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TREATABILITY STUDY WORK PLAN FOR OPERABLE UNIT 1 REMEDY DESIGN LABORATORY STUDIES VITRIFICATION PART 1B: WORK PLAN APPENDICES

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APPENDICES

TREATABILITY STUDY WORK PLAN FOR OPERABLE UNIT 1

4047

FERNALD ENVIRONMENTAL MANAGEMENT PROJECT
FERNALD, OHIO

REMEDY DESIGN LABORATORY STUDIES

VITRIFICATION

Part 1B: WORK PLAN APPENDICES

GTS DURATEK/CATHOLIC UNIVERSITY OF AMERICA

NOVEMBER, 1992

U.S. DEPARTMENT OF ENERGY

01

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Receiving, Shipping and Handling of glasses and other radioactive samples

Glass Characterization

Leach Test Procedures

Analytical Procedures

Procedure for Characterization of Physical Properties and Carbon Content of Wastes and Glass

Gamma Counting

Procedure for TCLP Tests

Procedure for Analysis of TCLP Extracts

Melt Conductivity Determination

UNCONTROLLED

VSL Technical Procedure

Title: Glass Preparation and Minimelter

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Glass Preparation and Minimelter

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DOCUMENT REPLACEMENT RECORD
Glass Preparation and Minimelter
Rev #0
GLASS PREPARATION

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VSL-GP-1. WVN	Feed Preparation for Continuous Melter	4	Section 6.5
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VSL-GP-4.WVN	Sludge Assay for Continuous Melter Batch Preparation	1	Section 6.3
VSL-GP-5.WVN	Procedure for Making Radioactive Glass Bars	4	Section 6.7
VSL-GP-6.WVN	Procedure for Making Small Melts	5	Section 6.8
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VSL-MH-3.WVN	Calibration Procedure for the O-Haus 310 Balance	4	Section 5.2.1
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GLASS PREPARATION AND MINIMELTER PROCEDURES

1.0 PURPOSE

This procedure describes methods, materials, equipment and special conditions required to prepare the glassy material and its sampling into different geometry. This procedure provides the means for obtaining verifiable, reproducible, documented data from tests and experiments.

2.0 APPLICABILITY

This procedure applies to all employees trained and authorized to prepare the glassy material and its sampling into different geometry in the VSL. Specific activities covered by this procedure include:

(a) Determination of the volatile content of ingredients used in the preparation of glasses.

(b) Assaying of the ferric sludge used in the continuous melter feed.

(c) Preparation of the feed for the continuous melter.

(d) Operation of the continuous melter ("minimelter").

(e) Casting of crucibles of glass produced by the minimelter into glass bars.

(f) Preparation and melting of small melts of glass, when a small adjustment of composition compared to the minimelter production is necessary.

(g) Grinding of glass bars.

(h) Calibration of balances, sieves.

(i) Calibration of thermometers and thermocouples.

This procedure will be implemented by personnel from the VSL.

3.0 SAFETY

3.1 HAZARDS

3.1.1 Chemical Hazards.

The definition of a hazardous chemical is given in Section 8.3 of the CUA Chemical Materials Safety Manual, adopted from the OSHA Hazard Communication Standard. The CUA Chemical Materials Safety Manual is hereafter referred to as the CCMSM. This Standard and its application to CUA facilities are described in Sections 8.1 of the CCMSM, with the exceptions noted in Section 8.28.

3.1.2 Radioactive Hazard.

(a) radioactive isotopes used in the preparation of glasses; (b) a potential radioactive hazard exists on handling radioactive sludge; (c) all radioactive glass samples (bars and powder).

3.2 HAZARD MITIGATIONS

3.2.1 Identification of Hazards

The identification of chemical hazards should include an education program (Section 8.2 of the CCMSM). This program should include familiarization with the CCMSM as a whole, including the toxic properties of chemicals according to Sections 8.22, 8.23, 8.24 and 8.25 of the CCMSM, other hazards of chemicals classified under Sections 8.9, 8.10, 8.11, 8.12, 8.13 and 8.14 of the CCMSM, chemical handling and storage procedures (see paragraph 3.2.2 below), and emergencies (see paragraph 3.2.3 below). The education program will also include familiarization with the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University.

Other elements of hazard identification include the proper use of labels (Section 8.4 of the CCMSM) and of Material Safety Data Sheets (Sections 8.5, 8.26 and 8.27 of the CCMSM). Workplace chemical lists should be compiled, maintained, and made available for employees in accordance with Section 8.31. In addition, a copy of the CCMSM should be kept in the laboratory and its use should be included in the education program cited above. The same should apply to the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University.

3.2.2 Handling and Storage of Chemicals

Handling of chemicals should be conducted according to Section 8.6 of the CCMSM. Storage of chemicals should follow Section 8.8 of the CCMSM. Special precautions should be taken in handling and storage of incompatible chemicals, solvents, compressed gases, and radioactive materials according to Sections 8.10, 8.12, 8.13, and 8.14 of the CCMSM, respectively. Unstable chemicals according to Section 8.9 should be excluded from the glass melting laboratory except in cases where the introduction of such a material is required by the project manager. Such a requirement should be made in writing and specify the maximum quantity of the material and the maximum period it may be stored before being discarded. Shock-sensitive chemicals according to Section 8.11 should never be introduced into the laboratory under any circumstances. No work requiring the use of restricted areas according to Section 8.15 should be performed within the glass melting laboratory.

Waste disposal of hazardous chemicals should follow procedures established by the CUA Environmental safety Office according to Section 8.16 of the CCMSM.

3.2.3 Emergencies

Chemical spills should be dealt with according to Section 8.17 of the CCMSM. Fire and fire-related emergencies should be controlled according to Section 8.21 of the CCMSM. Cases of personal contamination should be treated according to Section 8.19 of the CCMSM, cases of injury and illness according to Section 8.18, and cases requiring minor first aid according to Section 8.20.

3.2.4 Radioactive Hazard Mitigation

All rules for control and monitoring of personnel exposure and environmental radiation are described in Chapter 3 of the radiation safety manual of The Catholic University of America.

Application of these rules is the responsibility of each user working in the restricted area (rooms 37 to 40 of Hannan Hall) and the project managers, under the supervision of Dr. Warren E. Keene, Radiation Safety Officer of CUA.

3.3 PERSONNEL PROTECTION

Personnel protection should consist of two elements. The first is familiarization and compliance with the instructions of the CUA Chemical Materials Safety Manual (CCMSM), including identification of hazards (see paragraph 3.2.1 above), handling and storage of chemicals (see paragraph 3.2.2 above), and dealing with emergencies (see paragraph 3.2.3

above). The second element is familiarization and compliance with the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University. The overall chemical hygiene program (Section 8.34 of the CCHP), based on the OSHA Regulation on Occupational Exposure to Hazardous Chemicals in Laboratories, should be followed. In detail, exposure reduction measures (Section 8.35 of the CCHP) will include environmental monitoring (8.35.1), inspections, maintenance and housekeeping (8.35.2), use of protective apparel and equipment (8.35.3), posting of signs and labels (8.35.4), and dealing with accidents and spills (8.35.5). Ventilation of both general laboratory spaces and of special areas and local devices such as chemical fume hoods and glove boxes should follow the instructions of Section 8.36 of the CCHP. Employees should take part in the safety information and training program which exists in the University according to Section 8.37 of the CCHP. The glass melting laboratory should not conduct operations with hazardous substances which require a special review of laboratory activities under Section 8.38 of the CCHP, viz. reproductive toxins, chemicals which have a high degree of acute toxicity, and chemicals considered select carcinogens. Employees should be informed about the medical program provided by the University in cases of work with hazardous substances according to Section 8.39 of the CCHP. Responsibility for chemical hygiene rests with all employees. Specific chemical hygiene responsibilities as applied to the glass melting laboratory are specified in Section 8.40 of the CCHP. Glass melting laboratory workers should be familiar with the basic rules and procedures for working with chemicals included in Section 8.41 of the CCHP and should work in full compliance with these rules and procedures. No work with chemicals of high chronic toxicity according to Section 8.41.4 of the CCHP should be carried out in the glass melting laboratory. Animal work, whether involving chemicals of high chronic toxicity according to Section 8.41.5 of the CCHP or not involving chemicals of this type, should not be carried out in the glass melting laboratory.

Aside from compliance with the general instructions described in the CUA radiation safety manual, the following rules apply to the protection of personnel working in the restricted area of the Vitreous State Laboratory:

1) All personnel working in the restricted area shall routinely undergo radiation safety training given by the radiation safety officer of CUA or provided at either Georgetown University or NIH or NIST (see section 4.1).

2) The restricted area should remain locked and only workers assigned to this area, and project managers should have access to this section. All other persons who need temporary access to the radioactive laboratory shall be accompanied by authorized personnel.

3) Film badges shall be worn by operation personnel upon entering the restricted area. Ring badges and rubber gloves shall be worn when handling radioactive material.

4) Shoe covers are to be worn in the melter room at all times.

5) Any spills must be cleaned up immediately, checked after cleaning with a survey meter, and the radiation safety officer shall be notified as soon as possible so that wipe tests may be performed. Immediately notify RSO of any unenclosed spills greater than about 100 ml.: phone 5206, home (703) 671-1659.

6) All samples shall be labelled with sample name, date and notebook/page of record indicating sample preparation. All new materials shall be entered into the inventory control file. Any material not in current use must be returned to the storage area in Room 40.

7) Respirators are to be worn as backup protection for operations involving the possible release of contaminated dust.

8) Repairs to equipment involving hazardous operations should not be performed unless under the direction and in the presence of one or more of the following: R. Mohr, H. Hojaji, I. Muller, W. Keene, P. B. Macedo. If alone and a hazardous situation occurs do only what is necessary to prevent a further hazard.

Refer to section II for emergency response. Notify Bob Mohr (202) 234-5953, Hamid Hojaji (301) 365-0999, Isabelle Muller (703) 534-5420 or Pedro Macedo (301) 229-0408 to obtain assistance. Notify Campus security and or the fire department of fire or serious accident. Security 5111. Fire and emergency dial 9 for outside line then 911.

4.0 REQUIREMENTS

4.1 TRAINING

All radioactive restricted area workers and, especially, all glass melter workers shall be trained both by the laboratory supervisor regarding their specific duties and by the radiation safety officer of campus.

Documents 10 CFR 19 and 10 CFR 20 are available to all restricted area workers.

Each restricted area worker must attend a course in radiation safety given at NIST, NIH or Georgetown University - College of Health Physics.

Training on each technical method and equipment used in the glass melting laboratory is given by the laboratory supervisor and/or by a trained and experienced person designated by the laboratory supervisor until the technician shows competence to use that method independently of direct supervision.

5.0 PREREQUISITES

5.1 EQUIPMENT LISTS

All equipment used for glass preparation and melter operation is listed on the following pages with notes indicating calibration requirements. Similar equipment has been grouped into the following categories: balances, furnaces, material handling, meters, and temperature devices.

BALANCES

Qty	Device	Manufacturer	Model No.	Calib. Req.
1	45kg Platform Scale	Circuits & Systems	BX100, #3422	I
1	1.2kg Balance	Mettler	PL1200 (=A11138)	III*
1	Dial-O-Gram Balance	Ohaus	310	II
1	Balance	Scientech	5200	III
1	Calibration Weights	Ohaus	50g, 100g, 200g	
1	Std. Weight, Class S	Fisher	1g, 20g	
1	Std. Weight, Class S	Troemner	1.5Kg	
1	Std. Weight, Class S	Troemner	10Kg	
1	Std. Weight, Class S	Troemner	20Kg	
1	Nominal Weight	Troemner	55lbs	

Calibration codes: QM= Qualitative Measurements Only; I= ± 0.1kg accuracy, every 6 months or as needed; II= ± 0.1 g accuracy, every 6 months or as needed; III= ± 0.01g accuracy, annually; *= calibrated by manufacturer representative

FURNACES

Qty	Device	Manufacturer	Model No.	Calib. Req.
1	Furnace	Deltech	DT-28-6	I

1	Furnace	Deltech	DT-28-12	I
1	Furnace	VSL	5" round	N.A.
1	Gold Furnace	Trans. Temp. Co.		N.A.
2	Laboratory Box Furnace	Neytech	85M	N.A.

Calibration codes: I= Thermocouple and controller unit calibrated every 6 months

MATERIAL HANDLING

Qty	Device	Manufacturer	Model No.	Calib. Req.
1	Brinell Microscope	Bausch & Lomb	20X VL6931	N.A.
1	Crucible, Cr_2O_3 or porcelain			N.A.
1	Crucible, Pt-Au, 430g		175ml volume	N.A.
1	Crucible, Pt-Au, 2.2kg		875ml volume	N.A.
2	Crucible Tongs	Fisher	15-207	N.A.
	Fireclay Crucibles, 400g	A.P. Green	185ml volume	N.A.
	Fireclay Crucibles, 1.4kg	A.P. Green	#5, 500ml volume	N.A.
1	Glovebox	Hamilton		N.A.
1	Glovebox	VAC	HE 113-135	N.A.
1	Glovebox			N.A.
	Graphite molds	VSL		N.A.
1	Green Goggles	Norton	Cyclops II, 787	N.A.
1	Grinder	Retsch/Brinkman	ZM1 = 41109	N.A.
	1 gallon metal can w/lid			N.A.
1	Pump	Masterflex	NE-7523-10	QM
1	Pump	Little Giant	LG 102	N.A.
1*	Respirator	3M	6200 w/series 2000 cartridges	N.A.
	Sample Cups, polyprop.	Oxford	4oz	N.A.

TPI-GP

Qty	Device	Manufacturer	Model No.	Calib. Req.
1	Brinell Microscope	Bausch & Lomb	20X VL6931	N.A.
1	Sieve	Fisher	40 mesh	I
1	Sieve	Fisher	60 mesh	I
1	Sieve	Fisher	100	I
1	Sieve	Fisher	200	I
	Spatula			N.A.
1	S. Steel Stirrer			N.A.
1	Tin snips	Craftsman	X4279	N.A.
	Weighing buckets			
	Weighing dishes			

• One worn by each operator involved in dust hazard procedure
Calibration codes: QM= Qualitative use only I= replaced every 100 uses, new ones calibrated before 1st use unless delivered by manufacturer with certificate of accordance with specifications outlined in ASTM E-11, USA Standard Z23.1.

METERS

Qty	Device	Manufacturer	Model No.	Calib. Req.
2	Ampmeter	Triplet	330-G	QM
2	Ampmeter	Allied		QM
1	Clip-On Ampmeter	Amprobe	RS-1, #BB240928	QM
1	DC Millivoltmeter	Digitec	268	None req'd.
4	Magnehelic manometer	Dwyer	2302	QM
1	Microvoltmeter	Keithley	177	I
1	Multimeter	BK Precision	2703	QM
1	Survey Meter	Eberline	SK-1	II

Calibration codes: I= Bi-Annually, calibrated externally; II= Annually by Radiation Safety Office

TEMPERATURE DEVICES

Qty	Device	Manufacturer	Model No.	Calib. Req.
1	Digital Thermometer	Omega	2166A, #8503	I
1	Digital Thermometer	Fluke	52	II
1	Programmer	Love	105	N.A.
1	Temp. Controller	Love	74, #06872	C
1	Thermometer		20 to 260°C	QM
1	Type K Thermocouple			none req'd.
4	Type S Thermocouples			IV
	Type S (Pt-Pt 10%Rh) Measuring Thermocouple			III
1	Universal Digital Controller	Honeywell	UDC300	N.A.

Calibration codes: I= calibrated along with Type S thermocouples to be used as temp. probes. 3 channels in use, ± 25°C, every 6 months or as needed; II= every 6 months or as needed; III= calibrated externally at NIST, test #235603; IV= bi-annually or as needed; C= controlling device

5.2 EQUIPMENT CALIBRATION

Calibration procedures for equipment listed in 5.1 are given on the following pages. Equipment is grouped in the same categories as in 5.1 and is listed in the same order.

5.2.1 BALANCES

CALIBRATION OF CIRCUITS AND SYSTEMS #3422 PLATFORM SCALE

Calibration of this balance should be performed every six months.

Calibration data should be logged in the current Calibration Logbook.

Calibration procedure requires a set of 1.5 kg, 10kg and 20 kg standards, traceable to NIST, as welll as a 23 kg (50 lb) nominal weight.

1. Allow at least 5 minutes after turning on the power for warmup then zero the balance.

2. For the first calibration data point, place the 1.5 kg standard on the center of the pan. Record the weight and then remove it.

3. Second calibration data point: repeat step 2 using the 10 kg standard instead.

4. Third calibration data point: repeat step 2 using the 20 kg standard instead.

5. Fourth calibration data point: repeat step 2 by placing both the 10 kg and 20 kg standards together for weighing.

6. The balance is now calibrated for the range 1.5 to 30 kg. Place the nominal 50 lb weight on the balance and record the reading.

7. Fifth calibration data point: place the 10 kg and 50 lb weights on the center of the pan and record the weight.

8. Sixth calibration point: place the 20 kg and 50 lb weights on the center of the pan and record the weight.

9. Prepare a new sticker giving date of calibration and due date of next calibration and give Logbook/page reference for the calibration data. Due date for the next calibration shall be 6 months from date of the just performed calibration. Sign calibration label and place it on the balance.

10. If the instrument does not meet the calibration requirements, as specified in the above Table, the laboratory supervisor and/or the factory should be consulted for corrective measures.

CALIBRATION OF O HAUS 310 DIAL-O-GRAM BALANCE

Note: Enter data in the current Equipment Calibration Log.

1. Clean the balance and check the tare with the weighing cup in place, adjusting if necessary.

2. Transfer the 50g, 100g and 200g calibration weights into the glove box via the pass through.

3. 1st point, 50g: Weigh the 50g weight on the scale and record the value.

4. 2nd point, 100g: Remove the 50g weight and weigh the 100g weight on the scale and record the value.

5. 3rd point, 150g: Add the 50g to the scale and record the value.

6. 4th point, 200g: Clear the scale, weigh the 200g weight and record the value.

7. 5th point, 250g: Add the 50g weight to the loaded scale and record the value.

8. 6th point, 300g: Remove the 50g weight from the scale, add the 100g weight to the scale and record the value.

9. Plot a graph of the observed values vs. the calibration weight values (actual values). Place the actual values on the horizontal axis.

10. The balance is considered within the required calibration if the values are within 1% of the standard calibration values. If the balance is out of calibration, repair or replace as is required.

11. If calibration is complete, fill out a VSL calibration sticker with the date of calibration, person who performed the calibration and date the next calibration is due. Place the sticker on the glove box window edge above the balance.

CALIBRATION OF SCIENTECH 5200 BALANCE

Note: Enter data in current Crucible Melts logbook. Two Fisher class S standard weights are needed for this procedure.

1. Turn-on balance, wait until self-test is completed and **0.00g** is displayed. If 0.00g is not displayed, press RANGE button to select HI range.

2. Place 1g standard weight on balance and record displayed weight in logbook.

3. Remove 1g standard weight on balance and replace with 20g standard weight. Record displayed weight in logbook.

4. Weights should not differ from standard values by more than 0.01g. If instrument is outside this range, the manufacturer should be consulted at:

   Scientech, Inc. (303) 444-1361
   5649 Arapahoe Ave. (800) 525-0522
   Boulder, CO 80303

5. If calibration is complete, fill out a VSL Calibration sticker and place on glovebox window.

5.2.2 CALIBRATION OF SIEVES

Note: Sieves are replaced after 100 samples of glass (250g or less) have been sieved. Each sieving is recorded in the last section of the current Glass Grinding log. Before new sieves are used they are calibrated by the following procedure. The Bausch & Lomb Brinnel Microscope is required for this procedure. The purpose of this procedure is to accurately measure and record the average dimensions of the mesh holes on a particular sieve. Though a brinnel microscope is referred to in the procedure, any other method of accurate inspection can be used.

1. Place each sieve on an overhead projector, light-table or other light source where they can be examined from above while illuminated from below.

2. Visually inspect the entire surface of the sieve in order to observe if the mesh size is approximately uniform over the whole sieve.

3. Randomly select 5 positions on the surface of the sieve and measure their dimensions according to the reticle scale marks in the scope. The scale measures 6mm in the microscope's field of view, marked in 0.1mm increments.

4. For each position, count the number of spaces (holes) in a 6mm (for 40, 60 and 100 mesh sieves) or 1mm (for 200 mesh sieve) section of the mesh. Record this value in the logbook. Then measure the diameter of the mesh wire and record this value also. Repeat this for each position (5 times in all).

5. Calculate the average number of spaces counted over the 5 positions.

6. Divide the length used (6mm = 6000μm or 1mm = 1000μm) by this average. This gives the dimension of 1 space plus 1 wire. Record this value (in μm) in the logbook.

7. Calculate the average wire diameter over the 5 positions. Convert the value from mm to μm by multiplying by 1000 and subtract this value from the result in step 6. This value is the width of one space in the sieve mesh. Record this in the logbook.

8. Repeat for each sieve. Sieves are now ready for use. And can be permanently placed in the grinding glovebox in room 39.

5.2.3 TEMPERATURE DEVICES

CALIBRATION OF DIGITAL THERMOMETERS AND TYPE S THERMOCOUPLES

Note: The Gold Furnace, controller and associated thermocouple, the Keithley 177 Microvoltmeter, and NIST calibrated Type S Measuring Thermocouple are needed for this procedure. During the calibration procedure, 3 thermocouples will be inside the Gold Furnace at the same time. These are the *control thermocouple* (connected to furnace controller), the *NIST thermocouple* and the *test thermocouple* (thermocouple being calibrated).

The cold junction of the NIST thermocouple should be immersed in an ice bath to a depth of 1 inch at all times.

1. Insert the control and NIST thermocouples in the furnace to immersion depths where the cross-section of the furnace possesses a uniform axial temperature profile. (Refer to Laboratory Notebook VSL-86-12 for details.)

2. Immerse Reference junction of NIST thermocouple in ice/water bath. Be sure that unmelted ice is always present in contact with the reference junction during the entire calibration procedure.

3. Insert the test thermocouple into the same end of the furnace as the NIST thermocouple. It should be parallel to the NIST thermocouple and inserted to the same depth.

4. Connect the NIST thermocouple to the microvoltmeter.

5. Connect the test thermocouple to the digital thermometer or controlling device it is being calibrated with. The two will always be calibrated as a unit. If the thermometer is a multi-channel unit, it is best to connect the test thermocouple to the channel it will be used with.

6. If a thermocouple is being calibrated on its own, connect it to the same microvoltmeter as the NIST thermocouple. Alternate between the two when taking readings. Using a thermometer record ambient temperature at the voltmeter terminals and use this to correct voltmeter reading with the aid of standard tables.

7. Set the furnace temperature to the desired value and turn on the power.

8. Wait about 10 minutes until the temperature is allowed to equilibrate.

9. Take 3 readings each from the test and NIST thermocouples at 5 minute intervals.

10. If more than one thermocouple is to be tested place the next thermocouple to be tested in the furnace, allow the temperature to equilibrate and repeat step 9.

11. Set the furnace to the next desired temperature and proceed with steps 8 through 10.

12. Repeat the calibration for a total of 4 temperatures that include the temperature range required.

13. At each temperature setting compare the temperatures corresponding to the readouts from the reference and the test thermocouples. Use the calibration table for the NIST thermocouple to convert its microvoltmeter readings into temperatures. Use standard tables, (e.g., in Omega Temperature Handbook) if necessary to convert test thermocouple readings into temperatures (i.e. if the test thermocouple is not being calibrated with a digital thermometer as a unit. If the two temperature measurements deviate by more than 10°C consult the Project Manager.

6.0 MINIMELTER AND GLASS PREPARATION PROCEDURES

6.1 OVERVIEW

All procedures used in minimelter operation and glass preparation are described in this section (6.2 through 6.9). The following summary is given to provide a clearer picture of the order and relationships between the detailed procedures given in 6.3 through 6.9.

I. Sludge Assay (6.3)

The proper proportion of Ferric Hydroxide sludge in a feed recipe cannot be calculated until the actual water content or dry material content of the sludge has been determined. This value varies over time as the solid material in the sludge tends to settle in the sludge drum over long periods of storage. Therefore an assay must be conducted each time a feed recipe is made.

II. Det. of Volatile Content of Ingredients (6.4)

Ingredients and reagents used in a recipe may lose volatiles when heated to melt or decomposition temperatures. Therefore the volatile content of all ingredients and reagents used in a feed recipe are determined before the actual recipe is calculated.

III. Feed Preparation (6.5)

After steps I and II have been completed and a recipe has been calculated, the actual feed is mixed. The non-radioactive feed is prepared first by mixing weighed-out sludge with ingredients and reagents. After a portion of the non-radioactive feed has been transferred into the glovebox in the melter room proportional amounts of Uranium and Thorium oxide are added, completing the recipe.

IV. Minimelter Operation (6.6)

Prepared radioactive feed from III is fed from the glovebox into the melter and glass is produced. Initially, a calculated amount of waste glass is produced and discarded to cope with the turnover of glass composition in the melt chamber. Subsequent glass, in filled crucibles, is then stored for later remelting. This glass

product will be referred to as "WVCM XX", where XX is the order number of the melt.

V. Casting Radioactive Glass Bars (6.7)

Glass produced in the minimelter in step IV or glass mixed for small melts in step VI are cast into bars for ease of use. For the minimelter product, filled crucibles are removed from storage, reheated and the glass is poured into graphite molds. For small melts, pre-mixed powdered ingredients are melted and stirred and the product poured into graphite molds.

VI. Making Small Melts (6.8)

Variations of the composition of glass produced in step IV in large batches can be achieved and studied by using a fraction of the produced glass as a basis (generally 90%) for new recipes (small melts). It is mixed with ingredients and reagents according to a new recipe and melted in one of the smaller furnaces. The glass sample will be referred to as "WVUTh XX", where XX is the order number.

VII. Radioactive Sample Grinding (6.9)

Glass powder is required both for characterization and for making small melts. Therefore glass bars that have been cast must be broken up, ground, and, if necessary, sieved (graded).

6.2 GLOVEBOX OPERATION

Three glove boxes are available to glass preparation activities:

- A large double workstation (4 gloves) in the minimelter room
- A single station glovebox dedicated to grinding and sieving
- A single station glovebox dedicated to weighing and mixing powders

Glove Boxes will be operated under slight negative pressure (sufficient to inflate the gloves). The pressure drop should always be at a minimum of -0.2 inches of water. The suction is provided by a line connected to the exhaust duct between the 80% filter and the room HEPA filter through a ball valve connected to the glove box. Each glovebox is equipped with its own HEPA filter, to be changed when the pressure drop is too low. The box is equipped with a chamber for passing materials in and out of the box. The chamber is equipped with inner and outer doors.

6.2.1 Glovebox Operation Procedure

1. Pre-Gloves (latex or vinyl) should always be worn when using a glovebox. Materials should be passed into and out of the GLOVEBOX via the following procedure:

   a. to bring objects INTO the glovebox, first insure that the inside door of the pass-through is sealed and there is negative pressure inside the box.

   b. unlatch the outside door of the pass-through and open it, placing the object(s) inside the pass-through chamber.

   c. close and latch the outside door.

   d. open the inside door and move object(s) from the pass-through chamber into the glovebox.

   e. close the inside door. During normal operation, both doors should remain closed.

   f. to REMOVE objects from inside the glovebox, make sure the outer door is closed and open the inner door.

   g. place object into the pass-through chamber and close inner door, making sure the door is properly sealed and the inside of the glovebox is under negative pressure.

   h. open the outer door and remove object. Pre-Gloves should be worn when handling objects removed from the glovebox. Objects removed from the glovebox should be bagged, rinsed or thrown-out into rad waste.

6.2.2 Checking Glovebox Gloves

1. Make sure gloves are completely inside glovebox and ballooned out as much as possible. If glovebox is equipped with a booster power, turning this on may help balloon gloves.

2. Fill gloves with tap water (as much as possible) and let sit for at least 10 minutes.

3. Insert a latex-gloved hand into the glove. Examine gloves through window, looking for dark water spots that indicate pinhole leaks.

4. To drain water from glove, place on empty bucket or other suitable container under gloveport and pull glove out into container.

5. Visually inspect port ring where glove is clamped to glovebox for tears or breakage.

6. If leaks are detected in step 3 or tears are found in step 5, replace the glove in question.

7. Glove boxes should be checked every three months following the above steps.

6.3 SLUDGE PREPARATION

The term *sludge* will apply to either Ferric Hydroxide sludge used to prepare batch recipes or radioactive sludge received from waste sites. The term *slurry* applies to a mixed recipe batch (often consisting of sludge and additives) ready to be fed into a continuous feed melter.

6.3.1 $Fe(OH)_2$ Sludge Assay For Continuous Melter Batch Preparation

Note: Record each preparation in the *WVCM Mix Book Log Notebook*

1. After measuring out the required amount of well stirred Ferric Hydroxide sludge, scoop out about 120 ml in a 4 oz sample cup.

2. Label a clean crucible without chips or cracks, weigh it and record the measurement.

3. Weigh a clean, dry 100 ml graduated cylinder, record weight.

4. Shake the sample well and pour 100 ml $\pm$ 5% into the graduated cylinder. Record to 0.5 ml.

5. Record the weight of the graduated cylinder plus sample.

6. Pour the sludge into the crucible and rinse the graduated cylinder with 10-15 ml deionized water and pour this into the crucible also.

7. Put the crucible and sludge in the drying oven (set to 90 $\pm$ 5 °C) for a few hours, or overnight to dry.

8. Put the dried sludge and crucible in the furnace (set to 800 $\pm$ 50 °C) for one hour.

9. Remove the crucible from the furnace and allow it to cool for at least one half hour, weigh it and record the final weight.

10. Subtract the weight of the empty graduated cylinder from the filled weight to obtain the weight of sludge.

11. Subtract the weight of the empty crucible from the final weight of the crucible and sludge to get the weight of the dried and fired sludge.

12. Record results and report them to the Project Manager.

6.3.2 Radioactive Sludge Drying and Assay

Note: If sample is newly received, make sure that procedures described in TPI.SH *Receiving, Handling, Storage and Shipment* Section 7.2, have been properly followed.

Make sure all implements used are thoroughly cleaned to avoid contamination of sample.

An assay should be performed to determine volatile contents of newly received sludge. Sludge is dried in bulk to be used in subsequent glass melts.

If you are running an assay, follow instructions below labeled **ASSAY**. If you are simply drying sludge, follow instructions below labeled **DRYING**. Instructions not specifically labeled are applicable to BOTH procedures.

1. Thoroughly mix sludge until homogeneous.

2. Weigh and label (sample name, date and empty weight) a clean, dry clay crucible. Record the weight in the Lab Notebook. For an ASSAY, use a small 400g clay crucible (unless otherwise specified by the Project Manager). For DRYING, use a large 1.4Kg crucible.

3. Place crucible in plastic bag (to minimize surface contamination during handling), pass into glovebox and fill to within 1" of rim with wet, mixed sludge.

4. Remove and weigh crucible. Record the wet weight of the sludge and crucible combined. Calculate and record the weight of total sample removed. Enter this value in the "Radioactive Material Chain of Custody Record." - Form # VSL - 16

5. Place crucible into an appropriate furnace (the large 1.4Kg crucibles will only fit in the Deltech DT-28-12 or the 5" Round furnace).

6. The nominal heating schedule is as follows but alternate heating schedules may be specified by the project manager.

   (i) Set furnace to 90 $\pm$ 5°C for 1 hour or until all free water has evaporated and sample appears dry.

   (ii) Set furnace to 450°C for 4 hours.*

7. Shut off furnace when final time is reached and allow sample to cool inside furnace.

   ASSAY: When sample has cooled to 110 ± 5°C, remove from furnace and place directly in desiccator to cool to room temperature.

8. When sample has cooled to room temperature, weigh and record the total dry weight of the sludge and crucible.

9. ASSAY: Calculate and record the percentage weight loss of the sample after drying.

10. Weigh and label (sample name, date, empty weight) a clean, sealable plastic jar and store the dry sludge inside.

6.4 DETERMINATION OF THE VOLATILE CONTENT OF INGREDIENTS USED IN THE PREPARATION OF GLASSES

1. Select a representative sample of the ingredient or reagent to be assayed.

   NOTE: An equivalent sample must be selected for chemical analysis of non-volatile components.

2. Add the sample to a weighed crucible. Determine the amount to yield about 1g for reagents or 10g to 100g for slurries.

   NOTE: For samples which are not expected to produce a stable stoichiometric compound after firing, additional reagents, as required, may be added. These will be given in individual instructions on a case by case basis.

3. Dry the crucible and contents in an oven if necessary to remove liquid water or water of hydration lost at low temperatures. Use an oven set at 90°C unless otherwise specified. The drying time will be given in individual instructions by the Project Manager.

4. Transfer the crucible and contents to the 5" round furnace (at room temperature) for heating. The nominal heating conditions are: heating rate of 4°C/min., to 800 $\pm$ 50 °C and hold for 1 hour.

   NOTE: Alternate heating schedules may be specified where appropriate.

5. Remove the crucible from the furnace and cool in a desiccator.

6. Weigh the crucible and contents to determine the weight loss. This is the volatile content in the original sample.

7. Write down the specific procedure and results in the *Crucible Melts Log Notebook.*

8. Include a copy of assay results in the chemical assays notebooks.

6.4.1 Thermogravimetric Analysis of Soils and Sludges

Note: If sample is newly received, make sure that procedures described in TPI.SH *Receiving, Handling, Storage and Shipment*, Section 7.2 have been properly followed.

Make sure that all implements used are thoroughly cleaned to avoid contamination of sample.

The analysis should normally be performed in a platinum crucible (Pt-Au or Pt-Rh) but clay crucibles may be used at the direction of the Project Manager. If using a platinum crucible ensure that no metallic objects come into contact with the hot crucible.

1. For sludges, thoroughly mix until homogeneous to ensure that a representative sample can be taken.

2. Weigh a clean dry crucible and record the weight in Lab Notebook. If a clay crucible is used, label it with the sample name, date, and empty weight.

3. Place crucible into a plastic bag, pass into glovebox and transfer into it approximately xx g of the soil or sludge to be analyzed.

4. Remove the crucible with sample from the glovebox, weigh, and record the weight. Calculate and record the weight of total sample removed. Enter this value in the "Radioactive Material Chain of Custody Record", form VSL-XXX.

5. Place crucible into furnace.

6. Set furnace to 90 ± 5°C for 1 hr, or until all free water has evaporated and sample appears dry.

7. At each analysis temperature, perform the following steps. The nominal sequence of temperatures and heating times is given below but alternative schedules may be specified by the Project Manager.

Temperature	Time
110°C	18 hrs
450°C	4 hrs
850°C	4 hrs
1150°C	4 hrs

(a) Set furnace to selected temperature and hold for appropriate period of time.

(b) Shut off furnace and allow sample to cool inside furnace.

(c) When sample has cooled to 110 ± 5°C remove from furnace and place in desiccator to cool to room temperature.

(d) Remove from desiccator and weigh and record total weight of dry sludge plus crucible.

(e) Calculate and record the percentage weight loss of the sample after drying.

(f) Repeat steps (a) through (e) for each scheduled temperature.

8. Weight and label (sample name, date, empty weight) a clean sealable plastic jar and store the final sample inside for any subsequent analysis (e.g. total chemical analysis).

6.5 FEED PREPARATION FOR THE CONTINUOUS MELTER

Note: Record non-radioactive portion of preparation in the *WVCM Mix Book Log Notebook*, and radioactive portion in the *Minimelter Records Log Notebook*

When weighing all chemicals, record the inventory number of the chemical (CUA# or VSL#) in the logbook next to the chemical name.

1. Weigh the clean, empty (dry) tank for mixing batch.

2. Add weight of sludge specified by the Project Manager, take sample and assay for solids content as per procedure in section 6.3.

3. Obtain remainder of recipe on basis of assay and target composition from the Project Manager.

4. Add specified weights of frit and non-radioactive chemicals.

5. Stir.

6. Split into two approximately equal batches, if necessary.

7. Transfer required amount of feed from large external tank to small tank in minimelt glovebox (weight determined by loss in weight of outside tank: ≈ 30 lbs ± 1%).

Record weight to ± 0.01 Kg

8. Weigh and add required amount of uranium and thorium oxides based on weight of slurry and recipe. Record to ± 0.1 gram.

6.6 MINIMELTER PROCEDURES

All minimelter data shall be recorded in the current *Minimelter Log Book*. A separate log of sample and melt materials entering and exiting the minimelter room will be kept.

I. GLOVE BOX CONNECTIONS

The glove box has several electrical outlets inside supplied through sealed electrical feedthroughs. Two instrument cords pass through a hole in the box sealed with RTV.

Three tubing feedthroughs penetrate the box wall. The first is used to pass non-radioactive feed into the box. The second is used to pass the radioactive feed to the melter. The third is used to pass water into the box. The tubing between the box and the melter is nylon reinforced vinyl tubing with a pressure rating of 300 psi. In the event of a clog the failure point of the feed tubing would be the silicone tubing which is entirely in the glove box. That tubing balloons and bursts at pressures of 20 psi.

This was confirmed by test. The connection of the reinforced vinyl tubing to the glove box was tested at an excess of 50 psi for five minutes with no signs of failure.

II. SETUP

1. Confirm normal conditions as below and record values*:

   a. melt temperature is 1100°C ± 20°C

   b. melt plenum temperature 800°C ± 50°C

   c. pour chamber temperature between 750°C and 875°C

   d. furnace pressure drop is 0.1" ± 0.03"

   e. primary filter combination pressure drop less than 1" (primary filter combination consists of roughing filter and an 80% filter)

f. Large HEPA filter pressure drop less than 0.3"

g. melt chamber globar current 20 amp

h. pour chamber globar current 25 ± 2 amp

i. pour tube heater 1 amp on meter

j. cooling water flowing in the feed pipe

k. check survey meter battery and confirm response to radioactive material.

* Record initial temperatures, heater values and time in run data table. Record pressures on comments page.

If conditions are not within normal limits determine cause if possible, notify the Project Manager and request instructions.

2. Read run objective instructions, sign and date.

3. Raise meltchamber setpoint to 1160°C, determine that the load current is less than 100 amp. If current is greater than 100 amp employ current limiter as required.

4. Set pour tube heater current to 2.0 A on meter.

5. Adjust pour chamber globar current as necessary to obtain a pour chamber temperature of 830°C ± 10°C. Current adjustments should be less than 2.5 amps per 5 minutes.

6. Tilt furnace using jack and 3" spacer. The long vertical edge of the spacer should be flush with the furnace edge.

7. Check to see that non radioactive feed is available in sufficient supply for the run--assume a feed rate of 30 lb per day of operation. If 60 lbs or less of feed remains notify the Project Manager that a new batch is needed and give an estimated time for depletion of current batch.

8. Observe level of feed in glove box (internal bucket). If feed is greater than 1/4 of bucket, turn on glove box feed bucket stirrer (speed setting 30, switch CW) and skip to step 10, if not, proceed as follows:

a. turn on stirrer in external supply bucket and stir for at least 5 minutes.

b. turn off stirrer and record weight of external feed bucket on comments page as "Outside Bucket, Initial Weight", then resume stirring.

c. insure that the intake end of the supply tube is inserted into the strainer pipe attached to the external feed bucket and the output end (in the glovebox) is inserted into the internal bucket.

d. using external pump, transfer nonradioactive feed into the glove box feed bucket until the scale underneath the external bucket registers a drop in weight approximately equivalent to the desired amount of nonradioactive feed (usually between 8 and 16kg, depending on how much water is to be added to the nonrad. feed in the internal bucket. Shut off pump.

e. shut off stirrer in external feed bucket.

f. record weight of external feed bucket on comments page as "Outside Bucket, Final Weight".

g. determine the weight of feed added to the glove box bucket as the difference of the weights determined in steps b and f (initial - final) and record on comments page.

h. using the weight from g calculate the weights of U and Th oxides to be added using the recipe supplied by the Project Manager. Record the results of the calculation on the comments page.

i. weigh the U oxide using the tared metal weighing cup. The weight should be within $\pm$ 0.1 g of the calculated value. Record the actual weight obtained on the comments page.

j. turn on the stirrer in the glove box feed bucket at a setting of 15 on the control dial.

k. open the funnel hole in the top of the feed bucket and place the funnel in it. Pour the U oxide powder in.

l. weigh Th oxide as in i and pour powder in feed bucket

m. remove funnel and close funnel hole

n. using paper wipes clean funnel, weighing cup, glove surfaces and other areas where powder may have fallen. Dispose of wipes in rad waste container, replace weighing cup on scale and funnel on shelf.

o. adjust stirrer speed setting to 30.

9. Flush out the non-radioactive feed from the external feed tube according to the following steps which should be carried out each time feeding from the external feed bucket into the glove box bucket is commenced.

a. Remove the intake end of the external feed tubing from the external feed bucket and insert it into a clean water pail.

b. Pump clean water for about 30-45 seconds until the tubing is clear from the feed. The discharge is fed into the glove box bucket.

10. Stir glove box bucket for at least 5 minutes before turning on glove box feed pump.

11. Before turning on glove box feed pump inspect the pump tubing for signs of damage indicated by evidence of feed leakage. If damage is noted replace the tubing according to the procedure below, if tubing is ok skip to step 11i. As preventative maintenance the tubing is to be replaced on the first operating day of each month and so noted in the comments page of the data log (replacement may be waived by the Project Manager if the current tube has had less than 3 weeks of service).

a. cut a 48" piece of Masterflex T-6411-17 silicone tubing and pass it into the glove box (see I).

b. remove as much feed from the existing tube as possible by running the pump while the intake end of the tubing is above the liquid level in the internal feed bucket. If this step is not possible due to excessively damaged and leaking tubing skip to step e and take extra care to avoid spilling feed from the tubing.

c. place intake end of tube in water pail and pump clean water for 2 minutes at a pump setting of 40 ml/min.

d. drain the tubing by reversing the pump for 30 seconds.

e. disassemble the pump head

f. remove the tubing from the bulkhead fitting and discard in the rad waste

container.

g. wipe up any spills and dispose of wipes.

h. install new tubing

i. secure tubing by applying silicone caulking (eg. RTV) to outside of pump head where both tubes enter the assembly. Caulking should be allowed to cure over night for maximum strength.

j. check to see that the tubing is secured in the protection pipe with its end approximately 1" from bottom of the bucket.

j. turn on pump at the setting specified in the run objective and confirm proper action i.e. no excessive air in lines, no crimping of pump tubing and delivery of feed into the furnace.

III. NORMAL OPERATIONS

1. Record run data every 20 minutes in data log. Data to be collected include: setpoint, electrode voltage, electrode current, melt chamber globar current, pour chamber globar current, pour tube meter reading, melt temperature, melt chamber temperature, pour chamber temperature and feed pump setting.

2. After recording data confirm that furnace is under negative pressure (pressure drop greater than .07"). Refer to section VI for corrective action if pressure drop is too low.

3. Monitor the glass level in the melter (melt chamber) at least every 20 minutes by gauging level with the edges of the electrodes.

4. The condition and size of the cold cap is to be monitored visually at frequent intervals and its size recorded every 20 minutes. If the cap exceeds 50%, adjust feed level to reduce the cold cap. Unusual conditions shall be noted in the comments page.

5. The level of the feed in the feed bucket should be monitored directly by lifting the lid. If the level is low (less than 1/4) feed should be added when convenient, refer to section II.

6. Observe the level of glass in the collection vessel. The vessel is to be removed before the level of glass reaches 1 inch from the lip. To gauge glass level in the ss waste crucible, line up insulation edge and lower edge of pour tube heater while observing the

glass level through the window. Glass level should be below that line of sight. To remove crucible:

a. obtain replacement crucible, a 3 qt. ss pot (used for waste only).

b. bring the furnace tilt to zero and wait for glass to stop dripping.

c. crucible removal requires wearing heat protection gloves and safety glasses or face shields and a respirator**.

d. unlatch door, remove and place on bricks

e. with black tongs slide crucible onto outer platform.

f. with pliers carry the crucible to the brick platform and place in 2 gal steel bucket and place lid on bucket.

g. put new crucible in place

h. replace and latch door

i. tilt furnace using jack and 3" spacer as in step I.6.

j. prepare a radioactive sample label for the bucket and record crucible removal on comments page in notebook. Label shall contain run #, feed #, date, approximate U and Th oxide content and data log reference.

k. after cooling, label bucket and take to storage.

Once the total amount of glass collected in ss crucibles reaches the turnover value for waste, clay crucibles are used to collect glass, as described in section 7 below.

7. Glass Sample Collection. (When total amount of waste glass collected in ss crucibles passes the turnover point specified by project manager).

a. level the furnace

b. place a numbered and dated clay sample crucible in special ss crucible having a holder for the clay crucible (the crucible # is the sample number for that day).

c. remove ss crucible from pour chamber as in III.6c-f.

d. place special ss crucible, containing clay crucible, in pour chamber

e. replace door

f. tilt furnace to 3" as in step I.7

g. observe pouring frequently until sample is collected (collection usually takes about 45 minutes for 400g crucible, 2.5 hours for 1.4kg crucible).

h. terminate pour by leveling furnace

i. after dripping has stopped remove special crucible. Separate clay crucible from special crucible by gripping clay crucible with pliers. Place the special crucible in a 2 gal. steel bucket to cool. Place clay crucible in 2 gal. steel bucket to cool for storage and later remelting. Cover with empty ss crucible to prevent splintered glass from escaping crucible as it cools.

j. place new or previous crucible in pour chamber as is appropriate.

k. Record the sample collection in comments page of data log, record sample transfer in sample log or prepare a radioactive sample label containing run #, feed #, date, approximate U and Th oxide content and data log page reference depending on sample disposition. The label is placed on the clay crucible when it cools down or on the steel bucket if crucible is placed in storage. Accuracy on the glass weighing should be + or minus 0.1g.

l. If sequential samples are desired, the procedure is modified as follows: in step i above, after the clay crucible is removed from the special crucible it is replaced with a new one and collection is resumed as in steps II 7 d-i. If is being stored a radioactive sample label for the steel bucket is prepared as in step k above which lists crucible numbers being stored in that bucket.

m. resume operations III.

8. Fill and empty the Pt-Au Crucible as follows:

a. Place the crucible in the refractory liner which is placed in a half SS crucible.

b. Follow the glass sample collection procedure as in step 7 of this procedure

c. After the crucible is filled and removed from the melter, place a piece of 1" white

insulation (WRP-ceramic fiber insulation) on top of the crucible. Allow the crucible to cool down to room temperature

d. Carefully remove the insulation and remove the crucible from the liner into the grinding glove box.

e. Invert the crucible and gently tap the crucible on its lips against the bottom of the glove box. If glass does not fall out then try to remove it from the top by inserting a screw driver into the glass surface cracks and forcing the pieces out.

f. After successive use the glass starts to wet the crucible; this is indicated by patches of glass on the crucible walls which remain. Take the crucible to the acid fume hood in room 336 for HF clean out.

9. Collect feed sample as follows (see II. 9. p.):

a. with pump running remove feed tube carefully from protection pipe and shut off pump

b. wipe outside of feed tube and dispose of wipes in rad waste container

c. collect 12 to 15 ml of feed in a sample vial by operating the feed pump in the reverse direction.

d. replace feed tube into protection pipe.

e. close sample vial

f. using damp wipes clean the outside of the sample vial and the gloves and dispose of wipes in rad waste container

g. remove vial from glovebox (refer to I for glovebox procedure).

h. note sample taking on comments page of data log

i. label sample with run #, feed #, date, log entry and approximate U and Th oxide content

j. when sample exits the minimelter room record it in the sample log book.

10. General operating rules:

    a. put tools away after use.

    b. clean up any spills immediately

    c. keep working area as neat as possible

    d. record on comments page of data log all observations of unusual events immediately following their occurrence.

    e. label all samples with sample name, date and notebook page indicating sample taking.

    f. shoe covers are to be worn in the melter room at all times

    g. respirators** are to be worn as backup protection for operations involving the possible release of contaminated dust.

    h. Film badges will be worn by operating personnel in the melter room. Ring badges will be worn when handling the U and Th bottles and glass samples.

    i. Repairs to equipment involving hazard should not be performed unless under the direction and in the presence of one or more of the following: R. Mohr, H. Hojaji, I. Muller, W. Keene, P. B Macedo.. If alone and a hazardous situation occurs do only what is necessary to prevent further hazard.

Refer to section VIII for emergency response. Notify Bob Mohr (202) 234-5953, Hamid Hojaji (301) 365-0999, Isabelle Muller (703) 534-5420 or P. Macedo (301) 229-0408 to obtain assistance. Notify Campus security and or the fire department of fire or serious accident. Security 5111. Fire and emergency dial 9 for outside line then 911.

Notify RSO of any unenclosed feed spills greater than about 100 ml.: phone 5206, home: (703) 671-1659.

IV. <u>SHUTDOWN</u>

1. Clean and flush feed and pump tubing as follows:

    a. run pump in reverse direction at maximum speed until as much of the feed as possible is removed from the tubing.

b. withdraw feed tube from feed pipe and place end in water container. Run pump in forward direction at maximum speed until tubing is refilled. Then reverse pumping direction until tubing empties.

c. repeat b until the tubing is cleaned.

d. pump any remaining contaminated water into the melter at a feed rate of 30 ml/min.

e. pump clean water into the melter for 15 minutes at 30 ml/min.

f. turn off pump and stirrer

g. replace feed tube in feed pipe securing it so that its end is approximately 1" from the bottom of the bucket.

h. wipe up any spills in the glove box with wipes and dispose of the wipes in the rad waste container.

2. Level furnace

3. Set pour chamber globar current to 23 amps

4. Set pour heater to 1 amp on dial

5. Set meltchamber setpoint to 1000°C

6. Inspect feed lines and note any signs of deposits that might lead to blockage. If there is buildup note in the maintenance log and notify R. Mohr on following day.

7. Prepare the next data page according to format used on previous data pages.

8. Check, sign and date the data pages for the current run. Reviewer shall also sign and date the data pages.

9. Put away tools etc., clean up any spills, and mop the lab floor.

10. Record the pressure drops across filters and furnace on the comments page of the data log.

V. FILTER CHANGEOUT--RSO presence is required
(Record in maintenance log.)

A. Roughing Filter

1. The roughing filter will be changed at or before the time the pressure drop across the primary filter combination is no greater than 1"

2. Respirators**, gloves and disposable coveralls are to be worn

3. Remove filter cover and place on paper towel

4. Attach drawstring bag to filter access port

5. Remove filter into bag

6. Twist bag and tape

7. Carefully remove bag from filter port into plastic bag, tape closed and dispose in rad waste

8. Visually inspect 80% filter and port opening

9. Carefully clean port opening with damp wipes and dispose in rad waste container

10. Push filter into port with pink side up

11. Replace filter cover

12. Wipe area with damp wipes and dispose of wipes in rad waste container

13. Dispose of gloves in rad waste container

14. Proceed to changeout of 80% filter.

B. 80% Filter

1. Collect tape, disposal bag and polyethylene sheet.

2. RESPIRATORS**, GLOVES and disposable coveralls are to be worn

3. Tape polyethylene sheets to the flanges on the long sides of the housing. Tape a bag to the flange end facing the room door.

4. Loosen the bottom nuts several turns

5. With one person pushing the old filter out with the new filter a second person receives the old filter into the bag.

6. The bag is carefully removed from the filter housing and closed with tape.

7. The polyethylene strips are removed to rad waste, the flanges are inspected for dust and wiped with damp wipes which are then disposed of in rad waste.

8. The new filter is seated properly on the flanges and the flange nuts tightened to compress the gasket slightly until no gaps are visible.

9. Dispose of the gloves in the rad waste

10. Record new pressure drops and filter changeout in the maintenance log and on filter housing.

C. HEPA Filter

1. It is not anticipated that the HEPA will require replacement during these experiments. If it becomes necessary to change the HEPA, a procedure will be developed and approved prior to changeout.

D. Filter at the Minimelter Exhaust Port

Note: Respirators are to be worn during this operation.

1. Check the filter periodically. A dark filter indicates that a change is required. Its original color is white.

2. Use the white side of the roughing filter which is used in the off gas filter housing and cut a strip approximately 25 1/2" long by 3" wide.

3. Carefully remove the dirty filter from the intake port and dispose of it in the rad waste container.

4. Clean the melter top surface and outside the intake port with damp wipes and dispose of

them in the rad waste container.

5. Insert the new filter. It should completely fill the gap at the intake port.

VI. ROUTINE MAINTENANCE OF THE OFF-GAS EXHAUST PORT FROM THE MINIMELTER PLENUM

Note: This procedure is to be performed every 3 months or when the furnace pressure drop falls below 0.06" during normal operations. Record in the maintenance log.

1. Check that auxiliary blower is functioning

2. Confirm that filter pressure drops are not outside limits given in section II

3. Confirm that there is air flow at the cool air intake

4. If none of the above conditions would explain a low pressure drop then cause is probably blockage of the exhaust port

5. RESPIRATORS** and GLOVES are required

6. Open clean out port and discard used foil gasket in rad waste

7. Using a metal rod push any blocking material down into the furnace

8. After cleaning is complete store rod in container for cleaning

9. Close cleanout port employing a new gasket

VII. CORRECTIVE ACTION FOR A CLOGGED FEED LINE
(Record in maintenance log.)

1. Perform line cleaning procedure described in step IV.1 above.

2. If the blockage is not removed by the above the feed pipe may have to be mechanically cleaned.

3. RESPIRATORS** and vinyl gloves are required.

4. With the furnace level remove the feed pipe and place in a double plastic bag in a 5 gal

metal can.

5. Using damp wipes clean the area around the furnace opening for the feed pipe and cover the opening with a metal cover.

6. Use a stiff wire, with the feed pipe inverted over the plastic bag, to clean the feed pipe.

7. Using damp paper wipes carefully clean the exterior of the feed pipe and dispose of wipes in rad waste.

8. Dispose of soiled gloves in rad waste and using new gloves replace the feed tube in the furnace.

9. Carefully clean with damp wipes the exposed surfaces of the feed tube and the surrounding area.

10. Dispose of wipes and vinyl gloves in plastic bag.

11. Using clean gloves close the plastic bag with tape and dispose of in rad waste.

12. Operate feed pump to determine whether blockage has been removed.

13. If a block remains the reinforced tubing must be removed, have its ends clamped, and be disposed of as contaminated material.

14. Replace the tubing and resume operation as in III.

VIII. EMERGENCY RESPONSE

VSL personnel are not specifically trained to handle medical, fire, electrical, chemical or radiation emergencies (with the exception of W. Keene who is trained to handle radiation related hazards). Therefore, response to an emergency should be limited to those actions by which the untrained individual may prevent further hazard and to the notification of the proper authorities.

Emergency phone #'s: Police, Fire Dept. and Ambulance 9 for outside line then 911; Campus Security 5111; W. Keene 5206, R. Mohr 5327 office or (202) 234-5953 home, H. Hojaji 6705 office or (301) 365-0999 home, P. Macedo 5327 office or (301) 229-0408 home, I. Muller 5331 office or (703) 534-5420 home.

Campus security is to be notified of all accidents involving building damage, personal

or building hazard and or personal injury.

A few possible hazards and the response to them are listed below:

1. Small fires of non electrical origin. Use fire extinguisher found on the wall of the melter room to extinguish the fire and notify campus security immediately. If the fire can not be immediately extinguished leave the room , close the door and call the DC fire department informing them of fire and the fact that radioactive materials are in the room. Turn off room ventilation blower using circuit breaker in press room or switch mounted outdoors next to blower. Sound building fire alarm. Do not turn off fume hood exhaust blower (s).

2. Small electrical fire. Remove power from electrical circuit using circuit breaker in press room for surface mounted electrical fixtures. Flush mounted outlets are controlled by circuit breakers in hallway. Use fire extinguisher found on east wall of the melter room to extinguish the fire and notify campus security immediately. If the fire can not be immediately extinguished leave the laboratory, close the door and call the DC fire department informing them of fire and the fact that radioactive materials are in the room. Turn off room ventilation blower using circuit breaker in press room or switch mounted outdoors next to blower. Sound building fire alarm.

3. Electrical malfunction causing obvious overheating of equipment or wiring as indicated by smells, smoke or temperature.

   Remove power from the equipment using disconnect or circuit breaker. Notify R. Mohr or the Project Manager for assistance.

4. Spill of radioactive feed. Radioactive feed might be released in the unlikely event of a feed line rupture. If this is the case and the pump is still operating turn off the pump motor if it can be reached without contaminating the operator. If the pump can not be reached remove power to the electrical circuit using the circuit breaker in the hallway. Notify W. Keene of a spill. For preliminary cleanup vinyl gloves and respirators** should be worn.

   Wipe up spill with paper towels and dispose of towels in rad waste. Before operations can be resumed W. Keene must survey the area and direct any additional cleanup efforts.

5. Personal injury requiring medical attention. Do not move injured person unless it is necessary to prevent further injury.

   Call for an ambulance and notify Campus Security.

6. Large fire. Isolate fire if possible by closing room door.

Call fire department notifying them of fire and the presence of radioactive materials. Turn off room blower using circuit breaker in press room or outside switch mounted near blower. Sound building fire alarm and notify Campus Security. Do not turn off fume hood exhaust blower (s).

6.7 PROCEDURE FOR MAKING RADIOACTIVE GLASS BARS

Note: Document work performed in the appropriate Log Notebook.

*Note: Three furnaces can be used to remelt glass for bar casting. The Deltech DT-28-6 is used for smaller melts (in the small 400g clay crucibles or the small Platinum crucible), the Deltech DT-28-12 and the VSL 5" Round furnaces are used for larger melts (in the large 1.4kg clay crucibles or the large Platinum crucible). If using either Deltech furnace, follow instructions below labeled **DELTECH**. If using the VSL 5" Round furnace, follow directions below labeled **ROUND**. Instructions not specifically labeled are applicable to BOTH furnaces.*

When pouring glass from a small melt that has already been prepared according to section 6.8 and is ready to pour, skip to Step 3.

1. Heat furnace to 1150°C±10°C, or to temperature specified by the Project Manager.

2. Placing Sample In Furnace:

   (a) Get a fireclay crucible containing the radioactive glass from the melter room or storage area in room 40 and record this in the appropriate storage log.

   (b) DELTECH: Open the furnace and place the crucible in the center of the platform. Close the furnace

   ROUND: Remove the insulating furnace lid and ceramic crucible cover from the furnace. Place the crucible into the center of the furnace and cover with the ceramic crucible cover. Place the insulating furnace lid on top of the furnace and cover the center hole with a small piece of insulation.

   (c) Record the time when you put the sample in, and the temperature before you started, and after you finished.

3. Put the graphite molds in the Neytech annealing furnace, turn it on, and adjust the temperature setting as necessary to obtain a reading on the S-type monitor thermocouple corresponding to 500°C±50°C (or a different temperature as specified by the project manager).

4. Pouring bars:

(a) Make sure you have the tin snips where you can reach them while pouring.

(b) Remove the molds from the furnace and put them on the ceramic fiber board leaving a spot to set the crucible down. Close the Neytech furnace.

(c) ROUND: If the stirrer has been used in the melt, unchuck the extension cable and swing the arm out of the way.

(d) DELTECH: Open the furnace.

ROUND: Remove the insulating lid from the furnace. Uncover the crucible and remove the ceramic crucible cover from the furnace. If the stirrer has been used in the crucible, remove it and place on a graphite plate to cool (Pt at high temperatures should NOT come in contact with other metal).

note: If using platinum crucibles, the next three steps should be carried out with an insulation material protecting the crucible even during the pouring.

(e) Remove the sample from the furnace and place next to the molds on the table.

(f) Get a good grip on the crucible for pouring (with the tongs).

(f) Pour the glass into the molds. When a mold is full and the string of glass from the crucible to the mold is thin, cut it with the tin snips.

(g) Put the molds and bars in the 500°C Neytech furnace.

(h) Put the empty clay crucible in a metal can with a lid to cool.

(i) DELTECH: Close the furnace. Set the temperature to 0°C, unless another bar is to be cast, in which case follow step 2.

ROUND: Place the ceramic crucible cover back inside the furnace. Place the insulating lid back on top of the furnace. Leave the furnace temperature control set at 1150 °C if more bars are to be poured later in the day, or turn down to 900 °C if the next set of bars is to be poured within a few days, otherwise leave off.

5. Turn off the annealing furnace after one hour. Let the bars cool slowly in the furnace overnight, or if you will be pouring more bars the same day, remove them when they cool to 220°C and put them in a metal can in case they shatter.

6. Weigh the bars on the Mettler scale and store them in a properly labeled zip lock bag, in the storage area in room 40. Make a proper entry in the Radioactive Glass Storage Log. Create a page in the appropriate Radioactive Glass Log and record the shape and weights of glass items produced.

7. Weigh the poured crucible. Calculate the glass left in the crucible by subtracting the empty weight of the crucible from the poured crucible weight. Record the poured crucible weight, remaining glass weight and total glass produced in the same lab book used to record your melting procedures.

6.8 PROCEDURE FOR MAKING SMALL MELTS

Note: Record the inventory number (CUA# or VSL#) or of each ingredient in the logbook next to the chemical name while weighing.

1. Obtain the recipe from the Project Manager

2. Check that all the necessary ingredients are available including WVCM series or other bulk glass that has been ground such that 95% of it passes through a 40 mesh sieve. (Grinding is described in 6.8)

3. Measure out each of the ingredients according to the recipe using the following procedure:

   a. Non-Radioactive Ingredients.

      i. Accurately weigh out the non-radioactive ingredient on the Mettler balance to within 1% of the required amount in a disposable sample dish.

      ii. Transfer the weighed ingredients into a pre-weighed clean empty sample cup and record the accurate weight.

   b. Radioactive Ingredients.

      i. In the glovebox, place a clean, marked jar (large enough to fit both the radioactive and non-radioactive ingredients) on the Scientech balance and zero the balance.

      ii. Weigh-out the radioactive ingredient into the jar according to the recipe. Weight should be within 1% of the required value. Record the RMIC#

of the radioactive ingredient in the logbook next to the ingredient name.

4. In the glove box add the non-radioactive ingredient to the bottle containing the radioactive ingredient. Close the lid and mix by shaking the bottle for several minutes. Transfer the mixture to a pre-weighed large clay crucible (only large clay or Platinum crucibles that accommodate the Pt stirrer are used for making small melts).

5. Large crucibles will only fit in either the VSL 5" round furnace or the Deltech DT-28-12 furnace. If using the Deltech furnace, follow steps labeled **DELTECH** below. If using the VSL 5" round furnace, follow steps labeled **ROUND** below. Unlabeled steps apply to both furnaces.

6. Remove the filled crucible from the glovebox, bagging the outside. If using the ROUND furnace, insert the stirrer deep into the mixture (do not use excessive force as the stirrer is very soft) with the plain end (no blades) up.

7. DELTECH: The Pt stirring rod should be set up inside the furnace. If not, swing the cable extension arm into position above the furnace. Open the furnace (it should be at room temperature) and place the stirring rod vertically inside the furnace with the plain end (no blades) at the top. Thread the plain end through the small hole at the top of the furnace, insert into the extension cable chuck and tighten. Adjust height of extension arm to upper position. Close the furnace.

8. Turn on the furnace power and set the controller temperature to 1150°C unless otherwise specified in the recipe or by the Project Manager.

9. DELTECH: When the furnace has attained the set temperature, open the furnace, center the crucible on the furnace platform and close the furnace. The stirrer and crucible should be lined up when the crucible is drawn into the furnace on the platform.

   ROUND: When the furnace has attained the set temperature, remove the insulating furnace lid and remove the ceramic crucible cover from inside the furnace. Place the crucible with stirring rod inside. Cover the crucible with the ceramic cover and stirrer guide tube. Be sure to thread the top end of the stirrer rod through the center hole of the ceramic cover and guide tube. A small section of the stirrer rod should protrude from the top of the guide tube.

10. Record the time the crucible enters the furnace and the setpoint.

11. ROUND: Cover the furnace with the insulating furnace lid, adjusting the stirrer and guide tube through the center hole of the lid.

12. ROUND: Swing the cable extension arm into position over the top of the stirrer. Tighten the chuck of the stirrer extension cable onto the top of the stirrer (it may be necessary to grasp the stirrer end with pliers to insert all the way into the chuck.

13. Make sure that the other end of the extension cable (the end that is not chucked to the stirrer rod) is coupled to the proper motor shaft. Tighten set screws on the motor shaft sleeve if necessary. The extension cable assembly should effectively transfer torque from the motor to the stirrer.

14. After melting for one hour set the rotation speed of the stirrer to #6 on the power controller* using the high torque output and stir for the appropriate time given in the recipe.

    *DELTECH: Make sure that the extension arm and stirrer have been lowered into the melt (lower position) BEFORE turning the stirrer on.

15. DELTECH: 5 minutes before the required melt time is complete, turn off the stirrer motor and raise the extension arm and stirrer to the upper position to allow the stirring rod to drip.

16. After the furnace has remained at the set temperature for the time required by the recipe, cast the melt into bars using the procedure in 6.7 (Procedure for Making Radioactive Glass Bars, steps 3 through 6). If using the ROUND furnace, first turn off the stirrer motor, unchuck the stirrer from the extension cable and swing the extension arm out of the way.

17. Record the weights of the crucible and the stirrer with the residual glass and calculate the glass yield from the weights of cast glass bars and residual glass. Record the weights in the appropriate Lab Notebook and radioactive glass sample log.

6.9 RADIOACTIVE SAMPLE GRINDING PROCEDURE

This procedure describes the grinding and sieving of radioactive glass samples:

1. Obtain sample and log it into the *Minimelt Sample Log Book* and into the grinder log book. Record date, glass batch, approximate description of material and reference to direct source i.e. received from whom and appropriate sample log reference, bar reference and crucible reference. When samples are combined for grinding, bars weighing a total of no more than 1 kg shall be combined.

2. Obtain containers for finished samples and for the remains and label them with date, glass batch, material description, bar reference and crucible reference.

3. Using disposable gloves place the containers and sample in the glove box pass through, close the outer pass through door and bring the above into the box.

4. The desired amount of material is first broken into pieces which will pass through the grinder aperture using a hammer and stainless steel plate. The breaking is done within a clean bag to contain the shattered material and to prevent contamination of the sample from previous samples. Up to 100g can be conveniently broken at one time.

5. The broken material size is tested by pouring through the grinder aperture into a clean sample cup.

6. The aperture is replaced in the grinder and the grinder lid is tightened and the grinder is turned on with speed switch at one and the timer at some convenient value.

7. When the grinder has reached speed as determined by the sound of the motor the broken glass is slowly poured into the grinder aperture. If the aperture clogs the clog may usually be removed using a spatula (use a spatula wider than the aperture). Feed all of the material into the grinder, turn it off and wait for it to stop. Stopping is confirmed by feeling the decay of grinder vibration.

8. Unscrew and remove the grinder lid. Remove the sample pan and transfer the ground material into a clean sample cup.

9. Repeat 6 through 8 as many times as specified by the project manager before sieving.

10. Transfer no more than 250g of the final ground product into the clean sieve stack

containing the desired sieves in proper order. For most tests the sieves will be 40,60,100 and 200 in that order from top to bottom. Underneath the bottom sieve should be a clean metal tray.

9. Shake the sieves for 2 minutes using up and down and side to side motion.

10. Pour the sized material into the appropriately marked containers. Material that has passed through 1 sieve but not the one underneath it is graded between the two sieves. For example material that has passed through the 100 mesh but is stuck on top of the 200 mesh is graded: 100-200. Material on the metal tray directly beneath a sieve is graded greater than that sieve. For example, if the 200 mesh is on the bottom, material on the metal tray directly underneath is graded: >200. If more of the smaller sizes are wanted the larger material can be recycled through steps 7-10.

11. Repeat steps 8-10 until the required amount of ground material has been sieved.

12. Fold and dispose of bag used for breaking the glass. Use the vacuum cleaner and brush to clean as much of the glove box, grinder and sieves as possible. Follow this with damp wipes which are disposed in waste bag. Use wipes to clean the exterior of the sample containers and place them into pass through and close inner door.

13. Using disposable gloves remove samples from pass through, rinse the outside of the bottles thoroughly.

14. Transfer samples and remains to the appropriate person and log the exit of the materials in the minimelt sample log. If sieves were used, log the amount of glass and type of glass passed through each sieve in the current *Sieve Use Record.*

** Where respirator use is specified it shall be a MSA COMFO II with type H Cartridge NIOSH # TC-21C-135.

7.0 SPECIMEN AND DATA TRACEABILITY

7.1 Flow Diagram for Glass Production

The following diagram (Figure 1) illustrates sample routing and sample documentation during glass production. Arrows indicate direction of flow as samples move from one procedure to another or to waste and storage. All operations directly associated with glass production are boxed with solid lines. The two glass melting steps are boxed and shaded. Three operations

associated with glass characterization are framed by broken lines.

7.2 Description of Steps Illustrated in Fig. 1

The following overview describes each boxed procedure in figure 1. Information given includes where each specimen comes from before reaching the described procedure, which log-book is used to record data on the specimen during the procedure, what numbers or labels if any are associated with the sample, and the form of the sample before and after the procedure.

I. Minimelt Glass: Sample arrives in the form of sludge, recorded in *WVCM Mix Book X.* During procedure, data is recorded in *Mini-Melt Log*. Sample exits in the form of glass, in steel cans or clay crucibles. Waste glass in steel cans are labeled MRxx where xx is a consecutively assigned number according to the minimelter waste log (see section IV below). Sample in clay crucible is labeled WVCM (example for West Valley Project; See the Project Manager for codes for other projects) followed by the run number of the melter and the crucible number. Sample is sent either to STORAGE, REMELT, or WASTE.

II. Storage: Sample reaches storage in several forms: glass in clay crucibles, glass bars and plates, sieved and graded. Sample comes from either the minimelter, bar/plate remelting, crushing/grinding, or small melts. Storage is recorded in *WVCM radioactive Glass Log , UTh Radioactive Glass Log* or *Misc Radioactive Glass Log* . Sample is sent either to REMELT or CRUSH and GRIND.

III. Remelt to Bars and Plates: Sample arrives in the form of glass in clay crucibles. Sample comes from either storage or minimelter. Remelting is recorded in *Crucible Melt Log*. Sample exits remelting in the form of cast bars and plates. Sample is sent to either STORAGE, WASTE, or CRUSH and GRIND.

IV. Waste: Sample reaches waste either as residual glass in clay crucibles or crushed and powdered remnants. Sample comes from minimelter, remelting, crushing/grinding, viscosity and leaching. Waste is recorded in *Minimelter Waste Log*. Waste is sent OUT for proper disposal by campus Radiation Safety Officer.

V. Crush and Grind: Sample arrives in the form of glass in bars or plates. Sample comes from remelting, storage or from small melts. Grinding is recorded in *WVCM Radioactive Glass Log*, *UTh Radioactive Glass Log* or *Misc Radioactive Glass Log*. Sample exits grinding in the form of graded or ungraded powder or crushed pieces. Sample is sent to either STORAGE, WASTE, VISCOSITY or LEACHING.

VI. Small Melts: Sample arrives as powdered WVCM XX glass from grinding. Sample is

mixed with other ingredients and labeled WVUTh XX, where XX is the run number. (WV is an example for West Valley Project. See Project Manager for code for other projects). Small melts are recorded in the *Crucible Melts log*. Sample exits to STORAGE, HEAT TREATMENT or CRUSH and GRIND.

VII. Heat Treatment, Viscosity, Leaching: These procedures fall under GLASS CHARACTERIZATION and are not covered in the GLASS PROCESSING procedures. Samples are recorded in WVCM, UTh or Misc Radioactive glass logs as leaving rooms 39-40. The recipient of the glass dates and signs the log. Logbooks used include:

All samples remain the property of the respective labs, and subsequent storage or disposal becomes their responsibility.

8.0 List of Logbooks

The following is a list of logbooks mentioned in section 7.2, with current VSL numbers given by the Administrative Office.

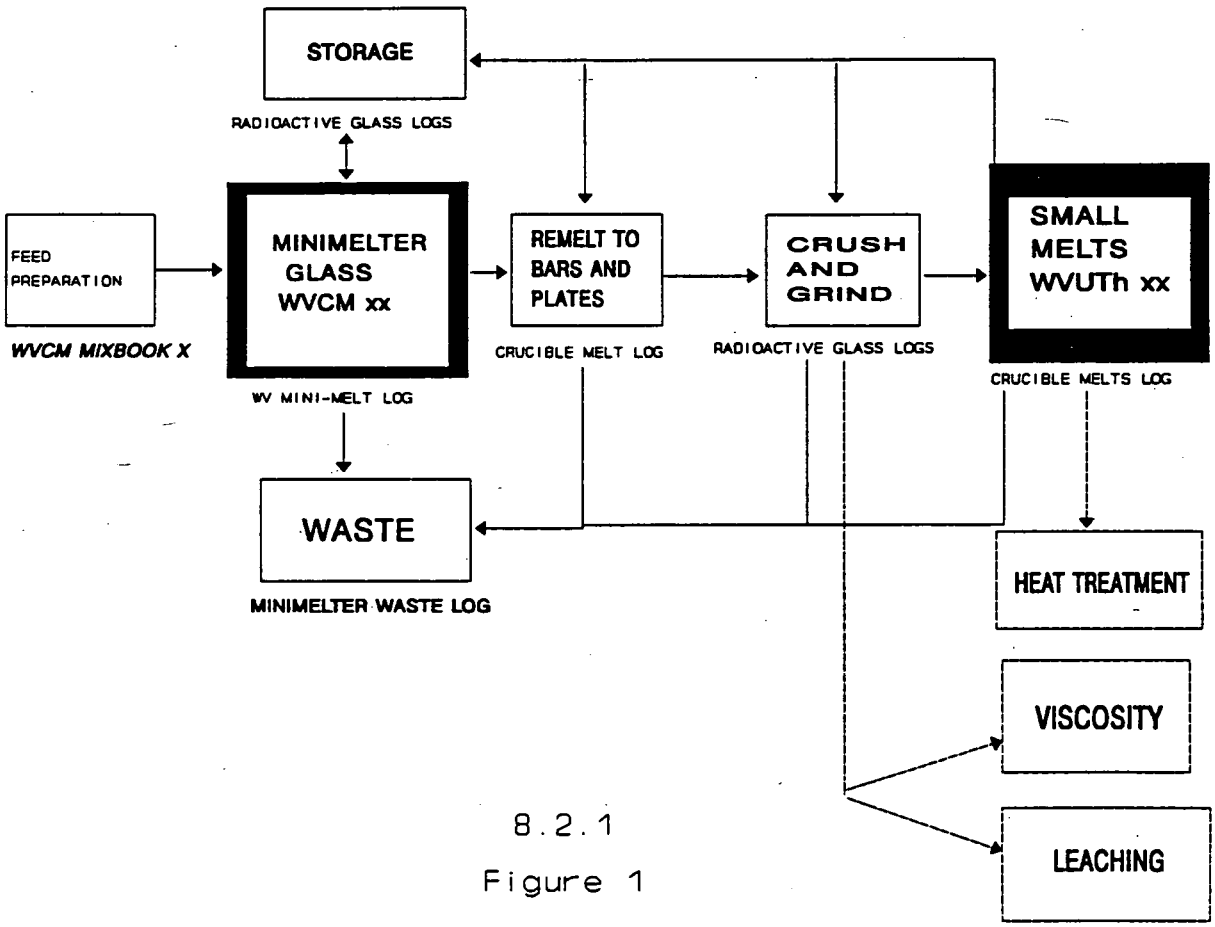
WVCM Mix Book X
Mini-Melt Log
WVCM Radioactive Glass Log
UTh Radioactive Glass Log
Misc Radioactive Glass Log
Crucible Melt Log
Minimelter Waste Log
Minimelter Maintenance Log
Sieve Use Log
Radioactive Glass Storage
Samples Receipt & Shipment Record
Equipment Calibration Log

9.0 REFERENCES

1) DOE/NE/44139-34
"Preliminary Results of Durability Testing with Borosilicate Glass Compositions" January, 1987.

2) "Radiation Safety Manual of the Catholic University of America" January, 1980; Warren E. Keene, RSO Washington, D. C. 20064.

3) PNL-3959
Materials and Design Experience in a Slurry-Fed Electric Glass Melter, S. M. Barnes, D. E. Larson, August 1987.


STORAGE
RADIOACTIVE GLASS LOGS
FEED PREPARATION
WVCM MIXBOOK X
MINIMELTER GLASS WVCM xx
WV MINI-MELT LOG
REMELT TO BARS AND PLATES
CRUCIBLE MELT LOG
CRUSH AND GRIND
RADIOACTIVE GLASS LOGS
SMALL MELTS WVUTh xx
CRUCIBLE MELTS LOG
WASTE
MINIMELTER WASTE LOG
HEAT TREATMENT
VISCOSITY
LEACHING

8.2.1

Figure 1

UNCONTROLLED

VSL Technical Procedure

Title: Gamma Counting on Germanium Detector

Document: TPI-RC

Issued: 9/92 Rev #0

Prepared by: Isabelle Muller Project Manager	Gerardo Del Rosario Project Technician
Approved by: Pedro B. Macedo Co-Director, VSL	C. Sivaprakash Quality Assurance Officer, VSL

ANALYSIS OF GAMMA EMITTING ISOTOPES USING THE GERMANIUM DETECTOR

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PROCEDURE FOR THE ANALYSIS OF GAMMA EMITTING ISOTOPES USING THE GERMANIUM DETECTOR

1.0 PURPOSE

This procedure describes the characterization of gamma emitting isotopes present in a sample relative to a calibration source of the same geometry. Based on the geometry of the calibration source, the identification and quantification of the isotopes are made through the Canberra Spectran-AT software executed under Microsoft Windows.

2.0 APPLICABILITY

This procedure applies to all VSL personnel trained and authorized to handle radioisotope-containing samples. Activities covered by this procedure include:

(a) Transfer of liquid nitrogen (LN2) from tank to cryostat
(b) Calibration of germanium (Ge) detector
(c) Gamma counting and analysis
(d) Documentation - log book and disk storage
(e) Sample accounting for traceability

3.0 SAFETY

3.1 Hazards

3.1.1 Chemical Hazards

The definition of a hazardous chemical is given in Section 8.3 of the CUA Chemical Materials Safety Manual, adapted from the OSHA Hazard Communication Standard. The CUA Chemical Materials Safety Manual is hereafter referred to as the CCMSM. This Standard and its application to CUA facilities are described in Sections 8.1 of the CCMSM, with the exceptions noted in Section 8.28.

3.1.2 Radioactive Hazards

The sources of radioactive hazards are the following:

(a) radioactive isotopes present in the sample
(b) mixed radionuclide calibration sources.

3.2 Hazard Mitigation

3.2.1 Identification of Hazards

The identification of chemical hazards should include an education program (Section 8.2 of the CCMSM). This program should include familiarization with the CCMSM as a whole, including the toxic properties of chemicals according to Sections 8.22, 8.23, 8.24 and 8.25 of the CCMSM, other hazards of chemicals classified under Sections 8.9, 8.10, 8.11, 8.12, 8.13, and 8.14 of the CCMSM, chemical handling and storage procedures (see paragraph 3.2.2 below), and emergencies (see paragraph 3.2.3 below). The education program will also include familiarization with the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University.

Other elements of hazard identification include the proper use of labels (Section 8.4 of the CCMSM) and of Material Safety Data Sheets (Sections 8.25, 8.26, and 8.27 of the CCMSM). Workplace chemical lists should be compiled, maintained, and made available for employees in accordance with Section 8.31. In addition, a copy of the CCMSM should be kept in the laboratory and its use should be included in the education program cited above. The same should apply to the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University.

3.2.2 Handling and Storage of Chemicals

Handling of chemicals should be conducted according to Section 8.6 of the CCMSM. Storage of chemicals should follow Section 8.8 of the CCMSM. Special precautions should be taken in handling and storage of incompatible chemicals, solvents, compressed gases, and radioactive materials according to Section 8.10, 8.12, 8.13, and 8.14 of the CCMSM, respectively.

Waste disposal of hazardous chemicals should follow procedures established by the CUA Environmental Safety Office according to Section 8.16 of the CCMSM.

3.2.3 Emergencies

Chemical spills should be dealt with according to Section 8.17 of the CCMSM. Fire and fire-related emergencies should be controlled according to Section 8.21 of the CCMSM. Cases of personal contamination should be treated according to

Section 8.19 of the CCMSM, cases of injury and illness according to Section 8.18, and cases requiring minor first aid according to Section 8.20.

3.2.4 Radioactive Hazard Mitigation

All rules for control and monitoring of personnel exposure and environmental radiation are described in Chapter 3 of the radiation safety manual of The Catholic University of America.

Application of these rules is the responsibility of each user working in the restricted area (rooms 37 to 40 of Hannan Hall) and the project managers, under the guidance of Dr. Warren E. Keene, Radiation Safety Officer of CUA.

3.3 Personnel Protection

Personnel protection from hazardous chemicals should consist of two elements. The first is familiarization and compliance with the instructions of the CUA Chemical Materials Safety Manual (CCMSM), including identification of hazards (see paragraph 3.2.1 above), handling and storage of chemicals (see paragraph 3.2.2 above), and dealing with emergencies (see paragraph 3.2.3 above). The second element is familiarization and compliance with the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University. The overall chemical hygiene program (Section 8.34 of the CCHP), based on the OSHA Regulation on Occupational Exposure to Hazardous Chemicals in Laboratories, should be followed. In detail, exposure reduction measures (Section 8.35 of the CCHP) will include environmental monitoring (8.35.1), inspections, maintenance and housekeeping (8.35.2), use of protective apparel and equipment (8.35.3), posting of signs and labels (8.35.4) and dealing with accidents and spills (835.5). Ventilation of both general laboratory spaces and of special areas and local devices such as chemical fume hoods and glove boxes should follow the instructions of Section 8.36 of the CCHP. Employees should take part in the safety information and training program which exists in the University according to Section 8.37 of the CCHP, and should be informed about the medical program provided by the University in cases of work with hazardous substances according to Section 8.39 of the CCHP. Responsibility for chemical hygiene rests with all employees.

In addition to compliance with the general instructions described in the CUA radiation safety manual, the following rules apply to the protection of personnel working in the restricted area of the VItreous State Laboratory:

1) All personnel working in the restricted area shall routinely undergo radiation safety training given by the radiation safety officer of CUA or in off-campus courses approved by the CUA RSO (see section 4.1).

2) The restricted area should remain locked and only workers assigned to this area, and project managers should have access to this section. All other persons who need temporary access to the radioactive laboratory shall be accompanied by authorized personnel.

3) Film badges shall be worn by operation personnel upon entering the restricted area. Ring badges and disposable gloves shall be worn when handling radioactive material.

4) Shoe covers are to be worn in the melter room at all times.

5) Any radioactive spill must be cleaned up immediately, checked after cleaning with a survey meter, and the radiation safety officer shall be notified as soon as possible so that wipe tests may be performed. Immediately notify RSO of any unenclosed spills greater than about 100 ml.: phone 5206, home (703)671-1659.

6) All samples shall be labeled with sample name, date and notebook/page of record indicating sample preparation. All new materials shall be entered into the inventory control file. Any material not in current use must be returned to the storage area in Room 40.

7) Respirators are to be worn as backup protection for operations involving the possible release of contaminated dust.

8) Repairs to equipment involving hazardous operations should not be performed unless under the direction and in the presence of one or more of the following: R. Mohr, H. Hojaji, I. Muller, W. Keene, P.B. Macedo. If alone and a hazardous situation occurs do only what is necessary to prevent a further hazard, and report the problem promptly to your supervisor.

TPI-RC

Refer to previous section 3.2.3 for emergency response. Notify Bob Mohr (202) 234-5953, Hamid Hojaji (301) 365-0999, Isabelle Muller (703) 534-5420 or Pedro Macedo (301) 229-0408 to obtain assistance. Notify Campus security and or the fire department of fire or serious accident. Security, x5111. Fire and emergency, dial 9 for outside line, then 911.

4.0 REQUIREMENTS

4.1 Training

All radioactive restricted area workers shall be trained both by the laboratory supervisor regarding their specific duties and by the CUA radiation safety officer.

Documents 10CFR19 and 10CFR20 are available to all restricted area workers. Each restricted area worker must attend a course in radiation safety given by or approved by the CUA RSO.

Training on each technical method and equipment used in radioactive counting is given by the laboratory supervisor and/or by a trained and experienced person designated by the laboratory supervisor until the technician shows competence to use that method without direct supervision.

5.0 PREREQUISITES

5.1 Equipment List

5.1.1 Canberra Gamma Spectroscopy System

Qty	Device	Model No.
1	Germanium Detector	GC 2020
1	Vertical Dipstick Cryostat	7500
1	Integrated Signal Processor	1510
1	System 100 Slave Board	4611
1	System 100 Software	Version 3.0
1	Spectran-AT Software	Version 4.0

5.1.2 Computer Accessories

Qty	Device	Model No.
1	Bently 286/12 System	ST 251-1
1	NEC Monitor	Multisync 2A
1	Epson Printer	LX 800
1	Microsoft Windows Software	Version 3.0
	Diskettes High Density 5.25"	
	Diskettes Low Density 5.25"	

5.1.3 Other Materials

Qty	Device	Manufacturer
	Marinelli Beaker, 500 mL	Ga-ma & Associates
	Vials, Screw Cap, 6 mL	
1	1.2 kg Balance, Model PL1200	Mettler
1	Graduate Cylinder, 500 mL	
1	Graduate Cylinder, 10 mL	
1	Oxford Macroset Pipetter	Fisher
1	Liquid Nitrogen Tank, 100 L	
1	Calibration Source, 500 mL	Analytics, Inc.
1	Calibration Source, 5 mL	Analytics, Inc.
	Disposable Latex Gloves or	
	Disposable Vinyl Gloves	
	Leather Work Gloves	

5.2 Equipment Calibration

5.2.1 Preparation

First-time users should familiarize, identify and locate the instruments and accessories listed in the equipment list. This is particularly important for the Canberra model 1510 unit which consists of the high voltage power source (HVPS), an amplifier, and multichannel analyzer (MCA), and of the detector and its preamp high voltage inhibit (HVI) LED indicators. The user must also be prepared for routine use of the detector cryostat liquid nitrogen (LN2) inlet line and the LN2 cylinder tank supply line during LN2 transfer.

Initially verify the following:

1) The computer, printer and the Canberra Model 1510 are switched on. If off, locate their individual power switches and turn on.

2) The detector preamp HVI LED indicators, if the indicator is green, the detector is cooled and ready for counting of sample. If the indicator is red, the detector has warmed up indicating low level or no LN2 in the cryostat. Prepare to transfer LN2.

5.2.2 LN2 Transfer

Warning: The extremely low temperature of LN2 (-196°C) can cause severe frostbite in contact with exposed skin. Some materials including rubber hoses may become brittle at -196°C and could shatter occasionally. Wear protective gloves, eye or face protection when handling LN2.

1) Check the level of LN2 in the cylinder tank from the level gauge glass located at the top of the tank. The position of the red portion of the spindle inside the gauge glass indicates the approximate amount of LN2 left in the 160 L tank. The tank has little or no LN2 if the spindle has dropped all the way down to the bottom of the gauge glass.

2) If there is enough LN2 in the tank, connect the end of the cryostat inlet port rubber hose to the "liquid" supply line of the LN2 cylinder tank.

3) Slowly open the valve of the "liquid" supply line of the

LN2 cylinder tank. The lines will quickly cool down and freeze. The filling up of the cryostat will take about 30 minutes to an hour if the detector was completely warmed up. Never allow any unprotected part of the body to touch anything that is, or may have been, in contact with LN2.

4) Close the valve when LN2 begins to come out of the vent line indicating a completely filled up cryostat. Stand clear of boiling and splashing LN2.

5) The detector will cool down sufficiently within 2 hours after filling, and the HVI LED indicator of the detector preamp should turn to green indicating that the detector is ready for calibration.

6) Allow the rubber transfer line to warm up to room temperature before disconnecting from the cylinder tank supply port.

5.2.3 Detector Calibration

1) Check that the HVI LED indicator of the detector preamp is green.

2) With the computer, printer, and the Canberra Model 1510 switched on, switch the power toggle on the HVPS area of the Canberra unit to the on position.

3) Set the voltage of the HVPS by turning the knob clockwise to 3500 V (3 on the small window and 50 on the knob dial). The voltage increment will illuminate the bar display.

4) Open the germanium (Ge) detector lead shield, place the calibration source of the desired geometry according to the Project Manager recommendation (500mL Marinelli, 5mL vial, etc.) on the detector and close the unit. The calibration source should be a mixed radionuclide source with at least 5 different energies including a crossover energy between 200 and 300 keV.

5) Get Windows and S100 programs activated on the computer and then execute the command MCA.EXE (refer to pages 1.21, 1.22 and 2.2 of the System 100 User's Manual Version 3.0).

6) Select the MCA board assignment and full memory size in the Welcome screen. For gamma (Ge detector) counting, use MCA #1 and 4k memory size.

7) At the initial MCA window, a spectrum of a previous counting might be on display. To erase the live and true time registers and all data channels including the set regions of interests (ROI) of the previous counting, use the Clear command on the top menu bar (see pages 2.9, 3.48 and 3.49 of the User's Manual).

8) Preset the count time using the Acquire menu on the top menu bar or press F11 accelerator key (refer to pages 2.8, 3.25 to 3.28 of the S100 User's Manual Version 3.0). Check the preset live time setting if acceptable, otherwise change the preset live time as recommended by the Project Manager.

9) Start the counting process through the Acquire command or press the F4 accelerator key.

10) Set the Regions of Interest (ROI) in the developing spectrum (see pages 3.5 to 3.7 of the User's Manual).

11) When the preset time is reached, save and name the file CALmmdd.MCA (mm=month, dd=day Ex. If calibration is done on April 9, the filename will be CAL0409.MCA) and store in the Calibration File diskette. Record and log this calibration exercise in the Detector Calibration Logbook (VSL #305-92).

12) Get a printout of the ROI Summary (refer to pages 3.16 to 3.19 of the User's Manual). This will become handy during the detector energy calibration.

13) The energy and efficiency calibrations of the detector are performed using the CALIB option of the SPECTRAN-AT (SPAT) program which is Microsoft Windows compatible and hence can be accessed within Windows. After getting a printout of the ROI Summary, close the MCA window from the control-menu box and go to the SPAT directory from the File Manager; open the directory and execute CALIB.EXE (refer to pages 29, 103, 108, 109, 112 and 137 of Microsoft Windows User's Guide Version 3.0).

14) Perform the energy calibration with the EN option and the efficiency calibration with the EF option of the calibration program. The program is clear, easy and prompts detailed dialogs for the user to enter all necessary data. Refer to the Spectran-AT V4 User's Manual.

15) Proceed to analyze the calibration spectra (see section 6.3). The calibration results and spectra (including

background, see section 6.2, step 5) should be examined and a person trained to do so such as the Project Manager or a trained technician assigned by him/her must be consulted so that any drift, shift or other change in system response is noted and acted upon.

16) Calibration of the detector is required when:

a) the detector warms up
b) there is power outage

Under normal continuous operation, calibration is done every 6 months. Once the calibration is performed and the logbook records filled-in, place a label on the 1510 unit giving the date of calibration, the geometry number and the next due date of calibration.

6.0 SAMPLE PREPARATION, COUNTING AND ANALYSIS

6.1 Preparation of Sample

Samples for radioactive analysis can be placed either in a 500ml Marinelli beaker, in a 6ml vial or any other container which is of the same exact geometry (including the physical state of the material, e.g. liquid, solid, slurry, soil, etc.) as one of the calibration sources currently on hand. Sample volume (for liquids) and weight (for solids) are first determined before placing in the desired containers, Marinelli beaker or vial. Label the samples properly and attach radioactive labels.

6.2 Counting of Sample

1) Do the initial checks as described in section 5.2.1 of this procedure.

2) Verify that the detector is calibrated from the calibration label on the 1510 unit and from the calibration logbook. Place the sample inside the lead shield.

3) Repeat steps 7, 8 and 9 on section 5.2.3 of this procedure.

4) When the preset time is reached, save and name the file with a relevant filename and store in the Ge Counting

diskette. Record and log this counting exercise in the Gamma Counting Logbook.

5) It may be necessary to run a background count of the spectrum which can be subtracted from the peak areas of the same energy of the sample. To achieve background counting, remove the sample from the shielding and start collection with the same preset time used for the sample. When counting is finished, save the file as BKmmdd.MCA and store in a BK File diskette. Background acquisitions should be done on a regular basis so that any change in system response can be noted and acted upon when the Project Manager checks the logbooks or if any emergency situation occurs.

6) Place all samples in the storage area (see section 8.0 below) and then proceed to analysis of the spectra using the Spectran-AT program.

6.3 Analysis of Spectra

1) On the computer, close the MCA system, access the SPAT directory and execute the ANALYZ.EXE program.

2) At the ANL> prompt, typing HELP would list all the available commands which includes the PEAK and QUAN analyses. The PEAK option performs nuclide identification while the QUAN option produces a complete radionuclide analysis report with measured and decay-corrected activities for each nuclide found included in the report. The Spectran-AT V4 User's Manual provides a thorough and detailed step by step explanation of the analyses options. It also describes the selection of the initialization parameters, such as the geometry, the smoothing function, the option to analyze multiplets, and others that are well explained. Example outputs are also given. The selection of the analysis library to be used will be set according to the Project Manager's instructions.

3) Output of report can be routed to a printer or ASCII disk file which creates a file named PEAK.WRI from PEAK analyses and QUAN.WRI from QUAN, MDA and LLD analyses. Specify the output listing device through the command ROUTE. See page 48 of the Spectran-AT manual.

7.0 DOCUMENTATION

The following logbooks are to be filled in with data corresponding to the activity performed:

a) Sample Log Book - to record sample data (see section 8.0).

b) Detector Calibration Logbook - to record the date of calibration, the calibration MCA file (CALmmdd.MCA), the type of detector calibrated, the calibrating source used, the geometry, and the name and signature of the person doing the calibration.

c) Gamma Counting Logbook - to record the date and time when counting was begun, the sample name, volume or weight and geometry, sampling time, the detector used, the total counting time, the MCA file name as saved and the name and signature of the person doing the counting.

The following data disks will be used to store MCA files:

a) Calibration File diskette - to store the detector calibration MCA file, CALmmdd.MCA.

b) Ge Counting diskette - to store MCA files of any counting activity made in the germanium detector.

c) BK File diskette - to store background MCA files.

8.0 SAMPLE TRACEABILITY

A description of all samples for gamma analysis should be entered in the "Sample Log Book", to record the sample name, origin/project, quantity (weight or volume), RMIC # (if sample came from an outside source), reference VSL Lab Notebook and page numbers (if in-house sample), date of log-in, date of log-out, originator, and other remarks.

After the gamma analysis is performed, the samples are to be stored in the radioactive storage area while the originator or responsible person is notified. If the originator wants his/her sample back, the sample shall be logged out, and originator must sign in the Sample Log Book, otherwise the sample remains stored in the radioactive storage area temporarily until the Project Manager advises for its proper disposal.

9.0 REFERENCES

1) Canberra Germanium Detectors User's Manual (0988)

2) Canberra System 100 User's Manual Version 3.0

3) Canberra Spectran-AT V4 CISE 526 User's Manual

4) Microsoft Windows Version 3.0 User's Guide

5) Radiation Safety Manual of the Catholic University of America; Warren Keene, RSO, Washington, DC January 1992.

Vitreous State Laboratory

The Catholic University of America

UNCONTROLLED

VSL Technical Procedure

Title: Receiving, Shipping and Handling of Glasses and other Radioactive Samples

Document: TPI-SH Rev #0

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1.0 PURPOSE

This Procedure describes specific actions which will ensure safe handling, storage, and tracking of received samples, and their associated data and shipment to the outside addressees.

The Procedure is written in compliance with the requirements set forth by the Section 13 of the Quality Assurance Manual, Document VSLQA1.MAN.

The Procedure is intended to meet NQA-1 Basic Requirement 13.

Persons accountable for maintenance of records on radioactive samples and their safe handling are the Catholic University Campus Radiation Safety Officer (CUA-RSO) and the Vitreous State Laboratory Radiation Safety Officer (VSL-RSO).

All technical employees of VSL are responsible for proper handling of radioactive and other hazardous materials and recording of all actions within the VSL.

2.0 APPLICABILITY

This Technical Procedure will refer to/apply to the following:

- Reception of samples at CUA-Radiation Safety Office
- Transfer of samples to VSL reception groups (Room #39)
- Sample traceability between VSL laboratories
- Storage of samples at VSL facilities
- Out-shipment preparation at VSL
- Shipment through CUA-Radiation Safety Office

3.0 SAFETY

3.1 HAZARDS

All samples received at CUA-VSL and handled at VSL laboratories are treated as potentially hazardous.

At VSL one encounters both chemical and radioactive hazardous materials.

3.1.1 Chemical Hazards.

The "hazardous chemicals", as referred to in this Procedure, are defined in Section 8.3 of the CUA Chemical Materials Safety Manual.

The CUA Chemical Materials Safety Manual is hereafter referred to as the CCMSM.

A copy of the CCMSM should be kept in every VSL laboratory.

The same applies to the CUA Chemical Hygiene Plan, which will be referred to as CCHP.

3.1.2 Radioactive Hazard.

Radioactive hazardous materials encountered at VSL are the following:

- radioactive isotopes used in the preparation of glasses;
- radioactive sludges;
- radioactive glass samples (bars and powder);
- radioactive liquid samples and
- radioactive liquids produced at VSL.

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3.2 HAZARD MITIGATIONS

3.2.1 Identification of Hazards

Procedures for identifications and handling of hazardous materials, as applied at VSL, are prescribed in Sections 8.22, 8.23, 8.24 and 8.25 of the CCMSM and in Sections 8.9, 8.10, 8.11, 8.12, 8.13 and 8.14 of the CCMSM.

Handling and storage of chemicals are described in Paragraph 3.2.2 of this Procedure. Emergency procedures are described in Paragraph 3.2.3 below.

A major element of hazard identification is the proper use of labels. At VSL, labeling of chemicals and other hazardous substances is done in accordance with Section 8.4 of the CCMSM, as well as in accordance with Material Safety Data Sheets provided by the manufacturers of the chemicals and other hazardous materials. (As regulated by Sections 8.5, 8.26 and 8.27 of the CCMSM).

Every VSL laboratory maintains the "Work place chemical list". The list is made available to employees in accordance with Section 8.31 of CCMSM.

3.2.2 Handling and Storage of Chemicals

Handling of chemicals should be conducted according to Section 8.6 of the CCMSM. Storage of chemicals should follow Section 8.8 of the CCMSM.

Special precautions should be taken in handling and storage of incompatible chemicals, solvents, compressed gases and radioactive materials in accordance with Sections 8.10, 8.12, 8.13, and 8.14 of the CCMSM, respectively.

Unstable chemicals, in accordance with Section 8.9 of CCMSM, should be excluded from the Glass Melting Laboratory except in cases where the introduction of such material is required by the project manager.

A special instruction for handling of each unstable chemical should be made in writing. It must specify the maximum quantity of the material and the maximum period it may be stored before being discarded.

Shock-sensitive chemicals, according to Section 8.11 of CCSMS, should never be introduced into the laboratory under any circumstances.

No work on materials as specified in Section 8.15 of CCSMS and requiring the use of restricted areas should be performed within the Glass Melting Laboratory.

At VSL, waste disposal of hazardous chemicals should be performed in accordance with procedures established by the CUA Environmental Safety Office, as described in Section 8.16 of the CCMSM.

3.2.3 Emergencies

The following are considered laboratory emergency cases:

- chemical spills, to be dealt with according to Section 8.17 of the CCMSM.
- fire and fire-related accidents, to be controlled according to Section 8.21 of the CCMSM.
- personal contamination, to be treated according to Section 8.19 of the CCMSM.
- injury, to be treated in accordance with Section 8.18 of CCSMS.

Cases requiring minor first aid should be treated in accordance with Section 8.20.

3.2.4 Radioactive Hazard Mitigation

Control and monitoring of personnel exposure and environmental radiation at VSL are performed in accordance with Chapter 3 of the Radiation Safety Manual (RSM) of the Catholic University of America.

Application of the rules prescribed by RSM is the responsibility of each user working in the restricted area (rooms 37 to 40 of Hannan Hall) and the project managers.

Dr. Warren E. Keene, CUA - Radiation Safety Officer (CUA-RSO) and Dr. Isabelle Muller, VSL Radiation Safety Officer (VSL-RSO) are responsible for ensuring that the work is done safely and in a manner consistent with applicable regulations.

3.3 PERSONNEL PROTECTION

Basic elements of personnel protection are the following:

- proper identification of hazards (cf. Section 3.2.1 above),
- proper handling and storage of chemicals and other hazardous materials (cf. Section 3.2.3 above) and
- proper dealing with emergencies (cf. Section 3.2.3 above).

To ensure reduction of the radiation exposure, the following measures, prescribed by Section 8.35 of the CCHP, must be implemented:

- environmental monitoring (8.35.1),
- inspections, maintenance and housekeeping (8.35.2),
- use of protective apparel and equipment (8.35.3),
- posting of signs and labels (8.35.4), and
- proper dealing with accidents and spills (8.35.5).

Ventilation of both general laboratory spaces and of special areas and local devices such as chemical fume hoods and glove boxes at VSL should be performed following the instructions of Section 8.36 of the CCHP.

The Glass Melting Laboratory at VSL should not conduct operations with hazardous substances which require a special review of laboratory activities under Section 8.38 of the CCHP, i.e.

- reproductive toxins,
- chemicals which have a high degree of acute toxicity, and
- chemicals considered select carcinogens.

Responsibility for chemical hygiene rests with all employees. Specific chemical hygiene responsibilities as applied to the Glass Melting Laboratory are specified in Section 8.40 of the CCHP.

Animal work, whether or not involving chemicals of high chronic toxicity, according to Section 8.41.5 of the CCHP, should not be carried out in the Glass Melting Laboratory at the VSL.

3.3.1 Special Rules Applicable at VSL

Besides general instructions described in the CUA Radiation Safety Manual, the following rules apply to the protection of personnel working in the restricted area of the VSL.

1) All personnel working in the restricted area shall routinely undergo radiation safety training given by the Radiation Safety Officer of CUA or provided at other facilities acceptable to the CUA-RSO, such as Georgetown University, NIH or NIST.

2) The restricted area should remain locked and only workers assigned to this area, and project managers should have access to this section. All other persons who need temporary access to the radioactive laboratory shall be accompanied by authorized personnel.

3) Film badges shall be worn by operation personnel upon entering the restricted area. Ring badges and rubber gloves shall be worn when handling radioactive material.

4) Shoe covers are to be worn in the Melter room at all times.

5) All spills must be cleaned up immediately. The contaminated areas should be checked after cleaning with a survey meter. The Radiation Safety Officer shall be notified as soon as possible, so that wipe tests may be performed.

   In the case of unenclosed spills greater than about 100 *ml* CUA-RSO should be immediately notified.

   CUA-RSO's CUA phone : (202) 319-5206
   home phone number : (703) 671-1659.

6) All samples have to be labelled with sample name, date and Logbook/page indicating sample preparation.

   All new sample have to be entered into the inventory control file. Any material not in current use must be returned to the storage area in Room 40.

7) Respirators have to be worn as backup protection for the operations involving the possible release of contaminated dust.

8) Repairs to equipment involving hazardous operations should not be performed unless under the direction and in the presence of one or more of the following:

   R. Mohr, H. Hojaji, I. Muller, W. Keene and P. B. Macedo.

9) If alone and a hazardous situation occurs do only what is necessary to prevent a further

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hazard.

To obtain assistance, immediately notify one of the following:

Bob Mohr	*Phone:*	*(202) 234-5953,*
Hamid Hojaji		*(301) 365-0999,*
Isabelle Muller		*(703) 534-5420 or*
Pedro Macedo		*(301) 229-0408.*

10) Notify Campus security of any fire or serious accident.

Campus Security Extension: 5111

4.0 REQUIREMENTS

4.1 TRAINING

All VSL employees working in the radioactive restricted area and, especially, all glass melter workers, must be trained in the radiation and chemical safety procedures by the laboratory supervisors and by the CUA-RSO.

Volume 10 of the Code of Federal Regulations, part 19 (10 CFR 19) and 10 CFR 20, specifying radiation safety regulations, is available to all restricted area workers and is on file with the VSL-RSO.

Each restricted area worker must attend a course in radiation safety at an institution acceptable to the CUA-RSO, such as NIST, NIH or Georgetown University.

Training on the technical methods applied in the Glass Melting Laboratory is given by the laboratory supervisor and/or by experienced persons designated by the laboratory supervisor. The training is deemed completed when the trainee formally demonstrates competence in the independent use of the technical methods taught.

5.0 PREREQUISITE

In order to ensure controlled introduction, distribution, safe handling and transfer of radioactive samples at VSL, measuring equipment has to be regulary maintained and calibrated. Besides measuring equipment other records and materials have to be maintained.

CUA-RSO should maintain or provide for the following:

- Logbook: RSO-1 (Radioactive Material Inventory Control);
- Form RSO-1 (Radioactive Material Inventory Control);
- Form RSO-6 (Inspection Report);
- Geiger-Mueller Survey Meter, with pancake probe;
- Wipes; and
- Tools appropriate for opening of containers with radioactive materials.

VSL-RSO should provide for the following:

- Logbook VSL-301-92;
- Disposable Gloves; and
- Tools appropriate for opening of containers with radioactive materials.

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6.0 OPERATIONAL PROCEDURES

6.1 EQUIPMENT LISTS

The equipment necessary for proper monitoring of radiation levels and measuring the amounts of samples used and distributed in various operations at VSL is listed in the Table below.

Calibration codes are appended to the Table.

Table: BALANCES and GEIGER COUNTERS

Qty	Device	Manufacturer	Model No.	Calib. Req.
1	45kg Platform Scale	Circuits & Systems	BX100, #3422	I
1	1.2kg Balance	Mettler	PL1200 (=A11138)	III*
1	Dial-O-Gram Balance	Ohaus	310	II
1	Balance	Scientech	5200	III
2	Calibration Weights	CUA	27g and 155g	
1	Std. Weight, Class S	Fisher	1g	
1	Std. Weight, Class S	Fisher	20g	
1	Std. Weight, Class S	Trommer	1.5 kg	
1	Std. Weight, Class S	Trommer	10 kg	
1	Std. Weight, Class S	Trommer	20 kg	
1	nominal weight		50 lb	
1	Geiger Counter	various	head: HP-260	

Calibration codes: QM= Quantitative Measurements Only; I= ± 0.1kg accuracy, every 6 months or as needed; II= ± 0.1 g accuracy, every 6 months or as needed; III= ± 0.01g accuracy, annually; *= calibrated by manufacturer representative

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6.2 PROCEDURES FOR EQUIPMENT CALIBRATION

6.2.1 BALANCES

CALIBRATION OF "Circuits and Systems #3422" PLATFORM SCALE

Calibration of this balance should be performed every six months.

Calibration data should be logged in the current Calibration Logbook.

Calibration procedure requires a set of 1.5 kg, 10 kg and 20 kg standards, traceable to NIST, as well as a 23 kg (50 lb) nominal weight.

1. Allow at least 5 minutes after turning on the power for warmup then zero the balance.

2. For the first calibration data point, place the 1.5 kg standard on the center of the pan. Record the weight and then remove it.

3. Second calibration data point: repeat step 2 using the 10 kg standard instead.

4. Third calibration data point: repeat step 2 using the 20 kg standard instead.

5. Fourth calibration data point: repeat step 2 by placing **both** the 10 kg and 20 kg standards together for weighing.

6. The balance is now calibrated for the range 1.5 to 30 kg. Place the nominal 50 lb weight on the balance and record the reading.

7. Fifth calibration data point: place the 10 kg and 50 lb weights on the center of the pan and record the weight.

8. Sixth calibration point: place the 20 kg and 50 lb weights on the center of the pan and record the weight.

9. Prepare a new sticker giving date of calibration and due date of next calibration and give Logbook/page reference for the calibration data. Due date for the next calibration shall be 6 months from date of the just performed calibration. Sign calibration label and place it on the balance.

10. If the instrument does not meet the calibration requirements, as specified in the above Table, the laboratory supervisor and/or the factory should be consulted for corrective measures.

CALIBRATION OF "Ohaus 310 Dial-O-Gram" BALANCE

Note: Enter data in the current Equipment Calibration Log.

1. Clean the balance and check the tare with the weighing cup in place, adjusting if necessary.

2. Transfer the 50 g, 100 g and 200 g calibration weights into the glove box via the pass through.

3. 1st point, 50 g: Weigh the 50 g weight on the scale and record the value.

4. 2nd point, 100 g: Remove the 50 g weight and weigh the 100 g weight on the scale and record the value.

5. 3rd point, 150 g: Add the 50 g to the scale and record the value.

6. 4th point, 200 g: Clear the scale, weigh the 200 g weight and record the value.

7. 5th point, 250 g: Add the 50 g weight to the loaded scale and record the value.

8. 6th point, 300 g: Remove the 50 g weight from the scale, add the 100 g weight to the scale and record the value.

9. Plot a graph of the observed values vs. the calibration weight values (actual values). Place the actual values on the horizontal axis.

10. The balance is considered within the required calibration if the values are within 1% of the standard calibration values. If the balance is out of calibration, repair or replace as is required.

11. If calibration is complete, fill out a VSL calibration sticker with the date of calibration, person who performed the calibration and date the next calibration is due. Place the sticker on the glove box window edge above the balance.

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CALIBRATION OF "Scientech 5200" BALANCE

Enter data in the current Calibration Logbook.

Two Fisher class S standard weights are needed for this procedure.

1. Turn on balance, wait until self-test is completed and **0.00 g** is displayed. If 0.00 g is not displayed, press RANGE button to select HI range.

2. Place 1 g standard weight on balance and record displayed weight in Logbook.

3. Remove 1 g standard weight on balance and replace with 20 g standard weight. Record displayed weight in logbook.

3. Weights should not differ from standard values by more than 0.01 g. If instrument is outside this range, the manufacturer should be consulted at:

Scientech, Inc. 5649 Arapahoe Ave. Boulder, CO 80303	(303) 444-1361 (800) 525-0522

6.2.2 GEIGER COUNTER

Geiger Counters are calibrated using a standard Cs-137 source, as specified by the ANSI N42-3-1969, which was reaffirmed in 1974.

The calibration is required annually and is performed by the RSO, as specified in the CUA Radiation Safety Manual. The Manual is incorporated by reference to the licenses: 08-02075-03 (by product material license), SUD-157 (source materials license), and SNM-164 (special nuclear material license).

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7.0 SPECIMEN CONTROL AND TRACEABILITY

This section describes proper procedures for receipt and distribution of radioactive materials by both CUA Radiation Safety Office and VSL Radiation Safety Office as well as within the VSL laboratories.

7.1 RECEIPT AND DISTRIBUTION OF RADIOACTIVE MATERIALS BY CUA-RSO

7.1.1 Purpose

The procedure for receipt and distribution of incoming radioactive material serves the following purposes:

- Provides a receipt to the delivering carrier.
- Creates the official CUA record of acquisition and distribution.
- Confirms that the shipment is correctly labeled.
- Determines whether the radiation exposure rate at the surface of the outer container, or contamination on that surface is within prescribed limits.
- Ascertains the radiation exposure rate at the surface of each package for the information of the CUA Authorized User.
- Ascertains the level of radioactive contamination of packaging material and the surface of the package.
- Assists in determining disposition of packaging material.

7.1.2 PROCEDURE

1. Receipt to the Carrier.

A receipt for the incoming shipment shall be provided to the delivering carrier. In most instances, the carrier will present a receipt form for signature. If not, a receipt shall be prepared, in duplicate, by the RSO. A signed and dated original shall be given to the delivering carrier; a signed and dated copy shall be kept with the RSO copy of the shipment documents.

2. Confirm Correct Shipment Labeling.

Radioactive material shipments are required to be labeled to identify the proper shipping name, UN number, isotope identity, radioactivity and transport index.

a. Examine shipping documents for consistency with labeling. Advise the RSO of any discrepancy.

b. Measure the radiation exposure rate at contact with all outer surfaces of the shipping container, using a Geiger-Mueller survey meter with pancake probe. Record the highest value

observed on Form RSO-6 (see Appendix A). Be alert for areas on the surface where the GMC response is significantly higher than elsewhere (hot spots). Such readings may provide the first indication of surface contamination. If hot spots are found, they should be wipe tested prior to opening the shipment. If wipe testing confirms the presence of surface contamination, advise the RSO.

c. Measure the radiation exposure rate at a distance of 1 meter from the outer surface of the shipping container. Record the observation on Form RSO-6 (see Appendix A) rounded up to the first decimal place. If the recorded value is greater than the transport index, advise the RSO.

3. Wipe Test Shipping Container.

Wipe test all outer surfaces of the shipping container. For small containers, wipe 100% of the surface; for large containers; obtain representative wipes on each surface. Record the results on Form RSO-6. Report results greater than 10 dpm/cm^2 (Beta/gamma or 1 dpm/cm^2 alpha) to the RSO.

4. Open Shipping Container.

a. Remove and examine any documentation found inside the top of the container. Compare the documentation with the shipment label with any shipping papers attached externally to the shipping container, and with any other information held regarding the shipment. Advise the RSO of any discrepancies noted.

b. Remove each package of radioactive material from the shipping container.Insure that there can be no additional packages hidden in the packing material. Compare the number of packages, isotope identity, activity, and package identification with the shipping documents. Report discrepancies to the RSO.

c. Wipe test the outer and inner surfaces of each container found inside the shipping container (except the inner surface of a primary container). Record the results on Form RSO-6.

d. Examine each radioactive material package and the packing material for evidence of leakage. Report evidence found to the RSO.

e. Measure the exposure rate at contact with each package, using the survey meter. Record the results on Form RSO-6.

f. If the radioisotopes in the shipment include gamma emitters, count 100%, or a representative sample, of all packaging material which cannot be wipe tested (e.g., vermiculite, clay granules, absorbent padding) using a Ge(Li) or Na(I) detector and multi-channel analyzer.

g. Dispose appropriately of the shipping container, intermediate containers, and packing material. Appropriate disposition may include:

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- Delivery to the Authorized User with the radioactive material package(s).
- Disposal to radioactive waste.
- Disposal to ordinary trash.
- Storage of reusable containers or packaging material.
- Return to vendor for recycling.

If in doubt, consult the RSO.

5. Complete the RSO Records

a. Complete the RSO-1 Form (Appendix A) with respect to each assigned RMIC number.

b. Complete a Form RSO-1, Radioactive Material Inventory Control, for each assigned RMIC number. For shipments involving multiple RMIC numbers, note all RMIC numbers marginally on the sheet with the lower number and use this sheet as the top sheet in the file.

c. Complete Form(s) RSO-6, Inspection Report.

d. Make a copy of each Form RSO-1 and RSO-6, and a copy of all shipping documents.

One copy of each form or document is retained in the RSO RMIC File; one copy is delivered to the VSL-RSO with the radioactive material.

Staple the documents together, with RSO-1 on top, followed by RSO-6 and all other documents.

Mark the top respective RSO-1 forms "RSO" and "USER."

7.1.3 DISTRIBUTION

1. Notify the VSL-RSO by telephone that the radioactive material is available and schedule a time for delivery or pickup by authorized VSL personnel.

2. At the time of transfer, obtain the signature and date of receipt of the Authorized User or his/her representative on the RSO copy of each Form RSO-1. Deliver the USER copy of Form(s) RSO-1 (and attached documents) to the recipient of the radioactive material.

3. Complete the RSO-1 log entry identifying the recipient and date of transfer.

4. File the RSO copy of the documentation.

7.2 RECEIPT AND DISTRIBUTION OF RADIOACTIVE MATERIAL BY VSL-RSO

Radioactive samples will be received at VSL by the VSL-RSO or authorized technician.

Restricted areas (room 35 through 40) are designated to receive all incoming radioactive materials (note: room 35 belongs to the CUA-RSO).

All sample numbers are to be recorded into "Receipt and Shipment" logbook VSL-301-92. These include:

- VSL assigned number (cf. sec. 7.2.3).
- RMIC number from the CUA-RSO.
- RI/FS number assigned by the originator.

An adequate description and weight of the received material will be documented in the log book together with the appropriate signatures and date. All sample quantities and weights shall be determined and their respective hazardous or non-hazardous status established by the CUA-RSO.

7.2.1 Non-Hazardous Samples

These samples will be opened on the bench and weighed.

7.2.2 Hazardous Samples

These samples will be opened in an appropriate size glove box and weighed.

Large 55 gallon drums require a large glove box located in room 37.

Smaller samples can be opened in the small glove boxes elsewhere in the laboratory restricted area.

7.2.3 Assignment of VSL ID-Code to Samples

Each separate sample will have an unique VSL ID-Code assigned to it.

The code will consist of:

- Originator's sample ID number (cf. Appendix C),
- Followed by the letter "R" only for chemically reduced glasses,

- Followed by the numerical value of the temperature (celsius) divided by 100.
- Followed by the letter "H" for heat treated glasses
- Followed by the number of hours of heat treatment.

Example: Shipment dated 4/2/92 RMIC #803
glass WVCM70-Ox-Cu-600C-3H becomes 70C6H3

7.2.4 Receipt and Distribution Forms

The VSL-RSO or other authorized person will fill out two forms:

- **"Hazardous/Mixed Waste Sample and Chain of Custody Record"** (Appendix B, Form VSL #16)

- **"Sample History"** (Appendix B, Form VSL #17)

7.3 INTERLABORATORY TRANSFER AND TRACEABILITY OF SAMPLES

Each sample, while being transferred from one to another laboratory at VSL, will be accompanied by the Sample History form (Form VSL #17).

While transferring sample from one laboratory to another, the following steps will be taken to ensure traceability of the samples:

1. The originating laboratory will sign and date the receiving laboratory's Logbook; including a description of the transferred material.

2. The receiving laboratory will sign and date the originating laboratory's Logbook; including a description of the received material.

Appendix B contains a set of forms which will be used in the manner described above for tracing samples between different laboratories at VSL:

1. Inventory of Experiments for PCT Laboratory,	Room: 336	(Form VSL #01)
2. Sample Inventory for PCT Laboratory,	Room: 336	(Form VSL #04)
3. Solution Sample Tracking	Room: 331	(Form VSL #05)
4. In/Out Logbook for Heat-treatment Laboratory,	Room: 38	(Form VSL #10)
5. Sample Inventory for Heat-treatment Laboratory,	Room: 38	(Form VSL #11)
6. Sample Inventory for S.E.M. Laboratory	Room: 434	(Form VSL #09)
7. Glass Sample Inventory	Room: 335	(Form VSL #12)
8. Sample Log-in book	Room: 38	(Form VSL #15)

Each form will either be glued into the existing Logbooks or will be used in separate three-hole binders, which will also be referred to as "Logbooks" and will be appropriately designated, kept and filed.

7.3.1 Tracing of Samples inside VSL laboratories

The laboratory manager of each VSL laboratory is responsible for

- appropriate data collection,

- maintaining unambiguous and easily understandable records of all operations performed on any glass or any leach test.

The records should be kept in such a way that data for each type of glass are easily retrievable and that an independent reconstruction and evaluation of the experimental activities can be performed.

Upon the entry of a sample (glass or leachant) into any VSL laboratory a new record should be established indicating the laboratory where the sample of the particular form was produced, the so called "Originating Laboratory", or the "Originating Logbook", into which previous operations or treatment are recorded.

Records should also contain the information on the persons performing transfer of the sample between the originating VSL Lab and receiving VSL Lab.

Appendix B contains a set of forms designed for keeping records of data collected during various sample preparation and characterization procedures within the VSL laboratories. The forms allow easy retrieval of data related to glass development activities.

Those forms are:

1. New PCT Test for PCT laboratory	Room: 336	(Form VSL #02)
2. Sampling of Leachant for PCT laboratory	Room: 336	(Form VSL #03)
3. Request for Analysis for Analytical Lab	Room: 331	(Form VSL #06)
4. Heat-treatment for Heat-treatment lab	Room: 38	(Form VSL #07)
5. Phase Content for SEM Lab	Room: 434	(Form VSL #08)
6. New Flow Test for Flow Test Lab	Room: 335	(Form VSL #13)
7. Sampling of Leachant for Flow Test Lab	Room: 335	(Form VSL #14)
8. Radioactive Glass REMELT/RECEIVE Sample Casting/Sieving Distribution	Room: 39	(Form VSL #21)
9. Distribution of Sieved Material	Room: 39	(Form VSL #22)
10 Batch Glass Melting Inventory	Room: 39	(Form VSL #23)
11. Glass Sample Inventory	Room: 331	(Form VSL #24)
12. Glass Dissolution	Room: 331	(Form VSL #25)
13. Receiving Samples for Flow Test	Room: 335	(Form VSL #26)

All forms will be used as pages of appropriate Logbooks or as inserts for properly named and designated three-hole binders.

Every form allows unique identification of the glass which is the subject of the test or operation described in the corresponding Logbook and provides reference to the previous steps, either in the glass preparation procedure or analysis performed.

Responsibility for the modification of the above forms rest with the project managers of the laboratories where the forms are used.

All form could be revised just like any other part of a Technical Procedure, as specified by the VSL QA Manual.

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7.3.2 Sample Division

When a single glass sample is divided into smaller sized samples ("branches"), a copy of the original Sample History form (Form VSL #17) must be attached to each new sample. This form includes the information on the number of "branches".

A record of the total number of divided samples and their individual weights will be entered into the laboratory notebooks (Logbooks) as well.

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7.4 STORAGE

Upon completion of all glass treatments, the samples and their Sample History forms (Form VSL #17) will be returned to the Glass Melting Laboratory for storage.

The laboratory manager will store the samples in room 40, having accounted for all recorded weights appearing on the Sample History form.

The incoming sample description is entered (ID code, weight, date, signature) into the storage Logbook VSL-303-92.

Each container with the radioactive sample will be accompained by the

- **"Hazardous/Mixed Waste Sample Daily Removal/Treatment Record and Chain-of-Custody Record"** form (Form VSL #19),

which will maintain the record of the amount of material removed or returned to storage.

7.5 PREPARATION FOR SHIPMENT OF RADIOACTIVE MATERIALS

Before shipping any material, the laboratory manager or VSL-RSO will generate a radioactivity report which will include the following information (also described in Section 7.6.8):

- The total curie content
- A RSR (Radiation Shipment Record) number (obtained from the CUA-RSO prior to pick-up)
- A description of the contents (quantities, composition, physical form, etc.)

Subsequent to the above, the shipped item is recorded in "Receipt and Shipment" log book VSL-301-92.

For packaging, refer to QA Manual, document VSL-QA-1, Section 13 and to Section 7.6 below.

The prepared package, with the radioactive sample, should be accompained by the filled-out forms RSO-19, RSO-22 and RSO-22A (see Appendix A) which should be obtained from the CUA-RSO.

The prepared package should be delivered, with the outer packaging open, to the CUA-RSO for final shipment.

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7.6 SHIPMENT OF RADIOACTIVE MATERIAL BY CUA-RSO

In accordance with the University licenses, granted by the NRC, only the Radiation Safety Office is authorized to ship radioactive material off-campus. This instruction provides information to guide the Authorized User in preparing radioactive material for shipment by the Radiation Safety Office.

7.6.1 Preparation of Packaging

Preparation of packaging for shipment of the radioactive material includes:

- insertion of the material into an appropriate primary container,
- sealing the container against leakage in transit,
- properly labeling the container,
- inserting the primary container into an appropriate secondary container (when required),
- preparing documentation which is desired to accompany the shipment,
- preparing a request for shipment (Form RSO-22), and
- arranging transfer of the prepared material to the Radiation Safety Office.

Preparation is the responsibility of the Authorized User desiring shipment.

"Primary container" is the container the inner surface of which is in direct contact with the radioactive material (or with the outer surface of a sealed source.

"Secondary container" is the container which is used to protect a primary container from the hazards of shipment and to confine the effects of leakage of the primary container to the interior of the secondary container.

A metal "paint can," together with absorbent/cushioning material, may be an appropriate secondary container when the primary container is a glass ampule.

7.6.2 Selection of the Primary Container

The Authorized User shall select a primary container which is appropriate to the chemical physical and radiological characteristics of the radioactive material to be shipped. If there is uncertainty, the Radiation Safety Office should be consulted.

7.6.3 Insertion of Radioactive Material into the Primary Container

The Authorized User shall insert and seal the radioactive material into the primary container in such a manner as to minimize the likelihood of contamination of the exterior surface of the container or leakage in transit.

The primary container will be wipe-tested by the Radiation Safety Office prior to packaging for shipment.

(Contaminated containers will be returned to the Authorized User for decontamination.)

7.6.4 Selection of the Secondary Container

The Authorized User shall determine whether a secondary container is required or desired, and provide an appropriate secondary container when the determination is affirmative. If there is uncertainty, the Radiation Safety Office should be consulted.

7.6.5 Insertion of the Primary Container into the Secondary Container

The Authorized User shall insert the primary container into the secondary container so that (1) the material may be transported safely to the Radiation Safety Office, and (2) the secondary container may be opened safely by the Radiation Safety Office for inspection and wipe testing.

7.6.6 Labeling

The Authorized User shall affix to the primary container (or to the secondary container if the primary container is too small) a label which includes as a minimum:

- standard radiation warning symbol
- word RADIOACTIVE
- identity and activity of each isotope present.

If the number of isotopes present makes this impracticable, the label shall show the total activity and the documentation shall include a list of isotopes and the activity of each.

Multiple primary containers shall be marked to show a sequential number and the total number of containers (e.g., "#2 of 4")

7.6.7 Documentation

The Authorized User is responsible for the preparation of documents which include all the information necessary for a proper shipment. A copy of the documentation shall be provided for retention by the Radiation Safety Office. As a minimum, the documentation shall include:

- number of primary containers in the shipment.
- identity and activity of each radioisotope in each primary container.
- net volume of liquid or net mass of solid material in each primary container.
- chemical and physical form of the material.
- any special instructions for opening the secondary and primary container(s).

A "Checklist for Preparation of Packages for Shipment" is provided by CUA-RSO as the form RSO-19 (Appendix A).

7.6.8 Request for Shipment

The request for shipment shall be made to the Radiation Safety Office, using the Forms RSO-22 and RSO-22A, submitted in triplicate. The form consists of three parts.

Part A shall be completed by the Authorized User requesting the shipment and the form delivered in triplicate to the Radiation Safety Office.

One copy will be returned to the Authorized User with Part B completed by the RSO to record receipt of the material by the RSO.

One copy will be returned to the Authorized User with Part C completed by the RSO to record delivery of the shipment to a commercial carrier for delivery to the consignee.

8.0 RECORD KEEPING

All shipment receipts, records, forms and Logbooks should be appropriately filed and maintained in a chronological or other order, transparent to an independent examiner.

Every record, while operational should be kept at a safe place in the laboratory or place where the corresponding operation is taking place.

Periodically or when a record form is completed, files shall be transferred to the Central Office for permanent storage and safe-keeping.

9.0 REFERENCES

1. Chemical Materials Safety Manual
The Catholic University of America, Washington, D.C., 1989

2. Chemical Hygiene Plan
(added to the Chemical Materials Safety Manual in February 1991)
The Catholic University of America, Washington, D.C., 1991

3. Title 10 Code of Federal Regulations parts 19 and 20
Office of the Federal Register,
National Archives and Records Administration, Washington, D.C.
(published every year)

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APPENDIX A: CUA-RSO Forms to be used by VSL

1.	Radioactive Material Inventory Control	(Form RSO # 1)
2.	Inspection Report - Incoming Radioactive Material	(Form RSO # 6)
3.	Checklist for Preparation of Packages for Shipment	(Form RSO #19)
4.	Request for Radioactive Material Shipment	(Form RSO #22)
5.	Description of Radioactive Material to be shipped	(Form RSO #22A)

Form RSO-1 THE CATHOLIC UNIVERSITY OF AMERICA RMIC #_____
6-79

RADIOACTIVE MATERIAL INVENTORY CONTROL

MATERIAL DESCRIPTION SECTION ISOTOPE_____

$T_{\frac{1}{2}}$:_____ STATE: SOLID LIQ GAS EMISSIONS: B^- B^+ x G

CHEMICAL FORM:_____ VOLUME OR MASS:_____

SPECIFIC ACTIVITY:_____ TOTAL ACTIVITY:_____

CUA ACQUISITION FROM:_____/DATE:_____

PURCHASE ORDER #:_____ OR TRANSFEROR LIC #:_____

USER INFO RECEIVED BY_____/DATE_____

DEPT:_____/AUTH USER:_____/AU #_____

AUTH EXP DATE:_____/MTRL STORED (RM/BLDG)_____

WITHDRAWAL AND DISPOSITION RECORD

DATE	BY WHOM (1)	ACTIVITY REMOVED	RUNNING INVENTORY	DISPOSITION OF WITHDRAWAL (2)

NOTES: (1) Enter "Decay" when correcting inventory record of short-lived isotopes for radioactive decay.
(2) If other than "radwaste" or to a supplementary RMIC, explain.

RETURN THIS FORM TO RSO WHEN INVENTORY REDUCED TO ZERO

USER'S REMARKS:

Form RSO-6

RADIATION SAFETY OFFICE
THE CATHOLIC UNIVERSITY OF AMERICA
INSPECTION REPORT--INCOMING RADIOACTIVE MATERIAL

Req. No._____ P.O. No. _____

Isotope_____ Activity _____

Package Received Date( ) Time( ) By _____

Package Inspection Completed Date( ) Time( ) By _____

Consignee Notified Date( ) Time( ) By _____

Inspection Results

1. External Dose Rate: Contact at highest point _____ mr/hr

at a 3 feet _____ mr/hr

2. Wipe Test (wipe entire surface of appropriate containers)

RMIC#	ID	Dimension (cm)	Area (cm2)	cpm Gr.	cpm Net	dpm eff=	dpm per 100 cm2	S.E* mr/hr
	Bg	-	-	-		-	-	-

*Surface Exposure Rate

LLD = 4.65 sqrt(Bg) + 2.71 =

Form CUA RSO 19 THE CATHOLIC UNIVERSITY OF AMERICA

CHECKLIST FOR PREPARATION OF PACKAGES FOR SHIPMENT

A. Shipment Identification RSR No._____

1. Ship to: Via:

_____ _____

_____ _____

_____ _____

License No._____

2. Description of Packages & Contents:

_____

_____

_____

B. Checklist:

(Initial) Prior to the first shipment of any package, the shipper shall determin by inspection or appropriate test:

_____ 1. The packaging meets the specified quality of design and constructio

_____ 2. The effectiveness of the shielding and containment are within the limits applicable to or specified for the package design.

Prior to each shipment of any package, the shipper shall ensure by examination or appropriate tests that:

_____ 1. The packaging is proper for the contents to be shipped.

_____ 2. The packaging is in unimpaired physical condition except for superficial marks.

_____ 3. The closure devices of the packaging, including any required gasket are properly installed and secured and free of defects.

_____ 4. For fissile materials, any moderators and neutron absorbers, if required, are present in proper condition.

_____ 5. Any special instructions for filling, closing, and preparing the package for shipment have been followed.

_____ 6. All closures, valves, and other openings of the containment system through which the radioactive contents might escape are properly closed and sealed.

_____ 7. Ensure incoming labels have been removed from package and proper labels and placards have been affixed for shipment.

_____ 8. Ensure shipment is braced so as to prevent leakage or shift of loading under conditions normally incident to transportation.

_____ 9. External radiation and contamination levels are within the allowable limits.

_____ 10. For waste shipments, verify there is no free standing liquid.

_____ 11. Proper notifications and acceptance of site receiving shipment have been made.

DATE:_____ _____

Authorized Signature

FORM CUA RSO-22

REQUEST FOR RADIOACTIVE MATERIAL SHIPMENT

NOTE: Deliver 3 copies of this form and related Form(s) RSO-22A to the Radiation Safety Office. One copy will be returned to you promptly with Part B completed by the Radiation Safety Office as your receipt for the material; a second copy will be returned to you with Part C completed as your record of shipment.

PART A (To be completed by the Authorized User requesting shipment)

Authorized User_____ Date of request_____

Applicable User Authorization Number_____

Shipping Date Requested_____ Ship by: (Circle one) **SURFACE** **AIR**

Number of primary containers to be shipped_____

Number of Form(s) RSO-22A attached_____

Consignee Information:

Nameofindividual_____

Telephone: Voice_____Fax_____

Shipping Address (Shipment can not be made to a P. O. Box)

_____

_____

_____

SIGNATURE OF AUTHORIZED USER_____

PART B Material received by CUA RSO (date-time)_____

s/s_____

PART C (To be completed by the Radiation Safety Office) **RSR No.**_____

Shipping Container Used_____

Consignee contacted for shipment authorization (date-time)_____

Common Carrier_____ BL/AB_____

Picked up (date-time)_____

FORM CUA RSO-22A _____ of _____

DESCRIPTION OF RADIOACTIVE MATERIAL TO BE SHIPPED

NOTE: If all primary containers in this shipment have identical descriptions, attach one Form RSO-22A to Form RSO-22. If primary containers in this shipment have differing descriptions, attach one Form RSO-22A for each description and number each Form 22A in the space provided above.

1. Date of applicable Form RSO-22 _____

2. Primary container identification _____

3. Primary container description _____

4. Authorized User's descriptor(s) _____

5. Physical form of the material (circle applicable) SOLID LIQUID GAS

6. Description of the radioactive material (e.g., aqueous solution, glass bars)

7. Does the material contain component(s) which are:

   CHEMICALLY HAZARDOUS _____ BIOLOGICALLY HAZARDOUS _____

8. If either hazard was indicated in (6), identify and quantify hazardous components.

9. Chemical form of the radioactive material _____

	per container	total for this sheet
10. Volume of liquid	_____	_____
11. Volume and pressure of gas	_____	_____
12. Net mass of solid material	_____	_____

13. Identity and activity of each radioisotope per container and for this sheet (A separate sheet providing this information may be attached if convenient.)

_____

_____

_____

15. Specify any special precautions or instructions to guide the consignee in opening secondary or primary containers.

APPENDIX B: Internal VSL Forms

1.	Hazardous/Mixed Waste Sample and Chain of Custody Record	Room: VSL	(Form VSL #16)
2.	Sample History	Room: 39	(Form VSL #17)
3.	Inventory of Experiments for PCT Laboratory,	Room: 336	(Form VSL #01)
4.	Sample Inventory for PCT Laboratory,	Room: 336	(Form VSL #04)
5.	Sample Inventory for Analytical Laboratory,	Room: 331	(Form VSL #05)
6.	In/Out Logbook for Heat-treatment Laboratory	Room: 38	(Form VSL #10)
7.	Sample Inventory for Heat-treatment Laboratory,	Room: 38	(Form VSL #11)
8.	Sample Inventory for S.E.M. Laboratory	Room: 434	(Form VSL #09)
9.	Sample Inventory for Flow Test L	Room: 335	(Form VSL #12)
10	Sample Inventory for Viscosity Laboratory	Room: 38	(Form VSL #15)
11.	New PCT Test for PCT laboratory	Room: 336	(Form VSL #02)
12.	Sampling of Leachant for PCT laboratory	Room: 336	(Form VSL #03)
13.	Request for Analysis for Analytical Lab	Room: 331	(Form VSL #06)
14.	Heat-treatment for Heat-treatment lab	Room: 38	(Form VSL #07)
15.	Phase Content for SEM Lab	Room: 434	(Form VSL #08)
16.	New Flow Test for Flow Test Lab	Room: 335	(Form VSL #13)
17.	Sampling of Leachant for Flow Test Lab	Room: 335	(Form VSL #14)
18.	Radioactive Glass REMELT/RECEIVE Sample Casting/Sieving Distribution	Room: 39	(Form VSL #21)
19.	Distribution of Sieved Material	Room: 39	(Form VSL #22)
20	Batch Glass Melting Inventory	Room: 39	(Form VSL #23)
21.	Glass Sample Inventory	Room: 331	(Form VSL #24)
22.	Glass Dissolution	Room: 331	(Form VSL #25)
23.	Receiving Samples for Flow Test	Room: 335	(Form VSL #26)
24.	Hazardous/Mixed Waste Sample Daily Removal/Treatment Record and Chain-of-Custody Record"	Room: 39	(Form VSL #19)
25.	Phase Content of Fernald Glasses	Room: 434	(Form VSL #18)
26.	Form VSL #20, under preparation		

Form VSL #01: **INVENTORY of EXPERIMENTS** *Laboratory:* PCT *Room:* 33

West Valley FULL GLASSS ID	

Immersion Date	Experiment	Originating Logbook	Page	Recorded in Logbook	Page	Signed

Verified by:	Date:

VSL Technical Procedure TPI-SH *Rev #0* *DATE 06/92*

Form VSL #02: **NEW PCT TEST** *Laboratory:* PCT *Room:* 336

EXPERIMENT:	IMMERSION DATE:	TIME:

No	Short Sample NAME	Full Glass ID	Originating Logbook	Container ID	Container Weight	Container +Glass Weight	Actual Glass Weight	Total Weight (g)	Weight 1st day (g)
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

Requirements: Nominal Glass Weight (g): Leachant:

Comments: Balance:

Pipet: pH Meter:

Performed by:	Verified by:	Date:	Logbook:	Page: 114

Form VSL #03: **SAMPLING of LEACHATE** *Laboratory:* PCT *Room:* 33(

Experiment:	Sampling Date:	Sampling Time:
pH METER:	Buffer #1, pH	Buffer #2, pH Slope
Sampling volume:	Manufacturer:	Manufacturer:
Dilution:	Lot #:	Lot #:

No.	SAMPLE NAME	Original Weight (g)	Weight prior to Sampling	Weight after Refill (g)	pH	
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						

Comments:
Pipet: Balance:

Performed:	Verified:	Date:	Logbook: 115	Page:

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Form VSL #04: **SAMPLE INVENTORY** *Laboratory:* PCT *Room:* 336

DATE	Full Glass ID	Experiment Name, No. of Samples	Brought in by: Received by :	Originating Lab Logbook/page	MESH Quantity (gr)	Logbook/page

Verified by:	Date:	Logbook:	Page:

VSL Technical Procedure TP-SH *Rev #0* *DATE 06/*

Form VSL #05: SOLUTION SAMPLE TRACING *Laboratory:* Analytical *Room:* 3

ate in	Exeriment NAME	No. of Samples	Lab or Project	Originating Logbook/page	RUN Name	Analytical Lab Operator	**Date out**	**Results out to**

Verified by:	Date:	Note: Unshaded columns, filled by the Originating Lab	**Note: Shaded columns filled by the Analytical Lab**

VSL Technical Procedure TPI-SH__ *Rev #0* *DATE 06/92*

Form VSL #06: **REQUEST FOR ANALYSIS** *Laboratory:* Analytical *Room:* 331

Experiment NAME:	Request made by:	Anal. Lab RUN NAME:
Number of Samples:	Date / / Time :	Anal. Lab Operator
How long can sample stand:	How soon are results required:	

Elements / ions to be analyzed and their expected levels

Element	Approx. Conc. (mg/L)	Element	Approx. Conc. (mg/L)	Element/Ion	Approx. Conc.(mg/L)
Si		Mn		F^-	
B		Mg		Cl^-	
Fe		Li		NO_3^-	
Sr		K		SO_4^{2-}	
Ca		Ni		PO_4^{3-}	
Na		Cr			
Al		Zr			
P		U			
Ti		Th			
Ba					

Comments:

Form VSL #07: **HEAT TREATMENT** *Laboratory:* Heat-treatment *Room:* 3

Full GLASS ID:	Date in:	In/Out Logbook/page:
SPL ID:	**Weight (gr)**	Hazard:
REMARKS:		

Premelting	no	yes	T(C°)	dT(C°)	time:
Heat-Treat	no	yes	T(C°)	dT(C°)	time:
Annealing	no	yes	T(C°)	dT(C°)	time:
REMARKS:					

CUTTING	No	Yes	REMARKS:
POLISHING	No	Yes	REMARKS:

New SPL ID:	Date out:	In/Out Logbook/page
Performed by:	Verified by:	Date:

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Form VSL #08: **PHASE CONTENT** *Laboratory:* SEM *Room:* 434

Full GLASS ID:	Heat treatment **Temperature:**	Heat treatment **Time:**

PHASES	CONCENTRATION OR RANGE (VOL%)	OBSERVATIONS ON MORPHOLOGY OR OTHER CHARACTERISTICS
Spinel		
Haematite		
Lithium Phosphate		
Augite		
Calcium Phosphate		
Rh, Pd, Ru		

Remarks:

Performed by:	Verified by:	Date:	Logbook:	Page:

Form VSL #09: **SAMPLE INVENTORY** *Laboratory:* S.E.M. *Room:*

DATE	Full Glass ID	Originating Logbook	Page	Brought in by:	Received by:

Verified by:	Date:	Logbook:	Page:

VSL Technical Procedure TPI-SH *Rev #0* *DATE 06/92*

Form VSL #10: **IN/OUT LOGBOOK** *Laboratory:* Heat-treatment *Room:* 38

Date In/Out	Full GLASS ID	SPL ID	Originating Logbook/page	Destination Logbook/page	Quantity (gr)	Received from: Received by :	Logbook : Page :

Verified by:	Date:	Logbook:	Page:

Form VSL #11: **SAMPLE INVENTORY** *Laboratory:* **Heat-treatment** *Room:* 38

Date	Full Glass ID	SPL ID	Quantity (g)	New Glass ID	In/Out Logbook/page	Logbook/page

Verified:	Date:	Logbook:	Page:

VSL Technical Procedure TPI-SH *Rev #0* *DATE 05/92*

Form VSL #12: GLASS SAMPLE INVENTORY *Laboratory:* Flow Test *Room:* 335

DATE IN	Full Glass ID	Experiment Name	No. of Samples	Received from: / Received by:	Originating Lab / Logbook/page	Type / Qntity (g)	Flow Test Logbook/page
Verified by:		Date:	Logbook:				Page:

Form VSL #13: NEW FLOW TEST, Powder *Laboratory:* Flow Test *Room:* 33

Experiment:	Immersion Date:	Immersion Time:	Temperature (°C)

. olume of Leachant (ml)	Sample Volume (ml)	Date of Change of Interval :	Initial	Second	Third	Fourth

No	Full Glass ID	Originating Logbook/Page	Container Material/size	MESH	Leachant Type	Leachant Volume (ml)	Initial Weight Full Container	Actual Glass Weight (g)	Specific Area (cm^2/g)	Total Area (cm^2)	Weight 1st day (gr)
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											
25											
26											
27											
28											
29											
30											

eachant #1	Leachant #2	Leachant #3	Termination Date:

Performed by:	Verified by:	Date:	Logbook:	Page:

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rm VSL #14: SAMPLING of LEACHATE *Laboratory:* Flow Test *Room:* 335

XPERIMENT				Sampling No.
rrent Sampling Interval			Sampling Date:	Sampling Time:
H METER:	Buffer #1 pH	Buffer #2 pH Slope	Buffer #3 pH	Buffer #4 pH
ampling volume (mL):	Manufacturer Exp	Manufacturer Exp	Manufacturer Exp	Manufacturer Exp
oom Temperature (°C):	Lot #	Lot #:	Lot #	Lot #

	Container Weight before Sampling	Container Weight after Sampling (g)	pH	No	Container Weight before Sampling	Container Weigth after Sampling (g)	pH	
				16				
				17				
				18				
				19				
				20				
				21				
				22				
				23				
				24				
				25				
				26				
				27				
				28				
				29				
				30				

rformed by:	Verified by:		Date:

Form VSL #15: SAMPLE LOGIN Book *Laboratory:* Viscosity and Conductivity *Room:*

Date In	Full Glass ID	Type Analysis	Submitted by / Received by	Originating Logbook/page	Results Out Date	Remarks
Verified by:			Date:	Logbook:		Page:

Form VSL #16: **Hazardous/Mixed Waste Sample Receipt/Shipment and Chain-of-Custody Record**

DATE RECEIVED:	**RECEIVED FROM:**
Project:	**Project Manager:**
Waybill Number:	**Sample Particulars**

Note: one line per item (even if the same RMIC or RSR number)

RMIC #	RSR #	VSL Full Glass ID	Container Description	Total Weight (g)	RI/FS #	Description of Sample	Activity (μCi)

Samples Stored at: _____ **Remarks:**

Samples intended for/ transferred to: _____

SAMPLE RECEIVED

Logbook/log ref.	Lab Supervisor	Lab Supervisor	Lab Supervisor

SAMPLE SHIPPED

Logbook/log ref.	Lab Supervisor	Lab Supervisor	Lab Supervisor
Verified by:	Date:	Logbook:	Page:

Form VSL #17: SAMPLE HISTORY

DATE RECEIVED	RECEIVED BY
PROJECT	INVESTIGATOR
VSL SAMPLE ID	HAZARD

PLANNING		FOLLOW-UP				
Action	O.K.	Laboratory - Room	Weight	Logbook	Signature	Date
INITIAL						
Ion Exchange						
GLASS MELTING						
Crucible						
Minimelter						
HEAT-TREATMENT						
Standard						
Annealing						
New GLASS ID **						
GLASS CHARACTERIZATION						
Viscosity						
Conductivity						
Phase Content						
Micro-structure						
LEACHING						
PCT						
TCLP						
Flow Test						
ANALYTICAL PROCEDURES						
DC Plazma						
Atomic Absorption						
ICPMS						
SHIPPING OUT						
Final STORAGE						

Verified by :	(**) Copy No.: of Total:	Date:

(**) Please note: If sample branching occurs, make an appropriate number of copies of this form and record ab

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Form VSL #18: **PHASE CONTENT of Fernald Glasses** *Laboratory:* SEM *Room:* 434

Full GLASS ID:	Heat treatment **Temperature:**	Heat treatment **Time:**

PHASES	CONCENTRATION OR RANGE (VOL%)	OBSERVATIONS ON MORPHOLOGY OR OTHER CHARACTERISTICS
Spinel		
Haematite		
Magnesium Silicate		
Augite		
Calcium Silicate		

Remarks:

Performed by:	Verified by:	Date:	Logbook:	Page:

Form VSL #19: **Hazardous/Mixed Waste Sample Daily Removal/Treatment Record and Chain-of-Custody Record**

Laboratory: Glass Preparation *Room:*

RECEIPT RECORD PAGE No.: _____ **PROJECT:** _____

Date	VSL Full ID	Quantity Removed from Storage	Quantity Used/ Treated	Quantity Returned to Storage	Balance in Custody	Where Used	Treated/ Disposed

Verified by:	Date:	Logbook:	Page:

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VSL Form #21 **Radioactive Glass REMELT/RECEIVED** *Laboratory:* Glass Preparation *Room:* 39

Sample Casting/Sieving Distribution

Refer to VSL bach name and number	Run - Crucible:
Notes	DATE (M/D/Y)
Shape (bars, plates, squares, etc):	Total WEIGHT (g)

Glass Casted or Received, List Separately Each Glass Item :

Item,description	Weight	Destination*	Book/Page	Signature	Date*
1					
2					
3					
4					
5					

Glass Crushed and Sieved: (*) leave blank if item sent to STORAGE

Item, description	40 - 60	100 - 200	UNGRADED	.....	Losses

Distribution of Sieved Material:

Grams:					
Date:					
Issued by:					
Destination:					
Logbook/page:					
Received by:					
Grams:					
Date:					
Issued by:					
Destination:					
Logbook/page:					
Received by:					
Verified by:		Date:	Logbook:		Page:

Form VSL #22: **Radioactive Glass REMELT/RECEIVED** *Laboratory:* Glass Preparation *Room:*

Sample Casting/Sieving Distribution

Distribution of Sieved Material		**RUN-Crucible**			
Sieved	40 - 60	100 - 200	Ungraded	.....	LOSSES
Grams:					
Date:					
Issued by:					
Destination:					
Logbook/page:					
Received by:					
Grams:					
Date:					
Issued by:					
Destination:					
Logbook/page:					
Received by:					
Grams:					
Date:					
Issued by:					
Destination:					
Logbook/page:					
Received by:					
Grams:					
Date:					
Issued by:					
Destination:					
Logbook/page:					
Received by:					
Grams:					
Date:					
Issued by:					
Destination:					
Logbook/page:					
Received by:					
Verified by:		Date:	Logbook:		Page:

Form VSL #23: **Batch Glass Melting Inventory** *Laboratory:* Glass Preparation *Room:* 39

Batch No.:		Record from: to:		
Removed Date	Destination Logbook/page	Signature	Full GLASS ID	Sample Weight (g)

Verified by: Date: Logbook: Page:

Form VSL #24: **GLASS SAMPLE INVENTORY** *Laboratory:* Analytical *Room:* 3

Date in	Sample Set Name	Full Glass ID	Glass Weight (g)	Originating Logbook/ page	Received from	Anal. Lab. Operator	Date out	Sample out to	Sample out by

Verified by:	Date:	Filled by Anal. Lab	Logbook:	Page:

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Form VSL #25: **GLASS DISSOLUTION** *Laboratory:* Analytical *Room:* 331

Date in	Full Glass ID	Anal. Lab Operator	Glass Weight (g)	Acid Volume (ml)					Approx. Temp (°C)	Time (hr)	Final Volume (ml)
				HF	HCl	HNO_3					

Verified: Date: | Filled in by Anal. Lab Logbook: Page:

Date in	Full Glass ID	Project	Originating Logbook	Page	Received from	Received by
Processing Atmosphere:						

FULL COMPOSITION (WEIGHT %)					
SiO_2		Fe_2O_3		TiO_2	
B_2O_3		MnO		ZrO_2	
P_2O_3		Cr_2O_3		UO_3	
Na_2O		NiO		ThO_2	
Li_2O		MgO		Ce_2O_3	
K_2O		CaO		Nd_2O_3	
Cs_2O		SrO		ZnO	
Al_2O_3		BaO		CuO	
				CoO	
				F	
Verified by:		Date:	Logbook:		Page:

APPENDIX C: Sample Identification Codes

Samples for the waste treatability studies will be identified by a contraction of the project (site) name, as follows:

West Valley

WVCM xx
WVUTh xx

Fernald

FE xx

Weldon Spring

WS xx

Oak Ridge

OR xx

Appendix D: List of Abbreviations

CCHP CUA Chemical Hygiene Plan

CCMSM CUA Chemical Materials Safety Manual

CUA Catholic University of America

CUA-RSO Catholic University of America-Radiation Safety Officer

GMC Geiger Muller Counter

NIH National Institute of Health

NIST National Institute of Standards and Technology

UN United Nations

VSL Vitreous State Laboratory

VSL-RSO Vitreous State Laboratory-Radiation Safety Officer

UNCONTROLLED

VSL Technical Procedure

Title: Procedure for Characterization of Physical Properties and Carbon Content of Wastes and Glass

Document: TPI-FC

Issued: 10/92 Rev #1

Prepared by: Noel Balitactac Laboratory Manager	Gerardo Del Rosario Project Technician
Approved by: Pedro B. Macedo Co-Director, VSL	Ian L. Pegg Project Manager
	C. Sivaprakash Quality Assurance Officer, VSL

Procedure for Characterization of Physical Properties and Carbon Content of Wastes and Glass

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TPI-FC

1.0 PURPOSE

This procedure describes the characterization of feed materials such as soil, sludge or fly ash in terms of its thermogravimetric properties, carbon content, specific gravity and particle size distribution. This procedure also provides the means for obtaining verifiable, reproducible, documented data from all tests performed.

2.0 APPLICABILITY

This procedure applies to all VSL personnel trained and authorized to handle radioisotope-containing samples. Activities covered by this procedure include:

(a) Thermogravimetric analysis
(b) Analysis of total carbon or total organic carbon
(c) Specific gravity determination
(d) Particle-size analysis - sieving
(e) Sample accounting for traceability

3.0 SAFETY

3.1 Hazards

3.1.1 Chemical Hazards

The definition of a hazardous chemical is given in Section 8.3 of the CUA Chemical Materials Safety Manual, adapted from the OSHA Hazard Communication Standard. The CUA Chemical Materials Safety Manual is hereafter referred to as the CCMSM. This Standard and its application to CUA facilities are described in Sections 8.1 of the CCMSM with the exceptions noted in Section 8.28.

3.1.2 Radioactive Hazards

The radioactive isotopes present in the sample (sludge, soil or fly ash) are the source of radioactive hazard.

3.2 Hazard Mitigation

3.2.1 Identification of Hazards

The identification of chemical hazards should include an education program (Section 8.2 of the CCMSM). This program should include familiarization with the CCMSM as a whole, including the toxic properties of chemicals according to Sections

8.22, 8.23, 8.24 and 8.25 of the CCMSM, other hazards of chemicals classified under Sections 8.9, 8.10, 8.11, 8.12, 8.13, and 8.14 of the CCMSM, chemical handling and storage procedures (see paragraph 3.2.2 below), and emergencies (see paragraph 3.2.3 below). The education program will also include familiarization with the CUA Chemical Hygiene Program (CCHPP) when the document describing this Program is finalized and approved for distribution by the University.

Other elements of hazard identification include the proper use of labels (Section 8.4 of the CCMSM) and of Material Safety Data Sheets (Sections 8.25, 8.26, and 8.27 of the CCMSM). Workplace chemical lists should be compiled, maintained, and made available for employees in accordance with Section 8.31. In addition, a copy of the CCMSM should be kept in the laboratory and its use should be included in the education program cited above. The same should apply to the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University.

3.2.2 Handling and Storage of Chemicals

Handling of chemicals should be conducted according to Section 8.6 of the CCMSM. Storage of chemicals should follow Section 8.8 of the CCMSM. Special precautions should be taken in handling and storage of incompatible chemicals, solvents, compressed gases, and radioactive materials according to Section 8.10, 8.12, 8.13, and 8.14 of the CCMSM, respectively.

Waste disposal of hazardous chemicals should follow procedures established by the CUA Environmental Safety Office according to Section 8.16 of the CCMSM.

3.2.3 Emergencies

Chemical spills should be dealt with according to Section 8.17 of the CCMSM. Fire and fire-related emergencies should be controlled according to Section 8.21 of the CCMSM. Cases of personal contamination should be treated according to Section 8.19 of the CCMSM, cases of injury and illness according to Section 8.18, and cases requiring minor first aid according to Section 8.20.

3.2.4 Radioactive Hazard Mitigation

All rules for control and monitoring of personnel exposure and environmental radiation are described in Chapter 3 of the radiation safety manual of The Catholic

University of America. Application of these rules is the responsibility of each user working in the restricted area (rooms 37 to 40 of Hannan Hall) and the project managers, under the guidance of Dr. Warren E. Keene, Radiation Safety Officer of CUA.

3.3 Personnel Protection

Personnel protection from hazardous chemicals should consist of two elements. The first is familiarization and compliance with the instructions of the CUA Chemical Materials Safety Manual (CCMSM), including identification of hazards (see paragraph 3.2.1 above), handling and storage of chemicals (see paragraph 3.2.2 above), and dealing with emergencies (see Paragraph 3.2.3 above). The second element is familiarization and compliance with the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University. The overall chemical hygiene program (section 8.34 of the CCHP), based on the OSHA Regulation on Occupational Exposure to Hazardous Chemicals in Laboratories, should be followed. In detail, exposure reduction measures (Section 8.35 of the CCHP) will include environmental monitoring (8.35.1), inspections, maintenance and housekeeping (8.35.2), use of protective apparel and equipment (8.35.3), posting of signs and labels (8.35.4) and dealing with accidents and spills (8.35.5). Ventilation of both general laboratory spaces and of special areas and local devices such as chemical fume hoods and glove boxes should follow the instructions of Section 8.36 of the CCHP. Employees should take part in the safety information and training program which exists in the University according to Section 8.37 of the CCHP, and should be informed about the medical program provided by the University in cases of work with hazardous substances according to Section 8.39 of the CCHP. Responsibility for chemical hygiene rests with all employees.

In addition to compliance with the general instructions described in the CUA radiation safety manual, the following rules apply to the protection of personnel working in the restricted area of the Vitreous State Laboratory:

1) All personnel working in the restricted area shall routinely undergo radiation safety training given by the radiation safety officer of CUA or in off-campus courses approved by the CUA RSO (see Section 4.1).

2) The restricted area should remain locked and only workers assigned to this area and project managers should have access to this section. All other persons who need temporary access to the radioactive laboratory shall be accompanied by authorized personnel.

3) Film badges shall be worn by operation personnel upon entering the restricted area. Ring badges and disposable gloves shall be worn when handling radioactive material.

4) Shoe covers are to be worn in the melter room at all times.

5) Any radioactive spill must be cleaned up immediately, checked after cleaning with a survey meter, and the radiation safety officer shall be notified as soon as possible so that wipe tests may be performed. Immediately notify RSO of any unenclosed spills greater than about 100 ml.: phone x 5206, home (703) 671-1659.

6) All samples shall be labeled with sample name, date and notebook/page of record indicating sample preparation. All new materials shall be entered into the inventory control file. Any material not in current use must be returned to the storage area in Room 40.

7) Respirators are to be worn as backup protection for operations involving the possible release of contaminated dust.

8) Repairs to equipment involving hazardous operations should not be performed unless under the direction and in the presence of one or more of the following: R. Mohr, H. Hojaji, I. Muller, W. Keene, P.B. Macedo. If alone and a hazardous situation occurs do only what is necessary to prevent a further hazard, and report the problem promptly to your supervisor.

Refer to previous section 3.2.3 for emergency response. Notify Bob Mohr (202) 234-5953, Hamid Hojaji (301) 365-0999, Isabelle Muller (703) 534-5420 or Pedro Macedo (301) 229-0408 to obtain assistance. Notify Campus security and or the fire department of fire or serious accident. Security, x 5111. Fire and emergency, dial 9 for outside line, then 911.

4.0 REQUIREMENTS

4.1 Training

All radioactive restricted area workers shall be trained both by the laboratory supervisor regarding their specific duties and by the CUA radiation safety officer.

Documents 10CFR19 and 10CFR20 are available to all restricted area workers. Each restricted area worker must attend a course in radiation safety given by or approved by the CUA RSO.

Training on each technical method and equipment used in radioactive counting is given by the laboratory supervisor and/or by a trained and experienced person designated by the laboratory supervisor until the technician shows competence to use that method without direct supervision.

5.0 PREREQUISITES

5.1 Equipment/Materials List

Thermogravimetric Analysis:

QTY	EQUIPMENT/MATERIAL	MANUFACTURER/MODEL
1	Furnace	Deltech/DT-28-12
1	Furnace	Deltech/DT-28-06
1	Laboratory box furnace	Neytech
1	Balance, 100g	Sartorius/B120S
1	Balance, 1.2kg	Mettler/PL1200
1	Crucible, Pt-Au, 430g	
1	Crucible, Inconel, 500g	
	Fireclay crucibles, 400g	
	Fireclay crucibles, 1.4kg	
	Disposable latex gloves or	
	Disposable vinyl gloves	
	Desiccator	
	Crucible tongs	
	Green goggles	
	Glovebox	

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Carbon Content Analysis:

QTY	EQUIPMENT/MATERIAL	MANUFACTURER/MODEL
1	Total organic carbon analyzer	Dohrmann/DC-80
1	Boat sampling module	Rosemount Analytical/183
3	Platinum boat, P/N 511-793	Rosemount Analytical
1	Stopwatch/Timer	
	Soap film bubble meter	
	Tweezers	
	20mL Glass or polystyrene vials	
	250mL Amber bottles	
	Spatula	
	Mortar and pestle, Diamonite	
	Quartz wool	
	Oxygen gas cylinder	

Note: Refer to the ASTM procedures in the appendix for the rest of the apparatus/equipment required in the specific gravity and particle-size analysis. For the calibration of the above listed equipment refer to the prerequisites section of the Glass Preparation and Minimelter procedure, TPI-GP.WVN.

6.0 Operational Procedures

6.1 Thermogravimetric Analysis of Soils, Sludges and Fly Ash

Note: The analysis should normally be performed in a platinum crucible (Pt-Au or Pt-Rh) but clay or inconel crucibles may be used at the direction of the Project Manager. If using a platinum crucible, ensure that no metallic objects come into contact with the hot crucible.

1. Sampling

Soil and fly ash are sampled by the method of quartering. Spread out the contents of the soil or fly ash from its bucket on a large plastic sheet

(obtain approval from Project Manager whether this can be done outside or inside the glovebox or laboratory hood, if outside the glovebox, wear the proper mask to avoid inhalation of hazardous particles), mark off quarters, and take scoop-fulls from each quarter collecting the desired sample volume. The samples obtained may contain large rocks, stones, soil aggregates, roots or vegetation. Consult the Project Manager whether these components are to be included or discarded from the samples for analysis.

For sludges, thoroughly mix the sludge in the drum until homogeneous to ensure that a representative sample is taken.

2. Weigh a clean dry crucible and record the weight in laboratory notebook, VSL #289-92.

3. Place crucible into a plastic bag, pass into glovebox and transfer into it the soil, sludge or fly ash to be analyzed.

4. Remove the crucible with sample from the glovebox, weigh and record the weight. Calculate the weight of the total sample removed and record this value in its corresponding "Daily Removal/Treatment and Chain-of-Custody Record", (Form VSL #19) compiled in laboratory notebook VSL #328-92.

5. Place the crucible with the sample into a furnace.

6. Drying of the samples is done in stages given in the table below unless otherwise specified by the Project Manager.

Temperature	Drying Time
110°C	18 hrs
450°C	4 hrs
850°C	4 hrs
1150°C	4 hrs

7. At each drying stage,

   (a) Set the furnace to the selected temperature and hold for the specified period of time.

   (b) At the conclusion of each drying time, shut off the furnace and allow the sample to cool inside the furnace.

   (c) When the furnace has cooled to around 200°C, (except for the first stage) remove the sample from the furnace and place in a desiccator to cool to room temperature.

   (d) Remove the sample from desiccator, weigh, and record the total mass of dry sample plus crucible.

   (e) Calculate and record the percent weight loss of the sample after drying.

8. After each drying stage, it may be necessary to take a sample of the dried material for analysis, e.g. 5 grams for chemical analysis, (per instruction of Project Manager). In this case, weigh and record the final mass of sample plus the crucible before proceeding to the next drying stage.

9. After completing the last drying stage, the dried material is transferred from the crucible to a clean sealable plastic jar and label indicating sample name (see section 7.0 below), and reference VSL Laboratory Notebook Number.

6.2 Analysis of Carbon Content

The measurement of the carbon content of sludge, flyash or soil samples is done using the Dohrmann DC-80 Carbon Analyzer attached with the 183 Boat Sampling Module (BSM). This system is capable of measuring the total carbon (TC), total organic carbon (TOC), and the inorganic carbon (IC) contents of wastewater, slurries, sludges and solid samples. Refer to the DC-80 Carbon Analyzer Equipment Manual and the Operation Manual of the Boat Sampling Module for additional information on the features and operation of the system.

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1. Start-up and calibration

Before attempting operation, verify and check the following:

- the IR detector has been on for at least 2 hours, for temperature stabilization. Ultimate stability is attained after 24 hours.

- the carrier gas (oxygen) is flowing at the detector "out" port in the range of 180-220 mL/min. This is checked using a soap film bubble meter. See Appendix E, page 10-1 of the DC-80 Manual.

- the TOC/POC mode switch on the DC-80 unit is in the TOC position.

- the detector/ppm switch on the DC-80 unit is in the ppm position.

- the disperser at the bottom of the sparger of the BSM is well submerged in acidified (with phosphoric acid, to pH 2) deionized water.

- the mist trap of the BSM should not be more than half filled with water. Empty out if necessary.

- inspect the tin/copper scrubber for presence of water. Replace if necessary.

(a) Turn on the combustion furnace of the Boat Sampling Module (BSM) by switching the power on located on the front panel of the module. Wait for about 15 minutes for furnace to stabilize at 800°C, factory preset temperature.

(b) Turn on the white power switch of the DC-80 unit. The readout will display "----" (indicates no printer connected) and the red indicator (error) is lit. Pressing the start button twice clears the readout to 0.0000 and lights up the green (ready) indicator.

(c) When the green LED indicator on the front panel of the BSM becomes lit, the furnace is stable at about 800°C. (Verify this temperature by following the test procedure specified on page 5 of the BSM Operation Manual).

(d) With the furnace at the desired operating temperature, advance the platinum boat into the furnace to bake for about 2 minutes. This eliminates any

carbonaceous material residing in the boat. Then, retract the boat until it is at the hatch port.

(e) Place tufts of quartz wool in the sample boat, then bake as described in step d. The wool helps promote even vaporization of sample while in the furnace.

(f) Calibrate the system by injecting 40 uL of Potassium Hydrogen Phthalate ($C_8H_5O_4K$, KHP) standard containing 2000 ppm C. This standard is prepared by dissolving 425 mg of KHP in 100 mL deionized water and 0.1 mL of concentrated phosphoric acid (refer to Appendix A, page 6-1 of the DC-80 Manual). Record this calibration data in logbook VSL #329-92.

2. Sample pretreatment

a) Samples containing particulates, such as slurries and suspensions, must be treated for the solids to pass through the needle of the syringe or the tip of a micropipette (consult the Project Manager if the particulates are to be included in the analyses). When the particulates must be included in the analyses, pulverize the sample using a mortar and pestle, or a Tissuemizer or any kind of homogenizer, if available. Samples which can not be pulverized must be analyzed as solid samples. After homogenization, proceed to acidify the sample.

b) For solid samples, pulverization results in greater homogeneity which allows efficient vaporization of the sample in the furnace. Weigh about 50 mg of the pulverized sample and add in 5 mL of deionized water in a 20 mL vial. Thoroughly mix the solution, then proceed to acidify the sample.

c) Acidify the sample with a few drops of concentrated phosphoric or sulfuric acid to pH 2-3, if analyzing for TOC. (For TC analyses, no acidification is necessary. Proceed to sample analysis.)

d) Sparge the acidified sample for 6 minutes in the sample sparging station of the BSM.

3. Sample analysis

a) Check the system calibration by injecting 40uL of 2000 ppm C KHP standard. Recalibrate the system if necessary. (Refer to page 3-5 to 3-9 of the DC-80 Manual).

b) Situate the sample boat at the hatch port.

c) Lift the hatch and transfer the sample to the boat using a micropipette or syringe. (Samples can also be transferred via the septum syringe port of the race tube near the hatch port.) Ensure complete transfer of sample from the syringe or micropipette by touching the needle or pipet tip to the quartz wool. If the boat was recently retracted from the furnace, allow at least 30 seconds for cooling before injecting the sample into it.

d) Close the hatch and wait for 1-2 minutes to allow detector baseline to stabilize.

e) Press the START button of the DC-80 unit and advance the boat into the furnace at about 2 inches per second.

f) Wait 2-8 minutes for the analysis to finish. When the detector signal is essentially back to original base line, the analysis terminates automatically and a beeping sound is activated. If the red (error) light did not come on, the analysis is successful. Record the reading on the display panel in logbook VSL #329-92. If the red light comes on, the analysis is erroneous. Refer to pages 3-9 and 3-10 in the DC-80 Manual for comments on error messages.

g) Retract the boat back to the hatch port when the analysis is finished. Allow the boat to cool down for at least 30 seconds before injecting another sample.

h) Do at least 5 trials per sample.

i) Correct the panel reading given in ppm C (by volume) to ppm C (by weight) in solid samples by correlating the amount of carbon and the concentration of the standard used in the calibration, to the reading obtained on a known weight of sample. (Refer to the sample calculation on pages 9 and 10 of the BSM Operation Manual).

j) Calculate the average of the corrected values and the standard deviation. Record all results in logbook VSL #329-92.

6.3 Specific Gravity

The specific gravity of soil, sludge and fly ash is determined by following ASTM D854-83 - Standard Test Method for Specific Gravity of Soils (see Attachment III

in the Appendix). To determint the specific gravity of glass, the glass sample is crushed to pass No. 10 (2.00 mm) sieve, and then ASTM D854-83 followed.

6.4 Particle Size Analysis

Particle size analysis of soil, sludge and fly ash is done using the ASTM D422-63 - Standard Test Method for Particle-Size Analysis of Soils, and ASTM D421-85 - Standard Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants (see Attachments II and I, respectively, in the Appendix).

7.0 Specimen Control/Traceability

Thermogravimetric Analysis: When a sample of sludge is taken from the drum or soil or fly ash is taken from its bucket for thermogravimetric analysis, the sample removed is recorded in their corresponding "Daily removal/treatment and chain of custody form", (Form VSL #19), compiled in the loose leaf binder, VSL #328-92. This sample removal is also documented in the users laboratory notebook, VSL #289-92 or VSL #300-92. After undergoing the final drying stage, the dried material is transferred to a container with a label indicating its sample name and the process the sample underwent. For example, FE1-D450 in the label, will stand for:

FE1 - Fernald Pit 5
D1150 - D for Dried, 1150 for 1150°C drying temperature

The label will also include the reference VSL laboratory notebook number and page where the process was recorded. This container with the dried sample is stored in the Fernald processed sample storage area temporarily located in room 39.

Carbon Content: All samples for analysis of carbon content (TOC or TC) will be logged in the logbook VSL #329-92 with a description of the sample, name, originator and its reference VSL laboratory notebook #. Any pretreatment process that the sample undergoes e.g. grinding or pulverization, or dissolution in water, shall be recorded in the logbook and the processed samples shall be labeled to reflect its name and reference logbook (VSL #329-92) and page number. All leftover and processed samples are saved and stored temporarily near the Dohrmann Carbon Analyzer while the originator or the responsible

person is notified. All samples are to be returned to the originator who must sign out the samples in the log book, VSL #329-92.

Specific Gravity: Sampling of materials for specific gravity test and its documentation is the same as stated above. Processed samples are not saved and are directly disposed of to a Fernald waste container.

Particle Size Analysis: Sampling of materials for particle size analysis and its documentation is the same as stated above. After sieving the samples, the fractions retained and passing each of the sieves used are collected and placed in a container with a label indicating the sample name and the sieve used, FEa-Sbxxx (a=1 for Pit 5, 2 for Pit 6, 3 for Clearwell, 4 for Fly ash RMIC 877, and 5 for Fly ash RMIC 878. For soils, FEa is replaced with their RMIC #. b=P for fraction passing, R for fraction retained; xxx=the mesh or sieve number.) For example, FE1-SP200 in the label will stand for:

FE1 - Fernald Pit 5
SP200 - Sieved Passing #200 mesh size sieve

or 815-SR10

815 - Fernald Soil with RMIC #815
SR10 - Sieved Retained at #10 mesh size sieve (passed the next coarser sieve used in the analysis)

The label has to include the reference laboratory notebook VSL # and page where the process was recorded. The samples generated from particle size analysis are stored in the Fernald processed samples storage area temporarily located in room 39.

8.0 Record Keeping

The following is a list of laboratory notebooks and forms currently in use where all data and activities pertaining to all of the analyses performed on Fernald samples described above are recorded.

1. VSL #289-92 - Thermogravimetric analysis
2. VSL #300-92 - Specific gravity and particle size analysis

TPI-FC

3. VSL #328-92 (Loose Leaf Binder) - Daily Removal/Treatment and Chain-of-custody record
4. VSL #329-92 - Carbon content (TOC/TC) analysis logbook
5. VSL #306-92 - Gamma counting record
6. VSL #305-92 - Detector calibration record
7. VSL #326-92 - Sample for radioactive counting logbook
8. VSL Form #19 - Daily Removal/Treatment and Chain-of-custody form

9.0 References

1) Radiation Safety Manual of the Catholic University of America; Warren Keene, RSO, Washington, DC, January 1992

2) 1991 Annual Book of ASTM Standards, Section 4, Volume 04.08, ASTM Philadelphia, PA, 1991

3) Dohrmann DC-80 Total Organic Carbon Analyzer Equipment Manual, Edition 11, July 1986, P/N 915-121

4) Installation and Operation of the 183 Boat Sampling Module, Rosemount Analytical Inc., P/N 915-240, January 1991

10.0 Appendix

List of Attachments

Attachment I - ASTM D421-85 - Standard Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants.

Attachment II - ASTM D422-63 - Standard Test Method for Particle-Size Analysis of Soils.

Attachment III - ASTM D854-83 - Standard Test Method for Specific Gravity of Soils.

OK

Standard Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[1]

4047

This standard is issued under the fixed designation D 421; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

Scope

1.1 This practice covers the dry preparation of soil samples as received from the field for particle-size analysis and the determination of the soil constants.

1.2 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of whoever uses this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

Referenced Documents

2.1 *ASTM Standards:*

D 2217 Practice for Wet Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

Significance and Use

3.1 This practice can be used to prepare samples for particle-size and plasticity tests where it is desired to determine test values on air-dried samples, or where it is known that air drying does not have an effect on test results relative to samples prepared in accordance with Practice D 2217.

Apparatus

4.1 *Balance*, sensitive to 0.1 g.

4.2 *Mortar and Rubber-Covered Pestle*, suitable for breaking up the aggregations of soil particles.

4.3 *Sieves*—A series of sieves, of square mesh woven wire cloth, conforming to Specification E 11. The sieves required are as follows:

No. 4 (4.75-mm)
No. 10 (2.00-mm)
No. 40 (425-μm)

4.4 *Sampler*—A riffle sampler or sample splitter, for quartering the samples.

Sampling

5. Expose the soil sample as received from the field to the air at room temperature until dried thoroughly. Break up the aggregations thoroughly in the mortar with a rubber-covered pestle. Select a representative sample of the amount required to perform the desired tests by the method of quartering or by the use of a sampler. The amounts of material required to perform the individual tests are as follows:

5.1.1 *Particle-Size Analysis*—For the particle-size analysis, material passing a No. 10 (2.00-mm) sieve is required in amounts equal to 115 g of sandy soils and 65 g of either silt or clay soils.

5.1.2 *Tests for Soil Constants*—For the tests for soil constants, material passing the No. 40 (425-μm) sieve is required in total amount of 220 g, allocated as follows:

Test	Grams
Liquid limit	100
Plastic limit	15
Centrifuge moisture equivalent	10
Volumetric shrinkage	30
Check tests	65

6. Preparation of Test Sample

6.1 Select that portion of the air-dried sample selected for purpose of tests and record the mass as the mass of the total test sample uncorrected for hygroscopic moisture. Separate the test sample by sieving with a No. 10 (2.00-mm) sieve. Grind that fraction retained on the No. 10 sieve in a mortar with a rubber-covered pestle until the aggregations of soil particles are broken up into the separate grains. Then separate the ground soil into two fractions by sieving with a No. 10 sieve.

6.2 Wash that fraction retained after the second sieving free of all fine material, dry, and weigh. Record this mass as the mass of coarse material. Sieve the coarse material, after being washed and dried, on the No. 4 (4.75-mm) sieve and record the mass retained on the No. 4 sieve.

7. Test Sample for Particle-Size Analysis

7.1 Thoroughly mix together the fractions passing the No. 10 (2.00-mm) sieve in both sieving operations, and by the method of quartering or the use of a sampler, select a portion weighing approximately 115 g for sandy soils and approximately 65 g for silt and clay soil for particle-size analysis.

8. Test Sample for Soil Constants

8.1 Separate the remaining portion of the material passing the No. 10 (2.00-mm) sieve into two parts by means of a No. 40 (425-μm) sieve. Discard the fraction retained on the No. 40 sieve. Use the fraction passing the No. 40 sieve for the determination of the soil constants.

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[1] This practice is under the jurisdiction of ASTM Committee D-18 on Soil and Rock, and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved July 26, 1985. Published September 1985. Originally published as D 421 – 35 T. Last previous edition D 421 – 58 (1978)[ε1].

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

OK

4047

Standard Test Method for Particle-Size Analysis of Soils[1]

This standard is issued under the fixed designation D 422; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

[1] NOTE—Section 19 was added editorially in September 1990.

1. Scope

1.1 This test method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 µm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 µm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Notes 1 and 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425-µm), or No. 200 (75-µm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (*1*) a high-speed mechanical stirrer, and (*2*) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20-µm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 µm.

2. Referenced Documents

2.1 *ASTM Standards:*

D 421 Practice for Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants[2]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[3]

E 100 Specification for ASTM Hydrometers[4]

3. Apparatus

3.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 % of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

3.2 *Stirring Apparatus*—Either apparatus A or B may be used.

3.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10 000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

3.2.2 Apparatus B shall consist of an air-jet dispersion cup[5] (Note 3) conforming to the general details shown in Fig. 3 (Notes 4 and 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft^3/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

3.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for hydrometers 151H or 152H in Specifications E 100. Dimensions of both hydrometers are the same, the scale being the only item of difference.

3.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 mL. The inside diameter shall be such that the 1000-mL mark is 36 ± 2 cm from the bottom on the inside.

3.5 *Thermometer*—A thermometer accurate to 1°F (0.5°C).

3.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of Specification E 11. A full set of sieves includes the following (Note 6):

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

Current edition approved Nov. 21, 1963. Originally published 1935. Replaces D 422 – 62.

[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103. Order Adjunct No. 12-404220-00.

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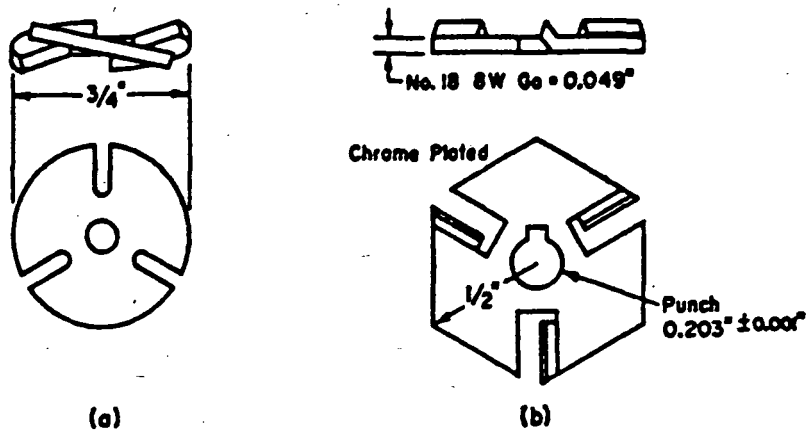


Metric Equivalents

in.	0.001	0.049	0.203	½	¾
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles

3-in. (75-mm)
2-in. (50-mm)
1½-in. (37.5-mm)
1-in. (25.0-mm)
¾-in. (19.0-mm)
⅜-in. (9.5-mm)
No. 4 (4.75-mm)
No. 10 (2.00-mm)
No. 20 (850-µm)
No. 40 (425-µm)
No. 60 (250-µm)
No. 140 (106-µm)
No. 200 (75-µm)

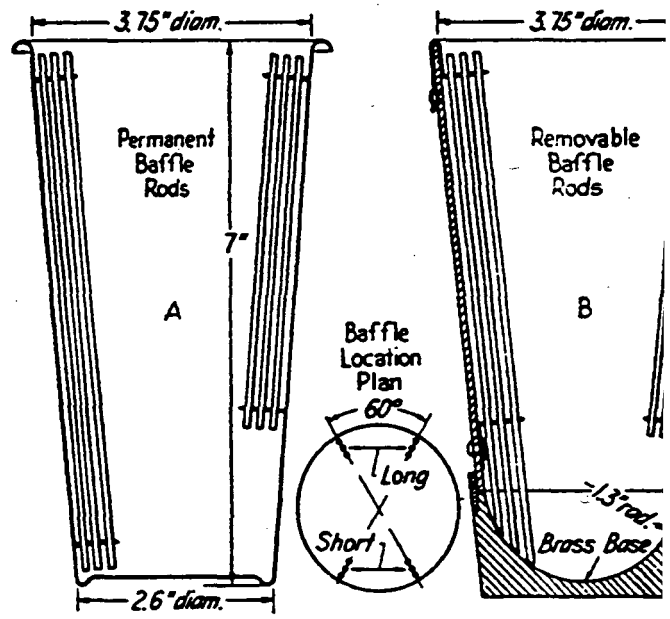
NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 17, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)
1½-in. (37.5-mm)
¾-in. (19.0-mm)
⅜-in. (9.5-mm)
No. 4 (4.75-mm)
No. 8 (2.36-mm)
No. 16 (1.18-mm)
No. 30 (600-µm)
No. 50 (300-µm)
No. 100 (150-µm)
No. 200 (75-µm)

3.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68°F (20°C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

3.8 *Beaker*—A beaker of 250-mL capacity.

3.9 *Timing Device*—A watch or clock with a second hand.



Metric Equivalents

in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus

4. Dispersing Agent

4.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

4.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to pr during the hydrometer test. For example, if the sedim tion cylinder is to be placed in the water bath, the distill demineralized water to be used shall be brought to temperature of the controlled water bath; or, if the sedi tation cylinder is used in a room with controlled tem ture, the water for the test shall be at the temperature o room. The basic temperature for the hydrometer test is (20°C). Small variations of temperature do not intro differences that are of practical significance and do prevent the use of corrections derived as prescribed.

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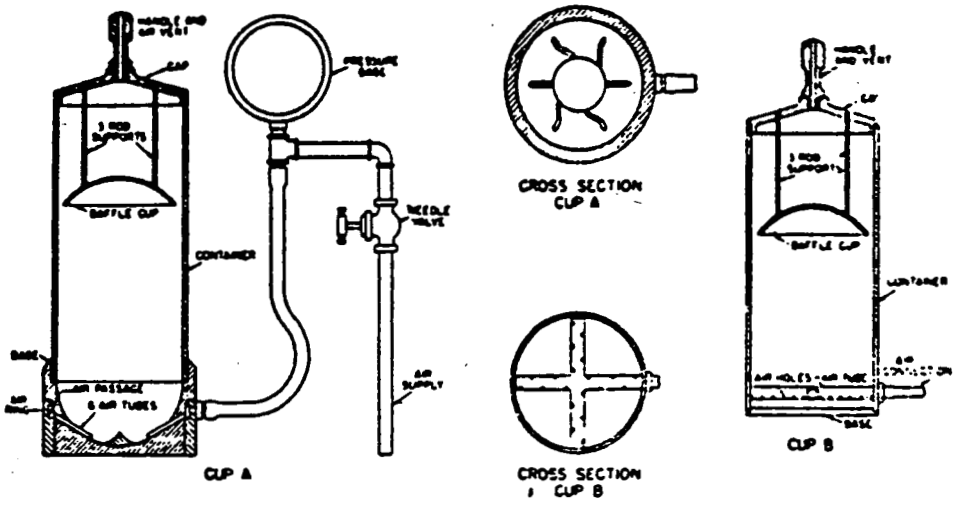


FIG. 3 Air-Jet Dispersion Cups of Apparatus B

5. Test Sample

5.1 Prepare the test sample for mechanical analysis as outlined in Practice D 421. During the preparation procedure the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Practice D 421, shall be sufficient to yield quantities for mechanical analysis as follows:

5.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
⅜ (9.5)	500
¾ (19.0)	1000
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

5.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

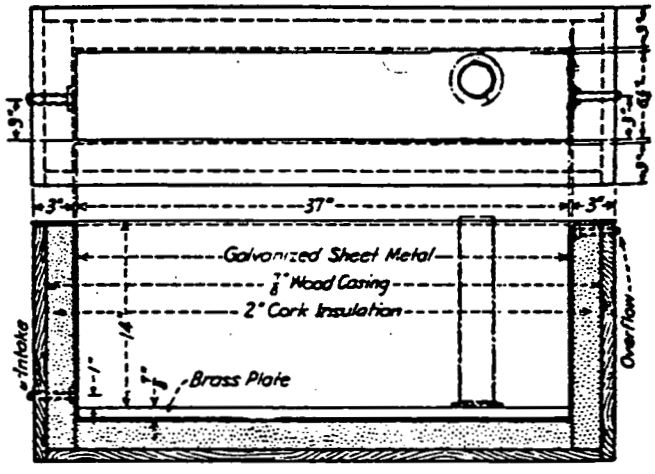
5.2 Provision is made in Section 5 of Practice D 421 for weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 12.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

Procedure

6.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in. (50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅜-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.



Metric Equivalents

in.	⅞	1	3	6¼	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath

6.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass % of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

6.3 Determine the mass of each fraction on a balance conforming to the requirements of 3.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

7. Determination of Composite Correction for Hydrometer Reading

7.1 Equations for percentages of soil remaining in suspension, as given in 14.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

7.1.1 Both soil hydrometers are calibrated at 68°F (20°C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

7.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

7.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

7.2 For convenience, a graph or table of composite corrections for a series of 1° temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

7.3 Prepare 1000 mL of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cyclinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the temperature of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one; for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

8. Hygroscopic Moisture

passing #10

8.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9°F (110 ± 5°C), and weigh again. Record the masses.

9. Dispersion of Soil Sample

9.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

9.2 Place the sample in the 250-mL beaker and cove 125 mL of sodium hexametaphosphate solution (40 Stir until the soil is thoroughly wetted. Allow to soak least 16 h.

9.3 At the end of the soaking period, disperse the sa further, using either stirring apparatus A or B. If st apparatus A is used, transfer the soil - water slurry fro beaker into the special dispersion cup shown in Fi washing any residue from the beaker into the cup distilled or demineralized water (Note 9). Add distill demineralized water, if necessary, so that the cup is than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handli water in the washing operation. Other devices include the wash bottle and a hose with nozzle connected to a pressurized distilled tank.

9.4 If stirring apparatus B (Fig. 3) is used, remov cover cap and connect the cup to a compressed air supp means of a rubber hose. A air gage must be on the between the cup and the control valve. Open the co valve so that the gage indicates 1 psi (7 kPa) pressure (10). Transfer the soil - water slurry from the beaker t air-jet dispersion cup by washing with distilled demineralized water. Add distilled or demineralized wat necessary, so that the total volume in the cup is 250 mL no more.

NOTE 10—The initial air pressure of 1 psi is required to preve soil - water mixture from entering the air-jet chamber when the mi is transferred to the dispersion cup.

9.5 Place the cover cap on the cup and open the control valve until the gage pressure is 20 psi (140 k Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispe for only 1 min. After the dispersion period, reduce the pressure to 1 psi preparatory to transfer of soil - water sl to the sedimentation cylinder.

10. Hydrometer Test

10.1 Immediately after dispersion, transfer the soil - w slurry to the glass sedimentation cylinder, and add disti or demineralized water until the total volume is 1000 m

10.2 Using the palm of the hand over the open end of cylinder (or a rubber stopper in the open end), turn cylinder upside down and back for a period of 1 min complete the agitation of the slurry (Note 11). At the en 1 min set the cylinder in a convenient location and t hydrometer readings at the following intervals of ti (measured from the beginning of sedimentation), or as m as may be needed, depending on the sample or the specifi tion for the material under test: 2, 5, 15, 30, 60, 250, a 1440 min. If the controlled water bath is used, the sedim tation cylinder should be placed in the bath between the and 5-min readings.

NOTE 11—The number of turns during this minute should approximately 60, counting the turn upside down and back as two tu

turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

10.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about 20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

10.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

11. Sieve Analysis

11.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-μm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9°F (110 ± 5°C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

12. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

12.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

12.2 To secure the total mass of soil passing the No. 4 (75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

12.3 To determine the total percentage passing for each size, divide the total mass passing (see 12.2) by the total mass of sample and multiply the result by 100.

Hygroscopic Moisture Correction Factor

13.1 The hydroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

Percentages of Soil in Suspension

14.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

14.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the air-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

TABLE 1 Values of Correction Factor, a, for Different Specific Gravities of Soil Particles[A]

Specific Gravity	Correction Factor[A]
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

[A] For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

14.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13): For hydrometer 151H:

$$P = [(100\,000/W) \times G/(G - G_1)](R - G_1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parentheses.

For hydrometer 152H:

$$P = (Ra/W) \times 100$$

where:

a = correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),

P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,

R = hydrometer reading with composite correction applied (Section 7),

W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 14.2), g,

G = specific gravity of the soil particles, and

G_1 = specific gravity of the liquid in which soil particles are suspended. Use numerical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_1.

15. Diameter of Soil Particles

15.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. According to Stokes' law:

$$D = \sqrt{[30n/980(G - G_1)] \times L/T}$$

where:

D = diameter of particle, mm,

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η = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),

L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (Table 2)),

T = interval of time from beginning of sedimentation to the taking of the reading, min,

G = specific gravity of soil particles, and

G_1 = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

15.2 For convenience in calculations the above equation may be written as follows:

$$D = K\sqrt{L/T}$$

where:

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

15.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A- and B-scales, the square root being indicated on the D-scale. Without ascertaining the value of the square root it may be multiplied by K, using either the C- or CI-scale.

16. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

16.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 14.2), and the result divided by 100.

16.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieves, including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 14.2).

16.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 12.2.

16.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 16.3) by the total mass of sample (as calculated in 14.2), and multiply the result by 100.

17. Graph

17.1 When the hydrometer analysis is performed, a graph of the test results shall be made, plotting the diameters of particles on a logarithmic scale as the abscissa and percentages smaller than the corresponding diameters

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[A]

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm	Actual Hydrometer Reading	Effective Depth, L, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

[A] Values of effective depth are calculated from the equation:

$$L = L_1 + \tfrac{1}{2}[L_2 - (V_B/A)]$$

where:

L = effective depth, cm,

L_1 = distance along the stem of the hydrometer from the top of the bulb mark for a hydrometer reading, cm,

L_2 = overall length of the hydrometer bulb, cm,

V_B = volume of hydrometer bulb, cm^3, and

A = cross-sectional area of sedimentation cylinder, cm^2

Values used in calculating the values in Table 2 are as follows:

For both hydrometers, 151H and 152H:

L_2 = 14.0 cm

V_B = 67.0 cm^3

A = 27.8 cm^2

For hydrometer 151H:

L_1 = 10.5 cm for a reading of 1.000

= 2.3 cm for a reading of 1.031

For hydrometer 152H:

L_1 = 10.5 cm for a reading of 0 g/litre

= 2.3 cm for a reading of 50 g/litre

TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis 4047

Temperature, °C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.1323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01255	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149

thmetic scale as the ordinate. When the hydrometer alysis is not made on a portion of the soil, the preparation the graph is optional, since values may be secured directly m tabulated data.

. Report

18.1 The report shall include the following:

18.1.1 Maximum size of particles.

18.1.2 Percentage passing (or retained on) each sieve, ich may be tabulated or presented by plotting on a graph ote 16),

18.1.3 Description of sand and gravel particles:

18.1.3.1 Shape—rounded or angular,

18.1.3.2 Hardness—hard and durable, soft, or weathered l friable,

8.1.4 Specific gravity, if unusually high or low,

8.1.5 Any difficulty in dispersing the fraction passing the . 10 (2.00-mm) sieve, indicating any change in type and ount of dispersing agent, and

8.1.6 The dispersion device used and the length of the persion period.

IOTE 16—This tabulation of graph represents the gradation of the ple tested. If particles larger than those contained in the sample were oved before testing, the report shall so state giving the amount and imum size.

8.2 For materials tested for compliance with definite cifications, the fractions called for in such specifications ll be reported. The fractions smaller than the No. 10 sieve ll be read from the graph.

8.3 For materials for which compliance with definite cifications is not indicated and when the soil is composed ost entirely of particles passing the No. 4 (4.75-mm) e, the results read from the graph may be reported as ows:

(1) Gravel, passing 3-in. and retained on No. 4 sieve ..... %
(2) Sand, passing No. 4 sieve and retained on No. 200 sieve ..... %
  (a) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve ..... %
  (b) Medium sand, passing No. 10 sieve and retained on No. 40 sieve ..... %
  (c) Fine sand, passing No. 40 sieve and retained on No. 200 sieve ..... %
(3) Silt size, 0.074 to 0.005 mm ..... %
(4) Clay size, smaller than 0.005 mm ..... %
  Colloids, smaller than 0.001 mm ..... %

18.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	.....
2-in.	.....
1½-in.	.....
1-in.	.....
¾-in.	.....
⅜-in.	.....
No. 4 (4.75-mm)	.....
No. 10 (2.00-mm)	.....
No. 40 (425-μm)	.....
No. 200 (75-μm)	.....

HYDROMETER ANALYSIS

0.074 mm	.....
0.005 mm	.....
0.001 mm	.....

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-μm) sieves may be substituted for No. 10 and No. 40 sieves.

19. Keywords

19.1 grain-size; hydrometer analysis; hygroscopic moisture; particle-size; sieve analysis

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American Association of Highway and Transportation Officials Standard
AASHTO No.: T 100

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Standard Test Method for Specific Gravity of Soils[1]

This standard is issued under the fixed designation D 854; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

[ε1] NOTE—Section 11 was added editorially in September 1990.

1. Scope

1.1 This test method covers determination of the specific gravity of soils by means of a pycnometer. When the soil is composed of particles larger than the No. 4 (4.75-mm) sieve, the method outlined in Test Method C 127 shall be followed. When the soil is composed of particles both larger and smaller than the No. 4 sieve, the sample shall be separated on the No. 4 sieve and the appropriate test method used on each portion. The specific gravity value for the soil shall be the weighted average of the two values (Note 1). When the specific gravity value is to be used in calculations in connection with the hydrometer portion of Method D 422, it is intended that the specific gravity test be made on that portion of the soil which passes the No. 10 (2.00-mm) sieve.

NOTE 1—The weighted average specific gravity should be calculated using the following equation:

$$G_{avg} = \frac{1}{\frac{R_1}{100G_1} + \frac{P_1}{100G_2}}$$

where:

G_{avg} = weighted average specific gravity of soils composed of particles larger and smaller than the No. 4 (4.75-mm) sieve,
R_1 = percent of soil particles retained on the No. 4 sieve,
P_1 = percent of soil particles passing the No. 4 sieve,
G_1 = apparent specific gravity of soil particles retained on the No. 4 sieve as determined by Test Method C 127, and
G_2 = specific gravity of soil particles passing the No. 4 sieve as determined by this test method.

1.2 The values stated in acceptable metric units are to be regarded as the standard.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 127 Test Method for Specific Gravity and Absorption of Coarse Aggregate[2]
C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials[3]
D 422 Method for Particle-Size Analysis of Soils[3]
E 12 Definitions of Terms Relating to Density and Specific Gravity of Solids, Liquids, and Gases[4]

3. Definition

3.1 *specific gravity*—the ratio of the mass of a unit volume of a material at a stated temperature to the mass in air of the same volume of gas-free distilled water at a stated temperature (per Definitions E 12).

4. Significance and Use

4.1 The specific gravity of a soil is used in almost every equation expressing the phase relationship of air, water, and solids in a given volume of material.

4.2 The term "solid particles," as used in geotechnical engineering, is typically assumed to mean naturally occurring mineral particles that are not very soluble in water. Therefore, the specific gravity of materials containing extraneous matter (such as cement, lime, etc.), water-soluble matter (such as sodium chloride), and soils containing matter with a specific gravity of less than one, typically require special treatment or a qualified definition of specific gravity.

5. Apparatus

5.1 *Pycnometer*—Either a volumetric flask having a capacity of at least 100 mL or a stoppered bottle having a capacity of at least 50 mL (Note 2). The stopper shall be of the same material as the bottle, and of such size and shape that it can be easily inserted to a fixed depth in the neck of the bottle, and shall have a small hole through its center to permit the emission of air and surplus water.

NOTE 2—The use of either the volumetric flask or the stoppered bottle is a matter of individual preference, but in general, the flask should be used when a larger sample than can be used in the stoppered bottle is needed due to maximum grain size of the sample.

5.2 *Balance*—Either a balance sensitive to 0.01 g for use with the volumetric flask, or a balance sensitive to 0.001 g for use with the stoppered bottle.

[1] This test method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock and is the direct responsibility of Subcommittee D18.03 on Texture, Plasticity, and Density Characteristics of Soils.

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[2] *Annual Book of ASTM Standards*, Vol 04.02.
[3] *Annual Book of ASTM Standards*, Vol 04.08.
[4] *Annual Book of ASTM Standards*, Vol 15.05.

Calibration of Pycnometer

6.1 The pycnometer shall be cleaned, dried, weighed, and the weight recorded. The pycnometer shall be filled with distilled water (Note 3) essentially at room temperature. The weight of the pycnometer and water, W_a, shall be determined and recorded. A thermometer shall be inserted in the water and its temperature T_i determined to the nearest whole degree.

NOTE 3—Kerosine is a better wetting agent than water for most soils and may be used in place of distilled water for oven-dried samples.

6.2 From the weight W_a determined at the observed temperature T_i a table of values of weights W_a shall be prepared for a series of temperatures that are likely to prevail when weights W_b are determined later (Note 4). These values of W_a shall be calculated as follows:

$$W_a' \text{ (at } T_x) = (\text{density of water at } T_x/\text{density of water at } T_i) \times (W_a \text{ (at } T_i) - W_f) + W_f$$

where:

W_a' = weight of pycnometer and water, g,
W_f' = weight of pycnometer, g,
T_i = observed temperature of water, °C, and
T_x = any other desired temperature, °C.

NOTE 4—This method provides a procedure that is most convenient for laboratories making many determinations with the same pycnometer. It is equally applicable to a single determination. Bringing the pycnometer and contents to some designated temperature when weights W_a and W_b are taken, requires considerable time. It is much more convenient to prepare a table of weights W_a for various temperatures likely to prevail when weights W_b are taken. It is important that weights W_a and W_b be based on water at the same temperature. Values for the relative density of water at temperatures from 18 to 30°C are given in Table 1.

7. Sampling

7.1 The soil to be used in specific gravity test may contain its natural moisture or be oven-dried. The weight of the test sample on an oven-dry basis shall be at least 25 g when the volumetric flask is to be used, and at least 10 g when the stoppered bottle is to be used.

7.2 *Samples Containing Natural Moisture*—When the sample contains its natural moisture, the weight of the soil, W_o, on an oven-dry basis shall be determined at the end of the test by evaporating the water in an oven maintained at 230 ±9°F (110 ±5°C) (Note 5). Samples of clay soils containing their natural moisture content shall be dispersed in distilled water before placing in the flask, using the dispersing equipment specified in Method D 422 (Note 6).

7.3 *Oven-Dried Samples*—When an oven-dried sample is to be used, the sample shall be dried for at least 12 h, or to constant weight, in an oven maintained at 230 ±9°F (110 ±5°C) (Note 5), cooled in a desiccator, and weighed upon removal from the desiccator. The sample shall then be soaked in distilled water for at least 12 h.

NOTE 5—Drying of certain soils at 110°C may bring about loss of moisture of composition or hydration, and in such cases drying shall be done, if desired, in reduced air pressure and at a lower temperature.

NOTE 6—The minimum volume of slurry that can be prepared by the dispersing equipment specified in Method D 422 is such that a 500-mL flask is needed as the pycnometer.

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8. Procedure

8.1 Place the sample in the pycnometer, taking care not to lose any of the soil in case the weight of the sample has been determined. Add distilled water to fill the volumetric flask about three-fourths full or the stoppered bottle about half full.

8.2 Remove entrapped air by either of the following methods: (*1*) subject the contents to a partial vacuum (air pressure not exceeding 100 mm Hg) or (2) boil gently for at least 10 min while occasionally rolling the pycnometer to assist in the removal of the air. Subject the contents to reduced air pressure either by connecting the pycnometer directly to an aspirator or vacuum pump, or by use of a bell jar. Some soils boil violently when subjected to reduced air pressure. It will be necessary in those cases to reduce the air pressure at a slower rate or to use a larger flask. Cool samples that are heated to room temperature.

8.3 Fill the pycnometer with distilled water, clean the outside and dry with a clean, dry cloth. Determine the weight of the pycnometer and contents, W_b, and the temperature in degrees Celsius, T_x, of the contents as described in Section 6.

9. Calculation and Report

9.1 Calculate the specific gravity of the soil, based on water at a temperature T_x, as follows:

$$\text{Specific gravity, } G \text{ at } T_x = W_o/[W_o + (W_a' - W_b')]$$

where:

W_o = weight of sample of oven-dry soil, g,
W_a = weight of pycnometer filled with water at temperature T_x (Note 7), g,
W_b = weight of pycnometer filled with water and soil at temperature T_x, g, and
T_x = temperature of the contents of the pycnometer when weight W_b was determined, °C.

NOTE 7—This value shall be taken from the table of values of W_a' prepared in accordance with 6.2, for the temperature prevailing when weight W_b was taken.

9.2 Unless otherwise required, specific gravity values reported shall be based on water at 20°C. The value based on water at 20°C shall be calculated from the value based on water at the observed temperature T_x, as follows:

$$\text{Specific gravity, } G \text{ at } 20°\text{C} = (K) \times (\text{specific gravity, } G \text{ at } T_x)$$

where:

TABLE 1 Relative Density of Water and Conversion Factor K For Various Temperatures

Temperature, °C	Relative Density of Water	Correction Factor K
18	0.9986244	1.0004
19	0.9984347	1.0002
20	0.9982343	1.0000
21	0.9980233	0.9998
22	0.9978019	0.9996
23	0.9975702	0.9993
24	0.9973286	0.9991
25	0.9970770	0.9989
26	0.9968156	0.9986
27	0.9965451	0.9983
28	0.9962652	0.9980
29	0.9959761	0.9977
30	0.9956780	0.9974

K = a number found by dividing the relative density of water at temperature T_x by the relative density of water at 20°C. Values for a range of temperatures are given in Table 1.

9.3 When it is desired to report the specific gravity value based on water at 4°C, such a specific gravity value may be alculated by multiplying the specific gravity value at temperature T_x by the relative density of water at temperature T_x.

9.4 When any portion of the original sample of soil is eliminated in the preparation of the test sample, the portion on which the test has been made shall be reported.

10. Precision and Bias

10.1 Criteria for judging the acceptability of specific gravity test results obtained by this test method on material passing the No. 4 (4.75-mm) sieve are given as follows (Note 8):

Material and Type Index	Standard Deviation[A]	Acceptable Range of Two Results (percent of mean)[A]
Single-operator precision:		
Cohesive soils	0.021	0.06
Noncohesive soils	[B]	[B]
Multilaboratory precision:		
Cohesive soils	0.056	0.16
Noncohesive soils	[B]	[B]

[A] These numbers represent, respectively, the (1S) and (D2S) limits as described in Practice C 670.

[B] Criteria for assigning standard deviation values for non-cohesive soils are not available at the present time.

10.2 *Bias*—There is no accepted reference value for this test method, therefore bias cannot be determined.

NOTE 8—The figures given in Column 2 are the standard deviations that have been found to be appropriate for the materials described in Column 1. The figures given in Column 3 are the limits that should not be exceeded by the difference between the results of two properly conducted tests.

11. Keywords

11.1 specific gravity

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This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, PA 19103.

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PROCEDURE FOR PCT TESTS

PRODUCT CONSISTENCY TEST (PCT)

1.0 SCOPE

This test procedure evaluates the relative chemical durability of both homogeneous and devitrified glasses by measuring the concentrations of the chemical species released from crushed glass to the test solution at 90°C.

This test procedure can specifically be used to evaluate whether the durability of glasses has been consistently controlled during production.

This test procedure can specifically be used to evaluate whether the radionuclide release properties of nuclear waste glasses have been consistently controlled during production.

2.0 APPLICABILITY

This procedure applies to all employees trained and authorized to perform this test in the VSL.

This test method provides data useful for (1) evaluating the chemical durability of glasses, (2) evaluating the radionuclide release properties of nuclear waste glasses, and (3) evaluating whether the glass durability or radionuclide release properties have been consistently controlled during long-term production. Accordingly, its applicability is widespread throughout manufacturing, research, and development. It can be used for specification acceptance.

This test method may be applicable to the evaluation of the chemical durability of glasses and the radionuclide release properties of nuclear waste glasses in natural environments. [7]

3.0 SAFETY

This test method must be performed in accordance with all applicable quality assurance requirements of the laboratory performing the test and using the data.

This procedure may involve hazardous materials, operations,

and equipment. All personnel who are authorized to perform this procedure must consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

3.1 Hazard Identification

All samples received at CUA-VSL and handled at VSL laboratories are treated as potentially hazardous. At VSL one encounters both chemical and radioactive hazardous materials.

The "hazardous chemicals", referred to in this procedure, are defined in Section 8.3 of the CUA Chemicals Materials Safety Manual (CCMSM). A copy of CCMSM is available in every VSL laboratory. Procedures for identifications and handling of hazardous materials as applied at VSL are prescribed in sections 8.22, 8.23, 8.24 and 8.25 of CCMSM and in sections 8.9, 8.10, 8.11, 8.12, 8.13 and 8.14 of the CCMSM.

A major element of hazard identification is the proper use of labels. At VSL, labeling of chemicals and other hazardous substances is done in accordance with Section 8.4 of the CCMSM, as well as in accordance with Materials Safety Data Sheets (MSDSs) provided by the manufacturers of the chemicals and other hazardous materials. Every VSL laboratory maintains the "work place chemical list". The list is made available to employees in accordance with Section 8.31 of CCMSM.

3.2 Handling and Storage of Chemicals

Handling of chemicals should be conducted according to Section 8.6 of the CCMSM. Storage of chemicals should follow section 8.8 of the CCMSM.

Special precautions should be taken in handling and storage of incompatible chemicals, solvents, compressed gases and radioactive materials in accordance with Sections 8.10, 8.12, 8.13, and 8.14 of the CCMSM respectively.

3.3 Emergencies

The following are considered laboratory emergency cases:

-chemical spills, to be dealt with according to section 8.17 of the CCMSM.

- fire and fire-related accidents, to be controlled according to section 8.21 of the CCMSM.

- personal contamination, to be treated according to section 8.19 of the CCMSM.

- injury, to be treated in accordance with section 8.18 of CCMSM.

Cases requiring minor first aid should be treated in accordance with section 8.20.

3.4 Radioactive Hazard Mitigation

Control and monitoring of personnel exposure and environmental radiation at VSL are performed in accordance with Chapter 3 of the Radiation Safety Manual (RSM) of the Catholic University of America. Application of the rules prescribed by RSM is the responsibility of each user working in the restricted area (rooms 37 to 40 of Hannan Hall) and the project managers.

Dr. Warren E. Keene, CUA - Radiation Safety Officer (CUA-RSO) and Dr. Isabelle Muller, VSL Radiation Safety Officer (VSL-RSO) are responsible for ensuring that the work is done safely and in a manner consistent with applicable regulations.

3.5 Safety Applicable to PCT Procedure

All appropriate precautions for operation of pressurized equipment must be taken. To ensure safe operation, the leach containers should be designed to withstand the vapor pressure of water at the test temperature with an appropriate safety factor. The thermal expansion of water must be taken into account when filling the leach containers. Between filling at 4°C and testing at 100°C water expands by 4 volume percent. Overfilling may lead to pressures inside the container that exceed the design limits, and could lead to the failure of one or more parts of the vessel.

4.0 Requirements

4.1 Training

Part of the sample preparation for PCT tests is done in the radioactive restricted area. All restricted area workers shall be trained both by the laboratory supervisor regarding their specific duties and by the radiation safety officer of campus. Each restricted area worker must attend a course in radiation safety.

Training on PCT procedure and equipment used in the PCT laboratory is given by the laboratory manager and/or by a trained and experienced person designated by the laboratory manager until the technician shows competence to use the method independently of direct supervision.

4.2 Definitions

leachant - The solution that is being used, or is intended for use in leaching.[3]
leachate - The solution resulting from a leach test[3]
ASTM Type I water - Purified water with a maximum total matter content of 0.1 mg/L, and a minimum electrical resistivity of 16.67 MΩ*cm at 25°C, and no detectable soluble silica (consult ASTM D 1193). [1]
open system tests - A system is open if, during the process under consideration, transfer of matter either into or out of the system takes place, e.g. O_2 and/or CO_2 diffusion into or out of the leach vessel. [6]
closed system tests - A system is closed if during the process under consideration no transfer of matter either into or out of the system takes place. [6]
set of samples - Those which have been tested simultaneously in the same oven.
sample blank - A cleaned test vessel which has been filled with the same amount of leachant as the sample vessels but contains no glass sample

5.0 Equipment and Supplies

5.1 Description of Equipment and Supplies

Qty	Device	Manufacturer	Model	Calib. Req.
1	Balance	Sartorius	LC 6200S	I
9	Oven	Blue-M	Stabil-Therm OV-490A,OV-490A-3	II
1	pH meter	Corning	245	III
1	Graduated Cylinder			IV

2	Pipettes (1.0 and 5-10 ml)	Oxford/Eppendorf		V
	Thermometers	Various	-20 to 150 °C, ± 1 °C	VI
	Syringes with SS needles			N.A.
	Stainless steel sections	Whitey	#304L-HDF4-150 #SS-4-P #SS-4-VCO-4 #SS-4-VCO-1-4	N.A.
		Cajon	#SS-4-VCO-3-47A	
		Swagelok	#SS-400-C	
	Three-layer rubber septa	Hamilton	76006	N.A.
	Syringe filter	Gelman	4497	N.A.
	Racks for holding stainless steel containers			N.A.

I: Calibration accuracy: ±100 mg
Calibration frequency: monthly

II: Temperature monitored daily with thermometers that are positioned as recommended by the manufacturer and meet NIST Specification, Monograph 150.

III: Calibration accuracy: +/-0.05 pH units
Calibration frequency: daily or every 25 measurements.
See VSL-LT

IV: Calibrated before first use.

V: Calibration accuracy: ± 5%
Calibration frequency: monthly

VI: Meeting NIST Specification, Monograph 150

Leach Vessels - 304L stainless steel leach vessels of 150 mL capacity designed to take internal pressures of at least 75 psi without leaking. The stainless steel vessels require a rubber disk

septa in order to remain sealed.

Mechanical Convection Oven - A laboratory oven suitable for operation up to 260°C and capable of maintaining ± 2.0°C throughout the entire interior of the oven at the test temperature.

Temperature Measurement - Thermistor probe and/or thermocouple with a facility for continuous monitoring of the temperature of the convection oven during the test duration.

Balance - a balance which will provide the following sensitivity: 0.25% of the largest and smallest masses to be measured including the mass of the reagents, sample, leachant, leachate, leach vessel, and any required combinations.

Weight Calibration Set - NIST Class S-type weight calibration set ranging between 100 mg - 1 kg, or equivalents, traceable to NIST.

Crushing Device - Any mechanical or manual crushing device which will avoid steel contamination in the crushed glass specimen. Grinding devices made of tungsten carbide, agate, sapphire, or dense alumina are acceptable.

Sieves - A nest of 3 inch, 5 inch, or 8 inch U. S. Standard ASTM stainless steel or brass sieves. The nest shall include the covers and pan, including the No. 100 (149 μm) and No. 200 (74 μm) sieves.

Flasks - Class A or calibrated volumetric labware.

Pipettes - Calibrated and precleaned pipette tips.

Syringes and Syringe Filters - Precleaned syringes and 0.45 μm syringe filters (for example Gelman #4497).

Sample Vials - Presterilized sample vials and caps.

pH Meter - pH meter with an accuracy of ± 0.2 pH units.

Milli-Q Water Purification System - Water purification system for producing ASTM-Type I water.

Ultrasonic Cleaner - Size to be compatible with cleaning of several leach vessels simultaneously.

High Pressure Air or Freon Pressurized Canister - To dry

sieves after washing; a drying oven is an acceptable alternative.

5.2 Calibrations

Note: See VSL-LT for detailed calibration procedures.

Calibrations - Initially calibrate all instruments used in this test. Perform periodic calibrations during use of the instrument to minimize possible errors due to instrumental drift.

Calibration and Standardization Schedule:

Thermistor or thermocouple - Calibrate annually with NIST Standards or ice/boiling water.

Balance - Have the balance calibrated on a monthly basis.
pH meter - Initially and after completion of a set of measurements with commercial buffer solutions that bracket the solution pH being measured.

5.3 Standards

Reference Glass - A reference glass of choice, similar in composition to the glass being tested, is to be run in triplicate along with each batch of glasses tested.

Multi-element Solution Standard - A reference solution of choice, containing the major elements of the composition of the leachate being tested, is to be submitted along with each batch of leachates for multi-element analysis. The reference solution standard should be traceable to NIST, or a comparable equivalent used, and should have a certified shelf life.

pH Buffers - commercial pH buffers to bracket the measured pH range of the leachant and leachate.

5.4 Reagents and Standards

Reagent Grade Acids - Reagent grade HNO_3 for cleaning of leach vessels.

High Purity Acid - High purity concentrated HNO_3 for acidification of leachates.

Solvents - Ethyl Alcohol 95% pure and reagent grade acetone.

ASTM Type I Water - Type I water shall have a minimal electrical resistivity of 16.67 M Ohm cm at 25°C (consult ASTM D1193).

The source water shall be passed through Milli-RO15 System which contains 6 cartridges (Ful-Flow, Teel, Lifegard, Rogard Pre filter and Milli-Ro-20 Spiral-Wound Reverse Osmosis) and Milli-Q System that consists of five cartridges (Lifegard, Super-C, two of Ion-Exchanger, and Millipore DI-12). Pass the purified water through an in-line conductivity cell to verity its purity.

5.5 Choice and Cleaning of Leach Vessel

Stainless Steel Vessels - Stainless steel vessels made of 304L are to be used in all tests.

Preparation of Vessels:

Identification of Vessels - A unique identifying number should be permanently etched on each leach vessel.

Identification of Vessel Cleaning History - Each batch of cleaned leach vessels will be labeled with a unique batch number. A log book of the leach vessel number and date the cleaning is completed shall be kept.

Cleaning of New Stainless Steel Vessels - New 304L stainless steel vessels shall be cleaned by the following procedure:

(a) Clean the vessels and lids (without the gaskets) ultrasonically in reagent grade acetone for 5 minutes.

(b) Clean the vessels and lids ultrasonically in 95% ethanol for 5 minutes.

(c) Rinse the vessels and lids 3 times with ASTM Type I water.

(d) Rinse the vessels and lids in 0.16M HNO_3 (1 wt% HNO_3) and heat on a hotplate 90°C for 1 hour.

(e) Rinse the vessels 3 times with ASTM Type I water.

(f) Submerge the vessels and lids in fresh ASTM Type I water for

1 hour.

(g) Rinse with fresh ASTM Type I water.

(h) Fill the vessel 80-90% full of ASTM Type I water. Close the lid and leave in a 90°C oven for a minimum of 16 hours.

(i) Remove the vessels from the oven, take an aliquot of the wter and measure the pH (consult ASTM D1293).

(j) If the pH is not in the range 5.0-7.0, repeat steps (f) through (i).

(k) If the 5.0-7.0 pH range cannot be achieved by 3 repetitions of steps (f) through (i) then repeat the cleaning and testing procedure starting at Step (d)

(l) Dry vessels and lids at 90°C for a minimum of 16 hours, and store inside a clean container until needed.

Cleaning of rubber septa:

(a) Handle the rubber septa only with clean tongs.

(b) Clean each rubber septa ultrasonically in 95% ethanol in 1 minute.

(c) Clean each rubber septa under flowing ASTM Type I water for 3 minutes.

Cleaning of Used Stainless Vessels - Used stainless steel containers shall be cleaned according to the following procedure:

(a) Remove as much as possible of the glass inside the container to a 20 ml vial, add leachate solution, then label the vial. Flush the container using flowing tap-water for 2 minutes, brush the inside using plastic brush for 2 minutes, and rinse in tap-water.

(b) Fill each container with 120 ml 30 wt% NaOH, seal, then place in a 150 ^{0}C oven for at least 16 hours (overnight is recommended).

(c) Remove one of the lids from the sealed container, empty, and

flush the container with flowing tap water for 2 minutes (both inside and outside the container). Remove the other lid from the container.

(d) Place containers in a large plastic bottle, fill with tap water and place the open bottle in a 90 ^{0}C oven for one hour.

(e) Drain containers and check the inside of each container for the remaining glass using a pen flashlight. If glass is found, repeat steps (a) through (d). Otherwise, proceed to next step.

(f) Place the containers in a large plastic bottle filled with 1.5 wt% HNO_3(take 85.7 ml 70% HNO_3 and dilute to 4000 ml) and place the open bottle in 90 ^{0}C oven for 16 hours.

(g) Drain the containers and rinse them with DI water 3 times. Place the containers in 1 wt% HNO3 (take 57.1 ml 70% HNO3 and dilute to 4000 ml) at 90 ^{0}C for 1 hour.

(h) Rinse each container with DI water 3 times and dry for one hour at 90 ^{0}C oven.

(i) Rinse with DI water 3 times.

(j) Reassemble containers and fill about 90% full with DI water and place them into oven overnight.

(k) Measure the solution pH. If the pH is in the range of 5 to 7, send 20 ml for analysis of Si and Fe.

(l) If the pH is out of above range, repeat steps (b) to (g).

If the Si concentration is more than 3 ppm, repeat steps (b) to (g).

(m) Dry vessels, lids, and gaskets at 90°C for a minimum of 16 hours and store inside a clean container until needed.

6.0 Test Procedure

6.1 Sample Preparation:

(a) **Sample Handling** - All glass must be handled with clean equipment and stored in clean container.

(b) **Choice of Appropriate Sample** - Samples of monolithic glass may either be fabricated individually or taken from large samples of glass. Choose representative monolithic sample free of surface contamination. If contamination is suspected flush the sample surface with ASTM Type I water and dry before crushing.

(c) **Choice of Sample Mass** - The reference ratio of leachant volume to sample mass (V_{soln}/m_{solid}) is 10 ± 0.1 mL/g. The volume of leachant is constrained by the volume of the leach vessel chosen. For example, a maximum volume of 100 mL and a sample mass of 10 g is recommended when using 150 mL vessels. Smaller amounts may be used, e.g. 4 g in 40 mL of leachant contained in a 40 mL vessel. Less than 1 g of specimen is not recommended.

(d) **Number of Sample Replicates** - A minimum of three replicate samples shall be used to provide estimates of experimental variability.

(e) **Crushing and Sieving Glass** - If the sample has dimensions larger than ¾", wrap the sample in a clean plastic bag and break it into smaller fragments with a hammer. For radioactive glass it may be necessary to use a steel mortar and pestle but caution must be exercised not to transfer steel particulates to the glass due to the known interactions of steel and glass in solution.

(f) Transfer glass fragments into a clean manual or mechanical grinder of choice. If a mechanical grinder such as a Tekmar® Grinding Mill is used with a tungsten carbide blade, grind in short bursts of approximately 5 seconds due to the brittle nature of the tungsten carbide blade. Do not use mechanical grinders with stainless steel blades unless they are 304L or 316L stainless steel due to the known interactions of lower carbon containing steels and glass in solution.

(g) Clean brass or stainless steel sieves, catch pan and lid before and after every use by flushing them with deionized water from all directions. Dry immediately with high pressure air, or in an oven. Do not use solvents and/or high temperatures as the mesh has a protective film to inhibit corrosion.

(h) Visually inspect the sieves for holes or tears before every use. Transfer crushed glass to the clean nest of sieves placing the fragments on a coarse sieve, e.g. 40 mesh in order to protect the finer sieves of 100 mesh (149 micron) below. The 200 mesh (74 micron) sieve should be under the 100 mesh sieve with a catch pan below.

(i) Place the lid on the nest of sieves and sieve for at least 5 minutes.

(j) Remove the 100 mesh sieve containing +100 mesh glass fraction. Then remove the 200 mesh sieve containing the -100 to +200 mesh fraction.

(k) Tap the 200 mesh sieve lightly over colored paper. For example if the glass is light colored paper, tap the sieve on dark paper. For dark glass, e.g. black nuclear waste glass, tap the sieve on white paper. If a significant amount of powder appears on paper, repeat steps (d) through (f) until no glass passes through the 200 mesh sieve.

(l) Transfer the -100 to +200 fraction of the sieved glass into a clean container labeled with the sample identification. The date and the name of the person preparing the sample should also appear on the container.

(m) If additional material is needed, recrush the +100 mesh size fragments or repeat step (a). When new glass fragments have been prepared repeat steps (c) through (g)

Enter sample identification, date, and name of the person preparing the sample on a sample log book.

6.2 Washing the 100-200 Mesh Glass:

(a) Place approximately 35-40 grams of sieved glass in a clean 200 mL glass beaker. Do not exceed the 20 mL mark on the beaker.

(b) Forcibly add 25-30 mL of ASTM Type I water from a squirt bottle to the glass. During this addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass.

(c) Allow the glass-water mixture to settle 15 seconds then decant off the water.

(d) Repeat steps (b) to (c).

(e) Repeat step (b).

(f) Prepare the ultrasonic cleaner by filling with water to

0.5". Place the beaker from step (e) in the ultrasonic cleaner for 2 minutes. After removing the beaker from the cleaner, decant the water from the beaker and discard.

(g) Repeat (f).

(h) Forcibly add 25-30 mL of absolute alcohol from a squirt bottle to the glass. During this addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass.

(i) Place the beaker from step (h) in the ultrasonic cleaner for 2 minutes. After the 2 minutes decant the alcohol from the beaker.

(j) Repeat step (i) two more times.

(k) Put the beaker full of cleaned glass in a 90°C oven overnight to dry. Glasses can be stored in a clean, sealed and labeled container until they are to be used. Before use the crushed glass must be redried at 90°C overnight so that the powders do not contain adsorbed water when weighed.

(l) Enter sample identification, date, and name of the person performing the sample washing on a sample washing log book.

6.3 Preparation of Standard Glass

(a) **Standard Glass Handling** - same as for sample glass.

(b) **Standard Glass Sample Size** - same as for sample glass.

(c) **Number of Standard Glass Replicates** - same as for sample glass.

(d) **Crushing, Sieving, and Washing of Standard Glass** - same as for sample glass.

6.4 Procedure

6.4.1 **Number of Sample Replicates** - All tests for each glass should be carried out at least in triplicate.

6.4.2 **Number of Standard Glass Replicates** - A standard glass test shall be run at least in triplicate as part of each "set" of samples.

6.4.2.1 A "set" of samples is considered to be those which are tested simultaneously in the same oven.

6.4.3 **Number of Vessel Blanks** - A blank is considered to be a cleaned test vessel which has been filled with the same amount of ASTM Type I water as the sample vessels but contains no glass. For each "set" of samples, two blanks from the same batch of cleaned vessels shall be used. If more than one batch of cleaned vessels is used in a "set" of samples, then two blanks from each batch of vessels will be used. Three blanks are recommended for tests in ground water and other leachants which have high concentrations of glass components.

6.4.4 **Leaching Procedure** - Each sample, standard, and blank shall be tested according to the following procedure:

6.4.4.1 Calibrate the pH meter. Collect a sufficient amount of fresh ASTM Type I water from the Milli-Q Water Purification System to fill all the leach vessels in the "set" of samples, including standards, and blanks being tested. Ensure that the Type I water meets the minimum electrical resistivity of 16.67 M Ω cm at 25°C. Record the resistivity of the water collected on each sample, standard, and blank. Determine the pH of an aliquot of ASTM Type I water collected. Use the water collected within 3-6 hours as slow absorption of gaseous species from the air can cause the initial conductivity and pH of type I water to slowly drift with time (consult ASTM D1125). Record the initial measured pH, the temperature at which the pH was measured, the pH values of the calibration solutions used, and the identification number of the pH meter used. Discard the aliquot of ASTM Type I water used for the pH measurement.

6.4.4.2 Check the balance calibration. Weigh the empty vessel with the lid. Record the vessel number and the initial weight.

6.4.4.3 Place the desired amount of prepared glass in the clean leach vessel. Replace the lid and reweigh the vessel, lid and sample. Record this composite weight. The difference between the two vessel weighings should be recorded as the weight of the glass.

6.4.4.4 Add ASTM Type I water equivalent to 10 times the mass of glass added as calculated in 6.4.4.3 so that $(V_{soln}/m_{solid}) = 10 \pm 0.1$ mL/g. Swirl to wet the glass. Close and seal the leach container with a wrench and reweigh. Record the total weight. For blanks add the same amount of water but no sample.

6.4.4.5 Shake each container thoroughly and place sample vessels horizontally into the preheated 90°C oven. The 7 day recommended test period starts at this time. Record that date and time (d:h:min).

6.4.4.6 Leave sample vessels in oven at test temperature between 16-24 hours before testing the tightness of the lids. Quickly retighten loose lids while the vessels are hot, weigh and record the weights again, and return immediately to oven for the remainder of the test duration. If the weight loss of a vessel is more than 1% of the total weight of its leachant, ASTM Type I water can be added to the vessel then retighten the vessel to continue the test. The sample should be discarded if the weight loss is more than 10% of its total leachant.

6.4.4.7 The testing period shall be controlled to within plus or minus 1% of the total time period of the test.

(a) One day before sampling stand the vessels vertically with the rubber septum at the top in a rack in the oven.

(b) On the day of sampling remove the loaded stainless steel containers from the oven. Try not to shake the containers. Weigh the container and record the weight.

(c) Unscrew the top using a wrench.

(d) While the vessel is still close to the test temperature remove about 4 ml of leachate from each container using a polypropylene syringe with a stainless steel needle which is inserted through the rubber septum. Detach the stainless steel needle from the syringe and attach the 0.45 μm syringe filter to the syringe. Filter the leachate through the filter into a clean vial. The sampling should be performed as soon as the vessel is removed from 90°C oven in order to obtain leachate concentrations which closely represent the 90°C equilibrium under the test conditions; this is very important for future data modelling.

6.4.4.8 Pipette 1.0 ml of the sampled leachate to a vial prefiled with 20.0 ml 1% HNO_3 solution which made from high-purity nitric acid and ASTM Type I water and label appropriately for analysis.

6.4.4.9 Pour the remaining 3 ml of the sampled leachate into a 4 ml plastic cup placed in a water bath at room temperature and measure pH. (It is very difficult to determine a reliable 90°C pH value. The leachate is, therefore, quenched to room temperature to

a obtain stable pH reading, which can be easily translated into 90°C pH value with the help of the geochemical code PHREEQE). Record pH measurement and temperature.

6.4.4.10 Inject enough fresh ASTM Type I water into the container using another clean polypropylene syringe with a stainless steel needle which is inserted through the rubber septum to recover the original weight of the vessel before sampling.

6.4.4.11 When sampling is finished, replace and tighten the stainless steel cap with a wrench. Weigh the container again and record the weight.

6.4.4.12 Return the containers to the oven and position them horizontally to continue the test.

6.4.4.13 Take 1 mL of the leachate used in the pH measurement and add 20 mL ASTM Type I water. Label this as back-up solution, and retain it until the primary solution is analyzed.

6.4.4.14 Measure cation and anion concentrations of glass leachates, standard glass leachates, blanks, and multielement solution standards using equipment calibrated with standards traceable to NIST if suitable standards exist. Report the precision and accuracy of the analytic techniques used. Although results should normally be accurate to within ± 10%, this will not be possible when concentrations in the solution approach detection limits. The detection limits for each analysis should preferably accompany the reported results.

6.4.4.15 For radioactive glasses, submit appropriately acidified aliquots for all desired radiochemical analyses.

6.4.4.16 Analysis of the solids on the filter or the remaining solid glass sample is optional. If solids analysis are not desired the filter and solid glass sample may be discarded. If solids analysis is desired, record the appearance of the specimen powder, e.g., visible changes in color, agglomeration, and gelatinization. Wash the specimen powder from the leach container with pure water onto a clean watch glass and dry at 90°C. A temperature of 90°C will only drive off adsorbed moisture and not water of hydration. After drying, store in a clean container or analyze.

7.0 Specimen Control/Traceability

Samples are received from the Glass Preparation Laboratory by logging out the samples on the glass log form maintained in the Glass Preparation Laboratory. The receiver signs, dates, and enters in the glass log form the PCT lab notebook number and page number where the sample receipt will be recorded. The traceability with the Glass Preparation Laboratory is thus maintained. VSL procedure TPI-SH deals with the sample traceability forms and other formats for recording the data.

8.0 Calculation and Reporting

8.1 **Use of Multielement Standard** - Calculate the average and standard deviation of the analytical results of the multicomponent standard. If the average values agree within 10% of the standard values then the analysis is considered acceptable.

8.2 **Use of Blanks** - Blanks are used to check if sources of contamination are present due to incorrect vessel cleaning or contamination. Corrections of up to 1% of any contaminant element is subtracted from the leachate concentrations for all samples using the data from the replicate blanks from the same batch of cleaned vessels. If corrections of >1% of any contaminant element occur, all the tests performed using vessels from that vessel cleaning batch must be repeated.

8.3 **Calculations of Leachate Concentrations** - Leachate concentrations, especially those for the major soluble elements in the glass (Li, B, Na, K, and Si) are calculated for the test glasses and the standard glass.

8.3.1 Calculate the final leachate volume (V_f) for each test including blanks. The amount of water loss is equal to the weight loss that occurred during heating.

8.3.2 Calculate the average blank concentration in appropriate units for each element, i, for each series of tests, from the following equation:

$$c_i(blank) = \frac{1}{(T)\,(V_i)} \sum_{m=1}^{T} c_i(m) \cdot V_f(m)$$

where

c_i (blank)	=	average blank concentration
c_i (m)	=	concentration observed in the mth blank replicate
V_f (m)	=	final volume of leachate measured in the mth blank replicate
V_i	=	initial volume of leachate in blank
T	=	total number of replicate tests performed

8.3.3 Calculate the corrected leachate concentration of each replicate from the following equation:

$$c_i^1(n) = \frac{c_i(n) \cdot V_f(n)}{V_i(n)} - c_i(blank)$$

where

c^1_i (n) = leachate concentration for the ith element in the nth replicate corrected for any volume change in the leachate during the test and for the average blank concentration.

c_i (blank)	=	average blank concentration
c_i (n)	=	concentration observed in the nth

sample replicate

$V_f(n)$ = final volume of leachate measured in the nth sample replicate

$V_i(n)$ = initial volume of leachate in the nth sample replicate

If $c^1_i(n)$ and $C_i(n)$ differ by more than 14% for boron, 17% for silicon, and 26% for sodium due to either volume changes or high concentrations in the blanks the data should not be used.

8.3.4 Calculate the average corrected leachate concentration for element i from the equation:

$$c_i(average) = \frac{1}{T}\sum_{n=1}^{T} c_i^1(n)$$

where

c_i (average) = the average corrected leachate concentration of element i observed in the test.

8.3.5 Calculate the standard deviation for the corrected leachate concentrations for various elements.

8.4 **Reporting and Deviations** - All data should be recorded in a laboratory data book, a floppy disk and a hard disk. Deviations from the test method and the expected effect on the results should be discussed.

8.5 Precision and Bias

8.5.1 **Precision:**

The data used to generate the measures of precision is the result of intra- and inter-laboratory round robins. These measures are typical of the methods applied to the glasses and standards used in the round robins, and are not all inclusive with respect to other types of glasses. The measures of precision were determined in accordance with procedures in ASTM Practice E691. These

measures are designated as follows:

Repeatability: the standard deviation for within-laboratory determinations.

Reproducibility: the standard deviation for between-laboratory determinations.

PCT Within-Laboratory Precision:

Approved Reference Glass ARM-1 (a simulated borosilicate nuclear waste glass). Within-Lab relative standard deviation for B was 2.3% and for Si was 1.8%.

PCT Between - Laboratory Precision:

Approved Reference Glass ARM-1 (a simulated borosilicate nuclear waste glass). Between-laboratory standard deviation (including within laboratory and between laboratory %RSD) for B was 12% and for Si was 10.2% for laboratories with varying analytical capabilities.

NIST Reference Glass SRM 623 (a borosilicate glass). Between-laboratory standard deviation (including within laboratory and between laboratory %RSD) for B was 19.8%.

8.5.2 **Bias:**

The average corrected leachate concentrations for the standard glass allow assessment of long term bias or variability of the test, e.g. how reproducible the experimental variables such as oven temperature, sieving, leachate analyses, etc. are over time. Use of a standard glass provides the basis for both within-laboratory and between-laboratory data comparisons.

9.0 REFERENCES and NOTES:

ASTM Standards:
D 1193 Specification for Reagent Water [1]
D 1193 Standard Test Methods for pH of Water [1]
D 1125 Test Methods for Electrical Conductivity and resistivity of Water [1]
E 691 Standard Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method [2]

Other Documents:
Nuclear Waste Materials Handbook: Test Methods [3]
Product Consistency Test Round Robin Conducted by the Materials Characterization Center-Summary Report [4]
Product or Consistency Test (PCT) for DWPF Glass [5]
Nuclear Waste Glass Product Consistency Test (PCT) Method [17]

1. Annual Book of ASTM Standards, Vol 11.01
2. Annual Book of ASTM Standards, Vol 14.02
3. DOE/TOC-11400, Revision 7, July 31, 1986.
4. Pacific Northwest Laboratory, PNL-6967, September 1989.
5. Part I. Test Development and Protocol, DPST-87-575, Savannah River Laboratory, Aiken, SC 29808, July 30, 1987.
6. American Geophysical Union, Dictionary of Geological Terms, 1976.
7. J.W. Shade and D.M. Strachan, Am. Ceram. Soc. Bull 65 [12] 1568-1573, 1986.
9. This does not preclude using natural groundwater in place of ASTM Type I Water if data on glass performance under repository relevant or other natural conditions is desired.
10. A nuclear-grade resin mixture of the strong base cation exchanger in the hydroxide form with a one-to-one cation to anion equivalence ratio, such as that available from the Millipore Corp, Bedford, Ma 01730; Barnstead Co., 225 Rivermoor St., Boston, MA 02131; Illinois Water Treatment Co., 854 Cedar St., Rockford, IL 61105; or Vaponics, Inc., 200 Cordage Park, Plymouth, NA 02360, is suitable.
11. An in-line filter such as those made by the Millipore Corp., Bedford, MA 01730; Gelman Instrument Co., 600 S. Wagner Road, Ann Arbor, MI 48106; and Schleicher and Schuell, Inc., 540 Washington St., Keene, NH 1003, has been found satisfactory.
12. G.L. McVay and C.Q. Buckwalter, Effect of Iron on Waste-Glass Leaching, "J. Am. Ceram. Soc., 66, 170-174 (1983).
13. The SA/V of the sample/leachant system shall be examined in the range in which the parameter (SA/V)*(time in days) is ≥100 cm^{-1} day to avoid short term test fluctuations. For 100-200

mesh particles of density of ~2.75 g/cc this converts to a (m/V) of 0.1 at 7 days or 0.7 (g/cc)*day. The 7 day recommended test duration, therefore, does not preclude longer test durations.

14. If presterilized sample vials are not available for the leachates at the termination of the leaching test, boil polyethylene specimen bottles and caps for 1 hour in ASTM Type I water. Allow the specimen bottles to remain in the water overnight but reduce the temperature so that boiling has stopped. Remove the bottles and caps and dry in an oven at 80°C.
15. Other acidification concentrations can be used if these are found to be more compatible with the acidification concentrations used in your analytic facility for cation analysis.
16. Precision and bias cited from references 4 and 5 are for Version 1.0 and 2.0 of the PCT which did not require sample washing. Better precision has been observed when samples are washed but sample bias remains the same (C.M. Jantzen and N.E. Bibler, in preparation).
17. C. M. Jantzen and N. E. Bibler, "Nuclear Waste Glass Product Consistency Test (PCT) Method, Version 3.0", Savannah River Laboratory.

UNCONTROLLED

VSL Technical Procedure

TITLE: Analysis Procedures

DOCUMENT: TPI-AL Rev # 1

ISSUED: 10/91 REV #: 0
REVISED: 10/92 REV #: 1

Documents Incorporated:

TPI03.WVN	Rev. #0
TPI-AL.WVN	Rev. #3
TPI-CA.WVN	Rev. #3
TPI-DP.WVN	Rev. #1

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ANALYTICAL LABORATORY PROCEDURES MANUAL

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1.0 PURPOSE

These procedures describe methods, materials, equipment and special conditions required to perform chemical analyses of liquid and/or solid samples. These procedures provide the means for obtaining verifiable, reproducible, documented data from tests and experiments.

2.0 APPLICABILITY

An indispensable part of the work carried out in our laboratories is chemical analysis. Various instruments are available for this purpose including a Spectrametrics DC plasma spectrophotometer (DCP), a Dionex Model 2010I ion-chromatograph, Inductively Coupled Plasma Mass Spectrometer (ICP-MS) and two Perkin Elmer Models 330 and 323 spectrophotometers.

Work with the DCP and DIONEX devices is performed in our "Analytical Laboratory". The other two instruments mentioned above, as well as other chemical analysis tools, are used in our "General Chemistry Laboratory". Analysis on ICP-MS is performed in Mass Spectrometry Laboratory.

These procedures apply to all employees trained and authorized to perform chemical analysis of liquid and/or solid samples in the VSL Analytical Facilities. Specific activities covered by these procedures include: a) acceptance of samples for analysis; b) preparation and handling of stock standard solutions and other reference materials; c) preparation of liquid and/or

solid samples for various types of analysis; d) calibration and operation of (i) the DC plasma emission spectrophotometer; (ii) the ion exchange chromatograph; (iii) the inductively coupled plasma-mass spectrometer; (iv) the Perkin Elmer 330 spectrophotometer; e) release and assurance of reliability of analytical results; f) record keeping; g) data processing of analytical data.

These procedures will be implemented by personnel from the VSL.

3.0 SAFETY

3.1 HAZARDS

The definition of a hazardous chemical is given in Section 8.3 of the CUA Chemical Materials Safety Manual, adopted from the OSHA Hazard Communication Standard. The CUA Chemical Materials Safety Manual is hereafter referred to as the CCMSM. This Standard and its application to CUA facilities are described in Sections 8.1 of the CCMSM, with the exceptions noted in Section 8.28.

3.2 HAZARD MITIGATION

3.2.1 Identification of Hazards

The identification of chemical hazards should include an education program (Section 8.2 of the CCMSM). This program should include familiarization with the CCMSM as a whole,

including the toxic properties of chemicals according to Sections 8.22, 8.23, 8.24 and 8.25 of the CCMSM, other hazards of chemicals classified under Sections 8.9, 8.10, 8.11, 8.12, 8.13 and 8.14 of the CCMSM, chemical handling and storage procedures (see paragraph 3.2.2 below), and emergencies (see paragraph 3.2.3 below). The education program will also include familiarization with the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University.

Other elements of hazard identification include the proper use of labels (Section 8.4 of the CCMSM) and of Material Safety Data Sheets (Sections 8.5, 8.26 and 8.27 of the CCMSM). Workplace chemical lists should be compiled, maintained, and made available for employees in accordance with Section 8.31. In addition, a copy of the CCMSM should be kept in the laboratory and its use should be included in the education program cited above. The same should apply to the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University.

3.2.2 Handling and Storage of Chemicals

Handling of chemicals should be conducted according to Section 8.6 of the CCMSM. Storage of chemicals should follow Section 8.8 of the CCMSM. Special precautions should be taken in handling and storage of incompatible chemicals, solvents, compressed gases, and radioactive materials according to Sections 8.10, 8.12, 8.13, and 8.14 of the CCMSM, respectively. Unstable chemicals according to Section 8.9 should be excluded from the

analytical facilities except in cases where the introduction of such a material is sanctioned by the laboratory manager in writing. In such a case the manager shall specify the maximum quantity of the material and the maximum period it may be stored before being discarded. Shock-sensitive chemicals according to Section 8.11 should never be introduced into the laboratory under any circumstances. No work requiring the use of restricted areas according to Section 8.15 should be performed within the analytical laboratories, except on temporary basis and only if adequate modifications have been made to the facilities and the specific procedures have been established. This includes all operations involving radioactive materials.

Waste disposal of hazardous chemicals should follow procedures established by the CUA Environmental safety Office according to Section 8.16 of the CCMSM. No radioactive waste shall be disposed of by analytical laboratories. All processed samples and their overflow should be returned to the originator of the samples for disposal. Any solid radioactive waste generated during operations is to be collected in separate containers and transferred to CUA Radiation Safety Office (RSO).

3.2.3 Emergencies

Chemical spills should be dealt with according to Section 8.17 of the CCMSM. Fire and fire-related emergencies should be controlled according to Section 8.21 of the CCMSM. Cases of personal contamination should be treated according to Section 8.19 of the CCMSM, cases of injury and illness according to Section 8.18, and cases requiring minor first aid according to

Section 8.20. All emergencies involving radioactive materials should be reported immediately to the RSO and dealt with according to the procedures outlined in the Radiation Safety Manual distributed by the RSO.

3.3 PERSONNEL PROTECTION

Personnel protection should consist of two elements. The first is familiarization and compliance with the instructions of the CUA Chemical Materials Safety Manual (CCMSM), including identification of hazards (see paragraph 3.2.1 above), handling and storage of chemicals (see paragraph 3.2.2 above), and dealing with emergencies (see paragraph 3.2.3 above). The second element is familiarization and compliance with the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University. The overall chemical hygiene program (Section 8.34 of the CCHP), based on the OSHA Regulation on Occupational Exposure to Hazardous Chemicals in Laboratories, should be followed. In detail, exposure reduction measures (Section 8.35 of the CCHP) will include environmental monitoring (8.35.1), inspections, maintenance and housekeeping (8.35.2), use of protective apparel and equipment (8.35.3), posting of signs and labels (8.35.4), and dealing with accidents and spills (8.35.5). Ventilation of both general laboratory spaces and of special areas and local devices such as chemical fume hoods and glove boxes should follow the instructions of Section 8.36 of the CCHP. Employees should take part in the safety information and training program which exists in the University according to

Section 8.37 of the CCHP. All personnel involved in handling of radioactive materials should be approved by the RSO and should participate in Radiation Safety Training Program provided by the RSO. Certain laboratory operations may require approval from the laboratory manager prior to implementation according to Section 8.38 of the CCHP. Employees should be informed about the medical program provided by the University in cases of work with hazardous substances according to Section 8.39 of the CCHP. Responsibility for chemical hygiene rests with all employees. Specific chemical hygiene responsibilities as applied to the analytical facilities are specified in Section 8.40 of the CCHP. Analytical laboratories personnel workers should be familiar with the basic rules and procedures for working with chemicals included in Section 8.41 of the CCHP and should work in full compliance with these rules and procedures. Animal work, whether involving chemicals of high chronic toxicity according to Section 8.41.5 of the CCHP or not involving chemicals of this type, should not be carried out in the analytical facilities.

4.0 REQUIREMENTS

4.1 TRAINING

All analytical laboratory personnel are trained in the understanding and use of the technical methods that they will use by the laboratory supervisor or by a trained and experienced person designated by the laboratory supervisor. Upon completion of training for a method, a technician is qualified to use that method independent of direct supervision.

4.2 CONTROL OF STANDARDS

Stock Standard Solutions

(1) All the instrumental analysis in the Analytical Laboratory are performed on relative basis by comparison with known standards.

(2) Sufficient stock of concentrated Standard Solutions should be kept in the Analytical Laboratory to allow at least 30 days of continuous work. (This means about 200 ml of solution containing approximately 1000 ppm of specimen.)

(3) The term "concentrated solution" means the solution contains 1000 $\pm$ 20% ppm of the analyte in question. No stock of more diluted standard solutions is allowed to be kept in the laboratory for a period exceeding 24 hours. Diluted solutions containing one or more of the following elements: Si, Fe, U, Th should be freshly prepared every 8 hours.

(4) Stock of standard solutions to be analyzed by atomic emission/absorption techniques should be purchased from major commercial sources. No use of solutions is allowed beyond expiration dates, if such dates are provided by the manufacturer or when the volume in the bottle falls below 1/8 of the total volume.

(5) Standard Solutions of anionic species for ion chromatography should be purchased from the National Institute for Standards and Technology in the form of 1000 ppm solutions.

All standard solutions of cations shall be analyzed relative to NIST standards on a monthly basis and a limit of $\pm$ 5% will be acceptable.

5.0 REFERENCES

Vogel, A. "Textbook of Quantitative Inorganic Analysis," 4th ed., Longman Scientific and Technical, England 1987.

Willard, H. H., Merritt, L. L. Jr., Dean, J. A. and Settle, F. A. "Instrumental Methods of Analysis," 6th Ed., D. Van Nostrand Co., New York 1981.

Boumans, P. W. J. M. (Ed.) Inductively Coupled Plasma Emission Spectroscopy," John Wiley and Sons, New York, 1987.

A. R. Date and A. L. Gray (Ed.) "Applications of Inductively Coupled Plasma - Mass Spectrometry," Champman and Hall, New York 1989.

The Catholic University of America Chemical Materials Safety Manual, January 1989.

The Catholic University of Chemical Hygiene Program, in preparation.

Radiation Safety Manual of the Catholic University of America, January 1980, W. E. Keene RSO.

6.0 OPERATIONAL PROCEDURES

6.1 SAMPLE PREPARATION

6.1.1 Preparation of Samples for Analysis

(1) Only liquid samples can be analyzed.

(2) With liquid samples, proceed as per procedure in section 6.1.2 below.

(3) Solid samples have to be converted into liquid form as per procedure in section 6.1.3 below.

6.1.2 General Solution Analysis

Procedure

Depending on what elements and ions are expected to be present, solutions are analyzed using a DC plasma spectrophotometer (DCP), and a Dionex ion exchange chromatograph. Generally, the DCP is used for Zr, Zn, P, Si, Ti, B, Mn, Fe, Co, Ca, Al, Cu, Mo, Ce, Sr, K, Na, Li, Mg, Ni, Nd and Cr analysis, and the Dionex to analyze anions such as F-, Cl-, NO2-, Br-, PO4—, NO3- and SO4—.

Qualitative handling of the solutions should be performed utilizing calibrated volumetric tools. These include volumetric flasks of various sizes and automatic dispensing pipettes (see Table in this section) with disposable tips (having fixed or variable volumes). To assure the accuracy of the volumetric tools, the following routine procedure should be applied:

Table

Qty	Device	Manufacturer	Model #	Calib. Req.
10	Auto. Pipettes			
1	50 microliter	Eppendorf	3130	I
1	100 microliter	Eppendorf	3130	I
1	200 microliter	Eppendorf	4700	I
1	1000 microliter	Eppendorf	4700	I
1	250 microliter	Eppendorf	4700	I
1	500 microliter	Eppendorf	4700	I
1	4 milliliter	Oxford	Macroset	I
1	5 milliliter	Oxford	Macroset	I
1	10 milliliter	Oxford	Macroset	I
1	0.025-10 milliliter	Rainin	EDP-25	II
	Volumetric flasks (PMP)	Various		N.A.

I: Calibration check performed every month according to the procedure described in section 6.1.4.

II: Calibration check performed according to the procedure described in section 6.1.4 every six months.

- volumetric flasks : visually examine every volumetric flask prior to use. If there is any mechanical damage to the flask or any permanent, visible solid residue inside, the flask should be discarded. Carefully rinse the flask after use with DI water to avoid deposition of solids.
- dispensing pipettes : gravimetric check of the calibration is required every month as per procedure in section 6.1.4). If the calibration cannot be adjusted, the tool should be replaced. If an adjustment has been performed, the calibration of the instrument should be immediately rechecked before it is returned to general use. The calibration check reports should be filed into the folder and kept in the Analytical Laboratory.

6.1.3 Glass and Sludge Dissolution for Analysis

Note: *Deviations from the methods described below may occur and are recorded in the Analysis Procedures Laboratory Notebook*

Dissolution of Glass, Sludge, Soil and Other Solids

(1) Weigh an amount of of solid as determined by the laboratory supervisor. In the cases of dry solids the solid should be previously ground into very fine powder and dried overnight at 105°C. Record the weight of the sample and transfer to a 125-ml narrow-mouth polyethylene bottle.

(2) Add several ml of one or more concentrated acids selected from the list of HF, HCl, HNO_3 and H_2SO_4 by the laboratory supervisor and record the volumes of each acid.

(3) Leave in sand bath for 2 to 3 hours at (70 $\pm$ 10)°C.

(4) In the case that dissolution is not complete, add further volumes of one or more acids selected from the list given in paragraph 2 by the laboratory supervisor and resume the heating at (90 $\pm$ 10)°C. Repeat as needed to produce complete dissolution. Record all details.

(5) If the glass is completely dissolved, add DI water to bring volume to 100 ml, and analyze the solution as per procedure in section 6.1.2.

6.1.4 Calibration of Pipettes

Method: Gravimetric using an analytical balance.

Frequency: Monthly except Rainin pipette which is six monthly.

Required Accuracy: As specified in individual procedures or as per instructions of the Project Manager.

(1) Obtain a "Pipette Calibration Check Record" form and supply requested information on pipette to be calibrated (e.g. manufacturer, model #, etc.).

(2) Tare out the weight of a 20 ml plastic vial on the balance.

(3) Pipet out deionized water from the pipette to be calibrated into the plastic vial. Record

nominal capacity, actual capacity (weight displayed on the balance), and percent error under the appropriate columns.

(4) Repeat steps (2) and (3) 5-15 times. For variable volume pipettes perform the measurement for 2-5 volume settings.

(5) Calculate the percent error as follows:

$$\% \text{ ERROR} = \frac{(\text{Actual capacity}) - (\text{Nominal capacity})}{(\text{Nominal capacity})} \times 100$$

(6) Examine results and report deviations from required accuracy to the Laboratory Manager.

(7) Sign and date and file form in the 'Calibration Records'

6.2 SOLUTION ANALYSIS WITH DC PLASMA

6.2.1 Solution Analysis with DC Plasma Emission Spectrophotometer (Water Matrix Only)

(1) A volume of at least 5 ml of the solution is required for analysis.

(2) Using a 2% HNO_3 solution as the matrix, prepare a Hi Std solution containing all of the elements of interest. The concentrations of elements in the Hi Std should somewhat (but no more than twice) exceed the expected concentrations of elements in the solution.

(3) Use a 2% HNO_3 solution as the Zero Std (Blank).

(4) Transfer the samples to be analyzed and the standard solutions to centrifuge tubes. At least two sets of standards will be required. Maximum of up to 32 samples can be analyzed per run.

(5) Load the automatic sampler carousel with the samples and the standard solutions according to the following sequence:

Position #1 : Zero Standard (always)
Position #2 : High Standard (always)
Position #3 - #12: Samples (maximum)

For higher positions the sequence can be repeated or terminated by the use of a zero standard followed by a hi standard.

Note: *There is a maximum of up to 10 samples allowed between two adjacent sets of standards. If certain series of samples consist of solutions having wide differences in concentration ranges (factor of 5 or more) and a cross-contamination is likely to occur, the samples can be separated by the zero standard. In that event, the zero standard has to be considered as a sample.*

(6) To perform the analysis and to obtain the report of the results, follow the procedure in section 6.3.2 below.

(7) If the sample (or samples) contain elements which have to be analyzed in a single element mode, perform the multiple element mode analysis first.

6.2.2 DC Plasma Emission Spectrophotometer Operating Procedure

Manufacturer: Spectrametrix Inc.
Model #: Spectra Span III-B

(1) Start-up procedure (to be performed daily or every 8 hours of continuous operation and recorded in the "DCP Maintenance" LN).

(a) Make sure that the plasma power supply switch (PLS) and the argon switch are in the OFF position. Set RUN/READY switch to RUN.

(b) Remove the shield covering the jet assembly.

(c) If the instrument was operated recently, wait a few minutes to give the electrodes sufficient time to cool.

(d) Replace all consumable elements of the jet assembly (i.e. ceramic sleeves, graphite anodes, and the stainless steel cathode).

(e) Turn argon gas ON on the tank and the regulator.

(f) To see if all the electrodes are properly set up, switch the RUN/READY switch to READY. All three electrodes should meet, with the cathode in the center of the far anode, making it bow slightly. If it is not in this READY position, move the cathode back and forth with the cylinder adjusting nut until the conditions above are satisfied.

(g) Turn the RUN/READY switch to RUN, and turn the gas switch off.

(h) Check if the aspiration hose and the waste hose are worn down; if they are,

change both of them, not just one, as this may variably affect the rate of inflow and rate of outflow and excessive moisture develop inside the atomizing chamber.

(i) Carefully replace the shield and protective cover.

(2) To Light the Spectrajet

(a) Make sure that the RUN/READY switch is on RUN.

(b) Turn up the argon gas switch.

(c) Make sure to have the proper gas pressures:

20 psi nebulizer

50 psi sleeves

(d) Turn the RUN/READY switch to READY.

(e) Push the PLS button.

(f) Set the peristaltic pump switch on and have DI water aspirating, as well as the pump clamp engaged.

(g) Once a constant sound is heard, watching the electrodes through the shield, flip the RUN/READY switch to RUN. The flame should appear and have the characteristic inverted "Y" appearance. If only a partial flame appears, or if no flame appears, quickly turn the RUN/READY switch back to READY, wait 5 seconds, and once again flip the switch to RUN. If, again, flame is not there or is not complete, try two more times, and if no success is achieved, flip to

READY, turn PLS button off, gas switch off, and readjust the electrodes. If after this the flame does not light, consult the manual.

(3) Element Programming

(a) At this point, choose the elements desired among the twenty elements on the cassette. To do this, flip the black switches corresponding to the elements in your high std up. The rest of the switches must be down.

(b) Now push in the light above the first black switch that is turned up. The LED display will now show information corresponding to that element.

(c) Set all the PMTs according to the temporary list posted.

(d) Now look at the first lit element. To check whether the correct high std value happens to be already entered from the last run, press DIS HI. The LED display will show the high std concentration for that element from the last run. If a change is desired, press C (to clear), enter the desired high std on the keyboard, and press ENT HI. Now the correct high std has been entered. Again to check, press DIS HI.

(e) To enter the correct low std, do as above, except with DIS LO, and ENT LO.

(f) Now push in the light above the second black switch that is up, and to the above procedure for the element. After the last element is completed, the DC Plasma is programmed to the high and low standards entered.

(4) Alignment of The Flame

(a) Visually inspect the flame - should have a stable, "straight-up" appearance. If not, adjust electrodes.

(b) Listen for an unusually high whistling sound - if present, adjust the gas flow to each anode and cathode with a small screwdriver. Caution should be exercised as this changes the shape, the focus, and the temperature of the flame.

(c) Look at the reflection of the flame on the entrance slit. The "Y" should have a sharp, in-focus appearance. If not, consult the trouble shooting section of the manual.

(d) The central point of the "Y" should be just below the third slit from the bottom, just illuminating the very bottom of the slit. To move the "Y" horizontally or vertically, use the silver set of knobs on the front part of the electrode assembly.

(5) Alignment of the Cassette

(a) Depending on the cassette used have a 10 ppm Cu or Ca solution aspirating.

(b) Turn up the Cu or Ca element switch, as well as the light above it.

(c) Turn the MODE switch to 1, the DIAG switch up, the REPEAT switch to 0, the AVG ONLY up and down, RST.

(d) Put the TIME setting high enough to read about 5000 on the LED display.

(e) Tune in the plasma position.

(f) AVG only up and down, RST.

(g) Optimize the Cu or Ca wavelength - turn the "vertical" and "horizontal" dials until the LED display is as high as it can get. Use the fine adjustment dials for this, as gentle rotation produces the best results.

(h) Once the cassette is aligned according to the Cu or Ca wavelength, switch the MODE switch to INT, TIME to 8, and REPEAT to 3.

(i) Other settings should be:

PRINT	up	BKGRD	down
DIAG	down	STD DEV	up
XMIT	up	LED	down
INT STD	down	ALL CHAN	up
CASSETTE	1		
TEST CURRENT on PMT			
AVG ONLY	down	RST	momentarily pressed

(j) Turn off the Cu or Ca element switch and turn on the light to one of the other elements in your run.

(k) AVG ONLY up and down, RST.

(l) Aspirate DI water.

(6) Standardization

(a) Divide the high and low standards to the appropriate test tubes on the carousel, with enough left in the flasks for calibration.

(b) Aspirate high std solution.

(c) Push A/R button.

(d) Once the A/R button goes off, HI STD button automatically comes on. Once this button goes off, the high std is calibrated.

(e) Have the low std (or blank) aspirating, for at least a minute since no carry-over from the previous high std is desired.

(f) Push LO STD button.

(g) Once this button goes off, the low std is calibrated.

(h) A printout on the high standard will show how accurate the high std was. If these are not satisfactory, both stds should be made new, and the DC Plasma restandardized.

(i) Aspirate DI water.

(7) Computer Programming

(a) Insert a disk with the printed side up and the notch toward the outside. Close the disk drive opening.

(b) Turn on the computer unit and the terminal.

(c) The computer monitor should appear; if not, turn on the reset button in the back of the computer unit.

(d) Type: DCP DATA

(e) Type in the date and the whole program, paying attention to the HS INT (number of samples in each block) in the parameter section, as well as the correct values for the high stds.

(8) Running Samples

(a) Insert the stainless steel aspiration tube into the sampler arm and push the Return button on the terminal. The computer will now take over and run the samples according to the scheme programmed.

(b) Watch for the steel tube being lowered directly into each tube on the carousel!

(c) Once the run is completed, the carousel will be reset into its starting position, calculations and the final printout should follow.

(d) Take the steel aspiration tube out of the sampler arm and aspirate with DI water

(9) Single Element Mode

(a) Replace multi-element cassette with single element cartridge.

(b) Use the first channel for all single mode elements.

(c) Refer to the "Handbook of the Spectral Lines of Elements for DC Plasma/Echelle Systems" and select the best line for the particular element of interest.

(d) Standardize the instrument with the standard solutions of that element (ref to para. 6 above)

(e) Run Samples as described in paragraph 8.

(10) Shutdown Procedure

(a) Take the aspiration tube out of DI water and aspirate air for about one minute.

(b) Press the PLS button so light is off.

(c) Flip the RUN/READY switch to READY

(d) Shut the argon gas off at the tank.

(e) Wait until the lines are bled, the regulator show '0', then flip the gas switch off or down. Then shut the argon gas off at the regulator.

(f) Turn the peristaltic pump switch off, and lower the pump clamp assembly.

(g) Set all PMT settings to '1'.

(h) Shut down the computer unit, the terminal, and the cooling water pump!

(11) Daily Maintenance (Record in "DCP Maintenance " LN)

(a) Replace all consumable elements of the jet assembly and align electrodes.

(b) "Peak" the system with Cu or Ca solution.

(12) Weekly Maintenance (Record in "DCP Maintenance" LN)

(a) Replace all plastic hoses.

(b) Rinse complete aspiration system with DI water.

(c) Rinse, clean and oil all ceramic rollers.

(d) Replace systems cooling water with fresh DI water.

(e) Clean the nebulizer using DI water and compressed air.

(f) Replace all consumable elements of the jet assembly

(g) Align electrodes

(h) "Peak" the system with Cu or Ca solution and align optics

(i) Seal all connections with teflon film tape

(j) Check channel's electronic function

6.3 ANALYSIS WITH ION EXCHANGE CHROMATOGRAPHY

6.3.1 Solution Preparation and Analysis

(1) About 2 ml of solution are required for analysis.

(2) Prepare the standard solution containing all the anions of interest, matching approximately the expected concentrations of anions in the sample to be analyzed.

(3) Refer to procedure in section 6.3.2 and perform the analysis of the standard solution first. Note if there is any change in retention time for each of the various anions by comparing the chromatogram with the most recent one. Then, perform the analysis of the sample.

(4) To obtain the results, Proceed as follows:

(a) identify the peaks of the appropriate anions by comparison with the standard output.

(b) measure the height of the peaks

(c) if no change in retention times has been observed (para. (3) above), obtain the results from the calibration curve (para. (5) below). If the retention times have changed, either compare the standard output with the sample output or construct the new calibration curve.

(5) Calibration curve is a graph showing the concentration of a particular anion species (in

ppm) against the appropriate peak height (in mm) which should be performed every 6 months.

6.3.2 Dionex Chromatograph Ion Exchange Operating Procedure

Manufacturer: Dionex
Model #: 2010i

(1) Operation Instructions for System Components

(a) Analytical Pump

(i) Eluent Section

Before beginning operation, prime the eluent manifold with the desired eluents (including regenerent and water, if used). After changing or refilling an eluent reservoir, reprime the corresponding eluent valve.

Procedure:

(1) Press STOP/START to stop the pump.

(2) Attach an empty 10 ml syringe to the luer adaptor on the priming valve.

(3) Turn the small handle on the priming valve to align with the syringe.

(4) If desired, fill the eluent reservoirs and connect them to the eluent bulkhead connectors on the pump.

Note: *Prime only one valve at a time.*

(5) Select an eluent (press the switch 'ON'). Use the syringe to draw liquid

from the reservoir, through the liquid lines and the eluent manifold, into the syringe. This flushes out any air or liquid trapped previously in the liquid line.

(6) Turn off the eluent valve just flushed. Remove and empty the syringe.

(7) Replace the syringe, select the next eluent, and repeat Step 5.

Note: *When making an eluent change while the pump is running, first select the new eluent, wait 1-2 seconds, then turn off the old eluent. This prevents the formation of bubbles in the eluent manifold during the change.*

(8) Repeat Steps 5 and 6 for the remaining eluents (if used).

(ii) Priming The Pump

The pump rarely loses prime when in continuous, routine use. However, if an eluent reservoir empties during operation, or if the pump has not been used for a month or more, it may be necessary to reprime the pump before resuming operation.

——————— CAUTION ———————

Bypass all columns before priming the pump to

avoid accidental system overpressing and possible column damage.

(1) Turn the knob on the pressure transducer housing counterclockwise approximately one-half turn. This opens the pressure transducer waste valve.

(2) Select one eluent (turn on the valve).

(3) Set FLOW SELECT to 1.0 ml/min.

(4) Set LOCAL/REMOTE to LOCAL.

(5) Set the LOCAL PRESSURE LIMIT SELECT to zero (00 x 10 psi) and the HIGH PRESSURE LIMIT SELECT to 500 (5 x 100 psi).

(6) Fill the 20 ml syringe with isopropyl alcohol. Connect the syringe to the priming valve luer adaptor.

(7) Turn the small handle on the valve to align with the syringe, opening the internal valve.

(8) Press START/STOP to start the pump.

(9) Slowly push the alcohol through the pump with the syringe. Ensure that alcohol is pushed through both heads by watching the Delivery Status LEDs: each LED must light at least once while pushing the alcohol through the pump.

Note: *Avoid pushing any air trapped in the syringe through the pump. This will result in the loss of prime.*

(10) Set FLOW SELECT to 4.0 ml/min and flush the pump with eluent for 2 minutes.

(11) Turn the handle on the priming valve to the right of the open position, closing the valve.

(12) Set FLOW SELECT to the desired flow rate.

(13) Turn the knob on the pressure transducer housing clockwise to close the waste valve.

---------- CAUTION ----------

Overtightening may damage the pressure transducer housing or the valve seal.

---

(14) Place the columns in line and set the pressure limits.

(15) Wait for the READY LED to light, indicating that the pump flow-control system is operating, before beginning an analysis.

(iii) Selecting The Pressure Limits

The low and high pressure limits automatically stop the pump in the event of system malfunction (e.g., overpressuring or a leak downstream from the pump). After priming the pump, select a high pressure limit 50-100 psi above the normal system operating pressure. The pump has an automatic safety high pressure limit of 1900 psi, above which it will not operate. Select a low pressure limit 100-200 psi below the normal system operating pressure.

Note: *The low pressure limit may be inactivated, as during priming, by setting the LOW PRESSURE LIMIT SELECT switches to zero.*

(b) Advanced Chromatography Module

(i) Routine Operation

During an analysis, Valve A is OFF, directing eluent flow through the injection valve to the separator, the suppressor (if used), and finally, through the detector cell to waste.

Turn Valve A On during on-line regeneration of the suppressor column. This reverses the flow through the suppressor and bypasses the injection valve, the separator, the cell, and all interconnecting tubing. See the Column manual for complete suppressor regeneration instructions. (**Note:** *this procedure does not apply for fiber type suppressor which is self-regenerating.*)

Periodically empty the waste container to prevent overflow. Take appropriate precautions when handling the strong acid and/or base waste from suppressor regeneration.

(ii) Making An Injection

The Advanced Chromatography Module is equipped with a 50 μl sample loop. Load sample into the sample loop through the SAMPLE PORT by the following method:

(1) Flush Sample Loop. Attach a syringe to the SAMPLE PORT. Insert the end of the tubing from the #2 bulkhead fitting into a small container of deionized water or eluent. Set the LOAD/INJECT switch to LOAD and draw at least 10 loop volumes (e.g., 0.5 ml for a 50 ul loop) of liquid into the syringe, flushing the sample loop completely.

(2) Load sample. Wipe off the end of the tube, then insert it into the sample container. Draw at least 10 loop volumes of sample into the syringe, filling the loop completely. Subsequent repetitive injections require only 5 loop volumes of sample.

(3) Inject Sample. After the detector baseline is stable, offset it to zero, and set the LOAD/INJECT switch to INJECT.

(c) Conductivity Detector

(i) Start-up

After installing the detector, or whenever the detector power has been off, follow this start-up procedure to reset the detector microprocessor.

(1) Check that all cables are correctly connected and that the detector is plugged into the power strip.

(2) Check that the power switch on the power strip is 'ON'.

(3) Turn on the POWER switch.

(4) Set the LOCAL/REMOTE switch to LOCAL.

(5) Turn 'OFF' the AUTO-OFFSET.

(6) Turn 'OFF' THE cell, then turn it to 'ON'.

(ii) Routine Operation

Check that CELL is on. Turn off the AUTO-OFFSET during system equilibration. Select an OUTPUT RANGE which will allow the baseline to be monitored on the recorder. After the chromatographic system is equilibrated, and the baseline is stable, select the OUTPUT RANGE sensitivity required for the analysis. Set TEMPERATURE COMPENSATION to the desired value (see Section iv). Then turn AUTO-OFFSET 'ON' just before beginning an analysis. The baseline may be reset to zero at any time by momentarily turning off AUTO-OFFSET then turning it back on.

(iii) Eluents

In general, eluents used with conductivity detection should have conductivities less than 50 μS for optimum detector sensitivity. In Ion ChromatographyTM, highly conductive eluent ions are removed from the eluent stream or converted to less conductive forms by the suppressor before entering the cell.

(iv) Temperature Compensation Optimization

The TEMPERATURE COMPENSATION setting for most eluents is approximately 1.7 %/°C. Optimize the Temperature Compensation setting for each column set and

eluent batch to minimize temperature-caused variations in the measured conductivity. This is especially important when operating at high sensitivities. The optimum setting for any eluent is determined as follows:

(1) Install a 30 cm (12 in) length of 0.5 mm ID tubing between the suppressor outlet (or Port #3 on Valve A) and the cell inlet.

(2) Prepare a beaker of room-temperature water and one of boiling water. Place the tubing from Step 1 in the room-temperature water.

(3) Install the columns.

(4) Set TEMPERATURE COMPENSATION to 2.0 %/°C.

(5) Begin pumping the eluent through the system.

(6) Select an OUTPUT RANGE which keeps the baseline on scale.

(7) After the system reaches equilibrium, and the baseline is stable, set the OUTPUT RANGE to 30 μS. Turn on AUTO-OFFSET. Position the recorder pen about halfway up the paper.

(8) Record the baseline for 1 tp 2 minutes.

(9) Remove the tubing from the room-temperature water and place it in the boiling water for 1 to 2 minutes. Note the recorder baseline shift. If the baseline shifts positive, the TEMPERATURE COMPENSATION setting is too low. If it shifts negative, the setting is too high.

(10) Place the tubing in the room-temperature water and allow the baseline to

restabilize for 1 to 2 minutes.

(11) Based on the results of Step 9, increase or decrease the TEMPERATURE COMPENSATION setting by 0.5 %/°C for the first trial and by 0.1 %/°C for successive trials.

(12) Repeat Steps 9 and 10 until the recorder baseline shift is at a minimum.

(13) Disconnect the tubing and reconnect the suppressor to the cell inlet as it was originally.

(14) Close the column compartment, covering the valve compartment.

(2) Performing Analysis

(a) Initial Operation

If a new column is installed, verify that the system is operating correctly by duplicating the test chromatograms sent with the columns. This also provides an opportunity to become familiar with operating the system before beginning actual analyses.

(1) Read Paragraph 1 above. It contains detailed operating instructions for each component.

(2) From the test chromatogram sent with each separator column, determine the eluent, flow rate, and full-scale setting used to analyze the test standard.

(3) Prepare the eluent and suppressor regenerant (if required) specified on the test chromatographs and connect them to the pump eluent manifold system.

(4) Select the flow rate indicated on the test chromatogram and allow the system to equilibrate with the eluent to be used in the analysis.

Note: *Normally, at least 20 column volumes of eluent are required for complete equilibration.*

(5) Set the conductivity detector TEMP COMP to 1.7.

Note: *The value 1.7 is the nominal Temperature Coefficient of the "standard anion eluent". Use this value with any eluent for the purposes of this test. During actual analysis, use the experimentally determined eluent temperature coefficient for optimum performance.*

(6) Turn on the recorder. Set the conductivity detector OUTPUT RANGE to a value that keeps the recorder pen on scale and monitors the base line. Once the baseline is stable, select the detector output range indicated on the test chromatogram.

(7) Load the sample loop with the standard solution shipped with the columns, offset the baseline, and inject the sample.

(8) Compare the chromatogram obtained with the test chromatogram.

(9) Calculate the efficiency and line resolution of the peaks indicated on the

test chromatogram. The calculated values should agree within 20-25% of those on the test chromatogram.

Note: *Slight differences are due to instrument variations and /or differences in temperature and eluent strength from test conditions.*

(b) Routine Operation

Routine operation consists of injecting samples using the procedure described in the paragraph above.

(c) Short-term Shutdown

Following completion of the daily operation, the system is normally set to stand-by state until operation is resumed.

(1) Turn off the pump, and recorder. Set the conductivity detector RANGE to '10k' μs and turn 'Off' CELL.

(2) Leave the POWER 'On'.

Note: *If the power is turned 'Off', the conductivity detector must be reset when operation is resumed.*

(d) Prolonged Shutdown

Use the following shutdown procedure if the system will not be used for two weeks or more.

(1) Prepare columns for storage.

——————— CAUTION ———————

Improper storage of columns may result in permanent degradation and loss of performance.

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(2) Remove the columns from the system, couple the column lines together, and flush the system with deionized water for 30 minutes at 9.9 ml/min.

(3) Turn off the POWER switch.

6.4 SOLUTION ANALYSIS WITH ICP-MS

6.4.1 Solution Preparation and Analysis

(1) Obtain the run information sheet and labels for the run from the Data Processing laboratory and obtain the samples from the Leach laboratory

(2) Prepare the standards required to be used in the run according to the instructions given on the run information sheet using the automatic pipette, 20 ml scintillation vials and polymethylpentane volumetric flasks.

(3) Add about 8 mls of the WASH solution (2% HNO_3 with deionized water) and about 8 mls of the ZERO solution (1% HNO_3 prepared by dilution of 70% HNO_3 with deionized water) to the required number of scintillation vials for the run.

(4) Arrange the vials in the order given on the run information sheet and label them with the sticky labels provided.

(5) Perform the analysis according to procedure in section 6.5.2 below.

6.4.2 Inductively Coupled Plasma-Mass Spectrometer Operating Procedure

Manufacturer: VG Instruments
Model #: PQI

A. <u>Turning on of Instrument</u>

(1) Open the two valves marked "Gas Supply" and "Pressure Buildup" on the argon cylinder.

(2) Ensure that the supply of argon is sufficient for one day's work by checking that the

liquid level marker in the central indicator on the cylinder is at least one quarter of the way up between the two points marked "N".

(3) Open the air by turning the black valve completely counterclockwise and then clockwise back again one quarter of a turn.

(4) Turn the intensity knob clockwise on the Tracor-Northern MCA of the instrument until the letters TN-7200 are of acceptable brightness.

(5) Ensure that the TIME switch on the Quadrupole Control panel is set to "EXT".

(6) Turn on the electric switch of the surge protection unit under the computer.

(7) Place about 100 mls of deionized water in a 120 ml urine sample cup and place the cup in the position allocated for it in the start-up and tuning rack of the autosampler.

(8) Set the start-up and tuning rack in the rightmost position of the autosampler tray.

(9) Ensure the cap has been removed from the cup.

(10) Turn on the autosampler.

(11) Load file #6 into the autosampler control keypad by hitting the "EDIT" key, the "6" key and then the "ENTER" key twice.

(12) Press the "START" key and the probe will enter the cup containing the deionized water.

(13) Load the peristaltic PVC tubes on to the head of the peristaltic pump and turn the pump on.

(14) Wait about one minute while deionized water is pumped through the instrument.

(15) Turn off the peristaltic pump.

(16) Turn on the computer and printer.

(17) At the prompt "C:\>" type "ICPMS" and strike "ENTER" key.

(18) After about 2 minutes the ICPMS options will appear on the screen and then load the instrument control program by striking the F1 key.

(19) Turn on the plasma of the instrument as follows:

WARNING: FAILURE TO EXACTLY FOLLOW THIS PROCEDURE MAY LEAD TO MELTING OF THE TORCH AND TO EXTENSIVE DAMAGE TO THE INSTRUMENT.

(a) Depress the three green buttons marked "COOL," "AUX" and "NEB" on the MS81 gas control panel.

(b) Adjust the flow rates of the gases to:

(i) 12 lit/min for the cooling gas

(ii) 2.5 lit/min for the auxiliary gas

(iii) 0.7 lit/min for the nebulizer gas

(c) Wait at least 5 minutes.

(d) Switch off the nebulizer gas.

(e) Wait until the flow indicator in the gas control panel flashes.

(f) Depress the red button marked "POWER" on the plasma radio frequency (RF)

generator.

(g) IMMEDIATELY ignite the plasma by depressing the red button marked "IGNITE" on the gas control panel.

(h) If the plasma does not form within 5 seconds depress the "IGNITE" button again.

(i) If the plasma still does not form IMMEDIATELY depress the yellow button marked "STANDBY" located on the plasma RF generator and consult with the project manager.

(j) IMMEDIATELY after the plasma is formed depress the green button marked "NEB" on the gas control panel.

(20) Turn the peristaltic pump on again in order to pass deionized water into the plasma.

(21) Hit the F4 key on the computer keyboard labelled "ICP CONTROL" followed by the F6 key labelled "TORCH POSITION".

(22) Once the torch has moved forward hit the "Esc" key.

(23) Hit the F5 key labelled "SAMPLE INTERFACE" followed by the F1 key labelled "EXPANSION PUMP".

(24) Wait until the expansion pressure reads "OK" and then hit the F3 key labelled "EXTRACTION ON/OFF".

(25) If the computer indicates that extraction lens 2 is not activated hit the F4 key labelled "EXTRACTION 1/2".

(26) Check that the expansion pressure reads "OK", then wait about one minute and then hit

the F2 key labelled "SLIDE VALVE".

(27) Ensure that the pressure reading on the Edwards vacuum controller gauge is below 5 x 10^{-6} mbar.

(28) If any problem exists call the Project Manager.

(29) Hit the Esc key and then hit the F6 key labelled "QUAD STATUS" in order to switch on the quadrupole analyzer. The value 106.5 should appear on the mass indicator of the quadrapole control panel of the instrument.

(30) Hit the F7 key labelled "DETECT MODE" followed by the F1 key labelled "HT ON/OFF".

(31) Check that the rate meter on the plasmaquad preamplifier panel shows a response of less than 50 Hz when the ratemeter is set to 100 Hz FSD. If the response is greater than 50 Hz call the project manager.

(32) Hit the "Esc" key followed by the "Q" and "Y" keys in order to return to the main ICPMS driver menu.

(33) Wait half an hour for the instrument to warm up.

B. Initialization of Analysis Run

(1) Load the start up and tuning rack of the autosampler with a urine sample cup containing about 100 mls of deionized water and the vials containing the tuning solution (TSO), the marker solution (MSO) and the WASH solution placing them in the positions marked on

the rack.

(2) Set the startup and tuning rack in the rightmost position of the autosampler tray.

(3) Load file #5 into the autosampler control keypad by striking the "EDIT" key, followed by the "5" key and then the "ENTER" key twice.

(4) Press the "START" key and the probe will enter the cup containing the deionized water.

(5) Set the quadrupole analyzer to 115 amu by turning the TIME switch on the Quadrupole control panel from "EXT" to "0.1" and adjusting the coarse and fine mass control dials.

(6) Press the "START" key again and the probe will enter the vial containing the tuning solution.

(7) Set the attenuation of the ratemeter in the Plasmaquad preamplifier panel to a full scale reading of 100 KHz.

(8) If the signal is greater than 20 KHz the response is acceptable otherwise call the project manager to tune the instrument.

(9) Return the TIME switch to "EXT".

(10) Press the "START" key again on the autosample control keypad and the probe will enter the WASH solution.

(11) Wait at least 3 minutes.

(12) Meanwhile load the peak jump isotope ratio program into the computer by carrying out the following:

(a) Hit the F6 key labelled "PJ ACQUIRE".

(b) Hit the F2 key labelled "IR PJ".

(13) Once the program is loaded hit the F9 key labelled "CALIB PARAM".

(14) Hit the F1 key labelled "PRINT INT" and then hit the "Y" key.

(15) Hit the F3 key labelled "PJ MARKER".

(16) Enter the name of the appropriate element menu and the message "waiting for marker sample" will appear.

(17) Press the "START" key again on the autosampler control keypad and the probe will enter the marker.

(18) Wait at least 2 minutes.

(19) Hit the F5 key labelled "ACQUIRE SAMP" in order to perform the marker run.

(20) Once the run is completed hit the F4 key labelled "MASS CAL" and then type "P" as the mass calibration type.

(21) Hit the "Esc" key.

C. Performance of Analysis Run

(1) Hit the F1 key labelled "QUEUE ACQUIRE".

(2) Enter the name(s) of the appropriate analysis file(s).

(3) Wait until the red light above the floppy disk drive A in the computer goes out and insert the first floppy diskette of the run and close the floppy diskette drive door.

(4) After the autosampler has been initialized, as indicated by an appropriate message on the

computer screen, press the "START" key on the autosampler control keypad and the probe will enter the WASH solution.

(5) Wait at least 5 minutes.

(6) Press the "START" key again and the probe will enter the urine sample cup containing deionized water.

(7) Wait at least 5 minutes.

(8) Press the "START" key again and the probe will move to the HOME position.

(9) Remove the startup and tuning rack from the autosampler.

(10) Set the five analysis racks into the autosampler.

(11) Load the racks with the first 70 samples of the analysis run as follows:

(a) Place the first sample in the forward left hand corner of the left most rack.

(b) Place the second sample behind it and continue placing samples in this way until the first column of seven sample locations is filled with vials.

(c) Repeat steps 11(a) and 11(b) for the next seven vials in the next column to the right and continue filling the racks in this way moving to the right of the autosampler until all 70 sample locations are filled with vials.

(12) Ensure that the caps have been removed from all of the sample vials.

(13) Hit any key on the computer keyboard two times.

(14) Wait about 30 seconds until the computer loads the controller of the autosampler. The message "ANALYSIS" will appear on the output display of the autosampler controller

keypad and the run will begin.

(15) Wait while the analysis is performed automatically.

(16) Replace the floppy diskette with another one when the message "COULD NOT WRITE THE FILE" appears on the computer screen and hit the "RETURN" key of the computer so that the analysis run can continue.

(17) After all the samples in the first autosampler tray (maximum 70 have been analyzed, replace the samples in the racks with the next samples to be analyzed by repeating steps (11) and (12).

(18) Queue a new procedure by performing steps (1) and (2).

(19) Repeat steps (17) and (18) until all the samples in the run have been analyzed.

D. Termination of Analysis Run

(1) Remove all sample racks from the tray of the autosampler.

(2) Switch the autosampler off, wait 10 seconds and then switch it on again.

(3) Set the startup and tuning rack back into the rightmost position of the autosampler tray.

(4) Remove the vials containing the tuning solution (TSO) and marker solution (MSO) from the rack so that only those containing the WASH solution and deionized water remain.

(5) Load file #7 into the autosampler control keypad by pressing the "EDIT" key followed by the "7" key and then the "ENTER" key twice.

(6) Press the "START" key and the probe will enter the vial containing the WASH solution.

(7) Wait about 5 minutes.

(8) Press the "START" key again and the probe will enter the deionized water.

(9) Hit the "Q" key followed by the "Y" key in order to obtain the ICPMS main driver program.

(10) Hit the F1 key labelled "INSTR CONTROL" to load the instrument control program.

(11) Hit the F1 key labelled "PUMPING STATE" and then answer the question on the screen by hitting the "Y" key.

(12) Disregard the message on the screen and hit any key to continue. The instrument will be turned off except for the plasma.

(13) Hit the "Q": key followed by the "Y" key and then repeat this step again in order to obtain the C:\> prompt on the computer screen.

(14) Type PARK <CR> and turn off the computer.

(15) Press the "START" key on the autosampler control keypad and the probe will return to the HOME position.

(16) Wait about 3 minutes.

(17) Turn off the peristaltic pump.

(18) Depress the switch marked "STANDBY" on the radio frequency generator in order to extinguish the plasma.

(19) Wait about 3 minutes until the front panel of the torch box cools to around room temperature.

(20) Switch off all the gases by depressing the buttons marked "NEB", "AUX" and "COOL" on the MS781 gas panel of the instrument.

(21) Release the peristaltic PVC tubes from the pump head.

(22) Dim the Tracor-Northern MCA in the instrument by turning down the button marked "INTENSITY".

(23) Turn off the autosampler.

(24) Turn off the surge protector below the computer.

(25) Close off the air supply by turning the black valve completely clockwise.

(26) Close off the argon by closing the two valves marked "Gas Supply and "Pressure Buildup" on the argon cylinder.

(27) Collect all diskettes and printouts and transfer them to the Data Processing Laboratory.

6.5 ANALYSIS WITH PERKIN ELMER 330 SPECTROPHOTOMETER

6.5.1 Solution Preparation and Silicon Analysis

Si-1 (Molybdate, direct)

Qty	Device	Manufacturer	Model	Calib. Req.
1	Spectrophotometer	Perkin Elmer	330	I
1	Balance	Mettler	AE100	II
3	Pipettes			
	200-100 μl	Labsystems	Digital	III
	5-10 ml	Oxford	Macro Set Transf.	III
	0.1-1 ml	Clay Adams	Selectapette	III

I: Calibrated with every analysis

II: Calibration accuracy: ± 1 mg
Calibration frequency: monthly

III: Calibration accuracy: ± 5%
Calibration frequency: monthly

Spectrophotometer Parameters

Cell: 1 cm x 1 cm x 4.5 cm (quartz)

Scan range: 840 nm - 620 nm

Peak wavelength: 812 nm

Base wavelength: 660 nm

Back correction range: 860 nm - 605 nm

Blank: DI water

Reagents

Silicon Standards: freshly prepare a set of silicon standards by dilution of an NBS traceable commercial standard in deionized water in order to produce one set of standards which covers the range of concentrations of Si to be used.

10% Ammonium molybdate: dissolve 2.5 g of Baker reagent grade ammonium molybdate in water, add 2.8 ml of concentrated reagent grade sulfuric acid dropwise while solution is stirring, dilute to 25 ml with water.

12.5% Tartaric acid: dissolve 3.125 g of reagent grade tartaric acid in water and dilute to 25 ml with water.

ANSA-bisulfite reducing agent:

Solution A: dissolve 3.125 g of reagent grade sodium bisulfite in 17.5 mL of water.

Solution B: dissolve 0.25 g of reagent grade anhydrous sodium sulfite in 5 mL of warm water and add 0.05 g of reagent grade 1-amino, 2-naphthol and 4-sulfonic acid (Eastman #360).

Mix solution A with solution B and dilute to 25 ml with water.

*Note: ANSA-bisulfite reagent should be stored in a cool dark place and prepared fresh every 2-4 weeks. All reagents should be prepared with deionized water using plasticware and stored in plastic bottles.

Procedure

(1) Prepare each sample by adding to a 25 ml plastic volumetric flask:

(a) recommended aliquot of sample (as per instructions of Laboratory Manager).

(b) Deionized water to neck of flask.

(c) 0.2 ml of 10% ammonium molybdate, shake well and wait 9-10 minutes.

(2) Add 1 ml of 12.5% tartaric acid, shake and wait 2 minutes.

(3) 0.2 ml of ANSA bisulfite reducing agent, dilute to 25 ml, shake well and wait 40 minutes.

(4) Perform the colorimetric measurement on the run of standards and samples as per procedure in section 6.5.2. Include at least one set of standards per run of samples determined.

(5) If concentrations of samples fall outside range of standards dilute the samples appropriately repeat the analysis.

6.5.2 PERKIN ELMER 330 SPECTROPHOTOMETER START-UP AND OPERATING PROCEDURE

1) Remove cover.

2) Remove pen cover.

3) Install appropriate cell holder.

4) Put in sample/reference compartment 2 identical cells filled with pure solvent.

5) Close sample compartment door completely.

6) Turn on line filter.

7) Turn power on. The instrument will calibrate itself for about 13 mins.

8) When the abscissa display stops at 500 nm and the recorder pen goes to 100% transmittance, turn off power to the D2 lamp immediately. Do not turn off D2 lamp if measuring under 350 nm.

9) Wait 10-20 minutes.

10) Press "ABS".

11) Press "SCALE" (e.g. 0-0.02).

12) Press "λ_1 - λ_2 SCAN" (according to element specifications).

13) Press "BACK CORR" (according to element specifications).

14) Align recorder pen with any vertical line on chart paper.

15) Press "λ_1 - λ_2 SCAN".

16) Press "SCAN".

17) Wait for cycle to be completed.

18) Remove cell from sample compartment, wash the cell with the sample to be analyzed and place sample in cell.

19) Place sample cell in sample compartment.

20) Close compartment door.

21) Press "WAVELENGTH MAX" and press "GOTO λ".

22) Input the appropriate values of the lower and upper scales to bring the peak into scale and press "SCALE".

23) Press "λ_1 - λ_2 SCAN".

24) Press "SCAN".

25) Analyze data as in the data processing section.

6.5.3 Procedure for Spectrophotometer Accuracy Verification

Equipment and Supplies

Qty	Device	Manufacturer	Model	Calib. Req.
1	Spectrophotometer	Perkin Elmer	330	I
1	Balance	Mettler	AE100	II
2	Pipettes 0.1-1 ml	Clay Adams	Selectapette	III
	5-10 ml	Oxford	Macro Set Transf.	III

Calibration Notes: I: Calibrated with every analysis; II: Calibration accuracy: ± 1 mg, Calibration frequency: monthly; III: Calibration accuracy: ± 5%, calibration frequency: monthly.

Spectrophotometer Parameters

Cell: 1 cm x 1 cm x 4.5 cm (quartz)

Scan range: 220 nm - 600 nm

Peak wavelengths: 257 nm, 350 nm

Base wavelengths: 235 nm, 313 nm

Back correction range: 200 nm - 620 nm

Blank: 0.001 n $HClO_4$

PROCEDURE FOR SPECTROPHOTOMETER ACCURACY VERIFICATION

Reagents

0.1 M $HClO_4$: in glacial acetic acid

1 g/l $K_2Cr_2O_7$: Dissolve 0.1 g of Potassium Dichromate of 99.9% purity or better (such as NBS Standard SRM136 or SRM935 Potassium Dichromate) in 100 ml of deionized water.

Procedure

Note: *This procedure should be performed monthly.*

(1) Prepare the following six standards in 100 ml flasks:

Standard #	0.1 M $HClO_4$ (ml)	1 g/l K_2/cr_2O_7 (ml)
1	1	0
2	1	2
3	1	4
4	1	6
5	1	8
6	1	10

(2) For each standard bring the final volume to the 100 ml mark on the flask neck with deionized water and shake thoroughly.

(3) Perform the colorimetric measurement as per procedure in section 6.5.2.

(4) Compare the measured absorbance values with those found in the NBS Special Publication Manual 260-54 Table 6 page 26 ("Standard Reference Materials" Certificaiton and Use of Acidic Potassium Dichromate Solutions as an Ultraviolet Absorbance Standard"). Log in results in the "Spectrophotometer Calibrations" L. N. and report to the Project Manager if the results fall outside a range of ± 5% of the NBS values.

7.0 Control of Samples and Release of Results

Acceptance of Samples and Release of Results

(1) Any sample entering the Analytical Laboratory Mass Spectrometry laboratory, in order to be analyzed, should be recorded in the Log-in/Log-out book. The recording is the sole responsibility of the originator of the sample. In addition, every sample should be accompanied by the standard "Request for Analysis" form, which provides adequate information about the sample (Format in procedure TPI-SH)

(2) After the completion of the analysis, any results in the form of printouts, compilations, tables, graphs, etc., as well as all of the remaining specimens (unless ordered by the originator to be discarded), should be transferred to the originator. The person receiving the results should acknowledge this in the Log-in/Log-out book. Any data remaining on the magnetic media should be transferred for further processing as explained below.

(3) Steps of Data Processing:

(a) Receive DCP disk from DCP operators.

(b) Transfer data files onto disk with data processing programs.

(c) Run processing programs to put data into files which are in a semi-matrix form (i.e. data printed in a chart).

(d) Data such as pH and Si Colorimetry should be taken from lab notebooks and entered manually.

(e) Print data in chart format.

(f) Give printouts to originator of samples.

(g) Destroy DCP files when processing is complete. Store data in chart format on data file diskettes.

8.0 DATA PROCESSING

This section includes a brief description and listings of computer programs used for data acquisition, data analysis, and data management.

1) Analytical Laboratory

A) Data Acquisition

The Analytical Laboratory uses a set of programs for recording and processing data from the DC Plasma Spectrophotometer. This set consists of a three part package. These three parts can be briefly described as follows: The first part records instrument readings, the second part performs any necessary calculations, and the last part prints out the results.

The first part of the package allows the operator to describe the conditions under which the instrument is being operated. The computer has only minimal control of the spectrophotometer and hence requires a fairly detailed description. After the description is entered, it will be recorded in a disk file. The first section of the program will next operate a carousel which presents samples to the instrument and record the readings made by the instrument on these samples into a disk file. When all samples have been measured control will be passed to the second part of the package.

The heart of the system is the second part of the package. The purpose of this part is

to use the instrument readings in the disk file to calculate a calibration curve for each sample and use this calibration to estimate a concentration for each sample. This calculation is based on fact that recalibration (or reslope) standards are mixed in with the samples on the carousel. Each sample is thus one of a small block of samples (typically three or four) surrounded by reslope standards. The instrument readings from the standards on either side of the block are used to construct the curve for that block of samples. The instrument readings on a sample and the calibration curve for that sample's block are used to calculate that sample's concentration. In the final stage of the calculation, the dilution factor for that sample is used to convert the measured sample concentration into the actual concentration of the sample before dilution. All of these results including standard deviations are placed in a final disk file. These final disk files can be used by subsequent programs if desired. When the final data file has been written the second part will pass control over to the third part.

The third part of the program set will read the final disk file and print a report of the sample concentrations with standard deviations. It will also make some elementary checks of the results and flag some conditions known to introduce errors in the analysis. Notable among these conditions is the case where the solution analyzed has a concentration higher than the high standard for the element.

Each of these packages was developed in order to ensure that all measurements and calculations in the analysis lab were made in a defined way. The automation of the calculations is particularly valuable in that errors of transcription and methodology are minimized.

The following programs are currently being used:

DCPDATA: Controls DCP equipment and calculates the averages of the concentrations.

REPRINT: This progrm reprints results of DCP.

OXIDE: This program calculates the percentage of oxides from PPM amounts.

RERUN: This program recalculates results of DCP test from data files on disk.

B) Data Analysis and Data Management

Various software manipulations must be performed in order to properly store the information gathered through the data acquisition programs on permanent storage disks and obtain data outputs in the desired format. Raw data from the DCP represent element concentrations and standard deviations expressed in ppm.

(1) A weekly schedule originating in the leaching laboratory and containing sampling information is sent to the Analytical Laboratory along with analysis request forms. This schedule is returned to the Data Processing Department along with a floppy disk containing raw data files from the DCP as well as results from the DIONEX if applicable. Similar tests are run together and given a reference name which is specified in the schedule sheet.

(2) Using the Reference name, raw data files are copied onto a scratch pod disk

(SPD). Names prefixed with a "D" denote files from the DCP. The suffix "D" and others with the exception of C, L, M and N indicate dilution files for DCP.

(3) The raw data transferred onto the scratch pod disk must be properly channeled into the permanent storage data disks and also used to produce printouts of results in the desired format. Two sets of programs are used for this purpose depending on whether samples from a single test or multiple test were run in the DCP.

To process the raw data, the data disk must have files containing all the initial pertinent information about a test run. These files are created using INDEX.

(4) The following programs are used for data management:

FASTDATA: This program combines DCP files into a single file that shows the concentration for each element and sample for a given test trial.

Leach test data processing programs are described in the technical procedure TPI-LT.

(2) General Chemistry Laboratory

Colorimetric Analysis:

Analytical methods for Colorimetric Analysis of Si, Fe and U have been in use in our laboratories for a considerable length of time. Detailed procedures for these tests are included in Section 6.5. Colorimetric data obtained from the Perkin Elmer Model 3 spectrophotometer

is translated into concentration data using a computer program called BLUEJ. This program uses absorption data of standards to calculate a calibration curve for the element being analyzed. The absorption data from the sample is used with this calibration curve to calculate concentration. The result is given in ppb.

9.0 ASSURANCE OF RELIABILITY OF ANALYTICAL RESULTS

Assurance of Reliability of Analytical Results

(1) The entire personnel of the Analytical Laboratory is required to be familiar with and to obey strictly all the individually relevant procedures included within the "Analytical Laboratory Procedures Manual".

(2) The final decision about acceptability of the results produced by the Analytical Laboratory is always the sole and exclusive responsibility of the originator of the sample.

(3) Internal acceptability criteria of the results produced by the Analytical Laboratory are as follows:

- (a) for single point measurements: originator's decision
- (b) for single-train measurements: originator's decision
- (c) for replicate-train measurements: scattering of average for every train not exceeding 3 standard deviations ('3σ' test).

(4) The analysis should be repeated in each one of the following cases:

(a) if so requested by the originator

(b) if a discrepancy of more than ± 50% is found between the actual and the expected (supplied by the originator) content of a certain analyte in the sample.

(c) if the results do not meet internal acceptability criteria (para. 3 above).

(d) if any form of analytical instrumentation malfunction is discovered. In this case immediate corrective action should be undertaken and internal investigation performed in order to evaluate to what extent and for how long such a malfunction has affected (if at all) the former analyses. If the discovery of a fault in the analytical system places in doubt results obtained and reported prior to such discovery, the originator(s) of the samples in question should be immediately notified.

10.0 RECORD KEEPING

The following record books should be kept and maintained in the Analytical Laboratory:

(1) Log-in/Log-out book to record sample admittance and release of results from the laboratory.

This book should consist of the following headings:

(a) Log-in Date - the date the sample was given to the Analytical Laboratory

(b) File Name - sample designation

(c) No. of Samples

(d) Originator

(e) Run Name - given if samples of different designations are analyzed within the same batch (run)

(f) Log-out Date - the day the results were received by the originator

(g) Person Receiving the Results - an originator or a person acting on his behalf

(2) Maintenance/Troubleshooting book for DCP Spectrophotometer.

This book should contain information on daily maintenance /troubleshooting or corrective action performed.

(3) Dionex IC book

This book, in addition to the maintenance/troubleshooting information (as described in paragraph 2 above), should contain the following records:

(a) Calibration chromatograms and curves

(b) Summary of analytical results (the table showing sample designation along with the concentrations of the analyzed anions)

(4) Maintenance/Troubleshooting Book for ICP-MS

This book should contain information on daily maintenance/troubleshooting or corrective

action performed.

(5) Calibration of Pipettes

All records of calibration data for pipettes, percentage of error detected should be maintained.

(6) Colorimetry Data

SiO_2 colorimetry data, verification of spectrophotometer accuracy and the spectral records obtained should be documented in addition to maintenance/troubleshooting log book of spectrophotometer.

(7) Other Records

Any round robin tests performed should be documented in a record book. Any general analysis performed in addition to above instrumental analysis should also be recorded in a separate book.

All the above laboratory record books are subjected to periodic surveillances as part of the Quality Assurance Program.

UNCONTROLLED

VSL Technical Procedure

Title: Leach Test Procedures

Document: TPI-LT Rev # 1

Issued: 10/91 Rev #0
Revised: 9/92 Rev #1

Documents Incorporated:

TPI-02.WVN	Rev #0
TPI-LT.WVN	Rev #4
TPI-DP.WVN	Rev #1

Date Issued: 10/91
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10.0 Record Keeping

DOCUMENT REPLACEMENT RECORD
(Replaced by Rev #0 of 10/91 and this manual)
Leaching Tests

Procedure	Description	Rev #	Replaced in this Manual by:
VSL-LT-1.WVN	Ground Sample Preparation	4	Section 6.1.1
VSL-LT-2.WVN	Monolithic Sample Preparation	2	Section 6.1.2
VSL-LT-3.WVN	Procedure for Cleaning Monolithic Samples	1	Section 6.3.1
VSL-LT-4.WVN	Cleaning Procedure for Teflon and Stainless Steel Parts	4	Section 6.3.2
VSL-LT-5.WVN	Procedure for Pulsed Flow Tests	4	Section 6.4.1
VSL-LT-6.WVN	Procedure for IAEA (High Dilution) Tests	4	Section 6.4.2
VSL-LT-7.WVN	Procedure for Cleaning Ross-Type pH Electrodes	4	Section 6.3.3
VSL-LT-8.WVN	Procedure for MCC-3 Tests	5	Dropped
VSL-LT-9.WVN	Procedure for MCC-1 Tests	3	Dropped
VSL-LT-10.WVN	Balance Calibration Check Procedure	5	Section 6.5.1
VSL-LT-11.WVN	Pipette Calibration Check Procedure	5	Section 6.5.2
VSL-LT-12.WVN	pH Meter Calibration Procedure	5	Section 6.5.3
VSL-LT-13.WVN	Procedure for Preparation of Deionized (DI) Water	5	Section 6.2.1
VSL-LT-14.WVN	Procedure for Preparation of Reference Grande Ronde Water (Synthetic Ground	NEW	Section 6.2.2

	Water)		
VSL-LT-15.WVN	Procedure for Preparation of EJ-13 Water	NEW	Section 6.2.3
VSL-LT-17.WVN	Procedure for Calibration of Oven Thermometers	NEW	Section 6.5.4
VSL-DP.3.WVN	Procedure for Running Pulsed Flow Data Analysis Programs	NEW	Section 8.1
VSL-DP-5.WVN	Procedure for Running IAEA Data Analysis Programs	NEW	Section 8.2

1.0 PURPOSE AND OBJECTIVES

These procedures describe methods, materials, equipment and special conditions required to perform leach testing on glasses and other solids. These procedures provide the means for obtaining verifiable, reproducible, documented data from tests and experiments.

The objectives of the leach tests are for: 1) evaluating the chemical durability of glasses; 2) evaluating the radionuclide release properties of nuclear waste glasses; and 3) evaluating whether the glass durability or radionuclide release properties have been consistently controlled during long-term production. Accordingly, its applicability is widespread throughout manufacturing, research, and development. These tests can be used for specification acceptance.

These test methods may be applicable to the evaluation of the chemical durability of glasses and the radionuclide release properties of nuclear waste glasses in natural environments.

2.0 APPLICABILITY AND CHARACTERISTICS TO BE TESTED

These procedures apply to all employees trained and authorized to perform leach testing on glasses and other solids in the VSL. Specific activities covered by these procedures include: (a) Ordering, assembly and cleaning of leach containers; (b) calibration, standardization and monitoring of environmental chambers, balances, volume measurement devices (pipettes), spectrophotometers and pH meters; (c) selection and ordering of chemicals to assure a

sufficiently high grade of purity; (d) Operation, maintenance and testing of water purification system; (e) preparation, storage, pH testing and sampling of leachants (including groundwater); (f) preparation and handling of test specimens, including crushing, sieving, washing, weighing and loading into the leach container in the cases of non-radioactive powdered samples only, and washing, measuring the dimensions, weighing and loading into the leach container in the cases of non-radioactive as well as radioactive monolithic specimens; (g) placing the necessary volume of leachant in the leach container, closing and weighing the leach container, and placing it in an environmental chamber; (h) removing all or part of the leachate at specified intervals and replacing it with fresh leachant; (i) measuring the pH of the leachate; (j) diluting the leachate whenever required with water or acid; (k) sending the leachate for chemical analysis, with a filled Request for Analysis form, for analysis in the analytical chemistry facility and/or the ICP-MS laboratory; (l) checking and compilation of analytical results; (m) documenting all operations in laboratory notebooks.

These procedures will be implemented by personnel from the VSL.

3.0 SAFETY

3.1 HAZARDS

The definition of a hazardous chemical is given in Section 8.3 of the CUA Chemical Materials Safety Manual, adopted from the OSHA Hazard Communication Standard. The CUA Chemical Materials Safety Manual is hereafter referred to as the CCMSM. This Standard and

its application to CUA facilities are described in Sections 8.1 of the CCMSM, with the exceptions noted in Section 8.28.

3.2 HAZARD MITIGATION

3.2.1 Identification of Hazards

The identification of chemical hazards should include an education program (Section 8.2 of the CCMSM). This program should include familiarization with the CCMSM as a whole, including the toxic properties of chemicals according to Sections 8.22, 8.23, 8.24 and 8.25 of the CCMSM, other hazards of chemicals classified under Sections 8.9, 8.10, 8.11, 8.12, 8.13 and 8.14 of the CCMSM, chemical handling and storage procedures (see paragraph 3.2.2 below), and emergencies (see paragraph 3.2.3 below). The education program will also include familiarization with the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University.

Other elements of hazard identification include the proper use of labels (Section 8.4 of the CCMSM) and of Material Safety Data Sheets (Sections 8.5, 8.26 and 8.27 of the CCMSM). Workplace chemical lists should be compiled, maintained, and made available for employees in accordance with Section 8.31. In addition, a copy of the CCMSM should be kept in the laboratory and its use should be included in the education program cited above. The same should apply to the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University.

3.2.2 Handling and Storage of Chemicals

Handling of chemicals should be conducted according to Section 8.6 of the CCMSM. Storage of chemicals should follow Section 8.8 of the CCMSM. Special precautions should be taken in handling and storage of incompatible chemicals, solvents, compressed gases, and radioactive materials according to Sections 8.10, 8.12, 8.13, and 8.14 of the CCMSM, respectively. Unstable chemicals according to Section 8.9 should be excluded from the leach testing laboratory except in cases where the introduction of such a material is required by the project manager. Such a requirement should be made in writing and specify the maximum quantity of the material and the maximum period it may be stored before being discarded. Shock-sensitive chemicals according to Section 8.11 should never be introduced into the laboratory under any circumstances. No work requiring the use of restricted areas according to Section 8.15 should be performed within the leach testing laboratory.

Waste disposal of hazardous chemicals should follow procedures established by the CUA Environmental safety Office according to Section 8.16 of the CCMSM.

3.2.3 Emergencies

Chemical spills should be dealt with according to Section 8.17 of the CCMSM. Fire and fire-related emergencies should be controlled according to Section 8.21 of the CCMSM. Cases of personal contamination should be treated according to Section 8.19 of the CCMSM, cases of injury and illness according to Section 8.18, and cases requiring minor first aid according to

Section 8.20.

3.3 PERSONNEL PROTECTION

Personnel protection should consist of two elements. The first is familiarization and compliance with the instructions of the CUA Chemical Materials Safety Manual (CCMSM), including identification of hazards (see paragraph 3.2.1 above), handling and storage of chemicals (see paragraph 3.2.2 above), and dealing with emergencies (see paragraph 3.2.3 above). The second element is familiarization and compliance with the CUA Chemical Hygiene Program (CCHP) when the document describing this Program is finalized and approved for distribution by the University. The overall chemical hygiene program (Section 8.34 of the CCHP), based on the OSHA Regulation on Occupational Exposure to Hazardous Chemicals in Laboratories, should be followed. In detail, exposure reduction measures (Section 8.35 of the CCHP) will include environmental monitoring (8.35.1), inspections, maintenance and housekeeping (8.35.2), use of protective apparel and equipment (8.35.3), posting of signs and labels (8.35.4), and dealing with accidents and spills (8.35.5). Ventilation of both general laboratory spaces and of special areas and local devices such as chemical fume hoods and glove boxes should follow the instructions of Section 8.36 of the CCHP. Employees should take part in the safety information and training program which exists in the University according to Section 8.37 of the CCHP. The leaching laboratory should not conduct operations with hazardous substances which require a special review of laboratory activities under Section 8.38

of the CCHP, viz. reproductive toxins, chemicals which have a high degree of acute toxicity, and chemicals considered select carcinogens. Employees should be informed about the medical program provided by the University in cases of work with hazardous substances according to Section 8.39 of the CCHP. Responsibility for chemical hygiene rests with all employees. Specific chemical hygiene responsibilities as applied to the leach testing laboratory are specified in Section 8.40 of the CCHP. Leach testing laboratory workers should be familiar with the basic rules and procedures for working with chemicals included in Section 8.41 of the CCHP and should work in full compliance with these rules and procedures. No work with chemicals of high chronic toxicity according to Section 8.41.4 of the CCHP should be carried out in the leach testing laboratory. Animal work, whether involving chemicals of high chronic toxicity according to Section 8.41.5 of the CCHP or not involving chemicals of this type, should not be carried out in the leach testing laboratory.

4.0 REQUIREMENTS

4.1 TRAINING

All leach testing laboratory personnel are trained in the understanding and use of the technical methods that they will use by the laboratory supervisor or by a trained and experienced person designated by the laboratory supervisor. Upon completion of training for a method, a technician is qualified to use that method independent of direct supervision.

4.2 DEFINITIONS

Leachant - The solution that is being used, or is intended for use in leaching.

Leachate - The solution resulting from a leach test.

ASTM Type I water - Purified water with a maximum total matter content of 0.1 mg/L, and electrical conductivity of 0.06 μmho/cm at 25°C, a minimum electrical resistivity of 16.67 MΩ*cm at 25°C, and no detectable soluble silica (consult ASTM D 1193).

Open system tests - A system is open if during the process under consideration, transfer of matter either into or out of the system takes place, e.g. O_2 and/or CO_2 diffusion into or out of the leach vessel.

Closed system tests - A system is closed if during the process under consideration no transfer of matter either into or out of the system takes place.

Set of samples - Those which have been tested simultaneously in the same oven.

Sample blank - A cleaned test vessel which has been filled with the same amount of leachant as the sample vessels but contains not glass sample.

Pipettes - Calibrated and precleaned pipette tips.

Syringes and Syringe Filters - Precleaned syringes and 0.45 μM syringe filters (for example Gelman #4497).

Sample Vials - Presterilized sample vials and caps.

pH Meter - pH meter with an accuracy of $\pm$ 0.2 pH units.

Milli-Q Water Purification System - Water purification system for producing ASTM-Type I

water.

Ultrasonic Cleaner - Size to be compatible with cleaning of several leach vessels simultaneously.

High Pressure Air or Freon Pressurized Canister - Pressure to be sufficient to dry sieves after washing.

Ion Selective Electrode - Ion selective electrode for F determination.

CALIBRATIONS

Calibrations - Initially calibrate all instruments used in this test. Perform periodic calibrations during use of the instrument to minimize possible errors due to instrumental drift.

Calibration and Standardization Schedule:

Thermister or thermocouple - Calibrate annually with NIST Standards or ice/boiling water.

Balance - Have the balance calibrated on a monthly basis.

pH meter - Initially and after with commercial buffer solutions that bracket the solution pH being measured.

4.3 Reagents and Standards

Reference Glass - A reference glass of choice, similar in composition to the glass being tested, is to be run in triplicate along with each batch of glasses tested. The reference glass should be traceable to NIST, or a comparable equivalent used.

Multi-element Solution Standard - A reference solution of choice, containing the major

elements of the composition of the leachate being tested, is to be submitted along with each batch of leachates for multi-element analysis. The reference solution standard should be traceable to NIST, or a comparable equivalent used and a certified shelf life.

pH Buffers - Commercial pH buffers to bracket the measured pH range of the leachant and leachate.

Ion Standard Solution - The reference solution standard should be traceable to NIST, or a comparable equivalent used.

Reagent Grade Acids - Reagent grad HNO_3 and HF for cleaning of leach vessels.

High Purity Acid - Ultrex high purity concentrated HNO_3 for acidification of leachates.

Solvents - Ethyl Alcohol - 95% pure, reagent grade acetone.

ASTM Type I Water - Type I water shall have a minimal electrical resistivity of 16.67 M Ohm cm at 25°C and a maximum of 20.0 M Ohm cm at 25°C (consult ASTM D 1193).

The source water shall be passed through Milli-RO15 System which contains 6 cartridges (Ful-Flow, Teel, Lifegard, Rogard Prefilter and Milli-Ro-20 Spiral-Wound Reverse Osmosis) and Milli-Q System that consists of five cartridges (Lifegard, Super-C, two of Ion-Exchanger, and Millipore DI-12). Pass the purified water through an in-line conductivity cell to verify its purity.

CHOICE OF LEACH VESSEL

Stainless Steel Vessels - Stainless steel vessels made of 304L are to be used in all tests.

PREPARATION OF VESSELS

Identification of Vessels - A unique identifying number should be permanently etched on each vessel.

Identification of Vessel Cleaning History - Each batch of cleaned leach vessels will be labeled with a unique batch number. A log book of the leach vessel number and date the cleaning is completed shall be kept.

Cleaning of New Stainless Steel Vessels - New 304L stainless steel vessels shall be cleaned by the following procedure:

Clean the vessels and lids (without the gaskets) ultrasonically in reagent grade acetone for 5 minutes. Clean the vessels and lids ultrasonically in 95% ethanol for 5 minutes. Rinse thoroughly with DI water. Let stand in a nitric acid solution overnight in a 90°C oven. The nitric acid solution is made by dissolving 1 gram of concentrated nitric acid (about 70% by weight, Analytical Reagent Grade) in 180 ml of de-ionized water or by diluting 4 ml of concentrated nitric acid to 1 liter with de-ionized water.

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6.0 PROCEDURE

6.1 SAMPLE PREPARATION

6.1.1 GROUND SAMPLE PREPARATION

Qty	Device	Manufacturer	Model #	Calib. Req.
1	Grinder	Retsch	ZM-1	N.A.
1	Sieves			I
1	Mortar & Pestle			N.A.
1	Balance	Mettler	AE100	II
1	Sieve shaker	Tyler		N.A.

Calibration codes: I: See TPI-GP, Section 5.2.2. Calibration to be performed once. II: Calibration accuracy: ± 1 mg. Calibration frequency: monthly.

Procedure

A. Non-radioactive samples

(1) Break glass sample received from the glass preparation group or the Project Manager into smaller pieces which will fit into the grinder.

(2) Grind the sample using a Retsch ZM-1 grinder.

(3) Fractionate the ground sample according to particle size -40 +60 mesh, <60 mesh, <325 mesh - using a stack of U.S. Standard Brass sieves which are placed

in a Tyler Portable Sieve Shaker.

(4) Store the powders in a plastic vial and identify each vial.

B. Radioactive samples (Alternative procedure: Section 6.9 in TPI-GP)

Note: Work must be performed inside designated glove box in Room 39 following procedures to handle radioactive materials as per instructions of CUA's RSO.

(1) Obtain bar sample from Glass Preparation group.

(2) Place bar into a plastic bag and break up into small pieces using a hammer.

(3) Grind glass pieces with a mortar and pestle.

(4) Fractionate the ground sample according to particle size - 40 + 60 mesh, < 60 mesh, < 325 mesh - using a set of U.S. Standard Brass sieves.

(5) Store the powders in plastic vials and identify each vial with a radioactive label. Keep loaded vials in Room 39.

C. Washing Procedure for Ground Sample

(a) Place approximately 15-20 grams of sieved glass in a clean 50 mL glass beaker. Do not exceed the 20 mL mark on the beaker.

(b) Forcibly add 25-30 mL of ASTM Type I water from a squirt bottle to the glass. During this addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass.

(c) Allow the glass-water mixture to settle 15 seconds then decant off the water.

(d) Repeat steps (b) and (c).

(e) Repeat step (b).

(f) Prepare the ultrasonic cleaner by filling with water to 0.5". Place the beaker from step 11.6.5 in the ultrasonic cleaner for 2 minutes. After removing the beaker form the cleaner, decant the water from the beaker and discard.

(g) Repeat (f).

(h) Forcibly add 25-30 mL of absolute alcohol from a squirt bottle to the glass. During this addition, the squirt bottle should be moved in a circular motion so that the wash stream agitates all the glass.

(i) Place the beaker from step (h) in the ultrasonic cleaner for 2 minutes. After the 2 minutes decant in the alcohol from the beaker.

(j) Repeat step (i) two more times.

(k) Put the beaker full of cleaned glass in a 90°C oven overnight to dry. Glasses can be stored in a clean, sealed and labeled container until they are to be used. Before use the crushed glass must be redried at 90°C overnight so that the powders do not contain absorbed water when weighed.

(l) Enter sample identification, date, and name of the person performing the sample washing on a sample washing log book.

6.1.2 Monolithic Sample Preparation

Qty	Device	Manufacturer	Model #	Calib. Req.
1	Faceting Machine	Graves	Mark IV	N.A.
1	Cutting Saw	Graves		N.A.
	Cutting Blades		Red Blazer	N.A.
	Cutting Blades	Felker	D200-L50-MBI/8	N.A.
	Diamond Laps	Various		N.A.
1	Hot plate			N.A.
	Mounting wax			N.A.
	Acetone			N.A.

Procedure

A. Blocks

(1) Prepare a flat surface on bulk glass using a 360 grit diamond polishing lap. This provides adequate stability for cutting. This step is not necessary if bars are available.

(2) Shape a parallel piped out of bulk sample using a diamond saw blade (Red Blazer) and smooth surface with a 360 grit diamond polishing lap.

(3) Using a diamond saw blade cut out blocks with dimensions about 15% - 20% greater then those required to give a surface area of 400 mm^2, (e.g. 9.5mm x 9.5mm x 9.5mm).

(4) Smooth out all surfaces using a 600 grit diamond polishing lap mounted on a MARK IV faceting machine, taking care that the end dimensions give a total surface area of $400 \pm 40 mm^2$. Parallelicity is insured by proper use of the faceting machine.

(5) Clean samples with acetone.

(6) Store sample in an envelope or vial identifying it with all pertinent information, i.e., source, sample I.D., dimensions and surface area. Include deviations if calculated.

(7) Document in the appropriate L.N. the preparation of the sample including deviations from this procedure. Record all pertinent information about the sample, i.e. source, sample I.D., dimensions and surface area, and the laboratory to which it was delivered and the person who received it.

B. Slides

(1) Prepare a flat surface on bulk glass using a 360 grit diamond polishing lap.

(2) Using a diamond saw blade prepare a surface perpendicular to the first one.

(3) Using mounting wax, attach the sample to the cutting jig and set stop such that slices ~1 mm thick are produced.

(4) Mount 200 grit diamond cutting blade (Felker D200-L50-MBI/8) on saw.

(5) Mount jig with sample on cutting table and carefully cut slides insuring that feed is slow (~0.3mm/sec.) and smooth.

(6) Using mounting wax, attach individual slices onto a microscope slide.

(7) Using the 200 grit diamond saw blade, cut out samples from the slices such that their total surface area is $400 \pm 40 mm^2$ (e.g. 17mm x 10mm x 1mm).

(8) Detach samples from glass slide by heating up gently, and clean with acetone.

(9) Store sample in an envelope identifying it with all pertinent information, i.e., source, sample I.D., dimensions and surface area. Include deviations if calculated.

(10) Document in the appropriate L.N. the preparation of the sample including deviations from this procedure. Record all pertinent information about the sample, i.e. source, sample I.D., dimensions and surface area, and the laboratory to which it was delivered and the person who received it.

6.2 LEACHANT PREPARATION

6.2.1 Procedure for Preparation of Deionized (DI) Water

Equipment and Supplies

Qty	Device	Manufacturer	Model	Calib. Req.
1	Milli-RO15 System - Containing the following cartridges	Millipore		
	1 Ful-Flow		RWS-5	I
	1 Teel			II
	1 Lifegard		CP20-010-03	III
	1 Rogard Prefilter		CDPR-012-F4	IV
	1 Milli-RO-20 Spiral Wound Reverse Osmosis Cartridge		CDRO-025-SI	V
1	Milli-Q System Containing the following cartridges	Millipore		
	1 Lifegard Prefilter		CP15-010-03	VI
	1 Super-C		CDFC-012-04	VII
	2 Ion-Ex		CDMB-012-02	VIII
	1 Millipore DI-12		CWDI-012-03	IX
1	Conductivity meter	Amber Science		X
	Polyethylene containers			N/A

Calibration notes:

I: Replace when the whole column turns from white to yellow (usually about every two weeks); II: Replace when the Ful-Flow cartridge is replaced; III: Replace when the pressure gauge reads less than 60 psi; IV: Replace when the pressure gauge reads more than 10 psi less than the pressure gauge of the RO cartridge; V: Replace once a year or when the percentage of divalent cations (Mg^{2+}, Ca^{2+}) removed falls below 80% and the percentage of monovalent cations (Na^{+}, K^{+}) removed falls below 65%; VI: Replace when the pressure differential between the first and second cartridge housings is greater than 10 psi; VII: Replace when the permanganate test given in section A.3 fails; VIII: Replace when the resistivity of the Meg-O-Meter falls below 16 M Ω cm; IX: Replace every 3-6 months when on output flow from Milli-Q system drops below 0.5 lit/min; ***(NOTE: All cartridge replacements must be recorded both in "Millipore Records" LN and on labels on the cartridge housings)***X: Self check prior to each use.

A. Specifications for Deionized Water

The definition of deionized water is that it must pass the following specifications.

(1) The resistivity of the water should be greater than 16 MΩ cm. This should be checked at least once a day and recorded on the sign-up sheet in Room 332.

(2) The concentration of silicon in the water should be not greater than 10 parts per billion. This should be checked by carrying out the procedure for silicon analysis (See Analytical Procedures Manual) once a month and the results should be recorded in the "Si Colorimetric Analysis" LN.

(3) The minimum color retention time of potassium permanganate should be 60 minutes. This test should be performed once a month according to the procedure given below.

(a) Prepare a potassium permanganate solution by dissolving 0.0316 g of $KMnO_4$ in 100 ml of water.

(b) Determine the consumption of potassium permanganate by adding 0.20 ml of $KMnO_4$ solution to a mixture of 500 ml of the reagent water and 1 ml of H_2SO_4 in a stoppered bottle. Consider the reagent water as having passed the test if the permanganate color does not disappear completely after standing for one hour at room temperature and record this information in the "Millipore Records" Laboratory Notebook.

B. Operation of the Millipore Water System

(1) Check the water level in the storage reservoir:

(a) If the water level in the tygon tube is lower than the mark on the tube, wait until the water level passes above the mark and then proceed to step 2.

(b) If the water level in the tygon tube is higher than the mark proceed to step 2.

(2) Turn on the pump.

(3) Wait for approximately 5-10 minutes.

(4) Check the resistivity on the resistivity meter:

(a) If the reading is greater than 16 MΩ cm go to step 5.

(b) If the reading is less than 16 M cm consult project manager concerning replacement of cartridges.

(5) Open the final valve and let 1-2 litres of water flow to be sure the line is clean.

(6) Rinse a clean conventional polyethylene container with a small quantity of fresh deionized water.

(7) Collect the required volume of deionized water.

(8) Close the valve and turn off the pump.

(9) Record the volume of water removed on the sign-up sheet in Room 332.

(10) Measure the conductivity of the water using the Amber Science conductivity meter which should be less than 2.5 μ mho cm^{-1}.

(11) Label the container clearly indicating the conductivity of the water and the date on which it was filled.

(12) Remove all unused deionized water after two weeks.

C. Additional Checks on the System

(1) Water from the Milli-RO system.

(a) Determine the concentration of Si in the water every month according to the procedure for silicon analysis (See Analytical Procedures) and record the results in the "Si Colorimetric Analysis" Notebook. If the concentration of silicon is greater than 100 ppb consult the Laboratory Supervisor or Project Manager.

(b) Submit a sample of the water for analysis using the DC Plasma method (see Analytical Procedures) every month and record the results in the "Millipore Records" Notebook.

(2) Deionized Water from the Milli-Q System

(a) Determine the concentration of Si in the water after consultation with the Project Manager on each occasion when important experiments are begun in which very low concentration levels of Si are important according to the procedure for silicon analysis. If the concentration of silicon is greater than 10 ppb consult the Laboratory Supervisor or Project Manager.

(b) Submit a sample of DI water for analysis and DC Plasma method (see Analytical Procedures) after each replacement of the ion exchange cartridge and record the results in the "Millipore Records" Laboratory. Notebook. If discrepancies of greater than 1 ppb

are found for the concentration of silicon from the Si analysis and DC Plasma analytical methods consult the Project Manager.

6.2.2 Procedure for Preparation of Ref. Grande Ronde

Equipment and Supplies

Qty	Device	Manufacturer	Model #	Calib. Req.
	Polyethylene Containers	Nalgene		N.A.
	Volumetric Flasks	Nalgene		N.A.
1	pH Meter	Fisher	Acumet-810 or 910	I

Calibration Notes:
I: Calibration Accuracy: ± 0.05 pH units; Calibration Frequency: Daily or every 25 measurements.

Chemicals

Na_2CO_3 (Sodium carbonate) dried at 120° for two hours and stored in a desiccator

$Na_2SiO_3 \cdot 9H_2O$ Reagent grade sodium silicate

NaOH 50 wt% solution of reagent grade sodium hydroxide

KCl Reagent grade sodium sulfate dried at 120°C for one hour and stored in a desiccator

Na_2SO_4	Reagent grade sodium sulfate dried at 80°C for one hour and stored in a desiccator
NaF	Reagent grade sodium fluoride dried at 80°C for one hour and stored in a desiccator
HCl	2.00 M Hydrochloric acid
$CaCl_2 \cdot 2H_2O$	Calcium chloride
$MgCl_2 \cdot 6H_2O$	Magnesium chloride

Procedure

(1) Prepare stock solution "A" as follows:

(a) Add 750ml of DI water to a 1 litre volumetric flask.

(b) Add the following chemicals to the above flask as per the quantities given below and stir until they are all dissolved.

Chemical	Quantity (g)/lit
$Na_2SiO_3 \cdot 9H_2O$	14.40
Na_2CO_3	3.80
NaF	2.95
Na_2SO_4	10.2

NaOH (50 wt %) 18.8

(c) Add DI water until the total volume equals exactly 1 litre.

(2) Prepare stock solution "B" as follows:

(a) Add 750 mls of DI water to a 1 litre volumetric flask.

(b) Add the following chemicals to the flask and stir until they are all dissolved.

Chemical	Quantity (g)/lit
KCl	0.262
$CaCl_2 \cdot 2H_2O$	0.4043
$MgCl_2 \cdot 6H_2O$	0.0107
HCl	171.6 ml of 2.00N

(c) Add DI water until the total volume equals exactly 1 litre.

(3) Take 25ml aliquots of Stock Solution A, and Stock Solution B and add to 900ml of DI water.

(4) Measure the pH of the solution.

(5) Bring the above solution to a pH of 9.75 ± 0.1 at room temperature by adding the appropriate quantities of either 0.1N HCl or 0.1N NaOH.

(6) Dilute this solution with DI water to exactly one litre.

(7) Measure the final pH of the solution and transfer a sample to the Analytical Lab for analysis.

(8) Store the remaining solution in a tightly capped polyethylene container.

6.2.3 Procedure for Preparation of EJ-13

A. Removal of Soluble Salts from Crushed Rock

(1) Place 50 g of crushed rock in a 1 litre Teflon bottle.

(2) Add 500 mls of DI water.

(3) Close the bottle and shake vigorously for 2 minutes.

(4) Place the closed bottle upright on a flat surface and allow to stand for at least one hour.

(5) Decant the liquid, taking a sample for anion analysis.

(6) Repeat steps (2) through (5) 3 more times.

(7) Transfer the four solution samples to the Analytical laboratory for analysis for chloride, nitrate and sulfate anions.

(8) If the last rinse solution shows less than 0.5 mg/L of each of chloride, nitrate, and sulfate it is to be concluded that the soluble salts have been removed. If the

Equipment and Supplies

Qty	Device	Manufacturer	Model	Calib. Req.
	Teflon bottles	Savillex		N.A.
1	Oven	Blue-M	Stabil-Therm OV-490A, OV-490A-3	I
1	Dark Storage bottle	Nalgene		N.A.
1	pH meter	Fisher	Acumet 810 or 910	II
1	Filter Holder, 47mm	Millipore		N.A.
1	Filter	Millipore/Nucleopore	0.1 μm	N.A.
	Crushed rock	Topopah Springs	<100 mesh	
	Tuff from Nevada Test site			
	J-13 Well water	from Nevada Test site		
	Sulfuric acid	Acs-Reagent Grade		

Calibration notes:

I: Temperature monitored daily with thermometers that are positioned as recommended by the manufacturer and meet NBS specification, Monograph 150; II: Calibration accuracy: ± 0.05 pH units, Calibration frequency: daily (see section 6.5.3).

last rinse solution shows more than 0.5 mg/L, repeat steps (2) through (5) and (7) above until a level of less than 0.5 mg/L is achieved.

TPI-LT

B. Preparation of Equilibrated J-13 Water (EJ-13 water)

(1) To the wet crushed rock (from which the soluble salts have been removed) contained in a 1 litre teflon bottle add J-13 well water until the bottle is about 90% full and mark the level.

(2) Cap the bottle tightly and place it in an oven preheated to 90°C.

(3) Allow the bottle to remain in the oven for about 3 weeks. Shake the bottle for about 1 minute twice a day for the first week, and once a day thereafter.

(4) After three weeks remove the bottle from the oven and allow it to cool for one hour in air.

(5) Filter the solution through a 0.1 micron Millipore or Nucleopore filter using the Millipore filter holder.

(6) Measure the pH of the final solution and store it in a plastic bottle. (Beware: Do not use transparent bottles since algae may grow in the liquid if it is stored in sun light). Transfer a portion of the final solution to the Analytical lab for analysis.

Note: i) The equilibrated liquid should be stable for several months; ii) If the procedure for preparing EJ-13 water had reached step (4) above and there is no immediate use for it, leave the teflon bottle in 90°C oven and when it is needed carry-out the following procedures:

(a) Remove the bottle from the oven and allow it to cool for 1 hour in air.

(b) If less than about 5% of the water has evaporated continue with step (5) above.

(c) If more than about 5% of the water has evaporated add DI H_2O slowly to bring the liquid to the original mark.

(d) Filter through a 0.1 micron Millipore or Nucleopore filter using the Millipore filter holder.

(e) Measure the pH of the final solution and store in a suitable plastic bottle (not transparent). Transfer a portion of the final solution to the Analytical lab for analysis.

C. Preparation of Equilibrated J-13 Water with pH Adjusted to 6.9 ± 1

(1) Take the required amount of the filtered EJ-13 solution and measure its initial pH.

(2) Add dilute sulfuric acid dropwise to the solution until the pH reaches 6.9 ± 1.

(3) If the pH falls below 6.0 add EJ-13 solution to bring the pH up again.

6.3 Cleaning Procedures

6.3.1 Procedure for Cleaning Monolithic Samples

Qty	Device	Manufacturer	Model #	Calib. Req.
1	Ultrasonic Cleaner	Bransonic	B-221	N.A.
1	Vacuum Oven	National Applicances	5831	N.A.
	Ethyl alcohol USP, absolute Reagent Quality			

(1) Place sample in a polystyrene vial.

(2) Add sufficient reagent grade ethanol to cover sample.

(3) Place in ultrasonic cleaner for 5 minutes.

(4) Transfer sample to a new vial; add fresh ethanol again.

(5) Repeat steps (2)-(4) three times.

(6) Place sample in vial in vacuum oven preheated to ~70°C and leave to dry for 20 minutes.

6.3.2 Procedure for Teflon and Steel

A. MCC procedure for cleaning new Teflon containers for IAEA tests. This procedure may be begun at step (3) for used containers.

Teflon containers: 60 ml Savillex-0102

Teflon baskets: Savillex 46SC

(1) Soak for one hour in 6 M HNO_3 + 0.2 M HF.

(2) Rinse with three container volumes of high-purity H_2O.

(3) Soak in 6 M HNO_3 for four hours at 50°C.

(4) Soak for 30 minutes in >60°C high-purity H_2O by full immersion.

(5) Soak for at least eight hours in fresh high-purity H_2O at 80°C by full immersion.

(6) Boil for 30 minutes in fresh high-purity H_2O by full immersion.

(7) Rinse with successive container volumes of high-purity H_2O until the pH of two successive rinse solutions are within 0.5 pH units of the original high-purity H_2O. A minimum of three rinses is required.

B. Procedure for cleaning Teflon containers used in pulsed flow tests. All parts are from Savillex. This procedure may be begun at step (3) for used containers.

Teflon segments Cat.#504

Teflon end cap Cat.#501

Teflon digestion ves. Cat.#560

Teflon transfer cap Cat.#501-37

(1) Soak for one hour in 6 M HNO_3 + 0.2 M HF.

(2) Rinse with three container volumes of high-purity H_2O.

(3) Boil in 6 M HNO_3 for four hours.

(4) Rinse with three container volumes of high-purity H_2O.

(5) Autoclave at 120°C for one hour in high-purity H_2O in stainless steel container.

(6) Boil for one hour in high-purity H_2O in a stainless steel beaker.

(7) Soak for a minimum of eight hours in high-purity H_2O in a polypropylene container at room temperature.

(8) Heat in a 200°C oven for five days.

(9) Boil for one hour in high-purity H_2O in a stainless steel beaker.

(10) Rinse with three container volumes of high-purity H_2O.

(11) Air dry at room temperature.

(12) Fill Teflon containers with 125 ml DI water, close lid, put rubber septum in place and leave in oven at 90°C.

(13) Measure pH of the DI water, record in 'Teflon Washing' L.N.

(14) If pH is <5.7 repeat steps 12 and 13; otherwise proceed to step 15.

(15) Air dry at room temperature.

C. Procedure for cleaning stainless steel containers used in pulsed flow tests:

All parts are from Whitey

Stainless Steel Sample Cylinder	Cat.# 304L-HDF4-150
Stainless Steel Pipe Plug	Cat.# SS-4-P
Stainless Steel VCO Nut	Cat.# SS-4-VCO-4
Stainless Steel VCO Male Connector	Cat.# SS-4-VCO-1-4

(1) Use as-received pickled or machined (rather than passivated) stainless steel vessels.

(2) Clean with analytical grade acetone for 5 minutes in an ultrasonic bath.

(3) Clean with reagent quality ethanol for 5 minutes in an ultrasonic bath.

(4) Rinse thoroughly with DI water.

(5) Let stand in a nitric acid solution overnight in a 90°C oven. The nitric acid solution is made by dissolving 1 gram of concentrated nitric acid (about 70% by weight, analytical reagent grade) in 180 ml of deionized water or by diluting 4 mls. of concentrated nitric acid to 1 litre.

(6) Rinse thoroughly with DI water.

(7) Dry overnight in an oven at 90°C.

6.3.2 Cleaning Ross Type pH Electrodes

MAINTENANCE

Weekly maintenance:

(1) Inspect electrode for scratches, cracks, salt crystal buildup, or membrane/junction deposits.

(2) Rinse off any salt build-up with DI water, and remove any membrane/junction deposits as directed in cleaning procedures below.

(3) Drain the reference chamber, flush it with fresh fill solution and refill the chamber.

Cleaning the electrode:

REFERENCE JUNCTION

(1) Rinse electrode with deionized water.

(2) Soak in 0.1M HCl or HNO_3 for half an hour.

(3) Remove electrode from acid solution, drain the reference chamber, flush it with fresh filling solution and refill the chamber.

(4) Rinse electrode with deionized water.

(5) Soak in storage solution for at least one hour before using.

6.4 Test Procedures

6.4.1 Pulsed Flow Tests

Equipment and Supplies

Qty.	Device	Manufacturer	Model#	Calib. Req.
1	Balance	Sartorius	Basic 4100S	I
1	Balance	Mettler	AE100	II
1	Balance	Scientech	5200	III
18	Ovens	Blue-M	Stabil-Ther, OV-490A, OV-490A-3	IV
1	pH Meter	Fisher	Accumet-810	V
1	pH Meter	Fisher	Accumet-910	V
1	Polypropelene Wrench Set	Savillex		NA
1	Grad. Cylinder	Naglene		VI
1	Plastic Funnel	Naglene		NA
	Thermometers	Various	-20 to 150°C, ± 1°C	V
	Dessicator			NA
	Metal Box	Hoffman Eng. Co.	NEMA Type 4, in-house Modif.	NA
	Syringes with gauge 20,4 inch hypodermic needles			NA
	Syringes with gauge 20, 6 inch hypodermic needles			NA
	Teflon sections	Savillex	#504, #501, #50-37	NA
	Stainless steel sections	Whitey	#304L-HDF4150	NA
		Whitey	#SS-4-P	
		Whitey	#SS-4-VCO-4	
		Whitey	#SS-4-VCO-1-4	
		Cajon	#SS-4-VCO-3-4TA	
		Swagelok	#SS-400-C	
	Three layer rubber septa	Hamilton	76-6006	
	O-ring	Whitey		NA
				NA

Calibration Notes: I: Calibration Accuracy ±0.1 g, Calibration frequency monthly; II: Calibration accuracy: ± 1 mg, Calibration frequency, monthly; III: Calibrations in TPI-GP; IV: temperature monitored daily with thermometers that are positioned as recommended by the manufacturer and meet NBS Specification, Monograph 150; V: Calibration accuracy ±0.05 pH units, Calibration frequency: Daily; VI: Check accuracy of volume by weighing a

measured volume of DI water prior to first use; VII: meeting NBS Specification , Monograph 150.

PROCEDURES FOR PULSED FLOW TESTS

Procedure

TEFLON CONTAINERS

A. Leachant: Deionized (D.I.) water

(1) Weigh sample. Record data in the lab notebook.

(a) If a powdered sample is required, grind as prescribed in section 6.1.1 and select 40-60 mesh powder.

(b) If a block sample is required, prepare following section 6.1.2, clean following section 6.3.1 and record dimensions and other information pertinent to the test in the lab notebook.

NOTE: ***In the case of radioactive samples prepared and weighed in the α- Lab, ~5 ml of DI water are added to the powder to facilitate transfer to the non-radioactive lab.***

(2) Transfer sample into a pre-cleaned 125 ml PFA teflon pipe section (Savillex #504) with a well tightened screw on end cap on one end (Savillex #501). Clean all Teflon parts as prescribed in section 6.3.2.

(3) In a 2L SS beaker, place 1L of DI water and bring to a boil. Bubble nitrogen into water and continue boiling for ~10 minutes.

(4) With a graduated cylinder measure 100 ml (95ml for radioactive samples) of boiled DI

water and pour into Teflon pipe section with sample; mark water level. To prepare blanks, eliminate sample. While container is open, blow nitrogen in to prevent CO_2 contamination.

(5) Close the Teflon pipe section using the screw on transfer cap (Savillex #501-37) with rubber septum secured in place and tighten it using the polypropylene wrench.

(6) Remove rubber septum. Blow nitrogen into container for ~2 minutes and secure septum in place again.

(7) Place ascarite in a dish which is placed in a Petri dish in the center of the desiccator below the desiccator plate. A plastic weighing boat may be used instead of the Petri dish for test temperatures below 70°C.

(8) Transfer closed pipe sections with samples, and blanks into the desiccator or metal box.

(9) With lid partially opened, introduce nitrogen into the desiccator for about 5 minutes. When using the metal box desiccator introduce nitrogen through the inlet valve keeping the outlet valve open; close both valves when finished.

(10) Close desiccator lid.

(11) Place desiccator or metal box in an oven preheated to the required test temperature as per instructions of the Project Manager.

(12) After 24 hours remove the dessicator or metal box from the oven and remove the pipe sections from the dessicator.

(13) Check and if necessary retighten the end and transfer caps of the teflon pipe sections.

(14) Repeat steps (8) through (11).

(15) At the specified intervals, determined for each test by the Project Manager, replace 25 ml of leachate or a different volume according to project manager's instruction with an equal volume of fresh leachant, using a syringe with a gauge 20, 4 inch hypodermic needle. Perform the exchange as follows:

(a) Remove desiccator or metal box from oven.

(b) Remove pipe sections from desiccator.

(c) Inserting needle through septum, extract 25 ml or appropriate volume of leachate.

(d) Transfer 3-4 ml of the sample leachate into a plastic cup placed in a water bath at room temperature and measure pH. Record measurement in the "pH Meas. on Flow Tests" L. N.

(e) Transfer the rest of the extracted leachate into a polystyrene vial appropriately labeled for analysis.

(f) Using a syringe and needle dedicated exclusively to use with DI water, transfer 25 or appropriate volume ml of DI water, boiled as prescribed in step 3, into the pipe section. Be sure the needle does not touch the liquid inside the container. If necessary add more DI water to each individual pipe section in order to bring the solution to the level marked in Step 4.

NOTE: New syringes and needles must be used for each new test.

The same syringe and needle may be used for duplicate or triplicate samples. In this case, wash the unit 3 times with ~10 mL of boiling DI water.

(g) When sampling is finished, check ascarite and rubber septums, return closed pipe section into the desiccator or metal box, blow nitrogen in for ~5 minutes, and put lid in place.

(h) Return desiccator or metal box to oven to continue test.

(16) Transfer the samples to the Analytical Laboratory for analysis according to the instructions of the Project Manager.

(17) Store results on floppy disk (refer to section 8.1) and file hard copy in the appropriate binder. Durability data binders are labelled according to name of tests contained within.

B. Leachant: Ground water or EJ-13 Solution.

(1) Weigh sample. Record data in the lab notebook.

(a) If a powdered sample is required, grind as prescribed in section 6.1.1 and select 40-60 mesh powder.

(b) If a block sample is required, prepare following section 6.1.2, clean following section 6.3.1 and record dimensions.

(2) Transfer sample into a pre-cleaned 125 ml PFA Teflon pipe section (Savillex #504) with a well tightened screw on end cap on one end (Savillex #501). Clean all Teflon parts as

prescribed in section 6.3.2.

(3) Add 100 ml of leachate into Teflon pipe section with sample. To prepare blanks eliminate sample.

(4) Close Teflon pipe section using screw on transfer cap with rubber septum secured in place. Tighten the transfer cap using the polypropylene wrench.

(5) Transfer closed pipe sections with samples, and blanks into the metal box or desiccator and put lid in place.

(6) Place a metal box or desiccator without ascarite in an oven preheated to the required test temperature as per instructions of the Laboratory Manager.

(7) Perform steps (12) through (16) of Section A, however, do not introduce nitrogen into the desiccator.

(8) Store results on floppy disk (refer to section 8.1) and file hard copy in the appropriate durability data binder.

C. Radioactive Samples (All leachants)

(1) Tightly fit the end cap on the appropriate side of the Teflon pipe section.

(2) Transfer the Teflon pipe section along with the transfer cap and rubber septum to the glove box in the laboratory where radioactive samples are stored.

(3) Weigh the required amount of glass powder in a plastic weighing boat or polystyrene vial on the scientech balance inside the glove box.

(4) Carefully transfer the glass powder into the Teflon pipe section together with 20 ml of leachant.

(5) Close the transfer cap and wipe the Teflon pipe section with a wet tissue in order to ensure that no glass powder remains on the outside of the container.

(6) Remove the Teflon container from the glove box and, while wearing disposable gloves, rinse and dry it again with DI water.

(7) Return the vessels containing the samples to the leach laboratory.

(8) Open the transfer cap and transfer 80 ml of boiling DI water under nitrogen or other leachant if leachant used is not DI water. Immediately close and tighten the transfer cap using the appropriate wrench.

(9) Continue the test from step 6 in Part A for DI water as the leachant or from step 5 in Part B for ground water or EJ-13 solution as the leachant.

STAINLESS STEEL CONTAINERS

D. Non-radioactive Samples

Note: 1) For tests with leachants other than Deionized water all procedures below are to be followed except that no nitrogen or ascarite is to be used.

2) No ground water is used in stainless steel vessels.

(1) Fit the stainless steel pipe plug on the appropriate end of the stainless steel sample

cylinder, using Teflon tape (according to the manufacturer's instructions). Tighten the pipe plug using an adjustable wrench and vice.

(2) Weigh the required amount of glass powder.

(3) Transfer the glass powder into the stainless steel sample cylinder together with 100ml DI water boiled under nitrogen if DI water is the leachant or with just fresh leachant in the case of other leachants.

(4) Immediately attach the stainless steel VCO male connector to the open end of the cylinder using the teflon tape.

(5) In the case of DI water as leachant introduce nitrogen through the hole of the VCO male connector for 1 minute.

(6) Immediately place a rubber O-ring on top of the VCO male connector and then place the rubber septum and secure it with the stainless steel VCO nut containing a stainless steel O-ring on the inside.

(7) Tighten the connector properly using an adjustable wrench and vice.

(8) If required by the Laboratory Manager's instructions screw the safety extender onto the vessel.

(9) Weigh and record the total weight of each individual stainless steel vessel.

(10) Place the stainless steel vessel horizontally or vertically in a dessicator or metal box containing a dish of ascarite, and introduce nitrogen.

(11) Place the above in a preheated oven and record the time as immersion time.

(12) After 24 hours, take out all vessels from the oven and weigh the stainless steel vessels.

(13) If the weight measured differs by less than 5% from the weight determined in Step 8 continue with the test otherwise consult the Laboratory Manager before proceeding.

(14) At the specified intervals sample the leachate as follows:

a) Remove all stainless steel vessels and place them vertically in a stand. Allow them to cool to room temperature.

b) Weigh all stainless steel vessels and record the weighing in the Flow Test LN.

c) If a safety extender is attached to the vessel remove the cap from the vessel.

d) Using a designated syringe and needle withdraw the exact volume of solution as specified by the Laboratory Manager.

e) Collect the above solution in a vial and label it properly (a part of this solution is to be used to measure pH and the remaining portion to be sent to Analytical Lab for analysis.

f) Replace with an exact amount of fresh leachant. ***(Note: In the case of DI water as leachant, it should be replaced with boiling DI water under nitrogen.)***

g) If a safety extender is attached to the vessel replace the stainless steel cap.

h) Weigh all the containers again and compare their weights with those before sampling. If the weight is less than the weight before sampling, add some leachant; if the weight is slightly higher than before, do not remove any solution.

i) Inspect all rubber septa and check if there are any holes present due to injection.

If holes are found, replace the rubber septa.

j) Return all the stainless steel vessels to the oven.

E. Radioactive Samples (All Leachants)

(1) Perform step (1) as in Part D of this procedure.

(2) Label each stainless steel vessel ensuring that all the parts are together, however, do not tighten the VCO male connector.

(3) Transfer the vessels to the Glass Preparation Laboratory, Room 39.

(4) Weigh the required amount of glass powder in a plastic weighing boat on polystyrene vial on the sartorius balance inside the glove box in Room 39.

(5) Carefully transfer the glass powder into the stainless steel vessel and lightly close the VCO male connector.

(6) Wipe the vessel with a wet tissue in order to ensure that no glass powder remains on the outside of the container.

(7) Return the vessels containing the samples to the leach laboratory.

(8) Place the vessels vertically in a stand in the fume hood.

(9) Open the VCO male connector and transfer 100 mls of fresh leachant to the vessel using a plastic funnel. (If DI water is the leachant 100 mls of DI water boiled under nitrogen should be used.)

(10) Rinse the plastic funnel after each transfer.

(11) Proceed as in Steps (4) - (13) of section D of this procedure.

6.4.2 PROCEDURE FOR IAEA (HIGH DILUTION) TESTS

Equipment and Supplies

Qty	Device	Manufacturer	Model #	Calib. Req.
1	Balance	Mettler	AE100	I
18	Ovens	Blue-M	Stabil-Therm OV-490A	II
1	pH Meter	Fisher	Accumet-810	III
1	pH Meter	Fisher	Accumet-910	III
	Grad. cylinder	Nalgene		IV
	Thermometers	Various	-20 to 150°C, ± 1°C	V
1	Teflon vessel	Savillex	#0102	N.A.
1	Teflon basket	Savillex	#46SC	N.A.

Calibration notes: I: calibration accuracy: ± 1 mg, calibration frequency: monthly; II: temperature monitored daily with thermometers that are positioned as recommended by the manufacturer and meet NBS Specification, Monograph 150; III: calibration accuracy: ± 0.01 calibration frequency: daily; IV: check accuracy of volume measured prior to first use; V: meeting NBS Specification, Monograph 150.

PROCEDURE FOR IAEA (HIGH DILUTION) TESTS

Procedure

(1) Weigh sample. Record data in the lab notebook.

(a) If a powdered sample is required, grind as prescribed in section 6.1.1 and select

40-60 mesh powder.

(b) If a block sample is required, prepare following 6.1.2, clean following section 6.3.1 and record dimensions.

(2) Transfer sample into a precleaned 60 ml PFA Teflon vessel (Savillex #0102). If block samples are being used, place a PFA Teflon basket (Savillex #46SC) inside the vessel. Clean all teflon parts as prescribed in section 6.3.2.

(3) Measure the required volume of leachant (40 ml) using a graduated cylinder and transfer into the teflon vessel.

(4) Screw on lid and tighten properly to minimize vaporization.

(5) Place closed teflon vessel in an oven preheated to the specified test temperature as per instructions of the Project Manager. Record the time of this operation as the "immersion time" in the lab notebook.

(6) Replace the entire volume of leachate with a similar volume (40 ml) of fresh leachant, daily to begin with, then weekly, and then monthly, or as specified for each individual test by the Laboratory Manager. Carry out this operation as follows:

(a) Remove the container from the oven and let it cool for about 10 minutes.

(b) If a powdered sample is being used, remove the lid and decant the liquid carefully if necessary using a disposable transfer pipette.

(c) If a block sample is being used, remove the lid, take out the

sample with clean plastic tweezers, empty the vessel, and return the sample to it.

(d) If requested by Laboratory Manager, transfer ~ 4 ml of the sampled leachate into a plastic cup placed in a water bath at room temperature and measure pH using a Ross-type electrode. Record pH reading in the "pH Data for IAEA Tests" L. N.

(e) Refill the container as per steps (3) and (4) and return to the oven.

(f) Record sampling time to the nearest 10 minutes in the 'pH Data for IAEA Tests' L.N.. If this operation is carried out on many samples, record the average time at which sampling is carried out as the sampling time for all.

(7) Analyze samples using DC plasma spectroscopy and colorimetry. Refer to Analytical Procedures.

(8) Store results on floppy disk (refer to section 8.3) and file hard copy in the appropriate binder. Durability data binders are labelled according to the tests contained within.

(9) Alternatively all data from this test may be recorded in the 'Glass Composition' LN and the 'pH Data for IAEA Tests' LN.

6.5 Calibrations

6.5.1 Balance

Frequency: Monthly for Mettlers and Sartorius; six monthly for all other balances except those which are for qualitative use only.

Required Accuracy: As specified in individual procedures or as per instructions of the Laboratory Manager.

(1) Obtain a set of Class S-type standard weights.

(2) Obtain a "Balance Calibration Check Record" form and supply requested information on balance to be calibrated (e.g. manufacturer, model #, etc.).

(3) Weigh the standard weights in the balance and record the nominal weight, actual weight, and percent error under the appropriate columns.

(4) Calculate the percent error as follows:

$$\frac{\text{(Actual weight) - (Nominal weight)}}{\text{(Nominal weight)}} \times 100 = \%\ \text{Error}$$

(5) Examine results and reports deviations from required accuracy to the Laboratory Manager.

(6) Sign and date and file form in the 'Calibration Records' binder.

6.5.2 Pipette

PIPETTE CALIBRATION CHECK PROCEDURE

Method: Gravimetric using a Mettler AE100, or a Sartorius Basic 4100S balance.

Frequency: Monthly except Rainin pipette which is six monthly.

Required Accuracy: As specified in individual procedures or as per instructions of the Project Manager.

(1) Obtain a "Pipette Calibration Check Record" form and supply requested information on pipette to be calibrated (e.g. manufacturer, model #, etc.).

(2) Take out the weight of a 50 ml plastic vial on the balance.

(3) Pipet out deionized water from the pipette to be calibrated into the plastic vial. Record nominal capacity, actual capacity (weight displayed on the Mettler balance), and percent error under the appropriate columns.

(4) Repeat steps (2) and (3) five times. For variable volume pipettes perform the measurement for 2-5 volume settings.

(5) Calculate the percent error as follows:

$$\% \text{ ERROR} = \frac{(\text{Actual capacity}) - (\text{Nominal capacity})}{(\text{Nominal capacity})}$$

(6) Examine results and report deviations from required accuracy to the Laboratory Manager.

(7) Sign and date and file form in the "Calibration Records" binder.

6.5.3 pH Meter

pH METER CALIBRATION PROCEDURE

Frequency: Daily or every 25 measurements
Required Accuracy: ± 0.05 pH units
Instruments: Fisher Accumet-810, 910 pH meters and Corning 245 pH meter.

(1) Remove the pH electrode from the routine storage solution.

(2) Wash the pH electrode and beaker with DI water.

(3) Adjust the instrument to the pH measurement mode.

(4) Adjust the temperature setting to room temperature.

(5) Choose two standard buffer solutions traceable to NBS Standards which covers the expected range of pH to be measured.

(6) With the slope dial on 100% calibrate the instrument with one of the standard buffer solutions. Allow the reading to stabilize. The pH should be corrected according to the pH-temperature table on the bottle. Record the buffer used, temperature and date in the appropriate LN.

(7) Wash the beaker and electrode with DI water and then insert the electrode in the second standard buffer solution. Adjust the slope dial such that the stabilized pH reading corresponds to the value of the pH expected from the pH-temperature table on the bottle. Again record the buffer used, temperature and date in the appropriate LN. The pH meter is now calibrated.

(8) Wash the electrode well with DI water before and between measurements of the pH of the samples.

(9) On completion of all measurements return the pH electrode to the routine storage solution and set the instrument to "Standby".

(10) If measurements fall outside the calibration range of pH meter, calibrate again with an appropriate buffer to cover the whole pH range of measurements. An accuracy of _+0.3 pH units is required for this final calibration.

6.5.4 Oven Thermometers

PROCEDURE FOR CALIBRATION OF OVEN THERMOMETERS

Equipment and Supplies

Qty	Device	Manufacturer	Model #	Calib. Req.
1	Standard Thermometer (Traceable to NIST)			I
1	Glass Beaker			N.A.

Calibration notes: I: calibrated by manufacturer

PROCEDURE

NOTE: This procedure shall be performed once a year on all oven thermometers.

1. Heat a tall beaker of water to about 55°C.

2. Place the standard thermometer (traceable to NIST) in the water so that the mercury bulb and the scale up to 30°C are immersed in the water.

3. Place the thermometers to be calibrated in the water to a depth of 76 mm as indicated by the appropriate mark on the thermometer for 76 mm partial immersion thermometers, or to a depth corresponding to 55°C on the scale for total immersion thermometers.

4. Record the temperature of the standard thermometer and the temperatures of the test thermometers.

5. Place a second thermometer adjacent to the standard thermometer such that the bulb of the second thermometer is centered halfway between the surface of the water and the temperature indicated on the standard thermometer.

6. Record the temperature indicated by the second thermometers.

7. Calculate the stem correction for the standard thermometer from the formula

$$\text{Stem correction} = 0.00016 \times N \times (T - ST)$$

where N is the number of degrees centigrade of the mercury column in the standard thermometer above the surface of the water. T is the temperature is °C indicated on the standard thermometer and ST is the temperature in °C indicated on the second thermometer.

8. Calculate the corrected standard temperature by adding the stem correction to T.

9. Repeat steps 1 through 8 for temperatures of around 70°C and 90°C.

10. A calibration accuracy of ± 1°C is acceptable.

Digital Ovens:

For digital oven calibration check, use a calibrated thermometer above and place it in a 125 ml Erlenmeyer flask filled with DI water and close with a rubber stopper. Keep the flask with the thermometer inside digital oven for overnight. Remove the flask from the oven and immediately read the temperature and record. Compare the temperature reading with the reading displayed on the digital oven. A plus or minus 1°C is acceptable.

7.0 SPECIMEN AND DATA TRACEABILITY

Samples arrive in the leach laboratory from the glass preparation laboratory. Each time a glass sample is received in the leach laboratory, the following particulars are documented in the laboratory notebook:

Number of the glass
Lab notebook # and the page # documented in the glass preparation laboratory

The glass sample is loaded into the leach vessel (stainless steel vessel with a permanent number inscribed on it). This part is done in the glass preparation laboratory (for safe radioactive handling purpose).

Sampling details of the leach for each glass are recorded in the appropriate laboratory

notebook and sent to analytical laboratory for analysis.

8.0 DATA PROCESSING

8.1 Pulsed Flow

PROCEDURE FOR RUNNING PULSED FLOW
DATA ANALYSIS PROGRAMS

Equipment List

Qty	Device	Manufacturer	Model	Calib. Req.
1	Computer	IBM compatible	386	N.A.
1	Monitor	Princeton	Max-12	N.A.
1	Keyboard	US Keyboard	US101	N.A.
1	Printer	Epson	FX286e	N.A.
1	Flow Software Package			N.A.

Procedure

Note: <CR> indicated the "enter" key on the keyboard.

(1) Turn the computer, monitor and printer on.

(2) Type E: <CR>

(3) Type CD\FLOW <CR>

(4) Type FLOWMAIN

(5) Type 1 to print the original data
Type 2 to calculate averaged data
Type 3 to sort the averaged data
Type 4 to calculate normalized data

Type 5 to search for sample names on the disk files
Type 6 to translate data from the present format to a format which may be read by LOTUS 1,2,3.
Type 8 to print an index of parameters for a test.

(6) Follow the instructions on the screen in order to run the desired program.

(7) At the end of the session type 0 and on obtaining the prompt E:\Flow> type OFF <CR> and the Y<CR>.

(8) Turn off the computer, monitor and printer.

8.2 IAEA

PROCEDURE FOR RUNNING IAEA DATA ANALYSIS PROGRAMS

Equipment List

Qty	Device	Manufacturer	Model #	Calib. Req.
1	Computer		IBM Compatible 386	N.A.
1	Monitor	Princeton	MAX-12	N.A.
1	Keyboard	US Keyboard	US101	N.A.
1	Printer	Epson	FX286e	N.A.
1	IAEA software program			N.A.

Procedure

Note: <CR> indicates the "enter" key on the keyboard.

(1) Turn on the computer, monitor, and printer.

(2) Type E: <CR>.

(3) Type CD\IAEA <CR>.

(4) Type IAEAMAIN.

(5)

Type 1	to print the original data.
Type 2	to calculate leach rates based on the original data.
Type 3	to average the leach rate data.
Type 4	to sort the means of the leach rates.
Type 5	to search for sample names on the disk files.
Type 6	to translate data from the present format to a format which may be read by LOTUS 1,2,3.
Type 7	to print an index of parameters for a test.
Type 8	to normalize the leach rate data.

(6) Follow the instructions on the screen in order to run the desired program.

(7) At the end of the session type 0 and on obtaining the prompt E:\IAEA > type OFF <CR> and then Y <CR>.

(8) Turn off the computer, monitor, and printer.

Section 9.0

ACCURACY AND RELIABILITY OF TEST RESULTS

The data used to generate the measures of precision is the result of intra- and inter-laboratory round robins. These measures are typical of the methods and applied to the glasses and standards used in the round robins, and are not all inclusive with respect to other types of glasses. The measures of precision were determined in accordance with procedures in ASTM Practice E691. These measures are designated as follows:

Repeatability: the standard deviation for within-laboratory determinations.

Reproducibility: the standard deviation for between-laboratory determinations.

Bias: the average corrected leachate concentrations for the standard glass allow assessment of long-term bias or variability of the test, e.g. how reproducible the experimental variables such as oven temperature, sieving, leachate analyses, etc. are over time. Use of a standard glass provide the basis for both within-laboratory and between data comparisons.

SECTION 10.0

RECORD KEEPING

The following records are to be maintained in the leach testing laboratory:

(1) Calibration Data:

All calibrations carried out for balances, pipettes, should be documented. The calibration data should include the weight/volume observed vis-a-vis marked data, the percentage error detected, pass/rejection, date of the calibration and the signature of the person carrying out the calibration.

(2) Leach test data for each of the leach test - pulsed flow, IAEA, should be documented.

(3) Sample preparation/cleaning records, details of sample preparation, cleaning of vessels.

(4) Leachant details:

Recipes for leachants prepared, and Millipore records should be maintained.

The above laboratory record books are subject to periodic surveillances as part of the Quality Assurance Program.

UNCONTROLLED

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EPA Toxicity Characteristic Leaching Procedure (TCLP) Applied to Waste Glasses at VSL

October 1990

1. Scope and Application

1.1 The unmodified EPA TCLP Method 1311 is designed to determine the immobility of both organic and inorganic contaminants present in liquid, solid, and multiphasic wastes.

1.2 This VSL modified version of the TCLP test is used specifically for solid waste glasses.

1.3 The waste glasses tested by this method are made at a temperature 1100°C or above in air. The amount of organics remaining in such glasses will be negligible. Therefore the procedure described here in complies with the EPA specification of EPA method 1311 when volatiles are not involved.

2. Referenced Documents

2.1 Method 1311 Toxicity Characteristic Leaching procedure, Federal Register, V.55, No. 61, 1990.

2.2 ASTM Standards

D1193 Specification for Reagent waster [1]
D1193 Standard Test Methods for pH of water [1]
D1125 Test Methods for Electrical Conductivity and Resistivity of Water [1]

4. Significance and Use

4.1 This test method provides data useful for evaluating the toxicity of a solid. A solid waste exhibits the characteristic of toxicity if, using this test method, the extract from a representative sample of the waste contains any of the contaminants listed in Table 1 at a concentration equal to or greater than the respective value given in that Table.

Table 1 - Maximum Concentration of Contaminants for the Toxicity Characteristic

Contaminant	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Mercury	0.2
Selenium	1.0
Silver	5.0

5. Apparatus

5.1 Extraction Bottles - Borosilicate glass bottles of 2.2 liter capacity (#3740-NGB, Associated Design and Manufacturing Company) or 2.2 liter polyethylene bottle (#3740-PB, Associated Design and Manufacturing Company).

5.2 Agitation Apparatus: The agitation apparatus, capable of rotating the extraction vessel in an end-over-end fashion at 30 ± 2 rpm, is of an EPA-approved design and purchased from Associated Design and Manufacturing Company (model 3740-BRE).

5.3 Filtration Devices: these devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm. The filter holder is capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation. The materials of construction of the filtration devices shall be made of glass, polytetrafluoroethylene or type 316 stainless steel.

5.3.1 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 µm or equivalent. Filters known to EPA which meet these specifications are identified in Table 2. Prefilters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1-L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

Table 2

Company	Location	Model	Pore size
Millipore Corporation	Bedford, MA (800) 225-3384	AP40 /	0.7
Nucleopore Corporation	Pleasanton, CA (415) 483-2530	211625	0.7
Whatman Lab. Products,Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dubin, CA (800) 334-7132 (415) 826-6010		

5.4 Sieves - 9.5 mm (0.375 inch), or finer, Teflon-coated sieve.

5.5 Crushing Device - any mechanical or manual crushing device which will avoid steel contamination in the crushed specimen. A grinding device made of tungsten carbide is one such example.

5.6 Laboratory Balance - Any laboratory balance accurate to within ±0.01 grams may be used (all weight measurements are to be within ±0.1 grams).

5.7 Weight Calibration Set - NIST Class S-type weight calibration set ranging from 100 mg to 1 kg.

5.8 Pipettes - Calibrated and precleaned tips.

5.9 Sample Vials - Precleaned sample vials and caps.

5.10 pH Meter - pH meter with an accuracy of ± 0.05 pH units at 25°C.

5.11 Milli-Q Water Purification System - Water purification system for producing reagent water.

6. Calibrations

6.1 Calibrations - Initially calibrate all instruments used in this test. Perform periodic calibrations during use of the instrument to minimize possible errors due to instrumental drift.

6.2 Calibration and Standardization Schedule:

6.2.2 pH meter - Before and after series of measurements with commercial buffer solutions that bracket the solution pH being measured.

6.2.3 Pipettes - once every month.

7. Standards

7.1 The reference solution standards should be traceable to NIST, or a comparable equivalent should be used, and should have a certified shelf life.

7.2 pH Buffers - Commercial pH buffers to bracket the measured pH range of the leachant and leachate.

8. Reagent

8.1 Reagent water, made from Millipore Super-Q or equivalent system.

8.1.1 ASTM Type I water - Type I water shall have a minimal electrical resistivity of 16.67 M Ohm cm at 25°C (consult ASTM D1193).

8.1.2 The source water shall be passed through Milli-R015 System which contains 6 cartridges (Ful-Flow, Teel, Lifegard, Rogard Prefilter and Milli-Ro-20 Spiral-Wound Reverse Osmosis) and Milli-Q System that consists of five cartridges (Lifegard, Super-C, two of Ion-Exchanger, and Millipore DI-12). Pass the purified water through an in-line conductivity cell to verify its purity.

8.2 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

8.3 Nitric acid (1N), HNO_3, made from ACS reagent grade.

8.4 Sodium hydroxide (2N), NaOH, made from ACS reagent grade.

8.5 Glacial acetic acid, HOAc, ACS reagent grade.

8.6 Extraction fluid:

8.6.1 Extraction fluid #1: Add 5.7 mL glacial HOAc to 500 mL of the appropriate water (See Step 5.1), add 64.3 mL of 1 N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

8.6.2 Extraction fluid #2: Dilute 5.7 mL glacial HOAc with ASTM Type I water to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

Note: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

8.7 Analytical standards prepared according to the appropriate analytical method.

9. Preliminary Evaluations

9.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle-size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500-mL beaker or Erlenmeyer flask.

9.2 Add 96.5 mL of reagent water (ASTM Type II or better) to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 10.0.

9.3 If the pH from Step 9.2 is >5.0 add 3.5 mL 1N HCl, slurry briefly, cover with a

watchglass, heat to 50°C, and hold at 50°C for 10 minutes.

9.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0 use extraction fluid #1. If the pH is >5.0 use extraction fluid #2. Proceed to Section 10.0.

10. Procedure

10.1 Weigh out 110 gram of the solid waste, reduce the size by grinding until the material will pass through a 9.5mm (0.375 inch) standard sieve.

10.2 Transfer 100.0 gram the ground solid material into an extractor bottle.

10.3 The amount of extraction fluid to be added to the extraction vessel is 20 times the weight of solid sample used, i.e. 2000.0 grams for 100 grams of solid waste.

Slowly add this amount of the appropriate extraction fluid (see Step 9.0) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e. temperature of room in which extraction takes place) shall be maintained at 22 ± 3°C during the extraction period.

Note: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

10.4 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as follows:

10.4.1 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder. Spread the waste sample evenly over the surface of the filter.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next 10-psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2-minute period) stop the filtration.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration.

10.5 The filtered liquid material obtained from Step 10.4 is defined as the TCLP

extract.

10.6 Following collection of the TCLP extract, add 20.0 mL of extract to each of six labeled sample vials. Measure the pH's of two of the six vials and record the pH's. Add 1.0 mL 1N HNO_3 to each of the four remaining vials to ensure pH < 2.0 of the resulting solutions. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4°C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic contaminants. If an analysis of the undigested extract shows that the concentration of any regulated metallic contaminant exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ±0.5 percent), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final analyte concentration} = \frac{(V_1)(C_4) + (V_2)(C_4)}{V_1 + V_2}$$

where:
V_1 = The volume of the first phase (L).
C_1 = The concentration of the contaminant of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the contaminant of concern in the second phase (mg/L).

10.7 Compare the contaminant concentrations in the TCLP extract with the thresholds identified in the appropriate regulations (Table 1). Refer to 11.0 for quality assurance requirements.

11.0 Quality Assurance Requirements

11.1 Maintain all data, including quality assurance data, and keep it available for reference or inspection.

11.2 A minimum of one blank (extraction fluid #1) for every 10 extractions that have been conducted in an extraction vessel shall be employed as a check to determine if any memory effects from the extraction equipment are occurring.

11.3 A matrix spike shall be performed for each waste unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. If more than one sample of the same waste is being tested, a matrix spike needs to be performed for every twenty samples and the average percent recovery applied to the waste characterization.

11.3.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

11.3.2 Matrix spike levels should be made at the appropriate regulatory threshold limits. However, if the extract contaminant concentration is less than one half the threshold limit, the spike level may be one half the contaminant concentration but not less than the quantitation limit or a fifth of the threshold limit.

11.3.3 The purpose of the matrix spike is to monitor the adequacy of the analytical methods used on the TCLP extract and to determine whether matrix interferences exist in analyte detection. If the matrix spike recoveries are less than 50%, then the analytical methods are not performing adequately or use of the methods is inadequate. Use of internal calibration quantitation methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the contaminant concentration in the TCLP extract.

11.3.4 Use of internal quantitation methods is also required when the contaminant concentration is within 20% of the regulatory level. (See section 11.5 concerning the use of internal calibration methods.)

11.3.5 Matrix spike recoveries are calculated by the following formula:

$$\text{Percent recovery} = \frac{A-B}{C} \times 100\%$$

where A = the concentration of the spike sample.
B = the concentration of the unspiked sample, and
C = the spike level

11.4 All quality control measures described in the appropriate analytical methods shall be followed.

11.5 The use of internal calibration quantitation methods shall be employed for a contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

11.5.1 The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

11.5.2.1 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The fourth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solutions, and may need dilution adjustment to maintain the signals in the linear range of the instrumental technique. All four aliquots are analyzed.

11.5.1.2 Prepare a plot, or subject data to linear regression, of instrumental signals or external-calibration-derived concentrations as the dependent variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

11.5.1.3 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject data to linear regression of the corrected instrumental signals or external-calibration-derived concentrations as the dependent variable versus the independent variable. Derive concentrations for unknowns using the internal calibration curve as if it were an external calibration curve.

11.6 Sample must undergo TCLP extraction within the following time periods (in days):

	From: Field collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable.

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization

if the waste exceeds the regulatory level.

12. SAFETY

12.1 This test method must be performed in accordance with all applicable quality assurance requirements of the laboratory performing the test and using the data.

12.2 This procedure may involve hazardous materials, operations, and equipment. All personnel who are authorized to perform this procedure must consult and establish appropriate safety and healthy practices and determine the applicability of regulatory limitations prior to use.

12.3 All appropriate precautions for operation of pressurized equipment must be taken. The thermal expansion of water must be taken into account when filling the leach containers.

13. APPLICABILITY

This procedure applies to all employees trained and authorized to perform this test in the VSL.

VSL Technical Procedure

UNCONTROLLED

Title: Glass Characterization

Document: TPI-GC Rev # 1

Documents Incorporated:

TPI04.WVN	Rev #0
TPI-GC.WVN	Rev. # New

Date Issued: 10/91 Rev # 0

Date Revised: 9/92 Rev # 1

Prepared by: Fr. Andrew Buechele Project Manager	Ian L. Pegg Project Manager
Approved by: Pedro B. Macedo Co-Director, VSL	C. Sivaprakash Quality Assurance Officer, VSL

GLASS CHARACTERIZATION

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REVISION TO THE TECHNICAL PROCEDURES MANUAL

Summary of Changes

Document: Glass Characterization Procedures

Rev. # and Date: Rev # 1 Date: 9/92

Document Code: TPI-GC

Section/Page	Location	Change
6.1	Equipment and Supplies	Revised
6.1.3	Calibration of Viscometer	Revised
6.1.4	Sample Control/Traceability	Introduced
6.2.1.3	Calibration of Digital thermometers and thermocouples	Revised
6.2.2.1	Equipment and Supplies	Revised

8.0 Data Analysis
9.0 Record Keeping

DOCUMENT REPLACEMENT RECORD

Glass Characterization

Procedure	Descriptions	Rev #	Replaced in this Manual by:
VSL-GC-1.WVN	Melt Viscosity Determination	1	Section 6.1
VSL-GC-2.WVN	Heat Treatment of Glass Samples	NEW	Section 6.2.1
VSL-GC-3.WVN	Preparation of Sample for Microscopic Examination	NEW	Section 6.2.2
VSL-GC-4.WVN	Procedure for Operation of the Scanning Electron Microscope	NEW	Section 6.2.3

10/91

1.0 PURPOSE

This procedure describes methods, materials, equipment and special conditions required to perform heat treatment, microstructural characterization, and melt viscosity characterization of nuclear waste glass. This procedure provides the means for obtaining verifiable, reproducible, documented data from tests and experiments.

2.0 APPLICABILITY

This procedure applies to all employees trained and authorized to perform heat treatment, microstructural characterization, and melt viscosity characterization of nuclear waste glass. Specific activities covered by this procedure include: (a) specimen preparation for heat treatment; (b) selection of container for heat treatment; (c) temperature measurement and control, thermocouple calibration; (d) control of redox state; (e) establishment of time-temperature schedule for heat treatments; (f) preparation of specimens for optical microscopy; (g) preparation of specimens for SEM examinations; (h) optical microscopy and photomicrography; (i) SEM examination and associated analytical procedures, namely:

1) X-ray EDS characterization, semi-quantitative and quantitative analysis, and elemental mapping.
2) X-ray WDS characterization, semi-quantitative and quantitative analysis, and elemental mapping.
3) Imaging techniques and photomicrography.

4) Image enhancement and processing.

5) Phase identification isolation and quantification through image analysis.

(j) documentation work, logging of images, photomicrographs, and analytical results in laboratory notebooks; (k) viscometer spindle calibration; (l) rotating spindle viscometer operation; (m) viscometer muffle furnace and temperature controller operation; (n) viscometer spindle cleaning procedure; (o) viscosity data analysis software.

This procedure will be implemented by personnel from the VSL.

3.0 SAFETY

3.1 HAZARDS

The definition of a hazardous chemical is given in Section 8.3 of the CUA Chemical Materials Safety Manual, adopted from the OSHA Hazard Communication Standard. The CUA Chemical Materials Safety Manual is hereafter referred to as the CCMSM. This Standard and its application to CUA facilities are described in Sections 8.1 of the CCMSM, with the exceptions noted in Section 8.28. The types of radioactive material encountered during heat treatment and specimen preparation for microstructural characterization include liquid, solid, and fine particulate. Materials used for viscosity determinations are generally monolithic glass bars but will from time to time include fine particulate samples.

3.2 HAZARD MITIGATION

3.2.1 Identification of Hazards

The identification of radioactive hazards shall be facilitated through an education program including the Radiation Health Physics course referred to in Section 4.1 and other specific directives.

All materials received by personnel in the heat treatment and microstructural characterization program shall be properly labeled and identified, if radioactive, by approved yellow radioactive hazard labels. A geiger counter shall be used regularly to monitor levels of radiation in the work area and identify any sources of radiation.

Viscosity measurements involve the use of hot, molten radioactive glass and all appropriate precautions should be exercised. Heat resistent gloves and tongs shall be used for handling furnace covers, spindles, crucibles, etc. Eye protection shall be worn at all times. A face mask is recommended during removal of the crucible from the furnace due to the potential for shattering. Personnel must ensure that the above-furnace exhaust system is operating over the course of all measurements.

Cleaning of the viscometer spindle involves the use of hydrofluoric acid (HF) solutions and personnel shall familiarize themselves with the particular hazards associated with that material (see Section 5.0). Acid resistent gloves, face protection, and aprons must be worn during this procedure. Personnel shall ensure that HF antidote gel is available near the work area before commencing work with HF.

3.2.2 Handling and Storage of Chemicals

Examination of liquid materials (solutions and suspensions) is not part of the microstructural or viscometric characterization program and shall not be carried out in areas designated for heat treatment, preparation, and examination of specimens. The only place where radioactive liquids will be encountered is in the cutting and polishing operations performed on solid specimens. All such work shall be carried out in a sealed glove box connected to an exhaust line through a HEPA filter. All solid materials resulting from such work shall be washed thoroughly in clean solvents and detergent solutions and dried prior to removal from the glove box to insure that no radioactive particulates or liquids are removed from the glove box with the samples. All radioactive wastes and materials contaminated with radioactivity during specimen preparations shall remain in the glove box until they can be removed for disposal or decontaminated using approved procedures.

Solid materials handled in this work shall not produce levels of radioactivity which create a serious danger to the personnel handling them. All standards set forth in the radiation safety manual of CUA by the radiation safety officer shall be adhered to at all times by those handling radioactive materials of any type. Cutting, grinding, polishing or pulverizing of solid radioactive materials shall only be carried out in a glove box using approved procedures.

Examination of particulate radioactive materials shall only be done if levels of radioactivity are low enough to permit handling without special precautions or if the particulates can be embedded in mounting resins or otherwise immobilized to prevent dispersion of such

materials in the environment and their ingestion or inhalation by handling personnel. Such embedding and immobilization and any further preparation likely to produce additional radioactive liquids or particulates shall be done in the glove box using the procedures outlined above for solid materials. Viscometric measurements made on initially particulate samples requires loading of the crucibles in a glove box and closing the crucible before removal to the viscometer furnace. The crucible should remain lightly covered until the sample has become molten.

3.2.3 Emergencies

Any accident which appears likely to release radioactivity in such a form that it can be ingested or inhaled by personnel, contaminate their person or clothing, or be dispersed in the environment shall be reported to supervisory personnel immediately and dealt with using approved techniques. The release must be confined and all contaminated items removed for decontamination or disposal by approved methods.

3.3 PERSONNEL PROTECTION

Personnel protection shall be effected through education in and familiarization with the hazards of and proper methods of handling radioactive materials. Approved radiation film or TLD badges shall be worn by personnel at all times when they are in areas where exposure to radioactivity above background levels is likely to occur, and these badges shall be collected and

processed monthly. A geiger counter or other approved means of detecting and monitoring levels of radiation shall be available to confirm that levels of radiation in the workplace are within acceptable limits and check for possible contamination of objects and surfaces in areas where radioactive materials are handled.

4.0 REQUIREMENTS

4.1 TRAINING

All glass characterization laboratory personnel are trained in the understanding and use of the technical methods that they will use by the laboratory supervisor or by a trained and experienced person designated by the laboratory supervisor. Upon completion of training for a method, a technician is qualified to use that method independent of direct supervision.

5.0 REFERENCES

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6.0 PROCEDURE

6.1 MELT VISCOSITY DETERMINATION

6.1.1 Equipment and Supplies

Qty	Device	Manufacturer	Model #	Calib. Req.
1	Viscometer	Brookfield	Synchralectic	I
1	Tube Furnace	Applied Test Systems		I
1	Controller	Love	Self-Tune Plus	N.A.
1	Spindle		Platinum	II
	Spindle extensions	Brookfield		N.A.
1	Multimeter	Keithley	177	III
1	Compensator	Omega		N.A.
1	Thermocouple	Chromel/Alumel type K		N.A.
1	Thermocouple	Pt/Pt-Rh type S		IV
4	Crucibles (#1,2,3,4)	Engelhard	Platinum	
	Crucibles	VWR	Alumina	
2	Thermometer	Fisher		
	Standard Viscosity Oil	Cannon	S2000	
	Standard Reference Material	NBS	SRM711	

Calibration codes: I. Calibration frequency: Every twenty uses/as necessary; Calibration accuracy ± 7% (see later in this procedure); II. Calibration is performed every twenty uses/as necessary using a standard oil of known viscosity (see later in this procedure); III. Calibration: Externally every two years. IV. Calibration: Every 40 uses/as necessary.

6.1.2 OPERATING PROCEDURE

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A. Viscosity Measurement

(1) Calibrate the spindle using the procedure given in Section 6.1.3.2.

(2) Weigh out accurately 85-110g of the glass sample into a platinum crucible and place it in the furnace.

(3) Turn on the power and set the controller temperature to 1150°C unless specified otherwise by the Project Manager.

(4) After attaining the set temperature, allow the furnace temperature to stabilize (about 7-30 minutes).

(5) Immerse the calibrated spindle into the melt and attach the spindle hook to the viscometer. Ensure that the spindle is completely immersed in the melt and the spindle tip is about 1 cm above the bottom of the crucible.

(6) Insert the Pt/Pt-Rh thermocouple such that the tip of the thermocouple is at the top of the glass melt. Allow furnace temperature to stabilize.

(7) Turn on the viscometer and set it to the lowest rotation speed.

(8) Allow the system to attain equilibrium (at least 60 seconds), and record the reading on the viscometer and the rotation speed in RPM.

(9) Proceed to the next higher rotation speed of the viscometer and continue as in step (8) until either 50 RPM is reached or the viscometer reading is off scale.

(10) Record the temperature indicated by the Pt/Pt-Rh thermocouple.

(11) Set the viscometer RPM to the lowest value and set the furnace to the next temperature of interest. Ensure that the furnace temperature has stabilized as in step (4).

(12) Repeat steps (7) through (11) until all measurements on the sample are completed.

(13) Remove the Pt/Pt-Rh thermocouple from the crucible.

(14) At the end of the viscosity run continue as in Section C.

B. Cleaning of the Spindle and Platinum Crucible

(1) Place cool platinum crucible (with used glass inside) upside down in an alumina crucible.

(2) Place alumina crucible in the Del-Tech furnace and raise temperature to 1150°C for 20 minutes, to allow the glass to drip off the platinum crucible.

(3) Remove crucibles from furnace and let cool. Label alumina crucible.

(4) Don safety equipment and place the spindle and crucible in an HF bath for 48 hours.

(5) Dip the spindle and crucible into the neutralizing solution, rinse thoroughly, and allow to dry.

C. Finishing the Viscosity Run

(1) Turn the viscometer off and set the furnace temperature to approximately 1150°C.

(2) Raise the spindle above the surface of the melt to allow excess glass to drip off for about 20 minutes.

(3) Turn furnace off. Allow to cool.

(4) Remove platinum crucible and spindle from furnace.

(5) Proceed to clean the spindle and platinum crucible according to the procedure given in section B.

6.1.3 Calibrations

6.1.3.1 Calibration of the Viscometer

(1) Weigh out accurately 120 ± 5g of the standard SRM-711 glass into a quartz crucible and place it in the furnace.

(2) Proceed as in section A, steps (3) through (12).

(3) Calculate the measure viscosity as in section 6.1.4 below for each temperature of the standard materials studied.

(4) Calculate the percentage error for each of the measured viscosity readings as compared to the nominal values.

(5) Examine the results and report deviation from the required accuracy of ± 7% to the Project Manager.

(6) Report the results in the viscosity calculation file.

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6.1.3.2 Calibration of the Spindle

(1) Place the standard oil (Cannon Instrument Company S2000 viscosity oil) in a platinum crucible.

(2) Insert the spindle in the oil as described in step (5) of section A.

(3) Turn on the viscometer and record the viscometer readings at the various rotation speeds as in section A from step (7) to (9).

(4) Observe if there exists any eccentricity of the spindle and if necessary replace it.

(5) Measure the oil temperature using a partial immersion thermometer and note it down.

6.1.4 Sample Control/Traceability

(1) All persons submitting samples to the viscometry/conductivity laboratory sign in their sample in the viscometry sample logbook.

(2) If sample is not used immediately, it is stored in the viscosity/conductivity lab. If sample is radioactive, it is stored behind lead shielding.

(3) Used samples (contained in their alumina crucibles) remain the viscosity/conductivity lab until retrieved by the persons submitting these samples. Radioactive samples are stored behind lead shielding.

6.1.5 Computerized Data Analysis

(1) Calculate the value of the constant K_1 using the nominal value of the viscosity of the standard oil and record it in the viscosity calculation file.

(2) From the results of the calibration of the spindle perform a least squares fit on the equation:

$$\tau + n K_1\omega + K_2 = 0$$

where τ is the viscometer reading,
K_1 and K_2 are instrument constants

n = shear viscosity of material under investigation

ω = rotation speed

and calculate the value of K_1.

3) From the viscosity measurements, using the value of K_1 derived from the spindle calibration, determine values of n and K_2 from the above equation by performing a least squares fit on the data at each temperature.

4) Using the data obtained in Step 3, perform a 3 - parameter least squares fit of the equation.

$$\text{Log } n = \frac{A}{T-T_0} + B$$

Where A, B and T_0 are fitting parameters.

5) Record all the raw and analyzed data in the viscosity calibration file.

Procedure for Running the Viscosity
Data Reduction Programs

Procedure

Note: <CR> indicates the "enter" key on the keyboard.

1) Turn the computer, monitor, and printer on.

2) Type C: <CR>.

3) CD\QBASIC <CR>.

4) Type VISCO <CR>.

5) Answer all the prompts and input the data as requested.

6) Repeat the calculation with different values of To, varying To in steps of 10°, until the variance is a minimum.

7) On answering no to the prompt of whether to perform another calculation, a printout of the latest set of data will be obtained and the program will be exited.

8) Turn off the computer.

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Equipment List				
Qty	Device	Manufacturer	Model	Calib. Req.
1	Computer	IBM or any Compatible	Any IBM compatib le	NA
1	Monitor	Any compatible Systems	Any compatib le equivale	NA
1	Keyboard	Compatible	NA	NA
1	Printer	Epson or HP Laser	NA	NA
1	Visco Software Package			

6.1.6 Record Keeping

) Viscometry data is recorded by hand in the appropriate notebook. Notebooks currently use are:

Fernald Viscosity & Conductivity
Viscosity & Conductivity - Glass Simulation
Calibration/Testing
West Valley: Viscosity
Argonne

) Results of the computerized analyses are posted on the v/c lab door for collection ' persons retrieving used samples. A notebook containing all results is stored in or near e v/c lab. Results are also stored by sample name in the v/c lab computer.

6.2 Microstructural Characterization of Glasses

6.2.1 Heat Treatment of Glass Samples

6.2.1.1 Equipment and Supplies

Qty	Device	Manufacturer	Model	Calib. Req.
1	Furnace	Lindberg	51848	NA
1	Annealing	Neytech	85M	NA
	Thermocouples		Type K	I
1	Digital Thermometer	Fluke	52	I
	Clay Combustion Crucibles	Leco		NA
	95% Platinum/5% Gold Crucible			NA
1	Crucible tongs			NA

Notes: I. Calibration to be carried out every six months or on every replacement with a new thermocouple. Calibration Accuracy: ± 10°C.

6.2.1.2 Procedure

Note: If the sample is radioactive steps 1-4 are to be carried out in the left hand glove box in Room 38

1. Place the glass sample in a plastic bag.

2. Break the glass into pieces small enough to enter the crucible by shattering the sample with a hammer.

3. If the sample comes in a crucible, remove pieces of the shattered crucible before continuing.

4. Transfer the glass pieces into the crucible by hand using a double pair of gloves.

5. Turn on the Lindberg Furnace.

6. Insert the thermocouple into the furnace through the hole in the door.

7. Set the controller on the front of the furnace to reach a temperature of 1100°C.

8. Wait for about 15 minutes for the temperature to reach 1100°C and stabilize.

9. Remove the thermocouple from the door of the furnace and open the door.

10. Place the crucible containing the glass sample into the oven, locate it in the proximity of the inserted thermocouple and close the door.

11. Replace the thermocouple in the furnace through the hole in the door.

12. Press the RECORD Button on the digital thermometer.

13. Wait for the required amount of time while the glass is remelted at 1100°C (Minimum 1 hour).

14. Press the VIEW button on the digital thermometer to check that the maximum and minimum temperatures fell in the range of 1125 to 1075°C. If not, consult the Project Manager.

15. Adjust the Lindberg Furnace controller to achieve the required temperature for heat treatment (this is generally in the range of 600 to 1000°C).

16. When the required temperature for heat treatment is reached record the time in the appropriate laboratory notebook.

17. Press the RECORD button on the digital thermometer.

18. Let the crucible remain in the furnace for the required time as determined by the Project Manager.

19. When the heat treatment is complete press the VIEW button on the digital thermometer to ascertain that the maximum and minimum temperatures did not vary by more than ± 10°C from the required temperature for heat treatment.

20. Consult the Project Manager if deviations are more than 10°C in the above step.

21. Ensure that the temperature of the annealing furnace is maintained between 460 and 560°C.

22. Remove the thermocouple from Lindberg Furnace.

23. Open the door of the furnace and remove the crucible using crucible tongs.

24. Transfer the crucible to the annealing furnace and securely latch the door of the annealing furnace.

25. Let the crucible containing the sample remain in the annealing furnace for a minimum period of 1 hour.

26. Remove the crucible from the annealing furnace using crucible tongs and allow the glass sample in the crucible to cool to room temperature.

6.2.1.3 CALIBRATION OF DIGITAL THERMOMETERS AND THERMOCOUPLES

Note: The Gold Furnace, controller and associated thermocouple, the Keithley 177 calibrated Microvoltmeter, and NIST calibrated Type S Measuring Thermocouple are needed for this procedure. During the calibration procedure, 3 thermocouples will be inside the Gold Furnace at the same time. These are the control thermocouple (connected to furnace controller), the NIST thermocouple and the test thermocouple (thermocouple being calibrated).

1. Insert the control and NIST thermocouples in the furnace to immersion depths where the cross-section of the furnace possesses a uniform axial temperature profile.

2. Immerse reference junction of NIST thermocouple in ice/water bath. Be sure that unmelted ice is always present in contact with the reference junction during the entire calibration procedure.

3. Insert the test thermocouple into the same end of the furnace as the NIST thermocouple. It should be parallel to the NIST thermocouple and inserted to the same depth.

4. Connect the NIST thermocouple to the microvoltmeter.

5. Connect the test thermocouple to the digital thermometer or controlling device it is being calibrated with. The two will always be calibrated as a unit. If the thermometer is a multi-channel unit, it is best to connect the test thermocouple to the channel it will be used with.

6. If a thermocouple is being calibrated on its own, connect it to the same microvoltmeter

as the NIST thermocouple. Alternate between the two when taking readings. Place a thermometer at the voltmeter terminals and record the ambient temperature with each voltmeter reading. Use this to correct for room temperature error with the aid of standard tables.

7. Set the furnace temperature to the desired value and turn on the power.

8. Wait about 10 minutes until the temperature equilibrates.

9. Take 3 readings each from the test and NIST thermocouples at 5 minute intervals.

10. If more than one thermocouple is to be tested place the next thermocouple to be tested in the furnace, allow the temperature to equilibrate and repeat step 8.

11. Set the furnace to the next desired temperature and proceed with steps 7 through 9.

12. Repeat the calibration for a total of 4 temperatures that include the temperature range required.

13. At each temperature setting compare the temperatures corresponding to the readouts from the reference thermocouples and test use the calibration table for the NIST thermocouple to convert its microvoltmeter readings into temperatures. Use standard tables (e.g. in Omega temperature handbook) if necessary, to convert test thermocouple readings to temperatures (i.e. if the test thermocouple is not being calibrated with a digital thermometer as a unit. If the two temperature measurements deviate by more than 10°C consult the Project Manager.

6.2.2 Preparation of Samples for Microscopic Examination

6.2.2.1 Equipment and Supplies

Qty	Device	Manufacturer	Model	Calib. Req.
1	Saw	Buehler	11-1180 Isomet	NA
1	Polisher	Buehler	69-1000 Minimet	NA
1	Evaporator	VSL		

Thermoplastic cement,
hot plate, sample mounting
Fixtures for saw, 1" dia.
aluminum or brass polishing
stubs

Lubricants:

Dia-cut
Kerosene or isocut fluid, or Lapping oil on Nylon; use only lapping oil on texmet
De-ionized water

Grinding/Polishing Media:

30μm Grid-abrade
12μm Grid-abrade
6μm Grid-abrade
15μm diamond paste on Texmet or preferably nylon (perforated texmet may better in some cases)
6μm diamond pasted paste on Texmet
1μm diamond paste on Texmet
0.05μm gamma alumina on microcloth

Cleaning supplies:

Ultrasonic cleaner, solution of Mr. Clean, tap water, deionized water, Methanol, acetone, tissue wipers.

Sample coating supplies:

Carbon electrodes, conductive paint.

6.2.2.2 Procedure

A. CUTTING AND POLISHING

Note: All cutting and polishing of radioactive samples must be performed in the two interconnected glove boxes located in Room 38.

1. If the sample is in a LECO clay crucible

(a) Cement the crucible containing the glass sample using thermoplastic cement to the

glass plate side of the sample holder.

(b) Attach the mounted sample to the sample holding arm of the Buehler Isomet Saw.

(c) Adjust the weights on the sample holding arm to produce moderate pressure of the sample against the saw blade.

(d) Position the sample over the blade of the saw so that the cut will be made in the region of the sample from which the slice is to be taken.

(e) Turn the power on and adjust the speed of rotation of the saw blade to produce the desired rate of cut.

(f) Ensure that there is sufficient quantity of Dia-cut lubricant for the blade in the reservoir.

(g) Gently lower the arm of the saw by hand until sample contacts the rotating blade.

(h) Cut through the entire sample but do not allow the blade to pass through the glass plate to which it is cemented.

(i) Lift the arm of the saw and repeat the cutting process after repositioning the sample for the desired thickness (2 or 3 turns of the micrometer screw).

(j) Remove the slice and free it of all fragments of clay.

(k) Proceed to Step 3.

2. If the sample is in a 95% Platinum - 5% gold crucible--

(a) Turn the crucible upside down and tap it gently so that the sample drops out of the crucible.

(b) Cement the sample using thermoplastic cement to the glass plate side of the sample holder.

(c) Proceed as in steps 1 (b) through 1 (j).

3. Wash the slice and remainder of the sample together with the sample holder in clean water and wipe dry to remove any fine particles before taking out of the glove box.

4. Remove the remainder of the sample from the mounting plate by placing it on a hotplate to melt the thermoplastic cement.

5. Store the remainder of the sample in an appropriately marked bag.

6. Mount the glass slices on a 1" diameter aluminum or brass polishing stub using thermoplastic cement.

7. Polish the glass slices using the Buehler Minimet Polisher.

8. Samples may also be embedded in epoxy or other resin using 1" or 1 and 1/4" O.D. molds. Mix the resin according to directions, and coat the mold with a light coating of petroleum jelly or mold release agent. After curing and before polishing on the Minimet a 3/16" hole must be drilled at dead center in the rear face of the embedded sample (use lathe in shop) to within 1/4"-3/8" of front face.

Note: Each step of the polishing process should be carried on as long as required to remove the scratches from previous step. A typical polishing procedure is given below:

(a) Grind the glass sample on a 30μm diamond Grid-abrade using water as the lubricant. Set the polisher to a speed setting of 6 and time setting of 5 and apply maximum pressure. Continue this process until the surface of the sample shows full contact with the grid surface.

(b) Clean the glass sample in an ultrasonic cleaner first using a solution of Mr. Clean and water for 30 seconds to 1 minute and then using water alone.

(c) Repeat step 7 (a) using a 12μm Grid-abrade, half pressure, speed setting of 5 and time setting of 5. Repeat 2-4 times until scratches from the previous step are removed.

(d) Carry out the cleaning process as in step 7 (b).

(e) Repeat the grinding/polishing process in step 7 (a) using a 6μm Grid-abrade, half pressure, a speed setting of 5 and a time setting of 5. Repeat 1-2 times to remove any scratches.

(f) Clean the glass sample as in step 7 (b).

(g) In the following steps, when kerosene oil or isocut are called for, use only enough to produce a small ring of fluid at the perimeter of the polishing pattern.

(h) Polish the sample using 15μm diamond paste on Nylon with Lapping oil as the lubricant. Do not use kerosene or Isocut on Texmet. It may be used on Nylon. Apply full pressure, use a speed setting of 5-9, and a time setting of 5. Repeat as necessary until scratches from the previous step are removed.

(i) Clean the sample with methanol or acetone using tissue wipers, and then in the ultrasonic cleaner using Mr. Clean and water or Ultramet solution. Again rinse with tap water in the ultrasonic cleaner before going to the next step.

(j) Repeat the polishing process in step 7 (h) using 6μm diamond paste on nylon with Lapping oil as the lubricant. Apply full pressure, speed 5 and a time setting of 9. Repeat till scratches from the previous step are removed.

(k) Clean as in step 7 (i).

(l) Repeat the polishing process in step 7 (j) using 1 μm diamond paste on Texmet with lapping oil as the lubricant. Apply full pressure, speed setting of 5 and a time setting of 9. Repeat till scratches from the previous step are removed.

(m) Repeat the cleaning process given in step 7 (i).

(n) Repeat the polishing process in step 7 (j) using 0.05μm gamma alumina on microcloth with deionized water a lubricant. Use a speed setting of 5 and a time setting of 4. Repeat till scratches from the previous step are removed.

(o) Repeat the cleaning process given in step 7 (i).

(p) Rinse the sample finally with tap water in the ultrasonic cleaner and wipe dry before removing the sample from the glove box.

B. COATING WITH CARBON

Note: I: If the evaporator system is in the shut down condition open the supply and return cooling water valves.

II: Specimens should be prepared for coating by painting the edge of the specimen and any remaining thermal cement with conductive paint so that a continuous conduction path will exist between the sample surface to be observed and the mounting stub.

1. Close the vent on the belljar.

2. Close the roughing valve.

3. Close the high vacuum valve.

4. Open the foreline valve.

5. Close the mechanical pump vent.

6. Start the mechanical pump.

7. Switch on the thermocouple gauge and note the foreline pressure.

8. Wait for the foreline pressure to reach 100 millitorr or less and then switch on the

diffusion pump.

9. Wait for 15 minutes.

10. Switch on the high vacuum gauge and verify that the pressure is in 10^{-5} torr range.

11. Vent the belljar by opening the belljar vent valve.

12. Cautiously unseat the belljar from the base plate and lift it clear of the work area.

13. Loosen the spring-loaded electrode holder and swing it aside.

14. Insert a 1/8th inch flat-ended carbon electrode in a stationary electrode holder and tighten.

15. Insert a 1/8th inch carbon electrode with a sharpened point in the spring-loaded electrode holder and swing the holder back into its operating position adjusting the location of the sharpened carbon to meet with the flat surface of the opposite carbon electrode.

16. When proper positioning has been achieved tighten the sharpened carbon in its holder and tighten the holder.

17. Release the restraint on the spring-loaded electrode holder allowing the carbons to come into contact.

18. Place the samples to be coated directly under the point of contact.

19. Place the polished brass plate adjacent to specimens. This is to be used as an estimator for the coating thickness.

20. Lower the belljar and make sure it is firmly sealed against the base plate by applying downward pressure by hand from the top.

21. Close the belljar vent.

22. Close the foreline valve.

23. Switch on the thermocouple gauge to monitor belljar pressure.

24. Open the roughing valve.

25. Wait for pressure in the belljar to drop to 10 millitorr or less.

26. Close the roughing valve.

27. Open the foreline valve.

28. Switch the thermocouple gauge to read foreline pressure.

29. Open the high vacuum valve slowly (Do not allow the foreline pressure to rise above 50 millitorr).

30. At the same time monitor the high vacuum gauge and try to keep the pressure below 10^{-3} torr by controlling the rate of opening of the high vacuum valve.

31. When the high vacuum valve is fully opened wait at least one minute until the pressure on the high vacuum gauge reads well below 10^{-4} torr.

32. Ensure that the current control knob top of the evacuator is fully counter clockwise.

33. Turn on the Filament Transformer Switch on the lower panel of the vacuum system and advance the current control until a dull red glow is visible at the contact point of the carbons.

34. Allow the system to outgas at this current setting for about 1 minute.

35. Advance the current control knob while monitoring the system current on the top panel meter until the current reaches about 30 amps. (Avoid looking directly at the point of contact between carbons. Some slight sparking is normal when sufficient evaporating current has been reached.)

36. Evaporate for 15 to 30 seconds until reflection on the polished brass plate dims slightly.

37. Turn the current control fully counter clockwise and wait for a minute for the system to cool slightly.

38. Switch off the Filament Transformer.

39. Close the high vacuum valve and vent the belljar.

40. Remove specimens, replace belljar and evacuate the system as in steps 20-31.

<u>To Leave the System in Operating Condition Ensure that:</u>

(a) Belljar vent and roughing valve are closed.

(b) High vacuum valve is open.

(c) Foreline valve is open.

To Shut down the System:

(a) Switch off the diffusion pump

(b) Wait for 15 minutes

(c) Close the high vacuum valve

(d) Close the foreline valve

(e) Turn off the supply and return cooling water

(f) Turn off the mechanical pump

(g) Open the mechanical pump vent

(h) Switch off all gauges

6.2.3 Procedure for Operation of the Scanning Electron Microscope

Manufacturer: JEOL
Model: JSM-35(C)

X-RAY ANALYZER AND IMAGE ANALYSIS SYSTEM

Manufacturer: Tracor Northern (TN)
Model: TN5500

A. OPERATION PROCEDURE FOR SEM

Note: The main power key switch for the SEM is always left ON. When the instrument is not in use the white switch at the lower right on the front panel of the instrument is left in the "unattended operation" position.

1. Press the white toggle switch down from the "unattended operation" position to the normal position.

2. Mount the specimen in the appropriate stage and thread the stage onto the specimen exchange rod.

3. Withdraw the rod fully into the specimen exchange cap until it locks in place.

4. Introduce the mounted specimen into the SEM through the airlock as follows:

   (a) Go to standby setting as below:

      (i) Ensure SEI-BEI switch is in the middle position, and no lights on SEI unit are ON

      (ii) Ensure that Gun Filament knob is turned all the way counter-clockwise

      (iii) Ensure that HV is shut off

   (b) Put the specimen exchange cap on the airlock chamber.

   (c) Push the red button next to the airlock chamber.

   (d) Wait until the light in the button goes OFF.

   (e) Set the specimen stage control knobs to the following positions: (Tilt: 0 degrees, x: 7.5, Y: 12.5, Rotate: 000)

(f) Open the sliding door all the way.

(g) Push the specimen into the specimen chamber until the stage seats fully on the tapered mounting post.

(h) Unscrew the rod, and withdraw it to the locked position.

(i) Close the sliding door.

(j) Push the red button, remove the airlock chamber specimen exchange cap and store on its holder.

(k) Cover the airlock with the protective dust cover.

5. Select the working distance--15mm for high resolution work and 39mm for routine work and x-ray analysis.

6. Turn on the high voltage switch.

7. Select the KV required (Normally 25 KV is selected for most routine and x-ray work).

8. Turn SEI-BEI switch to SEI. Two red lights on the SEI unit come on.

9. Set the condenser lens knob according to the KV as below: (guidelines only--readjustment may be required)

KV	Lens Position
30 KV or higher	2:00
20 KV - 29 KV	1:00
10 KV - 19 KV	12:00
1 KV - 9 KV	10:00

10. Advance the brightness and contrast knobs on the display monitor to at least half-scale.

11. Switch magnification to minimum (10 or 20).

12. Push in "Rapid 2" and select "Line Profile" by turning the switch (□,•,-) to ("_____").

Note: If you do not see the line on the screen adjust the "Brightness" below the screen and then "Brightness" on SEI

13. Increase Gun Filament control until the filament is saturated. Watch the movement of "Line Profile".

    Note: i) If the movement of line profile is too little, increase the "contrast" on the SEI unit.

    ii) The lifetime of a filament will be shortened if the Gun Filament knob is turned beyond the saturation point.

14. Adjust "Gun Bias" until "Load Current" is around 100μA. Readjust the filament saturation. When 10KV or lower is used, use the maximum load current which is less than 100μA.

15. Adjust the two upper "Beam Alignment" knobs and bring the signal line upwards on the monitor as much as possible. (Gun tilt alignment).

16. Adjust the "Medium" objective lens knob until the "Line Profile" has sharp peaks. (Focusing the image).

17. Adjust the "Brightness" and "Contrast" knobs on the SEI unit until the magnitude of the line Profile is about 1½ inch and the bottom of the line Profile is about the middle of the monitor screen.

18. Turn the switch (□,•,-) to "□" (picture). An acceptable image should be observed on the monitor. If there is no image or the contrast of the image is too low, adjust the CONTRAST on the display monitor.

19. Go to 20x magnification at 15mm working distance (or 10x at 39mm.) Check the position of the oval shaped image on the monitor screen. Adjust the two lower "Beam Alignment" knobs until this oval image is approximately at the center of the screen. Readjust the two upper "Beam Alignment" knobs for the maximum brightness.

    Note: This step is usually necessary only after changing a filament.

20. An acceptable image should be observed on the monitor. In order to lessen the noise level at "Rapid 2", turn the condenser lens knob 1-2 settings counter-clockwise. In order to obtain a sharper image at a magnification higher than 10000x, turn the condenser lens knob 1-2 settings clockwise.

21. Examine the image on the monitor and identify features of interest.

B. <u>PROCEDURE FOR OBTAINING A PHOTOGRAPH ON SEM</u>

1. Select the area to be photographed.

2. Focus the image carefully by going to higher magnification and using the "Rapid 1" (10 times higher magnification for low magnification 2-3 time higher magnification for high magnification. For magnification above 20000x focus should be done at the magnification in use.

3. Return to the desired magnification for photographing.

4. Push in the "Wave Form Monitor" and "Slow 1" buttons. If nothing appears on the screen, adjust "Brightness" knob below the screen.

5. Adjust the "Brightness" and "Contrast" knobs on the SEI unit. Set the wave form between the top line (white) and the bottom line (black) for the maximum contrast. If the soft contrast is preferred, set the signal between the top line and the second line from the bottom (grey).

6. Push in the "Normal" button in order to return to the image.

7. Insert film in the camera.

8. Push "Photo" and wait for light in "Photo" button to go off.

9. Process the film.

C. STIGMATOR ADJUSTMENT FOR MAGNIFICATION ABOVE 2000x ON SEM

1. Rock the fine focus knob back and forth between under and over focus conditions. Observe that the flow (or striation) of the image will be in the directions at right angles to each other as one goes from under to over focus.

2. Adjust the focusing just to the focus where the flow cannot be seen. This focus is somewhere midway between under and over focus.

3. Increase the "Magnitude" knob of the stigmator by half a turn, then adjust the "orientation" knob until the image becomes sharper.

4. Adjust the "Magnitude" and the "Orientation" controls in order to make the image sharper and repeat this procedure until no improvement in image quality is apparent. At this point going to under or over focus should produce only an uniform smearing of the image rather than perpendicular directions of flow.

5. Adjust the "Fine" objective lens knob for the sharpest image.

D. TO APPLY CORRECTION FACTOR FOR MAGNIFICATION WHEN THE SPECIMEN SURFACE IS NOT FLAT

Note: The working distance remains constant while any of the specimen stage movements are made (x and y translation, tilting and rotation), for a specimen with a relatively flat surface. Therefore, the magnification is always calibrated. In cases when a specimen has no flat surface proceed as follows:

1. In the case that a specimen has a roughness up to about 3mm, adjust the fine-z on the specimen stage such that the area of interest ia at the same level as the top of the specimen holder which is the calibrated level.

2. In cases when the area of interest on a specimen is either much higher or lower than the calibrated level, (more than 3mm roughness), apply a correction factor to the magnification as below:

   a) Measure the vertical distance between the area of interest on a specimen and the top level of the specimen holder which is the calibrated level

   b) Read the correction factor from Figure 2 of section SEMINST-5 of the JSM-35C Scanning Microscope Operator's manual

E. PROCEDURE FOR X-RAY ANALYZER AND IMAGE ANALYSIS SYSTEM (TN)

Note: Normally the TN computer is left ON at all times. When not in use, the SYQUEST cartridges are removed, and the Monitor and Video Copy Processor (VCP) are left OFF. (The rear panel switch of the VCP is left on at all times).

(i) To Start the TN System:

1. Insert Master Cartridge in Drive 1, close the door, wait until the light on the front panel turns from red to green.

2. Turn on the Monitor.

3. Turn on the front panel power switch of the VCP.

4. Ensure that the VCP is configured for analog RGB operation, Frame Mode, and Monitor Mode.

5. If data is to be stored insert appropriate disks in Drives 2, 3, and/or 4.

6. If the TN system does not come ON, carry out the following steps:

a) Ensure that the Printer in ON LINE

b) Press the RESET Switch

c) If still no response is obtained, turn the main power switch off, wait for 15 seconds, and turn it on again

d) If still no response is obtained, contact the Project Manager

(ii) X-ray Analysis:

1. Ensure that the specimen is at a working distance of 39mm.

2. Insert the x-ray detector (EDS) to the position marked 45.7mm.

3. Center the object of interest in the field of view on the SEM unit.

4. Set up Pulse Processing as below:

a) Press the SET UP on the Soft-pad.

b) Use the Omni control knob to select the desired program.

c) Press the "Enter Dialogue" soft key.

d) If the spectrum is to be obtained in the half memory for a voltage range of 0-10 Kev, select 2048 for the number of channels. If the spectrum is to be obtained for a voltage range of 0 to 20 Kev select 4096 channels.

e) Press the "RETURN" key.

f) Select the amplifier count rate: for maximum resolution use "LOW," for minimum acquisition times use "MED," or "HIGH."

g) Press RETURN.

h) When "Set up Complete" appears on the screen, press the "RUN" button on the soft-pad.

5. Set up the Pulse Height Analyzer (PHA) as below:

a) On the TN press the ACQUIRE button on soft-pad.

b) Select Program 1 (Display PHA status).

c) Press the RUN key on the soft-pad.

d) Check the deadtime displayed and adjust the condenser lens current on the SEM until deadtime reads about 30%.

e) Press the EXIT soft key.

f) Refocus the image if necessary.

6. Acquire an x-ray spectrum as below:

a) Position the beam on the object of interest (SEM).

Note: If the object of interest is smaller than about 5μm in diameter or thickness, the spectrum collected will not be exclusively from the object of interest, but will include some of the adjoining matrix as well. Operator's discretion is required in interpreting such a spectrum.

b) Press the ACQUIRE key on the soft-pad.

c) Enter the parameters for spectrum acquisition.

d) When "Set up complete" appears on screen, press the RUN key on the soft-pad.

e) Note that spectrum acquisition will begin. Accumulated spectral data will be displayed and updated during acquisition.

f) To halt the acquisition prematurely press the STOP key on the soft-pad.

g) Note that acquisition will terminate automatically when the condition set in step 6 (c) is satisfied.

h) Identify peaks by pressing the MARKERS soft-key and the Z-SELECT soft key. Use omni control on keyboard to scan the periodic table.

7. To print the spectrum on C.ITOH printer carry out the following steps:

a) Press the OUTPUT key on the soft-pad.

b) Select the PLOT SPECTRUM program.

c) Select plot size (one half page is usual).

d) Press RETURN. "SET UP COMPLETE" should appear.

e) Set paper on printer at top of the page.

f) Press the RUN button on the soft-pad. A spectrum will be printed out on the printer.

8. For microanalysis carry out the following steps:

a) Select the area to be analyzed.

b) Collect the spectrum in the first half of memory.

c) Load the SQ program.

d) Identify elements present.

e) Set up definitions.

f) Select elements to be analyzed.

g) Input the conditions of analysis. (Refer to the Tracor-Northern Operating Manual).

h) Type the command "QUANTIFY" followed by RETURN.

i) Results of analysis will be printed on the C.ITOH printer when complete.

iii) <u>IMAGE ANALYSIS:</u>

<u>To acquire a Video Image on the TN System:</u>

1. Press the upper sector of the "ENABLE" rocker switch on the trackball box.

2. Load the VISTA program.

3. Use the following soft-key sequence:

- Acquire
- Video
- Image 1 (or 2) as desired
- Select Resolution (128, 256, 512)

The image will now be acquired and displayed.

To determine an AREA Fraction

1. Display the image to be analyzed.

2. Use the soft key sequence "Process," "Gray," "Segment."

3. Attach the cursor to one extreme of the video level of the feature or phase of interest.

4. As the cursor is moved, video levels on the image between the cursor and the "attach" point will be colored in. Continue until the entire feature or phase is just colored in.

5. Read the percent of the image colored in directly off the display.

TO ACQUIRE X-RAY MAPS:

1. Use the soft-key sequence "SETUP," X-RAY."

2. Input the parameters for x-ray map acquisition.

3. Redefine any regions of interest that may result in ambiguous results.

4. Acquire maps using the soft-key sequence: "Acquire," "Maps (x-ray)," "64" (or "128" for higher resolution.)

5. Maps will be displayed as acquired, one line at a time.

F. SHUT DOWN PROCEDURE
For SEM unit

1. Set the SEI detector to OFF position.

2. Set the magnification knob to maximum.

3. Set the Scan Generator knob to Slow 2.

4. Set the CONTRAST and BRIGHTNESS knobs on the display monitor to minimum.

5. Turn the Gun Filament knob fully counter-clockwise.

6. Switch off the Accelerating voltage.

7. Switch the SEM unit to "unattended operation" by pushing the white button up.

For the TN system

1. Press the "Drive 1" button, wait until the flashing red light on the unit goes out.

2. Remove disk.

3. If a disk is present in Drive 2, repeat steps 1 and 2 above for it.

4. Remove floppy disks (if any) from drives 3 and 4.

5. Turn off the monitor.

6. Turn off the Video Copy Processor at the front panel switch.

7. Leave the computer ON.

SECTION 7.0

SPECIMEN CONTROL/TRACEABILITY

Specimens for glass characterization are obtained from the Glass Preparation Laboratory. Samples are recorded in WVCM, UTh or Misc Radioactive glass logs while leaving the Glass Preparation Laboratory. The recipient of the glass dates and signs the log and records the receipt in Log in the Electron Microscopy Laboratory. Samples prepared for SEM observation are stored in the SEM lab after observations are completed

SECTION 8.0

DATA ANALYSIS

Microstructural characterization studies consist of observing the crystal phases and carrying out the semi-quantitative analysis by Energy Dispersive Spectrometry. Output from this study consist of SEM micrographs of the crystal phases and spectra of the bulk glass and the phases. The observations made of the phases along with supportive micrographs and printed spectra

entered in the laboratory notebook maintained by the Project Manager. Data Analysis consist mainly of identifying and quantifying the crystal phases and confirming the identification with the EDS spectra. Technical literature, and textbooks in mineral chemistry are used for the identification of the crystalline phases.

SECTION 9.0

RECORD KEEPING

Details of heat treatment and sample preparation for Scanning Electron Microscope are recorded in the laboratory notebook "Heat Treatment and SEM Sample Preparation". Glass microstructural data and observations are recorded in a separate laboratory notebook. Formats for recording the crystalline phases observed are included in the Appendix at the end of this section.

Form VSL #18: **PHASE CONTENT of Fernald Glasses** *Laboratory:* SEM *Room:* 434

Full GLASS ID:	Heat treatment Temperature:	Heat treatment Time:

PHASES	CONCENTRATION OR RANGE (VOL%)	OBSERVATIONS ON MORPHOLOGY OR OTHER CHARACTERISTICS
Spinel		
Haematite		
Magnesium Silicate		
Augite		
Calcium Silicate		

Remarks:

Performed by:	Verified by:	Date:	Logbook:	Page:

VSL Technical Procedure TPI-SH *Rev #0* *DATE 06/92*

Form VSL #08: **PHASE CONTENT** *Laboratory:* SEM *Room:* 434

Full GLASS ID:	Heat treatment Temperature:	Heat treatment Time:

PHASES	CONCENTRATION OR RANGE (VOL%)	OBSERVATIONS ON MORPHOLOGY OR OTHER CHARACTERISTICS
Spinel		
Haematite		
Lithium Phosphate		
Augite		
Calcium Phosphate		
Rh, Pd, Ru		

Remarks:

Performed by:	Verified by:	Date:	Logbook:	Page:

UNCONTROLLED

Title: MELT CONDUCTIVITY DETERMINATION

Document: VSL-7.1-ANL

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Revisions

Rev. #	Date
NEW	11/90

Prepared by: Robert K. Mohr
Project Manager

Approved by: Pedro B. Macedo
Co-Director, VSL

Richard Sassoon
Quality Assurance
Officer, VSL

MELT CONDUCTIVITY DETERMINATION

Equipment and Supplies

Qty	Device	Manufacturer	Model	# Cal.Req
1	Controller	Love	49	N.A
1	Tube Furnace	VSL		I
1	Split Furnace	ATS Inc.	3320	I
1	Controller	Eurotherm	818P	N.A.
1	Multimeter	Keithly	177	II
1	Compensator	Omega		I
1	Thermocouple		Chromel/ Alumel type K	III
2	Thermocouple		Pt/Pt-Rh type S	I
	Thermocouple Protection tubes			N.A
	Crucible	U.S Quartz		
	Crucible	Engelhard	Platinum	N.A.
1	Thermometer	Fisher		
	Standard Ref. Mat.	NBS	SRM711	
1	Conductivity Probe	VSL	Pt	I
1	Impedance analyzer	Hewlett Packard	4194A	I

I. Calibration frequency: Every six months or sooner if required
Calibration accuracy +_ 7% (see later in this procedure)

II. Calibration: externally every two years

III. Replace after every third run in order to minimize oxidation.

PROCEDURE

A. Conductivity Measurement

(1) Perform instrument compensation using shorted and open terminals.

(2) Measure a precision resistor in the range of the glass resistance, the value to be within 1% of the quoted value.

(3) Perform instrument compensation using shorted and open probe.

(4) Weigh out enough glass to make up ~ 85 ml of melt (based on the density)

(5) Melt the sample at the upper temperature specified by the Project Manager

(6) Lower the probe electrodes into the melt (the electrode immersion depth is ~0.75 cm)

(7) Measure the resistance of the melt at a frequency of 100 Hz

(8) Lower the temperature and repeat step (7) at ~50°C intervals over the temperature range specified by the Project Manager

(9) Raise the temperature and repeat step (7) at ~100°C intervals over the measurement range

(10) Calculate the melt resistivity using the measured resistance and cell constant.

(11) Fit the data to a Fulcher equation. All data points should be within 20°C of the corresponding resistivity data point obtained from the Fulcher fit.

B. Cleaning of the Probe and Platinum Crucible

(1) Raise the probe above the melt and let the excess glass drip into the crucible

(2) Place the probe in a sealed can to cool to room temperature

(3) After the probe has cooled, place it in an HF bath to remove the remaining glass

(4) After the glass has been removed dip the probe in neutralizing solution, rinse it with deionized water and allow to dry

(5) Allow the furnace to cool to room temperature

(6) Remove the crucible and place it in a furnace at up to 1500°C

(7) After the glass temperature has stabilized pour out the glass into a clay crucible for disposal

(8) Place the platinum crucible in a sealed container to cool

(9) After the platinum crucible has reached room temperature place it in an HF bath to remove the remaining glass

(10) After the glass has been removed dip the crucible in neutralizing solution, rinse it with deionized water and allow it to dry

C. Calibration of the Conductivity Measurement System

(1) Perform instrument compensation using shorted and open terminals.

(2) Measure a precision resistor in the range of the glass resistance, the value to be within 1% of the quoted value.

(3) Perform instrument compensation using shorted and open probe.

(4) Measure the complex impedance of NaCl solutions of .01, .1 and .5 normality at 100, 200, 400 1000, 2000, 10,000, 40,000, and 100,000 Hz.

(5) Measure the temperature of the solutions using a thermometer.

(6) The probe immersion depth shall be 0.75 cm.

(7) Plot the real versus the imaginary impedance at each frequency and extrapolate to infinite frequency to obtain $R(\infty)$.

(8) Calculate the solution conductivities, σ, using the relations due to Stogryn (IEEE TRAN on MICROWAVE THEORY and TECH, Aug 1971 p733-736).

(9) Calculate the cell constant (L/A) from the relation: $L/A = R(\infty)\sigma$.

(10) Perform procedures A. (4)-(10) using SRM 711 glass

(11) The difference between reported and calculated resistivities of SRM 711 shall be within $\pm$ 15%.

Title: **PROCEDURE FOR ANALYSIS OF TCLP EXTRACTS**

Document: TPI-TCLP.AL

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PROCEDURE FOR
ANALYSIS OF TCLP EXTRACTS

1.0 Scope

This procedure describes the analytical methods for analyzing the TCLP extract samples. The TCLP extract samples are prepared in the VSL Solution Chemistry (PCT/TCLP) laboratory using the VSL procedure (VSL-8.1.ANL) based on EPA TCLP Method 1311. These extract samples are received in the VSL analytical laboratory for analysis of inorganic contaminants listed in Table 1.

This procedures describes the steps involved in the preparation of samples, quality control operations, preparation of reference standards, analysis on DC Plasma Spectrophotometer, data calculation and reporting.

2.0 Applicability

This procedure applies to all employees trained and authorized to perform this analysis in the VSL.

This analysis method provides data useful for evaluating the toxicity of a solid. A solid waste exhibits the characteristic of toxicity if, using this analytical method, the extract from a representative sample of the waste contains any of the contaminants listed in Table 1 at a concentration equal to or greater than the respective value given in that Table.

Table 1 - Maximum Concentration of Contaminants for the Toxicity Characteristic

Contaminant	Regulatory Level (mg/L)
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Mercury	0.2
Selenium	1.0
Silver	5.0

3.0 SAFETY

This analytical method must be performed in accordance with all applicable quality assurance requirements of the laboratory performing the analysis and using the data.

This procedure may involve hazardous materials, operations, and equipment. All personnel who are authorized to perform this procedure must consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

3.1 Hazard Identification

All samples received at CUA-VSL and handled at VSL laboratories are treated as potentially hazardous. At VSL one encounters both chemical and radioactive hazardous materials.

The "hazardous chemicals", referred to in this procedure, are defined in Section 8.3 of the CUA Chemicals Materials Safety Manual (CCMSM). A copy of CCMSM is available in every VSL laboratory.

Procedures for identifications and handling of hazardous materials as applied at VSL are prescribed in sections 8.22, 8.23, 8.24 and 8.25 of CCMSM and in sections 8.9, 8.10, 8.11, 8.12, 8.13 and 8.14 of the CCMSM.

A major element of hazard identification is the proper use of labels. At VSL, labeling of chemicals and other hazardous substances is done in accordance with Section 8.4 of the CCMSM, as well as in accordance with Materials Safety Data Sheets (MSDSs) provided by the manufacturers of the chemicals and other hazardous materials. Every VSL laboratory maintains the "work place chemical list". The list is made available to employees in accordance with Section 8.31 of CCMSM.

3.2 Handling and Storage of Chemicals

Handling of chemicals should be conducted according to Section 8.6 of the CCMSM. Storage of chemicals should follow section 8.8 of the CCMSM.

Special precautions should be taken in handling and storage of incompatible chemicals, solvents, compressed gases and radioactive materials in accordance with Sections 8.10, 8.12, 8.13, and 8.14 of the CCMSM respectively.

3.3 Emergencies

The following are considered laboratory emergency cases:

-chemical spills, to be dealt with according to section 8.17 of the CCMSM.

- fire and fire-related accidents, to be controlled according to section 8.21 of the CCMSM.

- personal contamination, to be treated according to section 8.19 of the CCMSM.

- injury, to be treated in accordance with section 8.18 of CCMSM.

Cases requiring minor first aid should be treated in accordance with section 8.20.

3.4 Radioactive Hazard Mitigation

Control and monitoring of personnel exposure and environmental radiation at VSL are performed in accordance with Chapter 3 of the Radiation Safety Manual (RSM) of the Catholic University of America. Application of the rules prescribed by RSM is the responsibility of each user working in the restricted area (rooms 37 to 40 of Hannan Hall) and the project managers.

Dr. Warren E. Keene, CUA - Radiation Safety Officer (CUA-RSO) and Dr. Isabelle Muller, VSL Radiation Safety Officer (VSL-RSO) are responsible for ensuring that the work is done safely and in a manner consistent with applicable regulations.

4.0 Requirements

4.1 Training

Training on TCLP extracts analysis procedure and equipment used in the analytical laboratory is given by the laboratory manager and/or by a trained and experienced person designated by the laboratory manager until the technician shows competence to use the method independently of direct supervision.

5.0 Equipment and Supplies

5.1 Standards

(1) All the analysis of TCLP extracts in the Analytical Laboratory are performed on relative basis by comparison with known standards.

(2) Sufficient stock of concentrated Standard Solutions should be kept in the Analytical Laboratory to allow at least 30 days of continuous work. (This means about 200 ml of solution containing approximately 1000 ppm of specimen.)

(3) The term "concentrated solution" means the solution contains 1000 ± 20 ppm of the analyte in question. No stock of more diluted standard solutions is allowed to be kept in the laboratory for a period exceeding 24 hours. Diluted solutions should be freshly prepared every 8 hours.

(4) Stock of standard solutions to be analyzed by DCP/Dionex

techniques should be purchased from major commercial sources. No use of solutions is allowed beyond expiration dates, if such dates are provided by the manufacturer or when the volume in the bottle falls below 1/8 of the total volume.

All standard solutions of cations shall be analyzed relative to NIST standards on a monthly basis and a limit of ± 5% will be acceptable.

5.2 Equipment

Qualitative handling of the solutions should be performed utilizing calibrated volumetric tools. These include volumetric flasks of various sizes and automatic dispensing pipettes (see Table in this section) with disposable tips (having fixed or variable volumes). To assure the accuracy of the volumetric tools, the following routine procedure should be applied:

- volumetric flasks : visually examine every volumetric flask prior to use. If there is any mechanical damage to the flask or any permanent, visible solid residue inside, the flask should be discarded. Carefully rinse the flask after use with DI water to avoid deposition of solids.

- dispensing pipettes : gravimetric check of the calibration is required every month as per procedure in section below). If the calibration cannot be adjusted, the tool should be replaced. If an adjustment has been performed, the calibration of the instrument should be immediately rechecked before it is returned to general use. The calibration check reports should be filed into the folder and kept in the Analytical Laboratory.

Qty	Device	Manufacturer	Model #	Calib. Req.
10	Auto pipettes			
1	50 microliter	Eppendorf	3130	I
1	100 microliter	Eppendorf	3130	I
1	200 microliter	Eppendorf	4700	I
1	1000 microliter	Eppendorf	4700	I
1	250 microliter	Eppendorf	4700	I
1	500 microliter	Eppendorf	4700	I
1	4 milliliter	Oxford	Macroset	I
1	5 milliliter	Oxford	Macroset	I
1	0.025-10 milliliter	Rainin	EDP-25	II
	Volumetric flasks	various		N.A.
	DCP Spectrophotometer	Spectrametrix	Spectra Span	With every run

I. Calibration check performed every month according to the following procedure

II. Calibration check performed every six months.

5.3 Calibration of Pipettes

Method: Gravimetric using an analytical balance.

Frequency: Monthly except Rainin pipette which is six monthly.
Required Accuracy: As specified in individual procedures or as per instructions of the Laboratory Manager.

(1) Obtain a "Pipette Calibration Check Record" form and supply requested information on pipette to be calibrated (e.g. manufacturer, model #, etc.).

(2) Tare out the weight of a 20 ml plastic vial on the balance.

(3) Pipet out deionized water from the pipette to be calibrated into the plastic vial. Record nominal capacity, actual capacity (weight displayed on the balance), and percent error under the appropriate columns.

(4) Repeat steps (2) and (3) 5-15 times. For variable volume pipettes perform the measurement for 2-5 volume settings.

(5) Calculate the percent error as follows:

$$\% \text{ ERROR} = \frac{(\text{Actual capacity}) - (\text{Nominal capacity})}{(\text{Nominal capacity})} \times 100$$

(6) Examine results and report deviations from required accuracy to the Laboratory Manager.

(7) Sign and date and file form in the 'Calibration Records'

6.0 Analysis Procedure

6.1 Sample/Standard Preparation and Quality Control

The following quality control operations have to be performed in the TCLP/analytical laboratory before and during the analysis:

(a) A minimum of one blank (extraction fluid) for every 10 extractions that have been conducted in an extraction vessel shall be employed as a check to determine if any memory effects from the extraction equipment are occurring.

(b) A matrix spike shall be performed for each waste glass unless the result exceeds the regulatory level and the data is being used solely to demonstrate that the waste property exceeds the regulatory level. If more than one sample of the same waste is being tested, a matrix spike needs to be performed for every twenty samples and the average percent recovery applied to the waste characterization.

(c) Matrix spike levels should be made at the appropriate regulatory threshold limits (See Table 1). However, if the expected extract contaminant concentration is less than one half

the threshold limit, the spike level may be one half the contaminant concentration but not less than the quantitation limit or a fifth of the threshold limit.

From the 1000 ppm stock standard solutions prepare the spiking solutions in the required concentration ranges according to Table 2. For example, solution #1 is prepared by pipetting 5 ml of 1000 ppm As, 5 ml of 1000 ppm Cr, and 1 ml of 1000 ppm Se into a volumetric flask and bringing to 100 ml with deionized water. Each of the three spiked sample are then prepared by adding 2 ml of spike solution to 18 ml of sample solution (i.e. 1:10 dilution of the spike solution by the sample solution).

	TCLP Level ppm	Spike	Spike ppm	ml of std made up to 100 ml	Dilution Factor	Standard Conc., ppm
Spike Solution #1						
As	5	1:10	50	5	20	1000
Cr	5	1:10	50	5	20	1000
Se	1	1:10	10	1	100	1000
Spike Solution #2						
Ag	5	1:10	50	5	20	1000
Ba	100	1:10	1000	100	1	1000
Cd	1	1:10	10	1	100	1000
Pb	5	1:10	50	5	20	1000
Spike Solution #3						
Hg	0.2	1:10	2	0.2	500	1000

Note: The reference standard solutions to be used for analyzing the spiked TCLP extract samples should have higher concentrations than the spiked concentration ranges. For e.g., to analyze a sample spiked with 5 ppm Arsenic the reference standard should have a concentration range of 7 ppm. Prepare the reference standard solutions from the stock standard solutions after taking the spiked concentrations into consideration.
see (j) below.

(e) The purpose of the matrix spike is to monitor the adequacy of the analytical methods used on the TCLP extract and to determine

whether matrix interferences exist in analyte detection. If the matrix spike recoveries are less than 50%, then the analytical methods are not performing adequately or use of the methods is inadequate. Use of internal calibration quantitation methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the contaminant concentration in the TCLP extract.

(f) Matrix spike recoveries are calculated by the following formula:

$$Percent\ recovery = \frac{A-B}{C} \times 100\%$$

where A = the concentration of the spike sample.
B = the concentration of the unspiked sample, and
C = the spike level

(g) The use of internal calibration quantitation methods shall be employed for a contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level. The following steps are to be followed:

(i) The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

(ii) The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The fourth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and 150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solutions, and may need dilution adjustment to maintain the signals in the linear range of the instrumental technique. All four aliquots are analyzed.

(iii) Prepare a plot, or subject data to linear regression, of instrumental signals or external-calibration-derived concentrations as the dependent variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

(iv) Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject data to linear regression of the corrected instrumental signals or external-calibration-derived concentrations as the dependent variable versus the independent variable. Derive concentrations for unknowns using the internal calibration curve as if it were an external calibration curve.

(h) The sample must be analyzed within the following time periods (in days):

	From field collection to TCLP extraction	From TCLP extraction to preparative extraction	Preparative extraction To: Determinative analysis	Total elapsed time
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

(i) A volume of at least 5 ml of the solution is required for analysis.

(j) Using a 2% HNO_3 solution as the matrix, prepare a Hi Std solution containing all of the elements of interest. The concentrations of elements in the Hi Std should somewhat (but no more than twice) exceed the expected concentrations of elements in the solution.

(k) Use a 2% HNO_3 solution as the Zero Std (Blank).

(l) Transfer the samples to be analyzed and the standard solutions to centrifuge tubes. At least two sets of standards will be required. Maximum of up to 32 samples can be analyzed per run.

(m) Load the automatic sampler carousel with the samples and the standard solutions according to the following sequence:

Position #1 : Zero Standard (always)
Position #2 : High Standard (always)
Position #3 - #12: Samples (maximum) including spiked sample and extraction fluid blank.

For higher positions the sequence can be repeated or terminated by the use of a zero standard followed by a hi standard.

Note: *There is a maximum of up to 10 samples allowed between two adjacent sets of standards. If certain series of samples consist of solutions having wide differences in concentration ranges (factor of 5 or more) and a cross-contamination is likely to occur, the samples can be separated by the zero standard. In that event, the zero standard has to be considered as a sample.*

If the sample (or samples) contain elements which have to be analyzed in a single element mode, perform the multiple element mode analysis first.

(i) Anlysis of the extracts is to be performed on DC Plasma spectrophotometer using the following DCP operating procedure.

6.2 DC Plasma Emission Spectrophotometer Operating Procedure

Manufacturer: Spectrametrix Inc.
Model #: Spectra Span III-B

Note: Refer to section 6.2.2 (pages 18 through 26) of VSL standard analytical procedure TPI-AL Rev # 1 of 10/92. This is a routine procedure for operating the DC Plasma Emission Spectrophotometer and is part of the analytical procedure manual.

7.0 Specimen Control/Traceability

Samples are received from the TCLP Laboratory and are accompanied by a Request for Analysis form (see TPI-SH for format). The samples are logged in the sample inward register in the analytical laboratory. After the analysis is performed the results are sent to the Laboratory Manager of TCLP laboratory for data evaluation. VSL procedure TPI-SH deals with the sample traceability forms and other formats for recording the data.

8.0 Data Processing

The Analytical Laboratory uses a set of programs for recording and processing data from the DC Plasma Spectrophotometer. This set consists of a three part package. The first part records instrument readings, the second part performs any necessary calculations, and the last part prints out the results. The details of this routine computerized data processing are contained in section 8.0 of the VSL standard analytical procedure TPI-AL, Rev # 1 of 10/92. This data processing operation is part of the routine procedure for analyzing samples on DC Plasma Emission Spectrophotometer contained in the procedure TPI-AL, Rev # 1 of 10/92.

From the analytical data of the spiked samples calculate the percentage recovery as in step (f) of section 6.1. Compare the contaminant concentrations in the TCLP extract with the thresholds identified in the appropriate regulations (Table 1).

9.0 References

Method 1311 Toxicity Characteristic Leaching Procedure, Federal Register, v. 55, No. 61, 1990

The Catholic University of America Chemical Materials Safety Manual, January 1989

The Catholic University of America Chemical Hygiene Program, in preparation

Radiation Safety Manual of The Catholic University of America, January 1980, W.E. Keene