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**101-SY Hydrogen Safety Project  
Chemical Analysis Support  
Window "C"  
Total Organic Carbon Analysis**

B. M. Gillespie  
R. W. Stromatt  
D. L. Baldwin  
F. V. Hoopes

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January 1992

Prepared for the U.S. Department of Energy  
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory  
Operated for the U.S. Department of Energy  
by Battelle Memorial Institute



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101-SY HYDROGEN SAFETY PROJECT  
CHEMICAL ANALYSIS SUPPORT

WINDOW "C"  
TOTAL ORGANIC CARBON ANALYSIS

B. M. Gillespie  
R. W. Stromatt  
D. L. Baldwin  
F. V. Hoopes

January, 1992

Prepared for  
the U.S. Department of Energy  
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory  
Richland, Washington 99352

MASTER

EP

## SUMMARY

Core samples taken from Hanford double-shell waste tank 101-SY during Window "C" (after the May 1991 gas release event) were analyzed for total organic carbon by the staff of the Analytical Chemistry Laboratory at Pacific Northwest Laboratory. Westinghouse Hanford Company submitted these samples as Sample Delivery Groups 0, 2, 5, 6, 8, 9, 11, 12, and 13.

The procedures used and the chain of custody were carefully documented. Results of the analyses will be used by the 101-SY Hydrogen Safety Project.

CONTRIBUTORS LIST

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CERTIFICATION STATEMENT

I certify that this data package is in compliance with the terms and conditions of the TPP 17667 and QA Plan MCS-027 for completeness. Release of the data contained in this hard copy data package has been authorized by the Analytical Chemistry Laboratory Project Manager or the Project Manager's designee, as verified by the following signature.

B. M. Gillespie

B. M. Gillespie  
101-SY ACL Project Manager

12-19-91

Date

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## 1.0 INTRODUCTION

This data package contains the results obtained by Pacific Northwest Laboratory (PNL) staff in the characterization of samples for the 101-SY Hydrogen Safety Project. The samples were submitted for analysis by Westinghouse Hanford Company (WHC) under the Technical Project Plan (TPP) 17667 and the Quality Assurance Plan MCS-027. They were from a core sample taken during Window "C" after the May 1991 gas release event. The analytical procedures required for analysis were defined in the Test Instructions (TI) prepared by the PNL 101-SY Analytical Chemistry Laboratory (ACL) Project Management Office in accordance with the TPP and the QA Plan.

The samples (Table 1) were submitted with the appropriate WHC Chain of Custody (COC) and Sample Analysis Request Forms. The samples were delivered to the "B" hot cells in the 325 Building in the 300 Area.

The requested analysis for these samples was for Total Organic Carbon. The quality control (QC) requirements for each sample are defined in the test instructions for each sample. The QC requirements outlined in the procedures and requested in the WHC SOW were followed.

Three appendices are provided: one for Test Instructions; one for Chain of Custody, Sample Analysis Request, and Sample Receipt Forms; and one that contains the primary organic analytical data.

TABLE 1.1. 101-SY Sample Numbers

WHC Sample Number      PNL ALO Sample Number

Sample Delivery Group #0

#101SY-W	91-2925
#101SY-D	91-2926
#SW LOOSE	91-2927

Sample Delivery Group #2

#451	91-6424
------	---------

Sample Delivery Group #5

#443	91-7320
------	---------

Sample Delivery Group #6

#538	91-7485
#634	91-7486

Sample Delivery Group #8

#631	91-7770
#650	91-7771
#712	91-7772
#667	91-7773

Sample Delivery Group #9

#641	91-7812
#601	91-7813

Sample Delivery Group #11

#672	91-7810
#661	91-7811
#776	91-8237
#789	91-8238
#788	91-8239
#766	91-8240
#754	91-8241
#619	91-8242

TABLE 1.1. (contd)

WHC Sample Number      PNL ALO Sample Number

Sample Delivery Group #12

#821	91-9279
#475	91-9280
#777	91-9281
#859	91-9282
#670	91-9283
#888	91-9284
#849	91-9285

Sample Delivery Group #13

#1062	91-10516
#1065	91-10517
#1068	91-10518
#1071	91-10519

## 2.0 TOTAL ORGANIC CARBON ANALYSIS RESULTS

The samples were prepared by procedure PNL-7-40.47, rev. 0, "Determination of TC, TOC, and TIC in Radioactive Liquids, Soils, and Sludges by Hot Persulfate Method." This procedure uses the oxidation/extraction method of hot acid persulfate oxidation. Evolved CO<sub>2</sub> is measured by a UIC Coulometric Carbon Analyzer coulometry detector. Samples are acidified with heated sulfuric acid to drive off all inorganic carbonate carbon as CO<sub>2</sub>. Excess potassium persulfate oxidant, along with a silver catalyst, is then added to the heated sulfuric acid solution. All organic carbon is oxidized to CO<sub>2</sub>, swept away by the carrier gas to the Coulometrics Analyzer, and the results calculated and displayed directly as  $\mu\text{g}$  carbon titrated.

Daily blank values were determined until consecutive blank runs differed by no more than  $\sim 10 \mu\text{g C}$ . Samples were run in duplicate. An  $\alpha$ -D-glucose spike was added to one sample daily in addition to the glucose standards analyzed daily in duplicate or triplicate. Reported values (summarized in Table 2) are corrected for percent recovery of standards.

Sample 91-6424/#451 was re-analyzed in duplicate due to instrument problems evident by low standard recoveries and erratic results during the first analysis. The correct results are shown in this report. Furthermore, small sample sizes were used because of titration cell solution discoloring and display run-on past the elapsed analysis time. These considerations should not affect the reported results. Low spike recoveries (<0%-69.3%) for 101-SY blind simulated samples 91-10516/#1068, 91-10517/#1065, 91-10518/#1068, and 91-10519/#1071 are, as of yet, unexplained. Previous spike recoveries for 101-SY samples have consistently been between 85% and 105%. These recoveries have an unknown effect on the reported blind sample results.

TABLE 2.1. Total Organic Carbon Analysis Results

<u>ALO ID</u>	<u>Cust ID</u>	<u>Size (g)</u>	<u>µg C Sample</u>	<u>µg/Kg Results</u>	<u>RPD Dups</u>	<u>Date Analyzed</u>
91-2925	W-1	0.1718	2489	$1.54 \times 10^7$	2	08/02/91
	-2	0.0787	1167	$1.58 \times 10^7$		
	Spike (added to sample)			105% Recovery		
91-2926	D-1	0.0622	1142	$1.96 \times 10^7$	11	08/02/91
	-2	0.0607	1001	$1.75 \times 10^7$		
	Standard			93.9% Recovery		
	Blank			131		
91-2927	SW Loose-1	0.0839	1420	$1.77 \times 10^7$	17	08/06/91
	-2	0.0563	1129	$2.10 \times 10^7$		
	Spike (added to sample)			96.3% Recovery		
	Standard			95.5% Recovery		
	Blank			138		
91-6424	#451-1	0.1275	1920	$1.61 \times 10^7$	4	08/12/91
	-2	0.1438	2076	$1.54 \times 10^7$		
91-7320	#443-1	0.1770	2250	$1.36 \times 10^7$	11	08/12/91
	-2	0.1348	1921	$1.52 \times 10^7$		
	Spike (added to sample)			85.1% Recovery		
	Standard			93.7% Recovery		
	Blank			100		
91-7485	#538-1	0.1210	1031	$0.91 \times 10^7$	9	08/13/91
	-2	0.1309	1228	$1.00 \times 10^7$		
91-7486	#634-1	0.0723	1074	$1.58 \times 10^7$	7.9	08/13/91
	-2	0.1292	1770	$1.46 \times 10^7$		
91-7770	#631-1	0.1011	874	$0.92 \times 10^7$	8	08/13/91
	-2	0.1634	1539	$1.00 \times 10^7$		
	Spike (added to sample)			97.7% Recovery		
	Standard			93.9% Recovery		
Blank			96			
91-7771	#650-1	0.1578	1914	$1.24 \times 10^7$	2	08/14/91
	-2	0.1508	1861	$1.26 \times 10^7$		

TABLE 2.1. (contd)

<u>ALO ID</u>	<u>Cust ID</u>	<u>Size (g)</u>	<u>µg C Sample</u>	<u>µg/Kg Results</u>	<u>RPD Dups</u>	<u>Date Analyzed</u>
91-7772	#712-1	0.1206	1775	1.51 x 10 <sup>7</sup>	6	08/14/91
	-2	0.1147	1793	1.60 x 10 <sup>7</sup>		
91-7773	#667-1	0.1291	1812	1.44 x 10 <sup>7</sup>	6	08/14/91
	-2	0.1419	2121	1.53 x 10 <sup>7</sup>		
	Spike (added to sample)			100.9% Recovery		
91-7812	#641-1	0.1206	1496	1.27 x 10 <sup>7</sup>	0	08/14/91
	-2	0.1394	1732	1.27 x 10 <sup>7</sup>		
91-7813	#601-1	0.1329	1584	1.22 x 10 <sup>7</sup>	2	08/14/91
	-2	0.1367	1671	1.25 x 10 <sup>7</sup>		
	Standard			97.2% Recovery		
	Blank			74		
91-7810	#672-1	0.0620	963	1.60 x 10 <sup>7</sup>	2	08/20/91
	-2	0.0453	686	1.56 x 10 <sup>7</sup>		
91-8237	#776-1	0.0281	420	1.54 x 10 <sup>7</sup>	6	08/20/91
	-2	0.0444	626	1.45 x 10 <sup>7</sup>		
	Spike (added to sample)			100.1% Recovery		
	Standard			97.0% Recovery		
	Blank			62		
91-7811	#661-1	0.0403	596	1.54 x 10 <sup>7</sup>	9.9	08/21/91
	-2	0.0632	1032	1.70 x 10 <sup>7</sup>		
91-8238	#789-1	0.0318	469	1.54 x 10 <sup>7</sup>	2	08/21/91
	-2	0.0224	323	1.51 x 10 <sup>7</sup>		
	Spike (added to sample)			97.0% Recovery		
	Standard			95.8% Recovery		
	Blank			72		
91-8239	#788-1	0.0701	931	1.38 x 10 <sup>7</sup>	9.1	08/22/91
	-2	0.0602	726	1.26 x 10 <sup>7</sup>		

TABLE 2.1. (contd)

<u>ALO ID</u>	<u>Cust ID</u>	<u>Size (g)</u>	<u>µg C. Sample</u>	<u>µg/Kg Results</u>	<u>RPD Dups</u>	<u>Date Analyzed</u>
91-8240	#766-1	0.0747	1010	$1.41 \times 10^7$	6	08/22/91
	-2	0.0666	959	$1.50 \times 10^7$		
	Spike (added to sample)			94.5% Recovery		
	Standard			96.0% Recovery		
	Blank			79		
91-8241	#754-1	0.0805	1204	$1.51 \times 10^7$	3	08/23/91
	-2	0.0504	733	$1.47 \times 10^7$		
91-8242	#619-1	0.1474	1744	$1.19 \times 10^7$	3	08/23/91
	-2	0.0649	739	$1.15 \times 10^7$		
	Spike (added to sample)			91.0% Recovery		
	Standard			99.2% Recovery		
	Blank			82		
91-9279	#821-1	0.0403	523	$1.32 \times 10^7$	6	09/04/91
	-2	0.0634	869	$1.40 \times 10^7$		
	Spike (added to sample)			98.7% Recovery		
91-9280	#475-1	0.0645	905	$1.43 \times 10^7$	4	09/04/91
	-2	0.0596	869	$1.49 \times 10^7$		
	Standard			98.1% Recovery		
	Blank			93		
91-9281	#777-1	0.0797	1271	$1.65 \times 10^7$	4	09/06/91
	-2	0.0525	872	$1.72 \times 10^7$		
	Spike (added to sample)			96.1% Recovery		
91-9282	#859-1	0.0353	564	$1.66 \times 10^7$	6	09/06/91
	-2	0.0549	829	$1.57 \times 10^7$		
	Standard			96.4% Recovery		
	Blank			85		
91-9283	#670-1	0.0771	1167	$1.61 \times 10^7$	5	09/10/91
	-2	0.0724	1146	$1.69 \times 10^7$		
91-9284	#888-1	0.0317	440	$1.48 \times 10^7$	4	09/10/91
	-2	0.0491	712	$1.54 \times 10^7$		
	Spike (added to sample)			95.6% Recovery		



TABLE 2.1. (contd)

<u>ALO ID</u>	<u>Cust ID</u>	<u>Size (g)</u>	<u>µg C Sample</u>	<u>µg/Kg Results</u>	<u>RPD Dups</u>	<u>Date Analyzed</u>
91-9285	#849-1	0.0568	757	$1.42 \times 10^7$	4	09/10/91
	-2	0.0584	813	$1.48 \times 10^7$		
	Standard			93.9% Recovery		
	Blank		85			
91-10516	#1062-1	0.1346	2708	$2.10 \times 10^7$	4.6	09/16/91
	-2	0.1199	2530	$2.20 \times 10^7$		
91-10517	#1065-1	0.0980	3030	$3.22 \times 10^7$	2	09/16/91
	-2	0.0677	2045	$3.15 \times 10^7$		
	Spike (added to sample)			36.4% Recovery		
	Standard			95.6% Recovery		
	Blank		90			
91-10518	#1068-1	0.0878	359	$4.25 \times 10^6$	0.5	10/02/91
	-2	0.1330	546	$4.27 \times 10^6$		
91-10519	#1071-1	0.1177	1422	$1.26 \times 10^7$	9.1	10/02/91
	-2	0.0795	1054	$1.38 \times 10^7$		
	Spike-1			<0% Recovery		
	-2			69.3% Recovery		
	Standard			96.2% Recovery		
	Blank		76			

Total Organic Carbon by PNL procedure 7-40.47, on instrument WC01713,  
325 Bldg., room 201.

### 3.0 COMPARISON OF BLIND SIMULATED SAMPLE RESULTS OBTAINED WITH THE PNL COMBUSTION AND THE HOT PERSULFATE METHODS

Because of the poor spike recoveries for the blind simulated samples using the Hot Persulfate method (procedure PNL-7-40.47), samples 91-10517/#1065 and 91-10518/#1068 were also analyzed by the PNL combustion method (procedure PNL-7-40.37). With this method the sample is heated at 500-750°C, oxidizing all organic carbon present to CO<sub>2</sub>. The CO<sub>2</sub> is measured also by a UIC Coulometric Carbon Analyzer. Results are corrected for α-D-glucose standard recovery and blank values.

Increased run times and small sample sizes were necessary to minimize TOC display run-on. No satisfactory end point was determined for these analyses, however. Carbon dioxide evolution above background levels continued well past the 20-30 minute run times. It is impossible to determine when organic carbon evolution stops, and when inorganic carbonate carbon evolution begins. Longer run times only increase the chance of carbonate carbon evolution being recorded. Higher furnace temperatures modestly increased CO<sub>2</sub> evolution, but did not noticeably decrease run times.

Results for sample 91-10517/#1065 were 11 to 28% lower than those reported using the Hot Persulfate method and were much less consistent. The result for sample 91-10518/#1068, on the other hand, was 22% higher than Hot Persulfate method results. No useful TOC data was generated using the combustion method because of the inability to determine an analysis end point and the variability in the generated values. Analysis was run on instrument WA92040, 325 Building, room 701.

APPENDIX A

TEST INSTRUCTIONS

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

DATE PREPARED: July 31, 1991 PREPARED BY: B. M. Gillespie  
SAMPLE NUMBERS: 91-2925 (101-SY-W), 91-2926 (101-SY-D) and 91-2927 (SW Loose)  
APPROVED BY: B. M. Gillespie DATE: 7-31-91  
Project Manager

CONTROLLING DOCUMENTS:

Project TPP: 101-SY Chemical Analysis Support Task TPP 17667  
Project QAPP: Hydrogen Safety Project QA Plan MCS-027  
Administrative Control Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on 101-SY tank samples that may be crust, slurry/sludge, saltcake and/or liquid samples and their accompanying blind and blank samples requiring Organic analysis.

This TI is based on the TPP 17667. Any deviations from the instructions contained in this TI will require prior approval from the Project Manager.

Upon sample receipt, samples and their extractants will be stored at  $4 \pm 2^{\circ}\text{C}$ , radioactive levels permitting.

All analyses are to be completed following the identified procedure and reported in the units of ug/L or ug/Kg. If deviations from the procedure must be made, the Project Manager must first be notified. Then the change is documented and this documentation must accompany the analytical data. All analytical data are returned to the ALO Project Support Office. Use of screen is optional for blanks.

REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Total Organic Carbon	PNL-MA-597, Vol. 7 Method 40.47	Stromatt (Baldwin)

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

SAMPLE PREPARATION INSTRUCTIONS

If samples aliquots must be transferred to other groups or out of the building, they must be transferred in accordance with PNL-ALO-010 (i.e. COC).

Respective representative sample aliquots, as spelled out in the procedures below, will be used for sample preparation as follows:

B-HOT CELL PREPARATION INSTRUCTIONS

The preparation and analysis of the samples is performed in a 'hot' cell due to high radiation dose rates.

<u>Task</u>	<u>Preparation Procedure</u>	<u>Task Leader</u>
Total Organic Carbon	7-40.47	Stromatt (Baldwin)

QUALITY CONTROL

Each sample shall be analyzed in duplicate with an analytical spike analyzed at the rate of one per day and a blank analyzed at the rate of one per day as per the TPP 17667.

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR ORGANIC ANALYSIS

DATE PREPARED: July 11, 1991 PREPARED BY: R. W. Stromatt

SAMPLE NUMBERS: 91-6424 (#451), 91-6425 (#578), 91-6426 (#579) 91-6427 (#580)  
and 91-6428 (#581)

APPROVED BY: *R. W. Stromatt* DATE: 7-11-91  
Project Manager

CONTROLLING DOCUMENTS:

Project TPP: 101-SY Chemical Analysis Support Task TPP 17667

Project QAPP: Hydrogen Safety Project QA Plan MCS-027

Administrative Control Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on 101-SY tank samples that may be crust, slurry/sludge, saltcake and/or liquid samples and their accompanying blind and blank samples requiring Organic analysis.

This TI is based on the TPP 17667. Any deviations from the instructions contained in this TI will require prior approval from the Project Manager.

Upon sample receipt, samples and their extractants will be stored at  $4 \pm 2^{\circ}\text{C}$ , radioactive levels permitting.

All analyses are to be completed following the identified procedure and reported in the units of ug/L or ug/Kg. If deviations from the procedure must be made, the Project Manager must first be notified. Then the change is documented and this documentation must accompany the analytical data. All analytical data are returned to the ALO Project Support Office. Use of screen is optional for blanks.

REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Volatile Organics	PNL-ALO-335	Stromatt
Total Organic Carbon	PNL-MA-597 Vol.7, Method 40.47	
<u>Screening Procedures</u>	<u>Procedure #</u>	<u>Task Leader</u>
Volatiles	PNL-ALO-332	Stromatt

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR ORGANIC ANALYSIS

SAMPLE PREPARATION INSTRUCTIONS

If samples aliquots must be transferred to other groups or out of the building, they must be transferred in accordance with PNL-ALO-010 (i.e. COC).

Respective representative sample aliquots, as spelled out in the procedures below, will be used for sample preparation as follows:

<u>Task</u>	<u>Preparation Procedure</u>	<u>Task Leader</u>
Volatiles	PNL-ALO-330	Stromatt

QUALITY CONTROL

The quality control requirements that require the CERCLA QC protocol outlined in the above procedures will be followed except no duplicate analyses or spike analyses are required by the WHC SOW or the TPP 17667.

Volatile sample analyses will be identified by the sample number only.

Data Reporting

<u>Task</u>	<u>Procedure</u>	<u>Task Leader</u>
Volatiles	PNL-ALO-350	Stromatt

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR ORGANIC ANALYSIS

DATE PREPARED: July 25, 1991

PREPARED BY: B. M. Gillespie

SAMPLE NUMBERS: 91-7320 (#443)APPROVED BY: B. M. Gillespie  
Project ManagerDATE: 7-25-91

## CONTROLLING DOCUMENTS:

Project TPP: 101-SY Chemical Analysis Support Task TPP 17667

Project QAPP: Hydrogen Safety Project QA Plan MCS-027

Administrative Control Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on 101-SY tank samples that may be crust, slurry/sludge, saltcake and/or liquid samples and their accompanying blind and blank samples requiring Organic analysis.

This TI is based on the TPP 17667. Any deviations from the instructions contained in this TI will require prior approval from the Project Manager.

Upon sample receipt, samples and their extractants will be stored at  $4 \pm 2^{\circ}\text{C}$ , radioactive levels permitting.

All analyses are to be completed following the identified procedure and reported in the units of ug/L or ug/Kg. If deviations from the procedure must be made, the Project Manager must first be notified. Then the change is documented and this documentation must accompany the analytical data. All analytical data are returned to the ALO Project Support Office. Use of screen is optional for blanks.

REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Total Organic Carbon	PNL-MA-597, Vol. 7 Method 40.47	Stromatt



101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR ORGANIC ANALYSIS

SAMPLE PREPARATION INSTRUCTIONS

If samples aliquots must be transferred to other groups or out of the building, they must be transferred in accordance with PNL-ALO-010 (i.e. COC).

Respective representative sample aliquots, as spelled out in the procedures below, will be used for sample preparation as follows:

<u>Task</u>	<u>Preparation Procedure</u>	<u>Task Leader</u>
Total Organic Carbon	7-40.47	Stromatt

QUALITY CONTROL

The quality control requirements that require the CERCLA QC protocol outlined in the above procedures will be followed except no duplicate analyses or spike analyses are required by the WHC SOW or the TPP 17667.

Each sample shall be analyzed in duplicate with an analytical spike analyzed at the rate of one per day and a blank analyzed at the rate of one per day.

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

DATE PREPARED: July 30, 1991 PREPARED BY: B. M. Gillespie

SAMPLE NUMBERS: 91-7485 (#538) and 91-7486 (#634)

APPROVED BY: B. M. Gillespie DATE: 7-30-91  
Project Manager

CONTROLLING DOCUMENTS:

Project TPP: 101-SY Chemical Analysis Support Task TPP 17667

Project QAPP: Hydrogen Safety Project QA Plan MCS-027

Administrative Control Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on 101-SY tank samples that may be crust, slurry/sludge, saltcake and/or liquid samples and their accompanying blind and blank samples requiring Organic analysis.

This TI is based on the TPP 17667. Any deviations from the instructions contained in this TI will require prior approval from the Project Manager.

Upon sample receipt, samples and their extractants will be stored at  $4 \pm 2^{\circ}\text{C}$ , radioactive levels permitting.

All analyses are to be completed following the identified procedure and reported in the units of ug/L or ug/Kg. If deviations from the procedure must be made, the Project Manager must first be notified. Then the change is documented and this documentation must accompany the analytical data. All analytical data are returned to the ALO Project Support Office. Use of screen is optional for blanks.

REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Total Organic Carbon	PNL-MA-597, Vol. 7 Method 40.47	Stromatt (Baldwin)

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

SAMPLE PREPARATION INSTRUCTIONS

If samples aliquots must be transferred to other groups or out of the building, they must be transferred in accordance with PNL-ALO-010 (i.e. COC).

Respective representative sample aliquots, as spelled out in the procedures below, will be used for sample preparation as follows:

B-HOT CELL PREPARATION INSTRUCTIONS

The preparation and analysis of the samples is performed in a "hot" cell due to high radiation dose rates.

<u>Task</u>	<u>Preparation Procedure</u>	<u>Task Leader</u>
Total Organic Carbon	7-40.47	Stromatt (Baldwin)

QUALITY CONTROL

Each sample shall be analyzed in duplicate with an analytical spike analyzed at the rate of one per day and a blank analyzed at the rate of one per day as per the TPP 17667.

**101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS**

DATE PREPARED: August 6, 1991

PREPARED BY: B. M. Gillespie

SAMPLE NUMBERS: 91-7770 (#631), 91-7771 (#650), 91-7772 (#712) and 91-7773 (#667)APPROVED BY: B M Gillespie  
Project ManagerDATE: 8-6-91**CONTROLLING DOCUMENTS:**

Project TPP: 101-SY Chemical Analysis Support Task TPP 17667

Project QAPP: Hydrogen Safety Project QA Plan MCS-027

Administrative Control Procedure: PNL-ALO-010

**INTRODUCTION**

This Test Instruction (TI) defines the scope of work to be completed on 101-SY tank samples that may be crust, slurry/sludge, saltcake and/or liquid samples and their accompanying blind and blank samples requiring Organic analysis.

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**REQUESTED ANALYSES**

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Total Organic Carbon	PNL-M-597, Vol. 7 Method 40.47	Stromatt (Balwin)

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

SAMPLE PREPARATION INSTRUCTIONS

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B-HOT CELL PREPARATION INSTRUCTIONS

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<u>Task</u>	<u>Preparation Procedure</u>	<u>Task Leader</u>
Total Organic Carbon	7-40.47	Stromatt (Baldwin)

QUALITY CONTROL

Each sample shall be analyzed in duplicate with an analytical spike analyzed at the rate of one per day and a blank analyzed at the rate of one per day as per the TPP 17667.

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

DATE PREPARED: August 8, 1991 PREPARED BY: B. M. Gillespie  
SAMPLE NUMBERS: 91-7810 (#672), 91-7811 (#661), 91-7812 (#641) and 91-7813 (#601)  
APPROVED BY: *B. M. Gillespie* DATE: 8-8-91  
Project Manager

CONTROLLING DOCUMENTS:

Project TPP: 101-SY Chemical Analysis Support Task TPP 17667  
Project QAPP: Hydrogen Safety Project QA Plan MCS-027  
Administrative Control Procedure: PNL-ALO-010

INTRODUCTION

This Test Instruction (TI) defines the scope of work to be completed on 101-SY tank samples that may be crust, slurry/sludge, saltcake and/or liquid samples and their accompanying blind and blank samples requiring Organic analysis.

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REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Total Organic Carbon	PNL-MA-597, Vol. 7 Method 40.47	Stromatt (Baldwin)

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

SAMPLE PREPARATION INSTRUCTIONS

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B-HOT CELL PREPARATION INSTRUCTIONS

The preparation and analysis of the samples is performed in a "hot" cell due to high radiation dose rates.

<u>Task</u>	<u>Preparation Procedure</u>	<u>Task Leader</u>
Total Organic Carbon	7-40.47	Stromatt (Baldwin)

QUALITY CONTROL

Each sample shall be analyzed in duplicate with an analytical spike analyzed at the rate of one per day and a blank analyzed at the rate of one per day as per the TPP 17667.

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

DATE PREPARED: August 15, 1991 PREPARED BY: B. M. Gillespie  
SAMPLE NUMBERS: 91-8237 (#776), 91-8238 (#789), 91-8239 (#788), 91-8240 (#766),  
91-8241 (#754) and 91-8242 (#619)  
APPROVED BY: B.M. Gillespie DATE: 8-15-91  
Project Manager

CONTROLLING DOCUMENTS:

Project TPP: 101-SY Chemical Analysis Support Task TPP 17667  
Project QAPP: Hydrogen Safety Project QA Plan MCS-027  
Administrative Control Procedure: PNL-ALO-010

INTRODUCTION

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All analyses are to be completed within the 14 day hold time following the identified procedure and reported in the units of ug/L or ug/Kg. If deviations from the procedure must be made, the Project Manager must first be notified. Then the change is documented and this documentation must accompany the analytical data. All analytical data are returned to the ALO Project Support Office. Use of screen is optional for blanks.

REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Total Organic Carbon	PNL-MA-597, Vol. 7 Method 40.47	Stromatt (Baldwin)



101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

SAMPLE PREPARATION INSTRUCTIONS

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B-HOT CELL PREPARATION INSTRUCTIONS

The preparation and analysis of the samples is performed in a "hot" cell due to high radiation dose rates.

<u>Task</u>	<u>Preparation Procedure</u>	<u>Task Leader</u>
Total Organic Carbon	7-40.47	Stromatt (Baldwin)

QUALITY CONTROL

Each sample shall be analyzed in duplicate with an analytical spike analyzed at the rate of one per day and a blank analyzed at the rate of one per day as per the TPP 17667.

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

DATE PREPARED: September 3, 1991 PREPARED BY: B. M. Gillespie  
SAMPLE NUMBERS: 91-9279 (#821), 91-8280 (#475), 91-9281 (#777), 91-9282 (#839),  
91-9283 (#670), 91-9284 (#888) and 91-9285 (#849)  
APPROVED BY: B.M. Gillespie DATE: 9-3-91  
Project Manager

CONTROLLING DOCUMENTS:

Project TPP: 101-SY Chemical Analysis Support Task TPP 17667  
Project QAPP: Hydrogen Safety Project QA Plan MCS-027  
Administrative Control Procedure: PNL-ALO-010

INTRODUCTION

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REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Total Organic Carbon	PNL-MA-597, Vol. 7 Method 40.47	Stromatt (Baldwin)

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

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Total Organic Carbon	7-40.47	Stromatt (Baldwin)

QUALITY CONTROL

Each sample shall be analyzed in duplicate with an analytical spike analyzed at the rate of one per day and a blank analyzed at the rate of one per day as per the TPP 17667.

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

DATE PREPARED: September 30, 1991 PREPARED BY: B. M. Gillespie

SAMPLE NUMBERS: 91-10516 (#1062), 91-10517 (#1065), 91-10518 (#1068),  
and 91-10519 (#1071)

APPROVED BY: [Signature] DATE: 9-30-91  
Project Manager

CONTROLLING DOCUMENTS:

- Project TPP: 101-SY Chemical Analysis Support Task TPP 17667
- Project QAPP: Hydrogen Safety Project QA Plan MCS-027
- Administrative Control Procedure: PNL-ALO-010

INTRODUCTION

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REQUESTED ANALYSES

<u>Requested Analysis</u>	<u>Procedure #</u>	<u>Task Leader</u>
Total Organic Carbon	PNL-MA-597, Vol. 7 Method 40.47	Stromatt (Baldwin)

101-SY SAMPLE TEST INSTRUCTION  
WINDOW C  
FOR TOTAL ORGANIC CARBON ANALYSIS

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<u>Task</u>	<u>Preparation Procedure</u>	<u>Task Leader</u>
Total Organic Carbon	7-40.47	Stromatt (Baldwin)

QUALITY CONTROL

Each sample shall be analyzed in duplicate with an analytical spike analyzed at the rate of one per day and a blank analyzed at the rate of one per day as per the TPP 17667.

APPENDIX B

CHAIN OF CUSTODY

APPENDIX B

CHAIN OF CUSTODY

B.1 WESTINGHOUSE CHAIN OF CUSTODY AND SAMPLE ANALYSIS REQUEST FORMS AND PNL  
SAMPLE RECEIPT FORMS

The following samples were received without sample analysis request form present:

<u>WHC Number</u>	<u>PNL/ALO Number</u>
<u>Sample Delivery Group #0</u>	
#101SY-W	91-2925
#101SY-D	91-2926
#SW LOOSE	91-2927

R 8422

Westinghouse Hanford Company	CHAIN OF CUSTODY 101-54 4-8-91
------------------------------	--------------------------------------

Company Contact Dan Harting Telephone 3-2532  
 Sample Collected by \_\_\_\_\_ Date \_\_\_\_\_ Time \_\_\_\_\_  
 Sample Locations \_\_\_\_\_  
 Ice Chest No. \_\_\_\_\_ Field Logbook and Page No. \_\_\_\_\_  
 Remarks H<sub>2</sub>s tamper seal  
For total organic carbon Analysis  
 Bill of Lading No. \_\_\_\_\_ Offsite Property No. \_\_\_\_\_  
 Method of Shipment \_\_\_\_\_  
 Shipped to 325 Building ATT: John Rzu - PNL  
 Possible Sample Hazards See RGR + Regulatory Guide 63

Sample Identification

<u>3 samples of R 8422</u>	<u>Aliquots of the</u>
<u>SW/Dry</u>	<u>original samples.</u>
<u>SW/Wet</u>	
<u>SW/Loose</u>	

Actual sample IDs received were:

- 101-54-W (91-2925)
- 101-54-D (91-2926)
- SW Loose (91-2927)
- RTS
- 4-8-91

Chain of Possession

Relinquished by: <u>Kenneth J. Patterson</u>	Received by: <u>E. F. Ruzo</u>	Date/Time: <u>4-8-91</u>
Relinquished by: <u>Rich L. Steele</u>	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:



Westinghouse Hanford  
Company

CHAIN OF CUSTODY

Company Contact Rev Crawford Telephone 3-1972

Sample Collected by Wayne Edmonson / KI Patton Date 7/10/91 Time \_\_\_\_\_

Sample Locations 101-SY Corel and auger samples

Field Chest No. \_\_\_\_\_ Field Logbook and Page No. \_\_\_\_\_

Remarks Samples submitted for VOA and TOC analysis

Bill of Lading No. \_\_\_\_\_ Offsite Property No. \_\_\_\_\_

Method of Shipment Doorstep carrier / overpack

Shipped to 325 Building Attn: Rick Steele - PNL

Possible Sample Hazards see RSR # 12103

Sample Identification

451 - R9217 Auger sample from Riser 13A for TOC

F578 - Hot Cell blank #1 for VOA

F579 - Hot Cell blank #2 for VOA

F580 - Hot Cell blank #3 for VOA

F581 - Hot Cell blank #4 for VOA

Chain of Possession

Relinquished by: <u>B.A. Chubb</u>	Received by: <u>T. J. Gonyea</u>	Date/Time: <u>7/10/91 6:56 pm</u>
Relinquished by: <u>T. J. Gonyea</u>	Received by: <u>Rick Steele</u>	Date/Time: <u>7/10/91 2050 hrs</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

## REQUEST FOR SPECIAL ANALYSIS (RSA) to PNL

Sample Point 101-SY B.A. Crawford / D.L. Herting		(2) Date/Time Issued 7/10/91 3:30pm	(3) Date/Time Required _____	(4) Charge Code _____ (5) Work Package W7A11
(6) Number of Samples 5	Dose Rate mRad/Hr _____	(7) Customer I.D. #451, #578 #579, #580 #581	(8) Laboratory I.D. _____	(9) Requester Name/Phone B.A. Crawford 3-1972 (11) Volume of Sample all in 20mL containers. (see below)
(10) Release _____ RPT				
(12) Determination	(13) Expected Range	(14) <del>Minimum Detection Level</del> <sup>OTR values</sup>	(15) wt. of sample Method Submitted	
#451 - (R9217 Auger sample) for TOC		200mR/hr	2.31g	
#578 - Hot Cell blank #1 for VOA		<5mR/hr	9.1g	
#579 - Hot Cell blank #2 for VOA		<5mR/hr	11.7g	
#580 - Hot Cell blank #3 for VOA		<5mR/hr	11.7g	
#581 - Hot Cell blank #4 for VOA		<5mR/hr	11.9g	
(16) Matrix (Other Metals or Anions Present)				
(17) Radioactivity Level (Actual <input type="checkbox"/> Estimated <input type="checkbox"/> Total Alpha _____ $\mu$ Ci/L Total Beta _____ $\mu$ Ci/L Total Gamma _____ $\mu$ Ci/L			(18) Additional Information (Measurement Uncertainty or Other Pertinent Information)	
			(20) Samples Received	
(19) Estimated Cost			By	Date
Laboratory Manager			From	Time
(21) Distribution of Final Results / Sample Disposal Instructions As per SMI				

SAMPLE RECEIPT FORM

Delivered by: WHC B-PIANT OVERPACK Date/Time: 2050 hrs 7/10/91

Received by: JK AAW RT STEELE

Customer Sample Number(s): #451 #574 #579 #580 #581

ALO Sample Number(s): 91-6424 91-6425 91-6426 91-6427 91-6428

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

2. Additional Shipping Forms (list):

OFFSITE RSR  
RSA

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

Present X Absent \_\_\_\_\_

If Present, Condition: GOOD

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

Notes: N/A

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

OK ~ 25°C (AMBIENT)

6. Condition of Sample Vials.

GOOD except LIDS have no septa

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

Agreement

8. Resolution of Problems or Discrepancies.

NA

RETURN COMPLETED FORM TO PROJECT MANAGER

Westinghouse Hanford  
Company

CHAIN OF CUSTODY

Company Contact Ben Crawford Telephone 3-1972

Sample Collected by Wayne Edmonson Date \_\_\_\_\_ Time \_\_\_\_\_

Sample Locations 101-SY

Ice Chest No. \_\_\_\_\_ Field Logbook and Page No. 326 7/24

Remarks Samples submitted for TOC and ~~Chloride~~ VOA analyses.

Bill of Lading No. \_\_\_\_\_ Offsite Property No. \_\_\_\_\_

Method of Shipment B-plant dig in N-55 Overpacks

Shipped to 325 Bldg Attn: Rick Steele

Possible Sample Hazards see RSR # 12221  
326 7/24

Sample Identification

#533 - Sec 24-101-SY (for VOA analysis) Core 1

#443 - RSR 12221 Auger sample

Chain of Possession

Relinquished by: <u>Bruce Herz</u>	Received by: <u>Mick Hanzy</u>	Date/Time: <u>7-25-91 6:23</u>
Relinquished by: <u>Mick Hanzy</u>	Received by: <u>Rick Steele</u>	Date/Time: <u>7-25-91 2115</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

**SAMPLE ANALYSIS REQUEST**

**PART I: FIELD SECTION**

Collector D.W. Edmonson Date Sampled        Time        hours  
 Company Contact BA Crawford Telephone (        ) 3-1972

Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
533	1 20ml I-chemical	13.22g sludge	VOA
443	1 20ml I-chemical	16.91 crust	TOC & chelators

Field Information\*\* \_\_\_\_\_

Special Handling and/or Storage VOAs bottled in 20 ml I-Chem jars as collected prior to preleaching discussions.

Possible Sample Hazards radioactive material, caustic matrix

**PART II: LABORATORY SECTION**

Received by \_\_\_\_\_ Title \_\_\_\_\_ Date \_\_\_\_\_

Analysis Required \_\_\_\_\_

\*\*Use back of page for additional information relative to sample location. A-6000-406 (05/90)

SAMPLE RECEIPT FORM

Delivered by: B-PLANT OVERPACK Truck Date/Time: 7-25-91 2150

Received by: J.K. RAW

Customer Sample Number(s): 533, 443

ALO Sample Number(s): 91-7319 & 91-7320

1. Customer Chain-of-Custody Form: Present X Absent \_\_\_\_\_
2. Additional Shipping Forms (list): ORSR  
SAR
3. Custody Seals on Shipping and/or Sample Containers and their Conditions.  
Present ✓ Absent \_\_\_\_\_  
If Present, Condition: GOOD

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

Notes: N/A

5. Condition of Shipping Container (i.e., broken container, dented, breached plastic bag, temperature of sample container as defined in Section 3.0 in PNL-ALO-051, etc.) GOOD (28°C)

6. Condition of Sample Vials. OK

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

8. Resolution of Problems or Discrepancies.  
N/A

RETURN COMPLETED FORM TO PROJECT MANAGER

Westinghouse Hanford  
Company

CHAIN OF CUSTODY

Company Contact: Bruce Hey (WHC) / Bev Crawford Telephone: 3-2529 / 3-1972

Sample Collected by: D.W. Edmanson Date: \_\_\_\_\_ Time: \_\_\_\_\_

Sample Locations: 101-54

Ice Chest No.: \_\_\_\_\_ Field Logbook and Page No.: \_\_\_\_\_

Remarks: \_\_\_\_\_

Bill of Lading No.: \_\_\_\_\_ Offsite Property No.: \_\_\_\_\_

Method of Shipment: B-plant pig in N-55 overpacks.

Shipped to: 325 Bldg. attn: John Rau

Possible Sample Hazards: Radioactive material (mixed waste)

Sample Identification

# 538 2.23g solids 1.3 Rad  $1.6 \times 10^3 \mu\text{Ci}$  crust "Anger" R-9257  $1.3 \times 10^{-5} \text{ } ^{235}\text{U}$

# 634 1.08g slurry  $7.9 \times 10^2 \mu\text{Ci}$   $1.4 \times 10^{-5} \text{ } ^{235}\text{U}$

Segment 7 Core  $\Rightarrow$  sample

Chain of Possession

Relinquished by: <u>Beverly A. Crawford</u>	Received by: <u>Walter J. Edmanson</u>	Date/Time: <u>7/30/91 6:15pm</u>
Relinquished by: <u>Walter J. Edmanson</u>	Received by: <u>John K. Rau</u>	Date/Time: <u>7-30-91</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

A-6000-07 (04/91)





101-54  
(17667)

SAMPLE RECEIPT FORM

7-30-91 JLR

Delivered by: WAL B. PLANT OVERPACK Date/Time: ~~7-30~~ 7-30-91

Received by: JK RAW / Hemi

Customer Sample Number(s): #538 + #634

ALO Sample Number(s): 91-7485 + 91-7486

1. Customer Chain-of-Custody Form: Present X Absent \_\_\_\_\_
2. Additional Shipping Forms (list): OFFSITE RSE
3. Custody Seals on Shipping and/or Sample Containers and their Conditions.  
Present X Absent \_\_\_\_\_  
If Present, Condition: GOOD
4. Sample Tag(s) ID Numbers if not Recorded on the Chain-of-Custody Record or on Sample Vial.  
Notes: N/A
5. Condition of Shipping Container (i.e., broken container, dented, breached plastic bag, temperature of sample container as defined in Section 3.0 in PNL-ALO-051, etc.) OK ~ 24°C (AMBIENT)
6. Condition of Sample Vials. OK
7. Verification of Agreement or Nonagreement of Information on Receiving Documents. AGREEMENT.
8. Resolution of Problems or Discrepancies. N/A

RETURN COMPLETED FORM TO PROJECT MANAGER

Westinghouse Hanford  
Company

CHAIN OF CUSTODY

Company Contact Bev. Crawford Telephone 3-1972

Sample Collected by K.T. Patterson/DW Edmonson Date \_\_\_\_\_ Time \_\_\_\_\_

Sample Locations 101-SY Core 22

Ice Chest No. \_\_\_\_\_ Field Logbook and Page No. \_\_\_\_\_

Remarks \_\_\_\_\_

Bill of Lading No. \_\_\_\_\_ Offsite Property No. \_\_\_\_\_

Method of Shipment B-plant pigs in N-55 overpacks.

Shipped to 325 Bldg. attn: Rick Steele or John Rau

Possible Sample Hazards Radioactive waste see PSR #

Sample Identification

#1631 2.42g seg 8 core 22 } packed in one pig  
#1650 9.43g seg 17 core 22 }

#712 5.79g R-9221 Anger sample } packed in one pig  
#1667 1.20g seg 16 core 22 }

Chain of Possession

Relinquished by: <u>Beverly A. Crawford</u>	Received by: <u>S.F. Wallace</u>	Date/Time: <u>8-7-91, 1840</u>
Relinquished by: <u>S.F. Wallace</u>	Received by: <u>John K. Rau</u>	Date/Time: <u>8-7-91, 2120</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:



~~6ms-691 200-20-1-167~~  
101-54 17667

SAMPLE RECEIPT FORM

Delivered by: B-PLANT OVERPAK TRUCK Date/Time: 8-7-91 2120  
Received by: Joh K. Brown  
Customer Sample Number(s): # 631, # 650, # 712, # 667  
ALO Sample Number(s): 91-7770, 91-7771, 91-7772, 91-7773

- 1. Customer Chain-of-Custody Form: Present  Absent \_\_\_\_\_
- 2. Additional Shipping Forms (list): ORSR  
SAR

- 3. Custody Seals on Shipping and/or Sample Containers and their Conditions.  
Present  Absent \_\_\_\_\_  
If Present, Condition: GOOD

- 4. Sample Tag(s) ID Numbers if not Recorded on the Chain-of-Custody Record or on Sample Vial.  
Notes: 631, 650, 712, 667 (on vials & COC)

- 5. Condition of Shipping Container (i.e., broken container, dented, breached plastic bag, temperature of sample container as defined in Section 3.0 in PNL-ALO-051, etc.)  
GOOD 30°C (AMBIENT TEMP)

- 6. Condition of Sample Vials.  
GOOD

- 7. Verification of Agreement or Nonagreement of Information on Receiving Documents.  
JKR 7-7-91 N/A AGREEMENT

- 8. Resolution of Problems or Discrepancies.  
N/A

RETURN COMPLETED FORM TO PROJECT MANAGER

Westinghouse Hanford Company

CHAIN OF CUSTODY

Company Contact Bruce Heig Telephone 3-2529  
 Sample Collected by D.W. Edmonson Date \_\_\_\_\_ Time \_\_\_\_\_  
 Sample Location 101-SY tank  
 Ice Chest No. \_\_\_\_\_ Field Logbook and Page No. \_\_\_\_\_  
 Remarks \_\_\_\_\_

Bill of Lading No. \_\_\_\_\_ Offsite Property No. \_\_\_\_\_  
 Method of Shipment B-plant pigs in N-55 overpacks  
 Shipped to 325 Bldg attn: Rick Steele  
 Possible Sample Hazards Radioactive material in caustic matrix  
see attached RSR #13647

Sample Identification

BH 3/3	<del># 637</del>	<del>4.63g</del>	<del>seg 15</del>	<del>sludge</del>	} packed in one pig	<del>750 Rad/hr</del>
BH 3/3	<del># total</del>	<del>4.64g</del>	<del>seg 15</del>	<del>sludge</del>		<del>1000 Rad/hr</del>
	# 641	1.65g	seg 5	solids	} packed in one pig	1250 Rad/hr
	# 621	3.71g	seg 13	solids		500 Rad/hr

Chain of Possession

Relinquished by: <u>Bruce Heig</u>	Received by: <u>R. Lempman</u>	Date/Time: <u>8-8-91</u>
Relinquished by: <u>R. Lempman</u>	Received by: <u>John K. Bon</u>	Date/Time: <u>8-4-91 2100</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

## SAMPLE ANALYSIS REQUEST

### PART I: FIELD SECTION

Collector D.W. Edmonson Date Sampled      Time      hours  
 Company Contact Bruce Hey Telephone (      ) 3-2529

Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
<del>677</del>	<del>1 ea 20 mL vial</del>	<del>Solids</del>	<del>Chlorides, TOC BH 3/8</del>
<del>664</del>	<del>1 ea 20 mL vial</del>	<del>Solids</del>	<del>TOC BH 3/8</del>
677 (H)	1 ea 20 mL vial	Solids	TOC
601	1 ea 20 mL vial	Solids	TOC

Field Information\*\* \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Special Handling and/or Storage radioactive material in caustic matrix.

Possible Sample Hazards \_\_\_\_\_  
 \_\_\_\_\_

### PART II: LABORATORY SECTION

Received by \_\_\_\_\_ Title \_\_\_\_\_ Date \_\_\_\_\_  
 Analysis Required \_\_\_\_\_

101-54  
#17667

SAMPLE RECEIPT FORM

Delivered by: B-Plant OVERPACK TRUCK Date/Time: 8-8-91

Received by: John K. Rowland <sup>8-8-91</sup> <sub>JKR</sub>

Customer Sample Number(s): ~~#672~~, ~~#661~~, #641, #601

ALO Sample Number(s): ~~91-7810~~, ~~91-7811~~, 91-7812, 91-7813  
<sub>8-8-91</sub> <sub>JKR</sub>

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

2. Additional Shipping Forms (list): ORSR  
SAR

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

Present X Absent \_\_\_\_\_

If Present, Condition: GOOD

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

Notes: 601 & 641 WERE ONLY SAMPLES  
SHIPPED, AS CONTAINER FOR OTHERS  
WAS NOT AVAILABLE

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

6. Condition of Sample Vials. GOOD

7. Verification of Agreement or Managreement of Information on Receiving Documents. AGREE

8. Resolution of Problems or Discrepancies. N/A

RETURN COMPLETED FORM TO PROJECT MANAGER

Westinghouse Hanford Company

CHAIN OF CUSTODY

Company Contact Bruce Hey Telephone 3-2529

Sample Collected by Patterson/Edmonson Date — Time —

Sample Location 101-54 see notebook: WHC-N-313-7 page 43

Ice Chest No. — Field Logbook and Page No. —

Remarks —

Bill of Lading No. — Offsite Property No. —

Method of Shipment —

Shipped to —

Possible Sample Hazards Radioactive material in caustic solution. See PSR#13638

Sample Identification

#672	4.63g	Segment 15	750 mRad/hr.	} Riser 22A, Core 22
#661	4.64g	Segment 16r	1000 mRad/hr	
#776	4.58g	Segment 18	1125 mRad/hr	
#789	4.56g	Segment 19	1000 mRad/hr	
#788	5.92g	Segment 20	1050 mRad/hr	
#766	7.07g	Segment 21	1600 mRad/hr	
#754	6.18g	Segment 22	1000 mRad/hr	
#619	3.06g	Segment 4	500 mRad/hr	

Chain of Possession

Relinquished by: <u>Bruce Hey</u>	Received by: <u>John J. Throckmorton</u>	Date/Time: <u>8-15-91 1815</u>
Relinquished by: <u>John J. Throckmorton</u>	Received by: <u>John K. Brown</u>	Date/Time: <u>8-15-91 2105</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:



# SAMPLE ANALYSIS REQUEST

## PART I: FIELD SECTION

Collector Edmonson/Patterson Date Sampled        —        Time        hours  
 Company Contact Bruce Hey Telephone (        ) 3-2529

Sample Number	Number and Type of Sample Containers	<del>Type of Sample*</del>	Analysis Requested
672	4.63g Seg. 15, Core 22, Riser 22A		750m Rad/hr TOC
661	4.64g Seg. 16r, "	"	1000m Rad/hr TOC
776	4.58g Seg. 18, "	"	1125m Rad/hr TOC
789	4.56g Seg. 19, "	"	1000m Rad/hr TOC
788	5.92g Seg. 20, "	"	1050m Rad/hr TOC
766	7.07g Seg. 21, "	"	1600m Rad/hr TOC
754	6.18g Seg. 22, "	"	1000m Rad/hr TOC
619	3.06g Seg. 4, "	"	500m Rad/hr TOC

Field Information\*\* \_\_\_\_\_

Special Handling and/or Storage See laboratory notebook: WHC-N-313-7 page 43 for specific information on sample preparation.

Possible Sample Hazards Radioactive material in caustic solution. See RSE # 13638

## PART II: LABORATORY SECTION

Received by \_\_\_\_\_ Title \_\_\_\_\_ Date \_\_\_\_\_

Analysis Required \_\_\_\_\_

101-54  
#17667

PNL-ALO-051, Rev. 0  
Exhibit 1  
Page 1 of 1

SAMPLE RECEIPT FORM

Delivered by: B Plant Overpak Truck Date/Time: 8-15-91 2105

Received by: John K. Bow

Customer Sample Number(s): #672, #661, #776, #789, #788, #766, #754, #619

ALO Sample Number(s): 91-7810, 91, 7811, 91-8237 thru <sup>and 815-01</sup> 91-8242

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

2. Additional Shipping Forms (list): ORSR

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

Present X Absent \_\_\_\_\_

If Present, Condition: GOOD

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

Notes: N/A

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

GOOD AMBIENT TEMP 29°C

6. Condition of Sample Vials.

GOOD \*

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

AGREE

8. Resolution of Problems or Discrepancies.

N/A

\* Samples 661, #672 REC'D & NUMBERS VERIFIED. ALL OTHERS STILL IN CANS & REC'D SIR 8-15-91

RETURN COMPLETED FORM TO PROJECT MANAGER

WILL BE OPENED IN MORNING.

Westinghouse Hanford  
Company

CHAIN OF CUSTODY

Company Contact Bud Crawford Telephone 3-1972

Sample Collected by Wayne Edmonson / K.J. Patterson Date 9/3/91 Time     

Sample Locations     

Ice Chest No.      Field Logbook and Page No.     

Remarks samples submitted for TOC and Rheology / Physical tests

Bill of Lading No.      Offsite Property No.     

Method of Shipment Doorstep carriers and B-plant pig in overpacks

Shipped to 325 Building Attn: John Row - PNL

Possible Sample Hazards see RSR # 13674

Sample Identification

#821 Segment 14 Core 22 for TOC	_____
#475 Segment 14 Core 22 for TOC	_____
#777 Segment 23 Core 22 for TOC	_____
#859 Segment 24 Core 22 for TOC	_____
#1670 Segment 25 Core 22 for TOC	_____
#888 Segment 10 Core 22 for TOC	_____
#849 Segment 11 Core 22 for TOC	_____
#817 Auger R-9221 crust for Rheology / Physical tests	_____

Chain of Possession

Relinquished by: <u>Bud Crawford</u>	Received by: <u>Charlene De Boise</u>	Date/Time: <u>9/3/91 1831</u>
Relinquished by: <u>Charlene De Boise</u>	Received by: <u>John K. Ryan</u>	Date/Time: <u>9-3-91 2100</u>
Relinquished by:	Received by:	Date/Time:
Relinquished by:	Received by:	Date/Time:

A-6000-407 (04/90)

**SAMPLE ANALYSIS REQUEST**

PNL

PART I: FIELD SECTION

Collector D.W. Edmonson / K.J. Peterson Date Sampled      Time      hours  
 Company Contact B.A. Crawford Telephone (      ) 3-1972

Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
821	1 ea. 20 mL vial	3.64g. sludge	TOC
475	" " "	3.17g solids	TOC
777	" " "	4.14g sludge	TOC
859	" " "	4.08g sludge	TOC
670	" " "	6.59g sludge	TOC
888	" " "	4.31g solids	TOC
849	" " "	2.49g solids	TOC
817	1 ea. 125 mL jar	111.4g. 10-54 crust	Rheology & physical tests

Field Information\*\* \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Special Handling and/or Storage radioactive material in caustic matrix.

Possible Sample Hazards \_\_\_\_\_  
 \_\_\_\_\_

PART II: LABORATORY SECTION

Received by \_\_\_\_\_ Title \_\_\_\_\_ Date \_\_\_\_\_  
 Analysis Required \_\_\_\_\_

SAMPLE RECEIPT FORM

Delivered by: B-PLANT OUP-PAK TRUCK Date/Time: 9-3-91 2140

Received by: John K. Brown

Customer Sample Number(s): #821, #475, #777, #859, #670, #888, #849

ALO Sample Number(s): 91-9279, 91-9280, 91-9281, 91-9282, 91-9283, 91-9284, 91-9285

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

2. Additional Shipping Forms (list):  
ORSR

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

Present X Absent \_\_\_\_\_

If Present, Condition: GOOD

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

Notes: ON COC

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

GOOD 25°C AMBIENT

6. Condition of Sample Vials. UNKNOWN UNTIL PUT INTO CELL. (9-4-91 0841 SAMPLES IN CELL & VIALS OK)

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

AGREE

8. Resolution of Problems or Discrepancies.

N/A

RETURN COMPLETED FORM TO PROJECT MANAGER

Westinghouse Hanford  
Company

CHAIN OF CUSTODY

Company Contact B.A. Crawford Telephone 3-1972

Sample Collected by B.A. Crawford Date 9/13/91 Time \_\_\_\_\_

Sample Locations Laboratory 222-SA; Blind standard material WHC-N-313-7 p. 76ff

Ice Chest No. \_\_\_\_\_ Field Logbook and Page No. WHC-N-313-7 p. 76-80

Remarks TOC blind standards made in mock 101-Sy matrix.

Bill of Lading No. \_\_\_\_\_ Offsite Property No. \_\_\_\_\_

Method of Shipment B-plant sample truck

Shipped to Rick Steele 325 Bldg Hot Cells

Possible Sample Hazards caustic

Sample Identification

#1062 WHC-N-313-7 p. 85  
#1065 WHC-N-313-7 p. 86  
#1068 WHC-N-313-7 p. 86  
#1071 WHC-N-313-7 p. 87

Chain of Possession

Relinquished by: <u>Burnley A. Crawford</u>	Received by: <u>Joseph G. Francik</u>	Date/Time: <u>9/24/91 12:10 PM</u>
Relinquished by: <u>Joseph G. Francik</u>	Received by: <u>GRH em</u>	Date/Time: <u>9/30/91 9:45 AM</u>
Relinquished by: <u>GRH em</u>	Received by: <u>Rick Steele</u>	Date/Time: <u>9/30/91 10:50 AM</u>
Relinquished by:	Received by:	Date/Time:

A-6000-407 (04r)01

# SAMPLE ANALYSIS REQUEST

## PART I: FIELD SECTION

Collector B.A. Crawford Date Sampled 9/13/91 Time \_\_\_\_\_ hours  
 Company Contact B.A. Crawford Telephone (509) 373-1972

Sample Number	Number and Type of Sample Containers	Type of Sample*	Analysis Requested
1062	1 ea 22 ml	Mock 101-SY	TOC
1065	1 ea 22 ml	" Solids "	TOC
1068	1 ea 22 ml	" "	TOC
1071	1 ea 22 ml	" "	TOC

Field Information\*\* Caustic matrix

---

Special Handling and/or Storage \_\_\_\_\_

Possible Sample Hazards \_\_\_\_\_

## PART II: LABORATORY SECTION

Received by \_\_\_\_\_ Title \_\_\_\_\_ Date \_\_\_\_\_

Analysis Required \_\_\_\_\_

SAMPLE RECEIPT FORM

Delivered by: G.R. HEYNADEZ Date/Time: 9-30-91 1050

Received by: R.T. STEELE

Customer Sample Number(s): 1062, 1065, 1068, 1071

ALO Sample Number(s): 91-10516, <sup>91-10517</sup> 91-10517, 91-10518, 91-10519

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

2. Additional Shipping Forms (list):  
HAZARDOUS MATERIAL SHIPMENT RECORD (HMSR)  
SAMPLE ANALYSIS REQUEST

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

Present \_\_\_\_\_ Absent X

If Present, Condition: \_\_\_\_\_

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

Notes: N/A

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

6. Condition of Sample Vials.  
GOOD

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

8. Resolution of Problems or Discrepancies.  
N/A

RETURN COMPLETED FORM TO PROJECT MANAGER



B.2 PNL CHAIN OF CUSTODY FORMS

<u>91-10516</u> ALO SAMPLE NUMBER	<u>TOC</u> ANALYSIS REQUESTED	<u>101-SY #1062</u> SAMPLE DESCRIPTION
SENDER _____	_____	DATE _____
RECEIVER _____	_____	DATE _____

<u>91-10517</u> ALO SAMPLE NUMBER	<u>TOC</u> ANALYSIS REQUESTED	<u>101-SY #1065</u> SAMPLE DESCRIPTION
SENDER _____	_____	DATE _____
RECEIVER _____	_____	DATE _____

<u>91-10518</u> ALO SAMPLE NUMBER	<u>TOC</u> ANALYSIS REQUESTED	<u>101-SY #1068</u> SAMPLE DESCRIPTION
SENDER _____	_____	DATE _____
RECEIVER _____	_____	DATE _____

<u>91-10519</u> ALO SAMPLE NUMBER	<u>TOC</u> ANALYSIS REQUESTED	<u>101-SY #1071</u> SAMPLE DESCRIPTION
SENDER _____	_____	DATE _____
RECEIVER _____	_____	DATE _____

SEE ATTACHED HDR SIGNATURES

Original - Project Management Office  
Copy - Sender  
Copy - Receiver

Applicable Test Instruction

TI-101-SY-16

<sup>10/7/91</sup>  
BEST CHAIN OF CUSTODY

<u>91-10516</u> SAMPLE NUMBER	<u>TOC</u> ANALYSIS REQUESTED	<u>10154 #1062</u> SAMPLE DESCRIPTION
SENDER <u>[Signature]</u>		<u>10/2/91</u> DATE
RECEIVER <u>Bill Coll</u>		<u>10/3/91</u> DATE

<u>91-10517</u> SAMPLE NUMBER	<u>TOC</u> ANALYSIS REQUESTED	<u>10154 #1065</u> SAMPLE DESCRIPTION
SENDER <u>[Signature]</u>		<u>10/2/91</u> DATE
RECEIVER <u>Bill Coll</u>		<u>10/3/91</u> DATE

<u>91-10518</u> SAMPLE NUMBER	<u>TOC</u> ANALYSIS REQUESTED	<u>10154 #1068</u> SAMPLE DESCRIPTION
SENDER <u>[Signature]</u>		<u>10/2/91</u> DATE
RECEIVER <u>Bill Coll</u>		<u>10/3/91</u> DATE

<u>91-10519</u> SAMPLE NUMBER	<u>TOC</u> ANALYSIS REQUESTED	<u>10154 #1071</u> SAMPLE DESCRIPTION
SENDER <u>[Signature]</u>		<u>10/2/91</u> DATE
RECEIVER <u>Bill Coll</u>		<u>10/3/91</u> DATE

Original - Project Management Office  
 Copy - Sender  
 Copy - Receiver

Applicable Test Instruction TI-101-SY-16

APPENDIX C

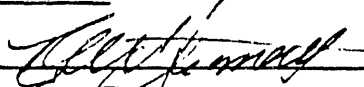

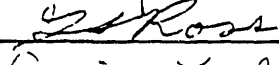
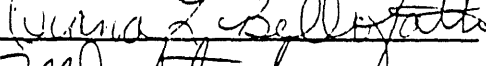

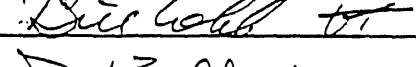
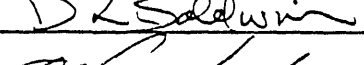
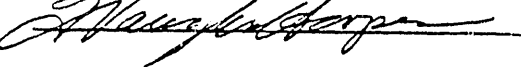
TOTAL ORGANIC CARBON RAW DATA

APPENDIX C

TOTAL ORGANIC CARBON RAW DATA

This section of the Primary Data Package contains the raw data forms as required by the Technical Project Plan 17667. The laboratory analyst signature list is provided below:

ORGANIC LABORATORY ANALYST SIGNATURE LIST

<u>Analyst Name</u>	<u>Initials</u>	<u>Written Name</u>	<u>Initials</u>
Robert W. Stromatt	RWS		RWS
Eric W. Hoppe	EWB		EWB
Gerald A. Ross	GAR		GAR
Diana L. Bellofatto	DLB		DLB
Marilyn J. Steele	MJS		MJS
William T. Cobb	WTC		WTC
David L. Baldwin	DLB		DLB
F. Vaughn Hoopes	FVH		FVH

108

7-30-91

DST TOC RESULTS (OLD WORK)

(batch run on 1-29-91 p 98)

W01713

STD 1	$\frac{(2702 - 22) \times 100}{22700 \times .1199} = 98.5\%$	$\frac{(2430 - 128) \times 100}{6000 \times .400} = 95.9\%$
STD 2	$\frac{(1978 - 22) \times 100}{18000 \times .1199} = 90.6\%$	$\frac{(2645 - 128) \times 100}{6800 \times .400} = 92.5\%$
STD 3	$\frac{(2372 - 28) \times 100}{20100 \times .1199} = 97.3\%$	$\frac{(2092 - 142) \times 100}{5200 \times .400} = 93.7\%$
	avg = 95.4%	avg = 94.0%
90-7815 #1 10Z AN-504	$\frac{(3954 - 22) \times 100}{.1958g \times .954 \times 10^6} = 2.11\%$	$\frac{(5120 - 128) \times 100}{.1958g \times .940 \times 10^6} = 2.71\%$ TC = 4.82%
#2	$\frac{(4013 - 22) \times 100}{.2087g \times .954 \times 10^6} = 2.00\%$	$\frac{(5342 - 128) \times 100}{.2087g \times .940 \times 10^6} = 2.66\%$ TC = 4.66%
90-7703 #1 105 AW-C-5	$\frac{(315 - 28) \times 100}{.1666g \times .954 \times 10^6} = 0.18\%$	$\frac{(595 - 142) \times 100}{.1666g \times .940 \times 10^6} = 0.29\%$ TC = 0.47%
#2	$\frac{(440 - 28) \times 100}{.2209g \times .954 \times 10^6} = 0.20\%$	$\frac{(728 - 142) \times 100}{.2209g \times .940 \times 10^6} = 0.28\%$ TC = 0.48%

8/1/91

BNW #58:00

Began running blanks & standards preparatory to running 101 by TOC samples. The cell current seemed very low and digital display was slow. Also organic blank was abnormally high.

The cell frit was found to be quite brown in color. So cleaned it thoroughly with 8M HNO3 and drew it through the frit with a vacuum line. Rinsed well and resumed work.

8-2-91 Reviewed pages 27 thru 108 for completeness and legibility, checking ~ 10% of the calculations. No deficiencies found.

\* Low stds. + erratic results. due evidently to various leaks in connections, + insufficient AgNO3 delivery because of bad pipetter. Replaced connections and got new pipet for AgNO3

Changed H2SO4 Trap  
Made up new AgNO3  
+ 2M H2SO4  
For Digestion.  
Replaced septum  
w/ Stopper.

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By DL Baldwin Date 8-21-91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_

BNW

8/2/91  
2/16

10/34 Samples for TOC

② Balance

RESULTS  
P.113

Glucose  
#818

(91-2925)

	SPL. SIZE	TIC	TOC
BIK 1)	-	21	128
2)	-	17	134
STD 1)	5.4 mg	.	2112 - 131 / 2160 = 92%
2)	4.9 mg	.	2008 / 1960 = 95%
W-1)	.1718 g.	1280	2620 = 15,500 PPM
2)	.0787 g.	601	1298 14,800
SPL. SPLIT 3)	.1574 g. + 3.8 mg.	1210	4034
(91-2926) D-1	.0622 g.	47	1273 18,400
D-2	.0607 g.	39	1132 16,500
STD. 3)	5.7 mg.		2265 - 131 / 2380 = 94%

8/6/91  
2/16

	SPL. SIZE	TIC	TOC
BIK 1)	-	22	152
2)	-	28	138
SPD. 1)	5.1 mg.		2069 - 1407 / 2040 = 94% Rec.
2)	4.2 mg.		1752 / 1680 = 96%
(91-2927) SW-Loose 1)	.0839 g.	1970	1558 16,800 PPM.
2)	.0563 g.	1275	1067 16,400
SPiked spl. 3)	.0559 g + 5.4 mg.	1190	3255

\* 8/7 thru 10/91 system down ex p. 108

8/12/91  
2/16

RESULTS  
P.114

RNYW  
#53268

	SPL. ID	SPL. SIZE	TIC	TOC
BIK 1)	-	-	16	110
2)	-	-	20	100
STD 1)		4.3 mg.		1695 - 105 / 1720 = 92%
2)		2.8 mg.		1168 / 1120 = 95%
91-6424 #451 1)		.1275 g.	1165	2020 = 15,000 PPM
2)		.1438 g.	1338	2176 14,400
91-7320 #443 1)		.1770 g.	1690	2350 = 12,700
2)		.1348 g.	1360	2021 14,200
SP + SPL. 3)		.1674 g. + (4.4 mg)	1525	3856
STD. 3)		4.2 mg.		1663 - 105 / 1690 = 93%

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_

Entered By DK Baldwin Date 8-26-91

Disclosed To and Understood By \_\_\_\_\_

Signed 1. \_\_\_\_\_ Date \_\_\_\_\_

2. \_\_\_\_\_ Date \_\_\_\_\_

110

8/13/91

~~980~~ ②

SPL. ID	SPL. SIZE	TIC	TOC
BIK 1)	-	15	106
2)	-	19	96
STD. 1)	2.2 mg.		915 - 100 ÷ 880 = 93% R
2)	2.8 mg.		1155 - ÷ 1120 = 94% R

RESULTS  
P. 115

91-7485, #538 1)	.1210 g.	1621	1127 = 8,500 PPM TIC
2)	.1309 g.	1754	1324 9,300

91-7496 #634 1)	.0723 g.	825	1170 14,800 PPM
2)	.1292 g.	1167	1866 13,200

91-7770 #631 1)	.1011 g.	702	970 8,600 PPM.
2)	.1634 g.	1089	1635 9,400
+SP.KC. 3)	.0924g + 2.9mg.	818	2062

STD. 3) 3.2 mg. 1302 - 100 ÷ 1280 = 94% R

Had to change cathode solution twice during the day because of off-color, + run away titrating after sloped line.

8/14/91

~~760~~ ②

BIK 1)	-	14	85
2)	-	12	74
STD. 1)	3.5 mg.		1458 - 80 ÷ 1400 = 98%
2)	3.1 mg.		1265 ÷ 1240 = 96%

91-7771 #650-1)	.1578 g.	1060	1988 = 12,000 PPM
2)	.1508 g.	1007	1935 12,300

91-7772 #712 1)	.1206 g.	1226	1849 = 14,700 PPM
2)	.1147 g.	1258	1867 15,600

91-7773 #667 1)	.1291 g.	1117	1886 = 14,000 PPM
2)	.1419 g.	1519	2195 14,900
+SP.KC. 3)	.0835 g. + 2.8mg.	820	2420

(change cath. + anode here)

91-7812 #641 1)	.1206 g.	1191	1570 = 12,400 PPM.
2)	.1394 g.	1213	1806 12,400

91-7813 #601 1)	.1329 g.	1318	1658 = 11,900 PPM
2)	.1367 g.	1385	1745 12,200

BNW  
#53268

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
STD Entered By 3) 4.0 mg. 1621 - 80 ÷ 1600 = 96% R  
Date \_\_\_\_\_

Disclosed To and Understood By \_\_\_\_\_

Signed 1. D. J. Sullivan Date 8-26-91

2. \_\_\_\_\_ Date \_\_\_\_\_



10/54 TOC CONT'D

bal. ④

Will use smaller sample size to hopefully prevent overloading scrubbers + turning the cascade solution odd colors.

8/20/91  
~~SPD~~

	<u>Spl. Id</u>	<u>Spl. Size</u>	<u>TIC</u>	<u>TOC</u>
	B1K 1)	-		68
	2)	-		62
	STD 1)	4.2 mg.		1688
	(Glucose B1F) 2)	2.9 mg.		1188
RESULTS P. 117	91-7810, #672 1)	.0620 g.	503	1025-
	2)	.0453 g.	450	748
	91-9237, #776 1)	.0281 g.	268	482
	2)	.0444 g.	320	688
	+SPIKE. 3)	.0210 g. + 2.0 mg.	240	1168

8/21/91  
~~SPD~~ ④

				<u>TOC</u>
	B1K 1)	-	-	80
	2)	-	-	72
	STD 1)	3.7 mg.		1517
	(Glucose #B1F) 2)	5.4 mg.		2098
RESULTS P. 117	91-7811 #661 1)	.0403 g.	219	668
	2)	.0632 g.	570	1104
	91-9238 #789 1)	.0318 g.	410	541
	2)	.0224	300	395-
	+SPIKE. 3)	.0399 g. + 3.8 mg.	433	2128
	STD 3)	2.5 mg.	-	1032

RAINW  
#55200

~~9/18~~ 9/23/91

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By [Signature] Date 9/23/91  
 Disclosed To and Understood By \_\_\_\_\_  
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 2. \_\_\_\_\_ Date \_\_\_\_\_

112

8/22/91

~~SPD~~ ④

	<u>Spl. Id</u>	<u>Spl. Size</u>	<u>TIC</u>	<u>TOC</u>
	BIK 1)	-		85
	2)	-		79
	Glucose # BIF 1)	1.8 mg.		765
	2)	2.8 mg.		1170
RESULTS P. 118	91-8239, #788 1)	.0333 g.	395	614 (Appears too high)
	2)	.0701 g.	759	1010
	3)	.0602 g.	615	805
	91-8240, #766 1)	.0747 g.	620	1089
	2)	.0666 g.	641	1038
	+ spike. 3)	.0530 + 3.2 mg.	528	2032
	Std. 3)	4.3 mg.		1714-79/1720

8/23/91

~~SPD~~ ④

	<u>Spl. Id.</u>	<u>Spl. Size</u>	<u>TIC</u>	<u>TOC</u>
	BIK 1)	-		88
	2)	-		82
	Glucose # BIF 1)	2.2 mg.		968
	2)	2.8 mg.		1176
RESULTS P. 118	91-8241 # 754-1)	.0805 g.	735	1284
	2)	.0504 g.	610	815
	91-8242 # 619 1)	.1474 g.	1190	1826
	2)	.0649 g.	580	821
	+ spike. 3)	.1230 g. + 3.2 mg.	255	2675

BNW  
#53268

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By St. Joseph Date 8/23/91  
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 2. \_\_\_\_\_ Date \_\_\_\_\_

101 SY TOC CALCULATIONS / RESULTS

8-20-91

D. Baldwin 113

NOTE: As per the SAMPLE TEST INSTRUCTIONS, a spike will now be analyzed in addition to the system standards. The STI calls for a minimum of one spike and one blank per day as well as the samples in duplicate. Our procedure will correctly stay the same: 2 blanks, 2 standards, 1 spike, samples in duplicate, ending day with 1 standard + 1 blank. Results are to be reported in  $\mu\text{g/L}$  or  $\mu\text{g/kg}$ .

The spike can be used in either of two ways, as follows:  
 A.) To determine spike recovery (using std recoveries and sample values)  
 B.) To determine sample recovery (using std recovery only as measured)  
 Both ways can be measured, but is time-consuming.  
 BEST  $\rightarrow$  Simply measured A.) Spike Recovery (using sample value)

General Formula: % Recovery of Spike Added to Sample  

$$= \frac{\mu\text{g C}_{\text{FOUND}}(\text{SPK} + \text{SPL}) - \mu\text{g C}_{\text{FOUND}}(\text{SPL})}{\mu\text{g C}_{\text{ADDED}}(\text{SPK})} \times 100$$

8-26-91  
3-27-91

BATCH	RUN	ON	8/2/91	(INFORMALLY REPORTED ON 8-9-91)
STD	1		$\frac{(2112 - 131) \times 100}{5400 \mu\text{g} \times 0.400} = 91.7\%$	(TOC ONLY)
STD	2		$\frac{(2000 - 131) \times 100}{4900 \times 0.400} = 95.3\%$	
STD	3		$\frac{(2265 - 131) \times 100}{5700 \times 0.400} = 94.6\%$	
			avg = 93.9%	
91-2925	W-1		$\frac{(2620 - 131) \times 100}{0.1718 \text{g} \times 0.939 \times 10^6} = 1.54\%$	TOC
	W-2		$\frac{(1298 - 131) \times 100}{0.0787 \text{g} \times 0.939 \times 10^6} = 1.58\%$	TOC
$\bar{x} = 1.56\% \text{ TOC}$ $1.54 \times 10^7 \mu\text{g/kg}$ $1.58 \times 10^7 \mu\text{g/kg}$				
SPIKE:		$\mu\text{g C}_{\text{FOUND}}(\text{SPK} + \text{SPL})$	$= 4034 - 131 = 3903 \mu\text{g C}$	
		$\mu\text{g C}_{\text{FOUND}}(\text{SPL})$	$= 0.156 \times 1.574 \times 10^6 \times 0.939 = 2306 \mu\text{g C}$	
		$\mu\text{g C}_{\text{ADDED}}(\text{SPK})$	$= 3800 \times 0.400 = 1520 \mu\text{g C}$	
		% Recovery	$= \frac{(3903 - 2306)}{1520} \times 100 = 105\%$	Recovery
91-2926	D-1		$\frac{(1273 - 131) \times 100}{0.0622 \text{g} \times 0.939 \times 10^6} = 1.96\%$	TOC = $1.96 \times 10^7 \mu\text{g/kg}$
	D-2		$\frac{(1132 - 131) \times 100}{0.0607 \text{g} \times 0.939 \times 10^6} = 1.76\%$	TOC = $1.76 \times 10^7 \mu\text{g/kg}$

802669 #

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
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 2. \_\_\_\_\_ Date \_\_\_\_\_

114

8-26-91

BATCH RUN ON 8/6/91 (INFORMALLY REPORTED)

STD 1  $\frac{(2069 - 138) \times 100}{5100 \times .400} = 94.7\%$

STD 2  $\frac{(1756 - 138) \times 100}{4200 \times .400} = 96.3\%$

91-2927 SW LOOSE-1  $\frac{(1558 - 138) \times 100}{.10839g \times .955 \times 10^6} =$   
 -2  $\frac{(1267 - 138) \times 100}{.0563g \times .955 \times 10^6} =$

$1.77\% = 1.77 \times 10^7 \mu g/kg$   
 $\bar{x} = 1.94\%$   
 $2.10\% = 2.10 \times 10^7 \mu g/kg$

SPIKE:  $\mu g$  FOUND (SPK + SPL) =  $3255 - 138 = 3117 \mu g C$   
 $\mu g$  FOUND (SPL) =  $.0194 \times .0559 \times 10^6 \times .955 = 1036 \mu g C$   
 $\mu g$  ADDED (SPK) =  $5400 \times .400 = 2160 \mu g C$   
 $\%$  RECOVERY =  $\frac{3117 - 1036}{2160} \times 100 = 96.3\%$

System down 8/7 thru 8/10 explain. p 108

BATCH RUN ON 8/12/91 TOC ONLY

STD 1  $\frac{(1695 - 100) \times 100}{4300 \times .400} = 92.7\%$

STD 2  $\frac{(1168 - 100) \times 100}{2800 \times .400} = 95.4\%$

STD 3  $\frac{(1663 - 100) \times 100}{4200 \times .400} = 93.0\%$

$\bar{x} = 93.7\%$

91-6424 #451 -1  $\frac{(2020 - 100) \times 100}{.1275g \times .937 \times 10^6} =$

-2  $\frac{(2176 - 100) \times 100}{.1438g \times .937 \times 10^6} =$

$1.61\% = 1.61 \times 10^7 \mu g/kg$   
 $1.54\% = 1.54 \times 10^7 \mu g/kg$

91-7320 #443 -1  $\frac{(2350 - 100) \times 100}{.1770g \times .937 \times 10^6} =$

-2  $\frac{(2021 - 100) \times 100}{.1348g \times .937 \times 10^6} =$

$1.36\% = 1.36 \times 10^7 \mu g/kg$   
 $\bar{x} = 1.44$   
 $1.52\% = 1.52 \times 10^7 \mu g/kg$

SPIKE:  $\mu g$  FOUND (SPK + SPL) =  $3856 - 100 = 3756 \mu g C$   
 $\mu g$  FOUND (SPL) =  $.0144 \times .1674g \times 10^6 \times .937 = 2258 \mu g C$   
 $\mu g$  ADDED (SPK) =  $4400 \times .400 = 1760 \mu g C$   
 $\%$  RECOVERY =  $\frac{3756 - 2258}{1760} \times 100 = 85.1\%$

BNW  
#53268

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By D. J. Sabalnis Date 8-26-91  
 Disclosed To and Understood By \_\_\_\_\_  
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 2. \_\_\_\_\_ Date \_\_\_\_\_

BATCH	RUN ON	8/13/91	TOC ONLY	101-SY	8-26-91
STD 1	$\frac{(915-96) \times 100}{2200 \times .400}$	=	93.1 %	} $\bar{x} = 93.9 \%$	115
STD 2	$\frac{(1155-96) \times 100}{2800 \times .400}$	=	94.6 %		
STD 3	$\frac{(1302-96) \times 100}{3200 \times .400}$	=	94.2 %		
91-7485 # 538-1	$\frac{(1127-96) \times 100}{.1210 \times .939 \times 10^6}$	=	0.91 %	=	$9.1 \times 10^6 \text{ } \mu\text{g/kg}$
-2	$\frac{(1324-96) \times 100}{.1309 \times .939 \times 10^6}$	=	1.00 %	=	$1.00 \times 10^7 \text{ } \mu\text{g/kg}$
91-7486 # 634-1	$\frac{(1170-96) \times 100}{.0723 \text{ g} \times .939 \times 10^6}$	=	1.58 %	=	$1.58 \times 10^7 \text{ } \mu\text{g/kg}$
-2	$\frac{(1866-96) \times 100}{.1292 \text{ g} \times .939 \times 10^6}$	=	1.46 %	=	$1.46 \times 10^7 \text{ } \mu\text{g/kg}$
91-7770 # 631-1	$\frac{(470-96) \times 100}{.1011 \text{ g} \times .939 \times 10^6}$	=	0.92 %	=	$9.2 \times 10^6 \text{ } \mu\text{g/kg}$
-2	$\frac{(1635-96) \times 100}{.1634 \text{ g} \times .939 \times 10^6}$	=	1.00	=	$1.00 \times 10^7 \text{ } \mu\text{g/kg}$

SPIKE:  $\mu\text{g C FOUND (SPK + SPI)}$  = 2062 - 96 = 1966  $\mu\text{g C}$   
 $\mu\text{g C FOUND (SPI)}$  = .0096  $\times$  .0924  $\text{g} \times 10^6 \times .939 = 833 \text{ } \mu\text{g C}$   
 $\mu\text{g C ADDED (SPK)}$  = 2900  $\times$  .400 = 1160  $\mu\text{g C}$   
 $\% \text{ RECOVERY}$  =  $\frac{1966 - 833}{1160} \times 100 = 97.7 \%$

BATCH	RUN ON	8/14/91	TOC ONLY	101-SY	8-26-91
STD 1	$\frac{(1458-74) \times 100}{3500 \times .400}$	=	98.9 %	} $\bar{x} = 97.2 \%$	
STD 2	$\frac{(1265-74) \times 100}{3100 \times .400}$	=	96.1 %		
STD 3	$\frac{(1621-74) \times 100}{4000 \times .400}$	=	96.7 %		
91-7771 # 650-1	$\frac{(1988-74) \times 100}{.1578 \text{ g} \times .977 \times 10^6}$	=	1.24 %	=	$1.24 \times 10^7 \text{ } \mu\text{g/kg}$
-2	$\frac{(1935-74) \times 100}{.1508 \text{ g} \times .977 \times 10^6}$	=	1.26 %	=	$1.26 \times 10^7 \text{ } \mu\text{g/kg}$

- CONT NEXT PAGE -

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By D & Baldwin Date 8-26-91  
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PAINV  
 # 53268

— CONT' F. PREVIOUS PAGE —

91-7772 #712 -1  $\frac{(1849-74) \times 100}{.1206 \text{ g} \times .977 \times 10^6} = 1,51\% = 1,51 \times 10^3 \mu\text{g}/\text{kg}$   
 -2  $\frac{(1867-74) \times 100}{.1147 \text{ g} \times .977 \times 10^6} = 1,60\% = 1,60 \times 10^3 \mu\text{g}/\text{kg}$

91-7773 #667 -1  $\frac{(1886-74) \times 100}{.1291 \text{ g} \times .977 \times 10^6} = 1,44\% = 1,44 \times 10^3 \mu\text{g}/\text{kg}$   
 -2  $\frac{(2195-74) \times 100}{.1419 \text{ g} \times .977 \times 10^6} = 1,53\% = 1,53 \times 10^3 \mu\text{g}/\text{kg}$   
 $\bar{x} = 1,49\%$

SPIKE  
 $\mu\text{g C}_{\text{FOUND (SPK+SPL)}} = 2420 - 74 = 2346 \mu\text{C}$   
 $\mu\text{g C}_{\text{FOUND (SPL)}} = .0149 \times .0835 \text{ g} \times .977 \times 10^6 = 1215 \mu\text{g C}$   
 $\mu\text{g C}_{\text{ADDED (SPK)}} = 2800 \times .400 = 1120 \mu\text{g C}$   
 $\% \text{ RECOVERY} = \frac{2346 - 1215}{1120} \times 100 = 100,9\%$

91-7812 #641 -1  $\frac{(1570-74) \times 100}{.1206 \text{ g} \times .977 \times 10^6} = 1,27\% = 1,27 \times 10^3 \mu\text{g}/\text{kg}$   
 -2  $\frac{(1806-74) \times 100}{.1394 \text{ g} \times .977 \times 10^6} = 1,27\% = 1,27 \times 10^3 \mu\text{g}/\text{kg}$

91-7813 #601 -1  $\frac{(1658-74) \times 100}{.1329 \text{ g} \times .977 \times 10^6} = 1,22\% = 1,22 \times 10^3 \mu\text{g}/\text{kg}$   
 -2  $\frac{(1745-74) \times 100}{.1367 \text{ g} \times .977 \times 10^6} = 1,25\% = 1,25 \times 10^3 \mu\text{g}/\text{kg}$

Da Baldwin 8-26-91

Reviewed pages 113-116 Checked about 30% of calculations. All were correct. Work is neat and readable

*Billie Wheeler* 8-29-91

BNW  
 #58268

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By DK Baldwin Date 9-16-91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_

101 SY TOC CALCULATIONS / RESULTS

9-5-91  
117

BATCH RUN ON 8/20/91

STD 1  $\frac{(1688 - 62) \mu\text{g} \times 100}{4200 \mu\text{g} \times .400} = 96.8 \% \text{ Recov}$

STD 2  $\frac{(1188 - 62) \mu\text{g} \times 100}{2900 \mu\text{g} \times .400} = 97.1 \% \text{ Recov}$   
 $\bar{x} = 97.0 \% \text{ Recov}$

91-7810 #672 1)  $\frac{(1025 - 62) \mu\text{g} \times 100}{10620 \text{g} \times .970 \times 10^6} = 1.60 \% = 1.60 \times 10^7 \mu\text{g}/\text{kg}$   
 2)  $\frac{(748 - 62) \mu\text{g} \times 100}{10453 \text{g} \times .970 \times 10^6} = 1.56 \% = 1.56 \times 10^7 \mu\text{g}/\text{kg}$

91-8237 #776 1)  $\frac{(482 - 62) \mu\text{g} \times 100}{10281 \text{g} \times .97 \times 10^6} = 1.54 \% = 1.54 \times 10^7 \mu\text{g}/\text{kg}$   
 $\bar{x} = 1.50$   
 2)  $\frac{(688 - 62) \mu\text{g} \times 100}{10444 \text{g} \times .97 \times 10^6} = 1.45 \% = 1.45 \times 10^7 \mu\text{g}/\text{kg}$

BATCH RUN ON 8/21/91 DB 9-5-91

**SPIKE:**  
 $\mu\text{g C FOUND (SPK+SPK)} = 1168 - 62 = 1106 \mu\text{g}$   
 $\mu\text{g C FOUND (SPK)} = 10210 \text{g} \times .0150 \times .970 \times 10^6 = 305.5 \mu\text{g}$   
 $\mu\text{g C Added (SPK)} = 2000 \mu\text{g} \times .400 = 800 \mu\text{g}$   
 $\% \text{ Recovery} = \frac{1106 - 305.5}{800} \times 100 = 100.1 \%$

BATCH RUN ON 8/21/91

STD 1  $\frac{(1517 - 72) \mu\text{g} \times 100}{3700 \mu\text{g} \times .400} = 97.6 \%$

STD 2  $\frac{(2098 - 72) \mu\text{g} \times 100}{5400 \mu\text{g} \times .400} = 93.8 \% \quad \bar{x} = 95.8 \%$

STD 3  $\frac{(1032 - 72) \mu\text{g} \times 100}{2500 \mu\text{g} \times .400} = 96.0 \%$

91-7811 #661 #1)  $\frac{(668 - 72) \mu\text{g} \times 100}{10403 \text{g} \times .958 \times 10^6} = 1.54 \% = 1.54 \times 10^7 \mu\text{g}/\text{kg}$   
 2)  $\frac{(1104 - 72) \mu\text{g} \times 100}{10632 \text{g} \times .958 \times 10^6} = 1.70 \% = 1.70 \times 10^7 \mu\text{g}/\text{kg}$

91-8238 #789 1)  $\frac{(541 - 72) \mu\text{g} \times 100}{10318 \text{g} \times .958 \times 10^6} = 1.54 \% = 1.54 \times 10^7 \mu\text{g}/\text{kg}$   
 2)  $\frac{(395 - 72) \mu\text{g} \times 100}{10224 \text{g} \times .958 \times 10^6} = 1.51 \% = 1.51 \times 10^7 \mu\text{g}/\text{kg}$   
**SPIKE:**  
 $\mu\text{g C FOUND (SPK+SPK)} = 2128 - 72 = 2056 \mu\text{g}$   
 $\mu\text{g C Found (SPK)} = 10152 \times .0399 \times .958 \times 10^6 = 581 \mu\text{g}$   
 $\mu\text{g C Added (SPK)} = 3800 \mu\text{g} \times .400 = 1520 \mu\text{g}$   
 $\% \text{ Recovery} = \frac{2056 - 581}{1520} \times 100 = 97.0 \%$

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_

Entered By DL Baldwin Date 9-5-91

Disclosed To and Understood By \_\_\_\_\_

Signed 1. \_\_\_\_\_ Date \_\_\_\_\_

#53368  
ANIR

118 9-5-91

BATCH RUN ON 8/22/91

STD 1  $\frac{(765-79) \mu\text{g} \times 100}{1800 \mu\text{g} \times .400} = 95.3 \%$

STD 2  $\frac{(1170-79) \mu\text{g} \times 100}{2300 \mu\text{g} \times .400} = 97.4 \%$

STD 3  $\frac{(1720-79) \mu\text{g} \times 100}{4300 \mu\text{g} \times .400} = 95.4 \%$

$\bar{x} = 96.0 \%$

91-8239 #788 1)  $\frac{(614-79) \mu\text{g} \times 100}{10333 \mu\text{g} \times .960 \times 10^6} = 1167 \%$

see page 112  
Problem with Analysis  
Do Not Report

2)  $\frac{(1010-79) \mu\text{g} \times 100}{10701 \mu\text{g} \times .960 \times 10^6} = 1.38 \%$

1.38% =  $1.38 \times 10^7 \mu\text{g}/\text{kg}$   
1.26% =  $1.26 \times 10^7 \mu\text{g}/\text{kg}$

3)  $\frac{(805-79) \mu\text{g} \times 100}{10602 \mu\text{g} \times .960 \times 10^6} = 1.41 \%$

91-8240 #766 1)  $\frac{(1089-79) \mu\text{g} \times 100}{10747 \mu\text{g} \times .960 \times 10^6} = 1.50 \%$

1.41% =  $1.41 \times 10^7 \mu\text{g}/\text{kg}$   
1.50% =  $1.50 \times 10^7 \mu\text{g}/\text{kg}$

2)  $\frac{(1038-79) \mu\text{g} \times 100}{10666 \mu\text{g} \times .960 \times 10^6} = 1.50 \%$

Spike:

$\mu\text{g C FOUND (SPK+SPK)} = 2032 - 79 = 1953 \mu\text{g}$   
 $\mu\text{g C FOUND (SPK)} = 10530 \mu\text{g} \times .0146 \times .960 \times 10^6 = 743 \mu\text{g}$   
 $\mu\text{g C Added (SPK)} = 3200 \mu\text{g} \times .400 = 1280 \mu\text{g}$   
 $\% \text{ Recovery} = \frac{(1953 - 743)}{1280} \times 100 = 94.5 \%$

BATCH RUN ON 8/23/91

STD 1  $\frac{(968-82) \mu\text{g} \times 100}{2300 \mu\text{g} \times .400} = 100.7 \%$

STD 2  $\frac{(1176-82) \mu\text{g} \times 100}{2800 \mu\text{g} \times .400} = 97.7 \%$

$\bar{x} = 99.2 \%$

91-8241 #754 1)  $\frac{(1286-82) \times 100}{10865 \mu\text{g} \times .992 \times 10^6} = 1.51 \%$

1.51% =  $1.51 \times 10^7 \mu\text{g}/\text{kg}$   
1.47% =  $1.47 \times 10^7 \mu\text{g}/\text{kg}$

2)  $\frac{(815-82) \times 100}{10564 \mu\text{g} \times .992 \times 10^6} = 1.19 \%$

91-8242 #619 1)  $\frac{(1826-82) \times 100}{1474 \mu\text{g} \times .992 \times 10^6} = 1.15 \%$

1.19% =  $1.19 \times 10^7 \mu\text{g}/\text{kg}$   
1.15% =  $1.15 \times 10^7 \mu\text{g}/\text{kg}$

2)  $\frac{(821-82) \times 100}{10649 \mu\text{g} \times .992 \times 10^6} = 1.15 \%$

Spike:

$\mu\text{g C FOUND (SPK+SPK)} = 2675 - 82 = 2593 \mu\text{g}$   
 $\mu\text{g C FOUND (SPK)} = 1230 \mu\text{g} \times .0117 \times .992 \times 10^6 = 1428 \mu\text{g}$   
 $\mu\text{g C Added (SPK)} = 3200 \mu\text{g} \times .400 = 1280 \mu\text{g}$   
 $\% \text{ Recovery} = \frac{(2593 - 1428)}{1280} = 91.0 \%$

Project No. \_\_\_\_\_ Date of Work (2593-1428)/1280 = 91.0%

Entered By DK Beldwin Date 9-5-91

Disclosed To and Understood By \_\_\_\_\_

Signed 1. \_\_\_\_\_ Date \_\_\_\_\_

2. \_\_\_\_\_ Date \_\_\_\_\_

BNW  
#55208



Reviewed pages 117 and 118, checked about 30% of the calculations. All were correct. Details is legible and easily followed. *John Stacey 7-5-91*

9/4/91  
*CSA*

LOIS4 TOC

Spl. Id.	Spl. Size	IC	TOC
BIK 1)	-	-	102
2)	-	-	93
Std 1)	3.0 mg.		1366
# BIF 2)	6.9 mg.		2814
91-9277 #821 1)	.0403 g.	333	616
2)	.0634 g.	491	962
+ SPIKE 3)	.0406 g. + 3.4 mg.	320	1975
91-9280 #475 1)	.0645 g.	570	998
2)	.0576 g.	553	962

9/6/91  
*CSA*

BIK 1)	-		92
2)	-		85
Std 1)	3.2 mg.		1360
2)	2.8 mg.		1129
91-9281 #777 1)	.0797 g.	635	1356
2)	.0525 g.	401	957
3)	.0876 g + 4.9 mg.		3387
91-9282 #854 1)	.0553 g.	525	649
2)	.0549 g.	460	914

9/10/91  
*CSA*

BIK 1)	-		92
2)	-		85
Std. 1)	3.2 mg.	-	1367
2)	3.4 mg.	-	1385

Project # 05208

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By D. B. ... Date 9-16-91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_

120

2/10/91  
7:00

(Cont'd)

	Spl. Id.	Spl. S.C.F.	TC	TOC
91-9283 A670	1)	.0771 g	610	1252
	2)	.0724 g	600	1231
91-9284 A888	1)	.0317	260	525-
	2)	.0491	315-	797
	3) Spike	.0719 + 3.1 mg	525-	2442
91-9285 A849	1)	.0565	460	842
	2)	.0584	501	<del>898</del> 898

CALCULATIONS 10154 TOC

9-6-91

BATCH RUN ON 9/14/91 p 119

STD 1	$\frac{(126.6 \mu\text{g} - 93 \mu\text{g}) \times 100}{3000 \mu\text{g} \times 1.400}$	=	97.7 %	$\bar{x} = 99.1 \%$
STD 2	$\frac{(2814 \mu\text{g} - 93 \mu\text{g}) \times 100}{6400 \mu\text{g} \times 1.400}$	=	98.6 %	

91-9279 #821 #1)	$\frac{(616 \mu\text{g} - 93 \mu\text{g}) \times 100}{10403 \text{ g} \times .981 \times 10^6}$	=	1.32 %	$1.32 \times 10^7 \mu\text{g}/\text{kg}$ $\bar{T} = 1.36$ $1.40 \times 10^7 \mu\text{g}/\text{kg}$
2)	$\frac{(967 \mu\text{g} - 93) \times 100}{10634 \text{ g} \times .981 \times 10^6}$	=	1.40 %	

<u>SPIKE</u>	ug C FOUND (SPL + SPA)	=	1978 - 93	=	1885
	ug C AMP (SPA)	=	.0406 g x .0136 Fraction x .981 x 10 <sup>6</sup>	=	542
	ug C Added (SPA)	=	3400 x 1.400	=	1360
	% Recovery	=	(1885 - 542) / 1360 x 100	=	98.7 %

BATCH RUN (cont)

91-9280 #475 1)	$\frac{(978 \mu\text{g} - 93 \mu\text{g}) \times 100}{10645 \text{ g} \times .981 \times 10^6}$	=	1.43 %	$1.43 \times 10^7 \mu\text{g}/\text{kg}$ $1.49 \times 10^7 \mu\text{g}/\text{kg}$
2)	$\frac{(902 \mu\text{g} - 93) \times 100}{10596 \text{ g} \times .981 \times 10^6}$	=	1.49 %	

BNW  
#55268

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By D. Baldwin Date 9-16-91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_

BATCH RUN ON 9-6-91 p 119

7-16-91

STD 1  $\frac{(1360 \mu\text{g} - 85) \times 100}{3200 \mu\text{g} \times .400} =$

99.6 %

121

$\bar{r} = 96.4 \%$

STD 2  $\frac{(1129 \mu\text{g} - 85) \times 100}{2800 \mu\text{g} \times .400} =$

93.2 %

91-928 #777 1)  $\frac{(1356 \mu\text{g} - 85) \times 100}{.0797 \text{g} \times .964 \times 10^6} =$

1.65 % =  $1.65 \times 10^7 \mu\text{g}/\text{kg}$

$\bar{r} = 1.68$

2)  $\frac{(957 \mu\text{g} - 85) \times 100}{.0525 \text{g} \times .964 \times 10^6} =$

1.72 % =  $1.72 \times 10^7 \mu\text{g}/\text{kg}$

SPIKE

$\mu\text{g}$  (FND (SPL+SPK)) =

$3387 \mu\text{g} - 85 = 3302$

$\mu\text{g}$  (FND (SPL)) =

$.0876 \text{g} \times .0168 \text{ FND} \times .964 \times 10^6 = 1419$

$\mu\text{g}$  (Added (SPK)) =

$4900 \times .400 = 1960$

% Recovery =

$(3302 - 1419) / 1960 = 96.1 \%$

91-9282 #859 1)  $\frac{(644 \mu\text{g} - 85) \times 100}{.0353 \text{g} \times .964 \times 10^6} =$

1.66 % =  $1.66 \times 10^7 \mu\text{g}/\text{kg}$

2)  $\frac{(914 \mu\text{g} - 85) \times 100}{.0549 \text{g} \times .964 \times 10^6} =$

1.57 % =  $1.57 \times 10^7 \mu\text{g}/\text{kg}$

BATCH RUN ON 9-10-91 p 119-120

STD 1  $\frac{(1267 \mu\text{g} - 85) \times 100}{3200 \mu\text{g} \times .400} =$

92.3 %

$\bar{r} = 93.9 \%$

STD 2  $\frac{(1385 \mu\text{g} - 85) \times 100}{3400 \mu\text{g} \times .400} =$

95.6 %

91-9283 #670 1)  $\frac{(1252 \mu\text{g} - 85) \times 100}{.0771 \text{g} \times .939 \times 10^6} =$

1.61 % =  $1.61 \times 10^7 \mu\text{g}/\text{kg}$

2)  $\frac{(1231 \mu\text{g} - 85) \times 100}{.0724 \text{g} \times .939 \times 10^6} =$

1.69 % =  $1.69 \times 10^7 \mu\text{g}/\text{kg}$

91-9284 #888 1)  $\frac{(525 \mu\text{g} - 85) \times 100}{.0317 \text{g} \times .939 \times 10^6} =$

1.48 % =  $1.48 \times 10^7 \mu\text{g}/\text{kg}$

$\bar{r} = 1.51$

2)  $\frac{(797 \mu\text{g} - 85) \times 100}{.0491 \text{g} \times .939 \times 10^6} =$

1.54 % =  $1.54 \times 10^7 \mu\text{g}/\text{kg}$

SPIKE

$\mu\text{g}$  (FND (SPL+SPK)) =  $2442 \mu\text{g} - 85 = 2357$

$\mu\text{g}$  (FND (SPL)) =  $.0719 \text{g} \times .0151 \times .939 \times 10^6 = 1019$

$\mu\text{g}$  (Added (SPK)) =  $3500 \times .400 = 1400$

% Recovery =  $(2357 - 1019) / 1400 = 95.6 \%$

91-9285 #849 1)  $\frac{(842 \mu\text{g} - 85) \times 100}{.0568 \text{g} \times .939 \times 10^6} =$

1.42 % =  $1.42 \times 10^7 \mu\text{g}/\text{kg}$

2)  $\frac{(898 \mu\text{g} - 85) \times 100}{.0584 \text{g} \times .939 \times 10^6} =$

1.48 % =  $1.48 \times 10^7 \mu\text{g}/\text{kg}$

#53268

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_

Entered By D. Baldwin Date 9-16-91

Disclosed To and Understood By \_\_\_\_\_

Signed 1. \_\_\_\_\_ Date \_\_\_\_\_

2. \_\_\_\_\_ Date \_\_\_\_\_

9/16/91

Review of pages 119 - 121 Technical & Surveillance  
 All entries are neat & legible  
 All calculations were checked for accuracy and found to be correct

10/1/91

TOC Blind samples \*

(2)

Sample ID	Spl. size	IC	OC
Blk 1)	-		102
2)	-		90
Glucose STD. 1)	6.2 mg.		2439
LOT # BIF 2)	5.7 mg.		2302
91-10516 #1062 1)	.1346 g.	1451	2798
2)	.1199 g.	1398	2620
91-10517 #1065 1)	.0980 g.	1042	3120
2)	.0677 g.	753	2135
+ spike. 3)	.0377 g. + 4.8 mg.	117	1945
Glucose BIF			

\* samples did not dissolve even after the persulfate addition  
 The H<sub>2</sub>SO<sub>4</sub> tray appeared darker color after samples were run

10/2/91

Blk 1)			84
2)			76
Glucose STD 1)	3.6		1468
# BIF 2)	5.3		2105
91-10518 #1068-1)	.0878	968	435
2)	.1330	1675	622
91-10519 #1071 1)	.1177	1762	1498
2)	.0795	797	1130
+ spike 3)	.0995 + 3.0 mg.	1014	827
+ spike. 4)	.0529 + 8.2 mg.	572	3026
Glucose BIF			

BNW

#53268

Project No. M63056 Date of Work 10/1/91 - 10/2/91  
 Entered By [Signature] Date 10/2/91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_

CALCULATIONS - TOC BLIND SAMPLES

BATCH # 122 (9-16-91)

123

STD 1 (Glucose)  $\frac{(2439-90) \times 100}{6200 \mu\text{g} \times .400} = 94.72 \%$

STD 2  $\frac{(2302-90) \times 100}{5700 \mu\text{g} \times .400} = 97.02 \%$   
 $\bar{x} = 95.87 \%$

91-10516 #1062 1)  $\frac{(3793-90) \times 100}{.1346 \text{ g} \times .959 \times 10^6} = 2.10 \%$  =  $2.10 \times 10^7 \mu\text{g}/\text{kg}$

2)  $\frac{(2620 \mu\text{g} - 90 \mu\text{g}) \times 100}{.1149 \text{ g} \times .959 \times 10^6} = 2.20 \%$  =  $2.20 \times 10^7 \mu\text{g}/\text{kg}$

91-10517 #1065 1)  $\frac{(3120-90) \times 100}{.0980 \text{ g} \times .959 \times 10^6} = 3.22 \%$  =  $3.22 \times 10^7 \mu\text{g}/\text{kg}$   
 $\bar{x} = 3.18 \%$

2)  $\frac{(2135-90) \times 100}{.0677 \text{ g} \times .959 \times 10^6} = 3.15 \%$  =  $3.15 \times 10^7 \mu\text{g}/\text{kg}$

SPIKE (Glucose) 3)  $\mu\text{g C FOUND (SPL+SPK)} = 1945 \mu\text{g} - 90 = 1855 \mu\text{g}$   
 $\mu\text{g C FOUND (SPK)} = .0379 \text{ g} \times .0318 \text{ FCN} \times .959 \times 10^6 = 1156$   
 $\mu\text{g C Added (SPK)} = 4800 \mu\text{g} \times .400 = 1920 \mu\text{g}$   
 $\% \text{ Spike Recovery} = \frac{(1855 \mu\text{g} - 1156)}{1920} \times 100 = 36.4 \%$

BATCH RUN 10-2-91

STD 1 (Glucose)  $\frac{(1463 \mu\text{g} - 76 \mu\text{g}) \times 100}{3600 \mu\text{g} \times .400} = 96.7 \%$

STD 2 (")  $\frac{(2105-76) \times 100}{5300 \mu\text{g} \times .400} = 95.7 \%$   
 $\bar{x} = 96.2 \%$

DP 11/5/91  
BNW 53269

91-10518 #1068 1)  $\frac{(435-76) \times 100}{.0573 \text{ g} \times .962 \times 10^6} = 0.43 \%$

2)  $\frac{(622-76) \times 100}{.1330 \text{ g} \times .962 \times 10^6} = 0.43 \%$

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By DA Baldwin Date 10-5-91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_

121

cont previous page

911-10519 #1071 1)  $\frac{(1448.49 - 76.419) \times 100}{.11779 \times .962 \times 10^6} = 1,26\%$   
 $\bar{x} = 1,32$

2)  $\frac{(1130 - 76) \times 100}{.07959 \times .962 \times 10^6} = 1,38\%$

SPike (ajlucose)

mg C FOUND (SPK) =  $825.49 - 76 = 749.49$   
 mg C FOUND (SPK) =  $.0995 \times .0138 \times .926 \times 10^6 = 1271.49$   
 mg Carbon (SPK) =  $3000.49 \times .400 = 1200.49$   
 % SPIKE RECOVERY =  $\frac{(749 - 1271)}{1200} \times 100$  (No Good)

SPike (ajlucose)

mg C FOUND (SPK + SPIKE) =  $3026 - 76 = 2950.49$   
 mg C FOUND (SPK) =  $.05299 \times .0138 \times .926 \times 10^6 = 676.49$   
 mg Carbon (SPK) =  $8400.49 \times 0.400 = 3280.49$   
 % SPIKE RECOVERY =  $\frac{(2950 - 676)}{3280} \times 100 = 69.3\%$

DK Baldwin 11/5/91

BNW 53268

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
 Entered By DK Baldwin Date 11/5/91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_

BEGIN TOC FOR 101 ST SIMULATED WASTE.

10/3/91

SAMPLE #'s 91-10516 (#1062), 91-10517 (#1065), 91-10514 (#1069),  
91-10519 (#1071)

PROCEDURE 7-10.37

RUN TIME = 10 MINS

BALANCE: 360-06-01-023 2.00057g OK

\* CHANGED OUT FURNACE TUBE & CONDITIONED 10/3/91

BLANK ( $\mu\text{g C}$ ) (USING PE BOAT)

- 1) 6.81
  - 2) 6.97
- } AVG. = 6.89  $\mu\text{g C}$

STANDARDS (GLUCOSE; Lot # 06902PX)

g SAM	$\mu\text{g C (EXP)}$	$\mu\text{g C (DISPAY)} - \text{BLK}$	% RES
1) 0.00466g	2660	2709.25 - 6.89	102
2) 0.00294g	1,180	1237.54 - 6.89	104%
3) 0.00365g	1,460	1550.00 - 6.89	106%
4) 0.00319g	1,260	1300.00 - 6.89	103
5) 0.00477g	1,910	1947.49 - 6.89	102

BLANK

- 3) 14.80  $\mu\text{g C}$

*Buttall*

END 10/3/91

BNW  
#52996

10/4/91

101 ST SIMULATED SAMPLES (CONT'D)

PROCEDURE 7-10.37

RUN TIME = 10 MINS

BALANCE: 360-06-01-023

FURNACE T = 607°C

2.00015g OK

BLANK: ( $\mu\text{g C}$ ) (PE BOAT)

- 1) 11.42

Project No. M46655 Date of Work 10/3/91, 10/4/91  
 Entered By Buttall Date 10/3/91, 10/4/91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_

C03-019

10/4/91

TOC (CONT'D)

135

BLANK (µg C)

~~1) 4.08~~ 2) 9.08  
 3) 8.82 }  $AVG. = 8.95 \mu g C$  BLANK

STANDARD (GLUCOSE; Lot # 06802PX)

g SAM	µg C (EXP)	(µg C (DUB) - BLK)	% REC
1) 0.00750	1,800	1812.29	100.0%
2) 0.00823	3,290	3151.33 <del>3169.28</del>	95.4%
3) 0.00409	1,670	1572.91	96.2%

} 96.0%

SAMPLE

91-10518 (#1068)

EMPTY BOAT (g)	BOAT + SAM (g)	SAMPLE (g)	µg C - BLK	C µg/Kg
1) 3.78784	3.87859	0.09375	306.28	$3.27 \times 10^4 \mu g/kg$
2) 3.83909	3.87719	0.03810	193.55	$5.07 \times 10^4 \mu g/kg$

RUNS ARE NOT LONG ENOUGH FOR THIS SIZE SAMPLE TO  
 RECOVER ALL OF THE CARBON COMING OFF THE SAMPLE. ALSO, MUCH  
 CRUSTY RESIDUE IS LEFT OVER IN THE SAMPLE BOAT AFTER  
 10 MIN. RUN TIME.

Bill Cobb ETD 10/4/91

#52996

10/8/91

101 ST SIM SAM (CONT'D)

PROCEDURE 7-70.37  
 FURNACE T = 607 °C

BURNER 360-06-01-023  
 2.00006g HR

RUN TIME DETERMINATION:

SPL # 1068 :	EMPTY BOAT (g)	BT + SPL (g)	SPL (g)	RUN TIME	µg C
1)	3.81639	5.83699	0.02060	25 mins	179

CO3-020

Project No. M96655 Date of Work 10/4/91, 10/9/91  
 Entered By WT COBB Date 10/7/91, 10/9/91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_



136 10/8/91 TOC (CONT'D)

WTC 10/8/91  
~~Run time min. BLK. (µg C) (7.8 µg)~~

Run time determination: Porcelain Be

WTC 10/8/91	MIN/g Sam 0:01343	MINS	Rd OUT (µg C)	µg C/Kg
		10	77.85	5.797 x 10 <sup>6</sup>
		20	129.00	9.531 x 10 <sup>6</sup>
(No Blank Values Subtracted From µg C Results)		25	150.00	1.117 x 10 <sup>7</sup>
		30	168.00	1.251 x 10 <sup>7</sup>
		35	185.00	1.378 x 10 <sup>7</sup>
		40	200.00	1.489 x 10 <sup>7</sup>
		45	214.00	1.593 x 10 <sup>7</sup>
		50	227.00	1.690 x 10 <sup>7</sup>
		55	242.00	1.802 x 10 <sup>7</sup>

10/9/91

CHANGED OUT NOx SCRUBBER TO SEE IF BLANK VALUES WOULD DECREASE & BECOME LESS MORE CONSISTENT.

RUN FURNACE w/ BOMB & LABEL INSERTED UP TO 1000 °C FOR 1+ HRS TO PURGE SYSTEM. BROUGHT SYSTEM BACK DOWN TO 600 °C; BLANK VALUES APPEAR MUCH LOWER & MUCH MORE STABLE AFTER PURGE & NOx SCRUBBER REPLACEMENT.

Bill Coll 10/9/91

BNW  
#52996

TOC - 101 SY SIMULATED - CONT'D

PROCEDURE 7-YO.37  
 FURNACE T = 607 °C

RUN TIME =

BALANCE: 360-06-01-023  
 2.00023, OK

(10mm) BLK: (µg C) (Por Bl) -1) 5.50 µg C  
 2) 4.26 } 4.33 = AVG  
 3) 4.40 }

GLUCOSE STD:	EMPT BOMB (g)	BE + SPL (g)	SPL (g)	µg C EXP	DISPAT (µg C)	% REC
Var = 6502PK	1) 0.57028	0.57609	0.00540	2.320	2482	107

DISREGARDED

Project No. M46655 Date of Work 10/8/91  
 Entered By WT COBB Date 10/9/91  
 Disclosed To and Understood By \_\_\_\_\_  
 Signed 1. \_\_\_\_\_ Date \_\_\_\_\_  
 2. \_\_\_\_\_ Date \_\_\_\_\_

003-011

TOL (CONT'D)

	EMPTY BE (g)	SPL+BE (g)	SPL (g)	μC GR	(μC) DISP-BLK	% REC	137
STD: 2)	0.56937	0.57773	0.00836	3.344	3160	97.5	
3)	0.56944	0.57271	0.00327	1.308	1175	89.8	
4)	4.20826	4.21167	0.00341	1.364	1330	97.5	
5)	4.20815	4.20932	0.00117	468	422	90.2	} 91.6 % RECOV.
6)	4.20771	4.21197	0.00426	1.704	1585	93.0	

SPL: 1068

	EMP BE (g)	SPL+BE (g)	SPL (g)	μC-BLK	μC/Kg SPL
10 min 1)	4.20757	4.21686	0.00932	77.75	7.99 x 10 <sup>6</sup>

BLK:	mins	μC
5	4.75	
10	4.50	
15	13.75	
20	16.78	

			μC RDout		
2)	4.21113	4.21703	0.00590	(10min) 46.50 (15min) 59.09 (20min) 68.64 (25min) 77.51 (30min) 92.00	7.88 x 10 <sup>6</sup> 1.00 x 10 <sup>7</sup> 1.16 x 10 <sup>7</sup> 1.31 x 10 <sup>7</sup> 1.56 x 10 <sup>7</sup>

700°C 3)	4.21323	4.21789	0.00466	(10min) 48.0 (15min) 59.45 (20min) 66.31	1.03 x 10 <sup>7</sup> 1.25 x 10 <sup>7</sup> 1.92 x 10 <sup>7</sup>
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SPL: # 1065

600°C 1)	4.21422	4.22130	0.00708	(10 min) 108.20 (15 min) 125.36 (20 min) 135.71 (25 min) 144.00	1.53 x 10 <sup>7</sup> 1.77 x 10 <sup>7</sup> 1.92 x 10 <sup>7</sup> 2.03 x 10 <sup>7</sup>
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END 10/4/91

10/10/91

TOL (CONT'D)

FURN T = 608°C

MET PROCEDURE = 7-10.37

BALANCE 360-06-01-023 2.00032 GR

BLK: <del>CERT</del> BE;		μC
1)	6.95	4) 4.20
2)	5.41	5) 3.95
3)	4.81	6) 3.95

Project No. M46655 Date of Work 10/9/91, 10/10/91

Entered By Zuellett Date 10/9/91, 10/10/91

Disclosed To and Understood By COS-022 Date \_\_\_\_\_

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2. \_\_\_\_\_ Date \_\_\_\_\_

BNW  
# 52996

REC  
107  
LD

TOC (CONT'D)

BLK : (10 min R.T.; Pore BC & LADLE)  
 10 min 1) 4.96  $\mu\text{g C}$   
 15 min 6.70  $\mu\text{g C}$   
 20 min 8.38  $\mu\text{g C}$   
 25 min 9.94  $\mu\text{g C}$   
 30 min 11.75

SPL #1065 - SUCROSE

#	EMP BC (g)	BE + SPL(S)	SPL (g)	(t)	$\mu\text{g C} - \text{BLK}$	$\mu\text{g C} / \text{Kg SPL}$
1)	4.21291	4.21560	0.00269	10m	41.31-4.96	$1.35 \times 10^7$
				15m	52.0-6.70	$1.68 \times 10^7$
				20m	57.0-8.38	$1.84 \times 10^7$
				25m	61.54-9.94	$1.91 \times 10^7$
				30m	65.08-11.75	$1.98 \times 10^7$
				35m	69.25	$\sim 1.95 \times 10^7$

BLK : (Pore SPL BE + LDC)

10 min 2) 9.77  
 15 min 10.86  
 20 min 12.50  
 25 min 13.95  
 30 min 15.35

SPL : #1065

2)	4.21290	4.21604	0.00334	10m	50.09-4.96	$1.35 \times 10^7$
				15m	59.57-6.70	$1.58 \times 10^7$
				20m	65.93-8.38	$1.72 \times 10^7$
				25m	71.16-9.94	$1.93 \times 10^7$
				30m	75.67-11.75	$1.91 \times 10^7$
				35m	79.79	$\sim 1.99 \times 10^7$

SPIKE : #1065

SPL 1a) 4.21368 4.21570 0.00202 30m 273.35-11.75 =  $261.60 \mu\text{g C}$   
 with SPL + GLN 1b) 4.21570 4.21608 0.00038

BNW  
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BNW

Project No. 10157 - SIMILAR SPL M46655 Date of Work 10/10/91  
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10/10/91 TOC (CONT'D)

139

EXPECTED GLUCOSE RECOVERY:  $0.80 \times 0.00039 \text{ g Glucose}$   
 $= 0.000312 \text{ g} = \underline{150 \mu\text{g C}}$

EXPECTED SPL #1065 C RECOVERY: (ASSUMES  $2.0 \times 10^7 \mu\text{g C/Kg SPL}$ )

$0.00202 \text{ g SPL} = 2.02 \times 10^{-6} \text{ Kg}$

$2.02 \times 10^{-6} \text{ Kg SPL} \times 2.0 \times 10^7 \mu\text{g C/Kg SPL}$

$= \underline{40 \mu\text{g C}}$

$150 \mu\text{g C} + 40 \mu\text{g C} = 190 \mu\text{g C}$  EXPECTED RECOVERY

$\frac{261 \mu\text{g C (DISPWT - BLK)}}{190 \mu\text{g C (EXPECTED)}} = \underline{137\% \text{ REC.}}$

SUSPECT THERE WAS DEFINITE WEIGHING ERROR. BC WT WAS MUCH HIGHER 'EMPTY' THAN IN RUNS BEFORE. COULD ALSO SUGGEST MUCH BETTER SUCROSE RECOVERY WAS OBSERVED - AS CALCULATED BELOW:

$261 \mu\text{g C TOTAL} - 150 \mu\text{g C (glucose)} = 111 \mu\text{g C}$  FROM  $2.0 \times 10^7 \mu\text{g SPL}$

$\frac{111 \mu\text{g C}}{2.02 \times 10^{-6} \text{ Kg SPL}} = \underline{5.5 \times 10^7 \mu\text{g C/Kg SPL}}$

SPLICE: #1065 (30mins)

SPL	2a)	4.22104g	4.21299g	0.00195g	545.65 μg C - 11.75 = 533.90
GLU	2b)	4.21299g	4.21471g	0.00119g	

GLU  $(0.0007 \text{ g}) \text{ (CALC EXP)} = 470 \mu\text{g C}$

SPL  $1.75 \times 10^{-6} \text{ Kg} \times 2.00 \times 10^7 \mu\text{g C/Kg} = 39 \mu\text{g C}$

$\frac{533.90}{509} \times 100 = \underline{105\% \text{ REC.}}$

$= 509 \mu\text{g C EXP}$

Project No. M96655 Date of Work 10/10/91  
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003-024

BNW  
#52996

1.10

10/10/91

TOC (CONT'D)

BLK: 30 MIN RUN TIME, Porcel BC.

1) 13.52  $\mu\text{g C}$

END 10/10/91

10/11/91

TOC (CONT'D) 101ST SIMULATED SPL'S.

RUN TIME = 30 MINS BC = PORCEL. FURNACE T = 603 °C BALANCE: 360.0019  
2.00016g CR

BLK: 30 MINS  
10 MIN 1) 14.30  $\mu\text{g C}$   
2) 9.15  
3) 7.65 } 7.90  $\mu\text{g C}$   
30 MIN 2a) 6.85  
30 MIN 4b) 18.29

STD: GLUCOSE LOT # 6802 PX

#	EMPTY BC (g)	BE + SPL (g)	SPL (g)	$\mu\text{g C Wt}$	$\mu\text{g C REPT} - \text{BLK}$	% REC
1)	4.20965	4.21550	0.00585	2.340	2309.93 - 7.90	98.4
2)	4.20871	4.21253	0.00382	1.648	1629.77 - 7.90	98.7

SPL: #1065, 30 MINS RUN TIME

#	EMPTY BC (g)	BE + SPL (g)	SPL (g)	$\mu\text{g C Wt}$	$\mu\text{g C REPT} - \text{BLK}$	% REC
1)	4.20925	4.21092	0.00167	—	65.05 - 19.29	$2.77 \times 10^3$
2)	4.20940	4.21176	0.00236	—	71.34 - 19.29	$2.25 \times 10^3$
3)	4.20967	4.21283	0.00316	—	80.21 - 19.29	$1.96 \times 10^3$
4)	4.20968	4.21290	0.00322	—	96.15 - 19.29	$2.98 \times 10^3$

SPL: #

GLUCOSE 1)	4.20969	4.21044	0.00075	300	} 348 = 321.43 - 49 $\mu\text{g C}$ SOL	359.72 - 19.29
SPL 2a)	4.21044	4.21237	0.00193	48		

(USING  $2.5 \times 10^3 \mu\text{g C} / \mu\text{g SOL}$ ) =  $273.73 \div 300 \times 100$   
= 91% REC

BNW  
#52996

Project No. M 46655 Date of Work 10/10/91  
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003-025

10/11/91

RANGE OF RECOVERY BASED ON SPL VALUES:

141

µg C EXPECTED FROM SPL:

µg C / kg SPL = 1.96 x 10<sup>7</sup> - 2.77 x 10<sup>7</sup>

µg C EXPECTED ON READOUT: 37.8 - 53.4

% REC RANGE: 321.43 - 57.8 ÷ 300 x 100 =  
321.43 - 53.4 ÷ 300 x 100 =

94.5% - 89.3%

BLK: 30 min

1) 19.34 µg C.

END 10/11/91 WTC

10/14/91

CHANGE NO. SCRUBBER FILLINGS.

10/14/91

TOC - BELWIN FOR 200-BP-1 SPL'S 92-00306 & 92-00355

PROCEDURE 7-70.37 T = 610 °C BALANCE # 360-06-01-023  
5.00005<sub>2</sub> OK.

BLK: Run Time = 10 mins

BE = PECELAW

~~92-0306-T-4~~ & ~~92-0355-T-7~~ 92-0306-T-4 & 92-0355-T-7

NEW BE (SCALE)

- 1) 4.00 µg C
  - 2) 2.55
  - 3) 2.25
- AVG. = 2.40 µg C

STD: α-D-GLUCOSE LOT # 6802PX

Emp BE (g)	BE + SPL (g)	SPL (g)	µg C EXP	µg C - BLK	% REC
1) 4.12939	4.13546	0.00607	2,430	2397.00 - 240	98.5
2) 4.12916	4.13300	0.00384	1,540	1536.36 - 240	99.6
					Avg. = 99.0%

BNW  
# 55996

Project No. \_\_\_\_\_ Date of Work 10/11/91 / 10/14/91

Entered By Bert Call Date 10/11/91 / 10/14/91

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Signed I. \_\_\_\_\_ Date \_\_\_\_\_

\_\_\_\_\_ Date \_\_\_\_\_

1015Y TDC CALCULATIONS/RESULTS

WT CORB 10/25/91 149

NOTE: THESE SAMPLES WERE RUN BY THE PNL COMBUSTION METHOD (PROCEDURE 7-10.37) FOR COMPARISON WITH HOT CELL HOT PERCHLORATE METHOD RESULTS (PROCEDURE 7-10.47). LONGER RUN TIMES <sup>(20-30 min)</sup> & SMALL SAMPLE SIZES WERE NECESSARY TO MINIMIZE ORGANIC CARBON OUTPUT RUN-ON. HIGHER FURNACE TEMPERATURES WERE EXAMINED <sup>(200°C)</sup> TO SEE IF THEY COUNTERACTED THE CARBON RUN-ON PROBLEM BUT WERE NOT PERSUED AFTER INITIAL TESTS SHOWED LITTLE PROMISE.

BATCH RUN ON 10/9/91

STD	1)	$\frac{(3167 - 4.88) \times 100}{9360 \mu\text{g} \times 0.100}$	= 94.5 %
(10 min Run)	2)	$\frac{(1179 - 4.33) \times 100}{3270 \mu\text{g} \times 0.100}$	= 89.8 %
	3)	$\frac{(1337 - 4.33) \times 100}{2710 \mu\text{g} \times 0.100}$	= 92.5 %
	4)	$\frac{(426 - 4.33) \times 100}{1170 \mu\text{g} \times 0.100}$	= 90.2 %
	5)	$\frac{(1589 - 4.33) \times 100}{2260 \mu\text{g} \times 0.100}$	= 93.0 %

AVG. = 93.0 %

91-10518 #1068

(20 min Run)

1)  $\frac{(69.67 - 16.78) \mu\text{g C}}{5.70 \times 10^{-6} \mu\text{g} \times 0.9386} = 9.45 \times 10^6 \mu\text{g C/Kg SPL}$

BATCH RUN ON 10/11/91

STD	1)	$\frac{(2310 - 6.85) \times 100}{5850 \mu\text{g} \times 0.100}$	= 98.4 %
(10 min Run)	2)	$\frac{(1630 - 6.95) \times 100}{4120 \mu\text{g} \times 0.100}$	= 98.5 %

AVG. = 98.4 %

91-10517 #1065

(30 min Run)

1)	$\frac{(65.05 - 19.27) \mu\text{g C}}{1.69 \times 10^{-6} \mu\text{g} \times 0.987}$	= $2.81 \times 10^7 \mu\text{g C/Kg SPL}$
2)	$\frac{(71.37 - 19.29) \mu\text{g C}}{2.36 \times 10^{-6} \mu\text{g} \times 0.987}$	= $2.28 \times 10^7 \mu\text{g C/Kg}$
3)	$\frac{(90.21 - 19.29) \mu\text{g C}}{2.6 \times 10^{-6} \mu\text{g} \times 0.987}$	= $1.99 \times 10^7 \mu\text{g C/Kg}$

BNW #52996

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
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BATCH RUN 10/11/91 (CONT'D)

1)  $\frac{(98.15 - 19.29) \text{ H}_2\text{C}}{3.12 \times 10^4 \text{ H}_2\text{O} \times 0.999}$

$2.52 \times 10^7 \mu\text{g C/m}^3 \text{ H}_2\text{O}$

END 10/15/91 SIMULATED SAMPLE CALCULATIONS  
WTLOBB

Dh Baldwin 11-5-91

BNW  
#52996

Project No. \_\_\_\_\_ Date of Work \_\_\_\_\_  
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