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CALCINATION OF DILUTE ZIRCONIUM WASTE

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ABSTRACT

A method was developed for converting dilute zirconium wastes stored at the Idaho Chemical Processing Plant (ICPP) into granular, free-flowing solids using fluidized-bed calcination. Prior to calcination, the dilute zirconium wastes are blended with sodium-bearing waste so that the calcine produced contains as much as 5.3 mole % sodium; calcium nitrate is added to the blend to give a calcium-to-fluoride mole ratio of 0.7.

SUMMARY

Zirconium fluoride wastes stored in Tanks WM-182, -188, and -189 have roughly 50% of the zirconium and fluoride and 70% of the aluminum concentration contained in zirconium (Zr) wastes calcined in the Waste Calcining Facility (WCF) prior to the ninth campaign. In addition, WM-188 and -189 wastes contain roughly 200 ppm chloride whereas the Zr wastes calcined in the past (concentrated Zr wastes), more concentrated in zirconium, aluminum, and fluoride, contained about 20 times less chloride. Pilot-plant calcination studies were made to determine if the wastes stored in Tanks WM-182, -188, and -189 (dilute Zr wastes) could be calcined in the same manner as used for concentrated Zr wastes. Calcination of concentrated Zr wastes required:

a) addition of enough calcium nitrate ($Ca(NO_3)_2$) prior to calcining the waste to give a calcium-to-fluoride (Ca/F) mole ratio of 0.55 when calcining Zr wastes alone;

b) addition of enough $Ca(NO_3)_2$ prior to calcining the waste to give a Ca/F mole ratio of 0.7 when calcining a blend of Zr wastes and sodium-bearing (Na) wastes.

When calcining dilute Zr wastes having a Ca/F mole ratio of 0.55, fluidized-bed operation, properties of calcine solids produced, and volatility of chloride and fluoride from the calciner vessel were similar to that achieved when calcining concentrated Zr wastes. Pilot-plant calcination of both concentrated and dilute Zr wastes release roughly 80% of the chloride to the off gas. Calcination of the dilute Zr wastes in the WCF containing 200 ppm chloride (without blending with Na wastes) might allow the chloride concentration in the off-gas acid scrubbing solution to exceed the maximum allowable concentration for corrosion control (2600 ppm) whereas past calcination of the concentrated Zr wastes would not. The product rate obtained during calcination of the dilute wastes was 1/2 that of the concentrated wastes. Calcination of the dilute wastes in the WCF could lead to difficulty in building bed and maintaining bed height. This problem can be resolved by calcining a blend of dilute Zr wastes and Na wastes.

Blending dilute Zr wastes with Na wastes in a ratio that produces calcine containing 5.3 or less mole percent sodium and then adding $Ca(NO_3)_2$ to the blend to give a Ca/F mole ratio of 0.7 results in quality fluidization and should prevent the chloride concentration in the off-gas scrubbing system from exceeding 2600 ppm.

It is recommended that dilute Zr wastes to be calcined in the WCF be blended with Na wastes to produce calcine with a sodium mole percent of 5.3 or less and that the blend contain a Ca/F mole ratio of 0.7. Using the waste nozzle air ratio and oxygen-to-fuel ratio to control particle size is preferred over alternately calcining blended dilute Zr-Na wastes with unblended dilute Zr waste.

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I. INTRODUCTION

At the Idaho Chemical Processing Plant (ICPP) aqueous radioactive wastes are solidified in the Waste Calcining Facility (WCF) using a fluidized-bed calciner. The resulting calcine is suitable for long-term storage as granular solids. The WCF, during its ninth processing campaign prior to October 1979, calcined zirconium fluoride wastes from storage Tanks WM-182, -188, and -189. The wastes in Tanks WM-188 and -189 were a blend of first-cycle zirconium fluoride waste and stainless steel sulfate waste diluted with non-fluoride wastes. The waste in WM-182 was first-cycle zirconium fluoride waste diluted with non-fluoride wastes. The known compositions of the three diluted zirconium fluoride wastes and an undiluted zirconium fluoride waste are tabulated in Table I. The diluted zirconium fluoride wastes contained roughly 50% of the zirconium and fluoride and 70% of the aluminum found in first-cycle zirconium fluoride wastes calcined prior to Campaign nine. Zirconium fluoride wastes diluted in zirconium, aluminum, and fluoride concentrations would be more difficult to calcine than the undiluted waste because calcination of the dilute waste would be more likely to produce excessive fines and/or not to build bed. This report describes studies performed in 10- and 30-cm-diameter, fluidized-bed, pilot-plant calciners prior to the ninth processing campaign of the WCF. These studies were intended to determine a method for calcining diluted zirconium fluoride wastes (dilute Zr wastes).

Prior to the ninth processing campaign of the WCF, zirconium fluoride (Zr) wastes had been successfully calcined in the WCF by adding sufficient calcium nitrate ($Ca(NO_3)_2$) to the waste to give a calcium-to-fluoride (Ca/F) mole ratio of 0.55 prior to calcination or by calcining a blend of 3 1/2 volumes (vol) of Zr waste with 1 vol of sodium-bearing (Na) waste to which $Ca(NO_3)_2$ had been added to give a Ca/F mole ratio of 0.7. The composition of the Na waste used in the blend is given in Table I. Pilot-plant calciner studies were conducted to determine if dilute Zr wastes could be calcined in the same manner as undiluted Zr wastes had been successfully calcined in the WCF. During these studies: (a) a synthetic dilute Zr waste having a Ca/F mole ratio of 0.55 was calcined in 10- and 30-cm calciners, (b) synthetic blends of 3, 5, and 8 vol of a dilute Zr waste and 1 vol Na waste were calcined in a 10-cm calciner, and (c) a synthetic blend of 5 vol of a dilute Zr waste and 1 vol Na waste were calcined in a 30-cm calciner. Calciner operability, fluidized-bed operation, characteristics of calcined solids produced, and volatility behavior of fluoride, chloride, mercury, and sulfate were studied during these calciner runs.

TABLE I

MOLAR COMPOSITION OF WASTES CALCINED IN THE WCF

Constituent	Dilute Zirconium Fluoride Wastes as of 10-1-79 in Tanks			Zirconium Fluoride Waste in Tank	Sodium-Bearing Waste in Tank
	WM-182	WM-188	WM-189	WM-185[a]	WM-180[b]
H+	1.20	1.35	1.58	1.51	0.92
Aℓ	0.41	0.59	0.46	0.68	0.55
Zr	0.19	0.30	0.24	0.44	<0.05
B	0.096	0.17	0.12	0.20	0.022
Fe	0.007	0.01	0.008	0.005	0.06
Na	0.05	0.05	0.06	0.001	1.20
K	—	0.008	0.008	—	—
Mg	—	0.01	0.008	—	—
Mn	—	0.002	0.001	—	—
Hg	—	0.001	0.01	—	—
F	1.28	1.94	1.57	3.12	0.31
Cℓ	—	0.006	0.006	0.0003	0.026
SO_4	—	0.07	0.05	—	—
NO_3	2.58	2.63	2.64	2.36	4.02

a. This waste was calcined in the WCF during its eighth processing campaign.

b. Based on analyses of two samples from Tank WM-180 in June 1979.

II. EXPERIMENTAL EQUIPMENT AND PROCEDURES

Schematic diagrams and brief descriptions of the 10- and 30-cm-diameter calciners used to develop a calcination flowsheet for dilute Zr wastes are shown in Figures 6 and 8 in the appendix of ICP-1163.[1] A single synthetic waste (hereafter referred to as the dilute Zr waste) was used during flowsheet development studies to represent the wastes stored in Tanks WM-182, -188, and -189. The composition of this waste and of the synthetic Na waste used are shown in Table II.

1. TEN-CENTIMETRE-DIAMETER CALCINER STUDIES (RUNS DZrW4-1, -2, -3 and -4)

Run DZrW4-1 was made to see if calcination of the dilute Zr waste with a Ca/F mole ratio of 0.55 gave desirable calciner operability, fluidized-bed operation, calcined solids' characteristics, and volatile behavior of fluoride, chloride, and mercury. Runs DZrW4-2, -3, and -4 were made to study the effect of varying the dilute Zr to Na wastes blend ratio (using sufficient $Ca(NO_3)_2$ to give a Ca/F mole ratio of 0.7) on calciner and fluidized-bed operation, calcine properties, and fluoride, chloride, and mercury volatility. The process heat for the fluidized-bed operation was supplied by in-bed combustion; the calcination temperature was 500°C; kerosene was the fuel used in heating the calciner; and a propane torch was used to initially raise the temperature inside the calciner above the autoignition temperature of a kerosene-oxygen mixture.

Off gas leaving the 10-cm-diameter calciner passes through (in the order named) a primary cyclone to remove fines, a venturi scrubber (using 1-4M nitric acid) for further fines removal, a secondary cyclone, and finally a water-cooled condenser. About 2% of the total off gas was drawn through a caustic scrubber from a point between the secondary cyclone and condenser. Acid scrubbing solution was not recycled to calciner feed. The scrubbing solution was replaced with new acid at least every 8h; the solution was replaced more frequently when too many solids in the acid scrub tank caused plugging in the acid scrub system or caused excessive back-pressure on the acid scrub pump. Solution was also removed from the acid scrub tank to keep the tank at a desired original level. All solution removed from the acid scrub tank was placed in a single container. At the end of the run the scrubbing solution in the single container was stirred, sampled, and analyzed for some of its constituents.

Samples of calcine product, calcine fines, and caustic scrub were taken every 8h. Some samples of each type were analyzed for some of their constituents. The starting bed used for the run calcining the unblended dilute Zr waste was obtained from the calcination at 500°C of waste simulating the WM-185 waste (Table I). The starting bed used for runs calcining blends of the dilute Zr waste and Na waste was obtained from a 30-cm calciner run in which a blend of 5 vol synthetic WM-185 waste and 1 vol Na waste was calcined at 500°C. The mass mean particle diameter (MMPD) and bulk density were determined for the calcine product every 8 h; MMPD, bulk density, and attrition index were determined for the initial and final beds; photographs were taken of magnified particles of the final bed; bulk densities were determined for the fines every 8 h; and weight of product and fines were noted during each run.

TABLE II

TYPICAL COMPOSITION OF SIMULATED WASTES USED IN DILUTE ZIRCONIUM WASTES STUDIES

	Concentration (Molar)	
Constituent	The Dilute Zr Waste	Sodium-Bearing Waste
Zr	0.22	
Aℓ	0.47	0.51
H^+	0.7	1.2
B	0.092	0.008
Na		1.8
K		0.2
Mn	0.004	0.017
Hg	0.002	0.003
Fe	0.009	0.017
Cr	0.008	
Ni	0.002	
Mg	0.009	
NO_3	1.3	4.9
F	1.37	
Cℓ	0.008	0.036
PO_4		0.019
SO_4	0.1	0.06

Forty-hour runs were made to test calcination of the dilute Zr waste and blends of 3, 5, and 8 vol of the dilute Zr waste with 1 vol of Na waste. Solid $Ca(NO_3)_2$ was added to the dilute Zr waste and to blends prior to calcination to give Ca/F mole ratios of 0.55 and 0.70, respectively. The size of calcine particles was controlled between 0.3 and 0.6mm by varying the feed nozzle air ratio (NAR) during the runs. Appendix A summarizes the operating conditions of all 10-cm-diameter calciner runs.

2. THIRTY-CENTIMETRE-DIAMETER CALCINER STUDIES (RUNS 71 AND 74)

A 30-cm-diameter, fluidized-bed, in-bed combustion calciner was used to calcine the dilute Zr waste with a Ca/F mole ratio of 0.55 (Run 74) and a blend of 5 vol dilute Zr waste and 1 vol Na waste with a Ca/F mole ratio of 0.70 (Run 71) to show the desirability of these flowsheets for testing in the WCF. The calcination temperature was 500°C; kerosene was the fuel; and a propane torch was used to raise the temperature inside the calciner above the autoignition temperature of a kerosene-oxygen mixture.

Off gas leaving the calciner passes through (in the order named) a primary cyclone, a quench tower, a venturi scrubber (using 0.5-4M nitric acid), a knock-out pot, and a condenser. About 0.3% of the total off gas was pulled through a caustic scrubber from a point between the knock-out pot and the condenser. Acid scrub was not recycled to calciner feed. Any solution removed from the off-gas acid scrubber was placed in a sparged hold tank. The acid scrub hold tank was sampled every time the acid scrub was sampled (every 8h); whenever an acid scrub sample was analyzed, an acid scrub, hold-tank sample for the same time interval was analyzed. If the hold tank was dumped during a run, it was dumped immediately after it had been sampled at the end of a regular 8-h sampling period. The product, fines, and acid scrub were sampled at 8-h intervals; each batch of feed containing $Ca(NO_3)_2$ was sampled; and the caustic scrub (having operated 8h with new 6M sodium hydroxide) was sampled at COT 20, 36, 52, 60, 84, and 100h (for Run 71) or 108h (for Run 74). During 8-h intervals—ending at COT 20, 52, 84, and 100h —there was only one caustic scrubber containing 1000 mL of 6M sodium hydroxide (NaOH) in the scrubber train; during the interval ending at COT 36h, there were two scrubbers in series, the first containing 1000 mL of 6M NaOH, the second containing 500 mL of 6M NaOH; during the interval ending at COT 60h, there were two scrubbers in series, the first containing 1000 mL of 6M NaOH, the second 500 mL of 12M hydrochloric acid. The two caustic scrubbers in series were used to determine how much fluoride and chloride were escaping from a single scrubber. The caustic and acid scrubbers in series were used to determine how much mercury was escaping from a single caustic scrubber. Some samples of each type were analyzed for some of their constituents. Calcine generated from the calcination of 1.7M aluminum nitrate containing 0.01M boric acid at 500°C was used as a starting bed for Run 71. The final bed of Run 71 was used as a starting bed for Run 74. The MMPD and bulk density were measured every 8h and the attrition index was determined every 16h on calcine product; fines' bulk density was measured every 8h; photographs were taken of magnified particles of product at COT 64, 88, and 112h; and weights of products and fines were determined at various times.

Operating conditions for Run 71 are listed in tables and curves in Appendices B and E, and for Run 74 in Appendices F and I.

During both runs, corrosion coupons of type 304L stainless steel were present in the off-gas line and equipment.

III. CALCINATION OF DILUTE ZIRCONIUM WASTE

The dilute Zr waste (Table II) having a Ca/F mole ratio of 0.55 was calcined for 40h in a 10-cm-diameter calciner and for 112h in a 30-cm-diameter calciner. Both runs were voluntarily terminated.

1. TEN-Cm CALCINER STUDY (RUN DZrW4-1)

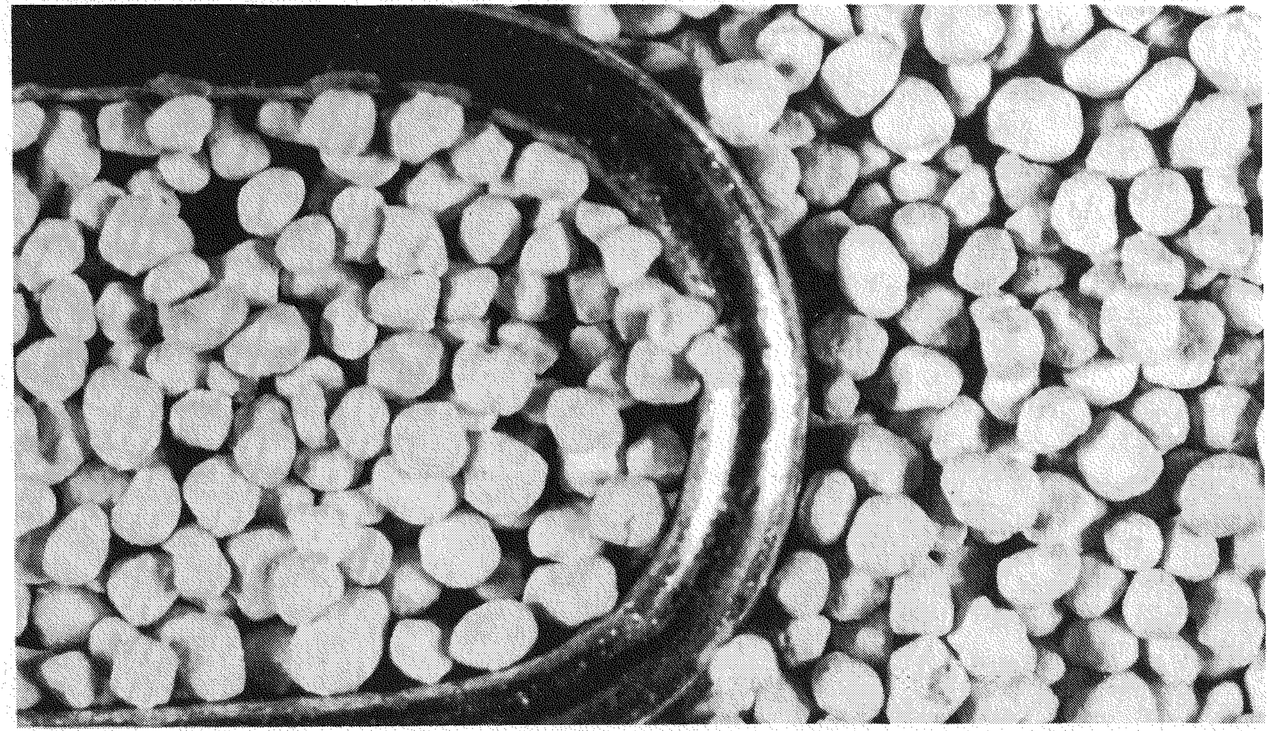
1.1 Fluidized-Bed Operation and Properties of Solids Generated

An initial NAR of 730 was used, but after 10h of run time, the NAR was decreased to 620 to prevent the product MMPD from decreasing below 0.3 mm during the 40h of run time. Excessive clinker formation did not take place in the bed. At the end of the run there were normal amounts of agglomerates inside the calciner stuck to calciner surfaces, nozzles, the propane torch, and thermocouple sheaths. The fines seemed to be sticky, causing primary cyclone plugging; the fines did not seem to be moist: they flowed out of the fines pot when it was given a sharp rap.

Photographs were taken of magnified, final bed material. No nodules, which reduce the quality of fluidized-bed operation, were found on the final bed material, and the surfaces of the material were relatively smooth. Figure 1 shows typical magnified calcine particles obtained by calcining the dilute Zr waste having a Ca/F mole ratio of 0.55 in a fludized-bed calciner. The figure also shows magnified calcine particles obtained by calcining at 500°C a Zr waste more concentrated in zirconium, fluoride, and aluminum (WM-185 waste of Table I) having a Ca/F mole ratio of 0.55. The pieces of metal shown in the photographs are paper clips. The surfaces of particles produced from the dilute Zr waste appear similar to those produced from the WM-185 waste. Particles produced from the calcination of WM-185 waste at 500°C in the WCF during its eighth campaign give high quality fluidized-bed operation.

Appendix A summarizes the data obtained from Run DZrW4-1 on fluidized-bed operation and properties of solids generated. The more important average data are shown in Table III. The values for product attrition index, product bulk density, and weight of final bed >14 mesh obtained during the calcination of the dilute waste are the same as would be expected from calcining a waste more concentrated in zirconium, aluminum, and fluoride (WM-185 waste of Table I) having the same Ca/F mole ratio and solidified in the same calciner operating under the same conditions.[2] The values for product rate (111 vs 200 g/h) and product-to-fines ratio (1.4 vs 2.0) are less than would be expected from calcining WM-185 waste having the same Ca/F mole ratio in the same calciner operating under the same conditions.[2] These low values for product rate and product-to-fines ratio indicate that difficulty could be experienced in maintaining bed level during calcination of the dilute waste in the WCF.

From the Dilute Zr Waste with a Ca/F Mole Ratio of 0.55



From WM-185 Waste with a Ca/F Mole Ratio of 0.55

Figure 1. Magnified Calcine Particles of Zr Wastes

TABLE III

FLUIDIZED-BED OPERATION AND PROPERTIES OF SOLIDS GENERATED DURING CALCINATION OF DILUTE Zr WASTES IN A TEN-cm CALCINER

Run #	Waste Calcined	Product Rate (g/h)	Product-to-Fines Ratio	Product Attrition Index (%)	Product Bulk Density (g/cm^3)	Weight of Final Bed >14 mesh (%)
DZrW4-1	Dilute Zr Waste	111	1.4	20	1.36	2.1
DZrW4-2	5 vol Dilute Zr Waste-1 vol Na Waste	177	4.7	82	1.64	0.7
DZrW4-3	8 vol Dilute Zr Waste-1 vol Na Waste	182	4.2	74	1.53	0.4
DZrW4-4	3 vol Dilute Zr Waste-1 vol Na Waste	233	6.5	76	1.61	4.0

1.2 Fluoride Volatility

Values summarizing the behavior of fluoride in calciner off gas during calcination of the dilute Zr waste in a 10-cm calciner are given in Table IV. During calcination of the dilute waste, 5.9% of the fluoride fed to the calciner was present in the off-gas acid scrub in excess of that which could be present as fluoride combined with calcium as CaF_2; 1.1% of the fluoride fed to the calciner escaped the acid scrubber as volatile fluoride uncombined with calcium as CaF_2. Thus 7% of the fluoride fed to the calciner was probably present in calciner off gas as volatile fluoride not entrained in fines. The zirconium, aluminum, and boron in the acid scrub were not present in sufficient quantities to complex the fluoride present (next to last row in Table IV). The material of construction in the WCF off-gas acid scrubber system is 304L stainless steel, and the acid scrubber is operated at 70°C. Based on a model developed at the ICPP, the corrosion rate of 304L stainless steel at 70°C in the acid scrub solutions produced during Run DZrW4-1 (not taking chloride concentrations of the scrub solution into consideration) would be roughly 127 μm/mo (5 mils/mo).[3] Corrosion rates greater than 51μm/mo (2 mils/mo) are considered undesirable.

1.3 Chloride Volatility

Values summarizing the behavior of chloride in calciner off gas during calcination of the dilute Zr waste in a 10-cm calciner are given in Table V. Chloride retention in calcine was very poor (<33%) during calcination of the dilute waste.

TABLE IV

FLUORIDE BEHAVIOR IN CALCINER OFF GAS DURING TEN-CENTIMETRE CALCINER RUNS

Run #	DZrW4-1	DZrW4-2	DZrW4-3	DZrW4-4
Waste Feed	Dilute Zr Waste	5 vol dilute Zr waste-1 vol Na waste	8 vol dilute Zr Waste-1 vol Na waste	3 vol dilute Zr waste-1 vol Na waste
Ca (M) in Acid Scrub	0.13	0.17	0.24	0.098
F (M) in Acid Scrub	0.72	0.25	0.26	0.15
Zr (M) in Acid Scrub	0.060	0.033	0.046	0.022
Al (M) in Acid Scrub	0.10	0.089	0.13	0.056
B (M) in Acid Scrub	0.039	0.031	0.028	0.024
% of F Fed to Calciner Uncomplexed in Acid Scrub (as CaF_2)	5.9	0	0	0
Is F in Acid Scrub Adequately Complexed for Acceptable Corrosion?[a]	no	yes	yes	yes
% of F Fed to Calciner Escaping Acid Scrubber as Gaseous Fluoride[b]	$1.1^{+0.4}_{-0.7}$	$0.22^{+0.09}_{-0.10}$	$0.34^{+0.04}_{-0.08}$	$0.13^{+0.05}_{-0.08}$

a. If the expression $\frac{F-4(Zr + B)}{2\ A\ell}$ (where F, Zr, B, and Aℓ are molar concentrations of these ions in acid scrub) is less than one, the fluoride is adequately complexed to prevent excessive corrosion (< 51 μm/mo or <2 mils/mo).

b. The numbers given represent the average, maximum, and minimum values obtained during a given run.

TABLE V

CHLORIDE BEHAVIOR IN CALCINER OFF GAS DURING TEN-CENTIMETRE CALCINER RUNS

Run #	DZrW4-1	DZrW4-2	DZrW4-3	DZrW4-4
Waste Feed	Dilute Zr Waste	5 vol dilute Zr waste-1 vol Na waste	8 vol dilute Zr Waste-1 vol Na waste	3 vol dilute Zr waste-1 vol Na waste
% of Total in:[a,b]				
Calcine Solids[c]	<33	80^{+6}_{-7}	84^{+5}_{-10}	88 ± 1
Fines	<12.2	30^{+5}_{-6}	22^{+10}_{-7}	11^{+2}_{-4}
Product	<20.6	54 ± 3	61^{+13}_{-19}	77^{+3}_{-2}
Acid Scrub	61	11	7.6	6
Off gas Leaving Acid Scrub[d]	12 ± 2	3 ± 1	3^{+3}_{-2}	2 ± 1

a. Percent of total found in calcine solids, acid scrub, and caustic scrub.

b. The numbers given represent the average, maximum, and minimum values obtained during a given run.

c. Fines plus product.

d. Based on concentration of chloride found in caustic scrub solution located downstream of acid scrubber.

1.4 Mercury Volatility

Average mercury retained in calcine solids (product plus fines) in Run DZrW4-1 amounted to 0.4% of the mercury fed to the calciner (Table VI). The average mercury material balance (total of mercury found in calciner streams divided by mercury fed to calciner times 100) was 47%. It was postulated that mercury and chloride escape from the WCF calciner vessel as a volatile mercury-chloride compound. If it was assumed that all mercury and chloride leaving the calciner were present as a volatile mercury-chloride compound during calcination of the dilute Zr waste in Run DZrW4-1, the compound would contain roughly 2 moles of chloride for each mole of mercury present.

2. THIRTY-Cm CALCINER STUDY (RUN 74)

2.1 Fluidized Bed Operation and Properties of Solids Generated

The attrition index (hardness) of calcine product generated during Run 74 reached a constant value of 6 to 10% (Figure 2). This calcine is soft and has about the same hardness as product produced from the calcination of zirconium waste more concentrated in aluminum, zirconium, and fluoride (having a Ca/F mole ratio of 0.55) in the same calciner at 500°C (such as the WM-185 waste shown in Table I). The particle size of product does not have a tendency to become too large when soft calcine product is produced. The product MMPD was maintained at about 0.47 mm for the first 56h of the run using a waste NAR of roughly 147; the waste NAR was increased from about 147 to 225 at COT 60h and the MMPD decreased from about 0.47 to 0.35 mm between COT 60 and 96h; at COT 96h the waste NAR was decreased to about 147 and the MMPD seemed to be increasing again (Figure 3).

The product-to-fines ratio ($\sim$2.5) (Figure 4) obtained during calcination of the dilute Zr waste in the 30-cm calciner seems to be about the same as that during the calcination of Zr waste more concentrated in zirconium, aluminum, and fluoride in the same calciner. The average production rate of product for Run 74 was 3.0 kg/h (Figure 5). The average production rate of three different runs calcining wastes similar to the WM-185 waste of Table I at 500°C under conditions similar to those of Run 74 (feed rates corrected to the rate used in Run 74) was 5.5 kg/h. Thus there was agreement between Run 74 and DZrW4-1 in that calcination of the dilute Zr waste gives a product rate less than what might be expected during calcination of the type of Zr waste fed to WCF previous to its ninth processing campaign. On the other hand, Run 74 gave a product-to-fines ratio that might be expected when calcining a Zr waste similar to those calcined in the WCF previous to its ninth campaign while DZrW4-1 gave a lower than normal product-to-fines ratio. The product rate observed in Runs 74 and DZrW4-1 could mean that during calcination of the dilute Zr waste in the WCF there would be problems building and maintaining bed level.

The bulk density of product produced during Run 74 (1.4 g/cm^3 - Figure 6) was higher than that normally obtained (1.2 g/cm^3) for product produced when Zr wastes more concentrated in aluminum, zirconium, and fluoride are calcined in the same calciner. The bulk density of fines (0.54 g/cm^3) and product particle density (2.4 g/cm^3) produced during Run 74 (Figure 6) were about the same as realized when calcining wastes similar to WM-185 (Table I).

TABLE VI

MERCURY BEHAVIOR IN CALCINER OFF GAS DURING
TEN-CENTIMETRE CALCINER RUNS

Run #	DZrW4-1	DZrW4-2	DZrW4-3	DZrW4-4
Waste Feed	Dilute Zr Waste	5 vol dilute Zr waste-1 vol Na waste	8 vol dilute Zr Waste-1 vol Na waste	3 vol dilute Zr waste-1 vol Na waste
% of Mercury Fed to Calciner in:				
Calcine Solids[a,b]	$0.40^{+0.13}_{-0.14}$	0.13 ± 0.01	$0.10^{+0.02}_{-0.01}$	17 ± 0.03
Fines[a]	$0.34^{+0.12}_{-0.06}$	$0.12^{+0.04}_{-0.02}$	$0.08^{+0.01}_{-0.03}$	$0.15^{+0.02}_{-0.03}$
Product[a]	0.06 ± 0.04	0.01 ± 0.002	$0.02^{+0.01}_{-0.002}$	0.1 ± 0.002
Acid Scrub	44	23	17	19
Off gas Leaving Acid Scrub[a,c]	$3.1^{+5.1}_{-3.0}$	$0.68^{+0.17}_{-0.16}$	$0.77^{+0.93}_{-0.49}$	$3.6^{+1.3}_{-2.1}$
% of Mercury Accounted for	47 ± 4	24^{+1}_{-0}	18 ± 1	22 ± 2

a. The numbers given represent the average, maximum, and minimum values obtained during a given run.

b. Fines plus product.

c. Based on concentration of mercury found in caustic scrub solution.

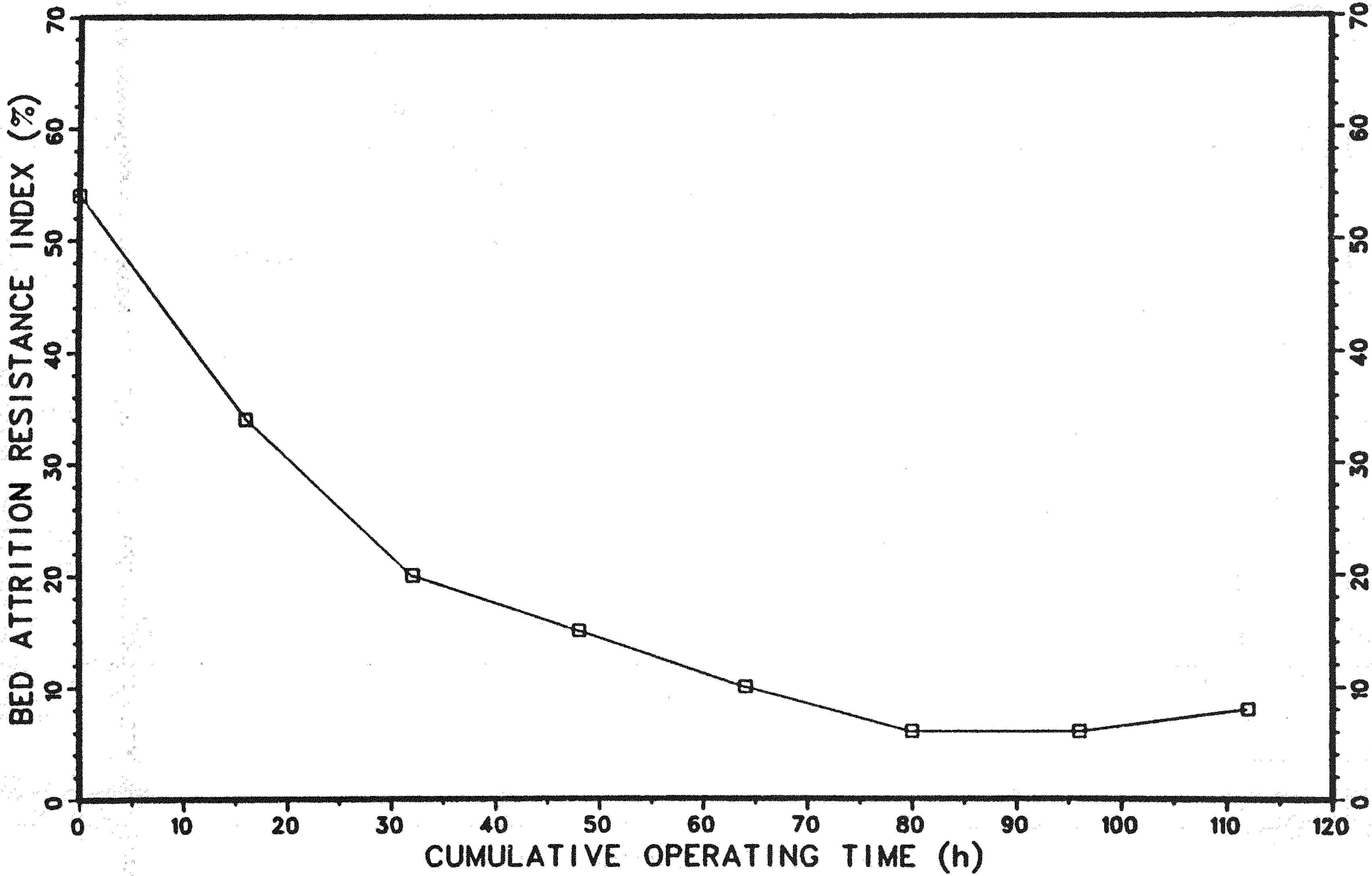


FIGURE 2. ATTRITION INDEX OF BED PRODUCED DURING RUN 74

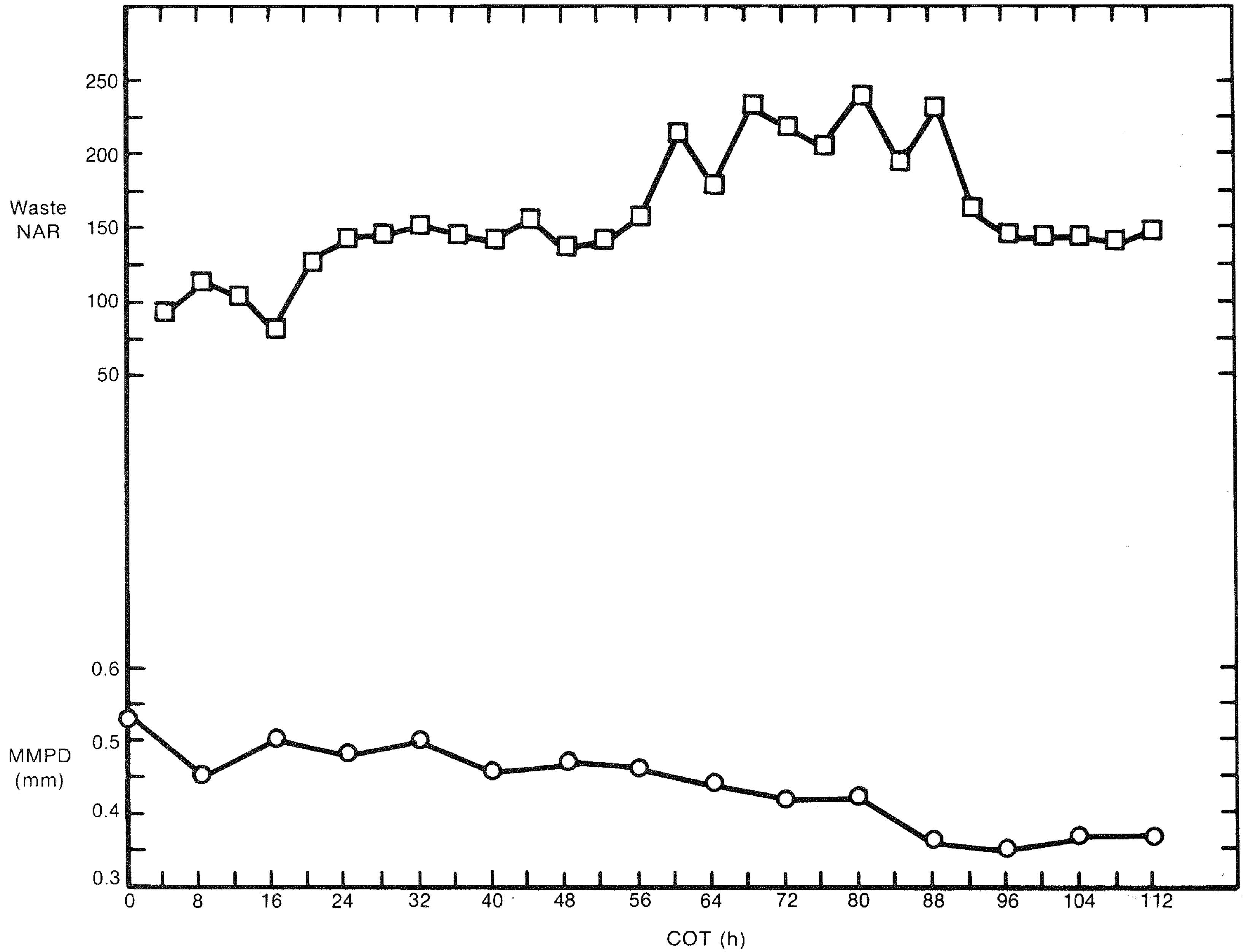


Figure 3. Variation of MMPD with Waste NAR during Run 74

ICPP-A-5089

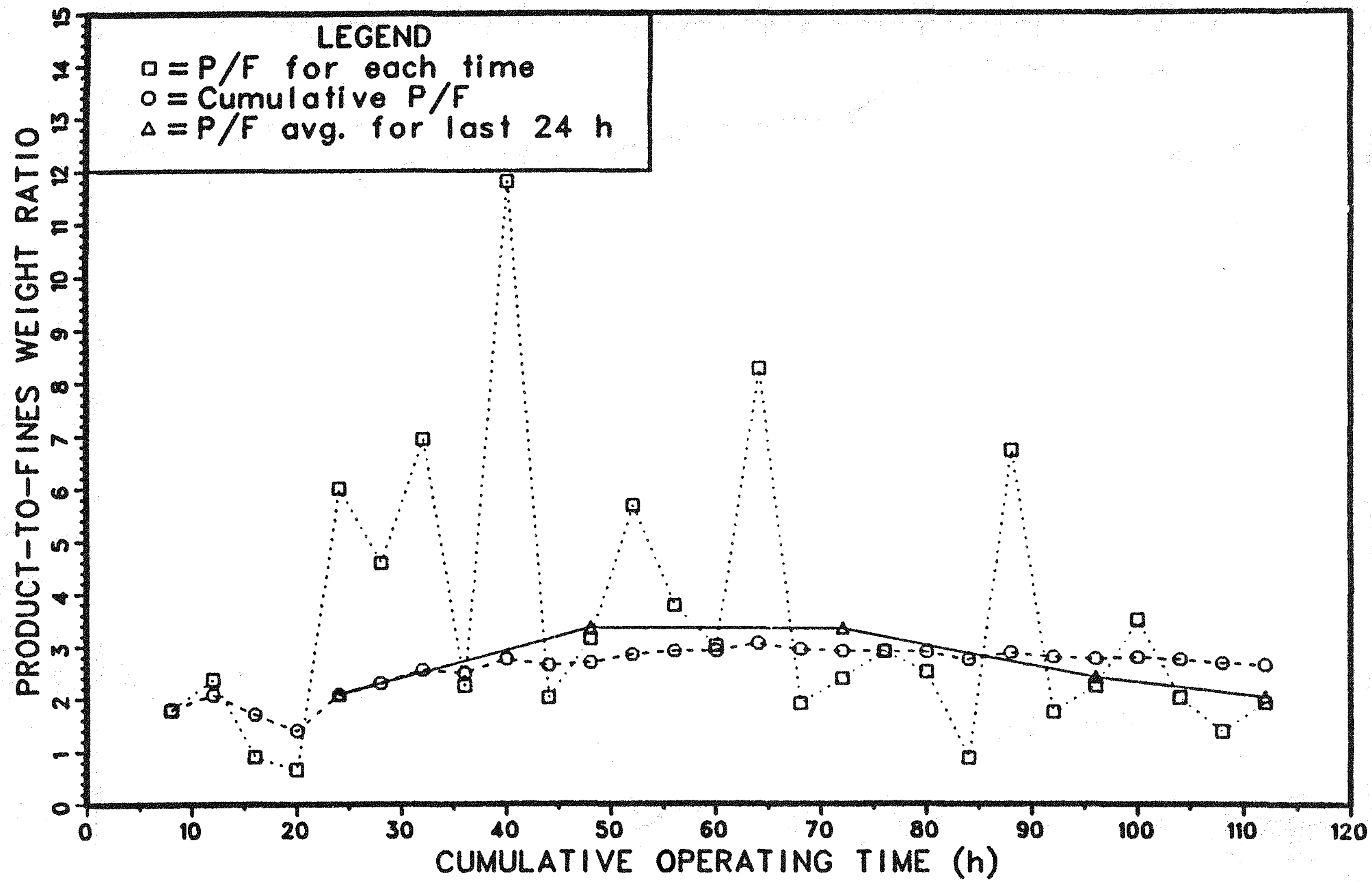


FIGURE 4. VARIATION OF PRODUCT-TO-FINES RATIO DURING RUN 74

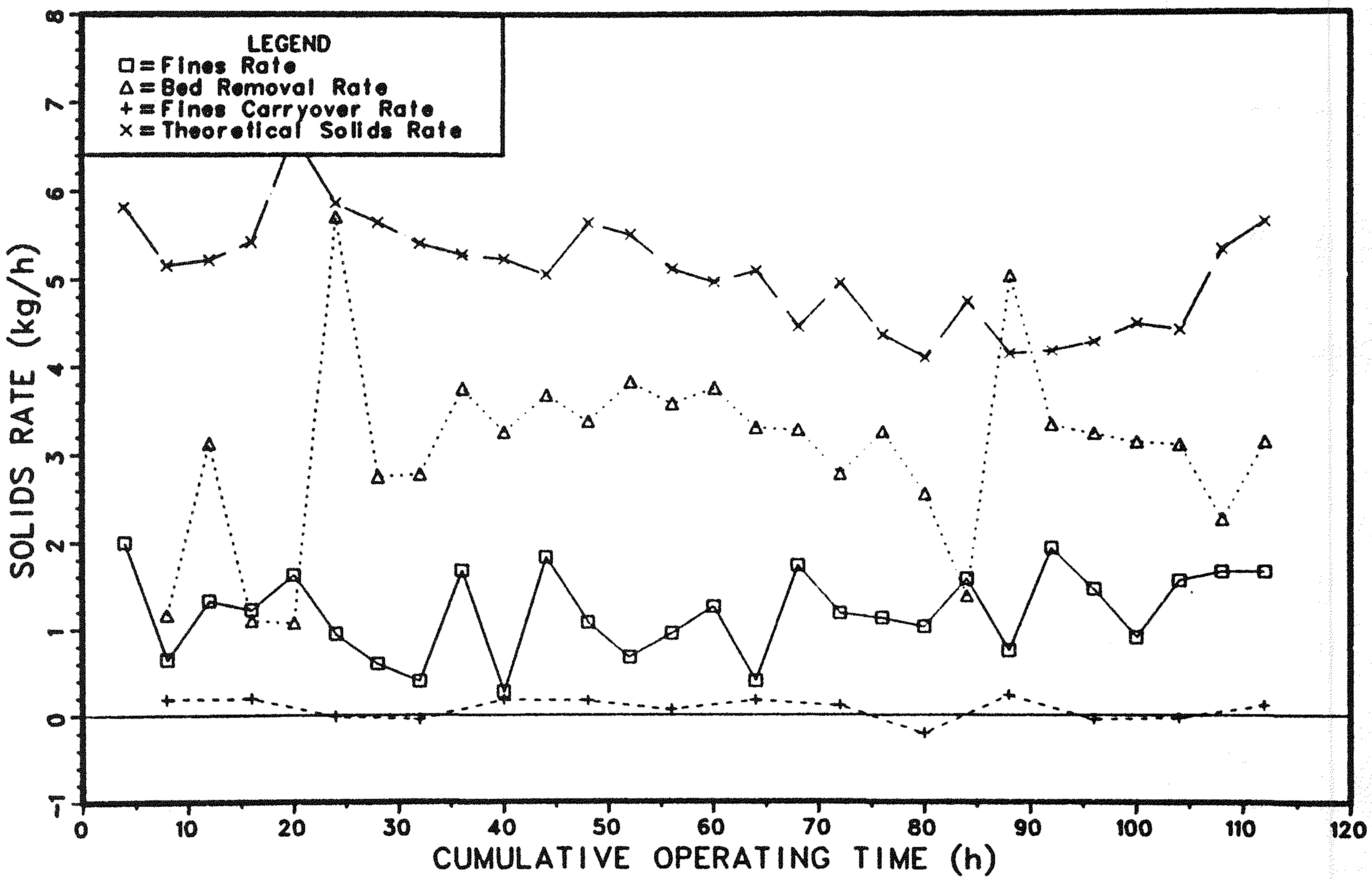


FIGURE 5. SOLIDS GENERATION DURING RUN 74

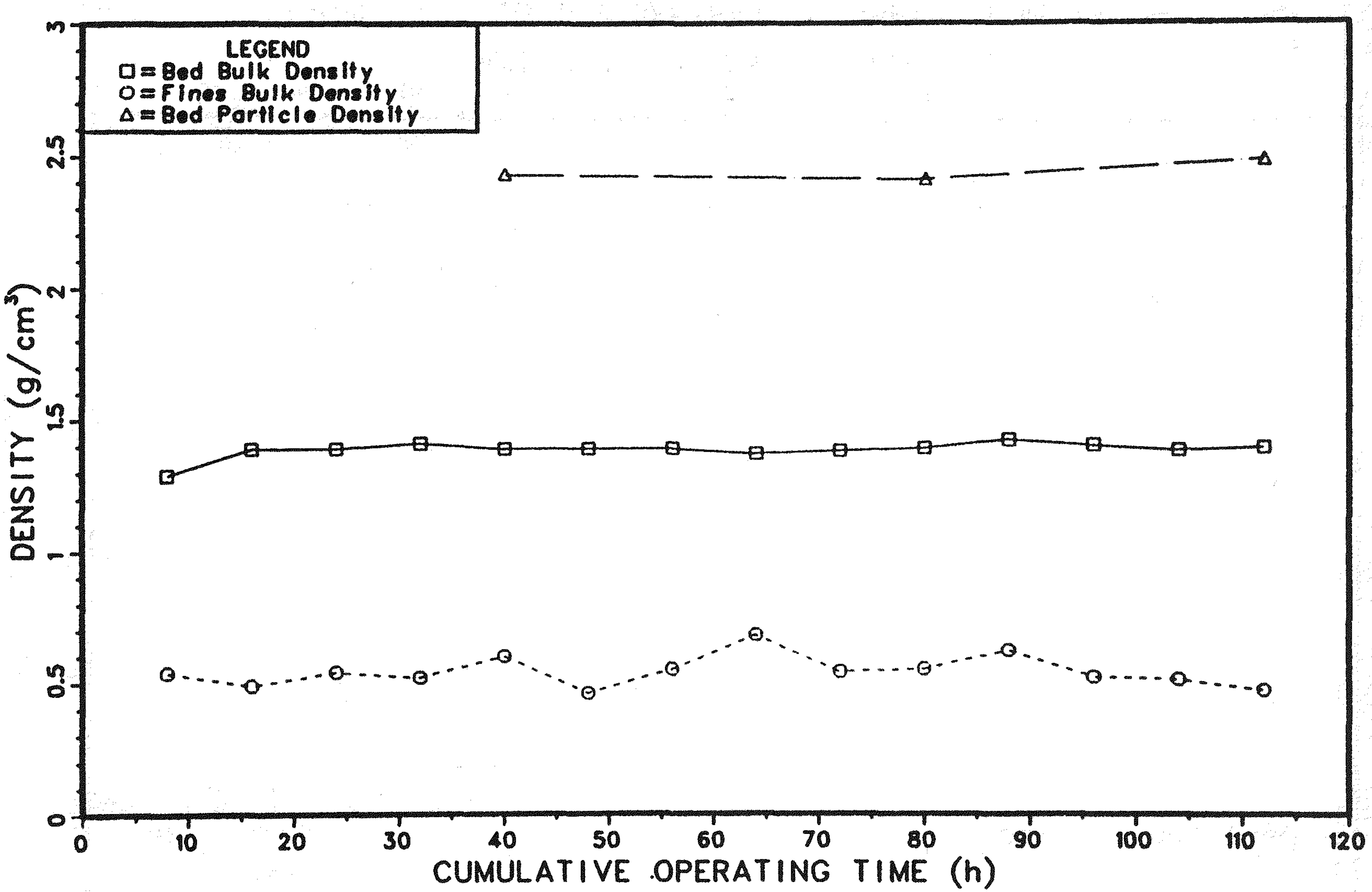


FIGURE 6. DENSITY OF SOLIDS PRODUCED DURING RUN 74

Pictures of product (taken at different times during the run) magnified 15x showed that the calcine was smooth and contained no nodules (particles in the upper photo of Figure 1 are from Run 74). Very little bed agglomeration occurred during the run. Detailed information on the operation of the fluidized bed and properties of solids generated is found in Appendix G. Final bed information not found elsewhere is summarized in Table VII.

Corrosion rates obtained from coupons present in the off-gas system are summarized in Table VIII. Corrosion rates were acceptable but were higher than in a 30-cm calciner run using, as calciner feed, a blend of 5 vol of the dilute Zr waste and 1 vol Na waste (Run 71, described later in the report). Corrosion rates in Run 74 are probably higher because more volatile chloride and fluoride was in the off gas of Run 74 than in the off gas of Run 71.

2.2 Fluoride Volatility

The composition of the off-gas acid scrub solution during Run 74 is given in Table IX. The distribution of fluoride, chloride, sulfate, and mercury between the product plus bed, fines, acid scrub, and off gas escaping the acid scrub (based on volatiles found in one caustic scrubber) for four different periods of operation is summarized in Table X. The composition of feed, product, fines, and caustic scrub is tabulated in Appendix H.

During calcination of the dilute Zr waste in Run 74, there was always fluoride in the off-gas acid scrub solution in excess of that which could be combined with the calcium present as CaF_2 (Table IX). Thus, fluoride was present in the off gas in a form different than CaF_2 and was being sorbed in the acid scrub solution. The zirconium, aluminum, and boron in the acid scrub solution were always in sufficient quantities to complex the fluoride present and to prevent excessive corrosion to off-gas acid scrub equipment. The volatile fluoride present in the off gas in a form other than CaF_2 varied between 0.4 and 1.8% of that fed to the calciner, averaging 1.1%. This is higher than the maximum 0.8% value obtained during the calcination of zirconium wastes similar to the WM-185 waste (Table I) and blends of this waste with Na waste (using calcium and/or magnesium nitrate to suppress fluoride volatility) in 10- and 30-cm calciners at 500°C.[2,4] Based on a model developed at the ICPP, the corrosion rate of 304L stainless steel at70°C in the acid scrub solutions produced during Run 74 would be roughly 36 µm/mo(1.4 mils/mo).[3] This corrosion rate is high but acceptable.

During one 8-h operating period, a second caustic scrubber was put in series with the one caustic scrubber normally used. Only 2% of the fluoride entering the first scrubber escaped. Thus, one caustic scrubber containing 1000 mL of 6M sodium hydroxide removes essentially all the fluoride from the off gas passing through it (0.3% of the total) during an 8-h period.

TABLE VII

FINAL BED INFORMATION IN RUNS 71 AND 74

Weight of Final Bed (kg)		Weight Percent of Bed >14 Mesh Particles		Weight Percent of Bed >1/2-inch Diameter Agglomerates	
Run 71	Run 74	Run 71	Run 74	Run 71	Run 74
91.7	80.5	0.63	0.81	0.28	0.22

2.3 Chloride Volatility

The chloride content of the feed used in Run 74 averaged 332 ppm. This is about 100 ppm greater than the dilute Zr waste stored in Tanks WM-188 and -189 (Table I). The chloride content of the off-gas acid scrub increased continuously during Run 74 until the concentration was approaching 2600 ppm by the end of 112h of operation (Table IX). Chloride concentration in the acid scrub solution should not exceed 2600 ppm in order to prevent excessive corrosion to the off-gas acid scrub system.[5] The chloride concentration of off-gas acid scrub solution will eventually reach a constant value depending upon the type of waste being fed to the calciner and the chloride concentration of that feed.[4] If the chloride value of acid scrub in Run 74 would reach 2600 ppm in a little greater than 112h of operation when calcining the dilute Zr waste containing 300 ppm chloride, it is probable that the acid scrub of the WCF when calcining dilute Zr waste containing 200 ppm chloride, would reach a constant chloride concentration greater than 2600 ppm.

Chloride retention in calcine solids (bed plus product plus fines) was very poor, averaging 20% (of the total chloride found in the calciner system) during the last 80h of the run (Table X). Calcination of the dilute Zr waste (Ca/F mole ratio of 0.55) in a 10-cm calciner also gave poor chloride retention in calcine solids (<33%).

Passing off gas through two caustic scrubbers in series during one 8-h operating period showed that only 3% of the chloride in the off gas entering the first scrubber was present in the off gas leaving that scrubber.

2.4 Mercury Volatility

Mercury retained in calcine solids amounted to <0.2% of the total mercury found in the calciner system (Table X). Very low mercury retention in calcine solids was observed during calcination of zirconium wastes similar to WM-185 waste of Table I and of blends of these wastes with Na waste.[2,4]

TABLE VIII

CORROSION OF OFF-GAS, STAINLESS STEEL 304L COUPONS DURING RUNS 71 AND 74

Coupon Position	Corrosion Rate		Type of Corrosion	
	Run 71	Run 74	Run 71	Run 74
Top of Condenser	(a)	2.84 μm/mo (0.11 mils/mo)	(b)	(b)
Scrub Tank Pump Exit	0.20 μm/mo (0.01 mils/mo)	11.79 μm/mo (0.46 mils/mo)	(b)	(b)
Quench Tower	2.35 μm/mo (0.09 mils/mo)	1.12 μm/mo (0.04 mils/mo)	(b) (c)	(b)
Bottom Gas Outlet of Condenser	0.12 μm/mo (0.004 mils/mo)	0.27 μm/mo (0.01 mils/mo)	(b)	(b)
Bottom of Knock-out Pot	0.68 μm/mo (0.03 mils/mo)	4.98 μm/mo (0.20 mils/mo)	(e) (d)	(b) (f)
Top of Knock-out Pot	1.66 μm/mo (0.07 mils/mo)	1.27 μm/mo (0.05 mils/mo)	(b) (d) (f) (g)	(b) (f)

(a) Threads on coupon broken during removal from calciner making corrosion rate determination impossible.

(b) Light general attack.

(c) Coupon contains stain not removed by ultrasonically cleaning in water and acetone plus boiling in 3 M nitric acid for 5 minutes.

(d) Coupon contains stain partially removed by ultrasonically cleaning in water and acetone plus boiling in 3 M nitric acid for 5 minutes.

(e) Light general attack with slight weld decay.

(f) Slight intergranular attack of weld metal.

(g) Exhibits shinny spot over weld area.

TABLE IX

COMPOSITION OF ACID SCRUB DURING RUN 74

COT (h)	F	Zr	Ca	B	Aℓ	NO_3	H^+	Cℓ	SO_4	Hg
	(M)							(ppm)		
0	0.08	(a)	(a)	(a)	(a)	(a)	(a)	25	40	125
32	0.17	0.021	0.078	0.021	0.056	2.40	2.26	527	630	1512
56	0.45	0.041	0.132	0.051	0.119	2.64	2.00	1230	1580	3889
80	0.81	0.062	0.325	0.072	0.152	2.83	1.76	2070	2480	5452
112	0.97	0.060	0.425	0.092	0.193	2.24	1.10	2540	3110	6910

(a) Not Analyzed

TABLE X

BEHAVIOR OF VOLATILES IN RUN 74

% of Total Volatile in (e):	Cℓ				Hg				F				SO_4			
Operating Period:	(a)	(b)	(c)	(d)	(a)	(b)	(c)	(d)	(a)	(b)	(c)	(d)	(a)	(b)	(c)	(d)
Fines	37.7	9.4	7.0	6.0	0.05	0.01	0.03	0.03	24.5	24.9	29.7	33.7	26.2	23.1	30.2	27.4
Product + Bed	9.4	6.4	15.7	13.6	0.08	0.05	0.12	0.02	73.5	69.7	65.2	62.6	71.5	72.9	66.5	69.5
Acid Scrub	26.0	57.4	36.4	24.1	75.8	89.4	63.3	64.4	1.3	4.7	4.2	2.4	1.6	3.4	2.2	1.3
Off Gas Escaping Acid Scrub (f)	26.9	26.8	40.9	56.3	24.1	10.6	36.6	35.5	0.75	0.71	0.97	1.3	0.69	0.68	1.1	1.8
% of Volatile in Acid Scrub which came from Fines (g)	4.2	0.91	0.45	0.14	0.006	0.001	0.002	0.0006	2.4	2.8	1.8	0.76	2.6	2.6	1.8	0.62
% of Total Volatile in Off Gas Escaping Acid Scrub coming from Fines (h)	0.096	0.095	0.11	0.078	0.0002	0.00009	0.0004	0.0003	0.20	0.19	0.30	0.43	0.18	0.18	0.28	0.36
% of Total Volatile Input Accounted for:	59.8	58.2	59.8	81.1	51.4	120	43.9	74.6	73.7	79.1	72.8	83.7	70.6	66.5	63.4	91.4

(a) The operating period covering the first 32h of the run based on analyses of acid scrub sample taken at COT 32h.
(b) The operating period between COT 32 and 56h of the run based on analyses of acid scrub sample taken at COT 56h.
(c) The operating period between COT 56 and 80h of the run based on analyses of acid scrub sample taken at COT 80h.
(d) The operating period between COT 80 and 112h of the run based on analyses of acid scrub sample taken at COT 112h.
(e) Assumes total concentration of volatile = conc. found in fines + conc. found in product and bed + conc. found in acid scrub + conc. found in caustic scrub.
(f) Based on component collected in a single caustic scrubber containing 6 M NaOH.
(g) Based on fines carryover and the concentration of volatile in the fines removed by the primary cyclone.
(h) Based on calcium in the caustic scrubber and the ratio of calcium to volatile in the fines removed by the primary cyclone.

Mercury material balances during calcination of zirconium wastes and blends of zirconium and Na wastes in 10- and 30-cm calciners generally have been poor.[2,4] The poor material balances had been assumed to be caused by sodium hydroxide being an inefficient mercury absorber, allowing most of the mercury in the off gas that had escaped the acid scrubber to pass through the caustic scrubber. During one 8-h operating period in Run 74, a hydrochloric acid scrubber (a good mercury absorber) was placed in series with and downstream from the caustic scrubber to determine how much mercury was escaping from the one caustic scrubber normally used. The average mercury material balance (not taking into account the 120% material balance obtained operating between COT 32 and 56h) in Run 74 using a single caustic scrubber was 57%. If a caustic scrubber and hydrochloric acid scrubber in series had been used, the average material balance would have been 64%. Thus mercury escaping the caustic scrubber does not explain the poor mercury material balances obtained.

If it is assumed that all mercury and chloride leaving the calciner was present as a volatile mercury-chloride compound during calcination of the dilute Zr waste in Run 74, the compound would contain roughly 4 moles of chloride per mole of mercury present. During Run 74, the mercury concentration in the off-gas acid scrub increased continually as the run progressed.

2.5 Sulfate Volatility

The sulfate retention in calcine solids was excellent, averaging 97% of the total found in the calciner system. The maximum sulfate concentration present in the acid scrub (3g/L) should not be enough to cause excessive corrosion. Passing off gas through 2 caustic scrubbers in series during one 8-h operating period showed that only 2% of the sulfate in the off gas entering the first scrubber was present in the off gas leaving that scrubber.

IV. CALCINATION OF BLENDS OF DILUTE ZIRCONIUM WASTE AND SODIUM WASTE

1. TEN-Cm CALCINER STUDIES (RUNS DZrW4-2, -3, and -4

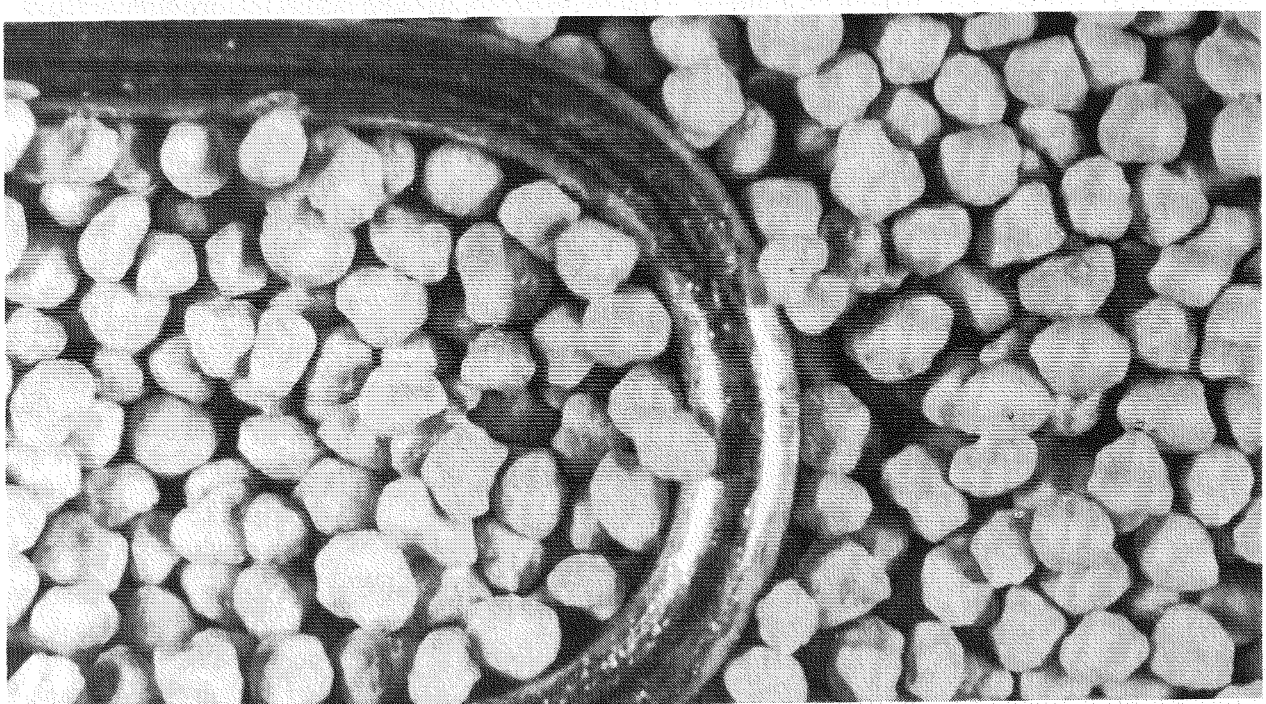
Forty-hour runs were made to test the calcination of blends of 1 vol Na waste with 3, 5, and 8 vol of the dilute Zr waste (Runs DZrW4-4, DZrW4-2, and DZrW4-3, respectively). The Ca/F mole ratio in the runs was 0.7. A high NAR ($\sim$1000) was used at the start of each run, and was adjusted to control product particle size. All runs were terminated voluntarily after 40h; excessive clinker formation did not take place during any of the runs. At the end of each run there were normal amounts of agglomerates inside the calciner stuck to calciner surfaces, nozzles, the propane torch, and thermocouple sheaths. Also in all runs, the fines seemed to be sticky, causing primary cyclone plugging; the fines did not seem to be moist: they flowed out of the fines pot when it was given a sharp rap.

1.1 Fluidized-Bed Operation and Properties of Solids Generated

Photographs were taken of magnified, final bed material. Figure 7 shows particles typical of calcining blends of 5 and 8 vol dilute Zr waste and 1 vol Na waste. The figure also shows magnified calcine particles obtained by calcining at 500^{o}C a blend of 5 vol Zr waste similar to WM-185 waste of Table I and 1 vol of Na waste. The surfaces of calcine from the blend containing the dilute Zr waste is similar in appearance to surfaces of calcine obtained from the blend containing Zr waste more concentrated in zirconium, aluminum, and fluoride. Final bed material produced during the calcination of blends of 5 and 8 vol dilute Zr waste with 1 vol Na waste contained a few nodules and was slightly rougher than material produced from calcining unblended Zr wastes (Figure 1). Particles from 5 and 8 to 1 blends should give good quality fluidized-bed operation. Final bed material produced from the calcination of the blend of 3 vol dilute Zr waste with 1 vol Na waste contained markedly more nodules than material produced during calcination of the other two blends but the non-noduled surfaces did not seem to be any rougher.

Table III gives average values for the more important properties of fluidized-bed operation and solids generated for Runs DZrW4-2, -3, and -4; more detailed information is given in Appendix A. Product produced by the calcination of the three blends had roughly the same bulk density as that produced by calcining blends of 3 to 8 vol WM-185 waste with 1 vol Na waste and the same hardness as that produced by calcining blends of 2, 3, and 4 vol WM-185 waste with 1 vol Na waste in the same calciner under about the same conditions.[2] The attrition index and bulk density of the calcine product produced by the calcination of blends of 3 to 8 vol dilute Zr waste with 1 vol Na waste seemed to be independent of the blend ratio; the product-to-fines ratio seemed to increase as the blend ratio decreased. Calcination of the 3 to 1 blend for 40h resulted in a final bed containing markedly more agglomerates than the final beds from the 5- and 8-to-1 blends (assumes particles in final bed that are greater than 14 mesh are agglomerates).

From Blend of 5 Vol Dilute Zr Waste - 1 Vol Na Waste; Ca/F Mole Ratio of 0.7



From Blend of 5 Vol Waste similar to WM-185-1 Vol Na Waste; Ca/F Mole Ratio of 0.7

Figure 7. Magnified Calcine Particles of Blended Zr and Na Wastes

1.2 Fluoride Volatility

Fluoride behavior in the off gas was similar during calcination of the 3 different blends. In all these runs, the amount of calcium in all acid scrubbing solution samples taken was enough to combine with all the fluoride present in the same samples to produce CaF_2 (Table IV). Thus all the fluoride present in the acid scrub system had probably been entrained as CaF_2 in fines contained in the off gas. The amount of volatile fluoride in the off gas not complexed with the calcium was small, varying between 0.13 and 0.34% of that fed to the calciner for the 3 different runs. Also in all 3 runs there was enough zirconium, aluminum, and boron present in the acid scrubbing solution to complex the fluoride present and thus prevent excessive corrosion (next to last row of Table IV).

1.3 Chloride Volatility

Chloride retention in calcine solids (product plus fines) was excellent (varying between 80 and 88%) in runs using blends of dilute Zr and Na wastes as calciner feed and seemed to be independent of the Na waste content of the blend within the blend ratio range studied in Runs DZrW4-2, -3, and -4 (Table V).

1.4 Mercury Volatility

Mercury retained in calcine solids (product plus fines) in all runs amounted to less than 0.2% of the mercury fed to the calciner (Table VI). If it was assumed that all the mercury and chloride leaving the calciner was present as a volatile mercury-chloride compound during calcination of the blends, the compound would contain roughly 2 moles of mercury for each mole of chloride present.

2. THIRTY-Cm CALCINER STUDY (RUN 71)

A blend of 5 vol dilute Zr waste and 1 vol Na waste (containing a Ca/F mole ratio of 0.7) was calcined for 112 h in Run 71. Detailed data for the run can be found in Appendices B, C, D, and E. Run 71 was voluntarily terminated. Very little bed agglomeration occurred during the run.

2.1 Fluidized-Bed Operation and Properties of Solids Generated

Product particle size was effectively controlled by varying a combination of the waste NAR and the oxygen-to-fuel ratio (Figure 8). Product MMPD increased from 0.41 to 0.60 mm when the waste NAR was roughly 350 and the oxygen-to-fuel ratio was about 1840; at COT 60h, the oxygen-to-fuel ratio was increased to 2000 (the waste NAR was left unchanged) and after a lag of 24h the MMPD decreased steadily.

The product was relatively hard with an attrition index averaging 49%; the product bulk density averaged 1.3 g/cm^3. These compare with an average attrition index and bulk density of 43% and 1.3 g/cm^3, respectively, from product obtained by calcining a blend of 5 vol of a Zr waste similar to WM-185 with

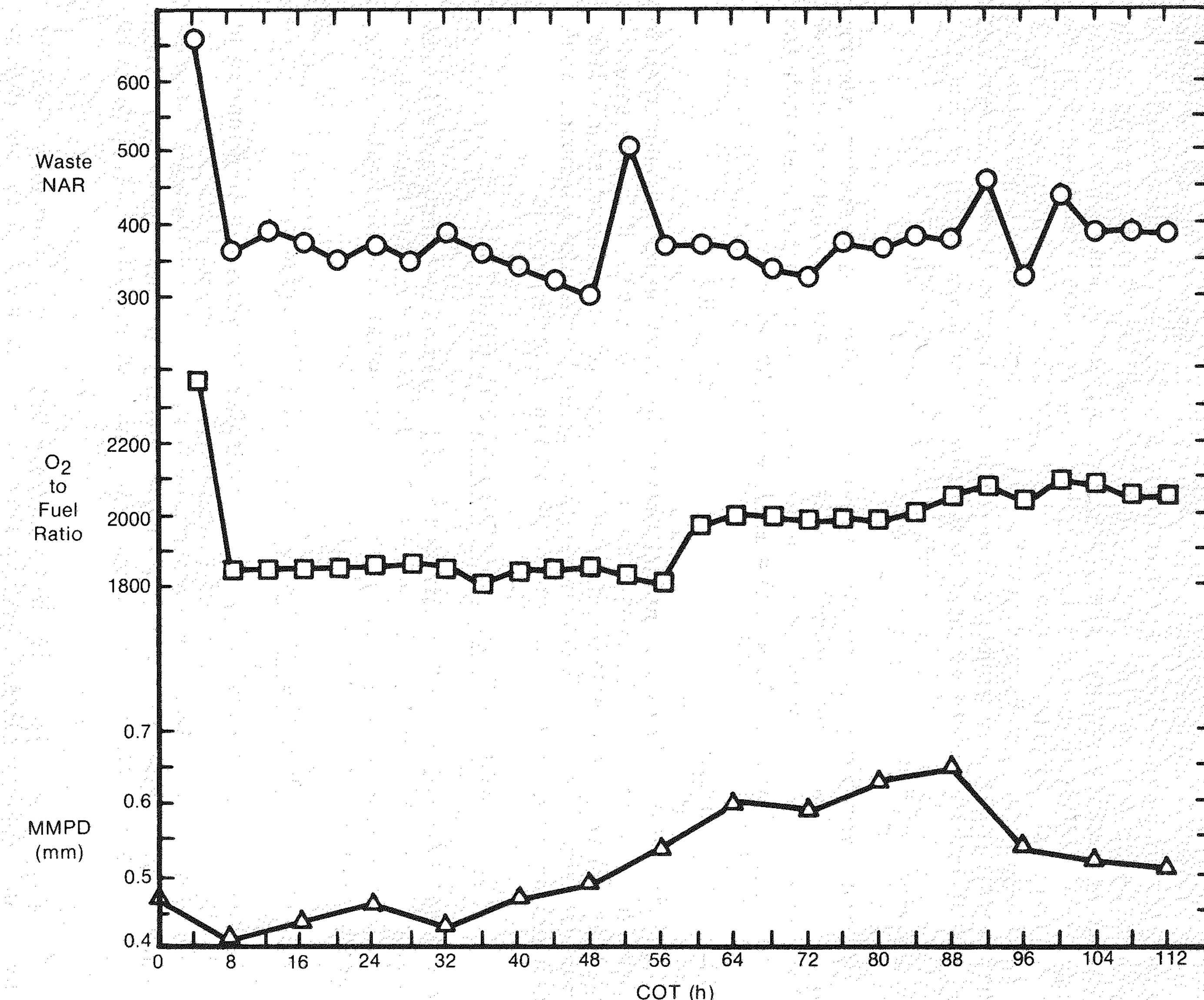


Figure 8. Relationship Between MMPD, Waste NAR, and O_2 to Fuel Ratio during Run 71

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1 vol Na waste at 500°C in the same calciner (Run 67).[2] The product-to-fines ratio for Run 71 averaged 4.5. Information on solids generation during Run 71 is summarized in Figure 9; the figure does not indicate fluidized-bed operation problems. Although the production rate of Run 71 was the same as when uncomplexed dilute Zr waste was calcined in Run 74, unlike Run 74 the product-to-fines ratio was high and the calcine product was hard. The combination of these characteristics should mean that there will be no difficulty in building bed and maintaining bed level when a blend of 5 vol dilute Zr waste and 1 vol Na waste is calcined at 500°C in the WCF.

In Figure 9, the times when the fines generation was low represents times when fines were plugging off-gas lines and/or equipment; sharp increases after these times of low fines generation rate represent times when the plugs were freed and the accumulated fines removed. There was no indication of dampness in the fines produced. Pictures of magnified product showed that the calcine had only a few nodules and was only slightly rougher than calcine produced from the calcination of unblended Zr waste (particles in the upper photo of Figure 7 are from Run 71).

Unlike product produced during calcination of blends of 3 and 5 vol Zr waste similar to WM-185 waste and 1 vol Na waste at 500°C in Run 67[2], the product from Run 71 showed only a slight tendency to set-up into very fragile agglomerates when allowed to cool without agitation.

Final bed information not found elsewhere is summarized in Table VII. Corrosion rates in the off-gas system were acceptable and are summarized in Table VIII.

2.2 Fluoride Volatility

The amount of calcium found in the acid scrub samples taken at COT 88 and 112 h was enough to combine with the fluoride present to form CaF_2, but the amount of calcium in the sample taken at COT 48 h was not enough to combine with the fluoride present to form CaF_2 (Table XI). This means, that during the time represented by the former two acid scrub samples, all the fluoride present in the acid had been CaF_2 in the fines entrained in the off gas, while during the time represented by the latter sample, much of the fluoride in the acid scrub had been in the off gas as a volatile species. The amount of fluoride escaping the acid scrub based on samples taken at COT 48 and 88 h was acceptable (0.4 and 0.3%, respectively, of the total fluoride present in the calciner system), while the fluoride escaping the acid scrub based on samples taken at COT 112h was higher than normal (1.2% of which only 0.05% was entrained in fines — Table XII). The higher fluoride volatility based on samples taken at COT 112h was probably due to the presence of insufficient calcium nitrate in the feed batch calcined between COT 108 and 112h (Table D-1 of Appendix D shows that this feed contained a calcium-to-fluoride mole ratio of only 0.547). In all acid scrub samples analyzed, there was enough zirconium, aluminum, and boron present to complex the fluoride and prevent excessive corrosion. During one 8-h operating period, a second caustic scrubber was put in service with the one caustic scrubber normally used. The fluoride found in the second caustic scrubber after this 8-h operating period was below analytical detection limits.

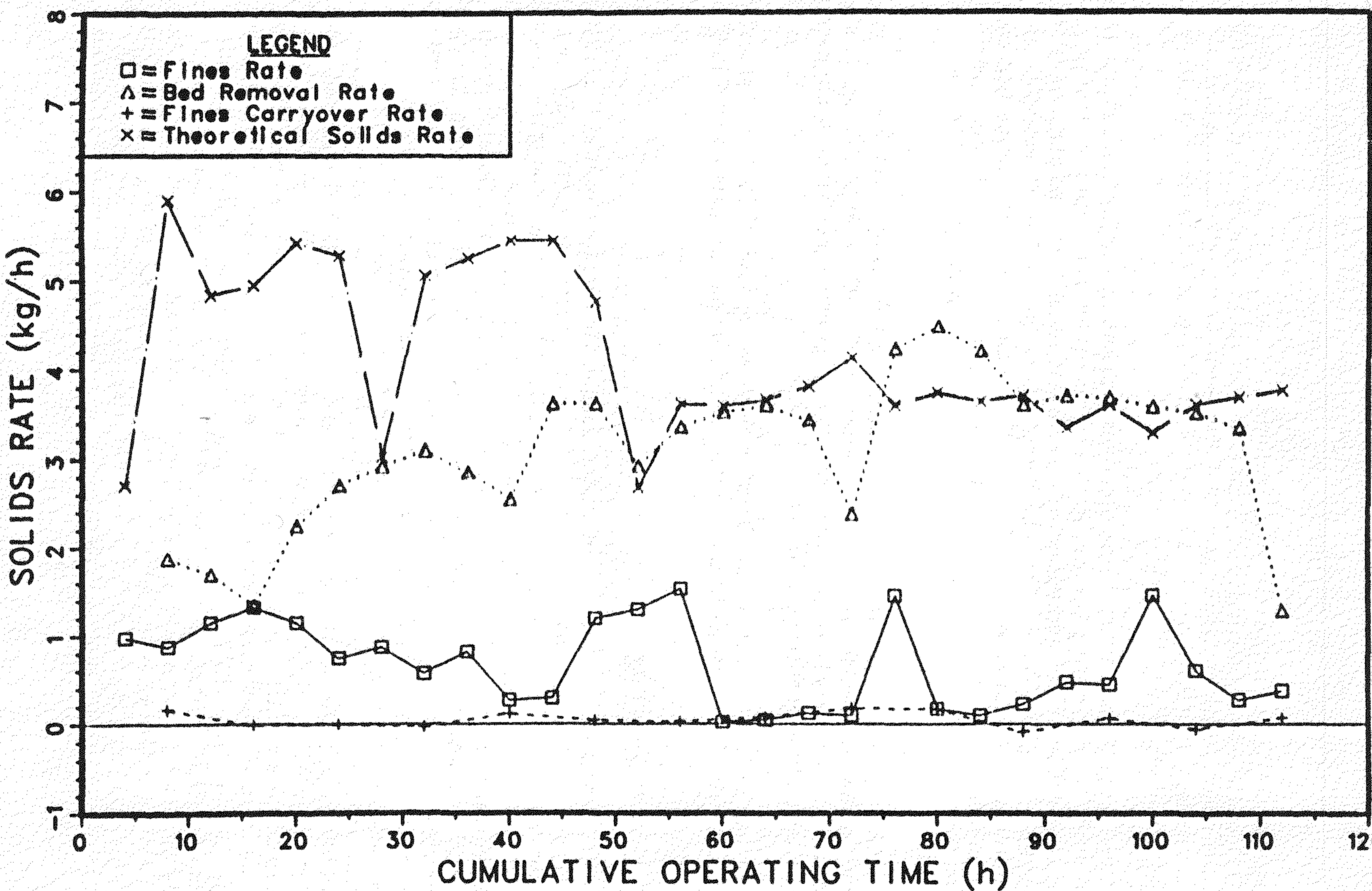

FIGURE 9. SOLIDS GENERATION DURING RUN 71

TABLE XI

COMPOSITION OF ACID SCRUB DURING RUN 71

COT (h)	F	Na	Zr	Ca	B	Aℓ	H	NO_3	Cℓ	SO_4	Hg
	(M)								(ppm)		
0	<10.8 ppm	(a)	(a)	(a)	(a)	(a)	(a)	(a)	16	19	0.3
48	0.12	0.081	0.011	0.025	0.032	0.044	1.18	2.00	372	1,100	763
88	0.20	0.139	0.015	0.100	0.030	0.074	1.60	2.89	553	1,470	1375
112	0.24	0.147	0.014	0.152	0.035	0.070	0.69	2.17	644	1,660	1224

(a) Not analyzed

TABLE XII

BEHAVIOR OF VOLATILES IN RUN 71

Operating Period:	Cℓ (a)	Cℓ (b)	Cℓ (c)	Hg (a)	Hg (b)	Hg (c)	Na (a)	Na (b)	Na (c)	F (a)	F (b)	F (c)	SO_4 (a)	SO_4 (b)	SO_4 (c)
% of Total Volatile in (d):															
Fines	28.2	17.4	22.1	0.3	0.2	0.5	16.8	14.5	18.6	18.4	9.0	12.7	14.4	13.8	17.8
Product + Bed	53.2	59.7	62.9	<0.1	<0.1	<0.1	78.5	82.1	78.5	78.5	89.1	82.8	81.5	83.8	79.4
Acid Scrub	9.6	4.5	9.4	74.0	50.0	36.1	4.7	3.4	2.9	2.6	1.6	2.5	3.1	1.5	1.7
Off Gas Escaping Acid Scrub (e)	9.0	18.4	5.7	25.7	49.8	63.4	-	-	-	0.45	0.33	2.0	1.0	1.0	1.1
% of Volatile in Acid Scrub which came from Fines (f):	19.8	2.9	0.25	0.02	0.04	0.005	1.2	2.5	0.22	1.3	1.5	0.14	0.98	2.5	0.20
% of Volatile in Off Gas Escaping Acid Scrub coming from Fines (g):	0.22	0.21	0.09	0.002	0.002	0.002	-	-	-	0.14	0.11	0.05	0.11	0.16	0.07
% of Total Volatile Input Accounted For:	132	89.2	77.0	28.7	25.6	11.5	65.5	81.7	80.7	78.1	81.6	60.8	64.9	70.2	77.0

(a) The operating period covering the first 48h of the run based on analyses of acid scrub sample taken at COT 48h.
(b) The operating period between COT 48 and 88h of the run based on analyses of acid scrub sample taken at COT 88h.
(c) The operating period between COT 88 and 112h of the run based on analyses of acid scrub sample taken at COT 112h.
(d) Assumes total concentration of volatile = conc. found in fines + conc. found in product and bed + conc. found in acid scrub + conc. found in caustic scrub.
(e) Based on component collected in a single caustic scrubber containing 6 M NaOH.
(f) Based on the fines carry-over and the concentration of volatile in the fines removed by the primary cyclone.
(g) Based on calcium in the caustic scrubber and the ratio of calcium to volatile in the fines removed by the primary cyclone.

2.3 Chloride Volatility

The chloride content of the blended feed used in Run 71 averaged 450 ppm. The chloride content of the acid scrub gradually increased from 16 to 644 ppm during the run (Table XI). This may have been a reflection of the fact that for some reason the chloride concentration of the last batch of feed used was greater than for the batch of feed used midway through the run, which, in turn, was greater than for the initial batch used (Table D-1 of Appendix D). The percent chloride retained in calcine solids (bed plus product plus fines) during Run 71 was excellent, averaging 81% (Table XII).

Passing off gas through 2 caustic scrubbers in series during one 8-h operating period showed that only 3% of the chloride in the off gas entering the first scrubber was present in the off gas leaving that scrubber.

2.4 Mercury Volatility

Mercury retained in calcine solids amounted to 0.5% or less of the total mercury found in the calciner system (Table XII). The material balance for mercury in the calciner system was low (<30% —Table XII). If it was assumed that all mercury and chloride leaving the calciner was present as a volatile mercury-chloride compound during Run 71, the compound would contain roughly 1 mole of mercury for every mole of chloride present.

During an 8-h operating period in Run 71, a hydrochloric acid scrubber was placed in series with and downstream from the caustic scrubber. Use of the hydrochloric acid scrubber increased the mercury material balance by 11%, which still left roughly 60% of the mercury fed to the calciner unaccounted for.

2.5 Sulfate Volatility

The sulfate retention in calcine solids was excellent, averaging 97% of the total found in the calciner system. The sulfate concentration present in the acid scrub (1.4g/L) should not be enough to cause excessive corrosion. The sulfate concentration in the caustic scrubber was always below the detection limit.

V. FLUIDIZED-BED OPERATION AND PROPERTIES OF SOLIDS GENERATED

Unblended WM-185 waste having a Ca/F mole ratio of 0.55 and a blend of 3 1/2 vol of a waste similar to WM-185 waste and 1 vol Na waste having a Ca/F mole ratio of 0.7 have been successfully calcined at 500°C in the WCF. Table XIII tabulates fluidized-bed and calcine solids characteristics that are important for the successful calcination of WM-185 waste, dilute Zr waste, and blends of these wastes with Na waste. If these characteristics are similar for wastes successfully calcined in the WCF and for dilute Zr wastes, then dilute Zr wastes should be calcinable in the WCF.

The only noticeable difference between WM-185 waste and the dilute Zr waste calcined in the same 10-cm calciner is the product-to-fines ratio and product rate for the dilute Zr waste are less than for WM-185 waste. The noticeable difference between these two wastes calcined in the same 30-cm calciner is the product rate for the dilute Zr waste is less than for WM-185 waste. Thus calcination of dilute Zr wastes in the WCF might result in difficulty in building and maintaining bed level. In every case where blends were calcined, the blend containing the dilute Zr waste had a lower product rate than the blend having the same Zr to Na waste blend ratio containing WM-185 waste. However, all blends containing dilute Zr waste have two characteristics important in building bed and maintaining bed level—a high product-to-fines ratio and a high product attrition index. Thus bed-building and maintaining bed level should not be a problem when calcining blends containing dilute Zr waste in the WCF.

When calcining blends containing 3 1/2 vol Zr waste similar to WM-185 waste and 1 vol Na waste in the WCF, controlling particle size was difficult because the product was hard. Removal of bed from the WCF calciner vessel during calcination of the same blend was also a problem because product particles were large and had a high bulk density. These problems will also be present when calcining blends containing dilute Zr waste in the WCF because particles produced from such blends are as hard as and have as high of a bulk density as those produced from blends containing WM-185 waste. The product attrition index seems to decrease for blends containing WM-185 waste as the blend ratio of Zr to Na waste increases from 3 to 1 to 8 to 1 but remains constant for blends containing dilute Zr waste. This could mean that calcine produced in the WCF during calcination of blends containing dilute Zr waste with a high ($\geq$5) Zr to Na waste blend ratio will be harder than calcine product from the comparable blend containing WM-185-type waste.

The surfaces of calcine produced during the calcination of a blend of 3 vol dilute Zr waste and 1 vol Na waste contained numerous nodules. Noduled calcine produces poor quality fluidized-bed operation. Thus the Zr to Na waste blend ratio should be greater than 3 to 1 when calcining blends of dilute Zr waste with Na waste in the WCF.

TABLE XIII

AVERAGE FLUIDIZED-BED OPERATING AND CALCINE SOLIDS CHARACTERISTICS OBTAINED DURING CALCINATION OF Zr WASTES AND BLENDS OF Zr AND Na WASTES [a,b]

Run #	Waste calcined	Product-to-Fines Ratio	Product Rate	Appearance of Calcine Surface	Product Bulk Density (g/cm^3)	Fines Bulk Density (g/cm^3)	Product Attrition Index (%)	Weight of Final Bed >14 mesh (%)
DZrW4-1	the dilute Zr waste	1.4	111g/h	smooth, no nodules	1.36	0.61	20	2.1
FV4-1	WM-185 waste	2.8	226g/h	smooth, no nodules	1.22	0.54	28	3.0[c]
DZrW4-4	3 vol dilute Zr waste-1 vol Na Waste	6.5	233g/h	slightly rough, many nodules	1.61	0.98	76	4.0
SBW4-9	3 vol WM-185 waste-1 vol Na Waste	5.6	292g/h	slightly rough, few nodules	1.68	0.65	80	1.1
DZrW4-2	5 vol dilute Zr waste-1 vol Na Waste	4.7	177g/h	slightly rough few nodules	1.64	0.48	82	0.7
SBW4-8	4 1/2 vol WM-185 waste-1 vol Na waste	2.4	274g/h	slightly rough few nodules	1.57	0.50	43	2.1
DZrW4-3	8 vol dilute Zr waste-1 vol Na waste	4.2	182g/h	slightly rough few nodules	1.53	0.51	74	0.4
SBW4-10	8 vol WM-185 waste-1 vol Na waste	4.1	331g/h	slightly rough few nodules	1.59	0.49	39	2.1
Run 74	the dilute Zr waste	2.5	3.0Kg/h	smooth, no nodules	1.39	0.54	8	0.8
Runs 63, 69,73[d]	WM-185 waste	2.8	5.5Kg/h	smooth, no nodules	1.21	0.55	11	0.2e
Run 71	5 vol dilute Zr waste-1 vol Na waste	4.5	3.0Kg/h	slightly rough few nodules	1.34	0.93	49	0.6
Run 67	5 vol Zr waste similar to WM-185 waste-1 vol Na waste	2.4	3.7Kg/h	slightly rough few nodules	1.34	0.84	44	0.8

a. Most of this information which is not found in the appendices of this report can be found in ENICO 1006[2] and 1016[4].
b. Calcination temperature = 500°C; Zr wastes have Ca/F mole ratio of 0.55; blends have Ca/F mole ratio of 0.7; first 8 runs in 10-cm calciner, remaining runs in 30-cm calciner; WM-185 waste is a Zr waste (Table I).
c. This value not available for Run FV4-1; value given is an average from a run calcining WM-185 waste with a Mg/F mole ratio of 0.55 and a run calcining WM-185 waste containing more Aℓ than normal with a Ca/F mole ratio of 0.55.
d. Values are average values from the 3 runs.
e. This value not available for Runs 63, 69, and 73; value given is from Run 65 calcining WM-185 waste with a Ca/F mole ratio of 0.7 (see ENICO - 1016[4]).

VI. BEHAVIOR OF VOLATILES

Zirconium fluoride wastes similar to WM-185 waste with a Ca/F mole ratio of 0.55 have been calcined in the WCF since the second processing campaign without giving unsatisfactory corrosion performance in the off-gas system due to loss of volatiles from the calciner vessel. The same can be said about the 4 1/2 months (between 4/17/78 and 9/29/78) during the eighth processing campaign of the WCF when a blend of 3 1/2 vol of Zr waste similar to WM-185 and 1 vol Na waste with a Ca/F mole ratio of 0.7 was calcined. Wastes simulating the Zr wastes (WM-185 waste) and blends of these wastes with Na waste that have been successfully calcined in the WCF were calcined in 10- and 30-cm calciners and the behavior of volatiles observed. It must be assumed that the behavior of volatiles during calcination of the dilute Zr waste and blends of this waste with Na waste is satisfactory if this behavior is similar to that experienced during calcination flowsheet development for wastes which have since been successfully calcined in the WCF. To facilitate this comparison, Table XIV summarizes fluoride and chloride behavior during calcination of: a) the dilute Zr waste, b) Zr waste more concentrated in aluminum, zirconium, and fluoride (WM-185 waste), and c) blends of these Zr wastes with Na waste.

Fluoride volatility (fluoride in off gas not complexed as CaF_2) from the calciner vessel (fluoride not found in bed plus product plus fines) during pilot-plant calciner runs has been acceptable when it is less than 1% of the fluoride fed to the calciner.[2,4] Acceptable fluoride volatility has also meant that the concentration of zirconium, aluminum, and boron in the acid scrub solution must be sufficient to complex the total fluoride concentration present.[2,4] During calcination of the dilute Zr waste in a 10-cm calciner: a) the fluoride volatility was 7% of that fed to the calciner; b) there was not enough zirconium, aluminum, and boron in the acid scrub to complex the fluoride present; and c) 304 L stainless steel in the acid scrub could have a corrosion rate as high as 127 μm/mo (5 mils/mo.). During calcination of the dilute Zr waste in a 30-cm calciner: a) the fluoride volatility was 2% of that fed to the calciner; b) there was sufficient zirconium, aluminum, and boron present in the acid scrub to complex the fluoride; c) stainless steel 304 L coupons in the off-gas system gave corrosion rates of less than 13 μm/mo (0.5 mils/mo.). During calcination of 3 to 8 vol dilute Zr waste and 1 vol Na waste in 10- and 30-cm calciners: a) the fluoride volatility was 0.5% or less of that fed to the calciner; and b) there was sufficient zirconium, aluminum, and boron present in acid scrub to complex fluoride present. Calcination of blends of 3 to 8 vol dilute Zr waste and 1 vol Na waste at 500°C in the WCF will cause no corrosion problems in the WCF off-gas system due to fluoride volatility. Whether this is true during calcination of the unblended dilute Zr waste in the WCF at 500°C is questionable.

Table XIV shows that: a) chloride volatility during calcination of blends of Zr and Na wastes at 500°C is much less (∼15% of the total found) than chloride volatility during calcination of unblended Zr wastes (>67% of the total found); b) unblended dilute Zr waste calcined at 500°C, has roughly the same chloride volatility as unblended WM-185 waste calcined at 500°C, and c) blends containing dilute Zr waste calcined at 500°C have roughly the same chloride volatility as blends containing WM-185 waste calcined at 500°C.

TABLE XIV

TYPICAL BEHAVIOR OF VOLATILES DURING CALCINATION OF Zr WASTES AND BLENDS OF Zr AND Na WASTES [a,b]

Run #	Waste Calcined	% of Volatile in Bed + Product + Fines		% of Volatile in Off-Gas Acid Scrub		% of Volatile escaping Off-Gas Acid Scrub[c]	
		F[d]	Cℓ[e]	F[f]	Cℓ[e]	F[f]	Cℓ[e]
DZrW4-1	the dilute Zr waste	93	<33	5.9	61	1.1	12
F V4-1	WM-185 waste	98	g	0	g	0.6	g
DZrW4-4	3 vol dilute Zr waste-1 vol Na waste	100	88	0	6	0.2	2
SBW4-9	3 vol WM-185 waste - 1 vol Na waste	99	92	0	6	0.7	<1.4
DZrW4-2	5 vol dilute Zr waste - 1 vol Na waste	100	80	0	11	0.2	3
SBW4-8	4 1/2 vol WM-185 waste - 1 vol Na waste	99	92	0	6	0.5	2
DZrW4-3	8 vol dilute Zr waste - 1 vol Na waste	100	84	0	8	0.3	3
SBW4-10	8 vol WM-185 waste - 1 vol Na waste	100	88	0	12	0.2	<1
Run 74	the dilute Zr waste	96	26	1.5	36	0.5	38
Run 69	WM-185 waste	100	7	0	9	0.1	84
Run 71	5 vol dilute Zr waste - 1 vol Na waste	99	81	0	8	0.5	11
Run 67	5 vol Zr waste similar to WM-185 waste- 1 vol Na waste	100	64	0	7	0.4	3

a. Most of this information which is not found in the appendices of this report can be found in ENICO 1006[2] and 1016[4].
b. Calcination temperature = 500°C; Zr wastes have a Ca/F mole ratio of 0.55; blends have Ca/F mole ratio of 0.7; first 8 runs in 10-cm calciner, remaining runs in 30-cm calciner; WM-185 waste is a Zr waste (Table I).
c. Based on volatile collected in a single caustic scrubber containing 6 M NaOH.
d. 100 - (% of volatile in acid scrub + % of volatile in caustic scrub).
e. Percent of total volatile found in calciner system.
f. Percent of fluoride fed to the calciner not carried over in fines as CaF_2.
g. Value not available.

During the eighth processing campaign of the WCF, a blend of Na and Zr wastes was alternately calcined with unblended Zr waste to control particle size. During the 4 1/2 months that the blend was calcined in this manner, the chloride concentration of the acid scrub approached but remained below 2600 ppm.[a] Chloride concentration of the acid scrub seemed to increase little if at all during calcination of the blend but increased noticeably when the unblended Zr waste was calcined. A model based on WCF chloride volatility experience shows that chloride concentration in the acid scrub reaches a constant value dependent on the initial chloride concentration of the calciner feed and type of feed; this constant value increases as the initial chloride concentration of the feed increases.[4] The Zr wastes calcined in the WCF during the eighth and previous campaigns contained on the order of 10 ppm chloride. If dilute Zr wastes containing approximately 200 ppm chloride are calcined as unblended wastes or are alternately calcined as a blend with Na waste, the chloride concentration in the acid scrub would reach a value greater than 2600 ppm and might do so within weeks.

Mercury volatility from the calciner vessel was extremely high (>99.4% of the mercury fed to the calciner) in all runs described in this report. The mercury material balance in runs calcining blends was poor (17-29%); the mercury material balance in runs calcining unblended dilute Zr waste was better (43-120%). Poor material balances were not caused by poor mercury absorption in the caustic scrubber. High mercury volatility from the calciner vessel has been experienced during calcination of all Zr wastes and blends of these wastes with Na waste in 10- and 30-cm calciner runs wherever mercury volatility has been studied.[2,4] A study on mercury volatility in the WCF during calcination of a blend of Na and Zr wastes showed that most of the mercury escaped the calciner vessel, but of the mercury escaping the calciner vessel: a) only 1.6 wt % got past the acid scrub system, b) only 0.12 wt % got past the silica gel adsorbers, and c) only 0.02 wt % got past the HEPA filters.[4]

Sulfate behavior during calcination at 500°C of dilute Zr waste and blends containing dilute Zr waste was observed for the two 30-cm calciner runs, but not for the 10-cm calciner runs. In both runs about 3% of the total sulfate found would have escaped from the calciner vessel (sulfate not found in bed plus product plus fines). The maximum sulfate concentration present in the acid scrub during the runs (1.4 to 3g/L) should not be enough to cause excessive corrosion.

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a. Chloride concentration of greater than 2600 ppm in the acid scrub causes excessive corrosion to materials of construction in the scrubbing system.[5]

VII. STABILITY OF BLENDS

It is important that a minimum amount of solids separate from solutions while they are stored in waste storage tanks (the solutions should be stable). Lines to remove solution from the storage tanks extend to within 7.6 to 15.2 cm (3 to 6 inches) of the bottom of the tanks. Everytime solution is removed from the tanks for calcination in the WCF, solids on the bottom of the tanks get stirred-up and some are entrained in the solution being removed. The solid content of the solution being withdrawn from the tank becomes greater as the depth of solids on the bottom of the tank increases. These solids can cause plugging problems in the calciner feed system.

Stability studies on solutions containing zirconium, fluoride, aluminum, and nitrate are reported in IDO-14522.[6] Stability studies on blends of WM-185 (Zr) waste and Na wastes are reported in ENICO-1006.[2] No stability studies have been made on blends of dilute Zr waste and Na waste.

Zirconium fluoride solutions, being 0.5M in aluminum, are stable up to a zirconium concentration of 0.8 M and a fluoride concentration of 4.2 M. Aluminum compounds precipitate from zirconium fluoride solutions being 0.5 M in aluminum when the acidity of the solution is greater than 1.5 M. Thus in Table I the dilute Zr wastes in storage Tanks WM-182 and -188 are stable. The dilute Zr waste in Tank WM-189 is not.

Calcium nitrate added to WM-185 waste to give a Ca/F mole ratio of 0.55 produces about 80g/L of solids (mostly $CaZrF_6$) in the waste.[2] Calcium nitrate added to dilute Zr waste would produce less than 80g/L solids because: a) the dilute waste is less concentrated in fluoride than WM-185 waste, b) less $Ca(NO_3)_2$ is used to give the dilute waste a Ca/F mole ratio of 0.55, and c) less $Ca(NO_3)_2$ added forms less $CaZrF_6$. The same would apply to a blend of WM-185 waste-Na waste and a blend of dilute Zr waste-Na waste if both blends had the same percent by volume Na waste. The solids formed when $Ca(NO_3)_2$ is added to a Zr waste or blend of the Zr waste with Na waste, including solids present before $Ca(NO_3)_2$ addition, are easily dispersed into solution. This would be true for blends stored in feed makeup tanks just prior to being fed to the WCF.

Space available in waste storage tanks at the ICPP will become very limited before the New Waste Calcining Facility (NWCF) comes on line. Since Zr and Na wastes are the two major wastes produced and stored at the ICPP, it may be desirable at some time to store blends of Zr and Na wastes in storage tanks. Based on stability studies using WM-185 wastes, blends of dilute Zr and Na wastes should become unstable within a few days. Table XV shows the depth of solids that would be produced and would settle to the bottom of a $1136m^3$ (300,000 gal) tank if $1080m^3$ (285,000 gal) of various blends of WM-185 and Na wastes were stored in them. Most waste storage tanks at the ICPP are $1136m^3$ tanks and a maximum of $1080m^3$ are usually stored in them. Table XV shows that the end of lines used to remove waste from storage tanks would protrude into solids if blends of 1 to 5 vol of WM-185 waste and 1 vol Na waste were stored at 35^oC in many ICPP waste storage tanks. Solids formed in blends of 2 to 10 vol WM-185 waste and 1 vol Na waste could not be easily removed from storage tanks as a slurry because they are present as flakes or granules.

TABLE XV

SOLIDS PRODUCED FROM STORAGE OF BLENDS OF WM-185 AND Na WASTES[a]

Blend	Storage Temperature (°C)	Depth of Solid on Bottom of tank cm	inches
1 vol WM-185 waste - 1 vol Na waste	35	30	12
2 vol WM-185 waste - 1 vol Na waste	35	10	4
3 vol WM-185 waste - 1 vol Na waste	35	15	6
4 vol WM-185 waste - 1 vol Na waste	35	18	7
5 vol WM-185 waste - 1 vol Na waste	35	13	5
10 vol WM-185 waste - 1 vol Na waste	24	2.0	0.8
	35	4.1	1.6
25 vol WM-185 waste - 1 vol Na waste	24	1.8	0.7
	35	3.6	1.4
50 vol WM-185 waste - 1 vol Na waste	24	1.0	0.4
	35	3.0	1.2
75 vol WM-185 waste - 1 vol Na waste	24	1.0	0.4
	35	3.0	1.2
100 vol WM-185 waste - 1 vol Na waste	24	1.0	0.4
	35	3.0	1.2

a. Assuming 1080m^3 (285,000 gal) of blend is stored in an ICPP 1136m^3 (300,000 gal) storage tank.

VIII. CAKING TEMPERATURES

Calcine stored in bins heated by fission product decay becomes difficult to remove from the bins when the temperature of the calcine rises above a given value (caking temperature) dependent upon the waste used to make calcine. It is imperative that the temperature of calcine stored in a bin not rise above the caking temperature if the calcine is to be retrieved.

The calcine from zirconium fluoride waste that will be stored in bins in the future may be heated as high as 400 to 500°C by fission product decay. Blending this waste with Na waste (a lower heat generating radioactive waste than Zr wastes) should produce calcine that will not be heated to as high a temperature as the unblended waste. Figure 10 shows the approximate caking temperatures of calcine produced by calcining the dilute Zr waste and blends of this waste with Na waste containing appropriate amounts of $Ca(NO_3)_2$. Appendix J shows how mole percent sodium in calcine granules (abscissa of Figure 10) is calculated. Caking temperatures of calcine were determined by heating the simulated calcine (bed granules without fines) in open crucibles and observing temperatures (to the nearest 50°C) at which the calcine begins to cake and stick to crucible surfaces. According to Figure 10, calcine produced from a blend of 4 1/2 vol of the dilute Zr waste stored in WM-182 with 1 vol of the Na waste stored in WM-180 (Table I) - having a Ca/F mole ratio of 0.7 - would have a caking temperature of approximately 550°C.

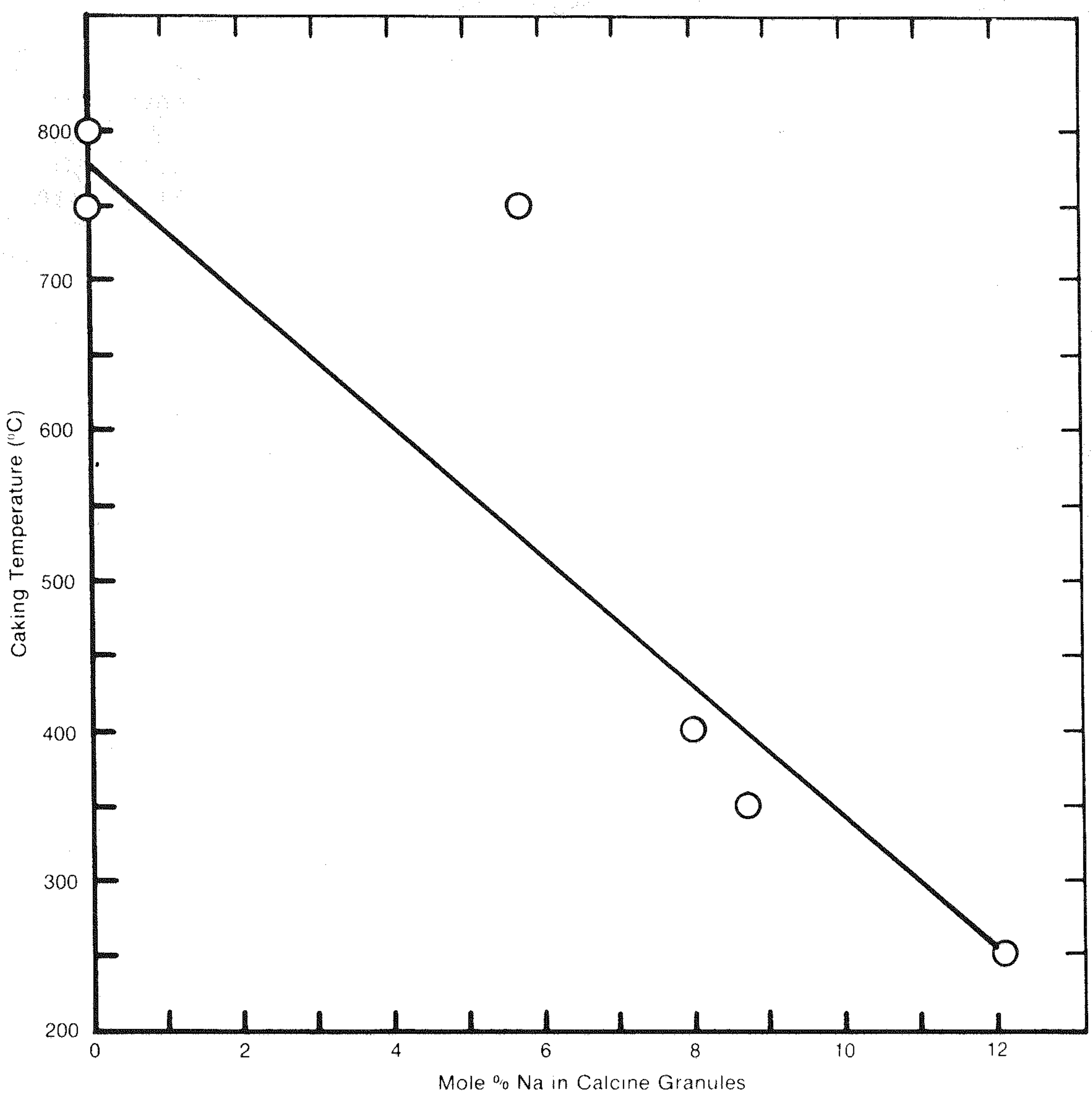

Figure 10. Variation of Caking Temperature with Sodium Content of Calcine

ICPP-A-5090

IX. COLLAPSED BED REFLUIDIZATION

Some problems have been experienced at the WCF in refluidizing the bed after it has been collapsed during a short shutdown. Difficulty in refluidizing the bed is probably caused by: a) calcine getting into the plenum (area below the distributor plate) and restricting fluidizing air flow; b) the calciner vessel walls contracting and compacting the bed during the cooling-down period; and c) the calcine particles sticking together slightly during cooling.

1. CALCINE IN PLENUM

Calcine can enter the plenum only through the fluidizing air holes, normally covered by bubble caps in the WCF. The bubble caps are designed to prevent backflow of solids when the bed is fluidized and when it is entirely stagnant. Sparge air or fluidizing air at a low rate could move the granules around enough to cause backflow into the plenum. Erosion or displacement of a bubble cap could also result in solids flow into the plenum.

2. VESSEL WALL CONTRACTION

It is not known how significant the effect of vessel wall contraction is on bed refluidization in the WCF. During cooling, the calciner vessel diameter will decrease 0.6 - 0.8cm and some compacting of the bed will occur.

3. CALCINE PARTICLES STICKING TOGETHER

Sodium-containing calcine particles have been observed to stick together slightly when cooled down in a bucket after being discharged from pilot-plant calciners.[2] The mechanism for the sticking is probably due to sodium nitrate (a low-melting compound) being present on the surface of the particles. Some brief pilot-plant tests showed that zirconium (WM-185)-sodium blend calcine would not stick together if cooled below 150^{o}C before being allowed to stagnate. This temperature could be different for other calcine types.

To determine how significant this particle cohesion is on bed refluidization, a test was conducted using the 30-cm-diameter calciner. The test consisted of simulating an emergency shutdown, allowing the bed to remain stagnant for two days, and then refluidizing the bed.

Run 71 was used for the experiment. When shutdown was initiated at 10:20 p.m. on Friday, the pilot-plant calciner had operated smoothly for 112h using a blend of 5 vol dilute Zr waste and 1 vol Na waste. Steady-state conditions had been reached, and the bed particle composition was probably almost identical to what the WCF would produce, except for the fission products.

Normal pilot-plant calciner shutdown consists of: switching from feed to nitric acid to purge the feed lines; shutting off acid, fuel, and oxygen; allowing the bed to cool while fluidized; and dumping the bed. For the test, shutdown consisted of: switching from feed to nitric acid for a few minutes and then shutting off acid, fuel, oxygen, and fluidizing air as rapidly as possible. The only air flow into the calciner over the weekend was from instrument purges.

For the refluidization test (beginning at approximately 8:30 a.m. on Monday) the fluidizing rate was slowly increased from 0 to 0.56m/s (1.8 ft/s) over about a 1-hour period (during Run 71 the fluidizing velocity below the fuel nozzle varied between 0.22 and 0.36 m/s or 0.72 and 1.2 ft/s). Pressure data were recorded throughout the test; however, the data were erratic and unreliable because of the caked bed. Fluidizing air was allowed to flow through the bed at the 0.56 m/s rate (maximum rate attainable in the 30-cm unit) for about one hour. The bed was not fluidized during this period; the air was channelling through the bed and slowly eroding the cake apart. During this period, a gasket in the fluidizing air line ruptured and the air was shut off. The gasket was repaired and the test restarted. The fluidizing rate was slowly increased to 0.36 m/s (1.2 ft/sec). As the rate was being increased, the air again channelled up through only part of the bed. After the air was at the 0.36 m/s rate for only a minute or two, the whole bed suddenly began fluidizing. The bed was allowed to fluidize for about 5 minutes and then the fluidizing test was repeated. As the air rate was increased, the bed began to fluidize at 0.14 m/s (0.5 ft/s) and was well fluidized at 0.18 m/s (0.6 ft/s). The bed was then drained; it flowed freely from the calciner. The back of the calciner was removed and the contents examined. Three or four very fragile cakes, 2 to 8 cm in diameter, remained on the distributor. If the bed had been allowed to fluidize for a few more minutes, these would have disintegrated also. Based on the examination, the calciner could have been safely started up after refluidization occurred. No agglomerates, which could not be easily broken up by fluidization, were formed by the bed collapse.

4. RECOMMENDATIONS FOR COLLAPSING WCF BED

a. When collapsing bed be sure fluidizing and sparge air are entirely off except for minimum purges necessary to prevent solids plugging of lines.

b. Do not collapse bed if bubble caps are damaged (or suspected to be damaged).

c. Cool bed to 150°C or lower before collapsing.

All of these recommendations cannot be met under some possible emergency conditions. The worst consequence of such a situation (if for some reason the bed cannot be refluidized) is the bed material would have to be dissolved; this probably does not warrant installation of an emergency fluidizing system.

5. RECOMMENDATIONS FOR REFLUIDIZING A COLLAPSED BED

Sometime an emergency condition may require the bed to be collapsed while still hot. Even though the particle-to-particle bonds are very weak in the resulting bed cake, the whole bed is much too large to lift, break up, and refluidize all at once. Based on the pilot-plant test, the recommended procedure is to put as much fluidizing air flow through the calciner as possible and to allow this flow to slowly erode the cake apart. In the pilot-plant, it took about three hours of channelling and erosion before the bed fluidized. In the WCF, this time could be quite different, depending on type of bed material, how long the bed has been collapsed, rate of air flow, and amount of moisture that has contacted the bed while stagnant. During erosion of the cake, bed material may get into the plenum and inhibit fluidizing flow. The plenum should be drained regularly during a refluidization attempt. If the bed cannot be refluidized after a couple of days of erosion, probably the only alternative is to dissolve it.

X. DISSOLUTION AND DISINTEGRATION OF CALCINE CLINKERS

It might be necessary to dissolve the bed in the calciner vessel of the WCF to remove it. Any clinkers in the bed would also have to be dissolved and/or disintegrated into particles small enough to be slurried from the calciner vessel. Studies were performed to find reagents that would dissolve and/or disintegrate clinkers formed from blends of dilute Zr waste and Na waste.

1. EXPERIMENTAL PROCEDURES

All of the synthetic clinkers used in the studies except two were made by melting calcine formed from Run DZrW4-3 (feed was a blend of 8 vol dilute Zr - 1 vol Na wastes) at 1150°C using a propane-oxygen torch. When the appropriate amount of calcine was molten, the torch was immediately removed from the calcine. One clinker was formed on the end of a piece of 1/4-inch stainless steel tubing by the method just described, except the torch was held on the molten calcine 5 minutes before being removed. Dissolution and/or disintegration of this clinker would represent removal of a clinker from a nozzle or a temperature rake (device for holding thermocouples in different bed locations) and also dissolution and/or disintegration of a clinker formed by exposure to a high temperature for a longer period of time than the clinkers previously described. Another clinker was formed on a stainless steel rod inserted (1cm above the fuel nozzle) into the bed of a 15-cm-diameter, fluidized-bed, in-bed combustion calciner. The bed of the calciner consisted of calcine from Run 71.

The following sequence of events were used to create the latter clinker: the bed was heated to 500°C and then cooled to 300°C with 4<u>M</u> nitric acid; the fuel and oxygen rate into the bed were increased and the fluidizing air was turned off; the fuel and oxygen rate were maintained high until the bed temperatures spread from 300 to 850°C. The temperature of the rod was >1370°C since it partially melted. The clinker was removed from the rod before treatment with reagents. This clinker was treated with reagents to determine if a clinker formed in a calciner would behave differently toward given reagents than a clinker formed in a propane-oxygen flame.

Synthetic clinkers were treated with combinations of nitric acid (HNO_3), aluminum nitrate ($A\ell(NO_3)_3$, sulfuric acid (H_2SO_4), hydrofluoric acid (HF), and boric acid (H_3BO_3). All reagent combinations were stable. Clinkers were exposed to each reagent for about 22h; 16mL of reagent combination was used per gram of clinker. Agitation of reagents containing clinkers was provided by gentle boiling, or if the reagent was maintained below the boiling point, by air sparging at 57 L/h (2 scfh). The dissolution plus disintegration rate of a clinker in a reagent combination was taken during the first 4h that the clinker was in contact with the reagents. The rate was determined by removing the clinker from the reagents, drying the clinker at 105°C, cooling to 24°C, weighing, and placing the clinker back into the reagent under the appropriate conditions. Clinkers undissolved or not disintegrated and fine undissolved solids remaining in the reagent after 22h were removed from the reagent, dried at 105°C, cooled to 24°C, and weighed. Some of these clinkers and fine undissolved solids were examined by X-ray diffraction and spectrochemical analysis.

2. RESULTS

The results of the dissolution of synthetic clinkers in various reagent combinations at different temperatures are summarized in Table XVI. The reagent combination of 6MHNO3-0.3MAℓ(NO3)3 has been used to dissolve calcine agglomerates and calcine in the WCF calciner vessel during decontamination of the vessel and will be referred to hereafter as the standard reagent combination. The standard reagent combination at 80°C dissolved and disintegrated the clinkers completely at a slow rate; the initial rate was equal to 10% of the initial clinker weight per hour with the rate decreasing as the dissolution progressed. The undissolved solids remaining after a dissolution were fine enough to be slurried in-and-out of vessels, through lines, and through valves. Addition of HNO_3, $Al(NO_3)_3$, HF, or H_3BO_3 to the standard reagent combination increased the dissolution rate only slightly, if at all. Decreasing the HNO_3 concentration of the standard reagent combination decreased the dissolution rate. Addition of H_2SO_4 to the standard reagent combination made the clinker inert to dissolution or disintegration. The amount of undissolved solids remaining after a dissolution using the standard reagent combination is roughly 3% of the initial clinker weight. Addition of $Al(NO_3)_3$ to the standard reagent combination increased the amount of undissolved solids remaining after a dissolution; addition of HF to the standard reagent combination decreased the amount of undissolved solids remaining after dissolution. Vigorous sparging during a dissolution seemed to be as effective in maintaining a good dissolution and disintegration rate as did increasing the dissolution temperature from 80 to 105°C. The standard reagent combination at 80°C was able to remove the clinker from the end of the piece of stainless steel tubing in 6 to 22h and to completely dissolve and disintegrate the clinker within 22h. The standard reagent combination at 80°C was also able to completely dissolve or disintegrate the clinker made in the calciner within 22h at a rate comparable to rates observed in dissolution of torch-made clinkers.

Major constituents present in clinkers (≥5 wt %) remaining undissolved and not disintegrated after 22h of exposure to reagents were (listed in order of decreasing concentration): zirconium, aluminum, calcium, and phosphorous. Major constituents present in undissolved solids (separate from clinkers) at the end of dissolutions were (listed in order of decreasing concentration): zirconium, phosphorus, and tin. Crystalline compounds found in undissolved solids were: $CaSO_4$, $NaZr_2(PO_4)_3$, ZrO_2, and $Ca_{0.15}Zr_{0.85}O_{1.85}$; undissolved solids from the clinkers made on the stainless steel tubing and rod also contained oxides of metals in the stainless steel. Crystalline compounds found in the undissolved and undisintegrated clinkers were: CaF_2, ZrO_2, and $CaSO_4$.

Apparently the clinkers dissolved and disintegrated as well as they did because essentially all the alumina present was amorphous not crystalline (not soluble in HNO_3). The undissolved solids (separate from the clinkers) remaining after dissolution of the clinker from the end of the stainless steel tubing and the clinker formed in the calciner (hereafter called the specially formed clinkers) contained from 0.5 to 1 wt % aluminum; the undissolved solids remaining after dissolution of the other clinkers contained from 0.02 to 0.05 wt % aluminum. Apparently heating the clinker on the tubing at 1150°C for a longer period of

TABLE XVI

DISSOLUTION AND DISINTEGRATION OF CLINKERS[a]

Clinker Source	Reagent Combination	Clinker Weight (g) Before Dissoln	Clinker Weight (g) After Dissoln	Dissolution Rate g/h	Dissolution Rate Wt % of Initial wt. of Clinker	Dissolution Temperature (^oC)	Undissolved Solid at end of dissolution (wt % of Initial wt. of clinker)
b	3M HNO_3-0.3M $A\ell(NO_3)_3$	9.4	3.0	0.3	3	97[c]	4.0[d]
b	6M HNO_3-0.3M $A\ell(NO_3)_3$	12.3	0.7	0.8	7	105[c]	3.3[d]
b	9M HNO_3-0.3M $A\ell(NO_3)_3$	18.9	0	3.2	17	110[c]	3.3
b	6M HNO_3-0.7M $A\ell(NO_3)_3$	16.6	0	1.7	10	105[e]	6.4
b	6M HNO_3-0.3M $A\ell(NO_3)_3$ -3M H_2SO_4	19.0	19.5	0	0	105[e]	0[d]
b	6M HNO_3-0.3M $A\ell(NO_3)_3$	14.8	0.2	1.6	11	80[e]	2.4[d]
b	9M HNO_3-0.3M $A\ell(NO_3)_3$	13.0	0.5	1.6	12	80[e]	2.2[d]
b	6M HNO_3-0.5M $A\ell(NO_3)_3$ -0.35M HF	17.2	0	2.4	14	100[e]	1.5
b	6M HNO_3-0.3M $A\ell(NO_3)_3$ -0.34M H_3BO_3	18.6	0	2.8	15	101[e]	2.3
f	6M HNO_3-0.3M $A\ell(NO_3)_3$	~6	0	g	g	80[e]	~12
h	6M HNO_3-0.3M $A\ell(NO_3)_3$	12.5	0	1.2	10	80[e]	11.8

a. Duration of dissolution = 22 h; 16 mL of reagent combination was used per gram of clinker.
b. Calcine treated with torch at 1150^oC; torch immediately removed when calcine became molten.
c. Boiling temperature of reagent combination.
d. This does not include the weight of clinker that has not been dissolved and/or disintegrated.
e. Below boiling temperature of reagent combination; reagents air sparged at 57 L/h (2 scfh).
f. Formed on end of tubing with torch at 1150^oC; torch removed after calcine remained molten for 5 minutes.
g. Not determined.
h. Formed on rod at >1370^oC in a calciner.

time than was used in forming other clinkers converted a small amount of amorphous alumina to crystalline alumina. The clinker made in the calciner was apparently subjected to higher temperatures and/or heated for a longer period at high temperatures than the other clinkers, and these extreme conditions caused a small amount of amorphous alumina to be converted to crystalline alumina. The undissolved solids remaining after dissolution and disintegration of the two specially formed clinkers were present in greater amounts than when other clinkers were dissolved because the specially formed clinkers contained more crystalline alumina and oxides of the components of stainless steel than did the solids formed from the other clinkers. It is not known why increasing the $A\ell(NO_3)_3$ concentration of the standard reagent combination produced more undissolved solids than normal during clinker dissolution. Adding HF to the standard reagent combination produced less solids because zirconium compounds are a major constituent of the clinkers and HF is usually more effective in dissolving zirconium compounds than other aqueous reagents. It is surprising that phosphorous is a minor to major constituent of clinkers since the blend used to make the calcine in the clinkers was <0.01$\underline{M}$ phosphate.

It is recommended that to dissolve clinkers in the WCF calciner vessel: a) 6-9$\underline{M}HNO_3$-0.3$\underline{M}A\ell(NO_3)_3$ at 80°C or higher temperature be used; b) the HNO_3 concentration of the dissolver solution not be allowed to fall below 6$\underline{M}$, c) the dissolver solution be sparged vigorously; and d) 13$\underline{M}HNO_3$ be added to the dissolver solution from time to time to compensate for steam dilution (used to heat solution in calciner vessel) and HNO_3 lost in dissolution.

XI. CONCLUSIONS AND RECOMMENDATIONS

The following conclusions were reached based on information presented in this report:

1. Dilute Zr waste can be calcined in a fluidized-bed at 500°C as a blend with Na waste if sufficient $Ca(NO_3)_2$ is added to give a Ca/F mole ratio of 0.7.

2. Calcine from blends of dilute Zr and Na wastes containing 5.3 mole percent sodium or less and a Ca/F mole ratio of 0.7 is relatively free from fluidized-bed agglomeration and has a caking temperature (determined by crucible method) of 550°C or greater.

3. The presence of $Ca(NO_3)_2$ in blends of 3 to 8 vol dilute Zr-1 vol Na wastes to give a Ca/F mole ratio of 0.7 effectively suppresses fluoride, chloride, and sulfate volatility when the blends are calcined (about 0.4, 15, and 3%, respectively, of that fed to calciner is volatilized from calciner vessel).

4. Calcination of a blend of 3 vol dilute Zr waste and 1 vol Na waste (Table II gives waste compositions) might produce calcine too noduled and agglomerated to give good quality fluidized-bed operation. Calcination of blends of 5 and 8 vol dilute Zr waste and 1 vol Na waste produces calcine with few nodules and little tendency to agglomerate. Beds of the latter calcine should give good quality fluidized-bed operation.

5. During calcination of blends of 3 to 8 vol dilute Zr waste with 1 vol Na waste containing a Ca/F mole ratio of 0.7: a) the product-to-fines ratio seems to increase as the proportion of Na waste in the blend increases, and b) the attrition index and bulk density of the calcine product seem to be independent of the Na waste content of the blend.

6. Calcination of blends of 3 to 8 vol dilute Zr waste - 1 vol Na waste having a Ca/F mole ratio of 0.7 produce hard calcine (attrition index 70 to 80%) with a high bulk density ($1.6 g/cm^3$).

7. Calciner operability and calcine properties should be about the same during calcination of a blend of 5-8 vol dilute Zr waste and 1 vol Na waste (Ca/F mole ratio of 0.7) at 500°C as during the calcination of 3 1/2 vol WM-185 waste (Table I) and 1 vol Na waste (Ca/F mole ratio of 0.7) at 500°C. The latter blend is similar to the one successfully calcined in the WCF during its eighth processing campaign.

8. The undissolved solids content of dilute Zr-Na wastes blends containing a Ca/F mole ratio of 0.7 is sufficiently low so that plugging of calciner feed equipment should be a less frequent problem than during processing of unblended zirconium waste.

9. Size of calcine product during calcination of dilute Zr-Na wastes blends can be controlled by variation of waste NAR in the 10-cm calciner and by variation of the waste NAR and oxygen-to-fuel ratio in the 30-cm calciner.

10. Blends of dilute Zr and Na liquid wastes are unstable within a day.

11. Clinkers formed during calcination of dilute Zr and Na wastes blends can be dissolved and/or disintegrated in $6\underline{M}HNO_3 - 0.3\underline{M}\ A\ell(NO_3)_3$ at 80°C.

12. Calciner operability, fluidized-bed properties, and properties of calcine solids produced seem to be about the same during calcination of dilute Zr waste (Ca/F mole ratio of 0.55) at 500°C as during calcination of Zr wastes more concentrated in aluminum, zirconium, and fluoride (Ca/F mole ratio of 0.55) at 500°C except the product rate when calcining the former waste was 1/2 that obtained calcining the latter wastes.

13. Calcination of dilute Zr waste containing a Ca/F mole ratio of 0.55 results in a much higher chloride volatility (>70% of the chloride fed to calciner volatilized from calciner vessel) than normally obtained when calcining blends of Zr and Na wastes (15%) containing a Ca/F mole ratio of 0.7.

14. Calcination of dilute Zr waste containing a Ca/F mole ratio of 0.55 results in a slightly higher fluoride volatility than (1% of that fed to calciner volatilized from calciner vessel) and about the same sulfate volatility (3%) as normally obtained when calcining Zr wastes and blends of Zr and Na wastes.

The following recommendations were reached based on information presented in this report:

1. A dilute Zr-Na waste blend producing calcine having a sodium mole percent of 5.3 or less should be used to calcine dilute Zr waste. A blend producing calcine having a sodium mole percent of 5.3 will provide a maximum Na waste throughput rate in the WCF while maintaining suitable calciner operations. The calcine has a reasonably high caking temperature (550°C). This blend ratio provides a safety factor from blend ratios that might produce bed agglomeration or caking in the storage bins. (The method used to calculate sodium mole percent in calcine is described in Appendix J.).

2. Control particle size and agglomeration by using the appropriate waste NAR, oxygen-to-fuel ratio, and fluidizing air rate. Using waste NAR and the oxygen-to-fuel ratio to control particle size is preferable to alternately calcining the blended dilute Zr-Na wastes with unblended dilute Zr waste. Calcining unblended dilute Zr waste will cause the chloride concentration in the off-gas acid scrub solution to exceed its maximum allowable concentration (2600 ppm) and exceed it more rapidly than when calcining the unblended Zr wastes that were

solidified during the eighth and prior WCF campaigns. This is because the dilute Zr wastes (at least those in WM-188 and WM-189) contain much higher concentrations of chloride than the latter Zr wastes.

3. Use a Ca/F mole ratio of 0.7 in dilute Zr-Na wastes blends.

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APPENDIX A

INFORMATION FROM TEN-CENTIMETRE CALCINER RUNS

TABLE A-1

OPERATING CONDITIONS FOR TEN-CENTIMETRE CALCINER RUNS

Calcination temperature = 500°C; Solid $Ca(NO_3)_2$ = fluoride and chloride volatility suppresser; Waste feed rate = 2L/h; Run duration = 40 h; Starting beds: Run DZrW4-1 = calcined WM-185 waste; Runs DZrW4-2, -3, and -4 = Calcined 5 vol WM-185 waste - 1 vol Na waste; Ca/F mole ratio: 0.55 for Run DZrW4-1 and 0.70 for Runs DZrW4-2, -3, and -4. Nozzles are Spraying System Co. nozzles; Feed nozzle = 40100 liquid, 120 air; Fuel nozzle = 2050 liquid, 67-6-20-70° air.

Run #	DZrW4-1	DZrW4-2	DZrW4-3	DZrW4-4
Waste Feed	The dilute Zr Waste	5 vol dilute Zr waste-1 vol Na waste	8 vol dilute Zr waste-1 vol Na waste	3 vol dilute Zr waste-1 vol Na waste
Fluidizing Air Rate - L/s (scfm)(a)	0.93 ± 0(1.97 ± 0)	0.93 ± 0(1.97 ± 0)	0.93 ± 0(1.97 ± 0)	0.92 ± 0(1.96 ± 0)
Fluidizing Velocity below Nozzle - m/s (f/s)(a)	0.33 ± 0(1.1 ± 0)	0.33 ± 0(1.1 ± 0)	0.33 ± 0(1.1 ± 0)	0.33 ± 0(1.1 ± 0)
Feed Atomizing Air Pressure - kPa(psig)(a)	62 ± 7(9 ± 1)	105^{+5}_{-2} $(15.3^{+0.7}_{-0.3})$	116^{+43}_{-6} $(16.8^{+6.2}_{-0.8})$	273^{+3}_{-8} $(39.6^{+0.4}_{-1.1})$
Feed Atomizing Air Rate - L/s(scfm)(a)	$0.36^{+0.05}_{-0.009}$ $(0.76^{+0.10}_{-0.02})$	0.57 ± 0(1.20 ± 0)	0.57 ± 0(1.20 ± 0)	0.52 ± 0(1.11 ± 0)
Fuel Nozzle O_2 Rate - L/s(scfm)(a)	$0.37^{+0.009}_{-0.04}$ $(0.78^{+0.02}_{-0.08})$	0.37 ± 0(0.79 ± 0)	0.37 ± 0(0.79 ± 0)	0.37 ± 0(0.79 ± 0)
Kerosene Feed Rate - L/h (a)	$0.57^{+0.03}_{-0.02}$	$0.62^{+0.03}_{-0.05}$	$0.60^{+0.03}_{-0.02}$	$0.68^{+0.05}_{-0.07}$
NAR (a)	634^{+97}_{-14}	1020 ± 0	1020 ± 0	935 ± 0
Bed Turn-over - %	67	80	80	84

(a) The numbers represent the average, maximum, and minimum values obtained during a run.

TABLE A-2

FLUIDIZED-BED CHARACTERISTICS DURING TEN-CENTIMETRE CALCINER RUNS

Run #	DZrW4-1	DZrW4-2	DZrW4-3	DZrW4-4
WASTE FEED	The dilute Zr waste	5 vol dilute Zr waste-1 vol Na waste	8 vol dilute Zr waste-1 vol Na waste	3 vol dilute Zr waste-1 vol Na waste
Product				
Production rate (g/h)	111	177	182	233
Density (g/cm^3)(a)	$1.35^{+0.03}_{-0.08}$	$1.64^{+0.09}_{-0.14}$	1.53 ± 0.03	$1.61^{+0.03}_{-0.06}$
MMPD (mm)(a)	$0.3295^{+0.0262}_{-0.0233}$	$0.5698^{+0.0173}_{-0.0315}$	$0.5111^{+0.0401}_{-0.0357}$	$0.4884^{+0.0059}_{-0.0093}$
FINES				
Production rate(g/h)	82	40	46	30
Density(g/cm^3)(a)	$0.61^{+0.17}_{-0.12}$	$0.48^{+0.05}_{-0.06}$	$0.51^{+0.04}_{-0.05}$	$0.98^{+0.12}_{-0.09}$
PRODUCT-TO-FINES RATIO	1.4	4.7	4.2	6.5
STARTING BED				
MMPD(mm)	0.4463	0.5383	0.5383	0.5383
Density(g/cm^3)	1.23	1.34	1.34	1.34
Attrition Index of - 32 +35 mesh fraction (%)	12	60	60	60
FINAL BED				
MMPD(mm)	0.3062	0.5871	0.5013	0.4839
Density(g/cm^3)	1.39	1.70	1.55	1.64
Attrition Index of -32 +35 mesh fraction (%)	20	82	74	76
Wt % of bed that is +14 mesh particles	2.4	0.72	0.44	5.3
Wt % of bed that is +1/2 - inch diam. agglomerates	5.3	0.36	0.46	5.1

(a) The numbers represent the average, maximum, and minimum values obtained during a run.

TABLE A-3

BEHAVIOR OF VOLATILES IN TEN-CENTIMETRE CALCINER RUNS

Run #	DZrW4-1			DZrW4-2			DZrW4-3			DZrW4-4		
WASTE FEED	The dilute Zr waste			5 vol dilute Zr waste–1 vol Na waste			8 vol dilute Zr waste–1 vol Na waste			3 vol dilute Zr waste–1 vol Na waste		
VOLATILE	F	Cl	Hg	F	Cl	Hg	F	Cl	Hg	F	Cl	Hg
% of total in (a):	(b)	(c)	(c)	(b)	(c)	(c)	(b)	(c)	(c)	(b)	(c)	(c)
Calcine Solids(d)(e)	93	<33	$0.40^{+0.13}_{-0.14}$	99.8	80^{+6}_{-7}	0.13 ± 0.01	99.7	84^{+5}_{-10}	$0.10^{+0.02}_{-0.01}$	99.9	88 ± 1	$0.17^{+0.03}_{-0.04}$
Fines	—	<12.2	$0.34^{+0.12}_{-0.06}$	—	30^{+5}_{-6}	$0.12^{+0.04}_{-0.02}$	—	22^{+10}_{-7}	$0.08^{+0.01}_{-0.03}$	—	11^{+2}_{-4}	$0.15^{+0.02}_{-0.03}$
Product	—	<20.6	0.06 ± 0.04	—	54 ± 3	0.01 ± 0.002	—	61^{+13}_{-19}	$0.02^{+0.01}_{-0.002}$	—	77^{+3}_{-2}	0.01 ± 0.002
Acid Scrub	5.9	61	92	0	11	97	0	7.6	95	0	6	83
Off Gas Leaving Acid Scrub(f)	$1.1^{+0.4}_{-0.7}$	12 ± 2	$6^{+10}_{-5.8}$	$0.22^{+0.09}_{-0.10}$	3 ± 1	2.8 ± 0.6	$0.34^{+0.04}_{-0.08}$	3^{+3}_{-2}	4^{+5}_{-2}	$0.13^{+0.05}_{-0.08}$	2 ± 1	16^{+5}_{-8}
% of Total Volatile Accounted for(a):	—	75^{+11}_{-5}	47 ± 4	—	57^{+18}_{-13}	24^{+1}_{-0}	—	74^{+14}_{-31}	18 ± 1	—	69^{+9}_{-12}	22 ± 2

(a) The numbers given represent the average, maximum, and minimum values obtained during a given run.
(b) Percent of volatile fed to calciner.
(c) Percent of total volatile found in calciner streams.
(d) Fines plus product.
(e) Fluoride in calcine solids = fluoride in feed - (fluoride in acid scrub + fluoride in caustic scrub).
(f) Based on component collected in caustic scrubber.

APPENDIX B

OPERATING CONDITIONS FOR RUN 71

TABLE B-1
MAJOR FLOWRATES
RUN 71

COT (HOURS)	FUEL (L/HR)	WASTE (L/HR)	FLUID AIR (M3/HR)	WASTE ATOM. AIR (M3/HR)	FUEL ATOM. O2 (M3/HR)	PURGES (M3/HR)	WET OFFGAS (M3/HR)	DRY OFFGAS (M3/HR)	OFFGAS DEW POINT (C)	SCRUB TO VENT. (L/HR)	VENTURI SCRUB RETURN (L/HR)	COND. TO DRAIN (L/HR)	SCRUB TANK ACC. (L/HR)	SCRUB RECYCLE TO FEED (%)
4	7.3	20.4	32.1	14.76	17.17	.40	99.3	63.9	74.5	51.82	126.00	29.40	4.84	0.0
8	6.8	41.6	33.4	14.83	12.48	.40	123.6	62.6	79.4	51.82	126.00	29.40	3.87	0.0
12	6.9	36.5	33.3	14.90	12.70	.40	117.1	62.3	79.4	51.82	135.00	28.70	1.29	0.0
16	6.9	37.7	33.2	14.76	12.71	.40	118.8	62.4	79.8	52.57	144.00	28.00	1.61	0.0
20	7.0	40.9	33.2	14.89	12.87	.40	122.7	62.3	80.7	52.57	97.00	21.00	.81	0.0
24	7.1	40.3	33.3	14.99	13.15	.40	123.5	63.7	79.2	53.32	105.00	29.50	.64	0.0
28	7.1	42.0	33.4	15.04	13.19	.40	121.7	59.8	81.0	52.57	109.50	31.55	1.94	0.0
32	7.0	38.2	33.3	15.01	12.86	.40	117.9	60.8	79.3	52.57	114.00	33.60	4.51	0.0
36	6.0	39.9	33.3	15.07	10.89	.40	116.9	59.1	80.4	52.57	120.60	32.55	3.06	0.0
40	5.9	41.1	33.4	14.93	10.93	.40	118.5	59.3	81.4	52.57	127.20	31.50	3.06	0.0
44	5.9	42.8	33.6	14.76	10.94	.40	120.6	59.3	81.8	52.57	115.20	27.75	8.22	0.0
48	5.9	47.5	33.6	15.19	10.97	.40	127.5	59.8	82.9	52.57	103.20	24.00	1.29	0.0
52	5.8	28.0	33.6	15.16	10.64	.40	101.9	58.8	77.5	52.57	112.80	25.80	2.74	0.0
56	5.7	38.6	33.6	15.21	10.34	.40	115.3	59.0	80.9	52.57	122.40	27.60	1.45	0.0
60	5.7	37.7	36.7	15.04	11.23	.40	117.9	62.8	79.8	52.57	124.50	26.70	1.61	0.0
64	5.6	38.4	36.4	14.99	11.15	.40	118.2	62.4	80.0	52.57	126.60	25.80	5.16	0.0
68	5.6	41.6	36.4	15.11	11.09	.40	122.4	62.6	81.0	52.57	119.40	24.15	.81	0.0
72	5.5	43.2	36.4	15.20	10.79	.40	124.3	62.6	81.4	52.57	112.20	22.50	-.16	0.0
76	5.6	37.7	36.4	15.11	11.09	.40	117.5	62.5	79.9	54.07	119.10	26.55	-.32	0.0
80	5.7	39.0	43.4	15.01	11.30	.40	126.4	69.6	78.4	53.32	126.00	30.60	-1.13	0.0
84	5.7	38.2	44.4	15.18	11.41	.40	126.5	70.8	77.6	52.57	124.80	28.10	2.58	0.0
88	5.8	38.2	43.2	15.06	11.90	.40	125.9	70.0	78.0	53.32	123.60	25.60	6.61	0.0
92	5.9	35.0	44.1	14.94	12.32	.40	123.0	71.0	74.0	53.32	120.30	26.60	2.10	0.0
96	5.8	44.9	44.3	15.06	11.83	.40	135.3	71.0	79.1	53.32	117.00	27.60	.81	0.0
100	5.8	34.3	44.5	15.15	12.16	.40	122.3	71.2	75.8	52.57	118.50	27.30	.81	0.0
104	5.8	38.2	44.7	15.10	12.07	.40	127.5	71.6	77.1	53.32	120.00	27.00	1.37	0.0
108	5.9	37.7	44.4	15.13	12.11	.40	127.0	71.4	77.2	53.32	122.50	27.00	1.94	0.0
112	6.0	37.7	44.4	15.13	12.39	.40	127.5	71.7	77.4	53.32	125.00	27.00	1.61	0.0

RECYCLE RATE IS BASED ON FEED WITH CALCIUM (SCRUB/FEED WITH CA + SCRUB)

TABLE B-2

CALCINER DATA

RUN 71

CDT (HOURS)	AVERAGE BED PRESSURE (KPA)	(PSI)	TEMPERATURE AVERAGE BED (C)	WASTE TO FUEL RATIO	O2 TO FUEL RATIO	O2 INPUT (% TH)	CO (%)	CO2 (%)	COMBUSTION EFFICIENCY (%)
4	121.7	16.2	491.+- 4.5	2.80	2364.	135.4	.95	.60	70.0
8	100.0	14.5	490.+- 2.2	6.13	1841.	98.5	.95	.60	70.0
12	105.0	15.2	494.+- 3.2	5.30	1846.	100.2	.95	.60	70.0
16	105.2	15.3	493.+- 3.0	5.48	1847.	100.3	.90	.40	66.1
20	105.3	15.3	494.+- 2.4	5.86	1845.	101.5	1.15	1.00	73.3
24	101.2	14.7	495.+- 3.5	5.69	1858.	103.7	.70	.20	61.9
28	104.0	15.1	492.+- 2.1	5.93	1864.	104.0	3.15	12.00	89.3
32	102.0	14.8	490.+- 2.7	5.47	1843.	101.4	3.55	11.40	88.4
36	104.0	15.1	492.+- 4.6	6.60	1832.	85.9	2.75	14.20	92.0
40	106.9	15.5	490.+- 5.5	6.94	1844.	86.2	2.85	16.60	92.8
44	107.1	15.5	491.+- 5.7	7.22	1845.	86.3	2.75	15.00	92.4
48	107.1	15.5	488.+- 5.8	8.01	1849.	86.5	2.85	17.00	93.0
52	108.1	15.7	487.+- 6.8	4.81	1830.	83.9	2.80	16.20	92.8
56	107.9	15.7	485.+- 8.7	6.77	1815.	81.6	3.00	16.80	92.6
60	107.8	15.6	491.+- 8.4	6.62	1972.	88.6	2.35	14.80	93.3
64	107.8	15.6	490.+- 9.2	6.88	1999.	88.0	2.35	15.40	93.5
68	108.5	15.7	491.+- 9.8	7.45	1988.	87.5	2.15	14.40	93.6
72	108.9	15.8	479.+-23.4	7.93	1977.	85.1	2.05	14.80	94.0
76	108.1	15.7	478.+-18.6	6.77	1989.	87.5	2.10	12.40	92.9
80	105.9	15.4	487.+- 7.0	6.85	1984.	89.2	3.00	17.60	92.9
84	104.3	15.1	489.+- 5.7	6.70	2002.	90.0	2.85	16.80	92.9
88	105.1	15.2	490.+- 6.7	6.56	2047.	93.9	3.00	16.60	92.5
92	94.1	13.6	491.+- 5.1	5.90	2077.	97.1	2.95	17.20	92.8
96	103.0	14.9	491.+- 4.3	7.73	2035.	93.3	3.05	17.20	92.6
100	102.3	14.8	490.+- 3.7	5.91	2091.	95.9	3.00	17.00	92.6
104	102.8	14.9	487.+- 4.2	6.56	2076.	95.2	3.05	17.40	92.7
108	103.5	15.0	465.+-43.3	6.36	2041.	95.5	3.15	17.80	92.6
112	104.7	15.2	490.+- 4.9	6.24	2050.	97.7	2.90	18.00	93.2

TABLE B-3

RUN 71 FEED BATCH DATA
(COT in hours; all volumes in litres)

COT	Feed Batch Number	Feed Batch Volume Prior to $Ca(NO_3)_2$ Addition	Feed Batch Volume After $Ca(NO_3)_2$ Addition
1	1	404	426
7	2	323	341
11	3	322	348
28	4	330	363
39	5	326	355
48	6	352	377
58	7	322	347
69	8	325	350
78	9	322	347
90	10	327	353
100	11	333	366
108	12	159	182

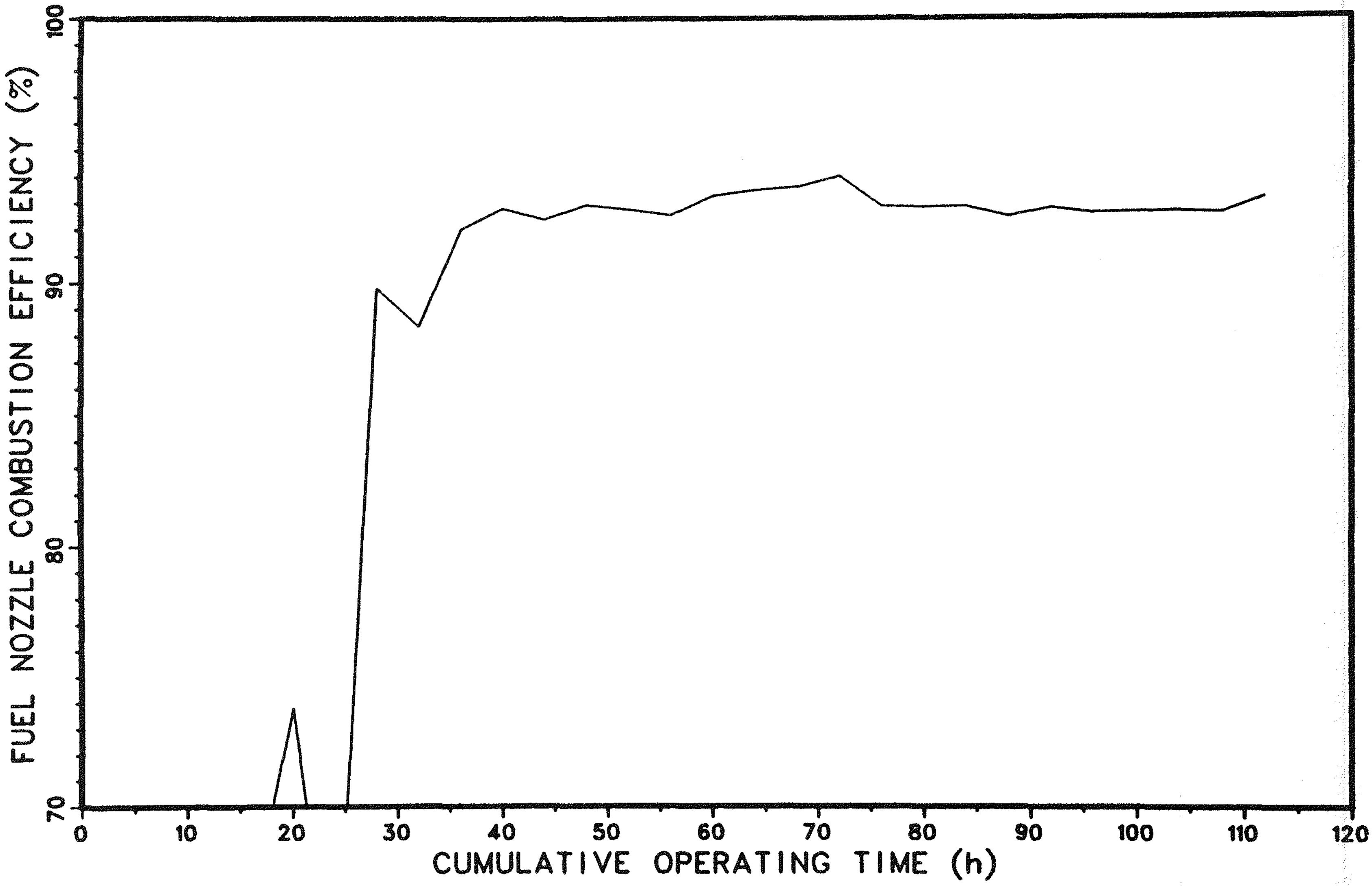

FIGURE B-1. FUEL COMBUSTION EFFICIENCY MEASURED DURING RUN 71

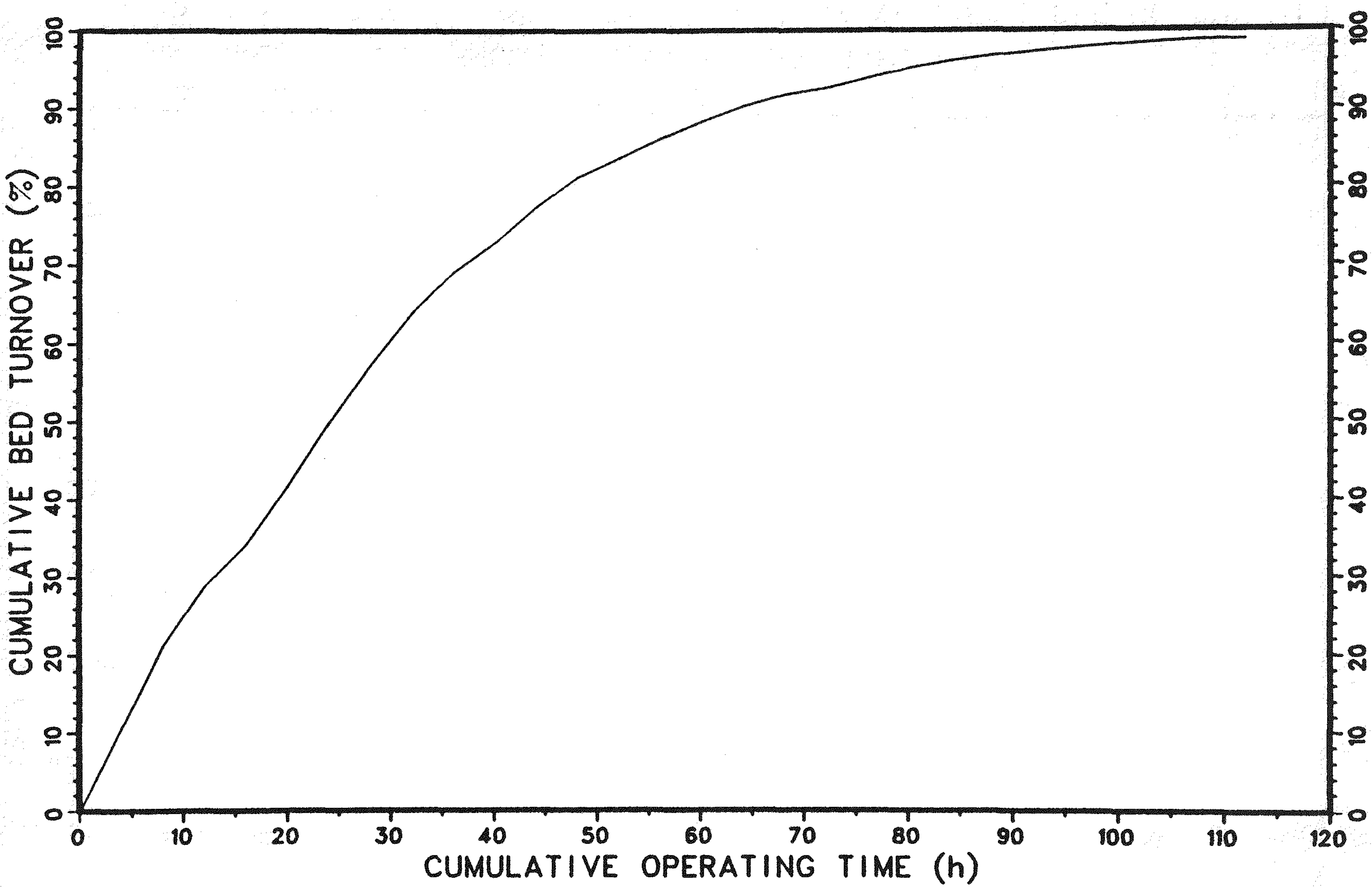

FIGURE B-2. BED TURNOVER OCCURING DURING RUN 71

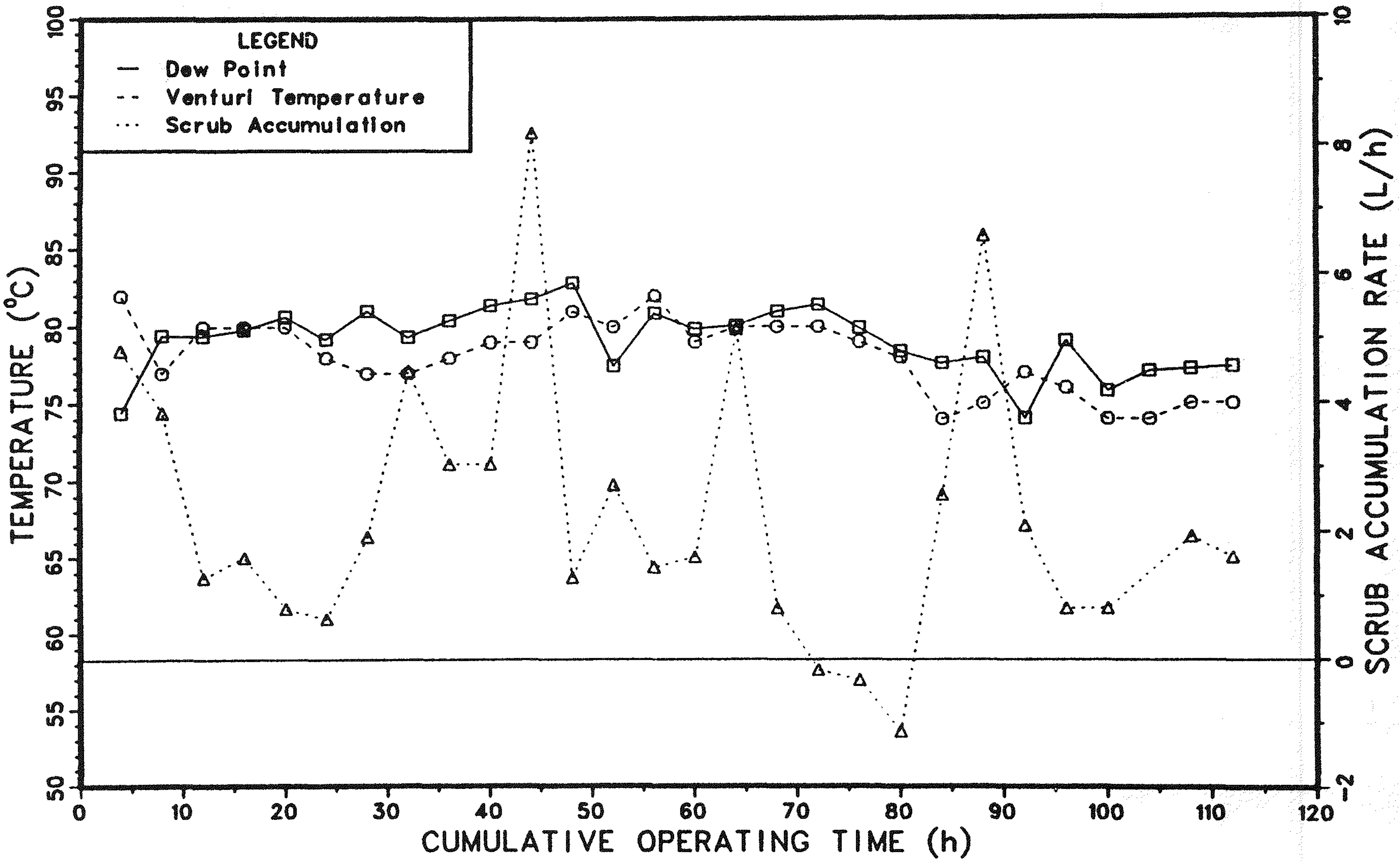

FIGURE B-3. SCRUB ACCUMULATION RELATIONSHIP TO DEW POINT AND VENTURI SCRUBBER TEMPERATURE DURING RUN 71

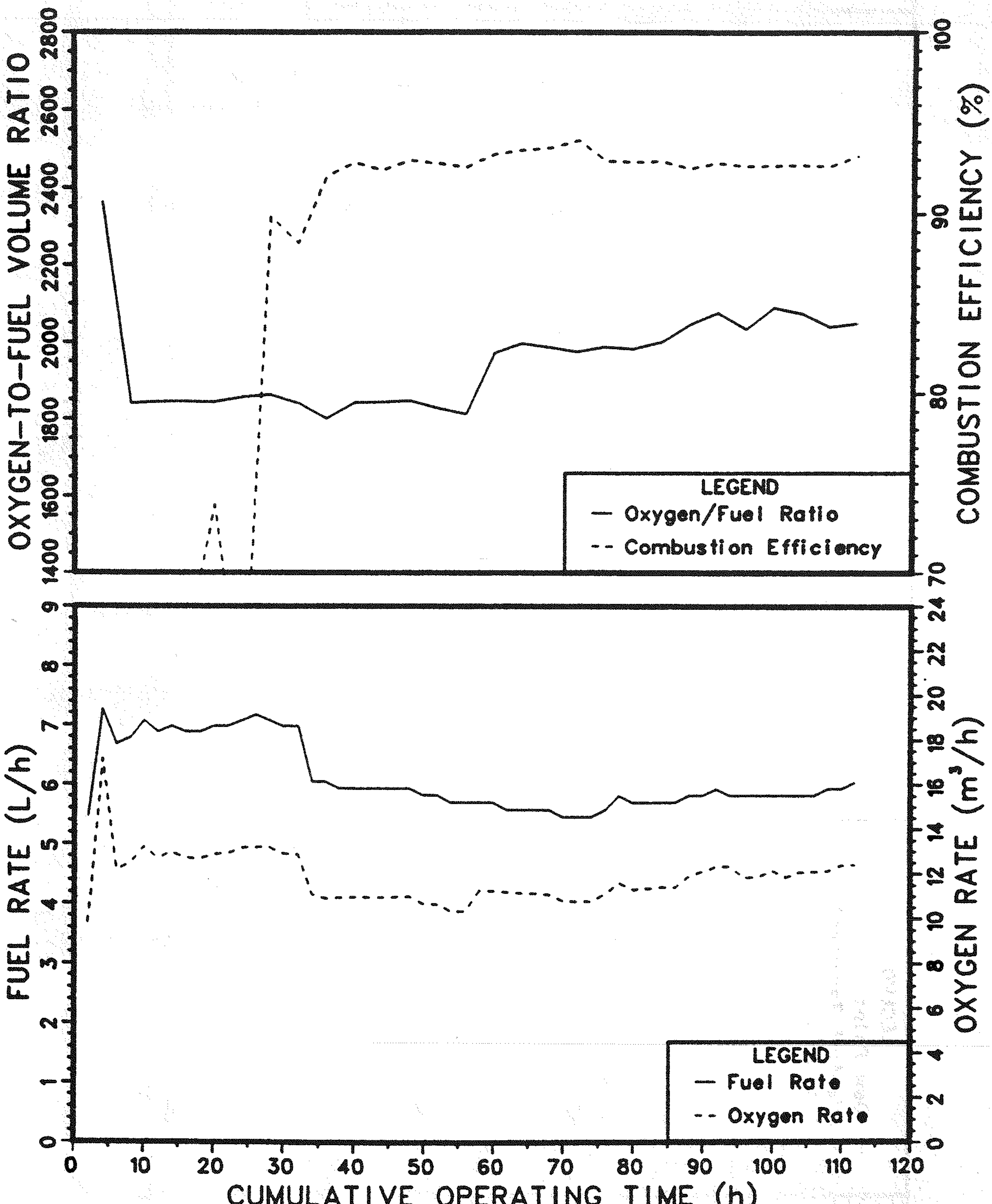

FIGURE B-4. IN-BED COMBUSTION SYSTEM PERFORMANCE DURING RUN 71

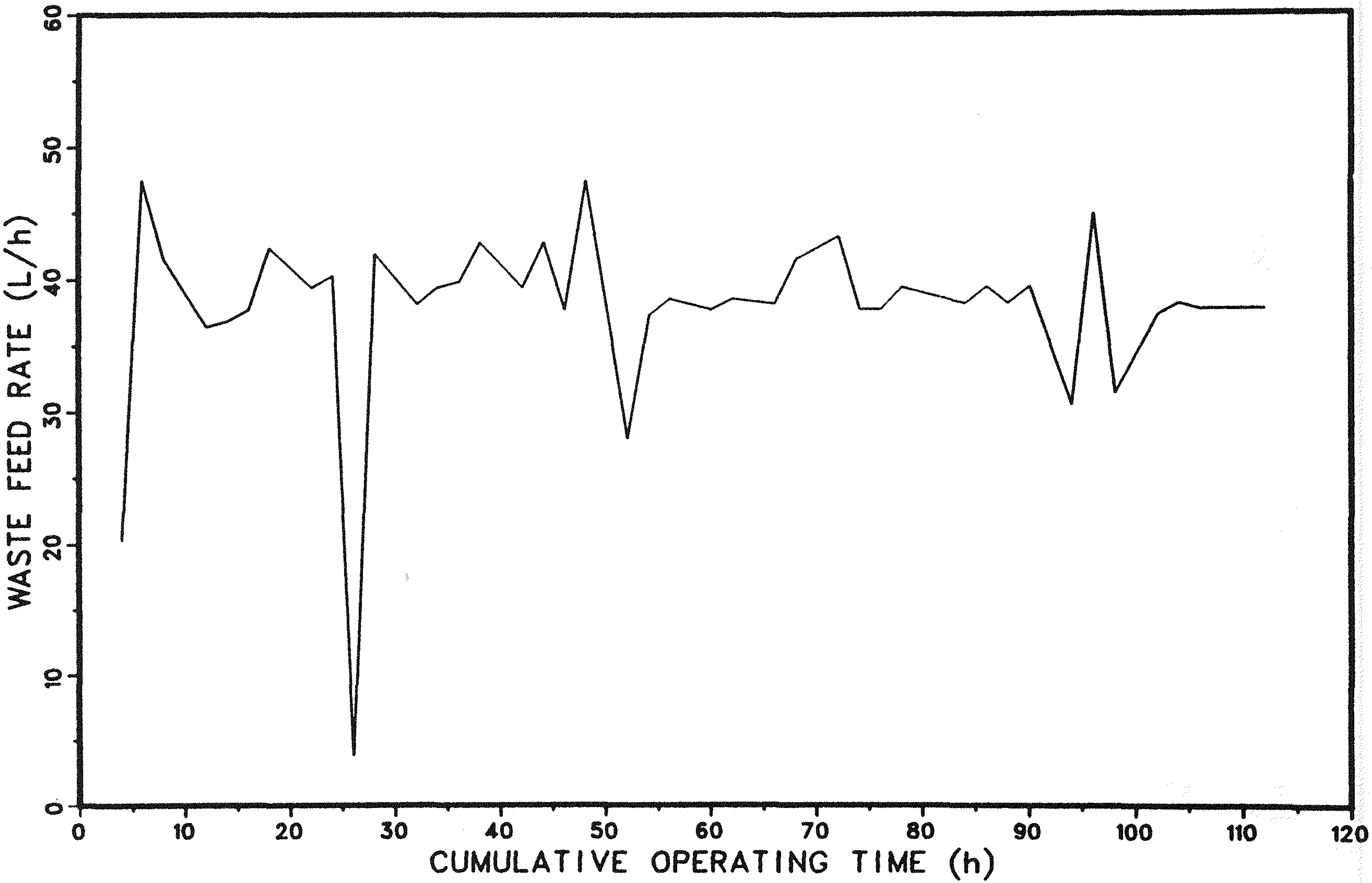

FIGURE B-5. WASTE FEED RATE DURING RUN 71

APPENDIX C

FLUIDIZED-BED OPERATION AND PROPERTIES OF SOLIDS GENERATED FOR RUN 71

TABLE C-1

PRODUCT SIZE DISTRIBUTION

RUN 71

CUMULATIVE OPERATING TIME (HOURS)	WEIGHT FRACTION RETAINED ON SCREEN — COMBINED SIZE RANGE (MESH) 14	16	20	24	28	32	35	48	65	100	150	200	-200	MMPD (MM)
0	.0075	.0038	.0150	.0469	.1316	.1917	.1992	.2782	.0902	.0338	0.0000	0.0000	0.0000	.47
8	0.0000	.0043	.0043	.0383	.0638	.1921	.2065	.3574	.2000	.0213	0.0000	0.0000	0.0000	.41
16	0.0000	0.0000	.0072	.0324	.1223	.1835	.1799	.3165	.1511	.0072	0.0000	0.0000	0.0000	.44
24	0.0000	0.0000	.0072	.0361	.1625	.1733	.1877	.3249	.1047	.0036	0.0000	0.0000	0.0000	.46
32	.0025	0.0000	.0025	.0200	.1200	.1450	.1700	.4300	.1050	.0050	0.0000	0.0000	0.0000	.43
40	0.0000	0.0000	.0059	.0354	.1535	.1732	.2598	.3425	.0276	.0020	0.0000	0.0000	0.0000	.47
48	.0033	0.0000	.0066	.0559	.1513	.2171	.3092	.2336	.0230	0.0000	0.0000	0.0000	0.0000	.49
56	0.0000	.0039	.0157	.0945	.1969	.3071	.2441	.1299	.0079	0.0000	0.0000	0.0000	0.0000	.54
64	0.0000	.0025	.0370	.1235	.3309	.3136	.1383	.0494	.0049	0.0000	0.0000	0.0000	0.0000	.60
72	0.0000	.0045	.0270	.1306	.3829	.2207	.1306	.0656	.0135	.0045	0.0000	0.0000	0.0000	.59
80	.0027	.0027	.0398	.2467	.3793	.1459	.1061	.0610	.0133	.0027	0.0000	0.0000	0.0000	.63
88	.0024	.0048	.0797	.2995	.2874	.1401	.0990	.0773	.0097	0.0000	0.0000	0.0000	0.0000	.65
96	0.0000	0.0000	.0505	.1448	.1953	.1684	.1818	.2189	.0494	0.0000	0.0000	0.0000	0.0000	.54
104	0.0000	.0039	.0391	.1055	.1758	.1914	.1992	.2422	.0391	.0039	0.0000	0.0000	0.0000	.52
112	0.0000	0.0000	.0290	.0766	.1656	.2070	.2381	.2402	.0414	.0021	0.0000	0.0000	0.0000	.51

<u>TABLE C-2</u>

FLUIDIZED BED DATA

RUN 71

CCT (HOURS)	FLUIDIZED DENSITY (G/ML)	WEIGHT (KG)	HEIGHT (CM)	MMPD (MM)	FLUIDIZING VELOCITIES BELOW FUEL NOZ (M/S)	ABOVE FUEL NOZ (M/S)	TOP OF BED (M/S)	THEORETICAL INCIPIENT (M/S)	REPLACEMENT TIME - 90% (HOURS)
4	.95	63.2	75.4	.41	.22	.42	.68	.07	76.
8	1.00	63.2	71.6	.41	.25	.47	.94	.07	78.
12	1.05	65.9	71.1	.43	.24	.45	.86	.07	89.
16	1.10	69.9	72.0	.44	.24	.45	.86	.08	119.
20	1.13	72.6	73.2	.45	.24	.45	.89	.08	74.
24	1.15	72.6	71.6	.46	.25	.47	.94	.08	62.
28	1.15	72.6	71.6	.45	.25	.45	.90	.08	57.
32	1.15	73.9	72.9	.43	.25	.45	.88	.08	55.
36	1.13	73.9	74.5	.45	.24	.45	.86	.09	60.
40	1.13	76.6	77.2	.47	.24	.44	.84	.09	69.
44	1.13	79.3	79.9	.48	.24	.44	.86	.10	50.
48	1.13	79.3	79.9	.49	.24	.44	.91	.10	50.
52	1.13	84.7	85.3	.52	.24	.43	.72	.11	67.
56	1.15	82.0	80.8	.54	.24	.43	.81	.12	56.
60	1.15	80.6	79.5	.57	.26	.44	.83	.14	53.
64	1.15	80.6	79.5	.60	.26	.44	.84	.15	52.
68	1.15	80.6	79.5	.59	.26	.43	.86	.15	54.
72	1.18	87.4	84.3	.59	.25	.42	.86	.14	85.
76	1.15	84.7	83.5	.61	.25	.43	.82	.15	46.
80	1.15	82.0	80.8	.63	.31	.44	.91	.16	42.
84	1.13	79.3	79.9	.64	.32	.45	.92	.16	43.
88	1.18	80.6	77.8	.65	.31	.45	.91	.17	52.
92	1.15	80.6	79.5	.59	.36	.50	1.00	.14	50.
96	1.18	80.6	77.8	.54	.33	.46	1.00	.12	51.
100	1.15	79.3	78.2	.53	.33	.46	.91	.11	51.
104	1.13	76.6	77.2	.52	.33	.46	.94	.11	50.
108	1.15	76.6	75.5	.52	.32	.44	.91	.11	53.
112	1.15	86.0	84.8	.51	.32	.45	.93	.11	155.

TABLE C-3

SOLIDS PRODUCTION DATA

RUN 71

CCT (HOURS)	FUEL NAR	WASTE NAR	FLUIDIZING VELOCITY (M/S)	PRODUCT RATE (KG/HR)	FINES RATE (KG/HR)	FINES TO SCRUB (KG/HR)	TOTAL SOLIDS RATE (KG/HR)	THEORY SOLIDS RATE (KG/HR)	PROD/FINE RATIC	ATTRITION INDEX	CYC EFF (%)	CUMULATIVE VCLUME REDUCTION
4	2180.	658.	.22	1.86	.98	.17	3.02	2.70	1.92	28.	85.	8.6
8	1901.	362.	.25	1.88	.88	.17	2.92	5.90	2.14	28.	84.	14.2
12	1815.	394.	.24	1.70	1.15	.08	2.93	5.00	1.48	28.	94.	15.4
16	1815.	377.	.24	1.35	1.33	-.01	2.67	4.95	1.02	28.	0.	16.4
20	1811.	350.	.24	2.25	1.15	-.00	3.40	5.52	1.96	32.	0.	16.0
24	1900.	373.	.25	2.70	.75	.00	3.45	5.28	3.60	35.	100.	15.7
28	1855.	349.	.25	2.93	.88	-.01	3.79	3.04	3.34	40.	0.	15.0
32	1871.	391.	.25	3.10	.58	-.02	3.65	5.19	5.39	44.	0.	14.8
36	1792.	368.	.24	2.85	.83	.05	3.72	5.26	3.45	46.	94.	15.0
40	1784.	344.	.24	2.55	.28	.12	2.95	5.57	9.27	47.	69.	15.6
44	1784.	326.	.24	3.63	.30	.09	4.01	5.45	12.08	49.	78.	14.8
48	1788.	303.	.24	3.63	1.20	.05	4.87	4.77	3.02	50.	96.	12.5
52	1756.	508.	.24	2.93	1.30	.04	4.26	3.13	2.25	53.	97.	13.1
56	1743.	370.	.24	3.35	1.53	.03	4.90	3.62	2.20	56.	98.	13.4
60	1894.	374.	.26	3.53	.03	.06	3.61	3.62	141.00	59.	30.	13.4
64	1920.	367.	.26	3.60	.05	.09	3.74	3.67	72.00	62.	37.	13.3
68	1897.	339.	.26	3.43	.13	.13	3.68	3.80	27.40	62.	48.	13.5
72	1884.	327.	.25	2.36	.10	.18	2.66	4.08	23.75	61.	35.	13.7
76	1908.	375.	.25	4.23	1.45	.18	5.85	3.60	2.91	61.	89.	13.5
80	1943.	368.	.31	4.48	.18	.17	4.82	3.74	25.57	60.	51.	13.6
84	1988.	386.	.32	4.20	.10	.04	4.34	3.66	42.00	59.	70.	13.2
88	2019.	380.	.31	3.60	.23	-.08	3.74	3.70	16.00	57.	0.	13.0
92	2294.	460.	.36	3.70	.48	-.01	4.17	3.55	7.79	56.	0.	12.8
96	2048.	329.	.33	3.68	.45	.07	4.19	3.60	8.17	54.	87.	12.6
100	2120.	437.	.33	3.58	1.45	.01	5.03	3.13	2.47	53.	99.	12.5
104	2092.	390.	.33	3.50	.60	-.05	4.05	3.60	5.83	52.	0.	12.5
108	2043.	392.	.32	3.33	.28	.01	3.61	3.68	12.09	51.	96.	12.7
112	2032.	388.	.32	1.28	.38	.07	1.72	3.75	3.40	50.	83.	13.2
TOTAL				340.7	76.0	6.2	423.	466.	4.48			

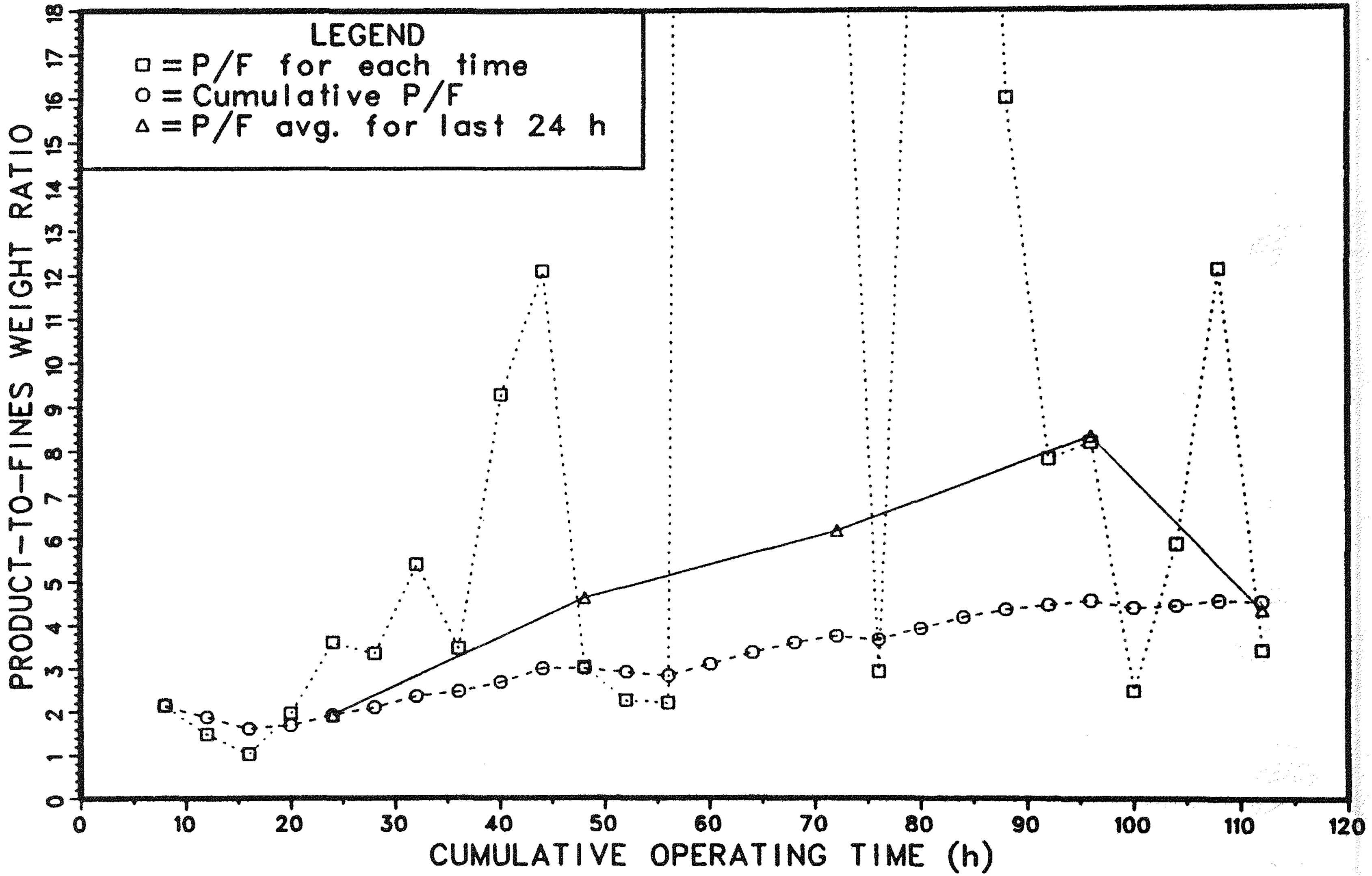

FIGURE C-1. VARIATION OF PRODUCT-TO-FINES RATIO DURING RUN 71

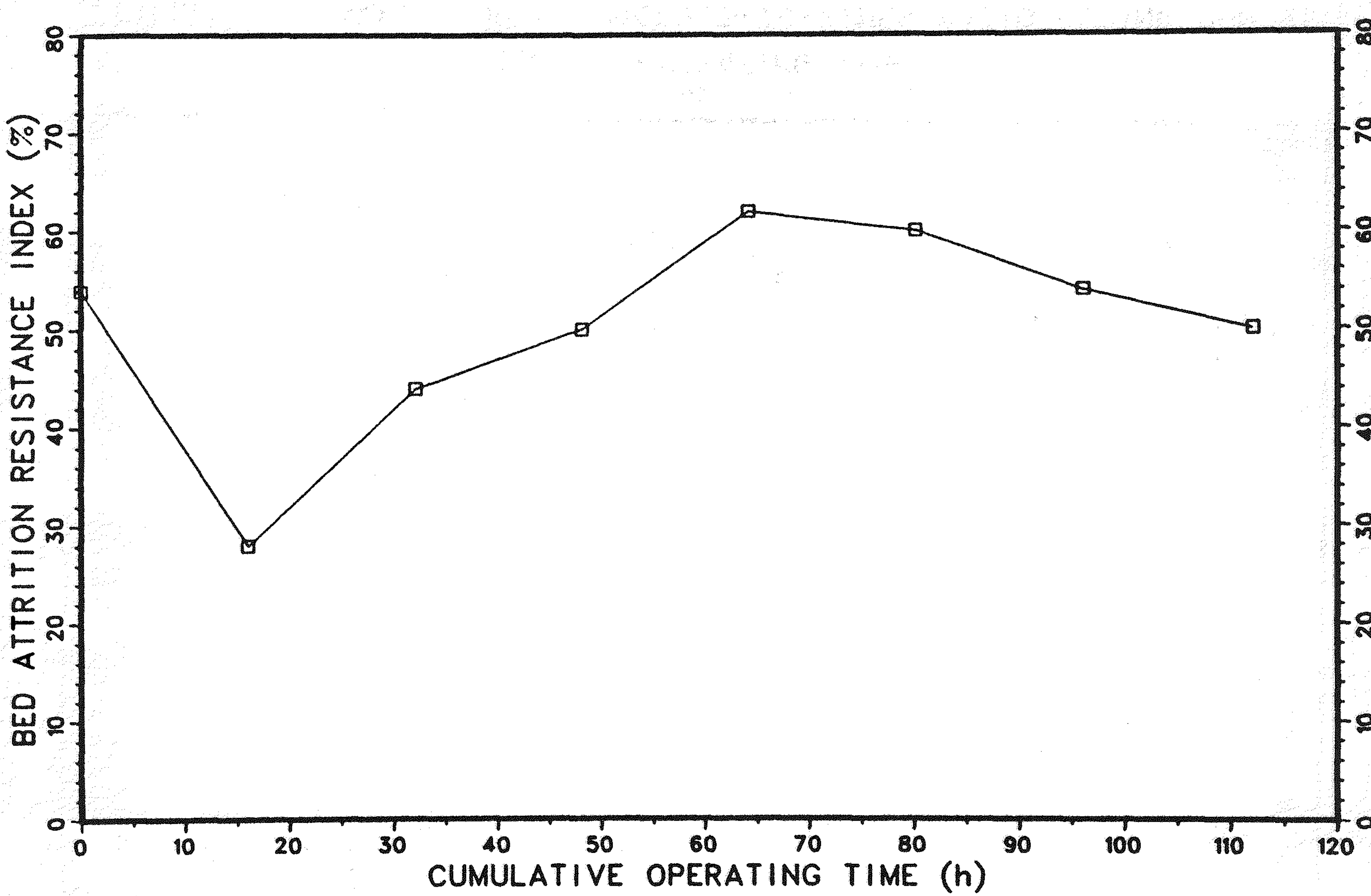

FIGURE C-2. ATTRITION INDEX OF BED PRODUCED DURING RUN 71

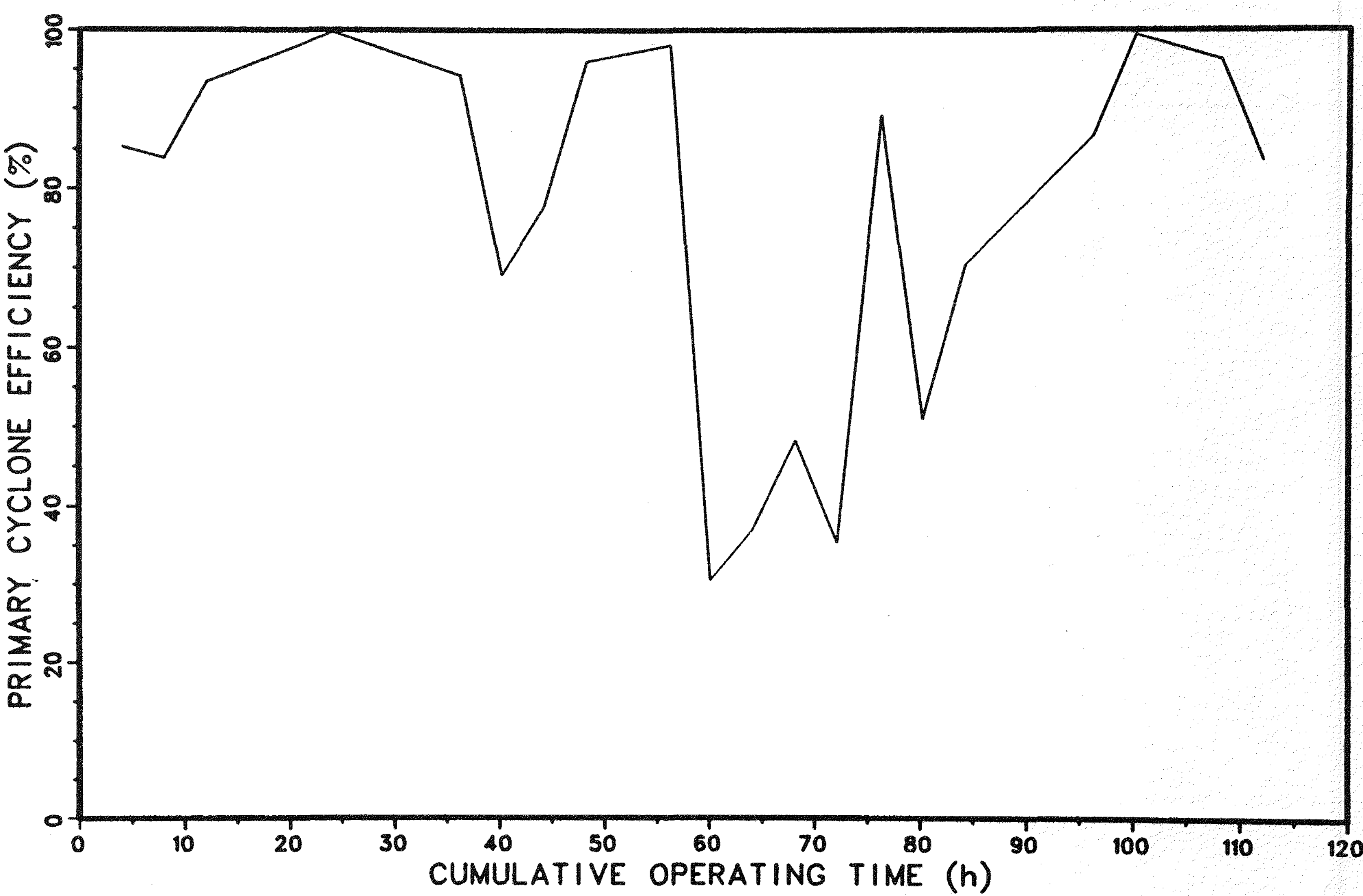

FIGURE C-3. CYCLONE EFFICIENCY MEASURED DURING RUN 71

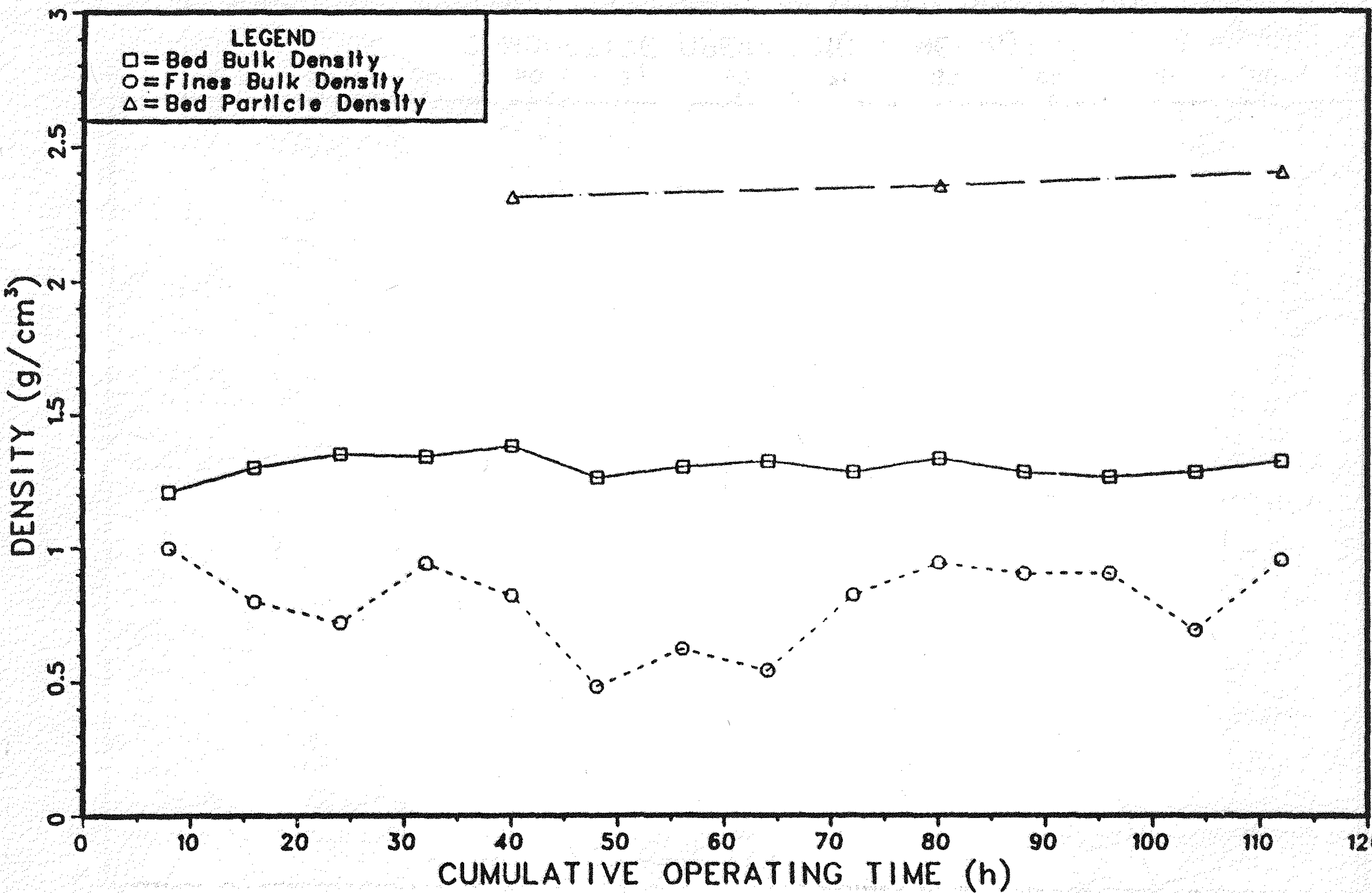

FIGURE C-4. DENSITY OF SOLIDS PRODUCED DURING RUN 71

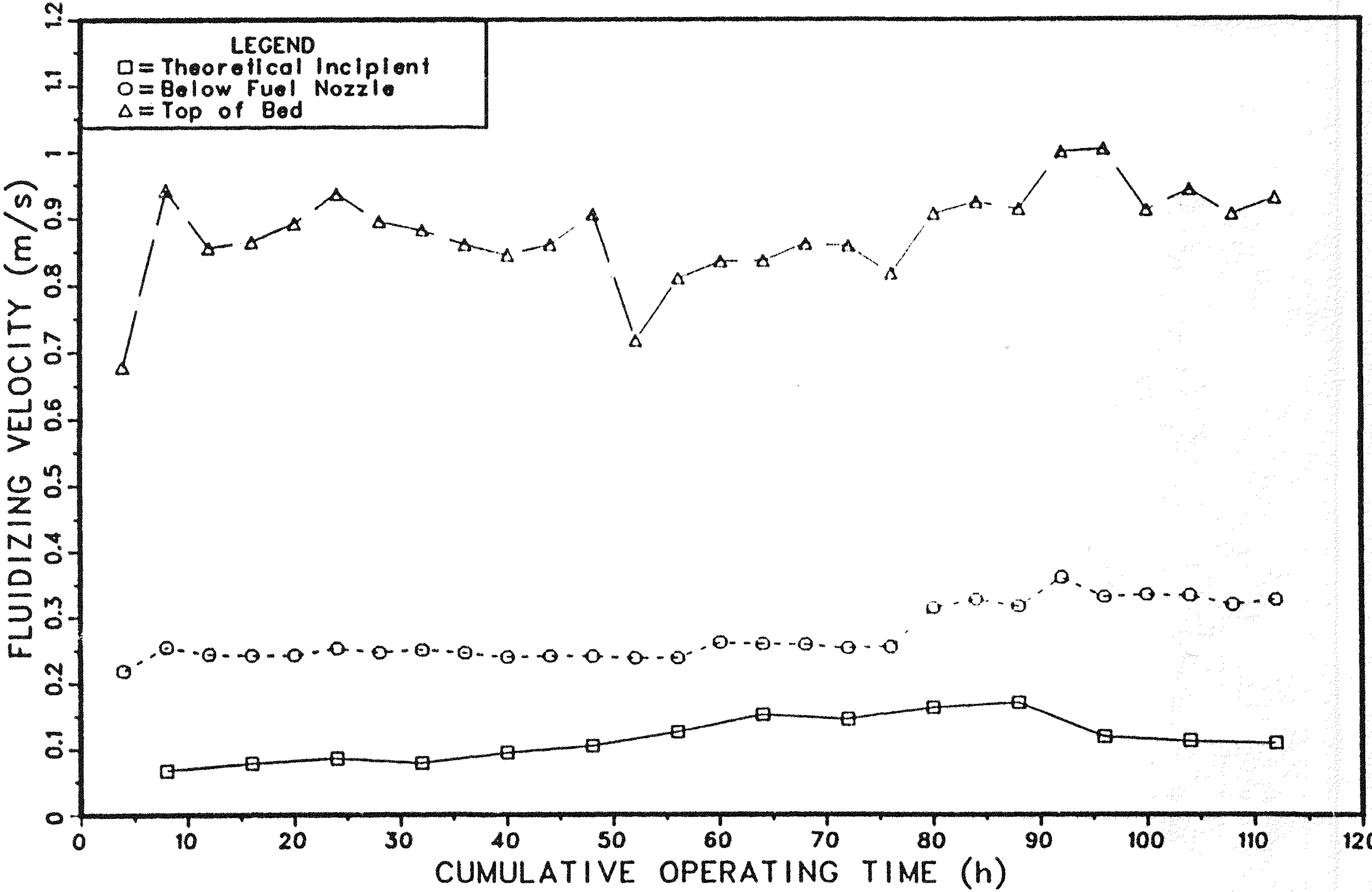

FIGURE C-5 INCIPIENT FLUIDIZING VELOCITY COMPARED TO FLUIDIZING VELOCITIES USED DURING RUN 71

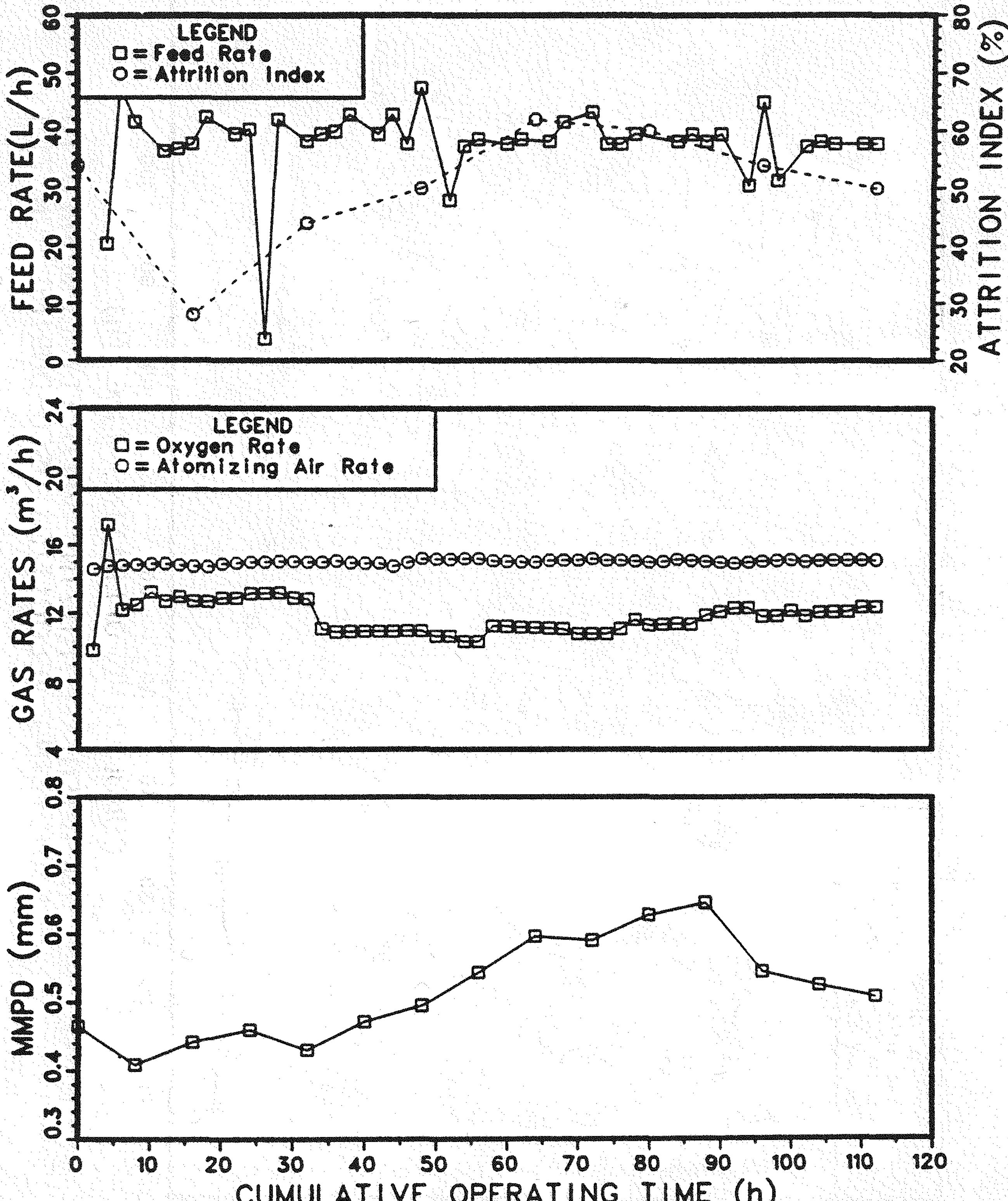

FIGURE C-6. EFFECT OF VARIOUS OPERATING CONDITIONS ON BED PARTICLE SIZE DURING RUN 71

APPENDIX D

ANALYSES OF SAMPLES TAKEN DURING RUN 71

TABLE D-1

ANALYSES OF RUN 71 FEED

Calciner Feed Concentration

COT (h)	H^+ (M)	F (M)	NO_3^- (M)	Aℓ (M)	Na (M)	Zr (M)	Ca (M)	Cℓ (μg/mL)	SO_4 (M)	B (M)	Hg (μg/mL)	Sp. Gr. (g/mL)	Mole Ratios Ca/F	Ca/Na	Aℓ/F
1	0.60	0.89	3.15	0.32	0.38	0.43	0.58	381	0.075	0.082	541	1.218	0.652	1.53	0.36
48	0.50	0.88	3.13	0.30	0.25	0.16	0.60	465	0.066	0.084	496	1.199	0.682	2.38	0.34
108	0.56	1.06	3.39	0.41	0.32	0.13	0.58	528	0.054	0.095	541	1.220	0.547	1.83	0.39

TABLE D-2

COMPOSITION OF FINES PRODUCED DURING RUN 71

COT (h)	F	Na	Zr	Ca	SO_4 (wt. %)	Aℓ	Cℓ	NO_3	Hg	Bulk Density (g/cm^3)
8	(a)	3.40	3.20	(a)	2.30	17.00	(a)	5.08	(a)	1.000
40	10.60	5.10	7.60	24.00	4.10	8.20	0.62	13.53	0.0020	0.820
80	8.29	5.30	8.10	27.00	5.60	6.90	0.49	12.51	0.0020	0.940
112	8.13	5.80	7.70	30.00	5.30	6.10	0.50	10.32	0.0015	0.950

(a) not analyzed

TABLE D-3

COMPOSITION OF PRODUCT PRODUCED DURING RUN 71

COT (h)	F	Na	Zr	Ca	SO_4 (wt. %)	Aℓ	Cℓ	NO_3	Hg	Densities (g/cm^3) Part.	Densities (g/cm^3) Bulk	BED VOID
0	1.50	(a)	(a)	(a)	0.57	(a)	(a)	(a)	0.0020	(a)	(a)	(a)
40	10.50	4.70	6.60	30.00	4.30	9.90	0.28	8.35	0.00002	3.05	1.38	0.55
80	13.50	4.20	8.80	31.00	4.20	6.80	0.27	8.17	0.00001	3.20	1.33	0.58
112	9.34	4.20	9.30	27.00	4.30	6.60	0.25	9.11	0.00001	3.20	1.32	0.59

(a) not analyzed

TABLE D-4

COMPOSITION OF ACID SCRUB HOLD TANK SOLUTION DURING RUN 71

COT (h)	F	Na	Zr	Ca	B	Aℓ (M)	NO_3	H+	Cℓ	SO_4 (mg/L)	Hg
48	0.13	0.080	0.012	0.100	0.031	0.052	1.96	1.09	287	1,030	852
88	0.14	0.083	0.012	0.105	0.029	0.052	2.03	1.12	280	1,130	763

TABLE D-5

COMPOSITION OF SCRUB SOLUTIONS DOWNSTREAM FROM NITRIC ACID SCRUBBER DURING RUN 71

COT (h)	F	Cℓ	Ca	Aℓ (mg/L)	Hg	SO_4	Scrubber Volume (b) (mL)
				First Caustic Scrubber			
20	23.4	18	17	5.6	16	<20	1,470
60	18.6	34	17	3.7	28	<20	1,770
100	89.1	3.1	4.6	<3.0	11	<20	1,250
				Second Caustic Scrubber			
36	<11	1.04	(a)	(a)	19	<20	520
				Hydrochloric Acid Scrubber			
60	(a)	(a)	(a)	(a)	95	(a)	490

(a) Not analyzed.
(b) At end of 8-h operating period.

APPENDIX E

RUN 71 TEMPERATURE AND PRESSURE DATA

TABLE E-1

RUN 71 THERMOCOUPLE LOCATIONS

TR901 THERMOCOUPLE LOCATIONS

1 -- OUTLET OF FEED COOLER
2 -- OUTLET OF SCRUB EM FLOWMETER
3 -- FEED ATOMIZING AIR
4 -- OUTLET OF VENTURI SCRUBBER
5 -- OUTLET OF QUENCH TOWER
6 -- INLET OF AIR PREHEATER
7 -- WATER OUTLET OF JET COND.
8 -- FUEL ATOMIZING O2
9 -- OUTLET OF PARTIAL COND.
10 -- FUEL
11 -- AMBIENT AIR
12 -- OUTLET OF DEMISTER
13 -- OUTLET OF HEAT EXCHANGER
14 -- SILICA GEL ADSORBER
15 -- INLET TO FINAL FILTER

TR902 THERMOCOUPLE LOCATIONS

1 -- PRODUCT POT
2 -- AMBIENT F OF FINES POT
3 -- PRIMARY CYCLONE OUTLET
4 -- VESSEL 2.2 METERS E WALL
5 -- VESSEL 1.87 METER E WALL
6 -- VESSEL 1.56 METERS E WALL
7 -- VESSEL 1.25 METERS E WALL
8 -- BED 78.7 CM W WALL
9 -- BED 78.7 CM S WALL
10 -- VESSEL 78.7 CM N WALL
11 -- BED 11.4 CM N WALL
12 -- VAPOR 97 CM E WALL
13 -- VAPOR 2.1 METER E WALL
14 -- VAPOR 1.79 METERS E WALL
15 -- FLUID AIR PREHEATER OUTLET
16 -- FINES POT INLET
17 -- VAPOR 1.49 METERS E WALL
18 -- BED 58.4 CM N WALL
19 -- VAPOR 1.26 METER W WALL
20 -- BED 11.4 CM S WALL
21 -- VESSEL 11.4 W WALL
22 -- BED 11.4 CM E WALL
23 -- BED 58.4 CM S WALL

TABLE E-2A

RUN 71 THERMOCOUPLE READINGS

INPUT DATA - TR901(1-15)   ALL UNITS ARE DEGREES C

LS	COT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	TIC 903	TIC 904
31	4	48	69	37	82	95	193	15	26	80	28	29	25	26	21	23	37	232
30	8	24	27	34	77	106	166	10	23	31	26	23	23	23	22	22	37	204
30	12	31	40	31	80	119	163	10	25	32	26	24	24	23	23	22	37	204
30	16	22	40	37	80	139	162	11	26	33	27	26	23	23	24	23	37	210
30	20	25	39	39	80	138	166	11	27	32	28	26	24	26	26	24	37	210
30	24	19	38	35	78	149	164	10	23	31	25	25	23	22	22	21	37	210
30	28	35	37	33	77	147	164	10	22	32	24	23	21	21	20	20	37	215
30	32	23	31	34	77	152	164	11	23	33	25	24	22	22	21	21	37	215
30	36	19	29	28	78	145	156	11	22	31	27	25	23	22	21	21	37	210
30	40	19	38	30	79	143	156	11	21	31	25	23	22	20	20	19	37	204
30	44	20	39	29	79	139	154	11	20	32	24	20	19	19	18	19	37	204
30	48	18	44	27	81	142	152	11	19	32	23	19	18	18	17	17	37	204
30	52	19	43	28	80	140	157	10	21	31	26	20	19	20	16	17	37	204
30	56	18	44	26	82	134	149	10	19	32	24	19	17	17	16	16	37	204
30	60	18	44	29	79	132	145	10	21	32	28	19	20	21	17	17	37	198
30	64	20	46	31	80	137	151	10	26	32	30	24	23	23	22	21	37	204
30	68	18	45	30	80	139	158	10	26	31	30	23	23	23	23	23	37	204
30	72	21	45	30	80	141	149	11	26	33	30	24	24	25	23	23	37	204
30	76	19	45	30	79	132	148	10	26	32	30	23	24	24	22	23	37	204
30	80	24	44	34	78	141	141	10	27	32	31	25	25	25	24	24	37	204
30	84	22	29	31	74	131	140	10	21	30	26	25	25	25	24	20	37	198
30	88	24	29	36	75	135	145	11	24	31	30	27	27	27	25	24	37	204
30	92	27	29	41	77	139	151	11	28	32	35	29	29	29	26	28	37	204
30	96	22	29	36	76	156	150	11	26	32	31	26	26	25	24	24	37	204
30	100	23	28	32	74	161	150	10	22	30	27	23	22	21	21	21	37	204
30	104	21	29	34	74	138	147	10	24	31	29	21	23	23	21	21	37	204
30	108	19	23	33	75	162	145	10	24	31	29	25	23	22	21	21	37	198
39	112	19	28	33	75	146	158	10	23	30	28	25	23	22	22	21	37	204

TABLE E-2B

RUN 71 THERMOCOUPLE READINGS

INPUT DATA - TR9C2(1-23)   ALL UNITS ARE DEGREES C

LS	COT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	DORIC	TRC905
41	4	284	19	364	323	378	331	424	490	495	430	488	427	397	438	95	96	453	494	475	486	443	485	496	500	504
40	8	293	17	378	317	370	318	404	491	491	428	489	422	388	432	91	81	442	490	466	490	447	486	493	499	500
40	12	290	23	379	322	376	322	404	495	495	431	493	424	393	435	93	85	444	496	468	493	452	488	498	502	500
40	16	288	23	375	320	375	320	404	493	495	430	492	425	393	434	94	55	443	495	445	492	451	487	496	500	498
40	20	332	25	376	323	378	323	407	493	495	432	492	427	394	435	94	64	444	496	472	493	454	490	497	499	498
40	24	345	21	379	325	379	321	408	494	497	431	493	426	397	437	97	45	445	497	472	492	453	490	500	501	500
40	28	319	15	370	319	373	315	403	493	494	427	491	424	390	430	90	37	441	494	468	493	451	488	493	499	501
40	32	297	17	370	319	373	315	406	492	493	428	491	424	390	428	90	62	440	492	468	490	446	485	489	502	500
40	36	350	20	360	307	355	307	400	492	497	490	490	425	418	412	87	55	430	495	465	490	45[illegible]	485	498	499	496
40	40	312	20	353	302	349	305	403	490	494	430	487	425	374	412	86	57	432	495	464	487	441	480	495	500	497
40	44	335	17	349	302	311	307	405	492	496	430	487	427	375	415	85	53	437	495	466	490	435	481	497	501	496
40	48	335	16	341	299	347	305	402	489	493	428	464	426	373	415	87	54	431	493	462	487	423	478	494	498	493
40	52	287	13	345	295	345	304	409	489	493	426	485	426	370	410	76	31	433	491	464	482	397	475	494	500	496
40	56	294	12	344	290	343	298	395	487	492	427	479	425	365	408	78	67	425	494	458	480	363	471	493	498	496
40	60	370	13	355	295	349	303	400	497	497	430	490	426	370	411	85	45	430	494	460	485	365	475	498	501	497
40	64	305	23	352	293	347	302	395	496	497	429	478	425	369	408	86	47	427	497	458	487	340	477	497	501	498
40	68	349	23	353	293	345	301	395	496	498	430	490	425	368	407	85	53	425	498	460	487	326	471	497	502	498
40	72	327	24	35[illegible]	294	346	304	400	494	495	430	435	429	368	410	85	80	428	496	461	478	323	460	494	499	493
40	76	324	18	343	290	344	302	397	497	494	426	458	425	367	407	80	53	428	488	464	463	309	455	492	499	497
40	80	305	20	350	294	345	300	392	488	492	427	478	425	369	410	84	50	424	495	454	483	337	478	492	502	497
40	84	318	22	352	296	347	303	397	492	494	428	483	425	373	412	84	52	425	492	457	488	352	480	495	499	496
40	88	367	24	351	298	348	304	397	492	494	430	485	426	374	411	90	60	429	495	461	487	364	478	497	500	498
40	92	339	27	356	302	352	307	404	494	494	432	494	425	376	414	94	52	433	494	468	488	384	481	494	500	495
40	96	350	22	364	303	350	306	405	493	494	431	488	428	377	415	95	91	432	495	465	488	415	484	495	501	496
40	100	344	15	356	300	347	304	404	491	491	424	488	425	374	382	90	79	429	492	462	485	412	485	495	502	497
40	104	300	18	352	296	343	300	395	490	490	426	487	423	370	408	90	70	425	489	458	484	405	479	490	496	495
40	108	370	22	350	297	341	302	398	402	402	430	490	425	374	410	94	76	430	495	467	491	420	480	495	498	497
49	112	296	22	357	304	351	311	416	493	492	430	490	429	380	420	96	81	439	495	473	486	420	482	495	498	496

TABLE E-3

RUN 71 PRESSURE GAUGE LOCATIONS

PRESSURE GUAGE LOCATIONS

PI901 -- STEAM TC JET	PI902 -- FEED SUPPLY	PI903 -- SCRUB SUPPLY	PI904 -- SCRUB TANK GUAGE PRES.
PI905 -- FUEL SUPPLY	PI906 -- FUEL NOZZLE LIQUID	PI907 -- OXYGEN SUPPLY	PI908 -- FEED NOZZLE LIQUID
PI909 -- FEED NOZZLE GAS	PI910 -- FLUIDIZING AIR PREHTR INLT	PI911 -- FLUIDIZING AIR SUPPLY	PI912 -- QUENCH TWR INLET (ABS)
PI913 -- VENT SCRB EXIT (ABS)	PI914 -- KNOCKOUT POT EXIT (ABS)	PI915 -- FEED ATOM AIR SUPPLY	PI917 -- PARTIAL COND EXIT (ABS)
PI918 -- STEAM JET INLET (ABS)	PI919 -- FUEL NOZZLE GAS	PI920 -- ATMOSPHERIC PRESSURE	PI922 -- INSTRUMENT AIR
PI923 -- 5C PSI AIR			

TABLE E-4A

RUN 71 PRESSURE GAUGE READINGS

INPUT DATA - PI(1-11), PSI AND KPA

LS	COT	901		902		903		904		905		906		907		908		909		910		911	
51	4	0.0	0.	9.4	65.	28.0	193.	3.1	21.	71.2	491.	5.0	34.	83.2	573.	8.5	59.	53.5	369.	4.5	31.	100.0	689.
50	8	0.0	0.	7.6	52.	27.0	186.	1.2	8.	72.0	496.	2.0	14.	83.5	575.	5.5	38.	53.0	365.	3.0	21.	100.0	689.
50	12	0.0	0.	9.0	62.	29.5	203.	1.8	12.	72.5	500.	3.0	21.	83.5	575.	5.5	38.	52.0	358.	3.7	25.	100.0	689.
50	16	0.0	0.	8.7	60.	27.5	190.	1.8	12.	72.5	500.	4.0	28.	84.0	579.	5.5	38.	52.0	358.	3.7	25.	100.0	689.
50	20	0.0	0.	6.0	41.	27.5	190.	2.0	14.	72.5	500.	2.0	14.	83.0	572.	5.5	38.	50.0	345.	3.8	26.	100.0	689.
50	24	0.0	0.	5.5	38.	27.5	190.	1.5	10.	72.5	500.	2.0	14.	82.1	566.	5.5	38.	51.0	351.	3.4	23.	100.0	689.
50	28	2.0	14.	7.2	50.	28.0	193.	1.4	10.	72.5	500.	2.0	14.	82.4	568.	5.6	39.	49.0	338.	3.3	23.	100.0	689.
50	32	2.0	14.	7.7	53.	27.5	190.	1.4	10.	72.6	500.	2.0	14.	81.5	562.	5.2	36.	53.0	365.	3.1	21.	100.0	689.
50	36	4.0	28.	6.8	47.	28.2	194.	1.5	10.	72.5	500.	2.0	14.	84.5	582.	5.0	34.	52.0	358.	3.4	23.	100.0	689.
50	40	4.0	28.	6.8	47.	29.5	203.	1.8	12.	72.5	500.	3.0	21.	85.0	586.	5.1	35.	51.0	351.	3.7	25.	100.0	689.
50	44	0.0	0.	6.0	41.	30.0	207.	2.0	14.	72.8	502.	1.0	7.	84.8	584.	5.2	36.	51.0	351.	3.7	25.	100.0	689.
50	48	2.0	14.	5.5	38.	29.0	200.	2.0	14.	72.9	502.	1.0	7.	84.9	585.	5.0	34.	48.5	334.	3.9	27.	100.0	689.
50	52	3.0	21.	6.9	48.	30.0	207.	2.0	14.	72.8	502.	1.0	7.	85.3	588.	4.8	33.	50.5	348.	4.0	28.	100.0	689.
50	56	3.0	21.	6.0	41.	29.0	200.	2.1	14.	73.0	503.	1.0	7.	85.0	586.	4.9	34.	50.5	348.	4.0	28.	100.0	689.
50	60	4.0	28.	6.2	43.	29.5	203.	2.1	14.	72.7	501.	3.0	21.	85.0	586.	5.1	35.	50.5	348.	4.0	28.	100.0	689.
50	64	4.0	28.	6.2	43.	29.5	203.	1.9	13.	72.6	500.	3.0	21.	85.2	587.	5.5	38.	51.0	351.	4.0	28.	100.0	689.
50	68	4.0	28.	6.0	41.	30.0	207.	1.9	13.	73.0	503.	3.0	21.	84.1	580.	5.0	34.	48.0	331.	4.0	28.	100.0	669.
50	72	4.0	28.	6.0	41.	30.0	207.	1.9	13.	73.0	503.	3.0	21.	84.0	579.	5.2	36.	49.0	338.	4.0	28.	100.0	689.
50	76	3.0	21.	6.0	41.	28.0	193.	2.1	14.	72.8	502.	1.0	7.	84.2	580.	5.4	37.	50.0	345.	4.0	28.	100.0	689.
50	80	3.0	21.	9.0	62.	28.0	193.	1.8	12.	73.0	503.	1.0	7.	83.2	573.	5.4	37.	47.0	324.	4.0	28.	100.0	689.
50	84	3.0	21.	8.9	61.	30.0	207.	1.6	11.	72.7	501.	3.0	21.	83.0	572.	5.3	37.	49.8	343.	4.0	28.	100.0	689.
50	88	3.0	21.	6.1	42.	30.0	207.	1.7	12.	72.7	501.	3.0	21.	83.0	572.	5.3	37.	46.5	320.	4.0	28.	100.0	689.
50	92	0.0	0.	8.2	57.	30.0	207.	1.7	12.	73.0	503.	0.0	0.	82.5	569.	5.4	37.	47.0	324.	4.0	28.	100.0	689.
50	96	0.0	0.	7.6	52.	30.0	207.	1.7	12.	73.0	503.	0.0	0.	82.5	569.	5.4	37.	47.5	327.	4.0	28.	100.0	689.
50	100	2.0	14.	6.0	41.	30.0	207.	1.5	10.	72.8	502.	1.0	7.	82.2	566.	5.4	37.	46.5	320.	3.9	27.	100.0	689.
50	104	3.0	21.	6.7	46.	30.0	207.	1.6	11.	72.8	502.	1.0	7.	81.5	562.	5.5	38.	49.5	341.	3.8	26.	100.0	689.
50	108	2.0	14.	6.3	43.	30.0	207.	1.5	10.	72.5	500.	3.0	21.	82.0	565.	5.5	38.	47.0	324.	3.7	25.	100.0	689.
59	112	3.0	21.	6.0	41.	30.0	207.	1.6	11.	72.5	500.	3.0	21.	82.0	565.	11.0	76.	47.0	324.	4.0	28.	100.0	689.

<u>TABLE E-4B</u>

RUN 71 PRESSURE GAUGE READINGS

INPUT DATA - PI(12-23) PSI AND KPA

LS	COT	912		913		914		915		917		918		919		920		922		923	
59	4	15.4	106.	13.4	92.	13.3	92.	100.0	689.	13.2	91.	12.9	89.	40.0	276.	12.3	85.	20.5	141.	49.0	338.
59	8	13.4	92.	12.7	88.	12.7	88.	100.0	689.	13.2	91.	12.8	88.	36.0	248.	12.3	85.	20.4	141.	49.0	338.
59	12	14.8	102.	13.4	92.	13.4	92.	100.0	689.	13.2	91.	13.1	90.	36.0	248.	12.3	85.	21.0	145.	49.0	338.
59	16	14.2	98.	13.5	93.	13.4	92.	100.0	689.	13.2	91.	13.1	90.	37.0	255.	12.3	85.	21.0	145.	49.0	338.
59	20	14.2	98.	13.4	92.	13.4	92.	100.0	689.	13.2	91.	13.1	90.	38.0	262.	12.3	85.	20.5	141.	48.0	331.
59	24	13.7	94.	12.8	88.	12.8	88.	100.0	689.	13.2	91.	12.9	89.	38.0	262.	12.3	85.	20.5	141.	49.0	338.
59	28	13.6	94.	12.7	88.	12.7	88.	100.0	689.	13.2	91.	12.7	88.	38.0	262.	12.3	85.	20.4	141.	49.0	338.
59	32	13.6	94.	12.8	88.	12.7	88.	100.0	689.	13.2	91.	12.7	88.	37.0	255.	12.3	85.	20.5	141.	49.0	338.
59	36	13.8	95.	13.1	90.	13.1	90.	100.0	689.	13.2	91.	12.7	88.	32.0	221.	12.3	85.	20.5	141.	48.0	331.
59	40	14.1	97.	13.5	93.	13.5	93.	100.0	689.	13.2	91.	13.1	90.	32.0	221.	12.3	85.	20.5	141.	48.0	331.
59	44	14.2	98.	13.5	93.	13.5	93.	100.0	689.	13.2	91.	13.1	90.	31.0	214.	12.3	85.	20.5	141.	48.0	331.
59	48	14.3	99.	13.6	94.	13.6	94.	100.0	689.	13.1	90.	13.1	90.	29.0	200.	12.3	85.	20.5	141.	48.0	331.
59	52	14.3	99.	13.6	94.	13.6	94.	100.0	689.	13.2	91.	13.6	94.	28.0	193.	12.3	85.	20.4	141.	48.5	334.
59	56	14.4	99.	13.7	94.	13.7	94.	100.0	689.	13.2	91.	13.2	91.	30.0	207.	12.3	85.	20.4	141.	48.0	331.
59	60	14.3	99.	13.5	93.	13.5	93.	100.0	689.	13.2	91.	13.2	91.	32.0	221.	12.3	85.	20.5	141.	48.0	331.
59	64	14.3	99.	13.6	94.	13.6	94.	100.0	689.	13.2	91.	13.5	93.	32.0	221.	12.3	85.	20.5	141.	48.0	331.
59	68	14.4	99.	13.6	94.	13.6	94.	100.0	689.	13.2	91.	13.5	93.	32.0	221.	12.3	85.	20.5	141.	48.8	336.
59	72	14.5	100.	13.6	94.	13.6	94.	100.0	689.	13.2	91.	13.5	93.	32.0	221.	12.3	85.	20.5	141.	48.7	336.
59	76	14.4	99.	13.6	94.	13.6	94.	100.0	689.	13.2	91.	13.6	94.	30.0	207.	12.3	85.	20.4	141.	48.5	334.
59	80	14.1	97.	13.2	91.	13.1	90.	100.0	689.	13.2	91.	13.4	92.	32.0	221.	12.3	85.	20.4	141.	48.5	334.
59	84	13.9	96.	13.0	90.	13.0	90.	100.0	689.	13.2	91.	13.2	91.	34.0	234.	12.3	85.	20.5	141.	48.5	334.
59	88	14.0	96.	13.0	90.	13.0	90.	100.0	689.	13.2	91.	13.2	91.	35.0	241.	12.3	85.	20.5	141.	49.0	338.
59	92	14.0	96.	13.0	90.	12.9	89.	100.0	689.	13.2	91.	13.1	90.	36.0	248.	12.3	85.	20.5	141.	48.5	334.
59	96	13.9	96.	13.1	90.	13.0	90.	100.0	689.	13.2	91.	13.0	90.	36.0	248.	12.3	85.	20.5	141.	48.5	334.
59	100	13.8	95.	12.9	89.	12.8	88.	100.0	689.	13.2	91.	12.8	88.	36.0	248.	12.3	85.	20.4	141.	49.0	338.
59	104	13.8	95.	12.6	88.	12.7	88.	100.0	689.	13.2	91.	12.8	88.	34.0	234.	12.3	85.	20.4	141.	48.5	334.
59	108	13.8	95.	12.7	88.	12.7	88.	100.0	689.	13.2	91.	12.7	88.	37.0	255.	12.3	85.	20.5	141.	49.0	338.
59	112	13.9	96.	12.8	88.	12.7	88.	100.0	689.	13.2	91.	12.8	88.	38.0	262.	12.3	85.	20.5	141.	48.5	334.

APPENDIX F

OPERATING CONDITIONS FOR RUN 74

TABLE F-1

MAJOR FLOWRATES

RUN 74

CCT (HOURS)	FUEL (L/HR)	WASTE (L/HR)	FLUID AIR (M3/HR)	WASTE ATOM. AIR (M3/HR)	FUEL ATOM. O2 (M3/HR)	PURGES (M3/HR)	WET OFFGAS (M3/HR)	DRY OFFGAS (M3/HR)	OFFGAS DEW POINT (C)	SCRUB TO VENT. (L/HR)	VENTURI SCRUB RETURN (L/HR)	COND. TO DRAIN (L/HR)	SCRUB TANK ACC. (L/HR)	SCRUB RECYCLE TO FEED (%)
4	7.8	49.6	33.7	4.71	12.83	.40	122.5	50.2	85.0	40.15	140.00	52.00	-10.16	0.0
8	7.8	43.5	33.6	4.96	12.77	.40	114.9	50.2	83.6	40.15	140.00	52.00	15.60	0.0
12	7.6	46.6	33.5	4.95	12.00	.40	117.9	49.5	84.2	40.15	147.50	51.00	-8.38	0.0
16	7.6	57.2	33.3	4.94	12.10	.40	131.0	49.5	86.2	40.15	155.00	50.00	-3.71	0.0
20	8.0	56.4	33.6	7.22	13.72	.40	135.2	54.2	84.6	40.15	287.50	40.60	14.35	0.0
24	7.4	49.2	33.4	7.19	11.06	.40	122.5	51.3	84.1	39.35	420.00	31.20	-7.26	0.0
28	7.3	48.8	33.5	7.23	10.38	.40	121.2	50.6	84.2	40.15	262.50	37.50	4.35	0.0
32	7.3	46.2	33.5	7.23	10.36	.40	118.1	50.7	83.6	38.55	105.00	43.80	-5.00	0.0
36	7.1	47.9	34.5	7.22	9.75	.40	120.5	51.4	84.2	38.55	95.00	44.90	12.09	0.0
40	7.2	48.3	33.6	7.22	10.37	.40	120.8	51.0	84.4	38.55	85.00	46.00	.81	0.0
44	7.3	44.5	33.2	7.16	10.64	.40	115.8	50.6	83.8	39.35	30.00	48.50	.32	0.0
48	7.3	50.5	33.3	7.18	10.90	.40	123.9	51.4	84.5	39.35	75.00	51.00	-.16	0.0
52	7.3	49.2	34.3	7.18	10.57	.40	122.9	51.9	84.5	39.35	78.00	54.00	-.81	0.0
56	7.3	43.7	33.3	7.17	10.61	.40	114.9	50.7	83.5	39.35	81.00	57.00	-1.77	0.0
60	7.3	44.9	33.3	9.90	10.93	.40	119.5	53.7	82.8	39.35	92.25	40.80	-3.23	0.0
64	7.4	53.8	33.5	9.88	11.25	.40	131.1	54.1	84.7	39.35	103.50	36.60	1.61	0.0
68	7.2	41.6	33.5	9.96	10.33	.40	114.6	53.2	82.3	38.55	94.75	38.10	-3.39	0.0
72	7.4	44.5	33.6	9.98	11.71	.40	120.1	54.7	82.7	39.35	86.00	39.60	6.61	0.0
76	7.4	47.3	33.5	9.96	11.26	.40	123.9	53.8	83.4	39.35	82.60	42.30	-3.55	0.0
80	7.3	40.3	34.4	9.91	10.60	.40	115.0	53.9	82.1	39.35	79.20	45.00	-3.87	0.0
84	7.4	49.4	34.4	9.92	11.79	.40	128.4	55.5	83.7	39.35	141.60	26.40	6.13	0.0
88	7.3	42.8	34.4	9.96	10.91	.40	118.7	54.4	82.2	39.35	204.00	7.80	.97	0.0
92	7.5	44.3	34.5	7.48	12.47	.40	120.1	53.3	83.3	39.35	130.50	18.00	-2.42	0.0
96	7.4	48.8	34.2	7.34	11.07	.40	123.7	51.7	84.7	39.35	57.00	28.20	-3.55	0.0
100	7.2	48.1	34.4	7.37	10.55	.40	122.6	51.7	84.9	39.35	70.50	64.05	14.67	0.0
104	7.3	47.1	34.5	7.23	11.06	.40	121.8	52.1	84.8	39.35	84.00	99.90	-2.26	0.0
108	7.4	48.5	34.4	7.17	11.24	.40	121.7	52.7	84.0	36.94	81.00	76.05	-3.55	0.0
112	7.4	48.3	34.5	7.22	11.42	.40	122.1	53.2	83.0	38.55	78.00	52.20	-7.74	0.0

RECYCLE RATE IS BASED ON FEED WITH CALCIUM (SCRUB/FEED WITH CA + SCRUB)

TABLE F-2

CALCINER DATA

RUN 74

COT (HOURS)	AVERAGE BED PRESSURE (KPA)	AVERAGE BED PRESSURE (PSI)	TEMPERATURE AVERAGE BED (C)	WASTE TO FUEL RATIO	O2 TO FUEL RATIO	O2 INPUT (% TH)	CO (%)	CO2 (%)	COMBUSTION EFFICIENCY (%)
4	104.4	15.1	470.+-49.5	6.36	1644.	82.1	2.65	20.00	94.3
8	104.1	15.1	489.+- 5.5	5.57	1637.	81.8	3.70	17.00	91.2
12	104.3	15.1	478.+-26.0	6.12	1574.	76.8	3.65	17.00	91.3
16	105.3	15.3	489.+- 3.7	7.51	1586.	77.4	3.50	16.20	91.3
20	102.0	14.8	497.+- 3.5	7.07	1721.	87.8	3.40	15.00	90.9
24	103.3	15.0	490.+- 2.5	6.69	1504.	70.8	3.60	15.40	90.7
28	103.3	15.0	490.+- 3.4	6.71	1430.	66.5	3.70	16.80	91.2
32	103.5	15.0	490.+- 3.1	6.36	1426.	66.3	3.70	15.40	90.5
36	104.8	15.2	491.+- 5.6	6.77	1378.	62.4	3.90	16.80	90.8
40	104.8	15.2	491.+- 3.5	6.74	1446.	66.4	3.90	15.60	90.3
44	105.4	15.3	483.+- 4.3	6.13	1465.	68.1	3.35	13.60	90.3
48	104.9	15.2	486.+- 3.2	6.95	1500.	69.7	3.85	17.80	91.3
52	105.6	15.3	489.+- 3.3	6.77	1456.	67.7	3.75	16.40	90.9
56	104.7	15.2	491.+- 3.2	6.01	1460.	67.9	3.60	15.80	90.9
60	103.3	15.0	494.+- 2.9	6.19	1505.	70.0	4.05	17.00	90.6
64	104.6	15.2	491.+- 3.2	7.23	1511.	72.0	3.70	15.60	90.7
68	103.9	15.1	492.+- 2.9	5.80	1440.	66.1	3.95	14.20	89.3
72	103.9	15.1	487.+- 3.8	5.98	1572.	74.9	3.95	18.00	91.2
76	103.2	15.0	489.+- 3.8	6.43	1531.	72.1	4.15	19.40	91.4
80	103.6	15.0	486.+- 2.7	5.55	1459.	67.8	4.00	19.00	91.5
84	104.2	15.1	490.+- 3.1	6.63	1583.	75.5	4.05	18.20	91.1
88	102.2	14.8	487.+- 2.8	5.90	1503.	69.9	4.05	18.20	91.1
92	104.3	15.1	486.+- 3.0	5.81	1635.	79.8	3.70	17.40	91.4
96	105.0	15.2	488.+- 3.1	6.63	1505.	70.9	4.10	19.80	91.6
100	106.4	15.4	486.+- 3.3	6.71	1472.	67.5	4.10	19.60	91.6
104	107.1	15.5	488.+- 3.0	6.48	1522.	70.8	4.10	17.60	90.7
108	105.2	15.3	484.+- 3.9	6.60	1528.	72.0	4.05	18.40	91.2
112	101.8	14.8	490.+- 3.4	6.49	1533.	73.1	3.45	16.00	91.3

TABLE F-3

RUN 74 FEED BATCH DATA
(COT in hours; all volumes in litres)

COT	Feed Batch Number	Feed Batch Volume Without $Ca(NO_3)_2$	Feed Batch Volume With $Ca(NO_3)_2$
1	1	322	343
7	2	322	343
16	3	322	343
24	4	355	384
32	5	326	350
40	6	315	340
49	7	312	355
57	8	326	348
66	9	319	347
74	10	358	387
83	11	320	344
91	12	321	335
98	13	313	337
100	14	320	345

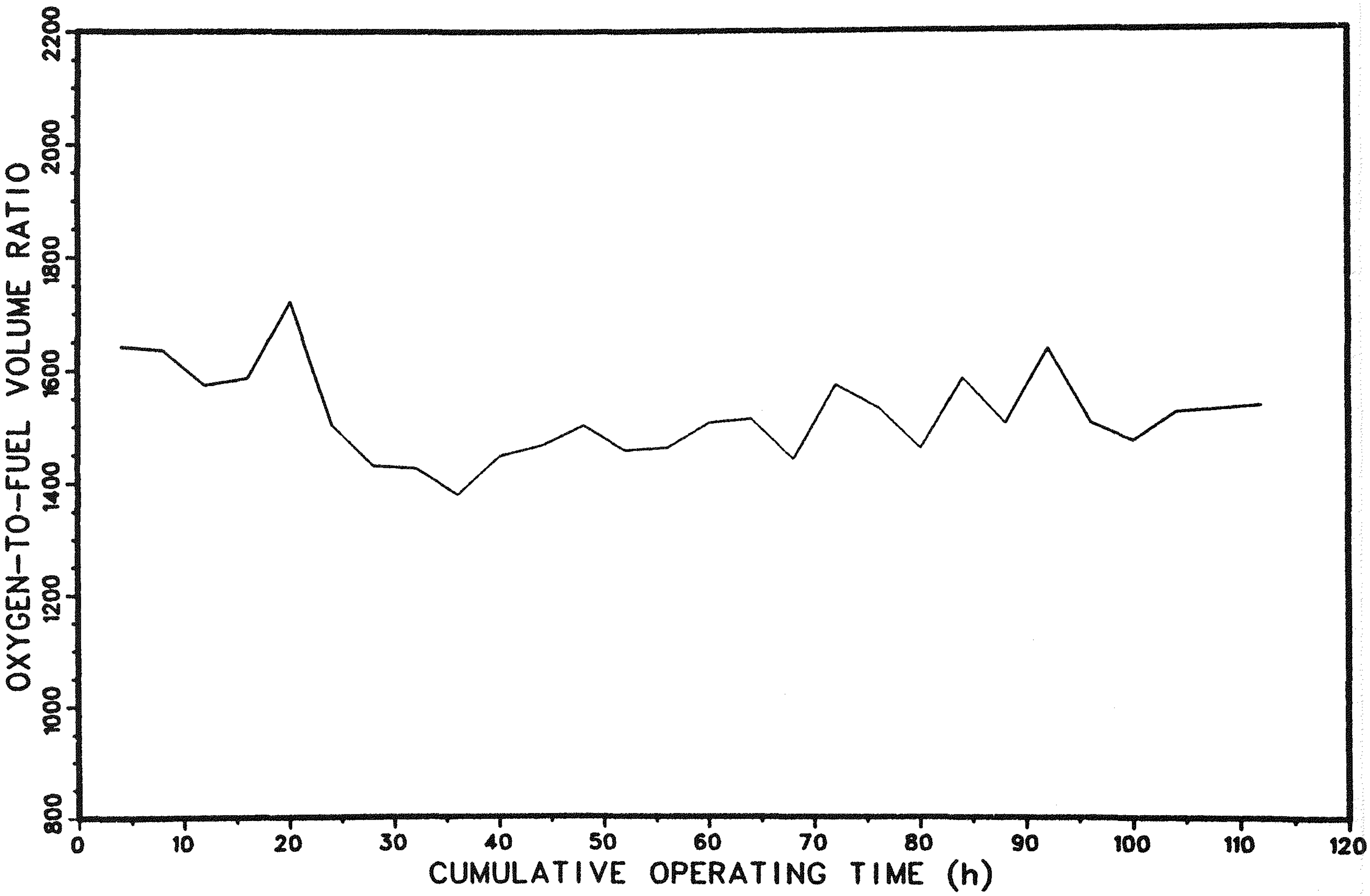

FIGURE F-1. OXYGEN-TO-FUEL RATIO USED DURING RUN 74

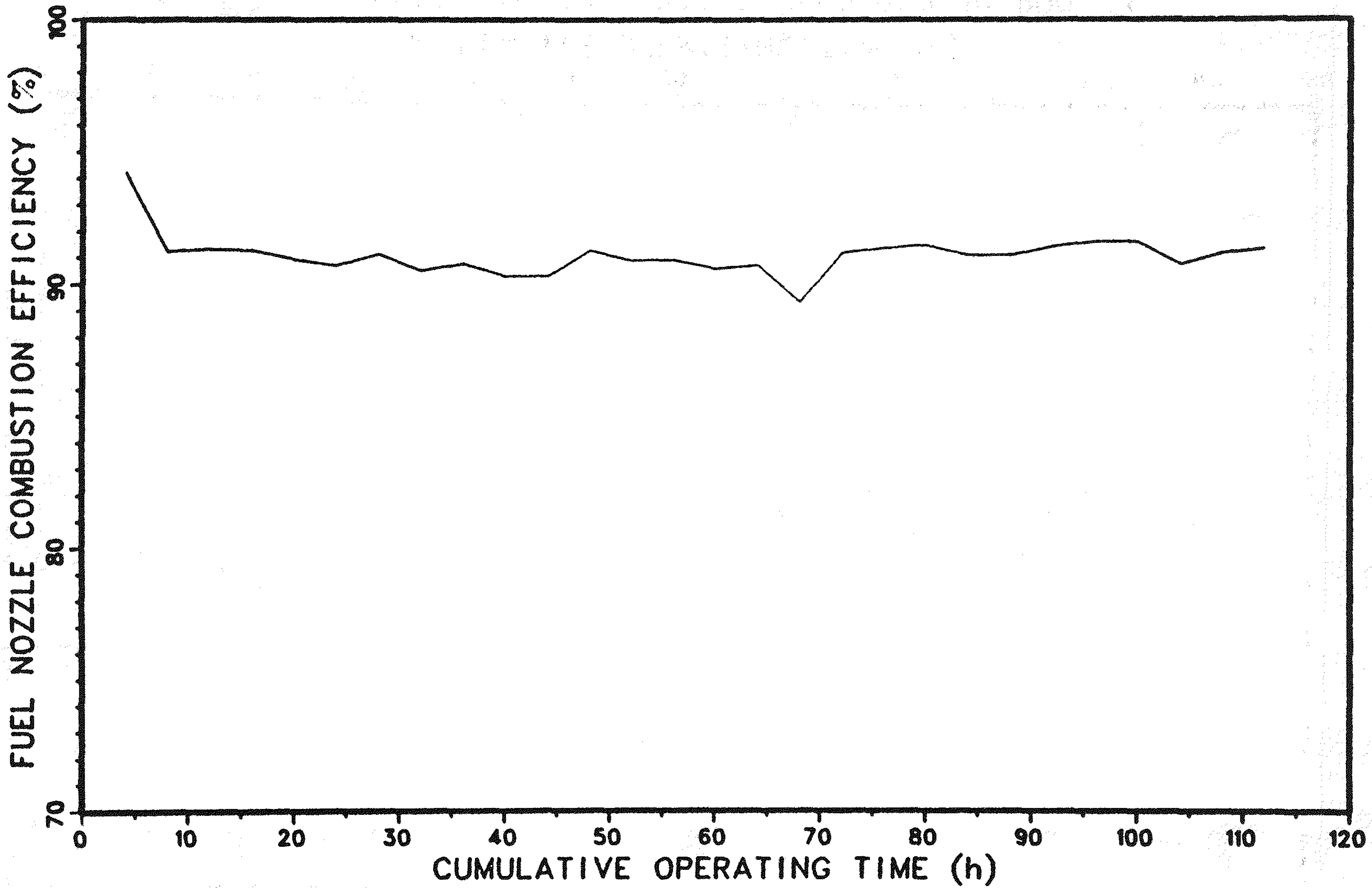

FIGURE F-2. FUEL COMBUSTION EFFICIENCY MEASURED DURING RUN 74

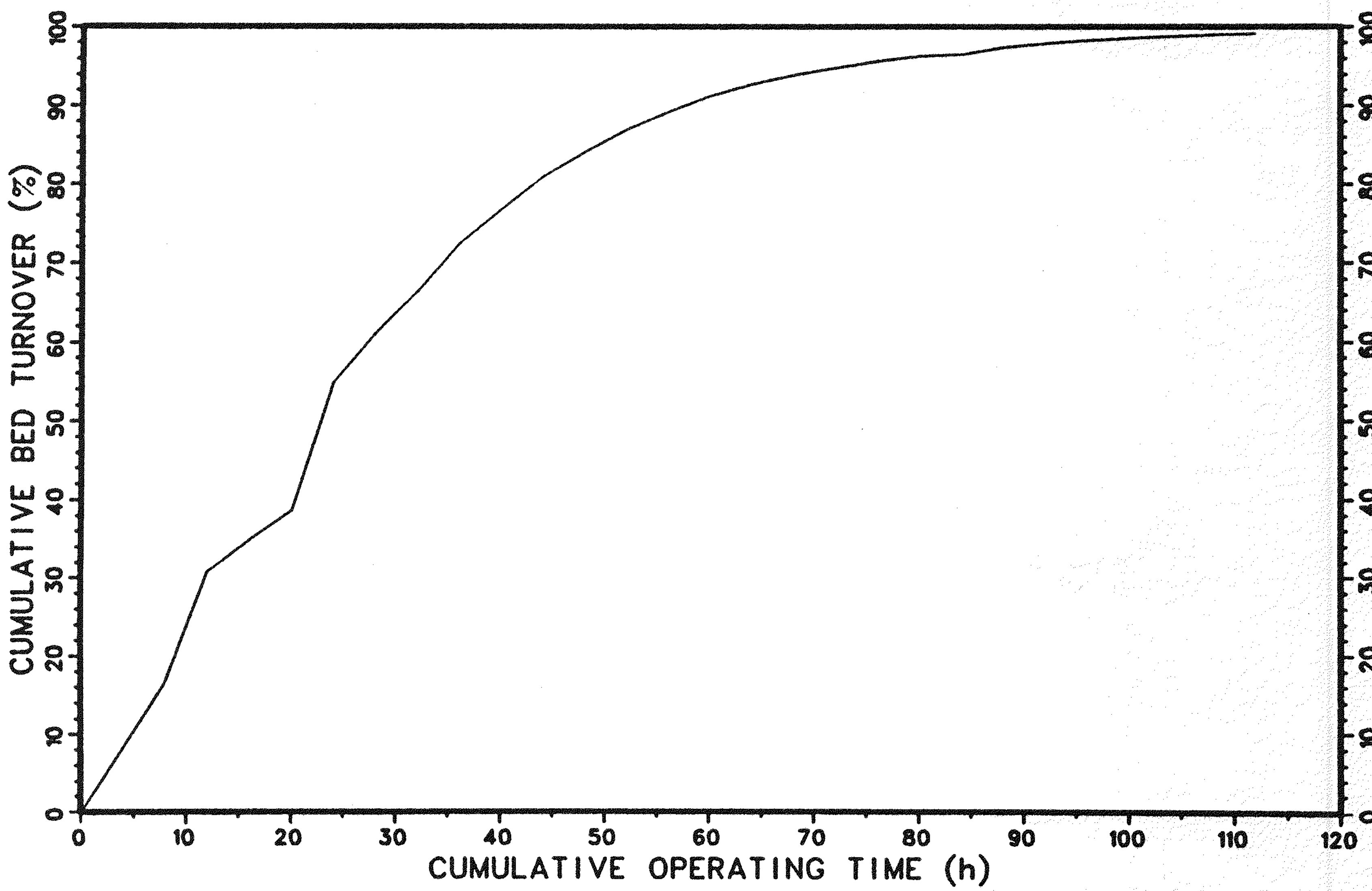

FIGURE F-3. BED TURNOVER OCCURING DURING RUN 74

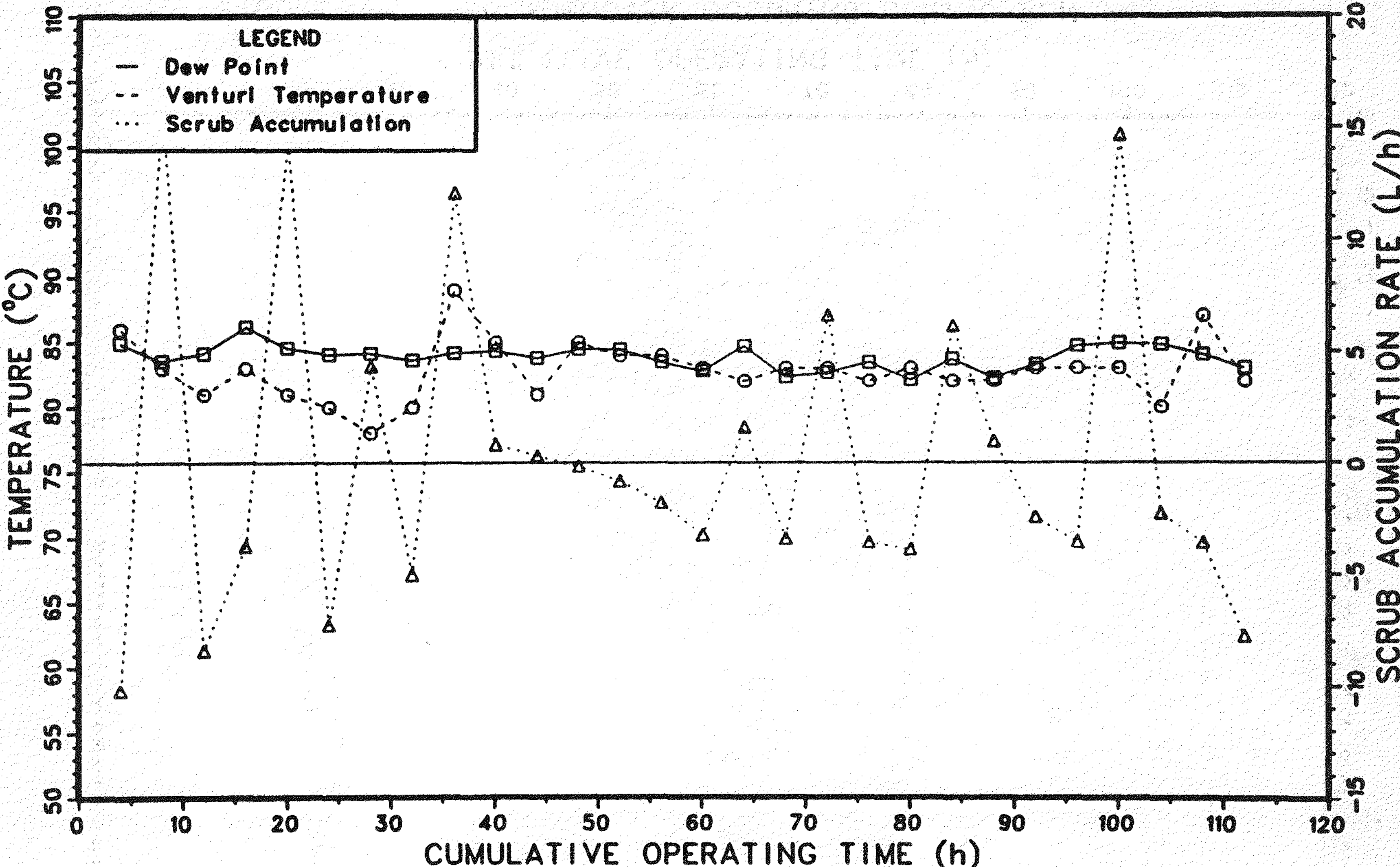

FIGURE F-4. SCRUB ACCUMULATION RELATIONSHIP TO DEW POINT AND VENTURI SCRUBBER TEMPERATURE DURING RUN 74

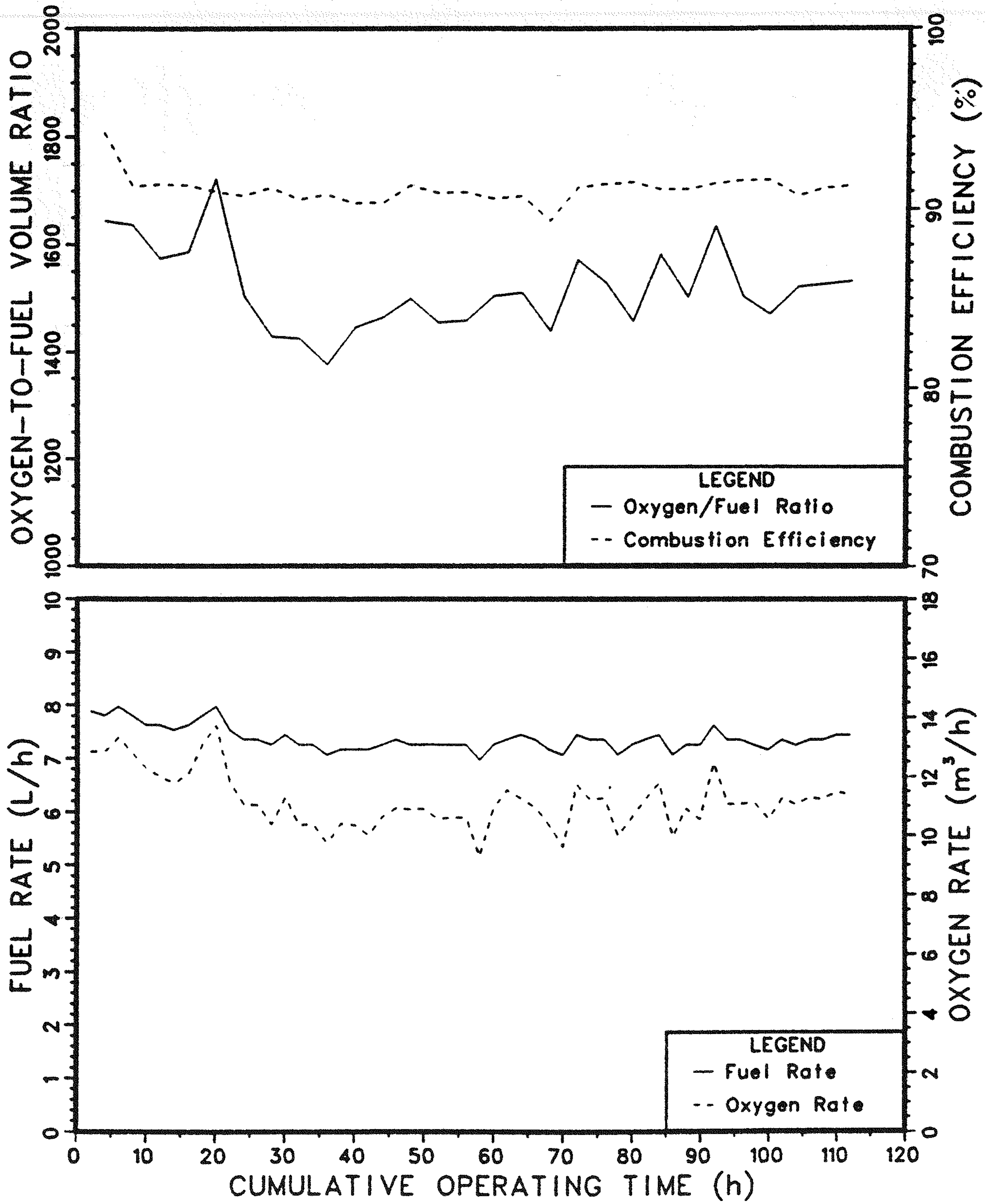

FIGURE F-5 IN-BED COMBUSTION SYSTEM PERFORMANCE DURING RUN 74

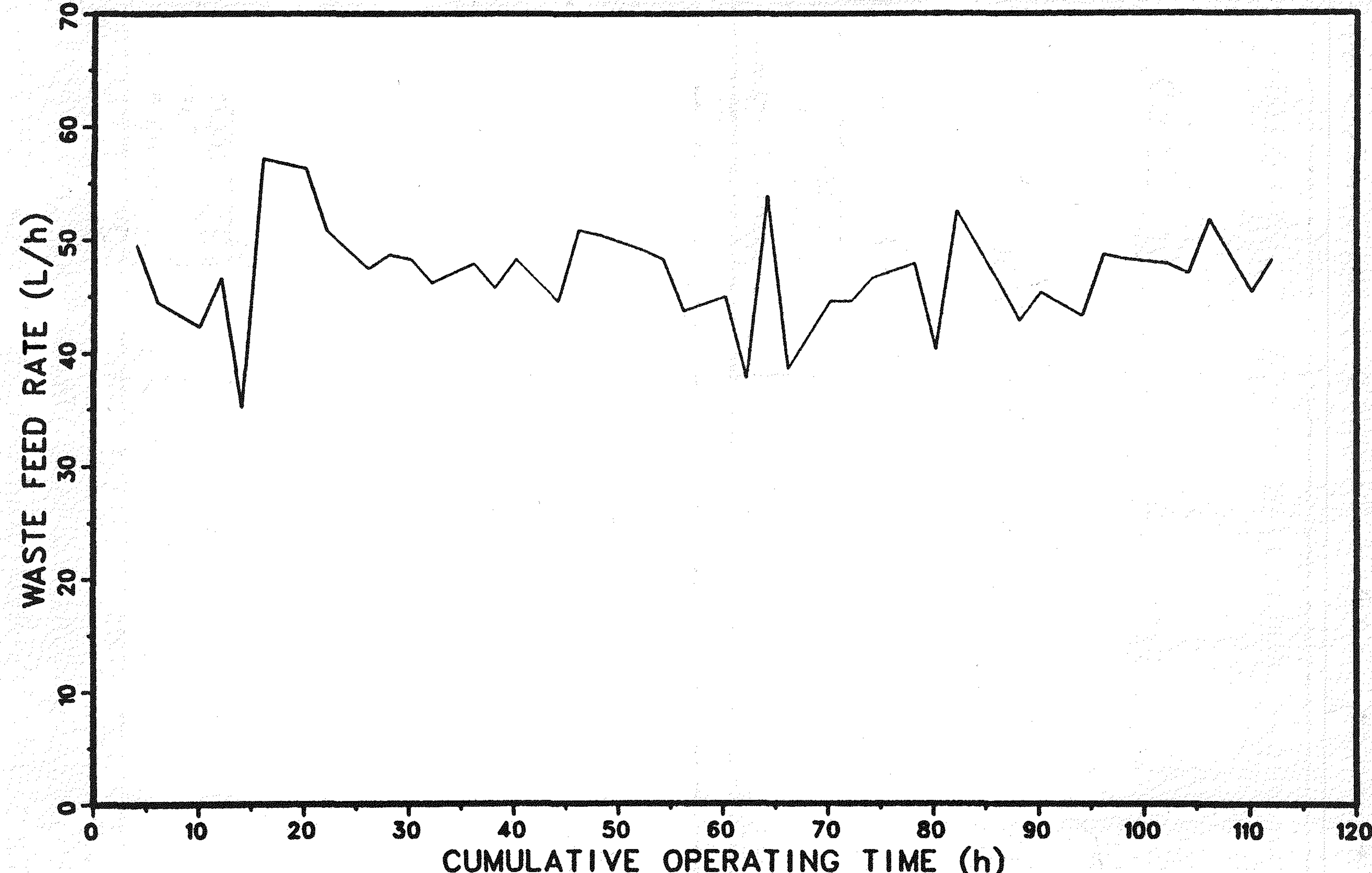

FIGURE F-6. WASTE FEED RATE DURING RUN 74

APPENDIX G

FLUIDIZED-BED OPERATION AND PROPERTIES OF SOLIDS GENERATED FOR RUN 74

TABLE G-1

PRODUCT SIZE DISTRIBUTION

RUN 74

WEIGHT FRACTION RETAINED ON SCREEN

CUMULATIVE OPERATING TIME (HOURS)	COMBINED SIZE RANGE (MESH)													MMPD (MM)
	14	16	20	24	28	32	35	48	65	100	150	200	-200	
0	0.0000	.0027	.0381	.1063	.1853	.1680	.2262	.2098	.0381	.0054	0.0000	0.0000	0.0000	.53
8	0.0000	.0037	.0185	.0519	.1296	.1926	.0815	.3741	.1111	.0296	.0074	0.0000	0.0000	.45
16	.0033	0.0000	.0163	.0358	.1661	.2508	.2704	.2280	.0261	.0033	0.0000	0.0000	0.0000	.50
24	0.0000	0.0000	.0141	.0459	.1519	.2120	.2580	.2580	.0459	.0035	.0035	.0035	.0035	.48
32	0.0000	.0032	.0195	.0584	.1721	.2240	.2500	.2370	.0325	.0032	0.0000	0.0000	0.0000	.50
40	0.0000	0.0000	0.0000	.0290	.1151	.2012	.2883	.3163	.0501	0.0000	0.0000	0.0000	0.0000	.46
48	0.0000	.0024	.0096	.0386	.1229	.2048	.2651	.3084	.0458	.0024	0.0000	0.0000	0.0000	.47
56	0.0000	0.0000	.0126	.0353	.1083	.1688	.2670	.3451	.0605	.0025	0.0000	0.0000	0.0000	.46
64	.0028	0.0000	.0084	.0309	.0843	.1489	.2669	.3792	.0758	.0028	0.0000	0.0000	0.0000	.44
72	0.0000	.0029	.0088	.0294	.0676	.1088	.2265	.4382	.1118	.0059	0.0000	0.0000	0.0000	.42
80	.0026	0.0000	.0077	.0255	.0638	.1148	.2398	.4362	.1029	.0077	0.0000	0.0000	0.0000	.42
88	.0070	0.0000	0.0000	.0070	.0211	.0493	.1479	.5493	.2042	.0141	0.0000	0.0000	0.0000	.36
96	.0024	0.0000	0.0000	.0047	.0071	.0284	.1256	.6232	.1872	.0190	.0024	0.0000	0.0000	.35
104	.0026	0.0000	.0052	.0103	.0236	.0515	.1675	.5722	.1546	.0155	0.0000	0.0000	0.0000	.37
112	.0035	0.0000	.0035	.0071	.0177	.0459	.1555	.5654	.1837	.0177	0.0000	0.0000	0.0000	.37

TABLE G-2

FLUIDIZED BED DATA

RUN 74

COT (HOURS)	FLUIDIZED DENSITY (G/ML)	WEIGHT (KG)	HEIGHT (CM)	MMPD (MM)	FLUIDIZING VELOCITIES: BELOW FUEL NOZ (M/S)	FLUIDIZING VELOCITIES: ABOVE FUEL NOZ (M/S)	FLUIDIZING VELOCITIES: TOP OF BED (M/S)	FLUIDIZING VELOCITIES: THEORETICAL INCIPIENT (M/S)	REPLACEMENT TIME - 90% (HOURS)
4	1.20	43.0	40.6	.45	.24	.37	.87	.08	85.
8	1.25	51.1	46.3	.45	.25	.38	.84	.08	101.
12	1.25	67.2	61.0	.47	.24	.37	.85	.09	50.
16	1.28	72.6	64.5	.50	.24	.38	.95	.11	152.
20	1.25	73.9	67.1	.49	.25	.39	1.02	.10	158.
24	1.25	73.9	67.1	.48	.25	.38	.90	.09	30.
28	1.25	73.9	67.1	.49	.25	.38	.89	.10	62.
32	1.28	76.6	68.1	.50	.25	.38	.87	.11	64.
36	1.28	75.3	66.9	.48	.25	.38	.88	.10	46.
40	1.30	75.3	65.6	.46	.24	.38	.86	.09	53.
44	1.25	73.9	67.1	.47	.24	.37	.83	.10	46.
48	1.25	76.6	69.5	.47	.24	.38	.90	.10	52.
52	1.30	76.6	66.8	.47	.25	.38	.89	.09	46.
56	1.20	73.9	69.8	.46	.24	.38	.84	.09	48.
60	1.50	72.6	54.9	.45	.25	.39	.89	.09	45.
64	1.25	72.6	65.8	.44	.24	.38	.96	.09	51.
68	1.25	71.2	64.6	.43	.25	.38	.84	.08	50.
72	1.25	71.2	64.6	.42	.25	.38	.88	.08	59.
76	1.23	71.2	65.9	.42	.25	.38	.91	.08	50.
80	1.20	65.9	62.2	.42	.25	.38	.84	.08	59.
84	1.20	76.6	72.4	.39	.25	.38	.94	.07	128.
88	1.25	65.9	59.7	.36	.26	.39	.88	.06	30.
92	1.20	65.9	62.2	.36	.25	.38	.87	.06	46.
96	1.25	67.2	61.0	.35	.25	.38	.90	.06	48.
100	1.30	67.2	58.6	.36	.25	.37	.87	.06	50.
104	1.23	67.2	62.2	.37	.24	.37	.86	.07	50.
108	1.18	69.9	67.4	.37	.25	.37	.87	.06	72.
112	1.18	71.2	68.7	.37	.26	.39	.91	.06	52.

TABLE G-3

SOLIDS PRODUCTION DATA

RUN 74

COT (HOURS)	FUEL NAR	WASTE NAR	FLUIDIZING VELOCITY (M/S)	PRODUCT RATE (KG/HR)	FINES RATE (KG/HR)	FINES TO SCRUB (KG/HR)	TOTAL SOLIDS RATE (KG/HR)	THEORY SOLIDS RATE (KG/HR)	PROD/FINE RATIO	ATTRITION INDEX	CYC EFF (%)	CUMULATIVE VOLUME REDUCTION
4	1615.	92.	.24	1.16	2.00	.19	3.35	5.81	.58	34.	91.	11.8
8	1616.	111.	.25	1.16	.65	.19	2.00	5.15	1.79	34.	78.	15.9
12	1558.	133.	.24	3.13	1.33	.19	4.64	5.22	2.36	34.	87.	13.7
16	1557.	83.	.24	1.10	1.23	.20	2.52	5.42	.90	34.	86.	13.7
20	1747.	127.	.25	1.08	1.63	.09	2.79	6.63	.66	31.	95.	14.6
24	1506.	143.	.25	5.70	.95	-.01	6.64	5.86	6.00	27.	0.	14.4
28	1432.	145.	.25	2.75	.60	-.03	3.32	5.64	4.58	24.	0.	14.8
32	1427.	153.	.25	2.78	.40	-.04	3.13	5.40	6.94	20.	0.	15.4
36	1361.	146.	.25	3.75	1.68	.07	5.49	5.28	2.24	19.	96.	15.2
40	1428.	144.	.24	3.25	.28	.18	3.71	5.23	11.82	18.	60.	16.4
44	1438.	155.	.24	3.68	1.83	.18	5.69	5.06	2.01	16.	91.	14.3
48	1481.	137.	.24	3.38	1.08	.17	4.62	5.63	3.14	15.	86.	13.0
52	1427.	140.	.25	3.83	.68	.12	4.62	5.50	5.67	14.	85.	14.0
56	1443.	159.	.24	3.58	.95	.07	4.59	5.11	3.76	13.	94.	14.6
60	1508.	216.	.25	3.75	1.25	.12	5.12	4.96	3.00	11.	91.	15.3
64	1493.	178.	.24	3.30	.40	.17	3.87	5.09	8.25	10.	70.	16.1
68	1433.	234.	.25	3.28	1.73	.14	5.14	4.45	1.90	9.	92.	15.0
72	1565.	219.	.25	2.78	1.18	.11	4.06	4.95	2.36	8.	91.	13.9
76	1534.	207.	.25	3.25	1.13	-.05	4.33	4.36	2.89	7.	0.	13.9
80	1454.	241.	.25	2.55	1.03	-.21	3.37	4.09	2.49	6.	0.	14.0
84	1573.	195.	.25	1.38	1.58	.01	2.96	4.73	.87	6.	99.	14.6
88	1518.	231.	.26	5.03	.75	.23	6.01	4.13	6.70	6.	76.	15.1
92	1619.	164.	.25	3.33	1.93	.09	5.34	4.16	1.73	6.	95.	14.0
96	1480.	145.	.25	3.23	1.45	-.05	4.63	4.27	2.22	6.	0.	13.2
100	1428.	146.	.25	3.13	.90	-.04	3.99	4.48	3.47	7.	0.	13.2
104	1467.	145.	.24	3.10	1.55	-.03	4.62	4.41	2.00	7.	0.	13.0
108	1501.	142.	.25	2.25	1.65	.04	3.94	5.32	1.36	8.	98.	12.7
112	1558.	149.	.26	3.13	1.65	.11	4.89	5.63	1.89	8.	94.	12.2
TOTAL				335.0	133.7	8.8	477.	568.	2.51			

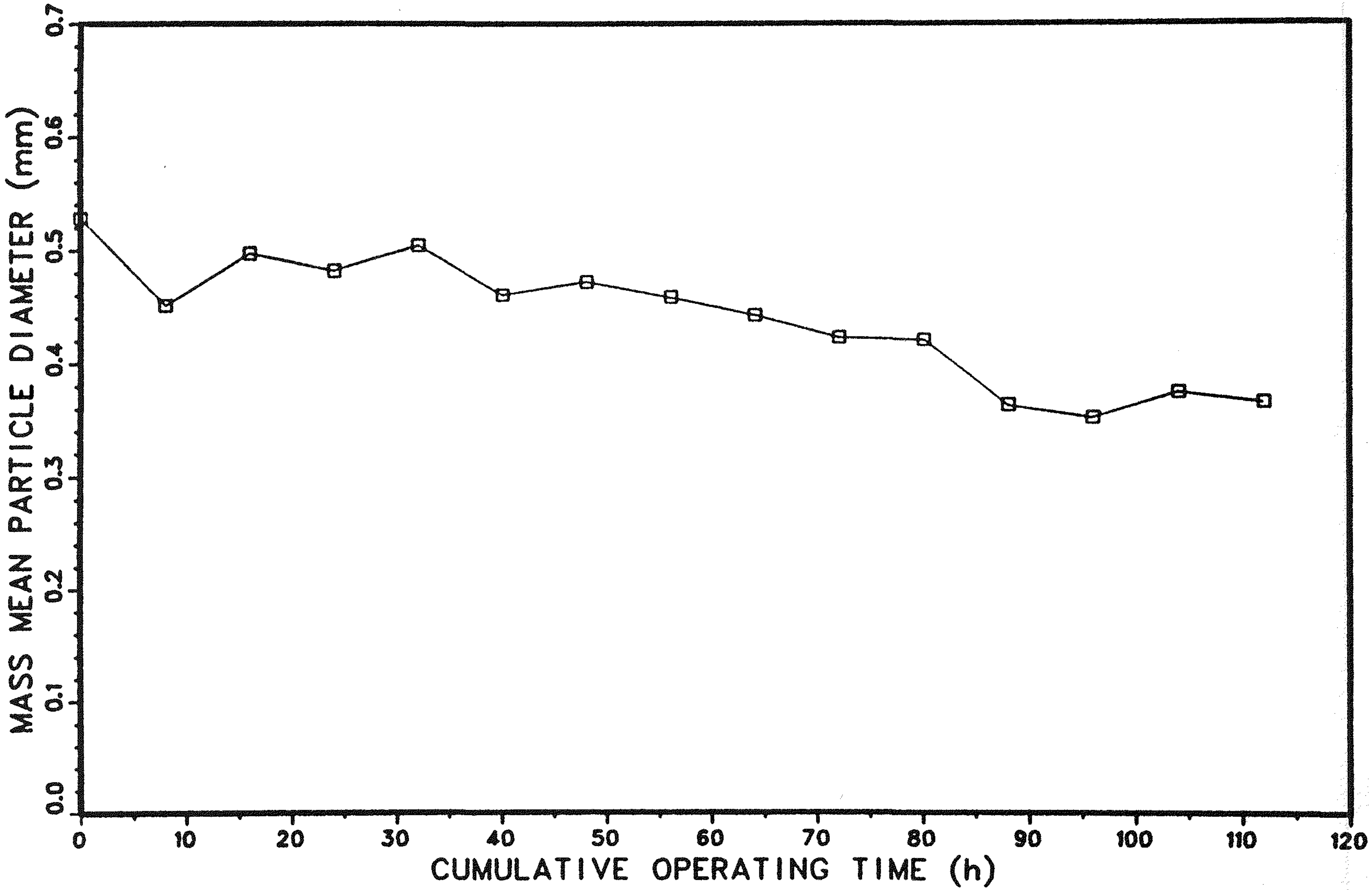

FIGURE G-1. BED MASS MEAN PARTICLE DIAMETER PRODUCED DURING RUN 74

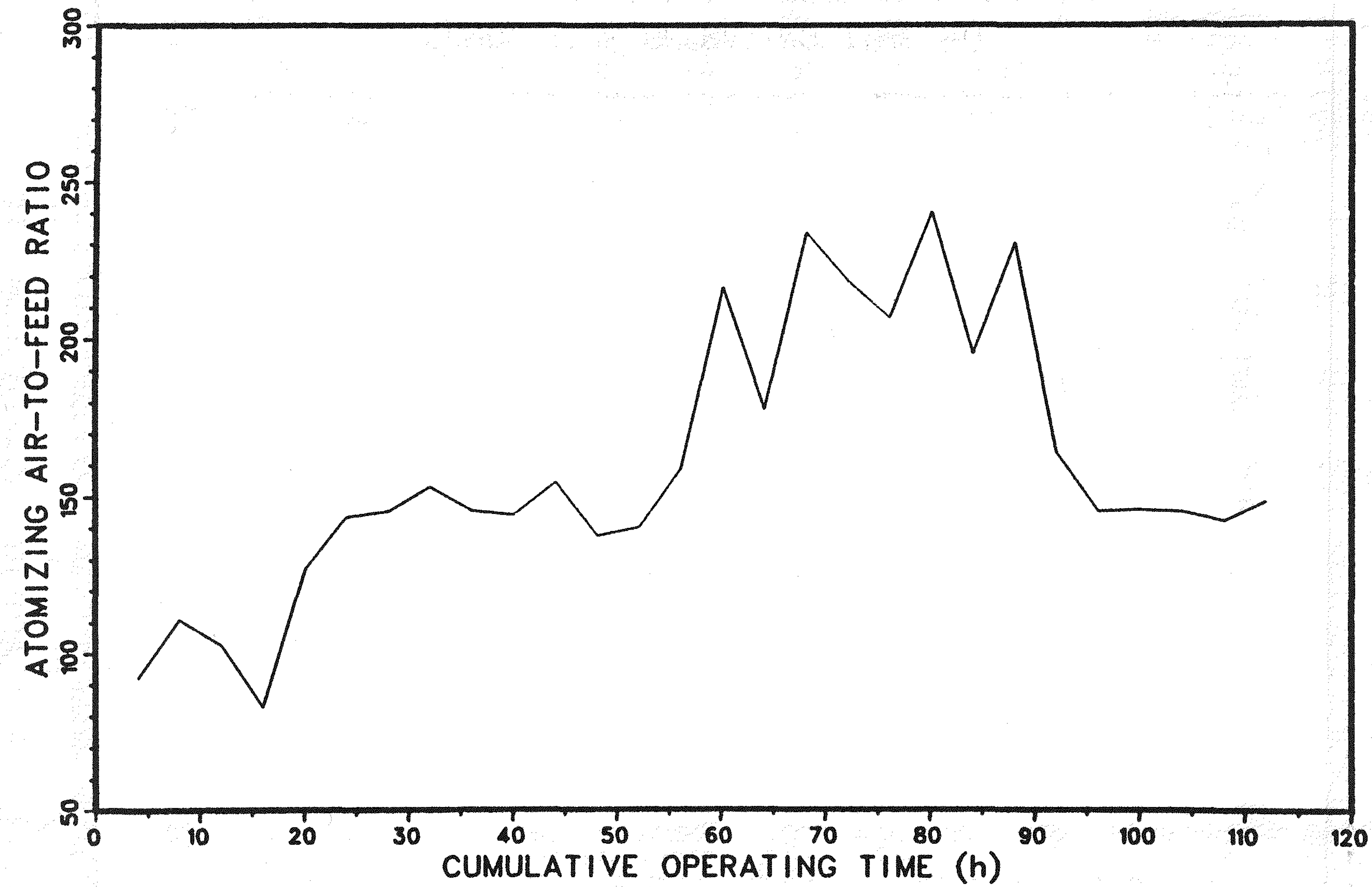

FIGURE G-2. FEED NOZZLE AIR RATIO USED DURING RUN 74

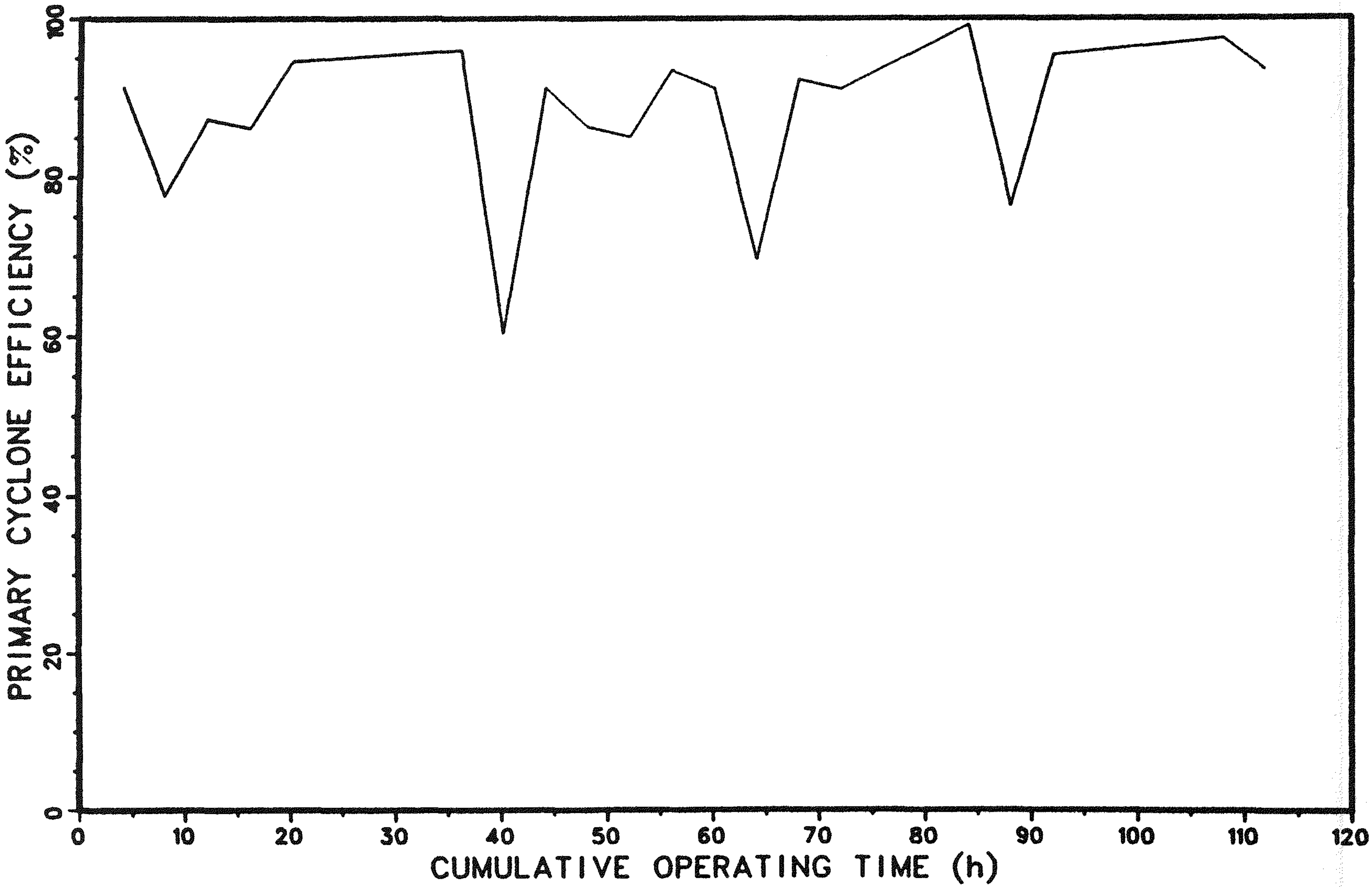

FIGURE G-3. CYCLONE EFFICIENCY MEASURED DURING RUN 74

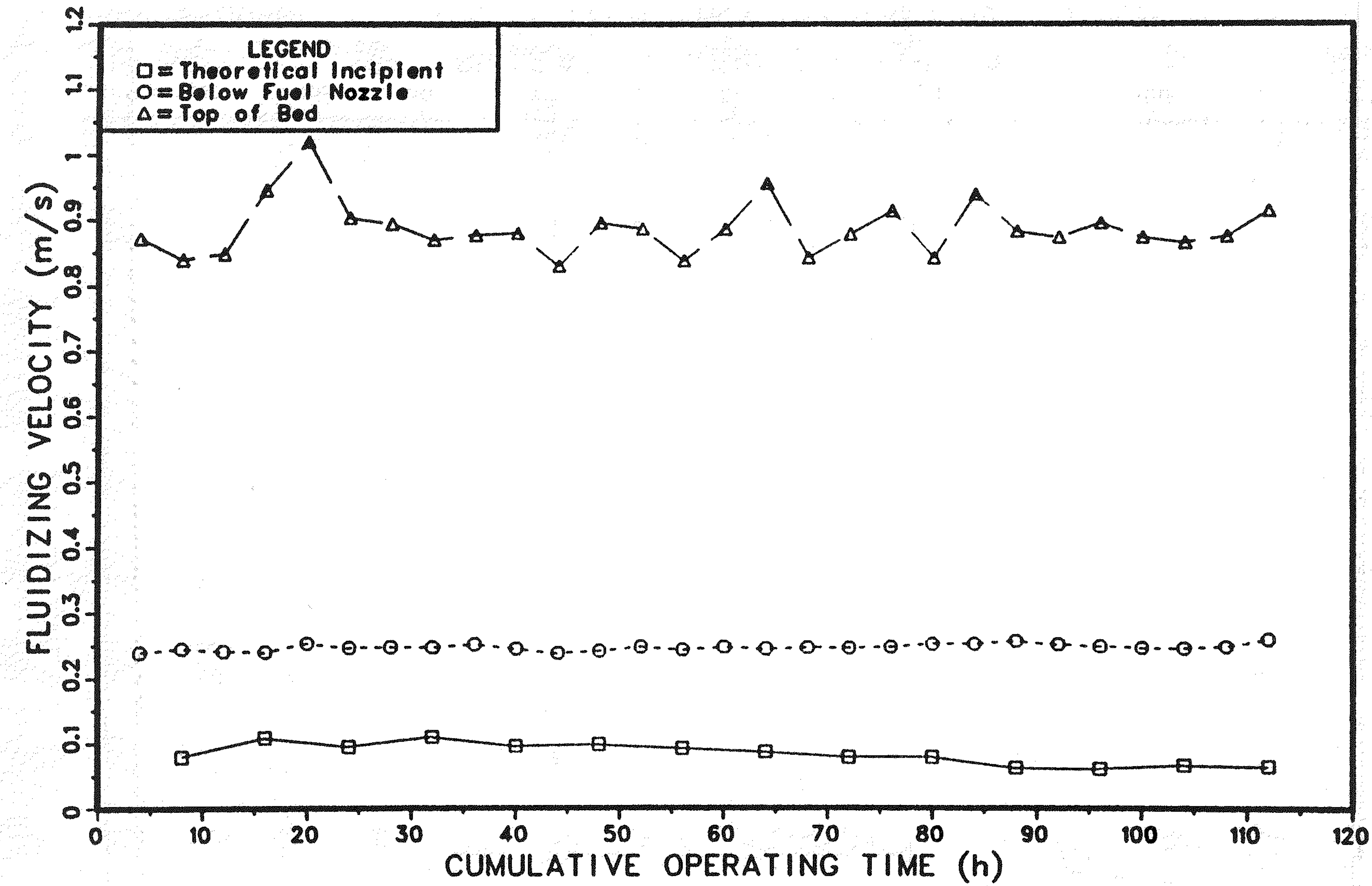

FIGURE G-4. INCIPIENT FLUIDIZING VELOCITY COMPARED TO FLUIDIZING VELOCITIES USED DURING RUN 74

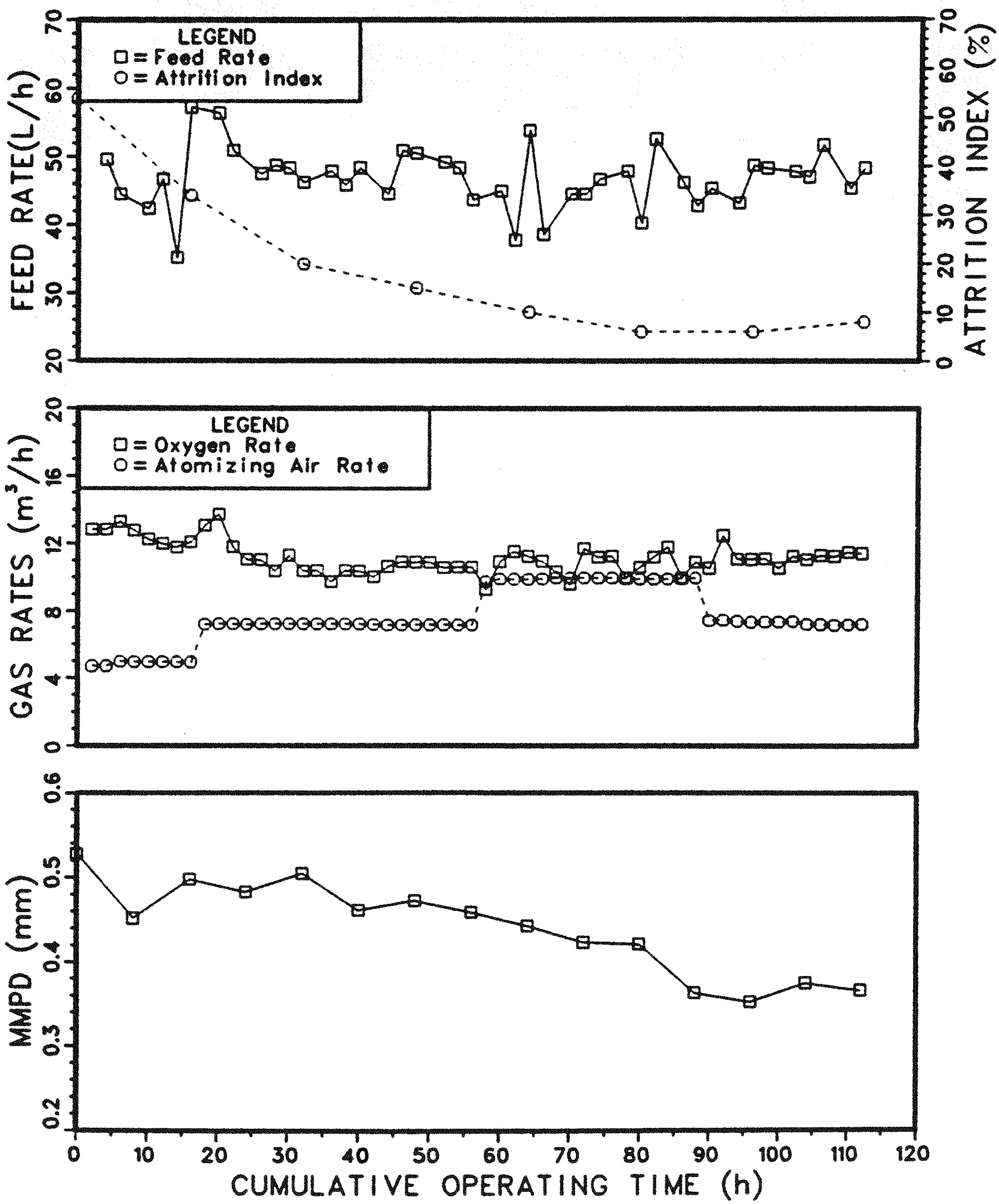

FIGURE G-5. EFFECT OF VARIOUS OPERATING CONDITIONS ON BED PARTICLE SIZE DURING RUN 74

APPENDIX H

ANALYSES OF SAMPLES TAKEN DURING RUN 74

TABLE H-1
ANALYSES OF RUN 74 FEED

COT	Calciner Feed Concentration											Mole Ratios	
(h)	H^+ (M)	F^- (M)	NO_3^- (M)	Aℓ (M)	Zr (M)	Ca (M)	Cℓ (µg/mL)	SO_4 (M)	B (M)	Hg (µg/mL)	Sp. G. (g/mL)	Ca/F	Aℓ/F
1	0.44	1.21	2.85	0.41	0.24	0.78	332	0.058	0.129	371	1.190	0.645	0.339
32	0.46	1.21	3.07	0.37	0.21	0.80	348	0.063	0.088	366	1.199	0.661	0.306
74	0.45	1.13	2.68	0.32	0.18	0.60	306	0.058	0.111	353	1.182	0.531	0.283
107	1.07	1.02	3.54	0.44	0.19	1.00	342	0.065	0.051	452	1.206	0.980	0.431

TABLE H-2

COMPOSITION OF FINES PRODUCED DURING RUN 74

COT (h)	F	Zr	Ca	SO_4	Al	Cl	NO_3	Hg	Bulk Density (g/cm^3)
	(wt.%)								
8	17.8	12.0	23.0	5.0	9.0	0.40	3.77	0.0005	0.54
40	20.3	14.0	26.0	4.2	10.0	0.07	0.86	0.0002	0.60
80	18.9	10.0	31.0	5.0	10.0	0.05	0.84	0.0002	0.55
112	20.3	14.0	27.0	5.0	9.3	0.05	1.24	0.0003	0.47

TABLE H-3

COMPOSITION OF PRODUCT PRODUCED DURING RUN 74

COT (h)	F	Zr	Ca	SO_4	Al	Cl	NO_3	Hg	Bulk Density (g/cm^3)	Bed Void
	(wt.%)									
0	14.6	(a)	(a)	3.7	(a)	0.30	(a)	0.00005	(a)	(a)
40	17.4	16.0	30.0	4.2	9.6	0.05	0.17	0.0002	1.39	0.43
80	17.0	15.0	22.0	4.2	9.0	0.05	0.26	0.0003	1.39	0.42
112	16.1	15.0	35.0	5.5	9.3	0.05	0.27	0.0001	1.39	0.44

(a) Not analyzed

TABLE H-4

COMPOSITION OF ACID SCRUB HOLD TANK SOLUTION DURING RUN 74

COT (h)	F (M)	Zr (M)	Ca (M)	B (M)	Aℓ (M)	NO_3 (M)	H^+ (M)	Cℓ (mg/L)	SO_4 (mg/L)	Hg (mg/L)
56	0.30	0.025	0.118	0.030	0.074	3.11	2.59	915	1140	3045
80	0.47	0.032	0.140	0.034	0.093	3.09	2.35	1100	1510	3455
112	0.55	0.032	0.128	0.045	0.104	3.07	2.27	1320	1660	3889

TABLE H-5

COMPOSITION OF SCRUB SOLUTIONS DOWNSTREAM FROM NITRIC ACID SCRUBBER DURING RUN 74

COT (h)	F (mg/L)	Cℓ (mg/L)	Ca (mg/L)	Aℓ (mg/L)	Hg (mg/L)	SO_4 (mg/L)	Scrubber Volume[b] (mL)
FIRST CAUSTIC SCRUBBER							
36	66.8	28.0	23	19	24	15	1875
84	125.6	99.0	83	43	49	49	2368
108	134.2	34.8	28	51	56	55	2350
SECOND CAUSTIC SCRUBBER							
36	18.4	16.8	(a)	(a)	21	8	463
HYDROCHLORIC ACID SCRUBBER							
60	(a)	(a)	(a)	(a)	83	(a)	670

(a) Not analyzed
(b) At end of 8-hour operating period

APPENDIX I

RUN 74 TEMPERATURE AND PRESSURE DATA

TABLE I-1

RUN 74 THERMOCOUPLE LOCATIONS

TR901 THERMOCOUPLE LOCATIONS

1 -- OUTLET OF FEED COOLER
2 -- OUTLET OF SCRUB EM FLOWMETER
3 -- FEED ATOMIZING AIR
4 -- OUTLET OF VENTURI SCRUBBER
5 -- OUTLET OF QUENCH TOWER
6 -- INLET OF AIR PREHEATER
7 -- WATER OUTLET OF JET COND.
8 -- FUEL ATOMIZING O2
9 -- OUTLET OF PARTIAL COND.
10 -- FUEL
11 -- AMBIENT AIR
12 -- OUTLET OF DEMISTER
13 -- OUTLET OF HEAT EXCHANGER
14 -- SILICA GEL ADSORBER
15 -- INLET TO FINAL FILTER

TR902 THERMOCOUPLE LOCATIONS

1 -- PRODUCT POT
2 -- AMBIENT S OF FINES POT
3 -- PRIMARY CYCLONE OUTLET
4 -- VESSEL 2.2 METERS E WALL
5 -- VESSEL 1.87 METER E WALL
6 -- VESSEL 1.56 METERS E WALL
7 -- VESSEL 1.25 METERS E WALL
8 -- BED 78.7 CM W WALL
9 -- BED 78.7 CM S WALL
10 -- VESSEL 78.7 CM N WALL
11 -- BED 11.4 CM N WALL
12 -- VAPOR 97 CM E WALL
13 -- VAPOR 2.1 METER E WALL
14 -- VAPOR 1.79 METERS E WALL
15 -- FLUID AIR PREHEATER OUTLET
16 -- FINES POT INLET
17 -- VAPOR 1.49 METERS E WALL
18 -- BED 58.4 CM N WALL
19 -- VAPOR 1.26 METER W WALL
20 -- BED 11.4 CM S WALL
21 -- VESSEL 11.4 W WALL
22 -- BED 11.4 CM E WALL
23 -- BED 58.4 CM S WALL

TABLE I-2A

RUN 74 THERMOCOUPLE READING

INPUT DATA - TR901(1-15)   ALL UNITS ARE DEGREES C

LS	COT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	TIC 903	TIC 904
31	4	20	38	31	86	142	136	11	21	11	24	18	21	24	20	20	-17	-17
30	8	19	36	36	83	140	140	11	20	9	24	20	20	19	19	18	-17	-17
30	12	19	38	37	81	89	148	11	19	9	23	21	19	19	19	18	-17	-17
30	16	17	35	36	83	173	156	11	19	10	23	20	20	20	20	17	-17	-17
30	20	20	41	35	81	163	187	11	20	11	24	20	20	20	20	19	-17	-17
30	24	17	41	34	80	110	156	11	20	13	23	20	20	20	19	19	-17	-17
30	28	21	39	34	78	84	155	10	20	12	24	21	20	19	18	18	-17	-17
30	32	18	28	34	80	113	158	11	21	13	24	21	20	20	19	19	-17	-17
30	36	17	38	35	89	200	158	11	20	16	20	21	21	20	20	19	-17	-17
30	40	17	37	35	85	209	161	11	20	15	20	20	20	20	19	19	-17	-17
30	44	22	35	34	81	201	162	11	20	17	25	22	20	20	19	19	-17	-17
30	48	18	38	38	85	205	162	11	23	21	27	24	22	23	21	22	-17	-17
30	52	24	28	38	84	200	160	10	24	17	28	25	23	23	22	22	-17	-17
30	56	18	34	29	84	199	163	11	22	19	25	25	22	22	22	23	-17	-17
30	60	22	34	34	83	208	160	10	21	16	25	22	20	20	22	22	-17	-17
30	64	17	37	38	82	194	160	11	20	15	25	22	20	21	21	21	-17	-17
30	68	18	33	33	83	191	161	11	20	14	25	22	21	20	20	20	-17	-17
30	72	20	32	32	83	190	166	11	20	13	25	20	20	19	19	19	-17	-17
30	76	28	32	33	82	197	167	11	21	13	26	22	20	20	20	19	-17	-17
30	80	19	32	31	83	187	162	10	19	10	23	20	19	19	18	18	-17	-17
30	84	25	32	33	82	190	189	10	19	11	23	21	19	19	17	17	-17	-17
30	88	20	37	33	82	176	165	11	19	12	23	23	19	19	20	18	-17	-17
30	92	21	37	29	83	167	164	11	19	11	24	21	19	19	19	18	-17	-17
30	96	23	38	40	83	155	165	11	25	23	30	26	23	25	21	23	-17	-17
30	100	25	37	38	83	154	163	10	22	15	26	23	22	22	22	21	-17	-17
30	104	25	37	34	80	163	161	10	19	12	24	22	22	20	20	18	-17	-17
30	108	24	36	36	87	162	171	11	19	15	24	21	21	21	19	19	-17	-17
39	112	23	33	35	82	163	176	11	21	14	25	21	21	21	20	19	-17	-17

TABLE I-2B

RUN 74 THERMOCOUPLE READINGS

INPUT DATA - TK9C2(1-23)   ALL UNITS ARE DEGREES C

LS	CUT	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	ODRIC	TKC905
41	4	265	17	553	297	337	280	352	362	462	486	495	363	373	392	75	92	396	493	426	491	440	485	504	505	498
40	8	270	20	551	314	363	302	373	480	490	410	488	395	398	416	86	100	425	495	452	468	437	464	495	499	498
40	12	278	18	550	324	370	311	394	488	490	420	420	401	423	422	90	105	432	490	433	467	445	481	493	495	498
40	16	275	18	551	324	371	311	400	490	491	425	489	422	406	427	94	92	436	490	464	487	446	481	492	495	498
40	20	260	14	547	333	385	328	430	500	500	423	497	430	416	443	103	66	455	497	484	496	455	490	499	504	510
40	24	298	15	550	320	370	310	398	491	491	423	491	421	403	425	94	75	435	492	463	469	449	485	492	502	501
40	28	297	18	552	322	372	313	401	491	494	425	491	424	407	427	95	71	432	492	464	486	451	484	493	498	500
40	32	394	19	382	325	373	315	403	492	491	425	490	424	406	428	95	70	437	491	465	490	450	484	494	500	500
40	36	396	16	382	325	373	313	401	494	493	424	490	425	406	425	96	70	439	493	466	489	447	479	495	500	500
40	40	436	16	381	325	372	313	401	494	493	424	489	425	407	428	97	75	435	494	467	490	447	485	494	500	500
40	44	430	15	390	332	383	326	413	484	486	421	484	423	413	435	92	62	456	484	462	480	442	475	488	494	494
40	48	385	17	380	325	373	314	405	488	488	423	488	424	407	423	95	123	437	486	463	465	443	479	487	497	496
40	52	454	21	384	320	374	315	406	491	491	425	488	426	408	429	98	80	440	490	467	487	447	483	493	496	498
40	56	392	20	366	327	375	377	405	494	493	423	491	427	409	431	100	75	440	493	468	490	448	485	494	499	500
40	60	395	19	369	330	377	318	405	496	496	425	494	426	411	432	100	95	440	494	467	492	451	488	496	499	504
40	64	435	19	385	327	373	315	403	494	492	425	492	426	410	429	99	99	440	492	465	489	453	485	494	499	501
40	68	429	19	382	325	373	314	403	492	494	424	493	425	406	428	98	89	440	493	466	490	455	486	494	499	501
40	72	376	14	365	327	376	315	406	491	491	421	488	421	408	429	95	93	438	496	464	485	450	480	489	499	500
40	76	352	19	385	327	375	317	411	494	492	425	489	425	410	430	87	82	441	488	468	487	455	482	499	496	500
40	80	392	17	364	324	373	314	405	487	488	424	485	424	407	428	98	81	437	488	465	484	451	481	488	493	496
40	84	396	18	400	339	393	331	431	494	490	425	490	428	424	447	107	134	453	490	462	489	456	484	492	494	498
40	88	350	18	386	327	376	316	406	490	489	426	487	425	410	432	102	104	440	486	464	468	454	482	490	493	498
40	92	330	13	385	325	375	314	402	486	486	421	487	422	408	428	95	117	437	486	463	484	445	480	489	496	500
40	96	277	19	380	323	371	313	400	490	490	423	487	422	405	425	96	104	435	488	463	486	451	482	491	495	498
40	100	338	20	383	325	374	315	403	489	486	426	486	422	408	427	100	92	436	485	463	484	450	480	490	490	498
40	104	285	19	550	325	374	315	406	491	491	425	487	425	408	429	97	100	439	489	463	487	450	483	491	497	498
40	108	286	21	551	327	377	320	416	487	406	426	486	426	411	432	104	104	442	486	470	477	451	480	485	498	496
49	112	315	15	550	329	379	321	417	490	495	425	491	427	416	436	105	105	447	491	477	489	450	485	486	498	500

PRESSURE GUAGE LOCATIONS

TABLE I-3

RUN 74 PRESSURE GAUGE LOCATIONS

PI901 -- STEAM TO JET	PI902 -- FEED SUPPLY	PI903 -- SCRUB SUPPLY	PI904 -- SCRUB TANK GUAGE PRES.
PI905 -- FUEL SUPPLY	PI906 -- FUEL NOZZLE LIQUID	PI907 -- OXYGEN SUPPLY	PI908 -- FEED NOZZLE LIQUID
PI909 -- FEED NOZZLE GAS	PI910 -- FLUIDIZING AIR PREHTR INLT	PI911 -- FLUIDIZING AIR SUPPLY	PI912 -- QUENCH TWR INLET (ABS)
PI913 -- VENT SCRB EXIT (ABS)	PI914 -- KNOCKOUT POT EXIT (ABS)	PI915 -- FEED ATOM AIR SUPPLY	PI917 -- PARTIAL COND EXIT (ABS)
PI918 -- STEAM JET INLET (ABS)	PI919 -- FUEL NOZZLE GAS	PI920 -- ATMOSPHERIC PRESSURE	PI922 -- INSTRUMENT AIR
PI923 -- 50 PSI AIR			

TABLE I-4A

RUN 74 PRESSURE GAUGE READINGS

INPUT DATA - PI(1-11), PSI AND KPA

LS	COT	901		902		903		904		905		906		907		908		909		910		911	
51	4	0.0	0.	4.9	34.	48.0	331.	5.6	39.	70.5	486.	3.0	21.	84.1	580.	7.5	52.	11.8	81.	4.7	32.	100.0	689.
50	8	0.0	0.	7.2	50.	50.7	349.	5.7	39.	69.0	475.	3.2	22.	83.0	572.	7.5	52.	13.0	90.	4.1	28.	100.0	689.
50	12	0.0	0.	3.8	26.	50.0	345.	5.3	37.	70.0	482.	3.5	24.	83.0	572.	7.0	48.	11.3	78.	4.0	28.	100.0	689.
50	16	0.0	0.	2.8	19.	50.0	345.	5.8	40.	70.0	482.	3.5	24.	84.5	582.	7.0	48.	14.0	96.	4.2	29.	98.0	675.
50	20	0.0	0.	3.0	21.	50.0	345.	7.4	51.	70.0	482.	4.0	28.	82.5	569.	7.0	48.	21.0	145.	4.1	28.	100.0	689.
50	24	0.0	0.	2.7	19.	48.0	331.	5.6	39.	70.5	486.	3.5	24.	87.0	600.	5.0	34.	25.0	172.	4.0	28.	99.0	682.
50	28	0.0	0.	3.0	21.	49.5	341.	7.3	50.	70.0	482.	3.3	23.	86.1	593.	5.0	34.	25.0	172.	4.2	29.	100.0	689.
50	32	0.0	0.	2.4	17.	32.5	224.	7.0	48.	70.5	486.	3.2	22.	85.9	592.	4.8	33.	24.0	165.	4.1	28.	100.0	689.
50	36	0.0	0.	3.0	21.	35.2	243.	1.5	10.	70.5	486.	3.1	21.	86.8	598.	5.0	34.	26.0	179.	4.1	28.	100.0	689.
50	40	0.0	0.	2.5	17.	35.0	241.	1.5	10.	70.5	486.	3.1	21.	85.8	591.	5.0	34.	27.0	186.	4.1	28.	100.0	689.
50	44	0.0	0.	3.2	22.	34.0	234.	1.6	11.	70.7	487.	3.0	21.	85.0	586.	5.4	37.	27.0	186.	4.2	29.	98.0	675.
50	48	0.0	0.	2.3	16.	33.0	227.	1.5	10.	71.0	489.	3.1	21.	85.0	586.	5.3	37.	26.0	179.	4.2	29.	100.0	689.
50	52	0.0	0.	2.4	17.	32.5	224.	1.6	11.	70.8	488.	3.1	21.	85.0	586.	5.4	37.	29.5	203.	4.3	30.	100.0	689.
50	56	0.0	0.	2.6	18.	33.5	231.	1.6	11.	70.4	485.	3.0	21.	85.0	586.	5.4	37.	28.0	193.	4.2	29.	100.0	689.
50	60	0.0	0.	3.0	21.	34.5	238.	1.6	11.	71.0	489.	2.8	19.	85.0	586.	5.0	34.	34.0	234.	4.0	28.	99.0	682.
50	64	0.0	0.	2.5	17.	35.0	241.	1.7	12.	70.5	486.	3.0	21.	85.0	586.	5.2	36.	35.0	241.	4.2	29.	100.0	689.
50	68	0.0	0.	2.5	17.	33.0	227.	1.7	12.	70.0	482.	3.0	21.	85.0	586.	4.7	32.	34.0	234.	4.1	28.	100.0	689.
50	72	0.0	0.	2.4	17.	35.0	241.	1.8	12.	71.0	489.	3.0	21.	83.0	572.	5.7	39.	30.0	207.	4.1	28.	100.0	689.
50	76	0.0	0.	2.5	17.	33.5	231.	1.8	12.	70.5	486.	3.0	21.	85.5	589.	5.5	38.	35.0	241.	4.2	29.	100.0	689.
50	80	0.0	0.	2.4	17.	34.5	238.	1.8	12.	70.5	486.	2.9	20.	83.8	577.	5.3	37.	34.0	234.	4.1	28.	98.5	679.
50	84	0.0	0.	2.0	14.	36.5	252.	1.8	12.	70.5	486.	2.9	20.	84.0	579.	6.0	41.	33.0	227.	4.2	29.	99.0	682.
50	88	0.0	0.	3.4	23.	31.0	214.	1.6	11.	70.5	486.	3.0	21.	84.0	579.	5.0	34.	33.0	227.	4.1	28.	100.0	689.
50	92	0.0	0.	3.4	23.	32.0	221.	1.8	12.	70.0	482.	2.8	19.	82.0	565.	17.5	121.	20.0	138.	4.2	29.	100.0	689.
50	96	0.0	0.	5.0	34.	28.0	193.	.8	6.	70.0	482.	2.7	19.	83.5	575.	6.0	41.	20.0	138.	3.9	27.	100.0	689.
50	100	0.0	0.	12.8	88.	32.5	224.	1.9	13.	70.5	486.	2.9	20.	84.0	579.	6.0	41.	21.0	145.	4.4	30.	100.0	689.
50	104	0.0	0.	7.0	48.	34.5	238.	2.0	14.	70.1	483.	2.9	20.	86.5	596.	7.0	48.	23.0	158.	4.4	30.	100.0	689.
50	108	0.0	0.	11.6	80.	34.5	238.	1.9	13.	70.0	482.	3.0	21.	84.5	582.	6.4	44.	23.0	158.	4.3	30.	99.0	682.
59	112	0.0	0.	6.1	42.	35.0	241.	1.5	10.	70.0	482.	3.0	21.	83.2	573.	5.6	39.	21.0	145.	4.1	28.	100.0	689.

TABLE I-4B

RUN 74 PRESSURE GAUGE READINGS

INPUT DATA - PI(12-23) PSI AND KPA

LS	COT	912		913		914		915		917		918		919		920		922		923	
59	4	14.1	97.	13.4	92.	12.3	85.	100.0	689.	13.2	91.	13.2	91.	39.0	269.	12.3	85.	20.5	141.	48.0	331.
59	8	14.2	98.	13.4	92.	13.2	91.	100.0	689.	13.2	91.	13.2	91.	40.5	279.	12.3	85.	20.5	141.	48.0	331.
59	12	14.0	96.	13.3	92.	13.2	91.	100.0	689.	13.3	92.	13.2	91.	39.0	269.	12.3	85.	20.5	141.	48.0	331.
59	16	14.0	96.	13.3	92.	13.2	91.	99.0	682.	13.3	92.	13.2	91.	39.0	269.	12.3	85.	20.5	141.	49.0	338.
59	20	14.1	97.	13.4	92.	13.4	92.	100.0	689.	13.3	92.	13.2	91.	42.0	289.	12.3	85.	20.5	141.	48.0	331.
59	24	13.9	96.	13.2	91.	13.1	90.	99.0	682.	13.3	92.	13.1	90.	30.0	207.	12.3	85.	20.5	141.	48.5	334.
59	28	14.0	96.	13.2	91.	13.1	90.	100.0	689.	13.3	92.	13.1	90.	32.0	221.	12.3	85.	20.5	141.	48.5	334.
59	32	14.0	96.	13.2	91.	13.1	90.	100.0	689.	13.3	92.	13.1	90.	30.0	207.	12.3	85.	20.5	141.	48.5	334.
59	36	14.1	97.	13.3	92.	13.2	91.	100.0	689.	13.3	92.	13.1	90.	30.0	207.	12.3	85.	20.5	141.	48.0	331.
59	40	14.1	97.	13.6	94.	13.5	93.	100.0	689.	13.3	92.	13.1	90.	30.2	208.	12.3	85.	20.5	141.	48.0	331.
59	44	14.2	98.	13.5	93.	13.4	92.	98.0	675.	13.3	92.	13.1	90.	34.0	234.	12.3	85.	20.5	141.	49.0	338.
59	48	14.2	98.	13.6	94.	13.5	93.	100.0	689.	13.3	92.	13.3	92.	33.0	227.	12.3	85.	20.4	141.	49.0	338.
59	52	14.2	98.	13.5	93.	13.4	92.	100.0	689.	13.3	92.	13.2	91.	32.0	221.	12.3	85.	20.5	141.	48.5	334.
59	56	14.1	97.	13.4	92.	13.4	92.	100.0	689.	13.2	91.	13.1	90.	33.0	227.	12.3	85.	20.5	141.	48.0	331.
59	60	13.9	96.	13.3	92.	13.2	91.	99.0	682.	13.2	91.	13.1	90.	24.0	165.	12.3	85.	20.5	141.	48.0	331.
59	64	14.1	97.	13.4	92.	13.3	92.	100.0	689.	13.2	91.	13.1	90.	22.0	152.	12.3	85.	20.5	141.	48.0	331.
59	68	14.1	97.	13.4	92.	13.3	92.	100.0	689.	13.2	91.	13.1	90.	30.0	207.	12.3	85.	20.4	141.	49.0	338.
59	72	14.2	98.	13.3	92.	13.3	92.	100.0	689.	13.2	91.	13.1	90.	36.0	248.	12.3	85.	20.4	141.	49.0	338.
59	76	14.2	98.	13.4	92.	13.3	92.	100.0	689.	13.2	91.	13.1	90.	32.0	221.	12.3	85.	20.5	141.	48.0	331.
59	80	14.2	98.	13.2	91.	13.1	90.	98.0	675.	13.2	91.	13.0	90.	35.0	241.	12.3	85.	20.5	141.	48.5	334.
59	84	14.2	98.	13.3	92.	13.2	91.	99.0	682.	13.2	91.	13.0	90.	36.0	248.	12.3	85.	20.5	141.	48.0	331.
59	88	14.0	96.	13.2	91.	13.2	91.	100.0	689.	13.2	91.	13.0	90.	34.0	234.	12.3	85.	20.5	141.	48.0	331.
59	92	14.2	98.	13.3	92.	13.2	91.	100.0	689.	13.2	91.	13.0	90.	39.0	269.	12.3	85.	20.4	141.	48.0	331.
59	96	14.2	98.	13.3	92.	13.2	91.	100.0	689.	13.2	91.	13.2	91.	37.0	255.	12.3	85.	20.4	141.	48.0	331.
59	100	14.4	99.	13.4	92.	13.3	92.	100.0	689.	13.2	91.	13.1	90.	33.0	227.	12.3	85.	20.5	141.	48.0	331.
59	104	14.5	100.	13.2	91.	13.2	91.	100.0	689.	13.2	91.	12.9	89.	33.0	227.	12.3	85.	20.5	141.	48.5	334.
59	108	14.3	99.	13.2	91.	13.3	92.	99.0	682.	13.2	91.	12.9	89.	33.0	227.	12.3	85.	20.4	141.	48.5	334.
59	112	14.5	100.	13.3	92.	13.3	92.	100.0	689.	13.2	91.	12.8	88.	39.0	269.	12.3	85.	20.5	141.	48.0	331.

APPENDIX J

CALCULATION OF SODIUM MOLE PERCENT IN CALCINE

In calculating the sodium mole percent of calcine produced from a Zr-Na wastes blend, only the major constituents are considered: aluminum, zirconium, fluoride, calcium, and oxide. During calcination of the blend, all the sodium (Na) is assumed to form Na_3AlF_6; the remaining fluoride (not used in Na_3AlF_6) is assumed to form CaF_2; the remaining aluminum (not used in Na_3AlF_6) is assumed to form Al_2O_3; the remaining calcium (not used in CaF_2) is assumed to form CaO; and the zirconium (Zr) is assumed to form ZrO_2.

As an example of this calculation, the mole percent of Na in a blend of 3 vol WM-185 waste (Table I) with 1 vol of the Na waste of Table II is calculated below. Concentration of major constituents in the blend are:

1. Sodium (Na) = $\dfrac{1.8\ \underline{M} \times 1}{4} = 0.45\underline{M}$

2. Aluminum (Al) = $\dfrac{(0.51\underline{M} \times 1) + (0.68\underline{M} \times 3)}{4} = 0.64\underline{M}$

3. Fluoride (F) = $\dfrac{3.1\underline{M} \times 3}{4} = 2.32\underline{M}$

4. Calcium (Ca) = Ca/F mole ratio is 0.7 or $\dfrac{Ca}{2.32\underline{M}} = 0.7$ or

   Ca = 0.7 x 2.32$\underline{M}$ = 1.62$\underline{M}$

5. Zr = $\dfrac{0.44\underline{M} \times 3}{4} = 0.33\underline{M}$

Thus, calcine from 1 L of blend contains: 0.45 moles of Na, 0.64 moles of Al, 2.32 moles of F, 1.62 moles of Ca, and 0.33 moles of Zr; but the moles of oxide (O) are unknown.

To calculate moles of 0:

1. 0.45 moles of Na, 0.15 moles of Al (1 mole of Al/every 3 moles of Na used), and 0.9 moles of F (2 moles of F/mole of Na used) are used to form Na_3AlF_6.

2. (2.32 - 0.9) moles of F and 0.71 moles of Ca (1 mole of Ca/every 2 moles of F used) are used to form CaF_2.

3. There are (0.64 - 0.15) moles of Al, (1.62 - 0.71) moles of Ca, and 0.33 moles of Zr to form oxides.

4. Moles of oxide = 0.74 moles as Al_2O_3 + 0.91 moles as CaO + 0.66 moles as ZrO_2 = 2.31 moles.

Thus mole percent of Na in calcine =

$$\frac{0.45(Na)}{0.45(Na) + 0.64(Al) + 2.32(F) + 1.62(Ca) + 0.33(Zr) + 2.31(0)} (100) = 5.9\%$$