
Oxidative Reduction of Glove Box Wipers with a Downdraft Thermal Oxidation System

**M. R. Phelps
W. A. Wilcox**

April 1996

**Prepared for
Westinghouse Hanford Company
and the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830**

**Pacific Northwest National Laboratory
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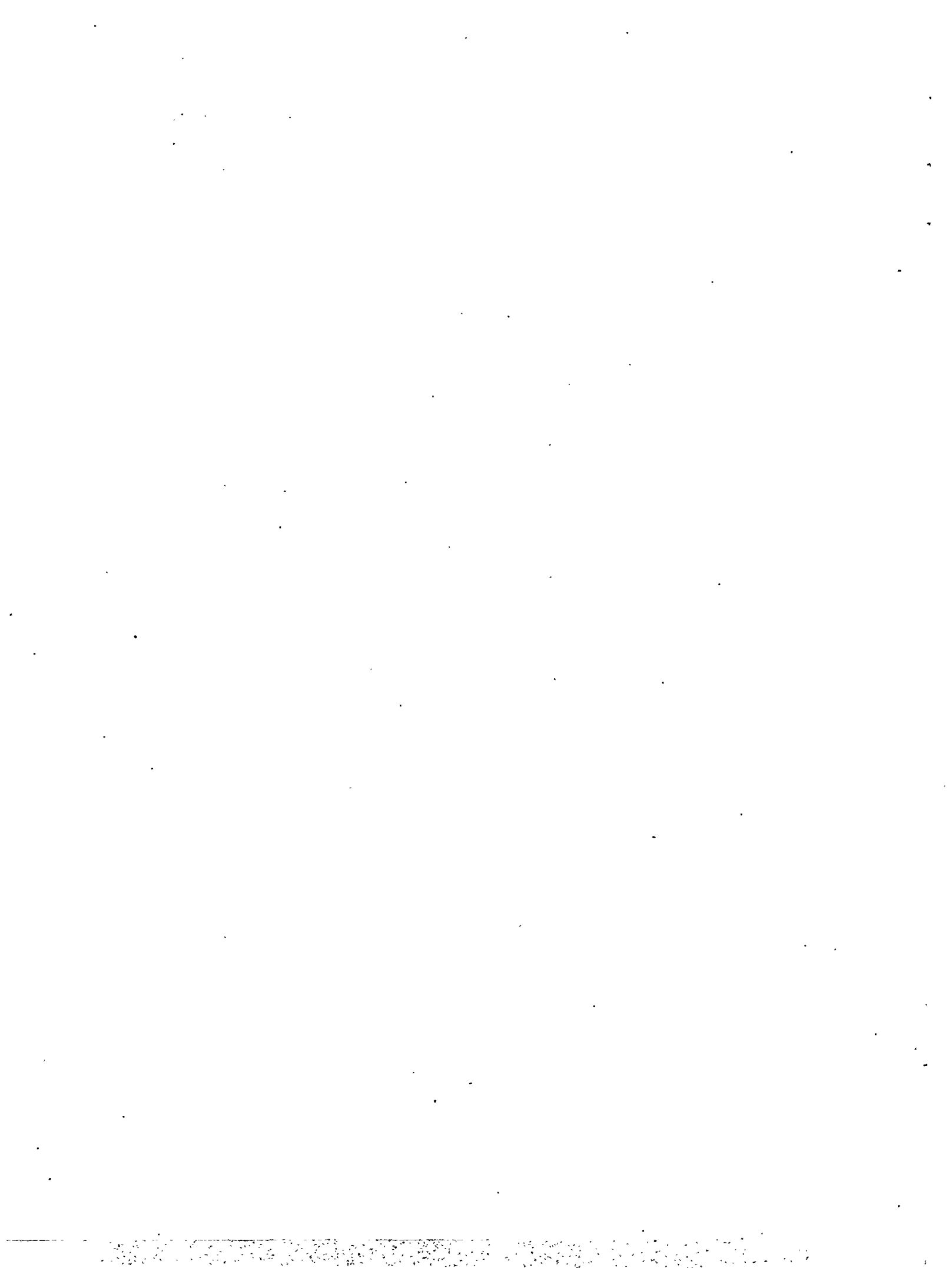
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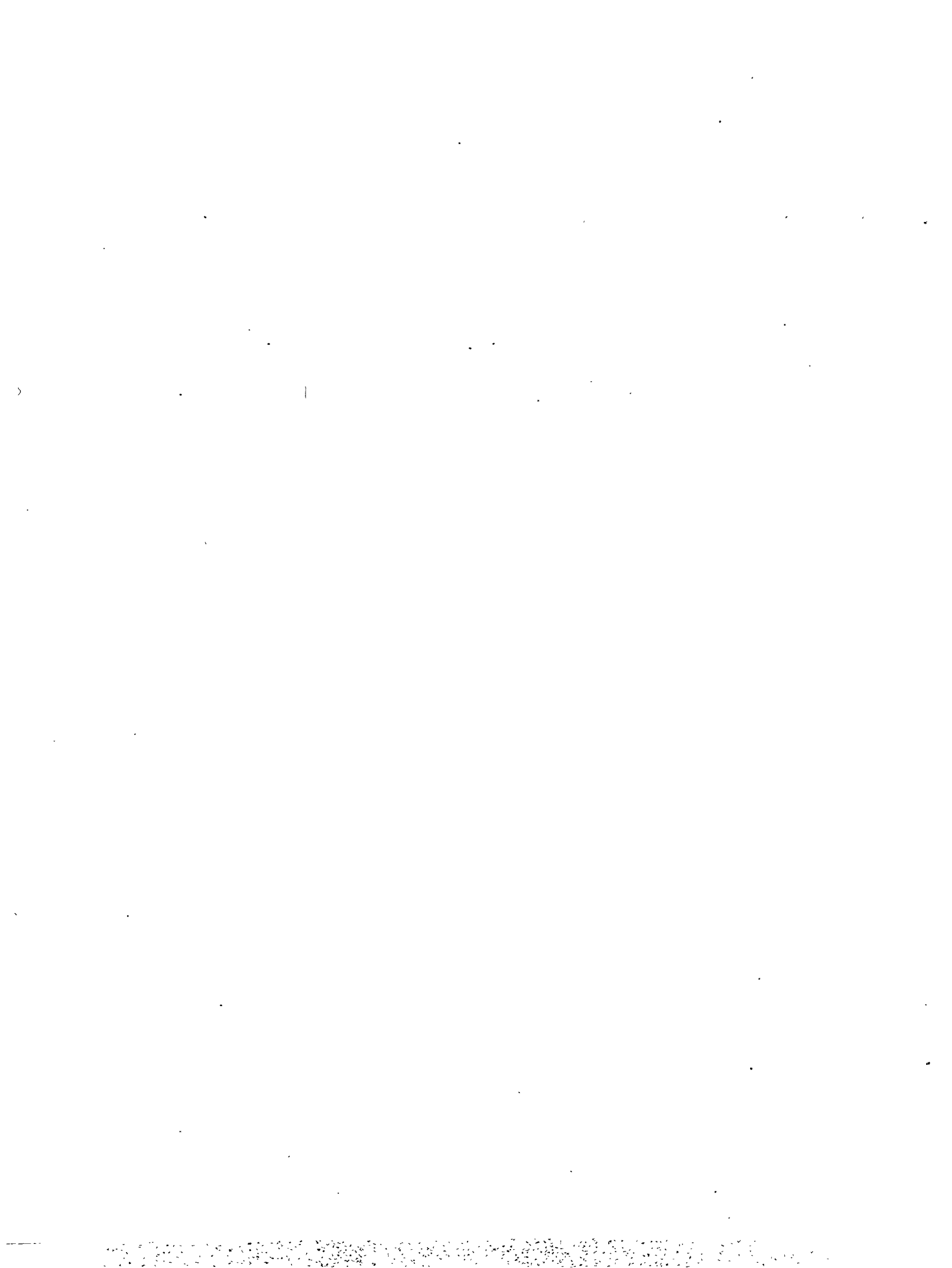
Pacific Northwest National Laboratory
Richland, Washington 99352



Summary

Wipers (rags) used for decontamination and glove box cleanup in the Plutonium Finishing Plant often become soaked with acid and plutonium-rich solutions. After use, these wipers are rinsed in a dilute NaOH solution and dried, but the formation of unstable nitrates and the hydrogen gas caused by hydrolysis are concerns that still must be addressed. This report gives the results of testing with a small downdraft thermal oxidation system that was constructed by Pacific Northwest National Laboratory to stabilize glove wiper waste, reduce the waste volume, and reclaim plutonium.

Proof-of-principle testing was conducted with eight runs using various combinations of rag moisture and chemical pretreatment. All runs went to planned completion. Results of these tests indicate that the thermal oxidation system has the potential for providing significant reductions in waste volume. Weight reductions of 150:1 were easily obtainable during this project. Modifications could result in weight reductions of over 200:1, with possible volume reductions of 500:1.



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1.0 Introduction

Wipers (rags) are often used for decontamination and glove box cleanup in the Plutonium Finishing Plant (PFP) at the U.S. Department of Energy's Hanford Site. During these operations, the wipers frequently become soaked with acid and plutonium-rich solutions. Although the wipers are rinsed in a dilute NaOH solution and dried after use, they are still considered a hazard. Principal concerns are the formation of unstable nitrates and the hydrogen gas caused by hydrolysis.

Thermal oxidation is one method considered for addressing these concerns. With an efficiently operating oxidation unit, the major benefits that could be realized include:

1. stabilization of glove box wiper waste to a safe form
2. large reduction in the waste volume
3. a method of collecting plutonium in a concentrated form, reducing equipment size and chemical volume required for recovery.

In 1979, a small thermal oxidation system was designed, fabricated, and tested for the Z-plant by Rockwell Hanford Operations with uncontaminated rags in the PFP pilot plant laboratory. Eleven runs were made using various combinations of rag moisture, air flow, and chemical pretreatment. Although only six of the runs went to planned completion, test results indicated that the potential for waste reduction is significant, with waste volume reductions of 100:1 easily obtainable. Changing priorities delayed issuing the test results until 1987. In the interim, the original system was disassembled, resulting in loss of some of the components.

In October 1991, Pacific Northwest National Laboratory^(a) constructed a small downdraft thermal oxidation system for the PFP (now operated by Westinghouse Hanford Company). Proof-of-principle (Phase I) testing of the oxidation chamber was completed in April 1992. Eight runs were made using various combinations of rag moisture and chemical pretreatment. All runs went to planned completion, with weight reductions of 150:1 easily obtainable. The experimental results and procedures are discussed in this report, along with recommendations for further development.

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2.0 Equipment

The thermal oxidation system consists of an oxidation chamber assembly, a filter assembly, scrubbing assembly, and two catch pots (see Figure 2.1). The oxidation chamber assembly consists of the chamber, grate, catch pot, and two ceramic fiber heaters. Instrumentation for the oxidation chamber assembly includes a flow meter to measure air flow into the chamber, a pressure gauge to measure vacuum pressure in the chamber, and thermocouples to measure temperatures within the chamber and the skin temperature of the chamber's body. A catch pot is located just below the main oxidation chamber body for collecting noncombustibles. The filter assembly houses two 5-micron, 304 stainless steel (SS) tubular filters, 10 in. long, and a catch pot for collecting particulates. Instrumentation for the filter assembly includes a differential pressure gauge to measure pressure drop across the filters and a thermocouple to monitor air temperature inside the filters. The scrubbing assembly includes a condenser and a back siphon trap. A differential pressure gauge measures the pressure drop across the scrubbing assembly.

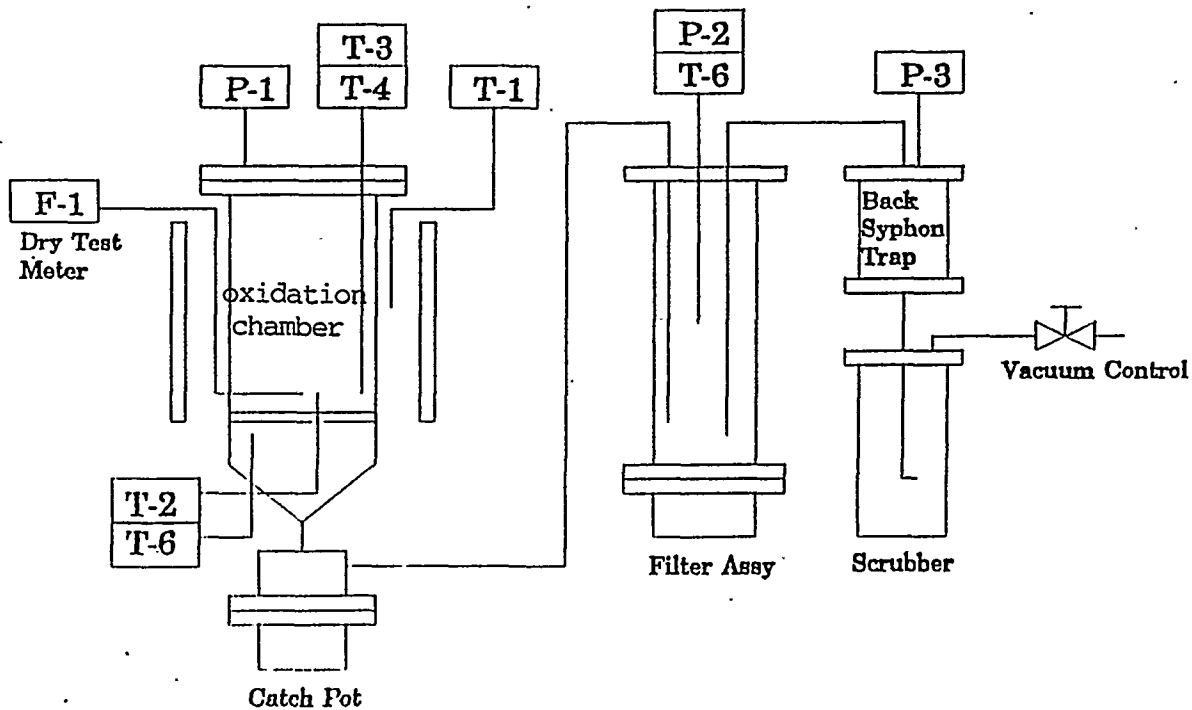
2.1 Oxidation Chamber

The main oxidation chamber (see Figure 2.2) is constructed of Schedule 40, 304 or 316 SS, 6-in. piping. Heat is supplied by two ceramic clamshell heaters strapped to the outer skin of the oxidation chamber body. Air is supplied through two 3/8-in. SS tubes, which are wrapped around the chamber body in the space between the chamber's skin and the clamshell heaters. The air supply tubes enter the oxidation chamber tangentially, 180 degrees apart from each other, at the top of the internal grate. The grate is constructed of 1/8 in. x 1 in. SS flat iron strips, which are separated by 5/16-in. SS washers. The distance between each strip is 3/4 in. A catch pot, connected to the chamber body with a 6 in. x 1 in. conical reducer, is located below the grate to catch ash and other noncombustibles.

A dry test meter measures air flow into the oxidation chamber, and an absolute pressure gauge measures inside pressure. Air is drawn through the system with a Model 1397 Welch vacuum pump connected to the scrubbing assembly. Thermocouples in the chamber measure the temperatures above and below the grate as well as the temperature inside the rag container (a round carton similar to 1/2-gal round ice cream cartons). Another thermocouple located in the space between a ceramic fiber heater and the chamber's skin indicates the temperature of the exterior wall.

The oxidation chamber is protected from overpressure with a spring-loaded, 6-in. SS blank flange attached at the top of the oxidation chamber body. The blank flange is removed prior to each run to load the rag container into the chamber and onto the grate. The blank flange is then reinstalled by slightly compressing each of four springs around the four bolts attaching the blank flange to the oxidation chamber body. A premeasured gauge is used to determine compressed spring length such that the blank flange should expand and relieve internal pressure at 35 psig.

Power to the ceramic heaters is controlled through a single variac. To limit the number of variables for each run, the variac was always set to supply a current of 19 amps to each heater.



- F-1 Gas Flow In
- P-1 Pressure: Combustion Chamber
- P-2 Pressure Differential: Filter Assembly
- P-3 Pressure Differential: Scrubber Assembly
- T-1 Temperature: Oxidation Chamber Skin
- T-2 Temperature: Center of Rag Carton
- T-3 Temperature: Above Grate
- T-4 Temperature: Above Grate
- T-5 Temperature: Below Grate
- T-6 Temperature: Inside Filters

Figure 2.1. Instrument Points and Flow Diagram

2.2 Filter Assembly

The filter assembly is constructed of 4-in. Schedule 40 glass piping (see Figure 2.2). The filter assembly is connected to the oxidation chamber assembly's catch pot with 1-in. Schedule 40 black piping. A catch pot, constructed of 4-in. Schedule 40 black piping, for collecting particulates is connected to the bottom of the main filter body with a 4-in. glass-to-steel pipe flange. Two 1/2 in. x 10 in. SS,

2.3

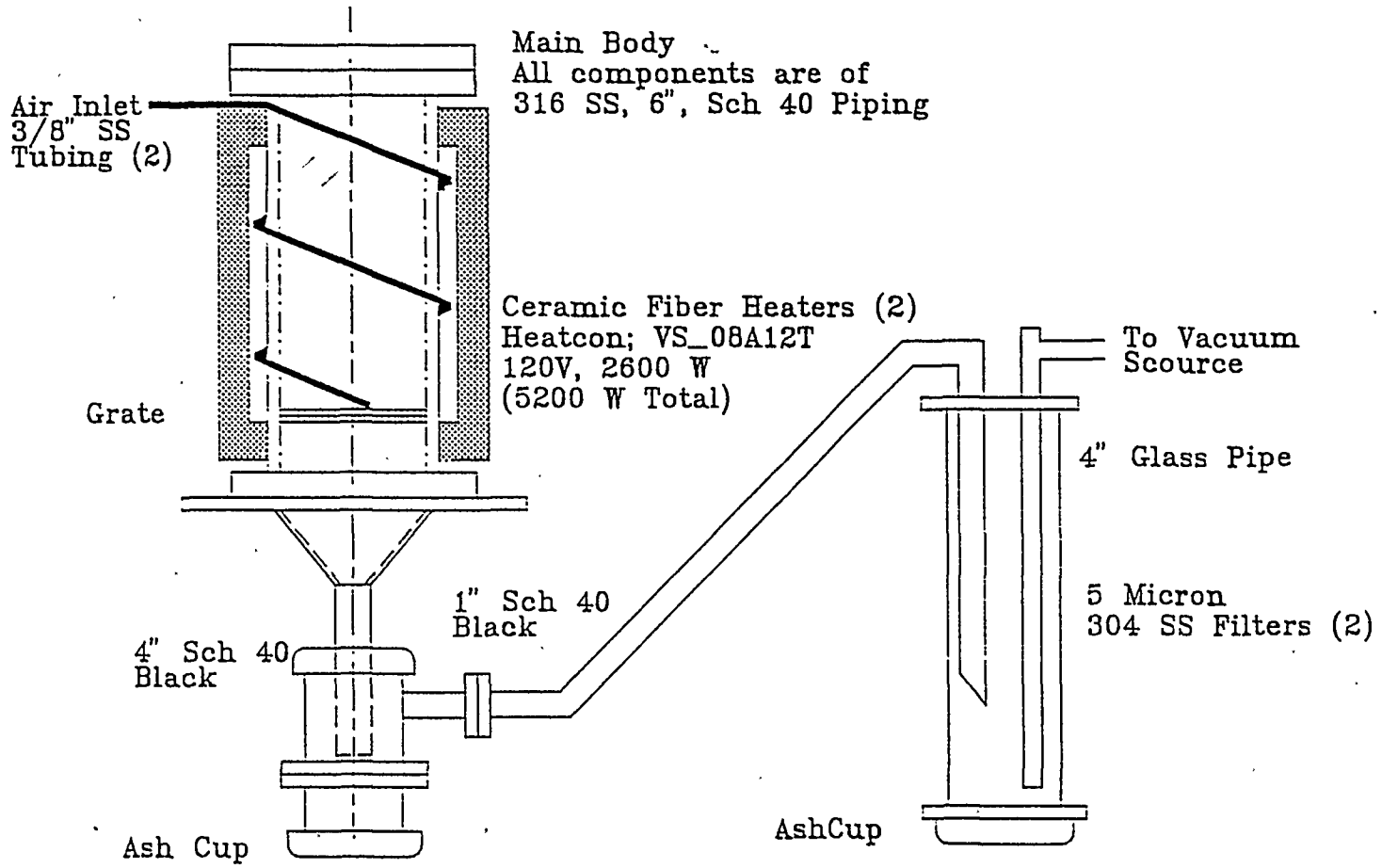


Figure 2.2. Downdraft Thermal Oxidation

5-micron tubular filters are housed inside the main filter body. For experimental runs, each filter was wrapped with a thin layer of kaowool to prevent plugging of the filters by condensing tars and particulates.

A differential pressure gauge measures the pressure drop across the filters to monitor filter loading. A thermocouple located inside one of the two filters monitors air temperature leaving the filter assembly. A septa port for obtaining gas samples is located above the other filter in the outlet gas stream.

2.3 Scrubbing Assembly

The scrubbing assembly consists of a scrubber housed in a single assembly. Cooling water feeds through the jacket of the scrubbing assembly to condense hot gases entering the assembly. Prior to each run, the scrubber was loaded with a measured amount of water. After each run, the water was removed and weighed.

A differential pressure gauge measures pressure drop across the scrubbing assembly. A thermocouple in the line exiting the scrubber monitors gas temperature entering the vacuum pump.

2.4 Vacuum Pump

A Model 1397 Welch mechanical vacuum pump supplies vacuum to the system. The pump is capable of a free air displacement of 500 L/min. Without any water in the scrubber assembly, the pump easily maintained a draw of 200L/min at the air inlet to the thermal oxidation chamber at 25 in. H₂O vacuum.

3.0 Experimental Procedures and Results

This section describes the eight experimental runs and the results obtained from the tests.

3.1 Leak Rate Test

The system was placed under 30 in. H₂O of vacuum, and the leak rate was observed. A vacuum drop to 10 in. was observed in 5.8 sec. Leak rate was determined to be 3.5 in. H₂O/sec (~28 L/m) at the beginning of the test series. Another leak rate check was made after Run #5 (see Section 3.3.5).

3.2 CHN Analysis of Glove Box Wipers

Two tests were made to determine the carbon, hydrogen, and nitrogen content of the rags used for this study (14 in. x 14 in. 100% cotton towels). Prior to testing, the rags were dried to remove all water content. The results are summarized in Table 3.1.

Table 3.1. CHN Analysis

<u>Sample Name</u>	<u>C Analysis % total wt</u>	<u>H Analysis % total wt</u>	<u>N Analysis % total wt</u>	<u>Average Total %</u>
100% cotton rags	43.28	6.35	0	
Repeat	42.42	6.28	0	42.85
Round Carton	46.17	7.21	0	
Repeat	46.34	7.18	0	46.26

3.3 Experimental Runs

Each run was started by turning the variac on to 80%, supplying 19 amps to each heater. After 8 min of heating, air flow through the system was started by pulling approximately 15 L/min of air through the system with the vacuum pump. When the temperature inside the carton of wipers started to climb, air flow through the system was turned to the maximum the vacuum pump could achieve. When a rapid temperature increase was observed in the carton of wipers, the variac was turned off and the experiment allowed to run to completion. After 107 min from startup, the vacuum pump was turned off and the experiment concluded. The oxidation chamber was always allowed to cool to at least 80°C before the interior was opened and examined.

Experimental results for the testing include a gravimetric analysis of the ash from each run. An offgas analysis from some of the runs was also made with a gas chromatograph. Ash was collected with a small hand-held vacuum, at the conclusion of each run, from the interior of the oxidation chamber, the grate, and the oxidation chamber's catch pot. Gas samples were taken periodically from the offgas line from the filter assembly. A carbon balance by gas analysis was attempted for some of the runs. The results are inconclusive, however, because none of the ash from the catch pots or the water from the scrubber was analyzed for carbon content. Also, gas analysis results in some cases tended to indicate that more carbon came out of the system than went in. This could be attributed to either a larger difference in carbon content between rags than results from a CHN analysis indicated, or an inaccurate calibration standard for the gas chromatograph. Further testing is needed to ascertain the source of the discrepancies.

3.3.1 Run #1: Dry Rags, Loosely Packed

A round carton and three rags were oven dried at 90°C for approximately 3 hr.

Carton wt	44.49 g
Rag wt	<u>170.03 g</u>
Total wt	214.52 g

The rags were loosely packed in a round carton and loaded into the oxidation chamber. A 1/4 in. x 1/4 in. SS mesh was placed between the bottom of the carton and the grate to facilitate ash recovery. The variac was turned on to 80%, supplying 19 amps to each heater. The vacuum pump was not initially turned on. A worksheet summary of the experiment is given in Table 3.2. Note: for Table 3.2 and the worksheet summaries for the other runs, temperatures are in degrees C; pressures are in inches H₂O. A graph of the temperature profile for Run #1 is included in Section 3.4. [Section 3.4 also includes graphs for Runs #2, 3, 5-8.]

After the experiment, the 6-in. blank flange at the top of the oxidation chamber assembly was removed and a small hand-held vacuum was used to collect the ash. When vacuuming was completed, the ash cup was removed to collect the ash and other noncombustibles. There was a white ash on a "glob" of dry black tar in the bottom of the ash cup.

Ash from grate	0.62 g
Ash from cup	0.05 g
Tar	<u>0.82 g</u>
Total	1.49 g

Table 3.2. Experimental Run #1

<u>Time</u>	<u>T1</u>	<u>T2</u>	<u>T3</u>	<u>T5</u>	<u>P1</u>	<u>AP2</u>	<u>Comments</u>
7:47	16.4	16.5	16.3	15.8	0	10	Variac on @ 80% (19 amps to each heater)
7:54	407	17.1	70	63	0	10	Air on @ 15 L/min
7:58	512	33	167	119	30	10	Air on @ 15 L/min
8:02	582	96	330	182	37	10	Air on @ 124 L/min
8:04							Smoke observed in filter assembly
8:07	662	223	689	365	37	10	
8:08	679	303	732	396	37	10	
8:10	730	430	878	506	37	10	
8:12	772	887	730	514	37	10	
8:13	781	979	744	533	37	10	
8:15	749	928	734	563	40	10	Variac turned off
8:20	596	664	600	533	36	10	Total air = 1900 L; rate = 132 L/min
8:27	447	339	439	442	32	10	Total air = 2750 L
8:33	359	320	350	370	28	10	Total air = 3610 L
8:43	256	233	250	278	25	10	Total air = 4970
8:58	167	158	163	188	22	9.5	Total air = 7000
9:20	101	104	101	116	21	9	Total air = 10,050
9:55	62	67	62	68	20	8.5	Total air = 14,900; vacuum pump off

Thermocouple placement for Run #1:

T1	Skin Temperature
T2	Inside Container
T3	Grate
T5	Below Grate

3.3.2 Run #2: Dry Rags, Tightly Packed

A round carton and six rags were dried at 90°C for 2 hr. The rags were rolled together to make a tight fit in the carton. This run was essentially a repeat of the first run except for the tight packing and number of rags. This experiment was done to determine if a significant difference between test results would occur as a result of using a different packing procedure. The stainless steel wire mesh was not placed in between the grate and carton. A worksheet summary of the experiment is provided in Table 3.3.

Table 3.3. Experimental Run #2

Time	T1	T2	T3	T5	P1	$\Delta P2$	Comments
13:15	20.6	21.2	20.4	20.6	0	0	Variac @ 80%
13:22	413	21.1	157	42	0	0	Air on @ 20 L/min
13:27	542	27.7	340	121	0	0	
13:29	578	74.5	391	169	34	11	Air on @ 132 L/min
13:35			577	413	35	12	Air total = 900 L; rate = 129 L/min
13:39	731	273	647	528	37	12	
13:48	822	601	783	670	43	12	Air @ 130 L/min
13:51							Variac off
13:56	655	776	678	634	42	13	T6 (air to vacuum pump) at 92.8°C
14:02	535	569	560	542	37	13	Total air = 4380 L
14:10	417	398	448	449	34	13	Air @ 142 L/min
14:19	302	289	315	336	30	13	Air @ 144 L/min
14:23					29	13	Total air = 7400 L
14:30	218	213	224	246	27	13	Air @ 145 L/min
14:42	157	162	163	181	25	12	
14:55	118	127	121	135	25	12	
14:57							Vacuum pump off; total air = 12,490 L

Carton wt 44.9 g
 Rag wt 325.2 g
 Total wt 370.1 g

280.0 g H₂O added to scrubber assembly.

The same procedure as used in Run #1 was used to collect and weigh the ash.

Ash from grate 0.53 g
 Ash from Cup 1.02 g
 Total 1.57 g

329.1 g H₂O collected from scrubber assembly.

3.3.3 Run #3: Wet Rags

A round container and five rags were dried overnight at a temperature of 90°C. The rags were then soaked in water and wrung out to simulate typical wet rags from storage drums. In order to minimize experimental variables, every attempt was made to duplicate Run #2 with regard to air flows and variac timing. The filter assembly was modified to include a septa for taking gas samples of the product gas. A worksheet summary of the experiment is included as Table 3.4. A worksheet summary of the gas analysis is included as Table 3.5. Gas analysis was by a Carle gas chromatograph.

Carton wt	46.9 g
Rag wt	279.4 g
H ₂ O wt	<u>69.9 g</u>
Total wt	403.1 g

280 g H₂O added to scrubber assembly.

306.5 g H₂O removed from scrubber assembly (26.5 g gain).

Table 3.4. Experimental Run #3

Time	T1	T2	T3	T5	P1	ΔP2	Comments
9:45	20.3	21.7	18.6	18.4	0	0	Variac on @ 80%
9:53	454	21.0	182	56.0	3	0	Air on @ 16 L/min
9:59	575	70	375	176	37	20	Air on @ 130 L/min
10:05	643	89	558	602	34	20	Air on full @ 122 L/min
10:10	724	89	652	663	35	20	Total air = 1570 L; air @ 118 L/min
10:17	823	150	826	690	40	16	Total air = 2280 L
10:20	846	448	806	702	40	16	Variac off; Total air = 2720 L
10:24	709	730	718	678	37	15	Total air = 3200 L; air @ 126 L/min
10:30	597	578	597	601	35	15	Total air = 4000 L; air @ 130 L/min
10:40	401	373	403	428	31	15	
10:45	336	316	339	368	28	15	Air @ 135 L/min
10:54	252	243	256	284	25	14	Total air = 7050 L; air @ 140 L/min
11:21	135	140	137	154	22	14	Total air = 10,650 L; air @ 142 L/min
11:25	120	128	124	138			Vacuum pump off; total air = 11,400 L

Table 3.5. Average Gas Analysis for Experimental Run #3

Air Through DTM	Representative Volume	CO₂	CO	C=C	CH₄	H₂
(L)	(L)	(L)	(L)	(L)	(L)	(L)
0-305	305	0.92				
305-1050	745	37.70	24.51	0.60	1.04	
1050-1925	875	40.62	31.68	0.96	2.63	2.19
1925-2700	775	57.74	4.11			
2700-3740	1040	47.53	2.18			
3740-4860	1120	8.06				
4860-9400	4540	7.3				
9400-11,400	2000	2.0				

3.3.4 Run #4: Simulated Loading with HNO₃, Na₂CO₃, and Fe(NO₃)₃

Fifty grams of 70% HNO₃ were added to 450 g tap water. Na₂CO₃ was added to the solution until fizzing stopped. This procedure was used to simulate neutralization procedures inside a glove box. Density of the resultant solution was 1.067 g/mL; 44.61 g of Fe(NO₃)₃·9H₂O were then added to the solution. The iron nitrate used came from a standard laboratory sample jar and was greenish in color. This could have been due to some cross contamination or possibly a mislabel. Although the discrepancy was noted prior to the experiment, a decision to use the sample was made and the experiment continued.

A round container and five rags were dried overnight at 90°C. The neutralized solution was dripped onto the rags and then left to sit in a covered beaker overnight. This procedure resulted in an uneven distribution of solution throughout the rags. A worksheet summary of the experiment is provided in Table 3.6.

Table 3.6. Experimental Run #4

Time	T1	T2	T3	T5	P1	ΔP2	Comments
7:45	16.2	15.8	16.1	16.4	0	0	Variac on 80%
7:53	437	15.7	134	55	1.5	0	Air on @ 8 L/min
7:58	563	19.8	304	155	2.0	0	
8:00	591	60	422	211	37	16	Air on full
8:08	739	92.6	765	545	33	30	Total air = 1140 L; air @ 110 L/min
8:13	812	92.3	870	656	35	30	Total air = 1650 L; air @ 110 L/min
8:16	843	97	845	692	39	24	Total air = 2050 L; air @ 115 L/min
8:19	865	127	856	717	40	20	Variac off; total air = 2370 L
8:22	757	415	791	705	39	20	Total air = 2730; air @ 123 L/min
8:26	663	678	691	651	37	19	
8:31	570	777	581	581	35	19	Total air = 3880 L; air @ 127 L/min
8:44	378	566	390	420	30	19	Total air = 5590 L; air @ 135 L/min
8:50	315	458	330	363	28	19	Total air = 6412 L; air @ 138 L/min
9:00	243	304	254	287	26	19	
9:10	189	227	202	229	25	18	Total air = 10,580 L; air @ 142 L/min
9:20	152	173	162	183	24	17	
9:24	140	161	154	170	24	17	Vacuum pump off; total air = 11,215 L

Prior to experiment, 280.5 g water added to the scrubber assembly.

Carton wt	44.85 g
Dry rag wt	296 g
Wet rag wt	<u>448.1 g</u>
Total wt	492.95 g

There was a large deposit of multicolored ash left sitting on the grate at the conclusion of this run. The volume of the ash was approximately 1/2 the volume of the original cylindrical carton. The ash was red, green, brown, white, and black in color. Most of the ash resembled the fibrous shape of the rag's cotton fibers. When touched, the ash crumbled to a fine powder. Because of the unusual results from this experiment and the discrepancy in the color of the iron nitrate, this experiment was repeated with some fresh iron nitrate. A worksheet for the gas analysis of Run #4 is shown in Table 3.7.

Table 3.7. Average Gas Analysis of Experimental Run #4

<u>Range</u>	<u>Representative Volume</u>	<u>CO₂</u>	<u>CO</u>	<u>C=C</u>	<u>CH₄</u>	<u>H₂</u>
(L)	(L)	(L)	(L)	(L)	(L)	(L)
0-226	226	6.71	2.58			
226-770	544	32.31	16.86	0.59	7.07	0.82
1475-2100	625	45.12				
2100-2775	675	40.16				
3465-4375	910	22.20				
4375-6345	1970	23.64	5.71			
6345-9035	2690	19.37				
9035-11,215	2180	5.45				
770-1475	705	54.50	3.95			
2775-3465	690	26.91				

3.3.5 Run #5: Rags Soaked in Fe(NO₃)₃ and Water

To simulate a different method by which rags might be treated in a glove box environment, dried rags were dipped into an acidic iron nitrate solution then quickly rinsed with water for neutralization. In preparation for the experiment, five rags and a cylindrical carton were dried overnight at 90°C; 293.0 g of a solution containing 89.22 g of Fe(NO₃)₃ in 70% HNO₃ were prepared. The dry rags were placed in the solution and allowed to sit for 5 min. The dry rags easily absorbed the iron nitrate, nitric acid solution. Each rag was then individually rinsed in the same beaker containing 1000 g of tap water. Each rinse lasted 30 sec with some agitation. The rinsed rag was wrung out over the beaker containing the rinse water. The rags were then stored overnight in a covered beaker. The rinse water was analyzed and found to contain 9.074 of Fe. The worksheet summary of the experiment is given in Table 3.8.

Table 3.8. Experimental Run #5

Time	T1	T2	T3	T5	P1	$\Delta P2$	Comments
7:45	20.7	17.7	18.9	19.0	0	0	Variac on @ 80%
7:48	293	18	30		2	1	Air on @ 15 L/min
7:52	437	18	98	58	3	1	
7:59	576	18	259	191	37		Air on full @ 170 L/min
8:01	589	34	286	223	33	16	Air @ 127 L/min
8:06	619	97	322	320	32	18	Total air = 1100 L
8:09	633	97	344	354	30	25	Total air = 1400 L; air @ 120 L/min; condensate coming into filter assembly
8:13	664	95	447	417	29	30	Total air = 1840 L; air @ 107 L/min
8:16	692	90	556	480	28	30	Total air = 2160 L; air @ 105 L/min
8:19	726	94	686	553	30	30	Total air = 2475 L; air @ 107 L/min
8:22	764	93	741	608	32	30	
8:26	812	91	778	663	35	30	Total air = 3235 L; air @ 110 L/min
8:31	857	90	821	716	38	28	Total air = 3780 L; air @ 112 L/min
8:35	880	89	831	741	40	25	Total air = 4235 L; air @ 116 L/min
8:38	889	90	821	716	38	28	Total air = 4580 L; air @ 116 L/min
8:41	896	221	834	761	40	23	Variac off; air @ 118 L/min
8:44	794	431	793	744	40	23	
8:48	691	572	709	683	38	23	Total air = 5770 L; air @ 124 L/min
8:52	612	786	630	621	35	23	Total air = 6270 L; air @ 124 L/min
8:56	541	759	554	563	33	24	Total air = 6770 L; air @ 127 L/min
9:04	428	493	469	467	31	24	Total air = 7900 L
9:10	369	357	375	410	29	24	Total air = 8650 L; air @ 131 L/min
9:22	257	252	264	297	25	20	Total air = 10,260 L; air @ 135 L/min
9:40	166	172	175	195	23	18	Air off; total air = 12,700 L

Carton wt	47.3 g
Rag wt	293.0 g
Liquid wt	<u>458.1 g</u>
Total wt	798.4 g

280 g water added to the scrubber assembly.

Ash from top of grate	2.29 g
Ash from ash cup	<u>1.47 g</u>
Total ash	3.67 g

486.0 g water removed from scrubber assembly (206.0 g gain).

The ash removed from the grate and the oxidation chamber ash cup was mostly reddish brown in color. White ash appeared as small chunks throughout the ash mixture. Approximately 0.4 g of fine red ash was also collected off of the chamber wall. The fine red ash that coated the wall also coated the inside of filter assembly and the kaowool covering the filters.

A worksheet outlining the gas analysis from Run #5 is included as Table 3.9.

Table 3.9. Average Gas Analysis of Experimental Run #5

<u>Range</u>	<u>Representative Volume</u>	<u>CO₂</u>	<u>CO</u>	<u>C=C</u>
(L)	(L)	(L)	(L)	(L)
0-590	590	1.30		
590-1195	605	2.06		
1195-1750	555	9.10	4.05	
1750-2260	510	18.82	8.72	0.20
2260-2780	520	33.12	10.40	
2780-3280	500	32.70	2.90	
3280-3875	595	39.03		
3875-4615	740	38.48		
4615-5610	995	33.23		
5610-6925	1315	29.85		
6925-8455	1570	25.55		
8455-10,305	1850	7.77		
10,305-12,700	2395	7.90		

Another check of the leak rate was made at the completion of Run #5 to compare with results from the leak rate test performed prior to Run #1. A blind flange was placed on the oxidation chamber side of the 1-in. line leading from the chamber assembly to the filter assembly. A vacuum pump and

rotameter were connected to one of the two 3/8-in. SS air inlet lines, and the other line was connected to a manometer. Vacuum was applied to the chamber assembly; a control valve to the vacuum pump was shut; and the leak rate determined by air flow through the rotameter. At room temperature and 29.5 in. H₂O of vacuum, the leak rate was 9.44 L/min. Further testing at different temperatures gave the following results:

Oxidation Chamber Outer Skin Temperature in deg C	Vacuum in inches H ₂ O	Leak rate in L/min
588	35	7.8
659	32.5	6.8
701	30	6.1
752	30	5.7
809	30	5.2
836	30	4.9

After cooling, the two heaters were removed, and the air inlet tubing examined. A small hole had developed in one of the lines at a point where the line was welded to the skin of the oxidation chamber. An attempt to repair the leak was unsuccessful. After the repair attempt, the leak rate was established once again at room temperature. At 29 in. H₂O of vacuum, the leak rate was 8.26 L/min.

3.3.6 Run #6: Dry Rags, Catalyst Placed in the 1-in. Line from the Oxidation Chamber Assembly to the Filter Assembly

A catalyst disk, 2 in. in diameter and 1 1/2 in. long, was used for the test. The catalyst was 0.3% platinum on a gamma alumina substrate coated on Dupont Torvex ceramic honeycomb and obtained from Strem Chemicals, Inc. The disk was placed in a 2-in. SS pipe, 3 in. long, that had been drilled and tapped for a thermocouple. A thermocouple was placed inside one of the honeycombs of the catalyst disk. The 2-in. SS pipe was placed in between the two flanges connecting the 1-in. offgas line from the oxidation chamber assembly to the filter assembly. A propane torch was used to preheat the catalyst to operating temperature before the beginning of the experiment. The kaowool around the SS filters was replaced prior to this experimental run.

When the blank flange was being removed from the 1-in. offgas line from the oxidation chamber assembly's catch pot, the line was found to be partially clogged with ash and condensed tars on the filter side of the flanged connection. The line was cleaned out and a catalyst placed between the two 1-in. flanges in an attempt to achieve a more complete conversion of the offgas to CO₂.

Six dry rags (268.74 g) were placed in a cylindrical carton (44.73 g) for a total loaded weight of 313.47 g; 280 g of water were added to the scrubber assembly. A worksheet for Run #6 is given in Table 3.10.

Table 3.10. Experimental Run #6

<u>Time</u>	<u>T1</u>	<u>T2</u>	<u>T3</u>	<u>T4</u>	<u>T5</u>	<u>P1</u>	<u>Comments</u>
8:46	18.7	18.6	18.6	18.7	40	0	Variac on @ 80%
8:54	412	19	201	72	57	2	Air on @ 16 L/min
8:57							Air on @ 47 L/min
9:00	539	71	390	218	105	32	
9:04	617	99	510	344	146	38	Total air = 580 L; air @ 147 L/min
9:10	702	141	621	513	305	40	Total air = 1500 L; air @ 172 L/min
9:14	746	391	674	619	315	40	Total air = 2200 L; air @ 176 L/min
9:18	755	807	683	604	319	40	Total air = 2900 L; air @ 178 L/min; variac off
9:24	573	616	611	548	302	40	Total air = 4000 L;
9:30	431	389	423	421	263	40	Total air = 5130 L; air @ 193 L/min
9:36	329	297	323	330	229	40	Total air = 6300 L; air @ 197 L/min
9:43	248	226	242	252	202	40	Air flow reduced from 204 L/min to 120 L/min
9:48	211	200	215	224	185		Catalyst torch turned off
9:56	174	170	179	188	156		Air turned off; total air = 9640 L

The catalyst bed never achieved operating temperature (350°C to 400°C) during the entire run. There is too much heat transfer from the offgases to the ash cup to facilitate placement of a catalyst in the offgas line from the ash cup.

Thermocouple placement for Run #6:

T1	Oxidation chamber skin
T2	Inside cylindrical carton
T3	Above grate
T4	Below grate
T5	Inside catalyst

0.20 g ash removed from the top of the grate; 0.65 g black char scraped from the ash cup. Total ash removed = 0.85 g.

292.3 g water removed from the scrubber assembly; however, an undetermined amount of water had leaked out through the lower ball valve during the night.

3.3.7 Run #7: Dry Rags, Catalyst Placed in Bottom of Conical Reducer

The catalyst disk from the previous run was removed from the 2-in. SS pipe that was placed in the 1-in. offgas line. The disk was then placed below the grate in the oxidation chamber assembly at the bottom of the conical reducer. A catch pot was placed above the catalyst in order to keep tars from falling into and fouling the catalyst. A thermocouple was placed inside the catalyst to monitor catalyst temperature. The SS filters were replaced with two new filters.

Six dried rags (356.13 g) were placed in a dry cylindrical carton (44.27 g) for a total loaded weight of 400.40 g. A worksheet of Run #7 is included here as Table 3.11. A worksheet of the offgas analysis is included as Table 3.12.

Table 3.11. Experimental Run #7

Time	T1	T2	T3	T5	P1	Comments
13:30						Torch turned on to preheat catalyst in cone
13:45	32.8	26.2	40.7	103		Variac on @ 80%
13:47	138	26	47	105	2	Vacuum pump on @ 11 L/min
13:50	319	27	112	123	2	
13:54	430	27	228	134	2	
13:58	517	29	336	167	2	Air @ 12 L/min
13:59	536	31	359	189	34	Air on full; catalyst temperature rising rapidly
14:01	582	77	432	288	32	Air @ 122 L/min; catalyst space red hot
14:04	630	95	530	333	32	Air total = 750 L; air @ 122 L/min
14:08	690	99	605	388	33	Air total = 1230 L; air @ 118 L/min
14:11	726	106	650	422	35	Air total = 1590 L; air @ 120 L/min
14:14	755	144	681	454	37	Air total = 1960 L; air @ 124 L/min
14:18	802	249	754	473	39	Air total = 2450 L; air @ 124 L/min
14:26	713	730	716	504	38	Air total = 3450 L; air @ 128 L/min
14:30	628	751	639	485	36	Air total = 3960 L; air @ 130 L/min
14:36	526	753	540	449	33	Air total = 4750 L; air @ 133 L/min
14:46	402	723	419	383	29	Air total = 6080 L; air @ 135 L/min
14:54	331	631	342	335	27	Air total = 7170 L; air @ 137 L/min
15:02	270	285	280	296	25	Air total = 8270 L; air @ 138 L/min
15:35		155	142	169	22	Air total = 12,800 L; vacuum pump off

Table 3.12. Average Gas Analysis for Experimental Run #7

Range	Representative Volume	CO₂	CO
(L)	(L)	(L)	(L)
0-915	915	112.4	16.93
915-1330	415	37.4	4.23
1330-1770	440	33.4	3.08
1770-2225	455	30.2	2.55
2225-2705	480	27.8	
2705-3230	525	22.6	
3230-3800	570	21.4	
3800-4390	590	17.3	
4390-5030	390	16.4	
5030-6205	1175	21.4	
6205-7815	1610	15.9	
7815-12,800	4985	25.9	

Thermocouple placement for Run #7:

T1	Oxidation chamber skin
T2	Inside cylindrical carton
T3	Grate
T5	Catalyst

The catalyst section only remained red hot for approximately 2 min. No smoke was ever observed in the filter assembly; however, the kaowool did become slightly discolored. The discoloration could have come from residue from the last run left in the ash cup or in the 1-in. offgas line.

Ash from on grate	1.20 g
Ash from reactor walls and baffle located above catalyst	0.45 g
Ash from ash cup	<u>0.00 g</u>
Total ash	1.65 g

3.3.8 Run #8: Catalyst, Rags Soaked in Fe(NO₃)₃ and Rinsed in Water

This run was done to simulate Runs 4 and 5 with a catalyst in the offgas. Five rags were prepared by dipping each one into 106.9 g of a neutralized solution containing 44.65 g of Fe(NO₃)₃*9H₂O. The neutralized solution was prepared by adding 50 g of 70% HNO₃ to 450 g

of tap water. Na_2CO_3 was added to the solution until fizzing stopped. After being dipped into the solution, each rag was allowed to sit for 15 min and then rinsed in 1.0 L of tap water. After rinsing, the rags were air dried for 2 hr then left sitting in a covered beaker overnight; 868.0 g of rinse solution were later analyzed and contained approximately 5.0 g of Fe.

Cylindrical carton	44.3 g
Damp Rags	<u>458.8 g</u>
Total	503.1 g

A torch was once again used to preheat the catalyst area prior to the experimental run. The scrubber assembly had 280.0 g of water added to it.

A worksheet summary for Run 8 is given in Table 3.13. A worksheet for the gas analysis is given in Table 3.14.

Table 3.13. Experimental Run #8

Time	T1	T2	T3	T5	P1	$\Delta P2$	Comments
9:44	26	19	32	106			Variac on @ 80%
9:46	158	19	43	96	1	0	Air on @ 13 L/min
9:54	458	20	254	142	2	0	
9:58	535	32	355	161	35	8	Air on full
10:00	558	68	403	222	30	10	Total air = 390 L; air @ 130 L/min
10:02	589	81	454	566	30	10	Total air = 640 L; air @ 120 L/min
10:05	630	85	537	323	30	10	Total air = 1000 L, air @ 120 L/min
10:08	674	90	584	379	30	12	Total air = 1370 L; air @ 120 L/min
10:12	722	88	656	417	33	12	Total air = 1840 L; air @ 117 L/min
10:18	789	193	747	472	36	12	Total air = 2550 L; air @ 117 L/min
10:22	822	489	773	507	38	15	Total air = 3025 L; air @ 120 L/min
10:26	837	639	782	531	40	12	Total air = 3525 L; air @ 122 L/min
10:30	843	791	780	550	40	16	Total air = 4025 L; air @ 126 L/min
10:35	840	797	774	560			Variac off
10:38	738	726	729	550	38	12	Total air = 5035 L; air @ 130 L/min
10:42	623	597	620	513	36	12	Total air = 5570 L; torch off catalyst area
10:45	553	535	556	483	33	11	Total air = 5950 L; air @ 132 L/min
10:55	395	395	403	392	29	11	Total air = 7300 L; air @ 135 L/min
11:05	295	305	304	325	25	13	Total air = 8660 L; air @ 138 L/min
11:06							Sharp crack (ping) heard. Weld cracked on air inlet line.
11:15	224	239	231	256	24	11	Total air = 10,050 L; air @ 138 L/min
11:27							EOR; total air = 11,730 L

Water removed from scrubber assembly	392.2 g
Ash removed from top of grate	0.85 g
Ash vacuumed out of reactor	1.80 g
Ash removed from reactor ash cup	<u>0.20 g</u>
Total ash removed from Run #8	2.85 g

Table 3.14. Average Gas Analysis of Experimental Run #8

<u>Range</u>	<u>Representative Volume</u>	<u>CO₂</u>	<u>CO</u>
(L)	(L)	(L)	(L)
0-445	445	7.74	3.0
445-885	440	24.9	5.3
885-1400	515	33.3	1.4
1400-1990	590	41.1	2.0
1990-2620	630	35.8	
2620-3230	610	29.3	
3230-3850	520	19.6	
3850-4485	635	14.8	
4485-5155	670	14.4	
5155-5980	825	5.7	
5980-7330	1350	5.1	
7330-9190	1860	6.5	
9191-10,920	1730	4.5	
10,920-11,730	810	1.5	

3.4 Tabulated Test Results

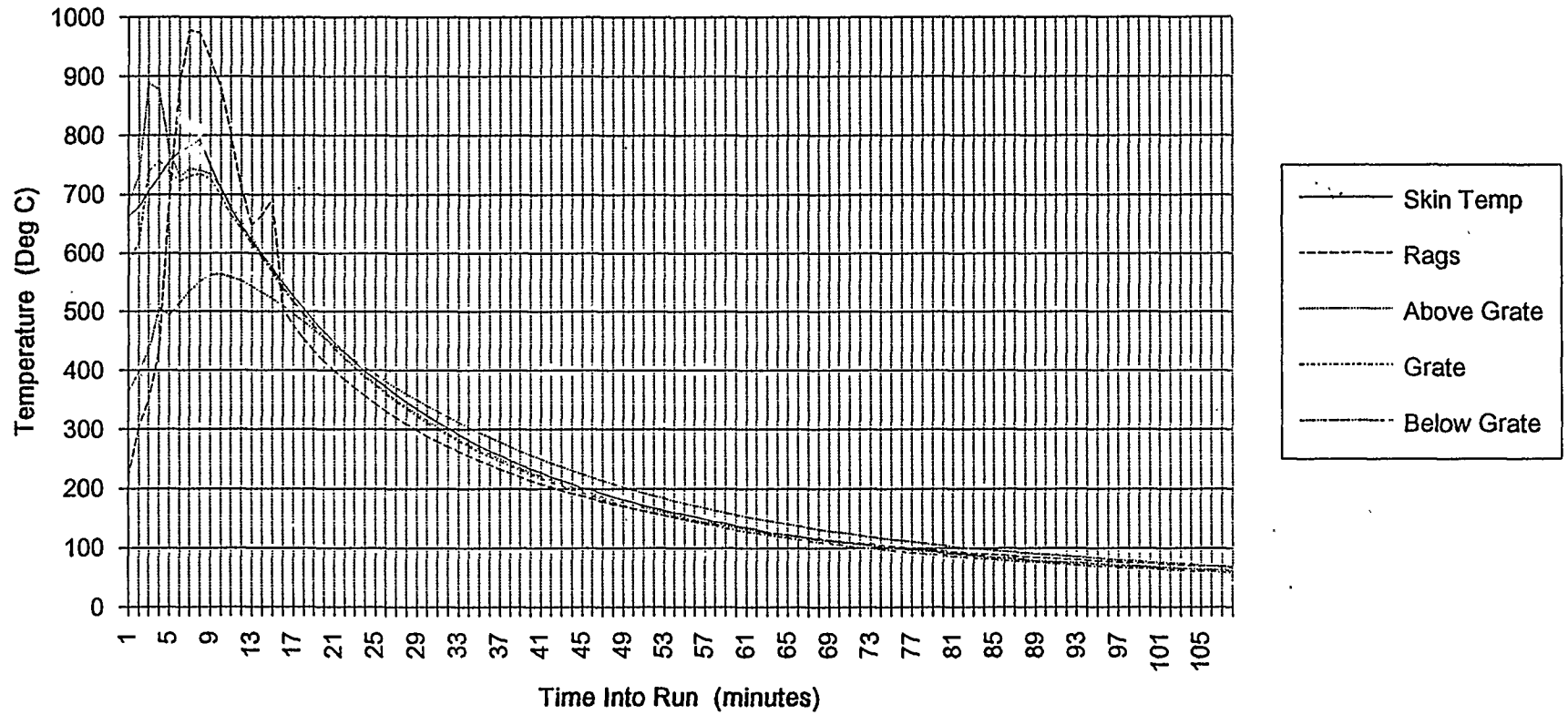
The results from the eight experimental runs were tabulated and are shown in Table 3.15. Graphs of temperature profiles for Runs 1-3 and 5-8 are also included in this Section.

Table 3.15. Tabulated Results of Experimental Runs #1 Through 8

Run #	Physical Condition	Chemical Preparation	Loaded Wt (g)	Ash Wt (g)	Wt Reduction	Scrubber Loading (g)
1	Dry, loose pack	None	214.52	1.49	144:1	
2	Dry, tight pack	None	370.1	1.55	239:1	49.1
3	Wet, tight pack	76.8 g H ₂ O	403.1	1.09	370:1	26.5
4	Wet, soaked in neutralized solution, tight pack	152.1 g 59.3 wt% H ₂ O 6.6 wt% 70% HNO ₃ 4.5 wt% Na ₂ CO ₃ 29.5 wt% Fe(NO ₃) ₃	448.1	not determined		
5	Wet, soaked in acid, rinsed in water	458.1 g 89.22 g Fe(NO ₃) ₃ dissolved in 10% HNO ₃ to make 200 g of acid solution	798.4	4.16	192:1	205
6	Dry, tight pack	None	313.47	0.85	369:1	12.3
7	Dry, tight pack	None	400.40	1.65	243:1	
8	Wet, soaked in neutralized solution, rinsed in water	227.46 g 59.3 wt% H ₂ O 6.6 wt% 70% HNO ₃ 4.5 wt% Na ₂ CO ₃ 29.5 wt% Fe(NO ₃) ₃	503.1	2.85	177:1	49.2

Downdraft Gasifier Run #1
Loose Packed Wipers (Dry)

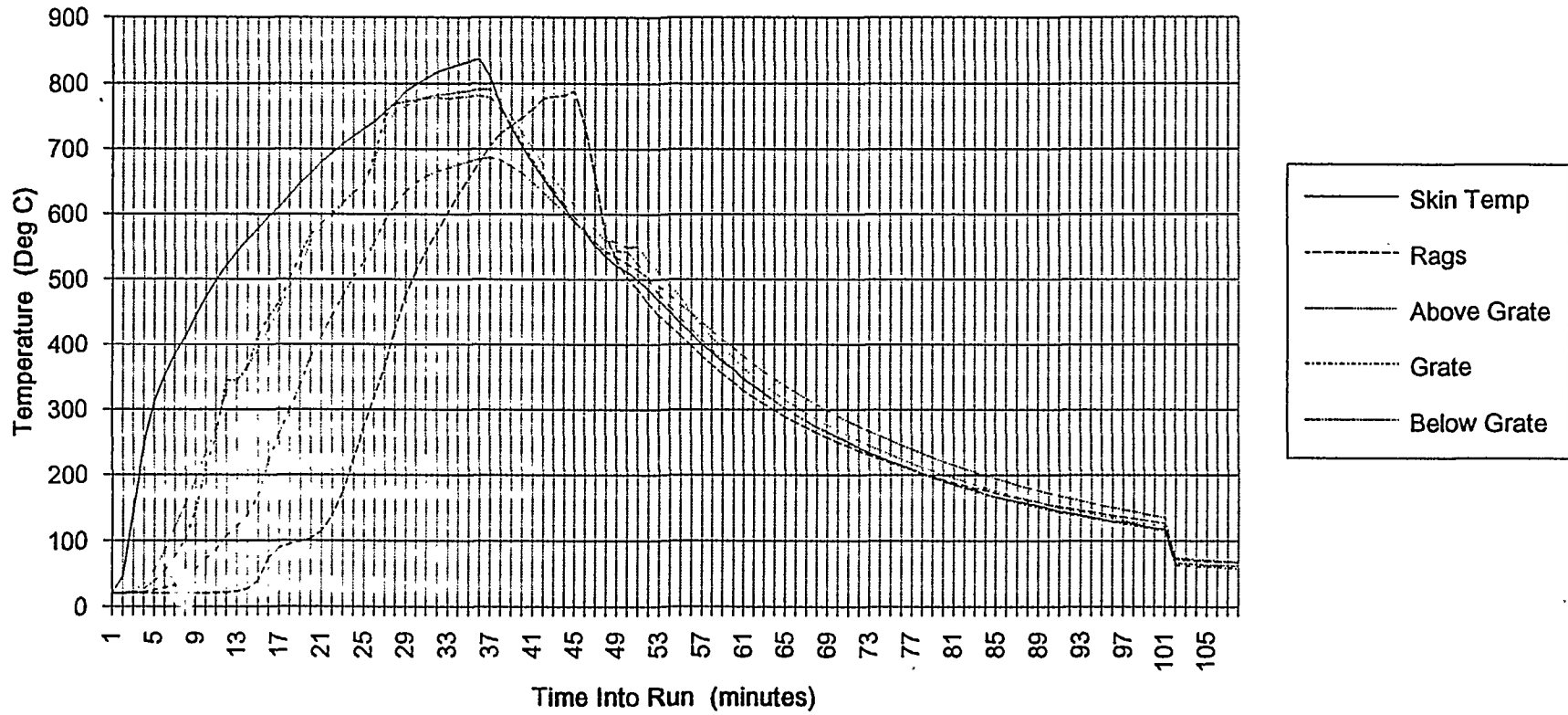
3.18



Downdraft Gasifier Run #2

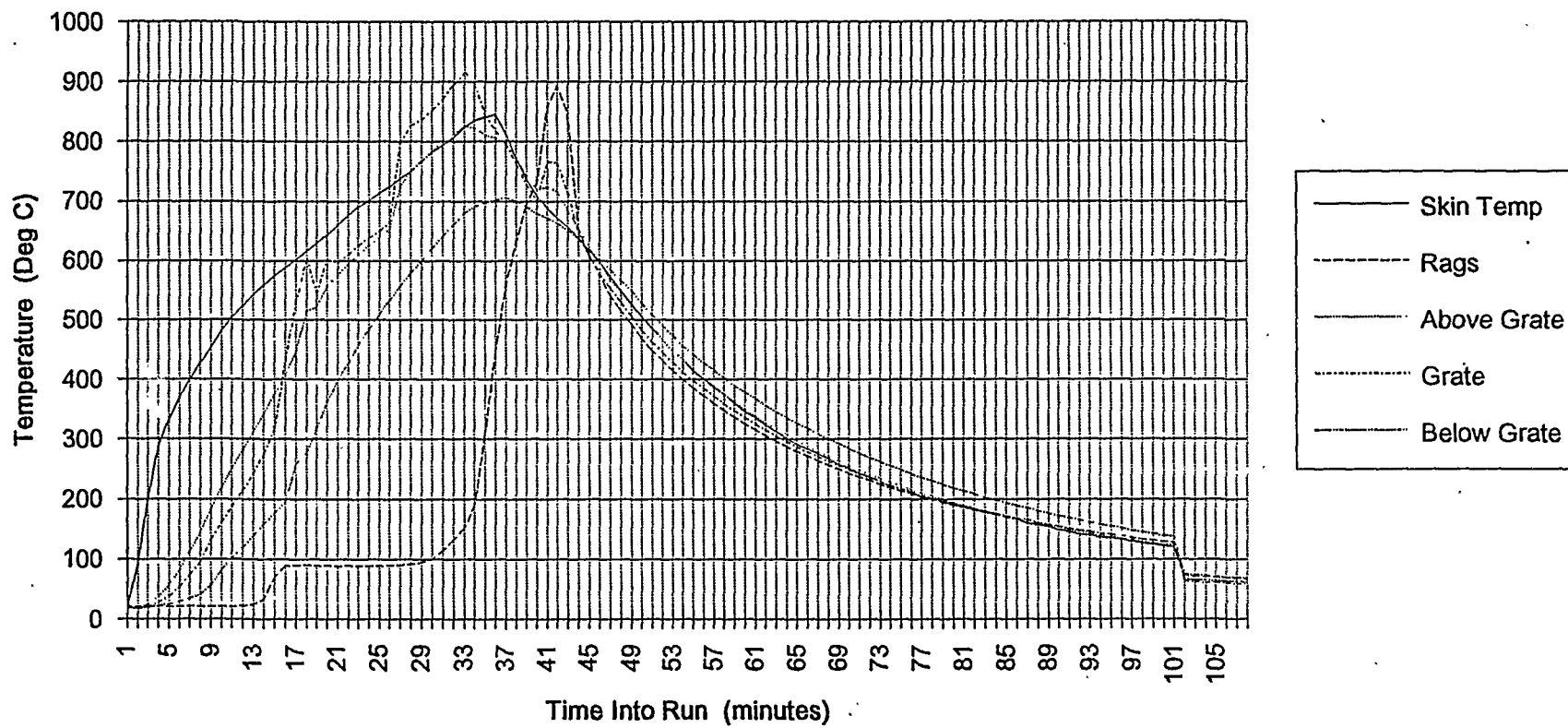
Compact Wipers (Dry)

3 19



Downdraft Gasifier Run #3

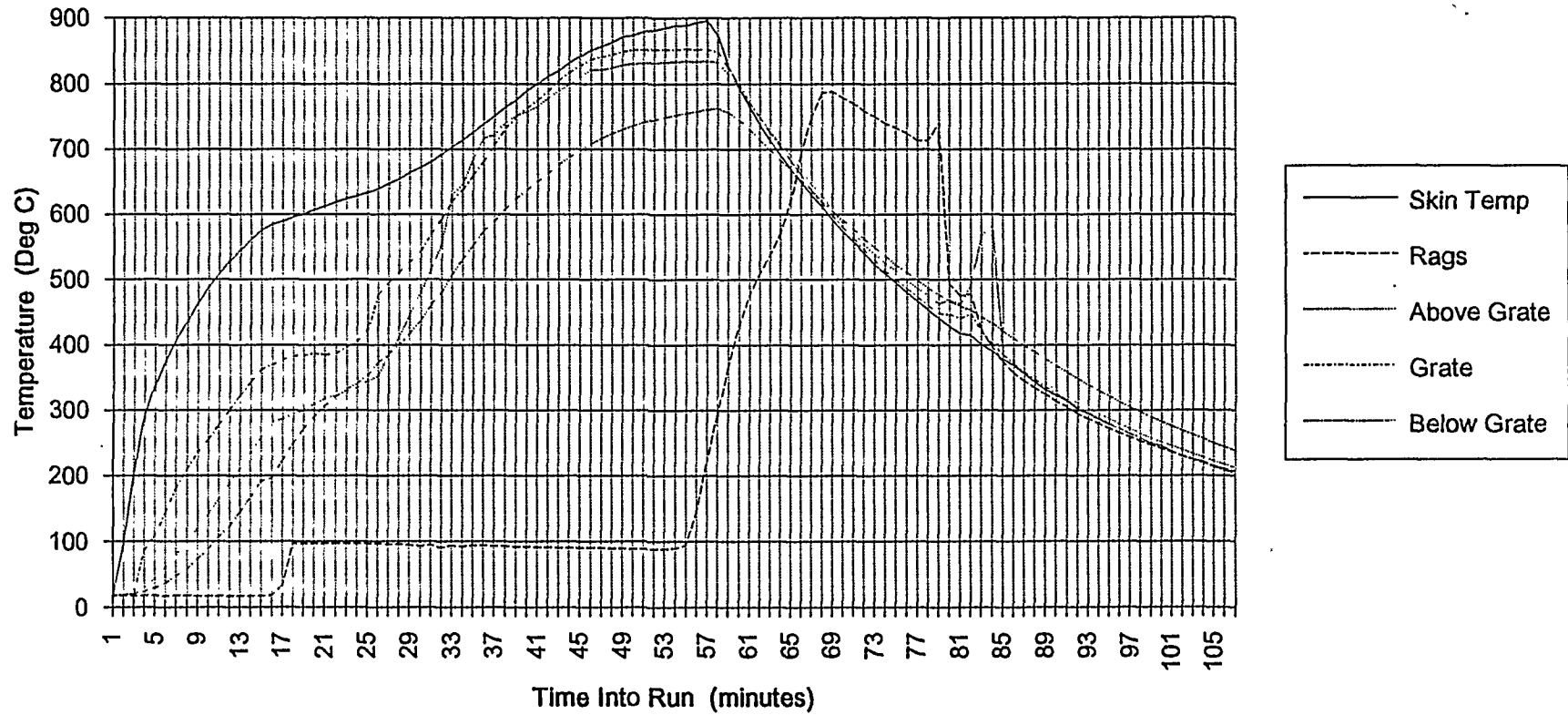
21.5 wt% H₂O



3.20

Downdraft Gasifier Run #5

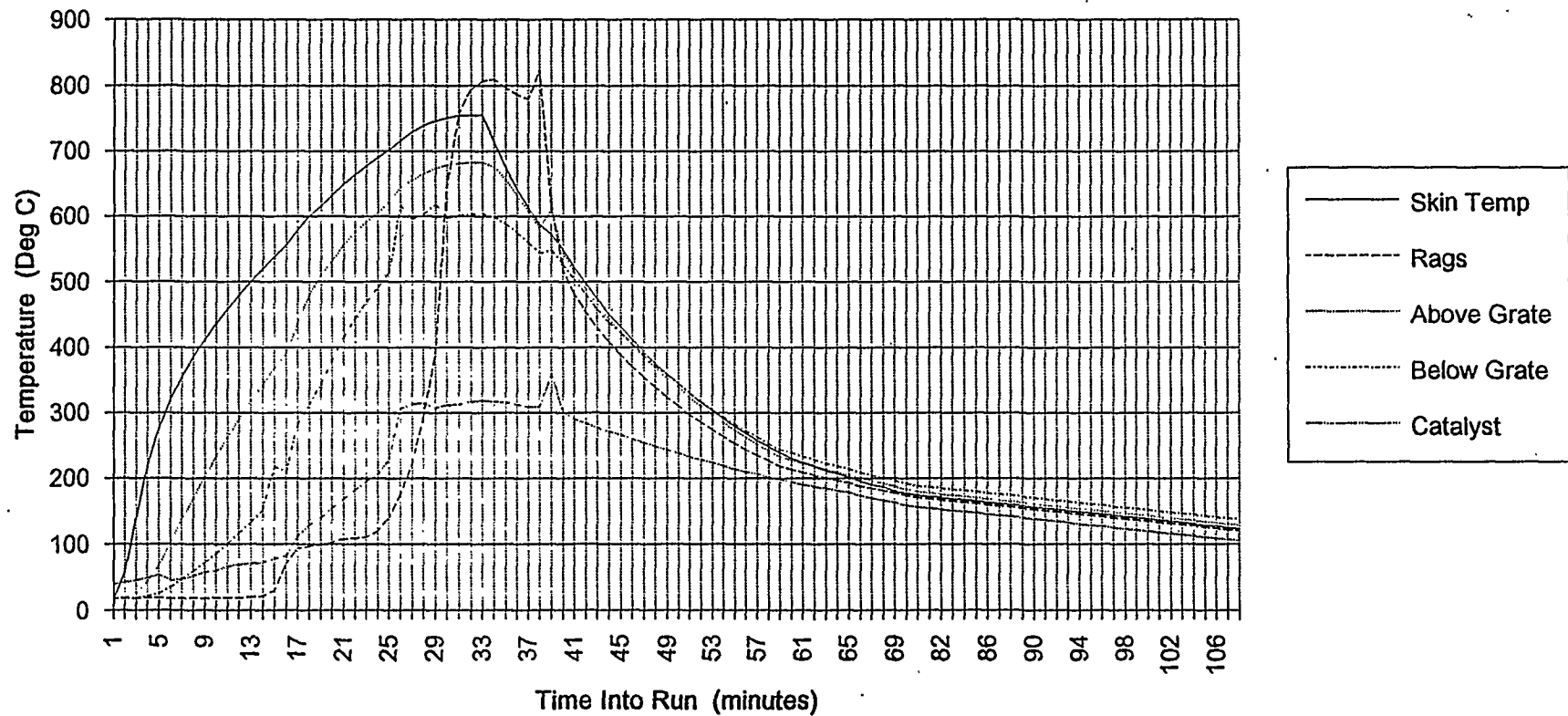
Wipers soaked in $\text{Fe}(\text{NO}_3)_3$, rinsed in H_2O , air dried



3.21

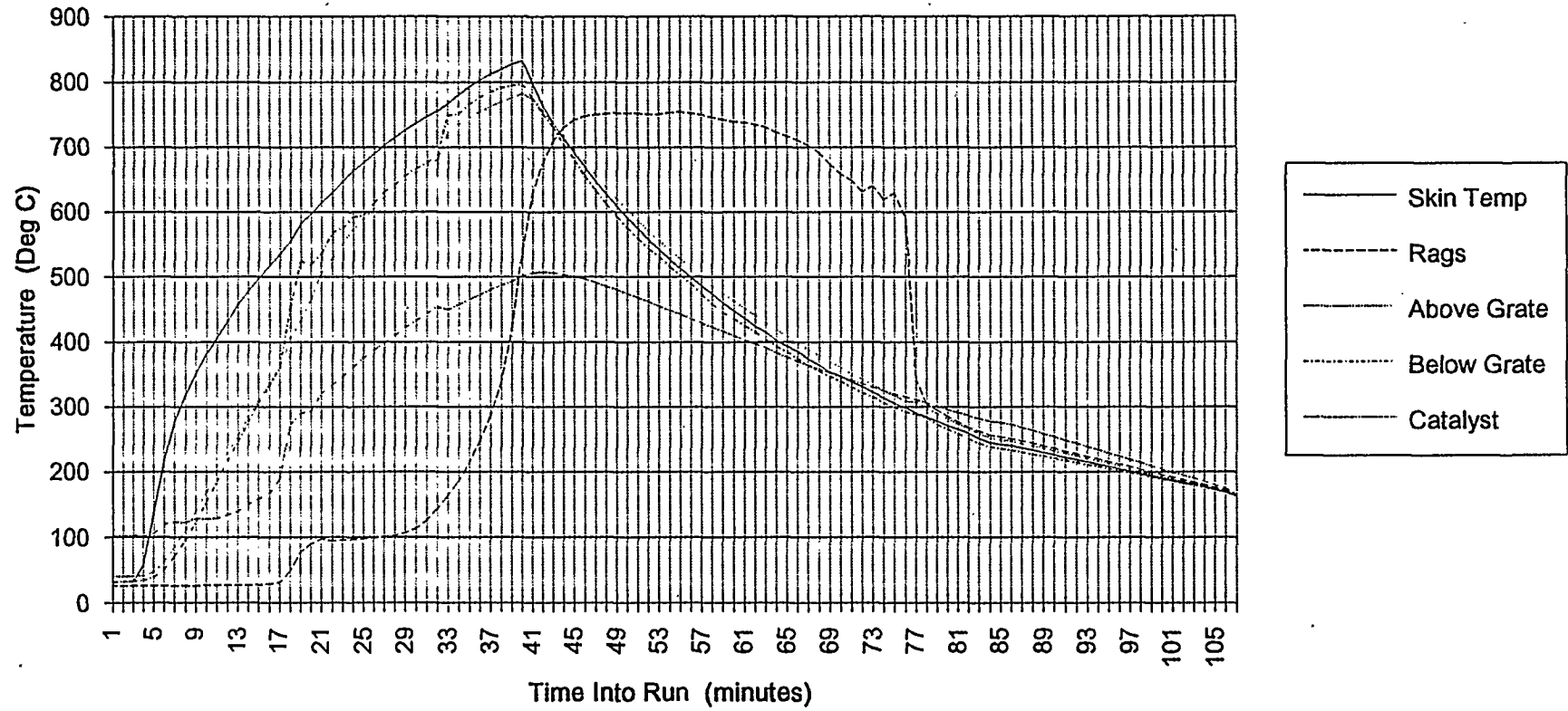
Downdraft Gasifier Run #6

Dry wipers, Catalyst between ash pot and filter assembly



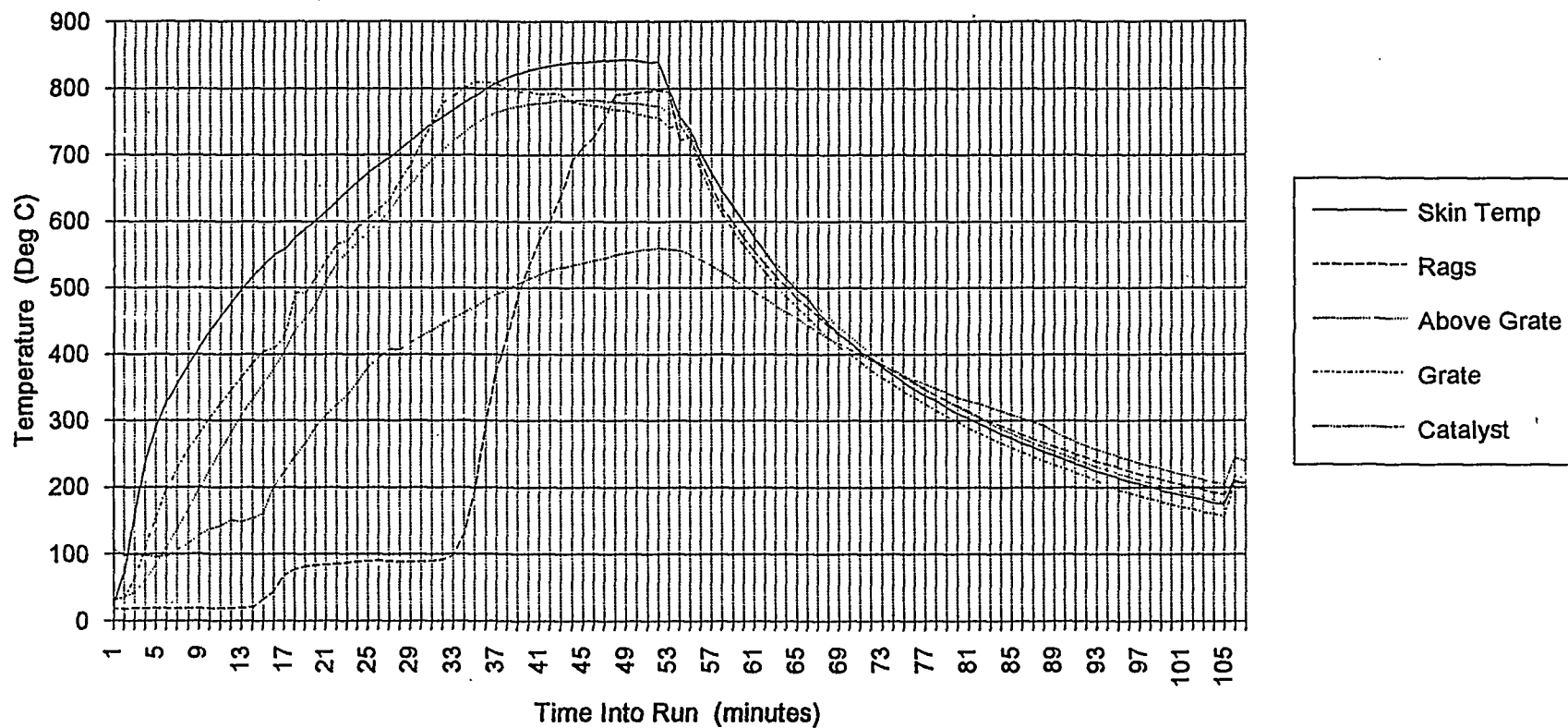
Downdraft Gasifier Run #7

Dry Rags; Catalyst below grate



Downdraft Gasifier Run #8

Soaked in Fe(NO₃)₃, Rinsed in H₂O, Air Dried



3.24

4.0 Conclusions and Recommendations

Results of this series of testing indicate that the potential for this type of thermal oxidation chamber is significant. Weight reductions of 150:1 were easily obtainable. With modifications, weight reductions of over 200:1 should be obtainable, with possible volume reductions of 500:1. Recommendations for increasing the efficiency of the system are noted as follows:

- The offgas leaving the main oxidation chamber is loaded with condensable tars. A high-temperature chamber located immediately after the main oxidation chamber could provide more complete thermal oxidation. The high-temperature chamber should be attached in such a manner as to prevent collection of tars and resins below the first stage of thermal oxidation.
- The offgas from the system should be analyzed for NO_x and combustible gas emissions.
- An efficient thermal oxidation system might be useful in plutonium recovery and recycling processes from contaminated plastics and other combustible materials. A series of tests need to be conducted with materials other than 100% cotton to make further evaluations.
- The filters were wrapped in kaowool to prevent plugging due to excessive loading. The kaowool worked as desired; however, a better solution to the problem needs to be found. One solution might be to cover the filters with paint roller rolls that have a cardboard inner liner. The rolls could easily be installed and removed by a worker in a glove box situation. The loaded rollers could then be disposed of by thermal oxidation.
- A loading assembly needs to be added to the original design in order to test the thermal oxidation system in a continuous mode.
- Air inlet tubes need to be configured and attached to the oxidation chamber body in a different configuration. The original design results in leaks in the tubing where it is attached (welded) to the oxidation chamber wall. A better design would be to bend the tube into several alternating "u" and "n" shaped portions and attach the tubing with conduit straps on the vertical legs.
- Ash collects on the grate. In a continuous mode operation, it would not take long before the ash would collect to the point where operations would have to halt to clear the grate of ash. A method of agitating the grate surface to knock off any loose ash needs to be found. One possible solution is a mechanical arm that could sweep the surface of the grate and break the ash into finer particles. The arm should be operational from outside the oxidation chamber while the system is running.
- A series of tests need to be conducted with simulated PFP glove box wipers in a system modified to run on a continuous basis to make any further evaluations.

