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SST
WASTE CHARACTERIZATION PROJECT

CORE 16 DATA REPORT

REV. 0

APPENDIX A
TEST INSTRUCTIONS

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**SST SAMPLE TEST INSTRUCTION FOR
CORE 16 SAMPLE RECEIPT AND PHYSICAL TESTING**

DATE PREPARED : May 8, 1990 PREPARED BY : Tom Jones

SAMPLE NUMBER : Core 16, Segments 1-5

APPROVED BY : Tom Jones Date 5/8/90
Project Manager

CONTROLLING DOCUMENTS :

TEST PLAN : WHC-EP-0210

PROJECT SOW : 16021

CONTROLLING PNL PROCEDURE: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on Segments 1-5 of Core 16. The scope of work includes segment extrusion, sampling, examination, and physical testing.

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

PRE-HOMOGENIZED SEGEMENT INSTRUCTIONS

- Collect solid sample for Particle Size Analysis (~1 gram)
- Collect solid sample for volatile organic analysis

<u>Requested Subsample</u>	<u>Procedure #</u>	<u>Wt. Req'd</u>	<u>Task Leader</u>
- Vol. Org. Screen (solid)	--	5 grams	Scheele

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**SST SAMPLE TEST INSTRUCTION FOR
CORE 16 SAMPLE RECEIPT AND PHYSICAL TESTING**

- If drainable liquid > 25 ml, collect Volatile Organic sample

<u>Requested Subsample</u>	<u>Procedure #</u>	<u>Wt. Req'd</u>	<u>Task Leader</u>
- Vol. Org. Screen (liq.)	--	5 grams	Scheele

- Photograph Segment
- Conduct Physical Tests on Pre-homogenized segment

<u>Requested Phy. Test</u>	<u>Procedure #</u>	<u>Task Leader</u>
- Weight	WHC-053-1	Scheele
- Volume	WHC-053-1	Scheele
- Density	WHC-053-1	Scheele
- Penetrometer	PNL-ALO-506	Scheele
- Slurry Only		
- Settling Rate	WHC-053-1	Scheele
- Vol% Settled Solids	WHC-053-1	Scheele
- Vol% Centrifuged Solids	WHC-053-1	Scheele

- Homogenize Segment following procedure PNL-ALO-135
- Prepare Samples for Transfer to B-Hot Cell
- Required Samples Per Segment (Segments 3 and 5 only)

Two 2.5 gram samples are to be taken from each homogenized segment. One sample is taken from the top of the mix and the other from the bottom. These samples are transferred to the B-Hot Cells for radiochem/inorganic analyses.

**SST SAMPLE TEST INSTRUCTION
FOR VOLATILE ORGANIC ANALYSES FOR CORE 16**

DATE PREPARED : May 10, 1990 PREPARED BY : S. G. McKinley

SAMPLE NUMBERS: 90-3514, 90-3516, 90-3518, 90-3520, 90-3522

APPROVED BY : [Signature] Date 5/10/90
Project Manager

COE # 213

CONTROLLING DOCUMENTS :

TEST PLAN: WHC-EP-0210 Project SOW: No. 16021

Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on Samples 90-3514, 90-3516, 90-3518, 90-3520, 90-3522. These samples were taken from Segments 1-5 of Core 16. This TI covers analysis of volatile organics in these segments.

This TI is based on the test plan identified above. Any deviation on the instructions contained in this TI requires prior notification of the Project Manager. Work will be completed at QA Level 2.

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

REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Volatile Organics	PNL-ALO-330 (Screen) PNL-ALO-335 (GC-MS)	Stromatt Stromatt

**SST SAMPLE TEST INSTRUCTION
FOR VOLATILE ORGANIC ANALYSES FOR CORE 16**

B-HOT CELL SAMPLE PREPARATION INSTRUCTIONS

Prepare four 1 gram aliquots of Samples 90-3514, 90-3516, 90-3518, 90-3520, 90-3522. Transfer to GC-MS laboratory for volatile organic analysis.

REQUESTED ANALYSES

<u>Sample #</u>	<u>Hex Scn</u>	<u>Head Sp Scn</u>	<u>GC-MS</u>	<u>Number of 1 g Sub-samples</u>
90-3514	X	X	X	4
90-3516	X in Dup		X	4
90-3518	X	X	X	4
90-3520	X		X in Dup	4
90-3522	X	X	X	4

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 PARTICLE SIZE ANALYSES**

DATE PREPARED : May 10, 1990 PREPARED BY : S. G. McKinley

SAMPLE NUMBERS : 90-3515, 90-3517, 90-3519, 90-3521, 90-3523

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APPROVED BY : [Signature] Date May 10, 1990
Project Manager

CONTROLLING DOCUMENTS :

TEST PLAN: WTC-ET-0210 Project SOW: No. 16021

Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on Samples 90-3515, 90-3517, 90-3519, 90-3521, 90-3523. These samples were taken from Segments 1-5 of Core 16. This TI covers particle size analysis of these segments.

This TI is based on the test plan identified above. Any deviation on the instructions contained in this TI will require prior approval from the Project Manager. All work will be completed at QA Level 2.

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

REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Particle Size Analysis	2-50.3 (Rev.0)	Merrill Burt

B-HOT CELL SAMPLE PREPARATION INSTRUCTIONS

Transfer samples to M.C. Burt for particle size analysis.

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 SEGMENT 3 HOMOGENIZATION TEST**

DATE PREPARED : May 15, 1990 PREPARED BY : Tom Jones

SAMPLE NUMBERS : 90-3617, 90-3618

APPROVED BY : Tom Jones Date May 15, 1990
Project Manager

CONTROLLING DOCUMENTS :

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

Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work for **Core 16 Segment 3 Homogenization Test**.

This TI is based on the test plan identified above. Any deviation on the instructions contained in this TI will require that prior notification be given to the Project Manager.

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

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 SEGMENT 3 HOMOGENIZATION TEST**

REQUESTED ANALYSES (on both samples and Methods Blank)

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
ICP Metals	PNL-SP-7	Urie
Gamma Energy Analysis	HTA-4-5	Kaye

REQUESTED ANALYSES (on sample 90-3617 only and Methods Blank)

Wt.% Solids	PNL-ALO-504	Steele
Total Beta	HTA-4-23, HTA-4-8	Kaye
Uranium	HTA-4-16	Kaye
Sr-90	HTA-4-8, HTA-4-11	Kaye
I-129	7-40.17, 7-40-26	Kaye
Tc-99	HTA-4-12	Kaye
Pu, Am, Np, alpha	HTA-4-15, HTA-4-5	Kaye
Pu, U, Mass Spec	HTA-4-40	Kaye

B-HOT CELL SAMPLE PREPARATION INSTRUCTIONS

Each sample received (90-3617 and 90-3618) is prepared in duplicate for ICP and gamma energy analysis using the potassium hydroxide fusion method (Procedure PNL-ALO-102). A methods blank will be run with the fusions. Suitable splits of each sample preparation are transferred to Kaye for radiochemical analyses, and Urie for ICP analysis.

An assessment of ICP and GEA data is made before proceeding with other radiochemical analyses.

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 SEGMENT 3 HOMOGENIZATION TEST**

The following identification system will be used for these samples:

90-3617-A-1 (Fusion)	and	90-3618-A-1 (Fusion)
90-3617-A-2 (Fusion Dup.)		90-3618-A-2 (Fusion Dup.)
90-3617-A-3 (Methods Blank)		

Methods Blank samples will be analyzed concurrently with other fusion samples for all analytes.

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 SEGMENT 5 HOMOGENIZATION TEST**

DATE PREPARED : May 15, 1990 PREPARED BY : Tom Jones

SAMPLE NUMBERS : 90-3619, 90-3620

APPROVED BY : 22 Jones Date 5/15/90
Project Manager

CONTROLLING DOCUMENTS :

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

Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work for **Core 16 Segment 5 Homogenization Test**.

This TI is based on the test plan identified above. Any deviation on the instructions contained in this TI will require that prior notification be given to the Project Manager.

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

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 SEGMENT 5 HOMOGENIZATION TEST**

REQUESTED ANALYSES (on both samples and Methods Blank)

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
ICP Metals	PNL-SP-7	Urie
Gamma Energy Analysis	HTA-4-5	Kaye

REQUESTED ANALYSES (on sample 90-3619 only and Methods Blank)

Wt.% Solids	PNL-ALO-504	Steele
Total Beta	HTA-4-23, HTA-4-8	Kaye
Uranium	HTA-4-16	Kaye
Sr-90	HTA-4-8, HTA-4-11	Kaye
I-129	7-40.17, 7-40-26	Kaye
Tc-99	HTA-4-12	Kaye
Pu, Am, Np, alpha	HTA-4-15, HTA-4-5	Kaye
Pu, U, Mass Spec	HTA-4-40	Kaye

B-HOT CELL SAMPLE PREPARATION INSTRUCTIONS

Each sample received (90-3619 and 90-3620) is prepared in duplicate for ICP and gamma energy analysis using the potassium hydroxide fusion method (Procedure PNL-ALO-102). A methods blank will be run with the fusions. Suitable splits of each sample preparation are transferred to Kaye for radiochemical analyses, and Urie for ICP analysis.

An assessment of ICP and GEA data is made before proceeding with other radiochemical analyses.

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 COMPOSITE PREP AND PHYSICAL TESTING**

DATE PREPARED : May 31, 1990 PREPARED BY : Tom Jones

SAMPLE NUMBER : 90-4178, -4179, -4180

APPROVED BY : 28 Jones Date 5/31/90
Project Manager

CONTROLLING DOCUMENTS :

TEST PLAN : WHC-EP-0210

PROJECT SOW : 16021

CONTROLLING PNL PROCEDURE: PNL-ALO-010

INTRODUCTION

This Test Instruction covers the preparation and physical testing of **Core 16 Composite**.

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

CORE COMPOSITE PREPARATION INSTRUCTIONS

A core composite will be prepared by combining 20 Wt.% of segments 1-5 of Core 16. The core composite will be homogenized following the same procedure as the one used for segment homogenization.

**SST SAMPLE TEST INSTRUCTION
FOR CORE 16 COMPOSITING AND PHYSICAL TESTING**

<u>Requested Subsample</u>	<u>Sample #</u>	<u>Wt. Req'd</u>	<u>Task Leader</u>
Homog. Check (2 samples)	90-4178 (top) 90-4179 (bot)	5 grams each	Steele
Chemical Testing	90-4180	75 grams	Steele
Physical Testing	----	60 grams	Scheele

<u>Requested Phy. Test</u>	<u>Procedure #</u>	<u>Task Leader</u>
- Density (in Dup)	WHC-053-1	Scheele
- TGA (in Dup)	RDS-TA-1	Scheele
- DSC (in Dup)	RDS-TA-1	Scheele
- Undissolved Solids (in Dup)	WTC-053-1	Scheele
- Viscosity (in Dup)	WTC-053-1	Scheele

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

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

SAMPLE NUMBERS : 90-4178, 90-4179

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

CONTROLLING DOCUMENTS :

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

Controlling Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work for **Core 16 Composite Homogenization Test.**

This TI is based on the test plan identified above. Any deviation on the instructions contained in this TI will require that prior notification be given to the Project Manager.

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

COMPOSITE PREPARATION

The Core 16 Core Composite will be prepared by taking 20 Wt% of each of the 5 segments and homogenizing as per PNL-ALO-135.

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

REQUESTED ANALYSES (on both samples and Methods Blank)

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
ICP Metals	PNL-SP-7	Urie
Gamma Energy Analysis	HTA-4-5	Kaye

REQUESTED ANALYSES (on Sample 90-4178 only and Methods Blank)

Wt.% Solids	PNL-ALO-504	Steele
Total Alpha	HTA-4-22, HTA-4-6	Kaye
Total Beta	HTA-4-23, HTA-4-8	Kaye
Uranium	HTA-4-16	Kaye
Sr-90	HTA-4-8, HTA-4-11	Kaye
I-129	7-40.17, 7-40-26	Kaye
Tc-99	HTA-4-12	Kaye
Pu, Am, Np, alpha	HTA-4-15, HTA-4-5	Kaye
Pu, U, Mass Spec	HTA-4-40	Kaye

B-HOT CELL SAMPLE PREPARATION INSTRUCTIONS

Each sample received (90-4178 and 90-4179) is prepared in duplicate for ICP and gamma energy analysis using the potassium hydroxide fusion method (Procedure PNL-ALO-102). A methods blank will be run with the fusions. Suitable splits of each sample preparation are transferred to Kaye for radiochemical analyses, and Urie for ICP analysis. The full suite of radiochemical analyses are done for Sample 90-4178 only. An assessment of ICP and GEA data is made before proceeding with other radiochemical analyses.

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

The following identification system will be used for these samples:

90-4178-A-1 (Fusion)	and	90-4179-A-1 (Fusion)
90-4178-A-2 (Fusion Dup.)		90-4179-A-2 (Fusion Dup.)
90-4178-A-3 (Methods Blank)		

Methods Blank samples will be analyzed concurrently with other fusion samples for all analytes.

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

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

SAMPLE NUMBERS : 90-4180

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

CONTROLLING DOCUMENTS :

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

Controlling Procedure: PNL-ALO-010

INTRODUCTION

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

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

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

REQUESTED ANALYSES

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

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

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

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

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

B-HOT CELL SAMPLE PREPARATION INSTRUCTIONS

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

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

SST SAMPLE TEST INSTRUCTION FOR CORE 16 COMPOSITE ANALYSES

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

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

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

SAMPLE IDENTIFICATION SYSTEM

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

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

The ICP Acid Leach Numbering System will be:

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

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

The AA Acid Leach Numbering System will be:

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

The Water Leach Sample Preparation aliquots numbering system will be:

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

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

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

The Mercury Preparation aliquots numbering system will be:

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

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

The SemiVolatile Organics numbering system will be:

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

The TOX/EOX numbering system will be:

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

The Cyanide numbering system will be:

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