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R. E. SIIVEIS		F 1 - L1			
<pre>U.S. Department of C. A. Babel</pre>	Energy, RL	S7-54	X		
Westinghouse Hanfo	rd Company				
J. N. Appel	<u> </u>	G3-21		X	
H. Babad		\$7-30	X		
K. E. Bell R. J. Cash		T6-06 S7-15	X X		
J. L. Deichman		H4-19	^	χ	
G. D. Forehand		\$7-31		χ̈́	¢
C. E. Golberg		H5-49		Х	
V. W. Hall		H4-21		X	
D. C. Hetzer		\$6-31	v	X	
L. Jensen G. D. Johnson		T6-07 S7-15	X X		
N. W. Kirch		R2-11	χ		
J. G. Kristofzski		T6-06	χ		
M. J. Kupfer		H5-49	X		
E. J. Lipke		S7-14	v	X	
N. G. McDuffie J. E. Meacham		S7-15 S7-15	X X		
P. M. Morant		37-13 H4-25	Ŷ		
B. C. Simpson		R2-12		X	
D. A. Turner		S7-15	X *-		
J. A. Voogd		R4-01	/	X	
Central Files		L8-04	2		
EDMC		H6-08	X	v	
LTIC OSTI		T6-03 L8-07	2	X	
TCRC		R2-12	2 2		
TFIC (Tank Farm In	formation Center)	R1-20	L	X	

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Environmental Prot Single-Shell Tank D. R. Einan 712 Swift Boulevar Richland, Washingt	Unit Manager rd, Suite 5		X		
U. S. Department of Jim Poppiti 12800 Middlebrook Trevion II, EM-36 Germantown, MD 20	Rd.			X	
Los Alamos Technic A. T. DiCenso 750 Swift Boulevar Suite # 4 Richland, WA 9935	rd		X		

ENGINEERING DATA TRANSMITTAL

Page 1 of 1. EDT Nº 612155

		 										
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Push Mode, Cores 81 and 82

Release Date:

5/18/95

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7. Abstract

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ANALYTICAL SERVICES

Project:

45-DAY SAFETY SCREEN RESULTS FOR TANK 241-U-204, PUSH MODE, CORES 81 AND 82

Date Printed:

MAY 17, 1995

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NARRATIVE

45-DAY SAFETY SCREEN RESULTS FOR TANK 241-U-204 PUSH-MODE CORE SAMPLES 81 AND 82.

This is the 45-Day report for the fiscal year 1995 tank 241-U-204 (U-204) push-mode characterization effort. Included are a summary of analytical results and copies of the differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) scans as requested in Reference 1. Other pertinent documentation will be included in the U-204 216-day report.

Summary

Core samples 81 and 82 from tank U-204, obtained by the push-mode core sampling method, were received by the 222-S Laboratories. Each core consisted of only one segment. Both core samples and the field blank were extruded, subsampled, and analyzed in accordance with Reference 1. Drainable liquids and the field blank were analyzed at the segment level for energetics by DSC, percent water by TGA, and total organic carbon (TOC) by furnace oxidation. In addition, the presence or absence of any separable, presumably organic, layer in drainable liquid samples was noted and none was observed. The solids were analyzed directly at the half segment level for energetics by DSC, percent water by TGA, and TOC by persulfate oxidation. Total alpha activity was determined on fusion digestions of the sludge subsamples. No immediate notifications were necessary on samples from cores 81 or 82.

Sample Receipt and Extrusion

U-204 Core 81, Segment 1

Segment 1 of core 81 was removed from riser 2 of tank U-204 on 4/5/95 at 0941 hours. The sample was shipped to the 222-S Laboratory on 4/6/95 and received at the 222-S Laboratory on the same day. Loading and extrusion of the sample in the hot cell took place on 4/7/95. No liner liquid was observed and no problems were noted in extruding the sample.

Collected during extrusion first was 174.56 g of turbid, orange-brown colored drainable liquid followed by approximately eight inches of sludge from the upper half of the sampler. The color of the sludge ranged from a medium brown color for the first two inches to a cream color for the last four inches. The solids had a smooth consistency and the last four inches to extrude appeared to contain less moisture, since this material maintained its shape in the extrusion tray unlike the first four inches. A total of 193.78 g of sludge was collected in one jar.

U-204 Core 82, Segment 1

The first and only segment of core 82 was removed from U-204 on 4/5/95 at 1828 hours. It was sent to, and received at, the 222-S Laboratory the following day. Extrusion took place on 4/7/95. No liner liquid was observed and no problems were noted during the extrusion.

Collected first during the extrusion was 96.28 g of turbid drainable liquid that was yellow in color. This was followed by six to seven inches of solid,

cream colored sample from the upper half of the sampler. The first three to four inches of the solids were wet and crumbly, while the final three inches were smooth and retained their shape. All solids were collected in one jar to yield 126.60 g.

U-204 Field Blank

The field blank was created on 4/5/95, shipped to the laboratory on 4/6/95, and received the same day. Extrusion took place on 4/7/95. There was no liner liquid and no problems were noted during the extrusion.

A mass of 267.75 g of drainable liquid was collected in two sample jars as a clear, colorless liquid. The material in jar 6782 was subsampled for analysis.

Analytical Results

The safety screening analytical results are presented in Table 1, which includes the LabCore sample number. Column 2 of the table indicates the sample preparation used, if any. As shown, analyses on fused samples are marked with a "F". Results for core 81 are designated as segment 81-1 in Table 1 and appear on the first page of the table. Results for core 82 are designated as segment 82-1.

Drainable liquid samples were not filtered prior to analysis as required in Reference 1. Analyses were inadvertently performed on the unfiltered liquid and were not subsequently performed on filtered samples since the samples showed no exotherm, the TOC content was below or near the detection limit, and the percent water by TGA was three to four times the action limit of 17%.

DSC (Energetics Content)

Analyses for DSC were performed under a nitrogen atmosphere using procedure LA-514-113, Rev B-1. No exotherms are observed for the sample and duplicate runs of the drainable liquid, field blank, or sludge samples, therefore no exotherms are calculated on a dry weight basis. The DSC profile of the drainable liquid samples from each core is very similar as is the profile of the sludge samples from each core. Following the endotherm associated with the heating and vaporization of water centered at roughly 102° C, each drainable liquid sample shows a much smaller endotherm centered at approximately 160° C. Each sludge samples also shows a water vaporization endotherm at about 100° C followed by a second endotherm of approximately equal magnitude centered at about 292° C. The DSC scans are provided as an attachment.

TGA (Moisture Content)

Weight percent water by TGA was performed under a nitrogen atmosphere using procedure LA-560-112, Rev. A-2. The moisture content in the sludge samples are similar with results ranging from 21.84 to 29.56% water. It appears that the core 82 sludge contains slightly less moisture than the core 81 sludge. At 10.2%, the relative percent difference (RPD) between the sample and

duplicate results from core 82 was slightly above the limit specified in Reference 1. No re-runs were performed. The water content in the two drainable liquid samples was very close. The core 81 liquid averaged 86.67% water while the core 82 liquid averaged 86.68% water. Percent water in the field blank sample was found to be 99.81, close to the expected 100%.

Total Alpha

Total alpha analyses were performed on fusion digestions of both sludge subsamples using procedure LA-508-101, Rev D-2. All results are at least a factor of 424 below the notification limit of 41 uCi/g. The sample and duplicate results from core 82 are below the detection limit of 0.0960 uCi/g. The sample result from core 81 was slightly above the detection limit and the duplicate result was below the detection limit. At 85.81%, the standard percent recovery on both sludge samples was outside of the $\pm 10\%$ limit specified in Reference 1. In addition, the spike recovery on the core 82 sludge sample was scarcely below the 90-110% limit at 89.40%. No re-runs were requested due to the very low activity of the samples.

Total Organic Carbon

Analyses for TOC were performed by direct persulfate oxidation on sludge, drainable liquid, and field blank samples per procedure LA-342-100, Rev. A-O. At 815 ug C/g, the sludge in core 81 was richer in TOC than core 82 sludge by a factor of approximately 6.5. The drainable liquids contained even less TOC. The core 82 liquid was less than the 80 ug C/mL detection limit and the core 81 liquid contained 82 ug C/mL. The RPD between sample and duplicate runs of the latter sample was 21.5%, which exceeds the Reference 1 limit of 10%. The 112% spike recovery on core 81 sludge also exceeds the Reference 1 limit of 90-110%. No re-runs were requested since all results were more than an order of magnitude lower than the notification limit of 30,000 ug C/g or 30,000 ug C/mL. The TOC content of the field blank was found to be below the detection limit of 80 ug C/mL.

Reference 1) WHC-SD-WM-TP-311, Rev. 0, "Tank 241-U-204 Tank Characterization Plan", dated March 23, 1995, Westinghouse Hanford Company, Richland, WA 99352.

SAMPLE DATA SUMMARY

Page: 1

Table 1. Analytical Summary for U-204, Cores 81 and 82. \$U-204\$

CORE NUMBER: 81 & 82 SEGMENT #: 81-1

SEGMENT PORTION: U Upper Half of Segment

FUNTION. U U	pper nacr or segment										
Sample#	R A# Analyte	Unit	Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
S95T000717	% Water by TGA using Mettler	%	99.59	n/a	29.12	29.56	29.34		n/a	n/a	n/a
S95T000717	DSC Exotherm using Mettler	Joules/g	104.7	n/a	0.00e+00	0	0.000			n/a	
S95T000717	TOC by Persulfate/Coulometry	ug/g	91.00	21.10	807.0	8.23e2	8.15e+02	1.96	112.0	80.00	n/a
S95T000721	F Alpha of Digested Solid	uCi/g	85.81	<4.34e-02	9.67e-02	<6.23e-2	n/a	n/a	92.80	9.60e-02	72.3

Drainable Liquid: Drainable Liquid

pramable bit	quid. Diamable Elquid						The second secon				
						**					
Sample# F	R A# Analyte	Unit	Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
S95T000723	% Water by TGA using Mettler	%	100.4	n/a	86.46	86.89	86.67	0.50	n/a	n/a	n/a
S95T000723	DSC Exotherm using Mettler	Joules/g	98.77	n/a	0.00e+00	0	0.000	n/a	n/a	n/a	n/a
S95T000723	TOC by Persulfate/Coulometry	ug/mL	94.33	35.50	90.80	7.32e01	8.20e+01	21.5	n/a	40.00	n/a

Table 1. Analytical Summary for U-204, Cores 81 and 82. U-204

CORE NUMBER: 81 & 82 SEGMENT #: 82-1

SEGMENT PORTION: U Upper Half of Segment

Sample#	R A#	Analyte	Unit	Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
S95T000718		% Water by TGA using Mettler	%	99.59	n/a	24.18	21.84	23.01	10.2	n/a	n/a	n/a
S95T000718		DSC Exotherm using Mettler	Joules/g	104.7	n/a	0.00e+00	0	0.000	n/a	n/a	n/a	n/a
S95T000718		TOC by Persulfate/Coulometry	ug/g	91.00	21.10	120.0	1.28e2	1.24e+02	6.45	n/a	80.00	n/a
S95T000722	F	Alpha of Digested Solid	uCi/g	85.81	<4.34e-02	< 4.36e-2	<4.40e-2	n/a	n/a	89.40	9.60e-02	49.0

D	rainabl	le Lic	guid:	Drainabl	e Liquid

Sample#	R A#	Analyte	Unit	Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
S95T000724		% Water by TGA using Mettler	%	100.4	n/a	87.18	86.18	86.68	1.15	n/a	n/a	n/a
S95T000724		DSC Exotherm using Mettler	Joules/g	98.77	n/a	0.00e+00	0	0.000	n/a	n/a	n/a	n/a
S95T000724		TOC by Persulfate/Coulometry	ug/mL	92.67	38.20	< 80	<80	n/a	n/a	n/a	80,00	n/a

17-may-1995 08:23:10

A-0002-1

Table 1. Analytical Summary for U-204, Cores 81 and 82. U-204

CORE NUMBER: 81 & 82 SEGMENT #: field blank

SEGMENT PORTION: Drainable Liquid

FUNITURE DIGI	Habite Eliquid						T				
Sample#	R A# Analyte	Unit	Standard %	Blank	Result	Duplicate	Average	RPD %	Spk Rec %	Det Limit	Count Err%
\$951000732	% Water by TGA using Mettler	%	99.63	n/a	99.01	100.6	99.81	1.59	n/a	n/a	n/a
S95T000732	DSC Exotherm using Mettler	Joules/g	100.9	n/a	0.00e+00	0	0.000	n/a	n/a		
S95T000732	TOC by Persulfate/Coulometry	ug/mL	92.67	38.20	< 80	<80	n/a	n/a	n/a	80.00	n/a

UNDIGESTED ANALYSES RESULTS - DIRECT

worklistrpt Version 2.0 02/21/95 05/08/95 11:39

LABCORE Data Entry Template for Worklist# 1215

ADP Book # 12N14-A Instrument: DSC01 Analyst:

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

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

				• •					
GROUP	PROJECT	S TYPE	SAMPLE#	R ATEST	MATR1X	ACTUAL	FOUND	DL	UNIT
		1 STD		DSC-01	SOLID	28.45	29.8	N/A	_ Joules/g
95000058	U-204	2 SAMPLE	\$951000717	0 DSC-01	SOLID	N/A	Ø		_ Joules/g
95000058	U-204	3 DUP	\$951000717	0 DSC-01	SOLID	_Ø_	<u>Ø</u> .	N/A	_ Joules/g
95000058	U-204	4 SAMPLE	\$951000718	0 DSC-01	SOLID	<u>N/A</u>	<u>Ø</u>	· ———	_ Joules/g
		5 STD		DSC-01	SOLID	28,45	29.6	N/A	_ Joules/g
95000058	U-204	6 DUP	\$951000718	0 DSC-01	SOLID	Ø	<u>Ø</u>	N/A	_ Joules/g

Final page for worklist # 1215

Verified by Blandina Valentuela 5/9/95

The dup for S95T000718 was analyzed of a day after the previous samples, was the technician did not have time to finish the work ust.

Data Entry Comments: S95T000717 produced two endotherms one at 99.1°C with a delta H 183.8 Tg and the second at 291.7°C with a delta H of 613.37/4. S95T000718 introduced two enaotherms on at 104.5°C with a delta H of 681.7 J/g and second at 296.9°C with a delta H of 743.4 J/g.
Units shown for QC (SPR & STD) may not reflect the actual units. DL = Detection Limit, S = Worklist Slot Number, R = Replicate' Number, A = Aliquot Code.

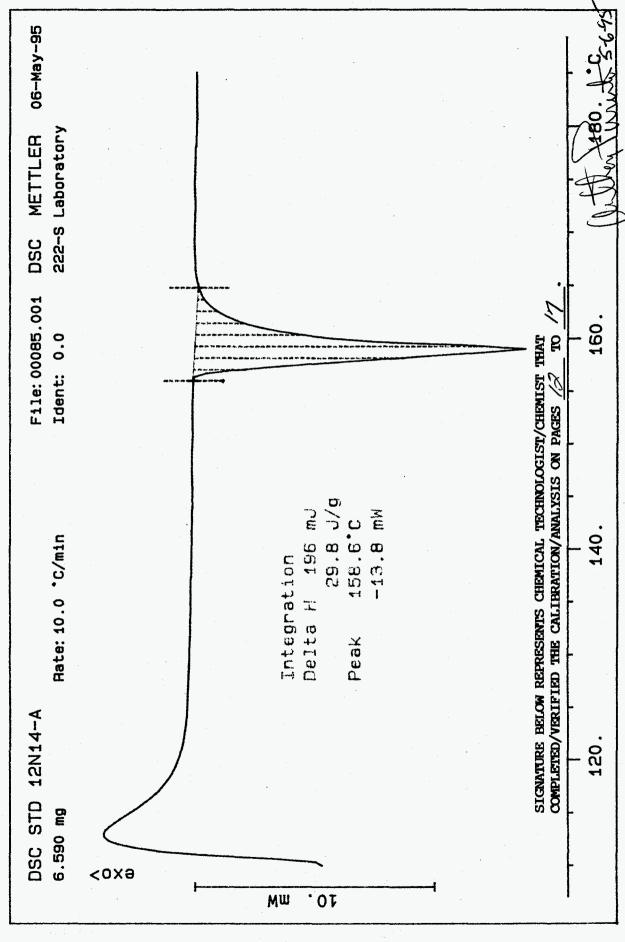
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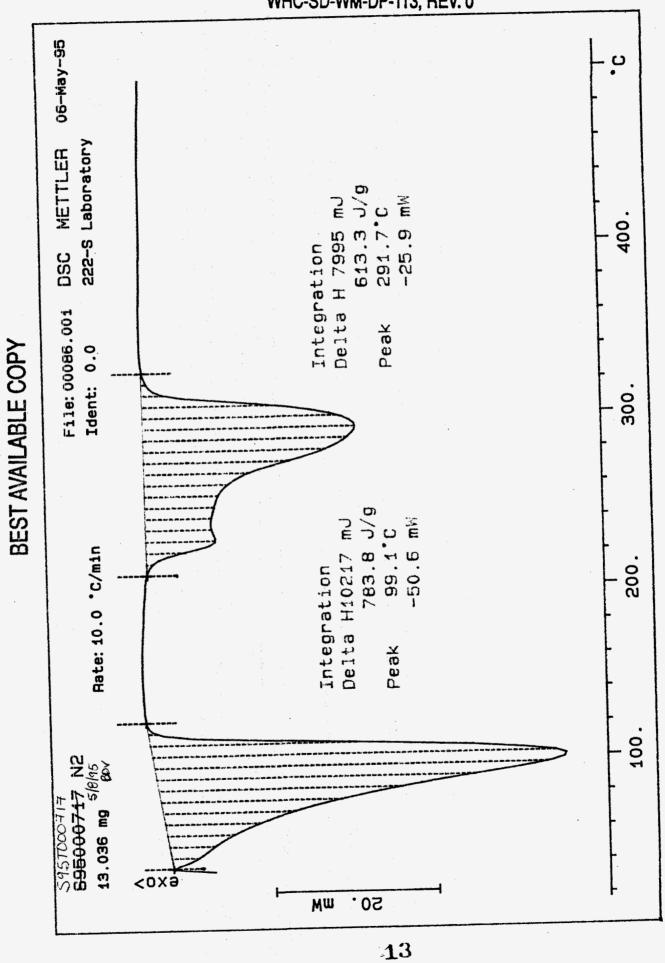
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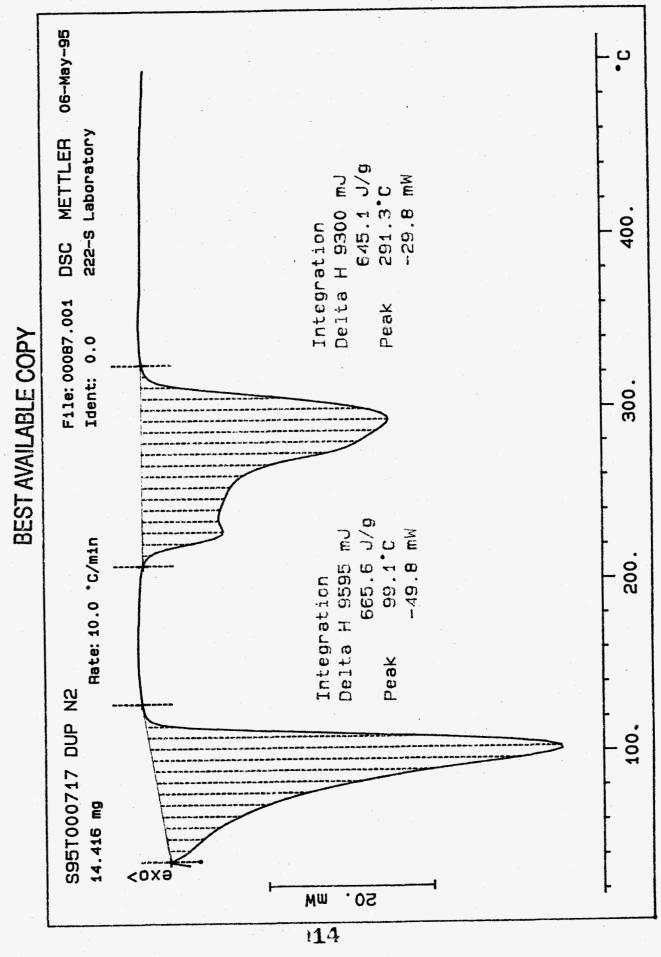
LABCORE Data Entry Template for Worklist# 1215

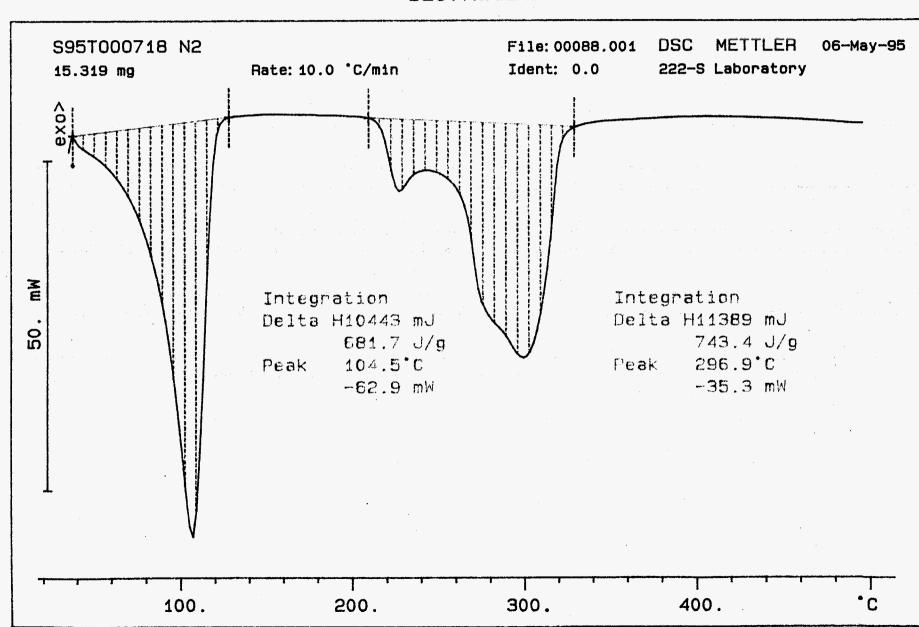
Analyst:	ADP	Instrument	: DSC01	· · · · · · · · · · · · · · · · · · ·	Book # 12	N14-	A
Method: LA-5	514-113 Rev/Mo	od <u>Bl</u>					
Worklist Com	ment: Please r	un U-204 DSC un	der N2. bdv				
GROUP PROJECT	S TYPE	SAMPLE# R A -	TEST	MATRIX AC	TUAL FOUND	DL	UNIT
	1 STD		DSC-01	SOLID		N/A	_ Joules/g
95000058 U-204	2 SAMPLE	S95T000717 0	DSC-01	SOLID	N/A		_ Joules/g
95000058 U-204	3 DUP	S95T000717 0	DSC-01	SOLID		N/A	_ Joules/g
5000058 U-204	4 SAMPLE	S95T000718 0	DSC-01	SOL ID	N/A	·	_ Joules/g
P5000058 U-204	5 DUP	S95T000718 0	DSC-01	SOLID		N/A	_ Joules/g
CM		Final pa	age for wo	rklist #	# 1215		
July Tu	rute Dat	-6-95	age for wo			Date	
Analyst Signar	ruth 5 ture Date	-6-95	age for wo		# 1215 Signature	Date	
Analyst Signar	ture Date	-6-95	age for wo			Date	
Analyst Signar	ture Dat	-6-95	age for wo			Date	
Analyst Signat	ture Date	-6-95	age for wo			Date	
Analyst Signat	ture Date	-6-95	age for wo			Date	

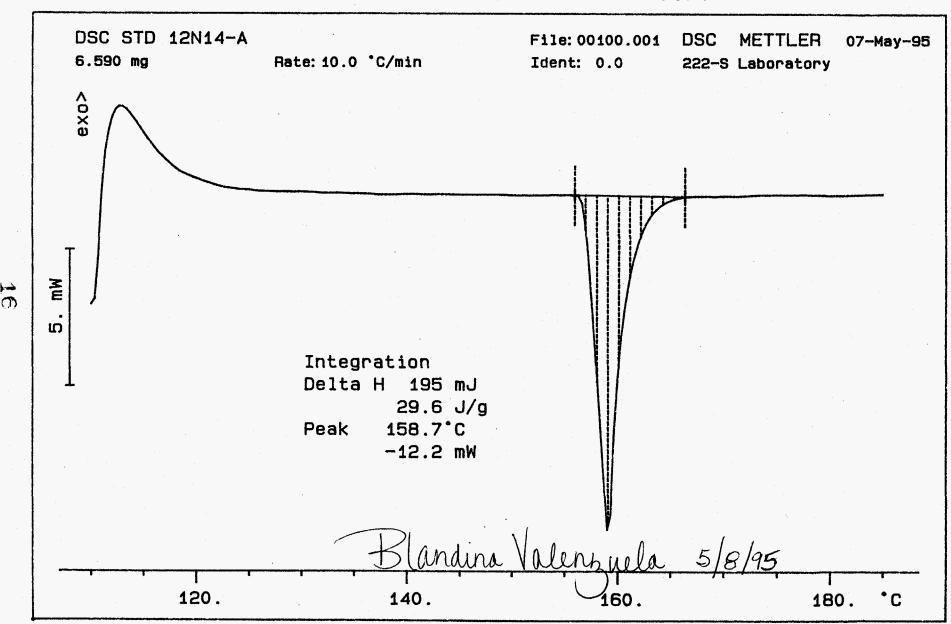
Data Entry Comments:		

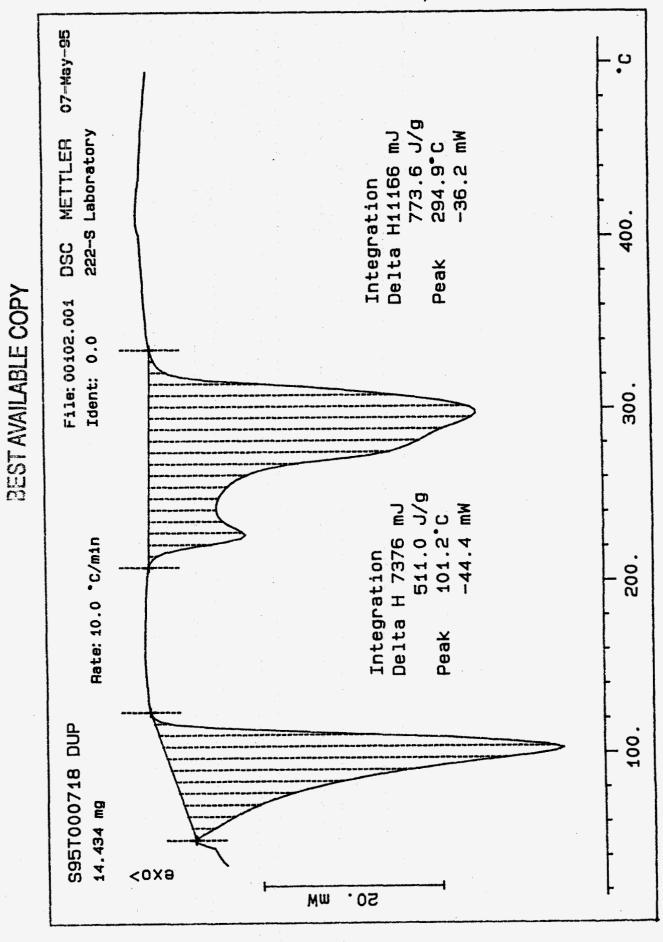










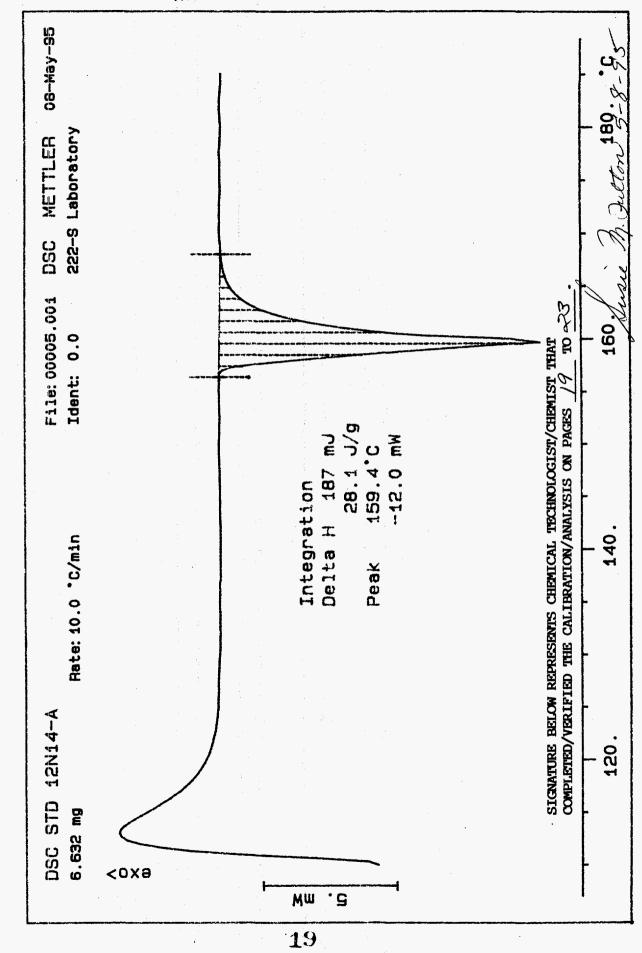


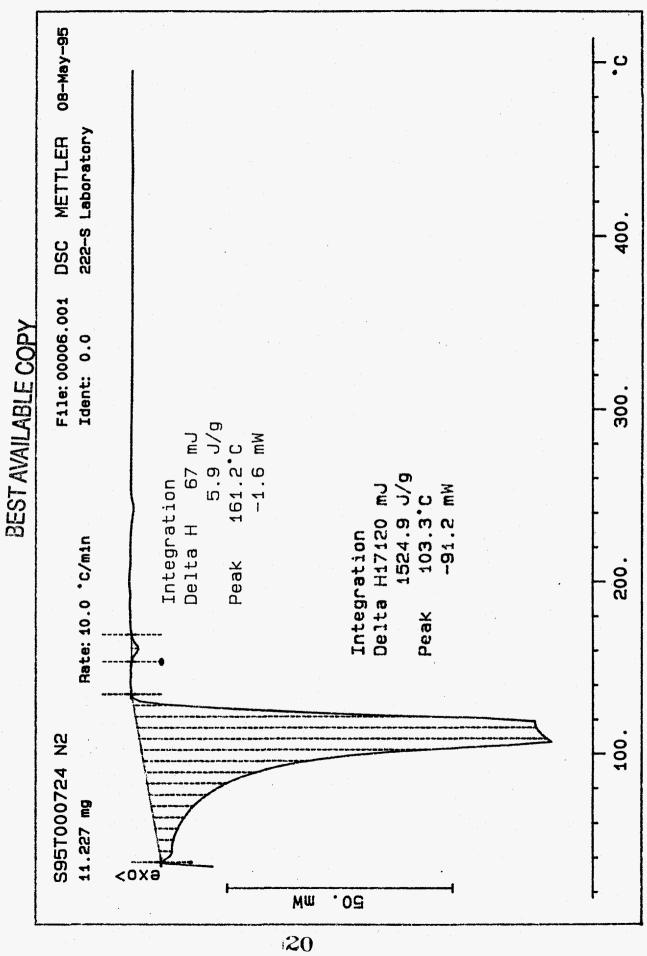
Page:

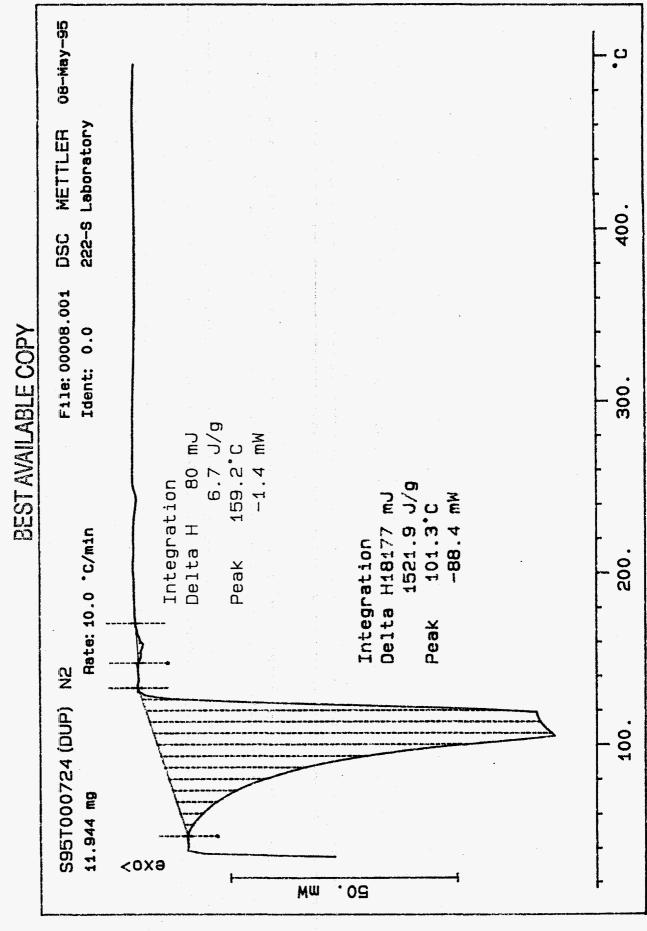
04/27/95 07:13

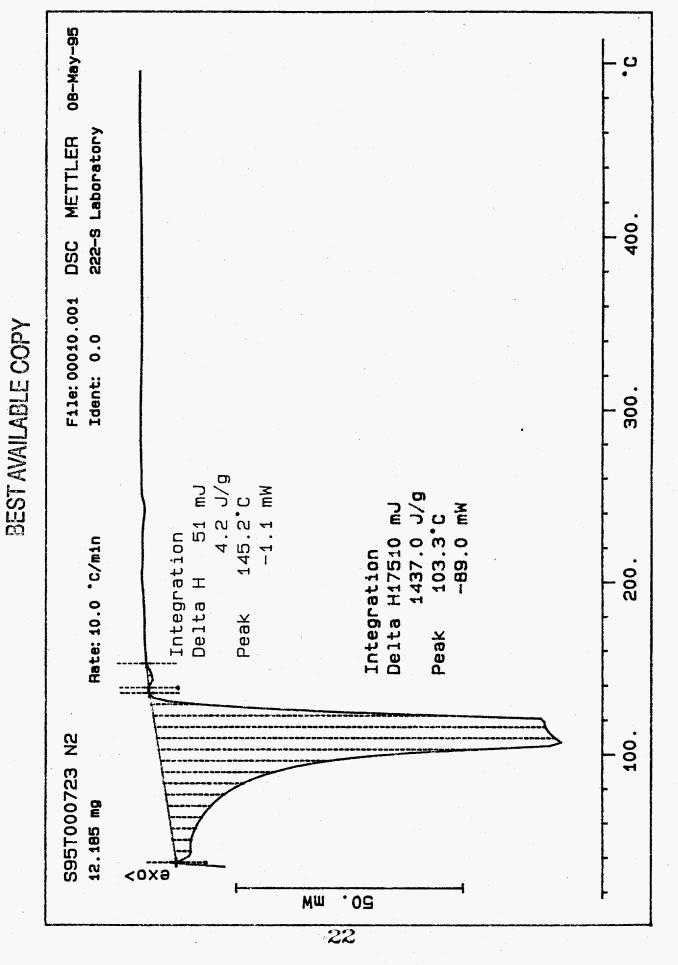
LABCORE Data Entry Template for Worklist# 1229

Analyst:	SHF	Instrumen	t: DSC01	· .	Book	# 12N	14-A	
Method: LA-5	14-113 Rev/Mo	d <u>B-1</u>						
Worklist Com	ment: Please ru	ın U-204 DSC un	der N2. bdv					
ROUP PROJECT	S TYPE	SAMPLE# R A	TEST	MATRIX	ACTUAL	FOUND	DL	UNIT
	1 STD		DSC-01	LIQUID	28.45	28.1	N/A	_ Joules/g
5000058 U-204	2 SAMPLE	S95T000723 0	DSC-01	LIQUID	N/A	_Ø		_ Joules/g
5000058 U-204	3 DUP	S95T000723 0	DSC-01	LIQUID	<u>Ø</u>	<u>Ø</u>	N/A	_ Joules/g
5000058 U-204	4 SAMPLE	S95T000724 0	DSC-01	LIQUID	N/A	Ø	-	_ Joules/g
95000058 U-204	5 DUP	s95T000724 0	DSC-01	LIQUID	_Ø_	<u>Ø</u>	N/A	Joules/g
Verified by	y Blanda	25-8-95 na Valenzuel	(d 3/7/78					

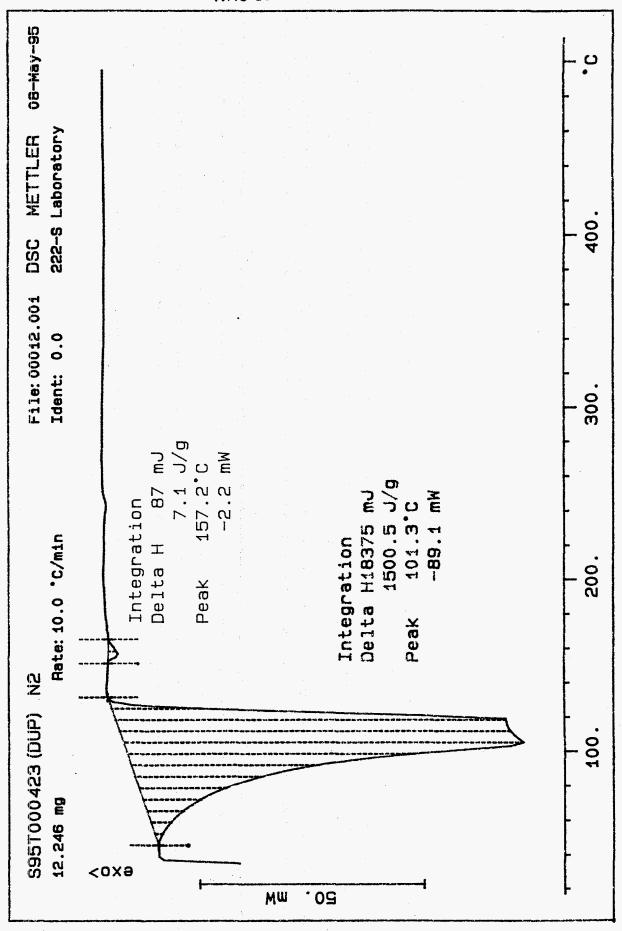












Page:

LABCORE Data Entry Template for Worklist# 1230

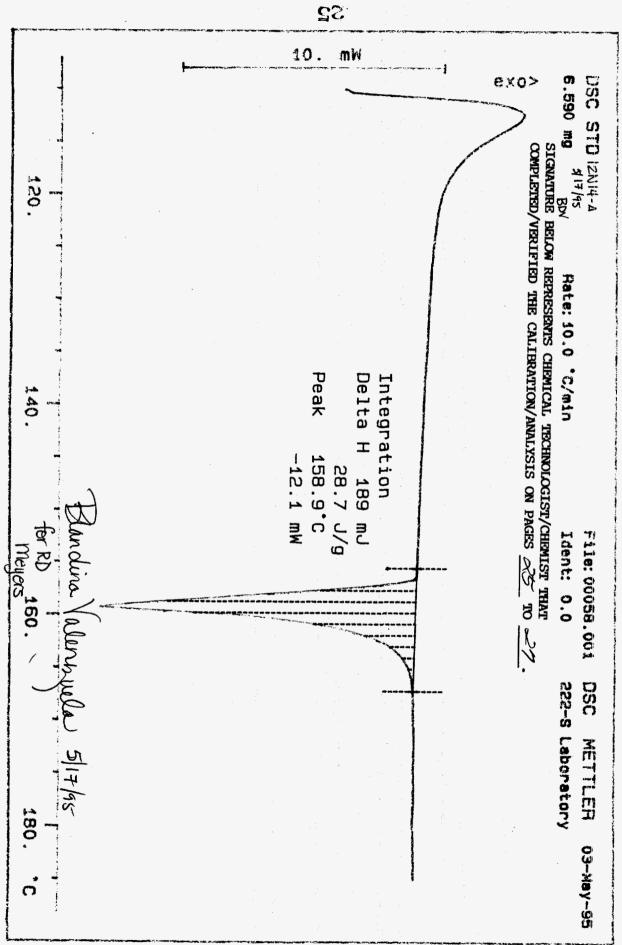
Rom Book # 12N/47 **Analyst: Instrument:** DSC01 Method: LA-514-113 Rev/Mod B-Worklist Comment: Please run U-204 DSC under N2. bdv R A -----TEST-----MATRIX ACTUAL **GROUP PROJECT** S TYPE SAMPLE# **FOUND** DL UNIT LIQUID 28.45 28.7 1 STD DSC-01 95000058 U-204 2 SAMPLE S95T000732 DSC-01 95000058 U-204 3 DUP S95T000732 0 DSC-01 Final page for worklist # 1230

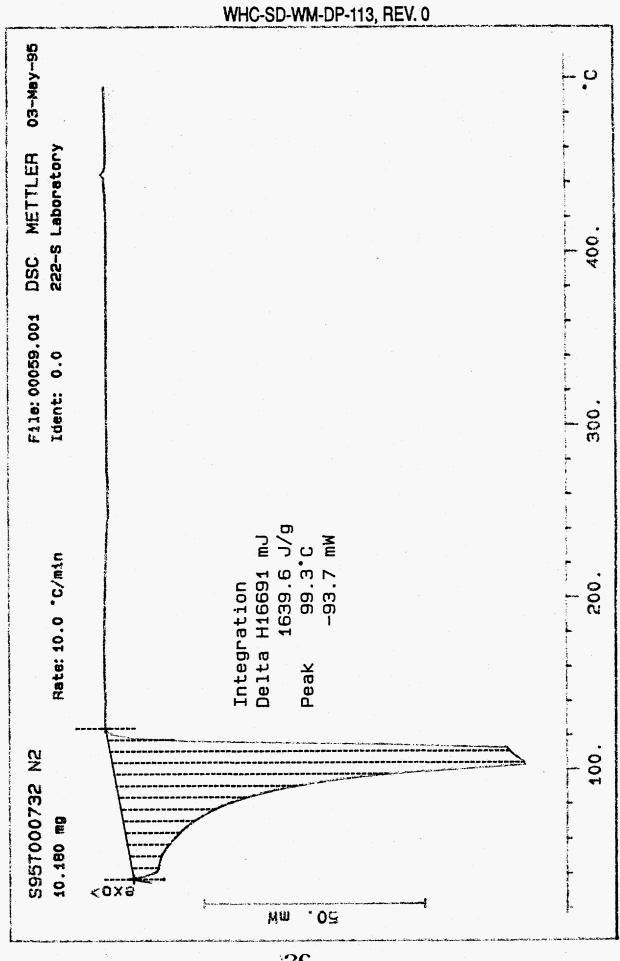
Verified by Blandina Valenzuela 5/4/95

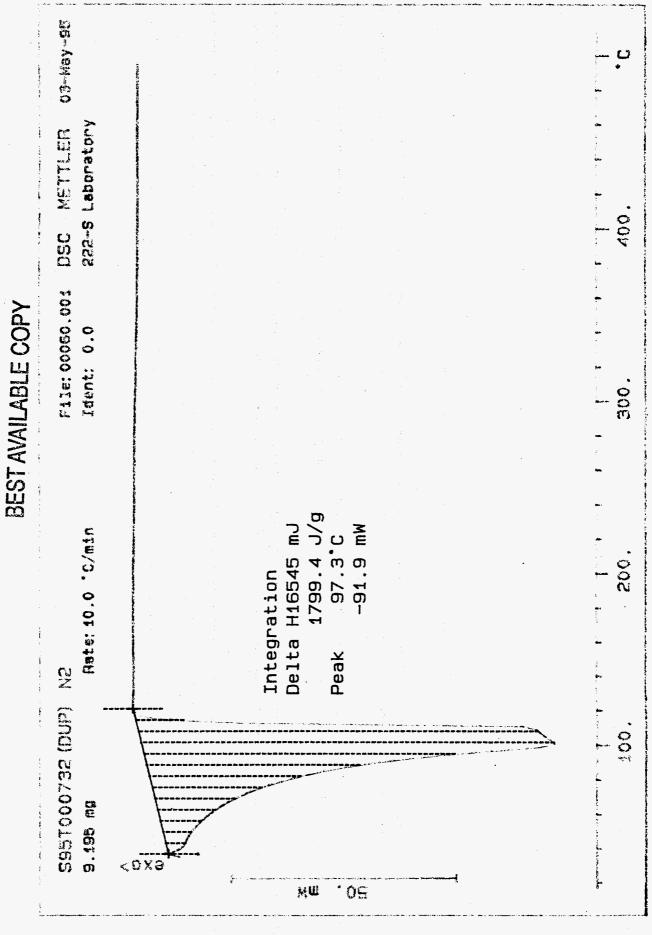
produced one endotherm at 99.3°C with a

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.









Page:

04/27/95 07:18

LABCORE Data Entry Template for Worklist# 1236

Book # 42 N8-A **Instrument:** TGA01 Analyst:

Method: LA-560-112 Rev/Mod

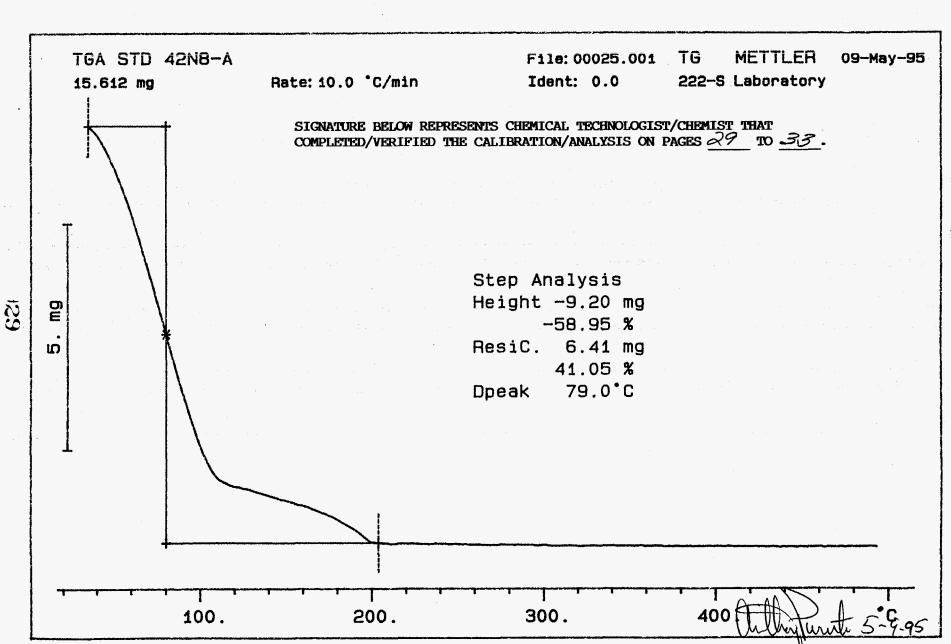
Worklist Comment: Please run U-204 TGA under N2. bdv

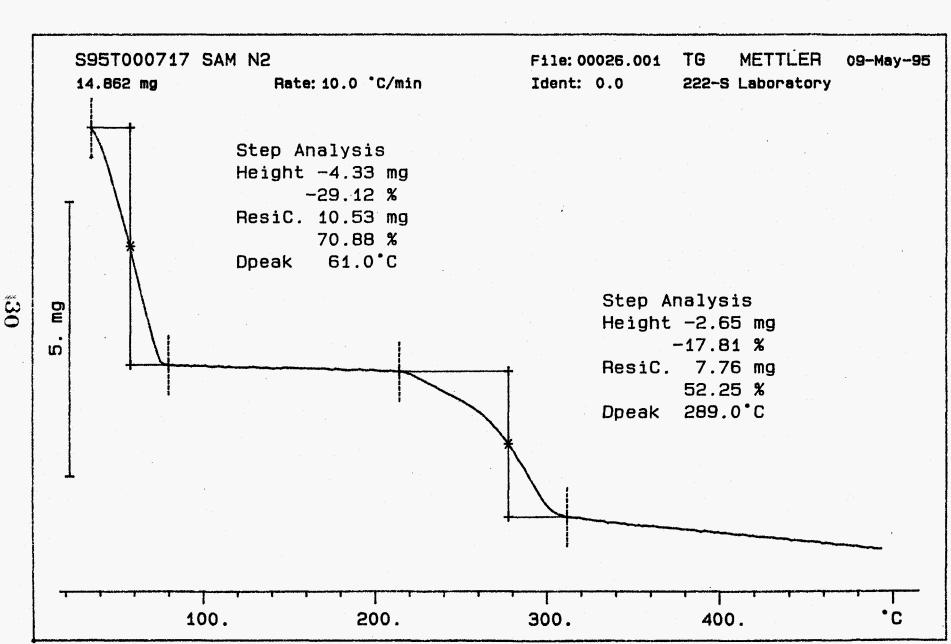
GROUP	PROJECT	S TYPE	SAMPLE#	R ATEST	MATRIX	ACTUAL	FOUND DL	UNIT
		1 STD		TGA-01	SOLID	59.19	58.95 N/A	%
95000058	U-204	2 SAMPLE	s95T000717	0 TGA-01	SOLID	N/A	29.12	%
95000058	U-204	3 DUP	s95T000717	0 TGA-01	SOLID	29.12	29.56 N/A	_ %
95000058	U-204	4 SAMPLE	s95T000718	0 TGA-01	SOLID	N/A	24.18	%
95000058	U-204	5 DUP	s95T000718	0 TGA-01	SOLID	24.18	21.84 N/A	%

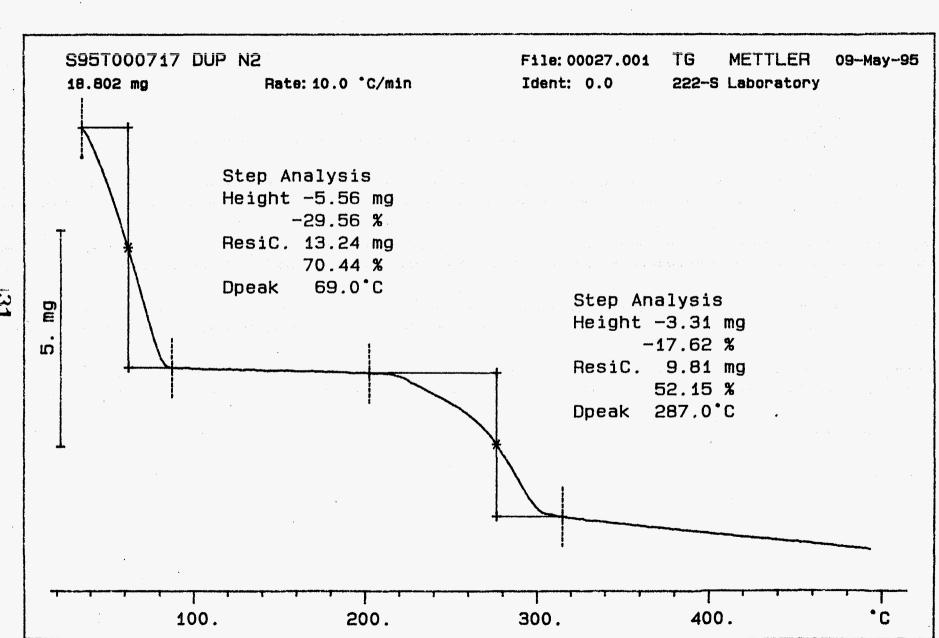
Final page for worklist # 1236

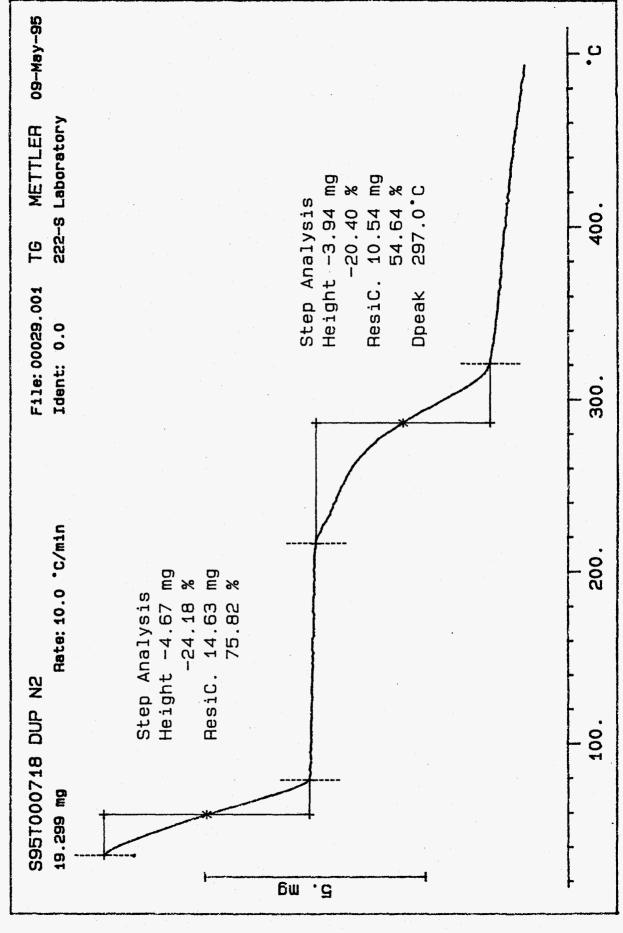
Verified by Blandina Valenzuela 5/9/95

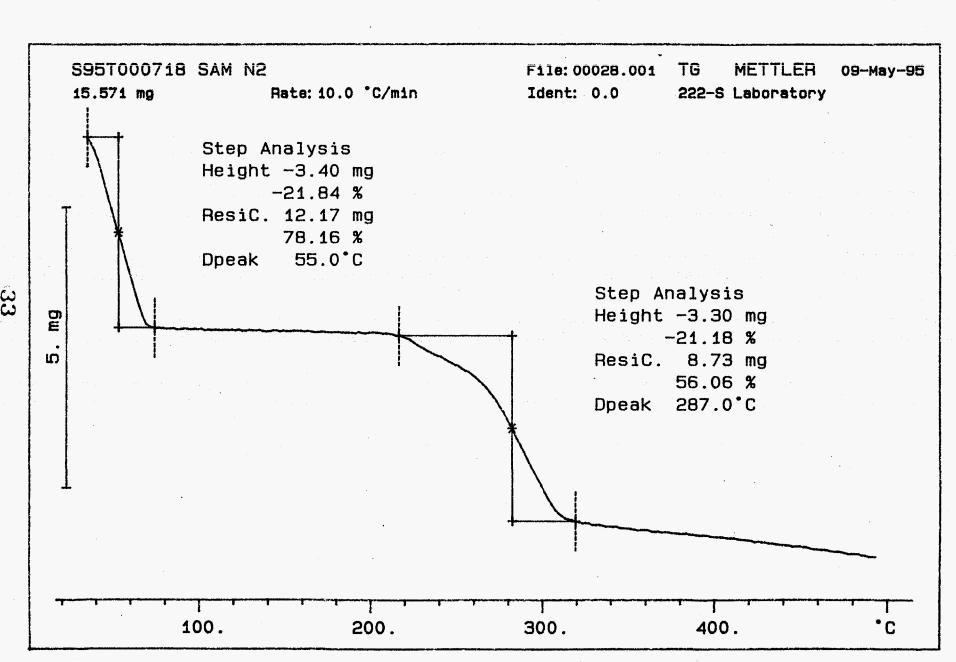
S95T000717 produced a second weight loss step of 17.81% at 289.00 Data Entry Comments: S95T000718 produced a second weight LOOS step of 20.40% at 297.0°C











95000058 U-204

95000058 U-204

95000058 U-204

3 DUP

5 DUP

4 SAMPLE

S95T000723 0

S95T000724 0

S95T000724 0

LABCORE Data Entry Template for Worklist# 1248

Book # 42N8-17 **Instrument:** TGA01 Analyst: Method: LA-560-112 Rev/Mod A-2Worklist Comment: Please run U-204 TGA under N2. bdv **GROUP PROJECT** S TYPE SAMPLE# R A -----TEST-----MATRIX ACTUAL FOUND DL UNIT LIQUID 59.19 59.42 1 STD TGA-01 86.46 95000058 U-204 2 SAMPLE S95T000723 0 TGA-01

Final page for worklist # 1248

TGA-01

TGA-01

TGA-01

LIQUID 86.46 86.89

LIQUID 87.18 86.18

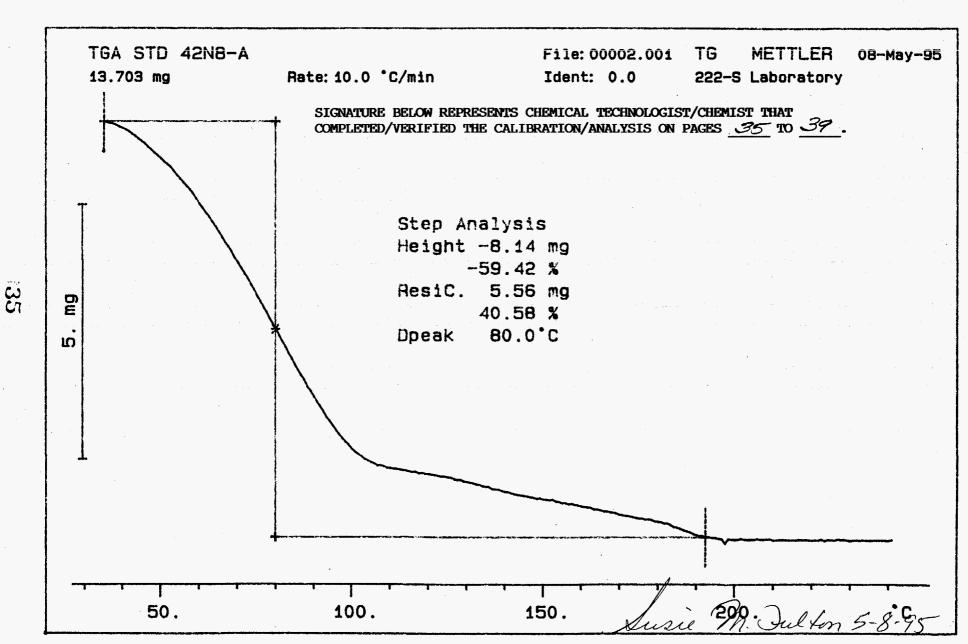
N/A

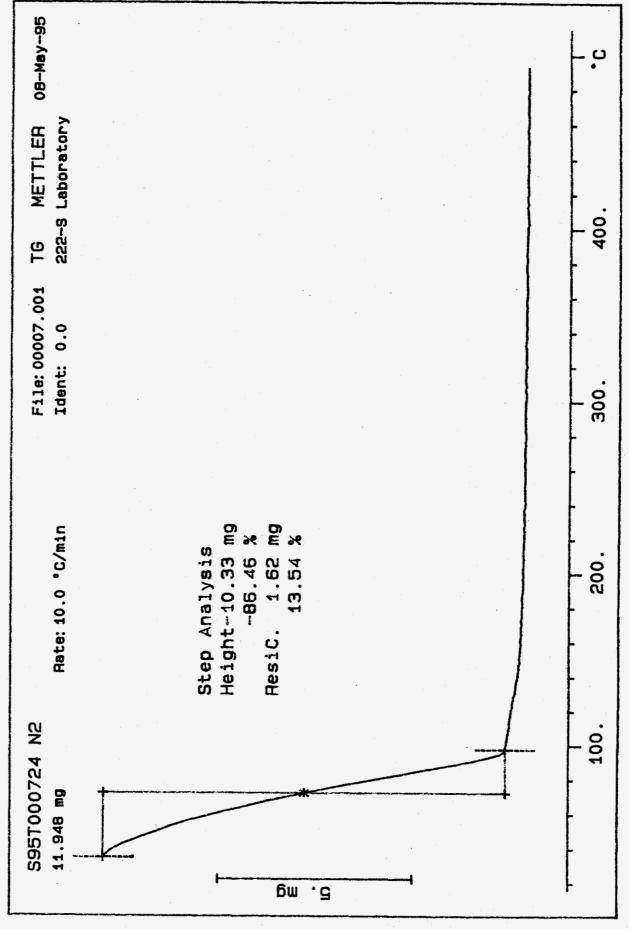
87.18

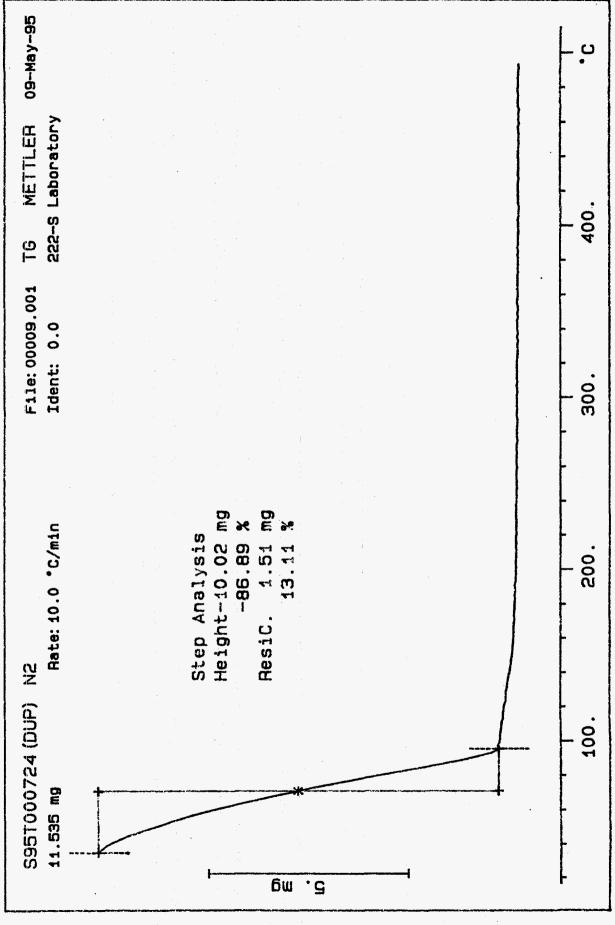
Analyst Signature Date
Verified by Blandina Valentuela 5/9/95

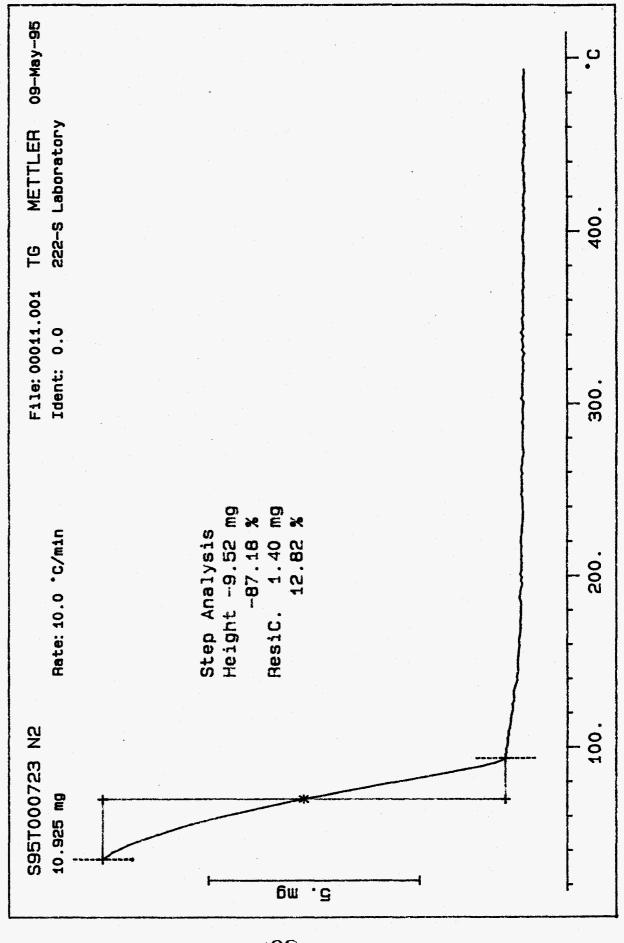
Data Entry Comments:

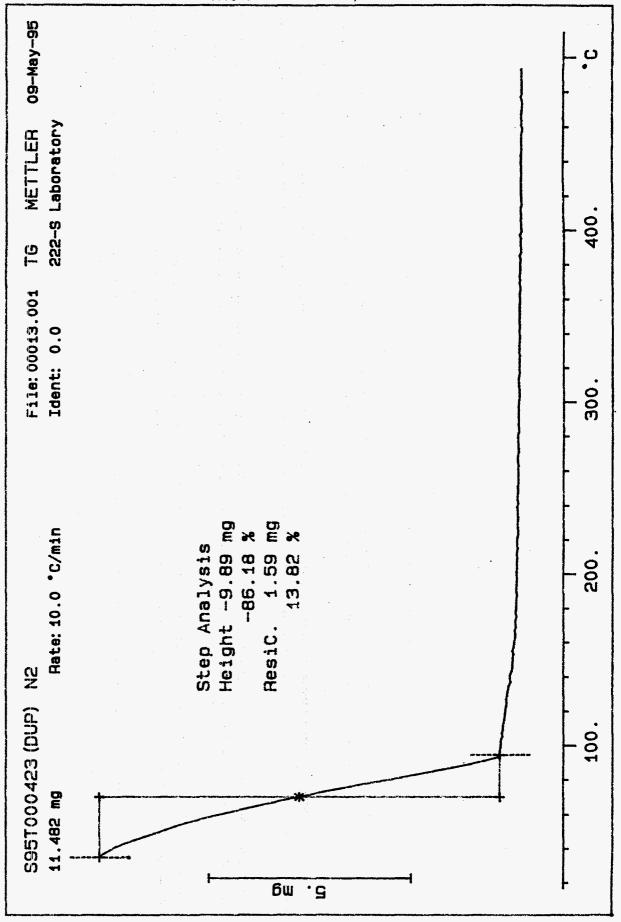
sampled are a bright yellow liquic











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LABCORE Data Entry Template for Worklist# 1249

Analys	t:	469	Instr	ume	nt: TGA01		Book	# 421	N8-A	
Method	d: LA-560	-112 Rev/Mo	od A-2			÷				
Workli	st Comme	ent: Please r	ın U-204 TO	GA ι	under N2. bdv					
GROUP	PROJECT	S TYPE	SAMPLE#	R A	TEST	MATRIX	ACTUAL 59.19	FOUND	DL	UNIT
		1 STD			TGA-01	LIQUID	58. 5/4/4	58.97	N/A	_ %
95000058	U-204	2 SAMPLE	s95T000732	0	TGA-01	LIQUID	N/A	99.01		_ %
95000058	U-204	3 DUP	S95T000732	0	TGA-01	LIQUID	99.01	100.56	N/A	_ %
,			Fin	al ı	page for wo	rklist	t # 124	49		
	Andre		9-95	•		E	Jon	9	5-9	-95
Analyst	Signatur	e Date	e \/ ^	-	0 -/0/-	Analy	st Signat	ture	Date	
lerifie	a by t	Blandu	ia Vale	nhi	ula 5/9/95					. •

