- ^e Calculated value.

D-112

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BUFFER SOLUTIONS (Continued)
SOLUTIONS GIVING ROUND VALUES OF pH AT 25°C
 Reproduced from "Electrolyte Solutions" by permission from
 Robinson and Stokes, authors, and Butterworth's Scientific
 Publications.

A*		B*		C*		D*		E*	
pH	z	pH	z	pH	z	pH	z	pH	z
1.00	67.0	2.20	49.5	4.10	1.3	5.80	3.6	7.00	46.6
1.10	52.8	2.30	45.8	4.20	3.0	5.90	4.6	7.10	45.7
1.20	42.5	2.40	42.2	4.30	4.7	6.00	5.6	7.20	44.7
1.30	33.6	2.50	38.8	4.40	6.6	6.10	6.8	7.30	43.4
1.40	26.6	2.60	35.4	4.50	8.7	6.20	8.1	7.40	42.0
1.50	20.7	2.70	32.1	4.60	11.1	6.30	9.7	7.50	40.3
1.60	16.2	2.80	28.9	4.70	13.6	6.40	11.6	7.60	38.5
1.70	13.0	2.90	25.7	4.80	16.5	6.50	13.9	7.70	36.6
1.80	10.2	3.00	22.3	4.90	19.4	6.60	16.4	7.80	34.5
1.90	8.1	3.10	18.8	5.00	22.6	6.70	19.3	7.90	32.0
2.00	6.5	3.20	15.7	5.10	25.5	6.80	22.4	8.00	29.2
2.10	5.1	3.30	12.9	5.20	28.8	6.90	25.9	8.10	26.2
2.20	3.9	3.40	10.4	5.30	31.6	7.00	29.1	8.20	22.9
		3.50	8.2	5.40	34.1	7.10	32.1	8.30	19.9
		3.60	6.3	5.50	36.6	7.20	34.7	8.40	17.2
		3.70	4.5	5.60	38.8	7.30	37.0	8.50	14.7
		3.80	2.9	5.70	40.6	7.40	39.1	8.60	12.2
		3.90	1.4	5.80	42.3	7.50	41.1	8.70	10.3
		4.00	0.1	5.90	43.7	7.60	42.8	8.80	8.5
						7.70	44.2	8.90	7.0
						7.80	45.3	9.00	5.7
						7.90	46.1		
						8.00	46.7		

F*		G*		H*		I*		J*	
pH	z	pH	z	pH	z	pH	z	pH	z
8.00	20.5	9.20	0.9	9.60	5.0	10.90	3.3	12.00	6.0
8.10	19.7	9.30	3.6	9.70	6.2	11.00	4.1	12.10	8.0
8.20	18.8	9.40	6.2	9.80	7.6	11.10	5.1	12.20	10.2
8.30	17.7	9.50	8.8	9.90	9.1	11.20	6.3	12.30	12.8
8.40	16.6	9.60	11.1	10.00	10.7	11.30	7.6	12.40	16.2
8.50	15.2	9.70	13.1	10.10	12.2	11.40	9.1	12.50	20.4
8.60	13.5	9.80	15.0	10.20	13.8	11.50	11.1	12.60	25.6
8.70	11.6	9.90	16.7	10.30	15.2	11.60	13.5	12.70	32.2
8.80	9.6	10.00	18.3	10.40	16.5	11.70	16.2	12.80	41.2
8.90	7.1	10.10	19.5	10.50	17.8	11.80	19.4	12.90	53.0
9.00	4.6	10.20	20.5	10.60	19.1	11.90	23.0	13.00	66.0
9.10	2.0	10.30	21.3	10.70	20.2	12.00	26.9		
		10.40	22.1	10.80	21.2				
		10.50	22.7	10.90	22.0				
		10.60	23.3	11.00	22.7				
		10.70	23.8						
		10.80	24.25						

- *A. 25 ml of 0.2 molar KCl + x ml of 0.2 molar HCl.
 *B. 50 ml of 0.1 molar potassium hydrogen phthalate + x ml of 0.1 molar HCl.
 *C. 50 ml of 0.1 molar potassium hydrogen phthalate + x ml of 0.1 molar NaOH.
 *D. 50 ml of 0.1 molar potassium dihydrogen phosphate + x ml 0.1 molar NaOH.
 *E. 50 ml of 0.1 molar tris(hydroxymethyl) aminomethane + x ml of 0.1 M HCl.
 *F. 50 ml of 0.025 molar borax + x ml of 0.1 molar HCl.
 *G. 50 ml of 0.025 molar borax + x ml of 0.1 molar NaOH.
 *H. 50 ml of 0.05 molar sodium bicarbonate + x ml of 0.1 molar NaOH.
 *I. 50 ml of 0.05 molar disodium hydrogen phosphate + x ml of 0.1 molar NaOH.
 *J. 25 ml of 0.2 molar KCl + x ml of 0.2 molar NaOH.
 Final Volume of Mixtures = 100 ml

BUFFER SOLUTIONS (Continued)
STANDARD VALUES OF pH AT TEMPERATURE 0-95°C

Temperature	Tetroxalate 0.05 molal	Tartrate 0.0341 molal (sat'd at 25°C)	Phthalate 0.05 molal	Phosphate ^a	Phosphate ^b	Borax 0.01 molal	Calcium hydroxide (sat'd at 25°C)
0	1.666	4.003	6.984	7.534	9.464	13.423
5	1.668	3.999	6.951	7.500	9.395	13.207
10	1.670	3.998	6.923	7.472	9.332	13.003
15	1.672	3.999	6.900	7.448	9.276	12.810
20	1.675	4.002	6.881	7.429	9.225	12.627
25	1.679	3.557	4.008	6.865	7.413	9.180	12.454
30	1.683	3.552	4.015	6.853	7.400	9.139	12.289
35	1.688	3.549	4.024	6.844	7.389	9.102	12.133
38	1.691	3.548	4.030	6.840	7.384	9.081	12.043
40	1.694	3.547	4.035	6.838	7.380	9.068	11.984
45	1.700	3.547	4.047	6.834	7.373	9.038	11.841
50	1.707	3.549	4.060	6.833	7.367	9.011	11.705
55	1.715	3.554	4.075	6.834	8.985	11.574
60	1.723	3.560	4.091	6.836	8.962	11.449
70	1.743	3.580	4.126	6.845	8.921
80	1.766	3.609	4.164	6.859	8.885
90	1.792	3.650	4.205	6.877	8.850
95	1.806	3.674	4.227	6.886	8.833

^a Solution 0.025 m KH_2PO_4 and 0.025 m Na_2HPO_4 .

^b Solution 0.008695 m KH_2PO_4 and 0.03043 m Na_2HPO_4 .

APPROXIMATE pH VALUES

The following tables give approximate pH values for a number of substances such as acids, bases, foods, biological fluids, etc. All values are rounded off to the nearest tenth and are based on measurements made at 25° C. A few buffer systems with their pH values are also given.

From Modern pH and Chlorine Control, W. A. Taylor & Co., by permission

ACIDS			
Hydrochloric, N.....	0.1	Oxalic, 0.1N.....	1.6
Hydrochloric, 0.1N.....	1.1	Tartaric, 0.1N.....	2.2
Hydrochloric, 0.01N.....	2.0	Malic, 0.1N.....	2.2
Sulfuric, N.....	0.3	Citric, 0.1N.....	2.2
Sulfuric, 0.1N.....	1.2	Formic, 0.1N.....	2.3
Sulfuric, 0.01N.....	2.1	Lactic, 0.1N.....	2.4
Orthophosphoric, 0.1N.....	1.5	Acetic, N.....	2.4
Sulfurous, 0.1N.....	1.5	Acetic, 0.1N.....	2.9
Acetic, 0.01N.....	3.4		
Benzoic, 0.01N.....	3.1		
Alum, 0.1N.....	3.2		
Carbonic (saturated).....	3.8		
Hydrogen sulfide, 0.1N.....	4.1		
Arsenious (saturated).....	5.0		
Hydrocyanic, 0.1N.....	5.1		
Boric, 0.1N.....	5.2		
BASES			
Sodium hydroxide, N.....	14.0	Lime (saturated).....	12.4
Sodium hydroxide, 0.1N.....	13.0	Trisodium phosphate, 0.1N.....	12.0
Sodium hydroxide, 0.01N.....	12.0	Sodium carbonate, 0.1N.....	11.6
Potassium hydroxide, N.....	14.0	Ammonia, N.....	11.6
Potassium hydroxide, 0.1N.....	13.0	Ammonia, 0.1N.....	11.1
Potassium hydroxide, 0.01N.....	12.0	Ammonia, 0.01N.....	10.6
Sodium metasilicate, 0.1N.....	12.6	Potassium cyanide, 0.1N.....	11.0
Magnesia (saturated).....	10.5		
Sodium sesquicarbonate, 0.1M.....	10.1		
Ferrous hydroxide (saturated).....	9.5		
Calcium carbonate (saturated).....	9.4		
Borax, 0.1N.....	9.2		
Sodium bicarbonate, 0.1N.....	8.4		
BIOLOGIC MATERIALS			
Blood, plasma, human.....	7.3-7.5	Gastric contents, human.....	1.0-3.0
Spinal fluid, human.....	7.3-7.5	Duodenal contents, human.....	4.8-8.2
Blood, whole, dog.....	6.9-7.2	Feces, human.....	4.6-8.4
Saliva, human.....	6.5-7.5	Urine, human.....	4.8-8.4
FOODS			
Apples.....	2.9-3.3	Gooseberries.....	2.8-3.0
Apricots.....	3.6-4.0	Grapefruit.....	3.0-3.3
Asparagus.....	5.4-5.8	Grapes.....	3.5-4.5
Bananas.....	4.6-4.7	Hominy (lye).....	6.8-8.0
Beans.....	5.0-6.0	Jams, fruit.....	3.5-4.0
Beers.....	4.0-5.0	Jellies, fruit.....	2.8-3.4
Beets.....	4.9-5.5	Lemons.....	2.2-2.4
Blackberries.....	3.2-3.6	Limes.....	1.8-2.0
Bread, white.....	5.0-6.0	Maple syrup.....	6.5-7.0
Butter.....	6.1-6.4	Milk, cows.....	6.3-6.6
Cabbage.....	5.2-5.4	Olives.....	3.6-3.8
Carrots.....	4.9-5.3	Oranges.....	3.0-4.0
Cheese.....	4.8-6.4	Oysters.....	6.1-6.6
Cherries.....	3.2-4.0	Peaches.....	3.4-3.6
Cider.....	2.9-3.3	Pears.....	3.6-4.0
Corn.....	6.0-6.5	Peas.....	5.8-6.4
Crackers.....	6.5-8.5	Pickles, dill.....	3.2-3.6
Dates.....	6.2-6.4	Pickles, sour.....	3.0-3.4
Eggs, fresh white.....	7.6-8.0	Pimento.....	4.6-5.2
Flour, wheat.....	5.5-6.5	Plums.....	2.8-3.0
Potatoes.....	5.6-6.0		
Pumpkin.....	4.8-5.2		
Raspberries.....	3.2-3.6		
Rhubarb.....	3.1-3.2		
Salmon.....	6.1-6.3		
Sauerkraut.....	3.4-3.6		
Shrimp.....	6.8-7.0		
Soft drinks.....	2.0-4.0		
Spinach.....	5.1-5.7		
Squash.....	5.0-5.4		
Strawberries.....	3.0-3.5		
Sweet potatoes.....	5.3-5.6		
Tomatoes.....	4.0-4.4		
Tuna.....	5.9-6.1		
Turnips.....	5.2-5.6		
Vinegar.....	2.4-3.4		
Water, drinking.....	6.5-8.0		
Wines.....	2.8-3.8		