FE-2007-25
Distribution Category UC-90d

FILTRATION PROCESS AND EQUIPMENT STUDIES FOR COAL LIQUEFACTION PROCESSES

QUARTERLY REPORT FOR SEPTEMBER - NOVEMBER 1976

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SUBMITTED BY:

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DECEMBER 16, 1976

ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION
UNDER CONTRACT E(49-18)-2007

OBJECTIVES

The ultimate objective of the subject two-phase program is to achieve major improvements in the filtration process and in the filtration equipment used in the coal liquefaction process. Reproducible doubling of the filtration rate, reduction of filter aid usage, and demonstration of these results on the pilot plant scale are ultimate goals. Only the first phase of the program has been contracted, and its immediate objectives are:

1. To develop basic knowledge of the coal oil filtration process and its dependence upon the various process parameters, and

2. To improve performance of filtration operations in existing pilot plants.

The Phase I program is to be implemented through:

1. Laboratory scale studies using a pressure precoat filter leaf system to simulate operation of a segment of a rotary pressure precoat filter, and

2. Consultation with and assistance to other ERDA contractors in efforts to optimize filtration procedures in their existing systems.

IMPORTANCE TO FOSSIL ENERGY TECHNOLOGY

Efficient solid-liquid separation is a "must" in coal liquefaction processes now under consideration. While other means of solid-liquid separation are being investigated, it would appear that filtration is most likely to succeed because of its inherent ability to produce a solids-free filtrate.

Since coal liquefaction processes constitute a large segment of the Fossil Energy Technology program, it is likely that filtration will be very important to the total Fossil Energy Technology effort.

TECHNICAL SUMMARY

During the quarter ending November 30, 1976, record flow rates for Tacoma Filter Feed in the order of 400 pounds/square foot of total filter area/hour were obtained. These rates were obtained at 600F and 40 psi differential pressure. The highest previous rate of 313 pounds/square foot/hour was obtained in August under similar conditions, but at 509F temperature.

As a result of having studied Tacoma Filter Feed in great detail, an experimental design was worked out to characterize filtration properties of other liquids in a minimum amount of time. Using this experimental design, filtration properties of uncentrifuged Synthoil and Wilsonville Filter Feed were determined.

Maximum rates for Tacoma, Wilsonville, and Synthoil materials were in the order of 400, 460, and 270 pounds/square foot/hour respectively at 600F, 35 psid, and 5 second immersions of the test leaf.

A series of runs was made during which the knife was not advanced to determine the amount of leakage, if any, that might occur around the edge of the precoat. After eighteen 5 second immersions during which a 3/32-inch

cake was allowed to build up on the precoat, the filtration rate was 45 pounds/square foot/hour.

Tests run on two grades of precoat material of different permeabilities indicate that lower knife cuts can be made with the lower permeability material without sacrificing rate.

One week was spent at the Pittsburgh and Midway Coal Company's plant in Fort Lewis, Washington.

EXPERIMENTAL DETAILS AND DISCUSSION

Record filtration rates for Tacoma Filter Feed of 388 and 438 (uncorrected, see below) pounds/square foot/hour (Runs B94 and B138, Table 1) were obtained. The 438 pound rate, however, included a timing error during immersion and should be corrected to approximately 400 pounds/square foot/hour. Both runs were made at approximately 600F, 40 psid, 5 second immersion, 10 mil blade advance, and a CELITE AF7 precoat. The highest previous rate of 313 pounds/square foot/hour was obtained in August under similar conditions, but at 509F.

The rate data from the series B79 through B87 clearly show the more mild (i.e., less than 40 psid differential pressure or less than 600F filtration temperature) sets of filtration conditions to be too mild to yield rates of greatest interest for the case of TFF 216.

Experimental Design

The series of tests typically run to gain filtration information are based on an augmented full factorial design (2^3), with center point. The principal variables (temperature, pressure differential, and blade advance) are treated at two principal levels each. The

augmentation includes insertion of (1) immersion time (3 levels) as a rider in every set of conditions, and (2) agitation (minimum and maximum feasible) as a second rider in the center point.

Wilsonville Filter Feed

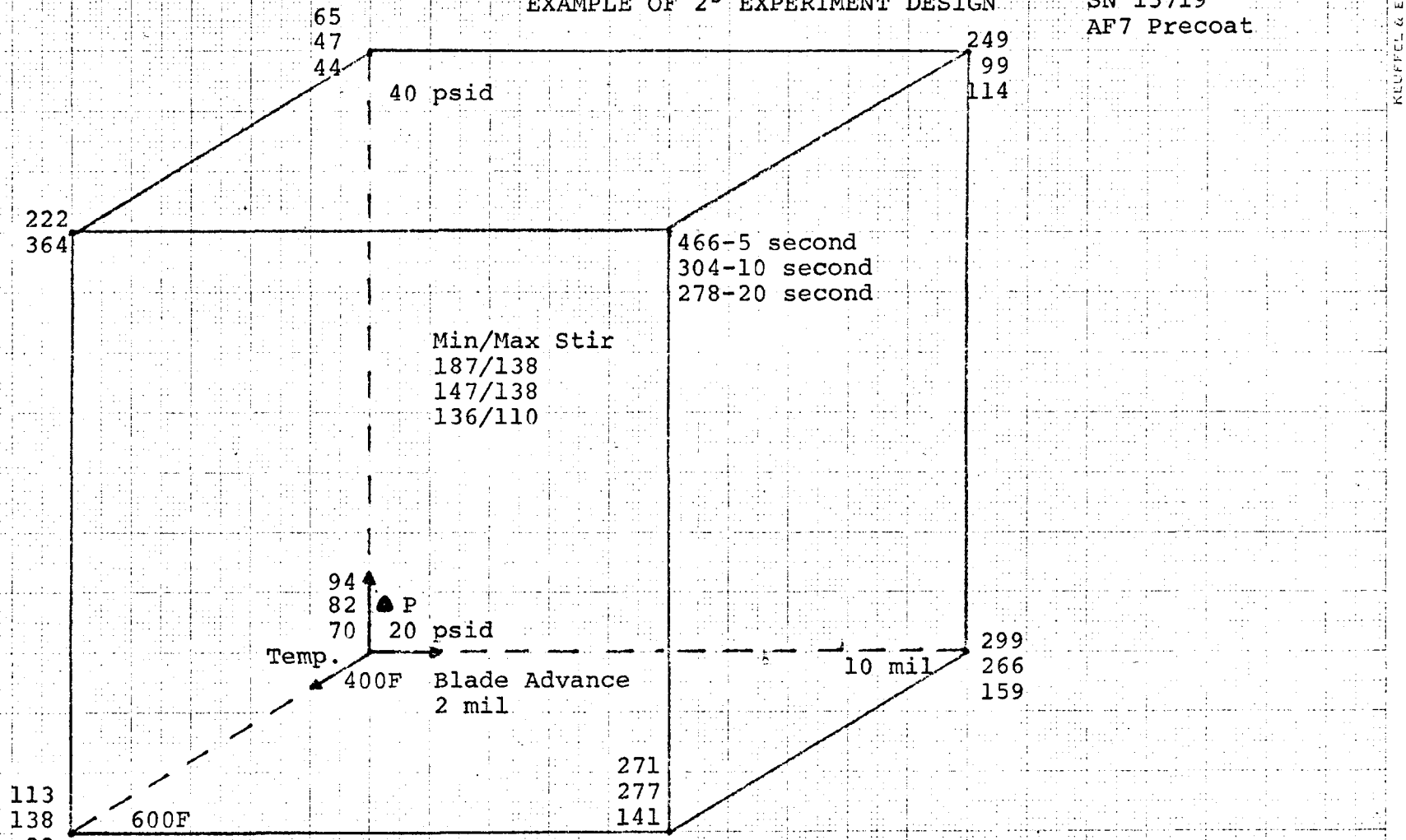
The data gathered from the above type of experimental design are shown on the cube in Figure 1 for Wilsonville Filter Feed (Runs C15 through C23, Table 1). The three vertical datum points at each apex of the cube and at the center are the result of the augmentation of three immersion times of the test leaf in the test liquid, the numbers from top to bottom representing immersion times of 5, 10, and 20 seconds respectively. The immersion time can then be translated into drum speed based on the assumption of a specific drum submergence. Thus, a submergence time of 5 seconds and an assumed drum submergence of 40 percent area can be translated into a drum speed of 4.8 rpm. Each datum refers to pounds of SRC and solvent filtered per square foot of total filter area (submerged and open) per hour.

The cube, front plane, represents all data taken at maximum temperature (600F), the right hand plane, all data at maximum rate of blade advance (mils per cut or drum revolution), and the upper plane, the maximum differential pressure run to date.

The overall results, although not entirely clear at present, indicate that the Wilsonville Filter Feed material filters more readily than the Tacoma Filter Feed under the same operating conditions. Further studies are planned including a rerun of the designed experiment with CELITE AF6 precoat material. The rate of 466 pounds/square foot/hour at 600F, 40 psid, and 10 mil blade advance should particularly be noted.

FIGURE 1 WILSONVILLE FILTER FEED
EXAMPLE OF 2^3 EXPERIMENT DESIGN

SN 15719
AF7 Precoat



Synthoil

A drum of uncentrifuged Synthoil (FB 45) was received from the Pittsburgh Energy Research Center of ERDA. The contents of the drum were warmed to near 200F and stirred by rolling for one hour. The drum was tapped and the contents divided among nine 5 gallon cans. Analysis of samples taken during the tapping showed very uniform ash contents averaging 3.86 percent and no trend in ash content from beginning to end of the operation.

The series B97 through B106 was designed as the basic exploratory experiment described above (2^3 with center point) and provided first experience with FB 45 (Synthoil). While the series provided useful familiarization with this rather different (from TFF) feed stock and served to demonstrate that reasonable filtration rates may be attained with this feed, the data are not clearly meaningful. There appears to have been some contamination of the charge with water (not yet confirmed), and a malfunction of the blade advance mechanism (discovered after the subsequent series) appears to have been in existence during this series.

The above Synthoil runs were rerun and data are shown as Runs B146 through C1. Again, a substantial amount of water was found in condensate from the Synthoil and in the filtrate, despite special precautions taken to assure that none was introduced in the work at Johns-Manville.

It is concluded that water was present in the Synthoil samples received, although it was not apparent as a second phase during transfer from the shipping drum to the 5 gallon paint cans in which it is stored.

Unusual shrinkage of the precoat was experienced, possibly due to the water present. However, two apparently reliable data points were generated in Run B146, and the subsequent runs of the series yielded essentially all data targeted. Run B146 was designed as the center point in the augmented 2^3 design, but because of the precoat shrinkage problem, the only meaningful data were those obtained with maximum stirring for 5 and 20 second immersions. Runs B148 and B150 were successful runs at the high temperature points of the design. Runs C1A and C1B were performed to assess (1) temperature dependence, by comparison with B148, and (2) the effect of stirring, C1A being performed with minimum feasible stirring and C1B with maximum feasible stirring. While in Runs B102A and B102B some suppression of rate by stirring was indicat this was not supported.

Analysis of the data has not been completed, but it is noted that filtration rates unexpectedly high for so viscous a fluid as Synthoil are observed under the most favorable conditions imposed. It appears that both the ΔP dependence and the temperature dependence of rate are greater than for the TFF 216. Blade advance dependence

appears greater at low ΔP, but smaller at high ΔP, as compared with TFF 216. Further, an anomoly appears in the ash contents of the various Synthoil filtrate samples. Analysis of the data is continuing.

Effects of Blade Advance Using Different Grades of Precoat Material

The series B109 through B116 was designed to assess dependence of rate upon blade advance when using an AF6 precoat (relatively fine), as compared to the case with AF7 in Runs B91 through B94. Post series examination of the precoat revealed a malfunction of the doctor blade mechanism. It is clear that actual cuts were achieved only in the last few instances. Data for Run B116 are probably meaningful. They support a modest suppression of rate by replacing AF7 with AF6. (Compare with Run B94.)

The series B91 and B94 was performed to explore the effect of blade advance under optimum conditions of temperature and pressure differential. A noteworthy increase of rate with blade advance is seen over the entire range covered. While it is estimated that "apparent rates" for this series were 8 to 10 percent below the "true rates", comparison of results with those of Run B87 reveals a probable advantage for the filtration under a differential pressure of 40 psid versus the 35 psid used in subject runs.

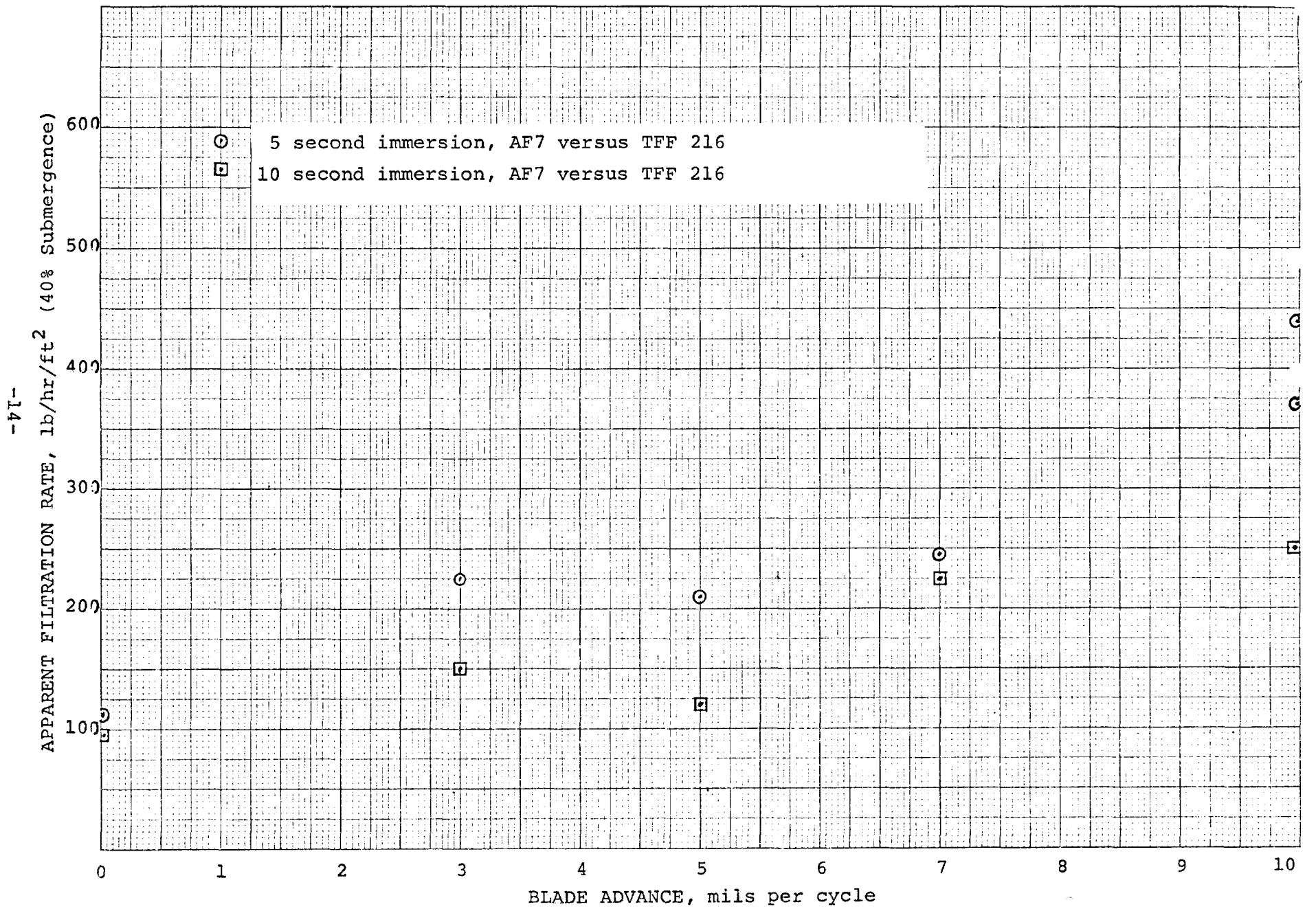
The series of Runs B120 through B127, was designed to refine the picture of dependence of filtration rate upon blade advance under near optimum conditions of temperature and pressure differential, but with more cycles at each set of conditions than for Runs B91 and B94. The apparent rate of 370 pounds/square foot/hour reported for 5 second immersions with intervening 10 mil cuts was that attained after sixteen cycles. The 251 pounds/square foot/hour reported for 10 second immersions is the average value of six such immersions (no trend evident) following nineteen 5 second immersions. Similar, though somewhat less extensive, sequences were employed to obtain the data for 7 mil and 5 mil advances. A rather severe leak from the vapor space of the main vessel of the apparatus forced the abortion of Run B127 and may have resulted in suppression of rate in Runs B123 and B125 due to excessive loss of lights from the charge.

Run B135 was a rerun of B127, but with a different charge and different precoat. Rates, being higher than those attained in B125 (5 mil advance), suggest that rates from the latter were indeed suppressed by the loss of lights, although differences in charge, precoat, or breakout may contribute to the discrepancy. Rates recorded for Run B138 (10 mil advance) may be as much as 10 percent high because of faulty timing of immersions. However, data from all these runs directed toward

delineation of the blade advance dependence are presented in Figure 2, together with those from Run 141, which was made with a cut after each immersion, but with no blade advance between cuts. It should be noted that no notable difference in rate was observed in Run B138 when pressure differential was increased from 34 to 40 psid. It, therefore, seems appropriate to consider data from Run B141 along with the other data in Figure 2.

Even with the several uncertainties associated with the data of Figure 2, a reasonably clear representation of blade advance dependence emerges. Note especially the much heavier dependence for 5 second immersions than for 10 second immersions. It is anticipated that some guidance in selecting most economical compromises between high filtration rate and long precoat life may be obtained from such plots as this.

FIGURE 2 DEPENDENCE ILTRATION RATE ON BLADE ADVANCE



Tacoma Filter Feed - Different Precoat Grades

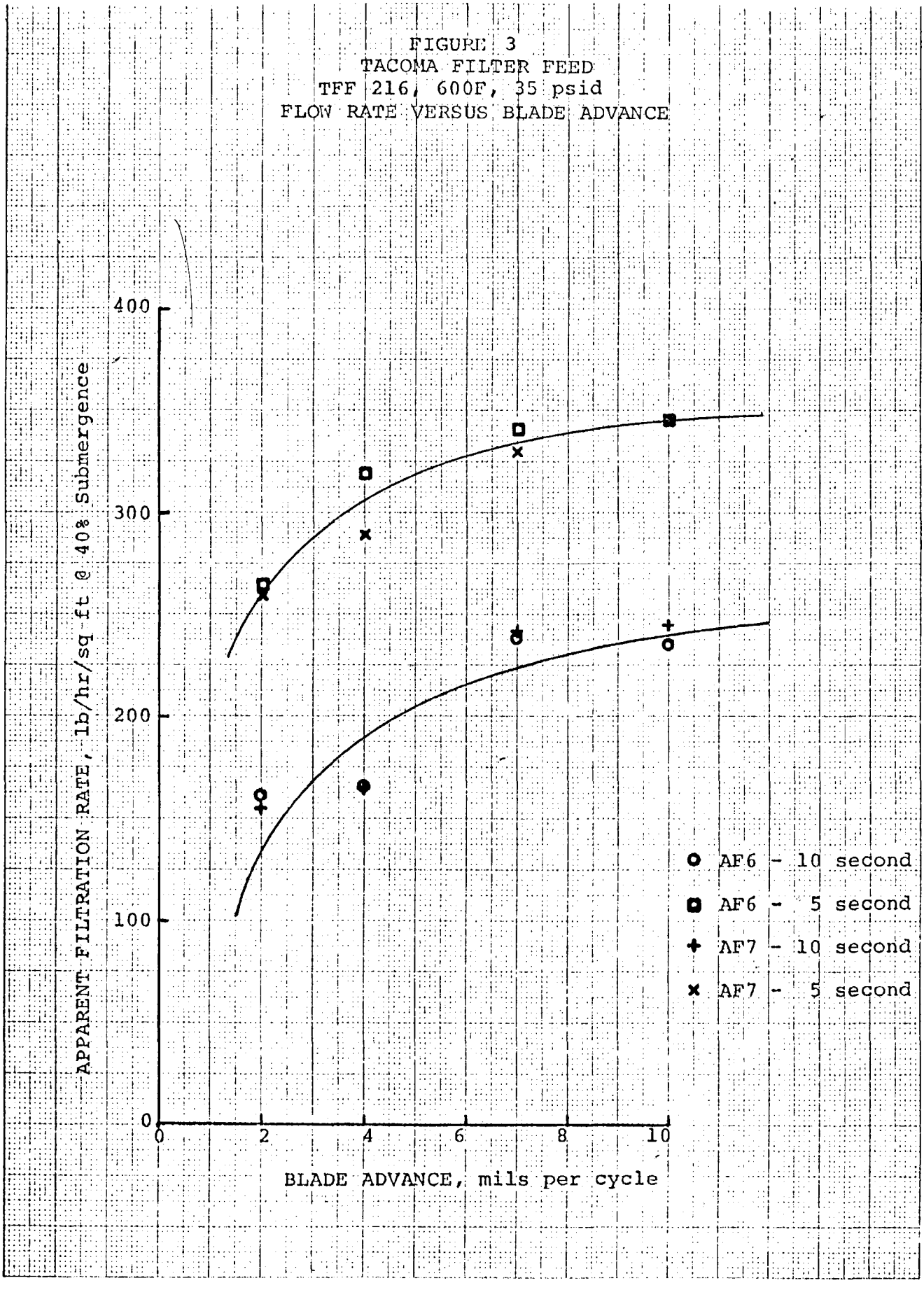
The purpose of the four run series C5 though C11 was to determine the filtration rates of Tacoma Filter Feed obtained with CELITE AF6 (CELITE is a registered trademark of the Johns-Manville Corporation) as the precoat material and then compare these rates with the rates obtained with CELITE AF7 as the precoat material. The precoat material CELITE AF6 with a median pore diameter of 5.5 microns might provide additional filtrate flow resistance when compared with CELITE AF7 with a median pore diameter of 7.5 microns. The data from these four runs are not reliable due to a suspected significant, unknown reduction in filter area as a result of a large breakout of precoat material sometime during the course of the runs.

Runs C27 through C33 were a rerun of Runs C5 through C11 with CELITE AF6 precoat material and can be compared with Runs C37 through C43 which were identical, but with CELITE AF7 precoat material. The rate data obtained are shown in Figure 3 and tabulated in a more concise from in Table 2. Note, rates as high as 345 pounds/square foot/hour at 40 percent drum submergence have been obtained.

The results indicate no apparent overall difference in filtration rate through precoats of CELITE AF6 and AF7. There may be a difference in rate for 5 second immersions at 4 mil knife advance per cycle. If this difference is

real, there may be less penetration of the fine coal solids into the CELITE AF6 precoat than into the CELITE AF7 precoat. Therefore, a series of runs are planned with CELITE AF5 (median pore diameter of 4 microns) as the precoat material to investigate this concept further. If the penetration of the fine coal solids is less with no significant reduction of filtration rate, filter aid economy can be increased.

FIGURE 3
TACOMA FILTER FEED
TFF 216, 600F, 35 psid
FLOW RATE VERSUS BLADE ADVANCE



Effects of Differential Pressure

Run B138, discussed in connection with Figure 1 above, was performed primarily in the interest of resolving the question of optimum pressure differential (ΔP) for filtration using the materials and conditions which have been of principal concern to the subject study to date. In earlier surveys of dependence upon this parameter, it has been clear that rate increases with ΔP up to 35 psid, but no further increase (indeed, some evidence of decrease) has been observed above about 40 psid. The possible advantage of 40 over 35 psid has been in question. In Run B138, eleven filtration cycles were run at a ΔP of 34 psid and eight similar cycles were run at 40 psid. No significant difference in rate was observed. Further experiments to refine the picture of ΔP dependence at levels above 35 psid are in order.

Filter Cake Permeability

Run B143 was performed to assess the permeability of the filter cake deposited from Tacoma Filter Feed (TFF) in a long series of short immersions, as would pertain at the exposed sidewall of the precoat on a rotary drum. The 45 pounds/square foot/hour value presented in Table 1 is that observed after eighteen 5 second cycles. Although the filtrate sample collected was too small to support very solid conclusions, it is clear that some flow

continued through the filter cake, which had built up to a thickness somewhat greater than 3/32 of an inch. It is possible that the continuing flow may be attributed to cracking of the cake due to drying during the blow-down stage of the cycle.

It is estimated that, if flow at the rate observed in Run B143 persisted through the partially blinded sidewall of the precoat in the laboratory scale apparatus, it could introduce a maximum error into the rate calculations of about 10 percent. This great an error would be likely to pertain only early in a series of runs and with small blade advances.

Immersion Times

It will be noted that, in a number of cases, filtration rates reported for 10 second immersions are lower than those for corresponding 20 second immersions. This relationship appears most frequently in the relatively low flow rate cases. In all cases, the immersions were run in the sequence: 20 second, 10 second, 5 second, 20 second. The phenomenon has not yet been explained. However, it may be related to the fact that the 10 second immersions in these necessarily brief sets are affected by their following the relatively long 20 second immersions, while the 20 second immersions follow those of only 5 seconds duration. Factors such as depth of penetration of fine coal solids into the precoat may be of

significance. Experiments to resolve the question are planned.

Support Efforts

Occasional required repairs, maintenance, and refurbishing of electrical and mechanical components of the rotary leaf pressure precoat filter system consumed significant staff time in September and October. However, no major equipment problems were encountered and no significant modifications were made.

TABLE 1. DATA FROM FILTRATION RUNS

RUN NO.	PRECOAT	CHARGE	TEMPERATURE °F	PRESSURE DIFFERENTIAL psid	BLADE ADVANCE mils	APPARENT FILTRATION RATE* 5 second	10 second	20 second	FILTRATE ANALYSIS PERCENT ASH	PERCENT SULFUR
B79	AF7	TFF 216	383	20	2	44	50	12	0.06	0.45
					10	39		24		
B81	AF7	TFF 216	409	40	2	55	44	13	0.07	0.42
					10	208		106		
B83A	AF7	TFF 216	497	30	5	249	162	113	0.04	0.43
B83B	AF7	TFF 216	497	30	5	239	158	108		
B85	AF7	TFF 216	604	20	2	148	96	124	0.02	0.39
					10	282	158	176		
B87	AF7	TFF 216	607	40	2	187	180	119	0.06	0.39
					10	388	229	195		
B91	AF7	TFF 216	605	35	3	124	101	108	0.04	0.40
					5	229	132	138		
B94	AF7	TFF 216	610	35	7	271	127	164	0.05	
					10	338	205	163		
B97	AF7	FB 45	399	20	2	18	8	13		0.28
					10	20	3	3		
B100	AF7	FB 45	402	40	2	29	22	3	0.09	0.48
					10	13	3	22		
B102A	AF7	FB 45	508	30	5	104	65	46	0.04	0.49
B102B	AF7	FB 45	508	30	5	86	57	34		
B104	AF7	FB 45	603	20	2	72	84	27	0.04	
					10	152	59	62		
B106	AF7	FB 45	601	38	2	177	87	61	0.05	0.80
					10	195	113	86		
B109	AF6	TFF 216	610	34	3	55	62	48		
B112	AF6	TFF 216	595	35	5	63	27	19		
B114	AF6	TFF 216	595	35	7	52	39	45		
B116	AF6	TFF 216	596	34	10	307	197	171		

TABLE 1. DATA FROM FILTRATION RUNS - NOVEMBER 1976

RUN NO.	PRECOAT	CHARGE	TEMPERATURE °F	PRESSURE DIFFERENTIAL psid	BLADE ADVANCE mils	APPARENT FILTRATION RATE* 5 second	10 second	20 second	FILTRATE ANALYSIS PERCENT ASH	PERCENT SULPHUR
C5[a]	AF6	TFF 216A	610	35	10	304	207		0.06	0.43
C7[a]	AF6	TFF 216A	610	35	7	288	202		0.04	0.41
C9[a]	AF6	TFF 216A	610	35	5	265	181		0.05	
C11[a]	AF6	TFF 216A	612	35	3	217	162		0.02	0.39
[a]Post Run examination indicated very serious breakout of precoat; rate data very suspect; See Runs C27 through C33										
C15	AF7	SN 15719	399	20	10	299	266	159	0.04	0.53
					2	94	82	70	0.04	0.54
C17	AF7	SN 15719	406	40	10	249	99	114	0.05	0.49
					2	65	47	44	0.04	
C19A	AF7	SN 15719	501	30	5	187	147	136	0.04	0.55
C19B	AF7	SN 15719	501	30	5	138	138	110	0.03	0.44
C21	AF7	SN 15719	600	21	10	271	277	141	0.02	0.47
					2	113	138	82	0.01	
C23	AF7	SN 15719	605	39	10	466	304	278	0.03	0.51
					2	222	364		0.04	
C27	AF6	TFF 216A	619	35	10	345	235		0.04	0.51
C29	AF6	TFF 216A	613	35	2	264	161		0.04	0.34
C31	AF6	TFF 216A	624	35	7	341	238		0.04	0.37
C33	AF6	TFF 216A	615	35	4	319	166		0.04	
C37	AF7	TFF 216A	615	35	10		250		0.12	0.27
C39	AF7	TFF 216A	608	36	2	259	154		0.12	0.40
C41	AF7	TFF 216A	613	35	10	345	244		0.10	
					7	330	241		0.09	0.38
C43	AF7	TFF 216A	610	35	4	289	164		0.12	0.43

TABLE 1. DATA FROM FILTRATION RUNS

RUN NO.	PRECOAT	CHARGE	TEMPERATURE °F	PRESSURE DIFFERENTIAL psid	BLADE ADVANCE mils	APPARENT FILTRATION RATE* 5 second	10 second	20 second	FILTRATE ANALYSIS PERCENT ASH	PERCENT SULFUR
B120	AF7	TFF 216	598	34	10	370	251		0.05	0.38
B123	AF7	TFF 216	598	35	7	244	228			
B125	AF7	TFF 216	600	35	5	210	118			
B127	AF7	TFF 216	600	35	3					
B135	AF7	TFF 216	609	35	3	225	150		0.07	0.41
B138	AF7	TFF 216	608	34 40	10	437 438			0.03	
B141	AF7	TFF 216	606	40	0	112	96		0.03	
B143	AF7	TFF 216	600	40	-	45		32	0.03	
B146	AF7	FB 45	508	30	5	128		70	0.02	0.44
B148	AF7	FB 45	603	40	10 2	269 171	133 91	105 ~70	0.10	0.46
B150	AF7	FB 45	606	20	10 2	185 76	90 37	71 24	0.08	
C1A	AF7	FB 45	508	40	10	171	121	76	0.08	
C1B	AF7	FB 45		40	10	198	~110	77		

TABLE 2

TFF 216, 600F, 35 psid

BLADE ADVANCE Mils Per Cycle	AF6 PRECOAT		AF7 PRECOAT	
	5 Second	10 Second	5 Second	10 Second
10	345	235	345	244
7	341	238	330	241
4	319	166	289	164
2	264	161	259	154

Apparent Filtration Rate

Apparent filtration rate terminology has been used in the discussions and some explanation of its meaning is desirable.

Rate is calculated in pounds/square foot/hour, on the basis of total area of a rotary precoat filter. Apparent rates are calculated on the assumption that full area of filter leaf was active. Since this is not true in many cases, true rates are generally higher than apparent rates.

The term "Apparent Filtration Rate", as defined above, is used to denote rates calculated from observed rates under the assumption that the entire precoat area as defined by the face of the filter leaf is presented unencumbered (not blinded) to the filter cake at the beginning of each immersion. Inspection of the cake/precoat after a series of runs typically reveals some degree of encumbrance which developed at some unidentifiable point in the series. Thus, "apparent" rates are in every case equal to or smaller than true rates in effect at the time of the measurement. From post-run inspections, it is estimated that apparent rates may be from 1 or 2 percent to 40 percent low. The 40 percent represents an extreme case; typically apparent rates are less than 25 percent low.

TASK III - CONFIRMATION TESTS AT GULF, FORT LEWIS, WASHINGTON

Original plans were to move the filter test equipment to Fort Lewis to develop laboratory data on fresh filter feed.

The process of dismantling the test equipment, packing, shipping to Fort Lewis, reassembly, and shakedown is estimated at four to six weeks. Therefore, to save time and expense (including living expenses for Johns-Manville personnel at Tacoma) arrangements are being made to have filter feed samples shipped hot from Fort Lewis to Denver where they will be tested before being allowed to cool.

A hot (200 to 300F) sample container is being assembled and plans are being made to obtain a fresh, hot sample of filter feed for testing in Denver during December 1976.

TASK IV - OPTIMIZATION OF PAMCO SRC FILTRATION

Monday afternoon, November 8 to noon, November 13, was spent at the Pittsburgh and Midway Coal Mining Company, Fort Lewis, Washington. Shortly after the writer's arrival, the plant was shut down until Friday. Because of the plant shut down, more time was spent in the areas outlined below. The writer considers this time very well spent and wishes to convey appreciation to PAMCO personnel for their assistance and hospitality.

The following was reviewed or accomplished:

1. Safety and health procedures were reviewed with plant industrial hygienist.

2. The Energy Research & Development Administration sponsored program being run by Johns-Manville, "Filtration Process and Equipment Studies for Coal Liquefaction Processes" was reviewed.

3. A seminar was given to approximately 25 PAMCO personnel on the elements of rotary drum precoat filtration.

4. Future rotary precoat test procedures and programs were discussed.

5. The rotary drum precoat filter station was gone over and recommendations were made for changes in precoat procedures.

6. The obtaining and transportation temperature of a hot sample of filter feed material for testing at Denver was discussed and sample points and transportation temperature decided upon.

7. Drawings of the Electric Power Research Institute sponsored 50 Square Foot Pilot Filter were reviewed.

8. It was the opinion of the writer and Fort Lewis personnel that it would not be practical in terms of time and operation to ship the rotary precoat test leaf from Denver to Fort Lewis.

9. Some few observations (due to the plant shut down during most of the writer's visit) were made during filtering and precoating on the 80 Square Foot Goslin-Birmingham filter.

The filter was started Friday afternoon using a precoat held over from Tuesday. Because of the age of the precoat, there was little to be gained in running tests on it. The filter was reprecoated Saturday and the writer was able to observe only part of the precoat application before having to leave for Denver.