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		Date: 06/01/95
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ENGINEERING DATA TRANSMITTAL

Page 1 of 1

1. EDT No 612159

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2	1	Cog. Mgr. J.G. Kristofzski	<i>J.G. Kristofzski</i>	6-13-95	76-04						
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		Safety									
		Env.									
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ANALYTICAL SERVICES

45-DAY SAFETY SCREEN RESULTS FOR TANK 241-C-204,
AUGER SAMPLES 95-AUG-022 AND 95-AUG-023

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WHC-SD-WM-DP-115, REV. 0

NARRATIVE

45-DAY SAFETY SCREEN RESULTS FOR TANK 241-C-204,
AUGER SAMPLES 95-AUG-022 AND 95-AUG-023

ANALYTICAL SUMMARY

Two auger samples from tank 241-C-204 (C-204) were received at the 222-S Laboratories and underwent safety screening analysis, consisting of differential scanning calorimetry (DSC), thermogravimetric analysis (TGA), and total alpha activity. The three samples submitted for energetics determination by DSC exceeded the notification limit. As required by the Tank Characterization Plan, the appropriate notifications were made within 24 hours of official confirmation that the limit was exceeded. Secondary analyses have been initiated. Results from secondary analyses will be included in a revision to this report.

A rag was caught in both auger samples. The rag material was segregated in the hot cell. None of the chemists nor analysts reported seeing any rag fibers contaminating the samples.

SCOPE

This document serves as the 45-day report deliverable for the tank C-204 auger samples collected on May 2, 1995 (samples 95-AUG-22 and 95-AUG-023). Each sample was received, extruded, and analyzed by the 222-S Laboratories in accordance with the Tank Characterization Plan (TCP) referenced below. Included in this report are the primary safety screening results (DSC, TGA, and total alpha) and copies of all DSC and TGA raw data scans as requested in the TCP. Photographs of the auger samples were taken during extrusion and, although not included in this report, are available.

The results of secondary analyses will be included in a revision to this report. The secondary analyses being conducted are described below.

SAMPLE RECEIPT, EXTRUSION, AND SUBSAMPLING

95-AUG-022

Sample 95-AUG-022 was collected from riser 7 (east coordinate) of tank C-204 using a 20-inch auger sampler. The sample was taken on May 2, 1995 at 1030 hours. It was received into the 222-S Laboratories on May 3 and extruded on May 4. Upon extrusion, it was evident that a rag had been caught by the auger. Some tank waste material was retrieved as well. A gob of waste (4.3 g) on flute 8 of the auger was subsampled as upper half solids. The rag material intermixed with waste was on flutes 11 through 18. All other flutes were bare. A total of 158.5 grams of solid material was collected, with no drainable liquid. Of that amount, 104.3 grams were segregated as rag material, 53.8 grams were segregated as lower half solids, and 4.3 grams were subsampled as upper half solids. The tank waste solids appeared dark brown.

It is anticipated that the archive material for this auger (sample S95T000892) will be used up in secondary analyses. Subsamples are identified in Table 1.

95-AUG-023

Sample 95-AUG-023 was collected from riser 7 (west coordinate) of tank C-204 using a 20-inch auger sampler. The sample was taken on May 2, 1995 at 1135 hours. It was received into the 222-S Laboratories on May 3 and extruded on May 5. As with sample 95-AUG-022, a rag was caught in the auger. Rag and tank waste material were recovered from flutes 13 through 18 of the auger. All other flutes were bare. A total of 135.0 grams of solid material were collected, with no drainable liquid. Of that amount, 93.9 grams of material were segregated as rag material, and 41.1 grams were segregated as tank waste solids. The tank waste appeared to be a mixture of yellow and dark brown solids. Upon subsampling (and incidental mixing), the material appeared brown. The sample was analyzed on a whole segment basis, as no change in strata could be seen and recovery was low. The archive sample (sample S95T000982) from this auger is expected to be used up in secondary analyses. Subsamples are identified in Table 1.

Table 1. C-204 Subsample Identification

Sample ID	Sample Description	Analyses
S95T000876	95-AUG-022 extrusion	extrusion
S95T000877	95-AUG-023 extrusion	extrusion
S95T000878	95-AUG-022 upper half solids, direct analysis	DSC/TGA
S95T000879	95-AUG-022 upper half fusion	fusion/alpha
S95T000880	95-AUG-022 rag material	archive
S95T000881	95-AUG-022 lower half solids, direct analysis	DSC/TGA
S95T000882	95-AUG-022 lower half fusion	fusion/alpha
S95T000883	95-AUG-022 upper half auger subsample	subsampling
S95T000884	95-AUG-022 lower half auger subsample	subsampling
S95T000885	95-AUG-022 lower half archive	secondary analyses
S95T000888	95-AUG-023 rag material	archive
S95T000890	95-AUG-023 whole segment solids, direct analysis	DSC/TGA
S95T000891	95-AUG-023 whole segment fusion	fusion/alpha
S95T000892	95-AUG-023 whole segment archive	secondary analyses
S95T000893	95-AUG-023 whole segment auger subsample	subsampling

ANALYTICAL RESULTS

Analytical results are summarized in Tables 5 and 6, with the applicable notification limits shaded. For tests where more than one replicate was performed, the results are presented in a another table for clarity (e.g. Tables 2, 3, and 4). The summary tables (created electronically from the laboratory sample management program) only include sample and duplicate results.

DSC (Energetics Content)

DSC analyses were performed under a nitrogen atmosphere using procedure LA-514-113, Rev. B-1. Exotherms exceeding the notification limit of 481 J/g were detected for all three samples. Safety program personnel were consulted for direction in running secondary analyses. The secondary analyses being conducted are discussed below.

Three LMCS control standards were run along with these samples, exhibiting recoveries ranging from 103.7 to 107.9 percent, all within the program's specified accuracy control limits of 90 to 110 percent recovery.

Results for S95T000890. The sample and duplicate results for sample S95T000890 (from 95-AUG-023) were 952.1 and 665.7 J/g respectively (on a dry weight basis). The relative percent difference (RPD) between sample and duplicate results was 35.4%. As this result was not within the TCP target of 10%, a triplicate sample was analyzed, with a result of 822.9 J/g. The triplicate result is not included in the summary tables, but is shown in Table 2 below. The scans for the sample and triplicate results appear similar in shape. The mean of the three results is 813.6 with a standard deviation of 143.4. Several factors could have contributed to this variability - the small sample size used for the DSC (typically 15-35 milligrams), the high moisture content of these samples, or insufficient homogenization. Also, contamination of rag material is a possibility (all visible rag material was segregated in the hot cell; however, individual rag fibers could have remained. None of the chemists nor analysts reported seeing fibers contaminating these samples). The DSC results for sample S95T000890 are presented in Table 2 and Table 6.

Table 2. Summary of DSC Results for S95T000890

Sample	Result (J/g)	Duplicate	Triplicate	Mean	Std. Dev.
S95T000890	952.1	665.7	822.9	813.6	143.4

Results for S95T000878 and S95T000881. Reproducible results for samples S95T000878 and S95T000881 were not obtained. The exotherms continued through 500°C (a baseline was not re-established). A modification to the analysis was initiated to increase the temperature limit to 600°C. Even in this case, the exotherms continued without no clear baseline at the limit of the test. The standards which were run with these samples exhibited acceptable recovery

(within 10% of the accepted true value). The empirical observation is that this is a real event. Safety program personnel were consulted when these high exotherms were observed, resulting in a selection of a suite of secondary analyses (discussed below).

The DSC analyzer can only integrate between fixed points on the graph; therefore, since the scans did not return to baseline, these data can only be reported as minimum values. Both samples (S95T000878 and S95T000881) were run in triplicate. The largest exotherm on sample S95T000878 was >1234.0 J/g (dry basis) on the duplicate analysis. The largest exotherm on sample S95T000881 was >1149 J/g (dry basis) on the triplicate analysis. As the scans for these samples did not return to baseline, the RPDs calculated in Table 5 are not applicable. The sample mean and standard deviation were also not calculated for these samples because only "greater than" values were obtained.

The endotherms for these samples were also quite large, dominating the scans up as far as 300°C. In an attempt to isolate the exotherms, subsamples from S95T000878 and S95T000881 were preheated to approximately 240°C by TGA (temperature raised at a rate of 10°C per minute) to remove water from the samples. The subsamples were then analyzed by DSC. These runs, marked as "test," can only be considered unofficial results. The result for S95T000878 was >1977.1 J/g and for S95T000881 was >962.4 J/g. The DSC results for samples S95T000878 and S95T000881 are presented in Table 3.

Table 3. Summary of DSC Results for S95T000878 and S95T000881

Sample	Result	Duplicate	Triplicate	"Test"*	Mean	Std. Dev.
S95T000878	>445.6	>1234.0	>696.5	>1977.1*	n/a	n/a
S95T000881	>647.3	>76.1	>1149.0	>962.4*	n/a	n/a

*"test" results are unofficial and should only be used with caution.

TGA (Moisture Content)

Weight percent water is calculated from weight loss by TGA. These analyses were performed under a nitrogen atmosphere using procedure LA-560-112, Rev. A-2. Results for the three samples and their duplicates ranged in value from 50.44 to 59.92 percent water by weight. Results for sample S95T000878 exceeded the RPD target of 10%. A triplicate analysis was performed, resulting in a determination of 59.48 weight percent water, compared to 58.32% and 50.44% for the sample and duplicate. The mean of the three results for sample S95T000878 is 56.08, with a standard deviation of 4.92. The TGA results for sample S95T000878 are presented in Table 4.

All TGA results were well above the safety screening minimum of 17 weight percent. Three LMCS control standards were run with these analyses, exhibiting recoveries ranging from 99.76 to 100.7 percent, which were within the program's specified accuracy control limits of 90 to 110 percent.

Table 4. Summary of TGA Results for S95T000878

Sample	Result (wt% H2O)	Duplicate	Triplicate	Mean	Std. Dev.
S95T000878	58.32	50.44	59.48	56.08	4.92

Total Alpha Activity

Analyses for total alpha activity were performed on three samples. Samples were prepared by fusion using procedure LA-549-141, Rev. C-3, and analyses were performed using procedure LA-508-101, Rev. D-2. A sample duplicate was performed on each sample. Sample and duplicate results ranged from 0.00643 to 0.0519 $\mu\text{Ci/g}$. The RPDs for samples S95T000879 and S95T000882 exceeded the TCP target of 10%. Since none of the results were more than ten times the detection limit, the variability is expected. As all results were below the safety screening limit of 41 $\mu\text{Ci/g}$ by a factor of approximately 800 or more, reruns were deemed unnecessary.

Two control standards were run, with recoveries of 105.7 and 90.5%, both within the TCP target of 90 to 110%. A spike was performed on sample S95T000879, with a recovery of 61.9%. This is outside of the TCP target recovery of 90 to 110%. Spike recoveries for alpha have typically been below the target criterion. The laboratory is proposing several minor changes to the methodology for this test to improve recovery in some cases. Since the sample results were far below the action limit, the poor spike recovery did not necessitate further testing (this method is for screening purposes - highly accurate results are not required far below the limit).

Secondary Analyses

Planning for secondary analyses was initiated once the exotherms exceeding the DSC criterion were observed. The strategy for secondary analyses was coordinated closely with safety program personnel. Conservation of sample was critical as only small archive samples remained after primary analyses. Two subsamples were submitted for Total Organic Carbon (TOC) determination per the TCP. These results will quantify the amount of organic material in the tank. The cyanide analyses were waived as the history of the tank did not include transfers of ferrocyanide streams. One remaining archive sample will be prepared for adiabatic calorimetry (by a method termed Reactive System Screening Tool). This method is called for as a secondary analysis in the TCP. The RSST result will provide a better understanding of the potential for propagating chemical reactions than the DSC. The final archive sample will be prepared for shipment to PNL for organic speciation. This will identify the organics present in the sample causing the high exotherm, as well as provide useful data for waste aging studies.

This will consume all remaining tank waste samples from tank C-204 (the segregated rag material will be retained for a period of time in the hot cell). Results of secondary analyses will be included in a revision to this report.

Responsible Project Coordinator: J. M. Conner

REFERENCE Schreiber, R. D., 1995, WHC-SD-WM-TP-307, Revision 0, "*Tank 241-C-204 Tank Characterization Plan*", dated March 6, 1995.

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SAMPLE DATA SUMMARY

Summary Tables - Preliminary Safety Screening Results
C-204

TABLE 5

CORE NUMBER: n/a
SEGMENT #: 95-AUG-022

SEGMENT PORTION: U Upper Half of Segment

Sample#	R	A#	Analyte	Unit	Action Limits		Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
					Lower	Upper									
S95T000878			% Water by TGA using Mettler	%	17.000	110.000	100.3	n/a	58.32	50.44	54.38	14.5	n/a	n/a	n/a
S95T000878			DSC Exotherm Dry Calculated	Joules/g Dry	-999.000	481.010	n/a	n/a	> 445.6	>1234.0	n/a	n/a	n/a	1.00e-04	n/a
S95T000878			DSC Exotherm using Mettler	Joules/g	-999.000	481.010	107.9	n/a	>195.7	>542.0	n/a	n/a	n/a	n/a	n/a
S95T000879	F		Alpha of Digested Solid	uCi/g	-999.000	41.010	90.54	<2.81e-03	6.43e-03	1.45e-2	1.05e-02	77.1	61.90	7.00e-03	73.8

L Lower Half of Segment: L Lower Half of Segment

Sample#	R	A#	Analyte	Unit	Action Limits		Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
					Lower	Upper									
S95T000881			% Water by TGA using Mettler	%	17.000	110.000	100.3	n/a	55.02	56.39	55.70	2.46	n/a	n/a	n/a
S95T000881			DSC Exotherm Dry Calculated	Joules/g Dry	-999.000	481.010	n/a	n/a	> 647.3	>76.1	n/a	n/a	n/a	1.00e-04	n/a
S95T000881			DSC Exotherm using Mettler	Joules/g	-999.000	481.010	107.9	n/a	>286.7	>33.4	n/a	n/a	n/a	n/a	n/a
S95T000882	F		Alpha of Digested Solid	uCi/g	-999.000	41.010	90.54	<2.81e-03	2.34e-02	1.21e-2	1.78e-02	63.7	n/a	7.00e-03	33.3

=> Limit violated
=> Selected Limit

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Summary Tables - Preliminary Safety Screening Results
C-204

CORE NUMBER: n/a
SEGMENT #: 95-AUG-023

TABLE 6

SEGMENT PORTION: W Whole Segment

Sample#	R	A#	Analyte	Unit	Action Limits		Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
					Lower	Upper									
S95T000890			% Water by TGA using Mettler	%	17.000	110.000	99.76	n/a	59.92	56.08	58.00	6.62	n/a	n/a	n/a
S95T000890			DSC Exotherm Dry Calculated	Joules/g Dry	-999.000	481.010	n/a	n/a	952.1	665.7	808.9	35.4	n/a	1.00e-04	n/a
S95T000890			DSC Exotherm using Mettler	Joules/g	-999.000	481.010	107.2	n/a	399.9	279.6	339.8	35.4	n/a	n/a	n/a
S95T000891	F		Alpha of Digested Solid	uCi/g	-999.000	41.010	105.7	1.40e-02	5.11e-02	5.19e-2	5.15e-02	1.55	n/a	7.00e-03	17.6

=> Limit violated
=> Selected Limit

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SAMPLE ANALYSES RESULTS

LABCORE Data Entry Template for Worklist# 1378

Analyst: SMF Instrument: DSC0 1 Book # 12N14-A

Method: LA-514-113 Rev/Mod B-1

Worklist Comment: Please run C-204 DSC under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			DSC-01	SOLID	<u>28.45</u>	<u>30.7</u>	<u>N/A</u>	Joules/g
95000069	C-204	2 SAMPLE	S95T000878	0	DSC-01	SOLID	<u>N/A</u>	<u>>195.7</u>		Joules/g
95000069	C-204	3 DUP	S95T000878	0	DSC-01	SOLID	<u>>195.7</u>	<u>>542.0</u>	<u>N/A</u>	Joules/g
95000069	C-204	4 TRIPL	S95T000878	0	DSC-01	SOLID	<u>>195.7</u>	<u>>305.9</u>	<u>N/A</u>	Joules/g
		5 STD			DSC-01	SOLID	<u>28.45</u>	<u>29.5</u>	<u>N/A</u>	Joules/g
95000069	C-204	6 SAMPLE	S95T000881	0	DSC-01	SOLID	<u>N/A</u>	<u>>286.7</u>		Joules/g
95000069	C-204	7 DUP	S95T000881	0	DSC-01	SOLID	<u>>286.7</u>	<u>>33.4</u>	<u>N/A</u>	Joules/g
95000069	C-204	8 TRIPL	S95T000881	0	DSC-01	SOLID	<u>>286.7</u>	<u>>508.8</u>	<u>N/A</u>	Joules/g

Final page for worklist # 1378

See attached for signatures 5/18/95
Analyst Signature BDV Date
Verified by Blandina Valenzuela 5/22/95

[Signature] 5-18-95
Analyst Signature Date

The exotherm values reported are a not the total energy produced from the reaction, the thermogram never returned back to the baseline. Therefore, the results should be considered greater than the reported values.

Data Entry Comments: S95T000878 produced one endotherm of 551.4 J/g at 106.8°C.
S95T000881 produced one endotherm of 484.4 J/g at 134.6°C

LABCORE Data Entry Template for Worklist# 1378

Analyst: SMF Instrument: DSC0 _____ Book # 12N14-A

Method: LA-514-113 Rev/Mod B-1

Worklist Comment: Please run C-204 DSC under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			DSC-01	SOLID	_____	_____	N/A	Joules/g
95000069	C-204	2 SAMPLE	S95T000878	0	DSC-01	SOLID	N/A	_____	_____	Joules/g
95000069	C-204	3 DUP	S95T000878	0	DSC-01	SOLID	_____	_____	N/A	Joules/g
95000069	C-204	4 SAMPLE	S95T000881	0	DSC-01	SOLID	N/A	_____	_____	Joules/g
95000069	C-204	5 DUP	S95T000881	0	DSC-01	SOLID	_____	_____	N/A	Joules/g

Final page for worklist # 1378

Smfulton 5-17-95
Analyst Signature Date

Analyst Signature Date

A triplicate was run on both samples.
5/18/95
BDY

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number,
R = Replicate Number, A = Aliquot Code.

BEST AVAILABLE COPY

SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 11 TO 18.

DSC STD 12N14-A

6.580 mg

Rate: 10.0 °C/min

File: 00015.001

Ident: 0.0

DSC METTLER

16-May-95

222-S Laboratory

exo >

10. mW

Integration

Delta H 202 mJ

30.7 J/g

Peak 158.9°C

-13.4 mW

120.

140.

160.

180. °C

Blandina Valenzuela for SM Fulton
5/17/95

BEST AVAILABLE COPY

S95T000878 N2
22.047 mg
Rate: 10.0 °C/min
File: 00017.001 DSC METTLER 16-May-95
Ident: 0.0 222-S Laboratory

exo >

Integration
Delta H 12157 mJ
551.4 J/g
Peak 106.8°C
-11.5 mW

10. mW

Integration
Delta H 4315 mJ
195.7 J/g
Peak 448.6°C
5.8 mW

Integration
Delta H 30 mJ
1.4 J/g
Peak 289.4°C
-0.5 mW

°C

BEST AVAILABLE COPY

BEST AVAILABLE COPY

S95T000878 (TRIPL) N2
 35.625 mg
 Rate: 10.0 °C/min
 File: 00021.001 DSC METTLER 16-May-95
 Ident: 0.0 222-S Laboratory

exov

Integration
 Delta H16071 mJ
 451.1 J/g
 Peak 162.5°C
 -20.2 mW

20. mW

Integration
 Delta H 250 mJ
 7.0 J/g
 Peak 282.7°C
 3.6 mW

Integration
 Delta H10896 mJ
 305.9 J/g
 Peak 505.9°C
 10.9 mW

BEST AVAILABLE COPY

DSC STD 12N14-A
6.746 mg
Rate: 10.0 °C/min
File: 00036.001
Ident: 0.0
DSC METTLER
222-S Laboratory
17-May-95

Blandina Valenzuela for SM Fulton 5/17/95

120. 140. 160. 180. °C

BEST AVAILABLE COPY

S95T000881 N2
25.005 mg

File: 00037.001 DSC METTLER 17-May-95
Ident: 0.0 222-S Laboratory

Rate: 10.0 °C/min

EXO

Integration
Delta H12113 mJ
484.4 J/g
Peak 134.6°C
-12.0 mW

Integration
Delta H 7168 mJ
286.7 J/g
Peak 462.7°C
8.9 mW

10. mW

BEST AVAILABLE COPY

S95T000881 (DUP) N2
 25.525 mg
 Rate: 10.0 °C/min
 File: 00039.001 DSC METTLER 17-May-95
 Ident: 0.0 222-S Laboratory

exo >

Integration
 Delta H14902 mJ
 583.8 J/g
 Peak 102.8 °C
 -11.8 mW

Integration
 Delta H 853 mJ
 33.4 J/g
 Peak 341.6 °C
 1.7 mW

BEST AVAILABLE COPY

S95T000881 (TRIPL) N2
 26.737 mg
 Rate: 10.0 °C/min
 File: 00041.001
 Ident: 0.0
 DSC METTLER
 222-S Laboratory
 17-May-95

exo >

Integration
 Delta H12822 mJ
 479.6 J/g
 Peak
 107.0 °C
 -12.8 mW

Integration
 Delta H13603 mJ
 508.8 J/g
 Peak
 560.0 °C
 14.4 mW

LABCORE Data Entry Template for Worklist# 1379

Analyst: SMP Instrument: DSC0 1 Book # 12N14-A

Method: LA-514-113 Rev/Mod B-1

Worklist Comment: Please run C-204 DSC under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			DSC-01	SOLID	<u>28.45</u>	<u>30.5</u>	<u>N/A</u>	Joules/g
95000069	C-204	2 SAMPLE	S95T000890	0	DSC-01	SOLID	<u>N/A</u>	<u>399.9</u>		Joules/g
95000069	C-204	3 DUP	S95T000890	0	DSC-01	SOLID	<u>399.9</u>	<u>279.6</u>	<u>N/A</u>	Joules/g
95000069	C-204	4 TRIPL	S95T000890	0	DSC-01	SOLID	<u>399.9</u>	<u>345.6</u>	<u>N/A</u>	Joules/g

Final page for worklist # 1379

See attached for signatures 5/11/95
Analyst Signature Date
Bdv

 5-12-95
Analyst Signature Date

Verified 5/12/95 James M. Fyfe

Data Entry Comments:

Sample produced one endotherm at 132.6°C with a
delta H of 422.2 J/g. Sample looked like stiff dark chocolate frosting.

LABCORE Data Entry Template for Worklist# 1379

Analyst: SMF Instrument: DSC0 _____ Book # 12N14-A

Method: LA-514-113 Rev/Mod B-1

Worklist Comment: Please run C-204 DSC under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			DSC-01	SOLID	_____	_____	N/A	Joules/g
95000069	C-204	2 SAMPLE	S95T000890	0	DSC-01	SOLID	N/A	_____	_____	Joules/g
95000069	C-204	3 DUP	S95T000890	0	DSC-01	SOLID	_____	_____	N/A	Joules/g

Final page for worklist # 1379

Sm Fulton 5-10-95
Analyst Signature Date

Analyst Signature Date

Triplicate was run. 5/11/95
BDV

Data Entry Comments:

sample is like stiff ^{dark} chocolate frosting

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 21 TO 24.

DSC STD 12N14-A

6.632 mg

Rate: 10.0 °C/min

File: 00033.001

DSC METTLER 10-May-95

Ident: 0.0

222-S Laboratory

exo>

10. mW

Integration
Delta H 202 mJ
30.5 J/g
Peak 158.6°C
-13.5 mW

120.

140.

160.

180.

°C

Susie M. Fulton 5-10-95

WHC-SD-WM-DP-115, REV.0

S95T000890 N2
25.214 mg

Rate: 10.0 °C/min

File: 00037.001

DSC METTLER

10-May-95

Ident: 0.0 222-S Laboratory

exo

Integration

Delta H10645 mJ

422.2 J/g

Peak

132.6°C

-13.3 mW

Integration

Delta H10084 mJ

399.9 J/g

Peak

450.7°C

14.7 mW

20. mW

°C

400.

300.

200.

100.

S95T000890 (DUP) N2
 20.209 mg
 Rate: 10.0 °C/min
 File: 00039.001 DSC METTLER 10-May-95
 Ident: 0.0 222-S Laboratory

exo

Integration
 Delta H10851 mJ
 536.9 J/g
 Peak 223.0 °C
 -9.8 mW

Integration
 Delta H 5651 mJ
 279.6 J/g
 Peak 336.6 °C
 12.1 mW

10. mW

100. 200. 300. 400. °C

S95T000890 (TRIPL) N2
 25.723 mg
 Rate: 10.0 °C/min
 File: 00041.001 DSC METTLER 10-May-95
 Ident: 0.0 222-S Laboratory

exo >

Integration
 Delta H 11839 mJ
 460.2 J/g
 Peak 136.5 °C
 -14.5 mW

Integration
 Delta H 8889 mJ
 345.6 J/g
 Peak 452.6 °C
 14.2 mW

10. mW

100. 200. 300. 400. °C

LABCORE Data Entry Template for Worklist# 1431

Analyst: BDV Instrument: DSC0 2 Book # —

Method: LA-514-113 Rev/Mod —

Worklist Comment: Calculated dry DSC for C-204. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
95000069	C-204	1 SAMPLE	S95T000878	0	DSC-02	SOLID	<u>N/A</u>	<u>>445.6</u>		Joules/g Dry
95000069	C-204	2 DUP	S95T000878	0	DSC-02	SOLID	<u>>445.6</u>	<u>>1234.0</u>	<u>N/A</u>	Joules/g Dry
95000069	C-204	3 TRIPL	S95T000878	0	DSC-02	SOLID	<u>>445.6</u>	<u>>696.5</u>	<u>N/A</u>	Joules/g Dry
95000069	C-204	4 SAMPLE	S95T000881	0	DSC-02	SOLID	<u>N/A</u>	<u>>647.3</u>		Joules/g Dry
95000069	C-204	5 DUP	S95T000881	0	DSC-02	SOLID	<u>>647.3</u>	<u>>76.1</u>	<u>N/A</u>	Joules/g Dry
95000069	C-204	6 TRIPL	S95T000881	0	DSC-02	SOLID	<u>>647.3</u>	<u>>1149.0</u>	<u>N/A</u>	Joules/g Dry
95000069	C-204	7 SAMPLE	S95T000890	0	DSC-02	SOLID	<u>N/A</u>	<u>952.1</u>		Joules/g Dry
95000069	C-204	8 DUP	S95T000890	0	DSC-02	SOLID	<u>952.1</u>	<u>665.7</u>	<u>N/A</u>	Joules/g Dry
95000069	C-204	9 TRIPL	S95T000890	0	DSC-02	SOLID	<u>952.1</u>	<u>822.9</u>	<u>N/A</u>	Joules/g Dry

Data entered + verified by **Final page for worklist # 1431**

Blandina Valenzuela 5/22/95
Analyst Signature Date

Analyst Signature Date

Data Entry Comments:

26

WHC-SD-WM-DP-115, REV. 0

The following DSC runs were performed in an attempt to better understand the exothermic characteristics of the C-204 samples. The samples were pre-dried before being run on the DSC. These are unofficial results. These test runs are described in the narrative.

Sample S95T000878	TEST
Sample S95T000881	TEST

COPY

S95T000878 (TEST) N2
 7.155 mg
 Rate: 10.0 °C/min
 File: 00055.001 DSC METTLER 19-May-95
 Ident: 0.0 222-S Laboratory

exo >

Integration
 Delta H14146 mJ
 1977.1 J/g
 Peak 441.0 °C
 18.0 mW

10. mW

COPY

S95T000881 (TEST) N2
 12.911 mg
 Rate: 10.0 °C/min
 File: 00057.001 DSC METTLER 19-May-95
 Ident: 0.0 222-S Laboratory

EXO >

Integration
 Delta H12426 mJ
 962.4 J/g
 Peak 547.8 °C
 12.3 mW

10. mW

LABCORE Data Entry Template for Worklist# 1374

Analyst: SMF Instrument: TGA0 1 Book # 42N8-A

Method: LA-560-112 Rev/Mod A-2

Worklist Comment: Please run C-204 TGA under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			TGA-01	SOLID	<u>59.19</u>	<u>59.39</u>	N/A	%
95000069	C-204	2 SAMPLE	S95T000878	0	TGA-01	SOLID	N/A	<u>58.32</u>		%
95000069	C-204	3 DUP	S95T000878	0	TGA-01	SOLID	<u>58.32</u> <u>50.44</u>	<u>50.44</u>	N/A	%
95000069	C-204	4 TRIPL	S95T000878	0	TGA-01	SOLID	<u>58.32</u>	<u>59.48</u>	N/A	%
		5 STD			TGA-01	SOLID	<u>59.19</u>	<u>58.77</u>	N/A	%
95000069	C-204	6 SAMPLE	S95T000881	0	TGA-01	SOLID	N/A	<u>55.02</u>		%
95000069	C-204	7 DUP	S95T000881	0	TGA-01	SOLID	<u>55.02</u>	<u>56.39</u>	N/A	%

Final page for worklist # 1374

See attached for signatures
Analyst Signature _____ Date _____

MS 5-17-95
Analyst Signature _____ Date _____

Verified by Blandina Valenzuela 5/22/95

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

LABCORE Data Entry Template for Worklist# 1374

Analyst: SMF Instrument: TGA0 _____ Book # 42 N8-A

Method: LA-560-112 Rev/Mod A-2

Worklist Comment: Please run C-204 TGA under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			TGA-01	SOLID	_____	_____	N/A	%
95000069	C-204	2 SAMPLE	S95T000878	0	TGA-01	SOLID	N/A	_____	_____	%
95000069	C-204	3 DUP	S95T000878	0	TGA-01	SOLID	_____	_____	N/A	%
95000069	C-204	4 SAMPLE	S95T000881	0	TGA-01	SOLID	N/A	_____	_____	%
95000069	C-204	5 DUP	S95T000881	0	TGA-01	SOLID	_____	_____	N/A	%

Final page for worklist # 1374

Sm-fulton 5-17-95
Analyst Signature Date

Analyst Signature Date

Data Entry Comments:

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number,
R = Replicate Number, A = Aliquot Code.

SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 29 TO 35.

TGA STD 42N8-A

18.284 mg

Rate: 10.0 °C/min

File: 00016.001

Ident: 0.0

TG

METTLER

16-May-95

222-S Laboratory

Step Analysis

Height-10.86 mg

-59.39 %

ResiC. 7.43 mg

40.61 %

Dpeak 81.7 °C

5. mg

50.

100.

150.

200.

°C

Blandina Valenzuela for SM Fulton
5/17/95

WHC-SD-WM-DP-115, REV.0

S95T000878 (DUP) N2

22.940 mg

Rate: 10.0 °C/min

File: 00020.001

TG

METTLER

16-May-95

Ident: 0.0

222-S Laboratory

Step Analysis

Height-11.57 mg

-50.44 %

ResiC. 11.37 mg

49.56 %

Dpeak 183.0 °C

31

5. mg

100.

200.

300.

400.

°C

S95T000878 (TRIPL) N2

10.260 mg

Rate: 10.0 °C/min

File: 00022.001

TG

METTLER

16-May-95

Ident: 0.0

222-S Laboratory

Step Analysis

Height -6.10 mg

-59.48 %

ResiC. 4.16 mg

40.52 %

Dpeak 173.0 °C

32

TGA STD 42N8-A

18.250 mg

Rate: 10.0 °C/min

File: 00035.001

TG

METTLER

17-May-95

Ident: 0.0

222-S Laboratory

Step Analysis

Height-10.73 mg

-58.77 %

ResiC. 7.52 mg

41.23 %

Dpeak 80.8 °C

Blandina Valenzuela for SM Fulton
5/17/95

S95T000881 N2

23.401 mg

Rate: 10.0 °C/min

File: 00038.001

TG

METTLER

17-May-95

Ident: 0.0

222-S Laboratory

Step Analysis

Height-12.88 mg

-55.02 %

ResiC. 10.52 mg

44.98 %

Dpeak 191.0 °C

S95T000881 (DUP) N2

19.399 mg

Rate: 10.0 °C/min

File: 00040.001

TG

METTLER

17-May-95

Ident: 0.0

222-S Laboratory

Step Analysis

Height-10.94 mg

-56.39 %

Resid. 8.46 mg

43.61 %

Dpeak 181.0 °C

35

5. mg

LABCORE Data Entry Template for Worklist# 1375

Analyst: SMF Instrument: TGA0 1 Book # 42N8-A

Method: LA-560-112 Rev/Mod A-2

Worklist Comment: Please run C-204 TGA under N2. bdv

GROUP	PROJECT	S TYPE	SAMPLE#	R A	-----TEST-----	MATRIX	ACTUAL	FOUND	DL	UNIT
		1 STD			TGA-01	SOLID	<u>59.19</u>	<u>59.05</u> <u>59.04</u>	<u>5/11/95</u> <u>BDV N/A</u>	%
95000069	C-204	2 SAMPLE	S95T000890	0	TGA-01	SOLID	<u>N/A</u>	<u>59.92</u>		%
95000069	C-204	3 DUP	S95T000890	0	TGA-01	SOLID	<u>59.92</u>	<u>56.08</u>	<u>N/A</u>	%

Final page for worklist # 1375

Smulter 5-10-95
Analyst Signature Date

L. Jones 5-12-95
Analyst Signature Date

Verified 5/12/95 Jean M. Lye

Data Entry Comments:

sample is like stiff dark chocolate frosting

Units shown for QC (SPK & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate Number, A = Aliquot Code.

SIGNATURE BELOW REPRESENTS CHEMICAL TECHNOLOGIST/CHEMIST THAT
COMPLETED/VERIFIED THE CALIBRATION/ANALYSIS ON PAGES 37 TO 39.

TGA STD 42N8-A

19.976 mg

Rate: 10.0 °C/min

File: 00034.001

TG

METTLER

10-May-95

Ident: 0.0

222-S Laboratory

Step Analysis

Height-11.80 mg

-59.05 %

ResidC. 8.18 mg

40.95 %

Dpeak 91.7°C

5. mg

50.

100.

150.

200.

°C

Susie M. Fulton 5-10-95

WHC-SD-WM-DP-115, REV. 0

S95T000890 (DUP) N2
27.826 mg

File: 00042.001

TG METTLER 10-May-95
222-S Laboratory

Ident: 0.0

Rate: 10.0 °C/min

Step Analysis
Height-15.60 mg
-56.08 %
ResidC. 12.22 mg
43.92 %
Dpeak 193.0 °C