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EVALUATION, ENGINEERING AND DEVELOPMENT OF ADVANCED CYCLONE PROCESSES

FINAL SEPARATING MEDIA EVALUATION AND TEST REPORT

(FSMER)

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DISCLAIMER

EXECUTIVE SUMMARY

"Evaluation Engineering and Development of Advanced Cyclone Processes" is one of the DOE-PETC sponsored advanced coal cleaning projects, which share a number of specific goals. These goals are to produce a 6% ash product, reject 85% of the parent coal's pyritic sulfur, recover 85% of the parent coal's Btu value, and provide products that are less than 30% moisture.

The process in this project, as the name implies, relies on a cyclone or cyclonic separator to achieve physical beneficiation based on the gravimetric differences between clean coal and its impurities. Just as important as the cyclonic separator, if not more so, is the selection of a parting liquid or medium for use in the separator. Selection of a separating medium is regarded as a significant portion of the project because it has a profound impact on the required unit operations, the performance of the separator, and economics of the process. The choice of medium especially influences selection of media recovery system(s), and the characteristics of clean coal and refuse products. Since medium selection is such an important aspect of the project, portions of the project are dedicated to the study, evaluation, and selection of the most desirable medium. Though separators are an important component, this project initially focused on media study, rather than the separators themselves. In coal processing, discussion of media requires description of the handling and recovery system(s), separation performance, interaction with coal, cost, and health, environmental and safety issues. In order to be effective, a candidate must perform well in all of these categories.

This Final Separating Media Evaluation Report presents a review of Phase I - Separating Media Evaluation, and Phase II - Separating Media Testing. Phase I evaluated the media on paper and in laboratory experiments. Separation performance data was collected during operation of a 1,000 lb/hr pilot plant and at several vendors' test facilities during Phase II. The report concludes by recommending that an aqueous calcium nitrate medium in association with a decanter centrifuge separator be used in Phase III - Process Optimization Testing. An organic mixture of methylene chloride and perchloroethylene was also evaluated but not recommended for Phase III. This document compares these two potential media. Various aspects of each medium were considered including separation performance, the regulatory atmosphere, risks to the environment and workers' health, availability and cost of major components and equipment to recover and regenerate the media as well as beneficiate the feed coal, and comparative maintenance and operating costs.

TABLE OF CONTENTS

not included with this report. In the appendix. ds 6-20-96

LIST OF TABLES

LIST OF FIGURES

1.0 INTRODUCTION

Work in Phase I, Media Evaluation, considered nearly 50 media candidates. Nine candidate media were selected for more detailed laboratory evaluation. At the conclusion of Phase I, an aqueous solution of calcium nitrate and an organic mixture of methylene chloride and perchloroethylene were selected for experimentation in Phase II, Media Testing. A number of separators were utilized in these tests. They were Krebs 2- and 4-inch cyclones, a PTI/DOE vertical centrifuge, Alfa-Laval (A-L) mini-clones, and an A-L centrifuge. This report represents the conclusion of Phase II and introduction to Phase III.

At the end of Phase I, the Project Team selected two media, an organic methylene chloride and perchloroethylene mixture, and an aqueous calcium nitrate solution for study in Phase II (Separating Media Testing) during which the media were tested with several separating devices. The results were compared to the washability of the test coal (Illinois No. 6) used during this phase. All tests were conducted in a 1,000 lb/hr closed loop circuit at Process Technology Incorporated's (PTI) facility except for several decanter centrifuge tests conducted at about 40 lb/hr at vendor facilities.

Throughout the report discussion, two methods of coal cleaning are deliberated. One uses an aqueous solution of calcium nitrate and the other an organic mixture of methylene chloride (mcl) and perchloroethylene (perc). In practice, calcium nitrate is an industrial grade fertilizer salt containing a small percentage of ammonia. Aqueous solutions of this salt can be adjusted anywhere from 1.0 (pure water) to about 1.50 sg at full saturation, depending on temperature. The organic liquid mixture can be adjusted from 1.33 to 1.6 sg, the specific gravity of mcl and perc, respectively. Utilizing these media are not a simple matter of replacing the current heavy media in commercial plants of today. Each requires different handling, equipment, and procedures for use in a coal preparation plant.

A complete process necessarily includes recovery and regeneration of the selected medium. Once contacted by the processing medium, product and refuse should be rinsed and the medium recovered for recycling. The entire layout of the plant, including coal handling and separation, medium formulation, recovery and regeneration equipment is termed a circuit. Aspects of the entire circuit must be considered when comparing media. Each circuit is discussed conceptually and compared to the other by several different measures.

This report presents the results of Phases I and II and provides the basis for recommending selection of a single medium for Phase III, Process Optimization Testing. Results from separation performance tests and filtration tests, circuit considerations, health and safety issues, and selected costs are discussed.

2.0 PERFORMANCE AND EFFICIENCY

Phase II tests were conducted using the Illinois No. 6 Seam coal. Complete results of these tests are contained in Quarterly Technical Progress Report #06. Sensitivity analysis and further study of these results will be conducted during Phase III, Process Optimization Testing. Data interpretation during Phase II determined media and conditions that gave the best results when compared to washability analyses and project goals.

Fourteen sets of tests were conducted with various operating conditions within each. The optimum results are indicated in Table 1. This table lists results according to the standard criteria of ash rejection, Btu recovery, and pyritic sulfur rejection. Both the organic and calcium nitrate media performed comparably well, though in different separators under different conditions.

Test 8 with the centrifuge represents achievement of the pyritic rejection goal, as do tests B14/T1 and T3 with a Krebs cyclone. The products from these tests were subjected to washability and partition curve analysis. These analyses represent the performance of the media. From these tests it was learned that a satisfactory separation is possible with either medium.

Throughout Phase II, performance with methylene chloride and perchloroethylene was better than with calcium nitrate in all separating devices. Mcl/perc was not tested in a decanter centrifuge, though it would be expected to perform well. A-L small diameter cyclones (mini-clones) tended to plug with either type of medium. With a 500 mesh feed, the A-L mini-clones performed better than the Krebs 2-inch cyclone, but this was not better then the Krebs cyclone on a 100 mesh basis. Calcium nitrate performed as well in the decanter as mcl/perc in a 2-inch Krebs cyclone. In other words, satisfactory performance using mcl/perc was obtained by using a Krebs 2-inch cyclone. Using calcium nitrate, satisfactory performance was obtained with a decanter centrifuge. Both combinations yielded grade/recovery relationships near the washability curve.

The comparison of the optimum tests to washability performance is shown in Figures 1A and 1B. Obviously the separation cannot be expected to do better than the washability. Partition curves from the optimum tests are shown in Figures 2, 3, and 4. These partition curves were used as a basis to simulate the separation of the four test coals by the indicated medium and separator (organic w/cyclone and aqueous w/decanter). The simulated results are shown in Table 2. The simulator takes into account

TABLE 1
COMPARATIVE PERFORMANCE RELATIVE TO RAW COAL
PHASE II ADVANCED CYCLONING PROJECT
OPTIMUM TEST RESULTS
Illinois No. 6 Seam Precleaned Coal
(Dry Basis)

	FINAL PRODUCT QUALITY			PERFORMANCE RELATIVE TO RAW COAL					
OPTIMUM TEST DESCRIPTION	ASH (Wt%)	SULFUR (Wt%)	PYRITIC SULFUR (Wt%)	YIELD (Wt%)	ENERGY RECOVERY (%)	ASH REMOVAL (Wt%)	SULFUR REMOVAL (Wt%)	PYRITIC SULFUR REMOVAL (Wt%)	PYRITIC SULFUR SEPARATION EFFICIENCY Index
Raw Coal (Phase I)	21.51	4.51	2.24	100.0	100.0	0.0	0.0	0.0	-
Precleaned Coal (Phase I)	11.05	3.60	1.43	77.0	86.5	60.4	38.5	50.8	267
100M - Washability (PTI)									
Float 1.50 Composite	4.83	2.63	0.34	67.6	81.5	84.8	60.6	89.8	1240
Float 1.60 Composite	5.49	2.66	0.39	70.0	83.7	82.2	58.8	87.9	1183
Float 1.80 Composite	6.03	2.69	0.43	71.4	84.9	80.0	57.5	86.2	1116
100M - MCL/PERC (PTI)									
2" Krebs (B14/T1)	6.26	2.69	0.39	62.2	75.2	83.8	64.9	90.5	978
2" Krebs (B14/T3)	7.23	2.77	0.44	67.2	80.1	79.7	61.4	88.5	1054
A/L Cyclones (B14/T7)	5.74	2.75	0.43	61.8	75.0	85.4	66.0	89.3	833
100M - $Ca(NO_3)_2$ (PTI)									
A/L Cyclones (B13/T16)	6.31	2.68	0.41	57.4	70.3	86.0	67.7	91.6	840
100M - $Ca(NO_3)_2$ (A/L)									
A/L Centrifuge (Test 8)	5.63	2.67	0.37	61.4	75.7	86.8	67.0	91.8	1089
100M - Water (HPM)									
Centrifloat (Test 10/3)	6.86	3.14	0.99	58.6	70.1	82.5	57.9	74.3	240
Centrifloat (Test 16/3)	7.61	0.35	1.13	60.6	72.2	81.3	55.8	71.0	228
Centrifloat (Test 14/1)	7.55	3.32	1.11	64.8	76.3	81.2	48.0	68.1	255
Centrifloat (Test 16/4)	7.70	3.38	1.15	64.7	77.2	79.9	52.2	68.3	256
Centrifloat (Test 14/4)	7.97	3.30	1.11	68.1	80.2	77.2	48.7	66.4	287
100M - Water (HPM)									
Centrifloat (Test 21/1)	6.76	3.08	0.86	59.5	70.6	81.4	57.6	75.9	275
Centrifloat (Test 22/3)	7.21	3.18	0.97	64.9	77.0	78.7	52.8	70.4	286
Centrifloat (Test 27/2)	7.14	3.20	0.96	70.1	82.5	76.6	47.8	66.2	322
Centrifloat (Test 27/3)	7.20	3.27	0.98	70.8	83.5	76.2	47.8	65.9	332
Centrifloat (Test 24/4)	7.41	3.29	1.05	72.7	84.8	73.5	44.5	61.9	315

NOTES: - Performance determined by combining results from precleaning and Phase II Test.
- Only tests achieving greater than 70% Energy Recovery and less than 8% product ash content were included in this table.

the sharpness of separation and shape of partition curves, changing somewhat with a change in medium gravity. A sharper separation (steeper partition curve at the d_{50} point) with decreasing medium gravity is an artifact of the program.

The Washability Report demonstrated that 100 mesh liberation could achieve the project goals for the four test coals. Separation performance and liberation are balanced at this point to achieve the project goals. The washability at 3/4 inch and 28, 100, 200 and 400 mesh were studied. It was found that 100 mesh was the largest topsize that could be used for all four test coals, representing the lowest common denominator to achieve the project goals. There would be no need to crush any finer than 100 mesh, but 100 mesh may be considered too fine for separation effectiveness. Fortunately, washability-type performance was closely approximated with the aforementioned optimum medium/separator combinations in Phase II. Therefore, 100 mesh was selected as the baseline top feed size for Phase III, Process Optimization Testing.

3.0 COST OF MEDIUM RECOVERY AND REGENERATION

Filtering and medium recovery tests were conducted using clean coal and refuse samples collected from Phase II. These experiments were meant to simulate the loss, recovery, and regeneration of medium in a commercial plant. These operations are critical to the process economics.

Calcium Nitrate Circuit

In conjunction with filtering to recover valuable medium, washing is used to rinse additional medium from the coal and refuse products. Washing can occur before the filtering stage by reslurrying with fresh water, on the filter with fresh water spray, or both. In either case the mother liquor (full strength medium) becomes dilute and less dense than the desired target gravity. Filter cake washing also lowers the ash of the clean coal product. All other factors remaining the same, the more wash water that is used the more medium recovered, and the more the water removal (thermal evaporation) costs are to regenerate the medium. This is best described as a functional relationship of the amount of fresh water used vs the residual amounts of medium remaining on the products. Thermal costs for medium recovery and regeneration need to be considered as well as the medium loss/make-up costs. The amount of residual medium on the plant products and the amount of wash water used reflect the medium make-up costs and the medium regeneration costs, respectively.

Filtration tests were conducted with two filter manufacturers, Outomec and Eimco who investigated aqueous phase filter

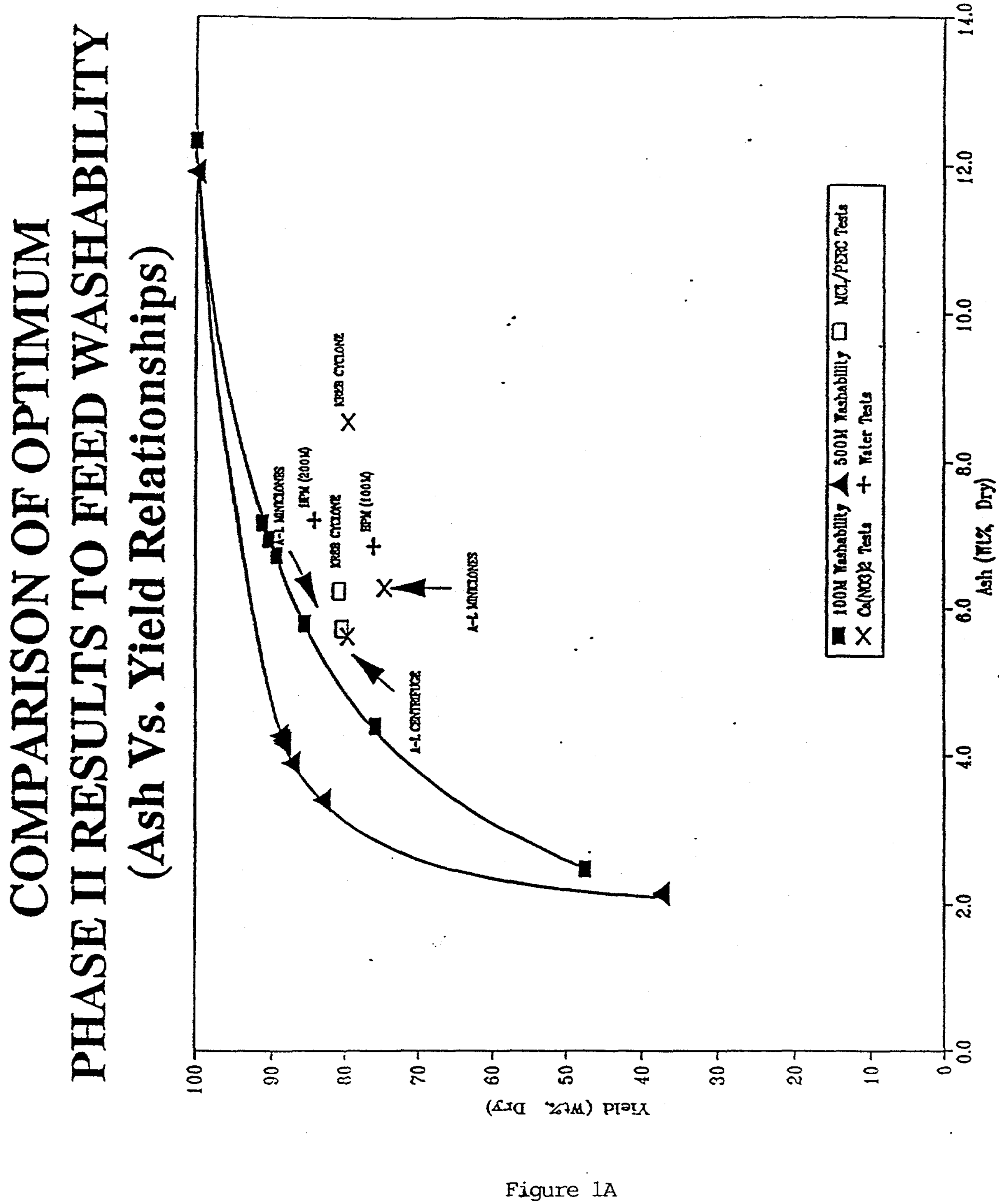

COMPARISON OF OPTIMUM
PHASE II RESULTS TO FEED WASHABILITY
(Ash Vs. Yield Relationships)
Yield (Wt%, Dry)
Ash (Wt%, Dry)
0.0
2.0
4.0
6.0
8.0
10.0
12.0
14.0
0
10
20
30
40
50
60
70
80
90
100
KREB CYCLONE
HPM (200M)
HPM (100M)
A-L MINICLONES
KREB CYCLONE
A-L MINICLONES
A-L CENTRIFUGE
100M Washability
500M Washability
NCL/PERC Tests
Ca(NO3)2 Tests
Water Tests

Figure 1A

COMPARISON OF OPTIMUM PHASE II RESULTS TO FEED WASHABILITY (Pyritic Sulfur Vs. Yield Relationships)

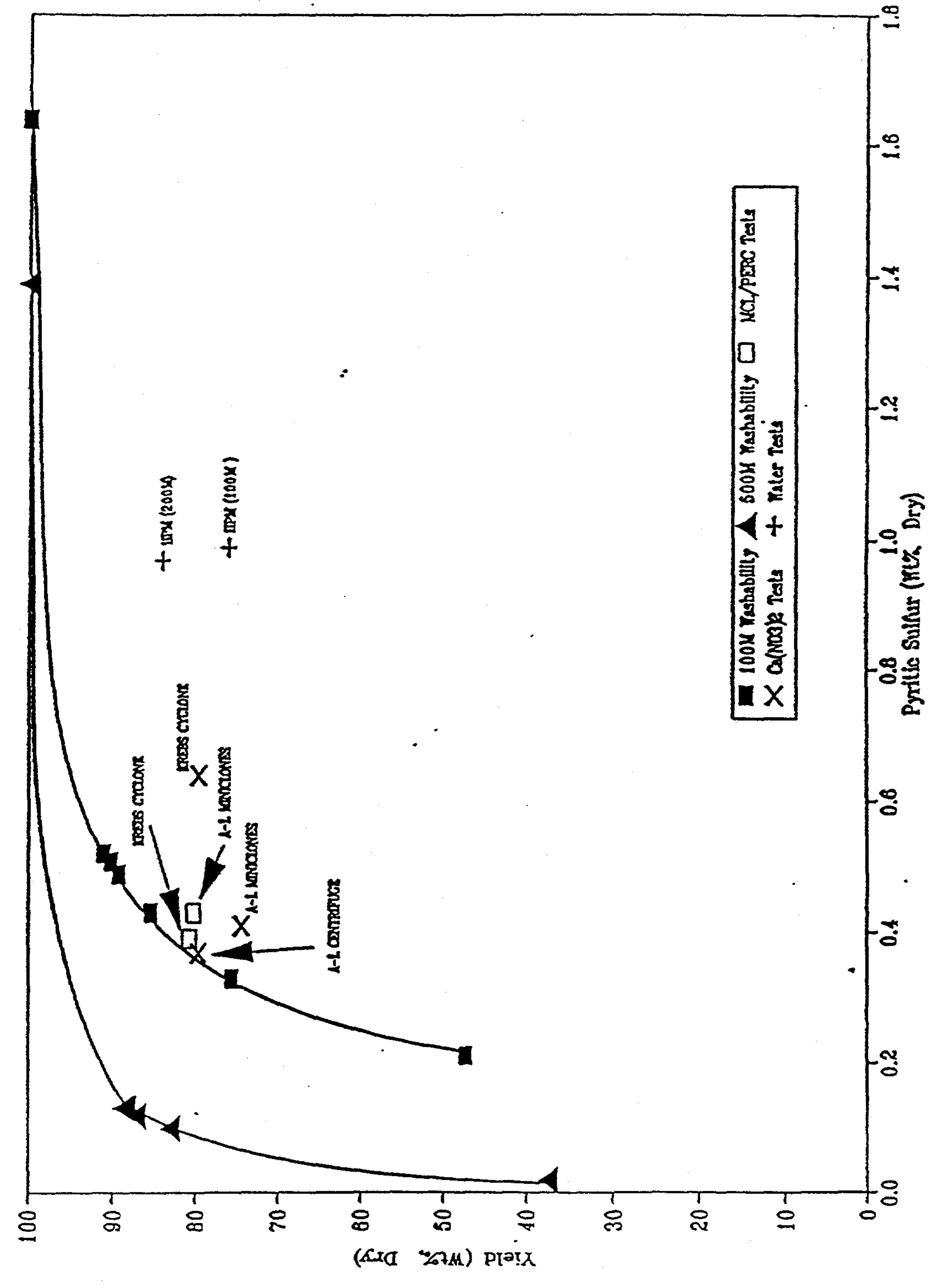
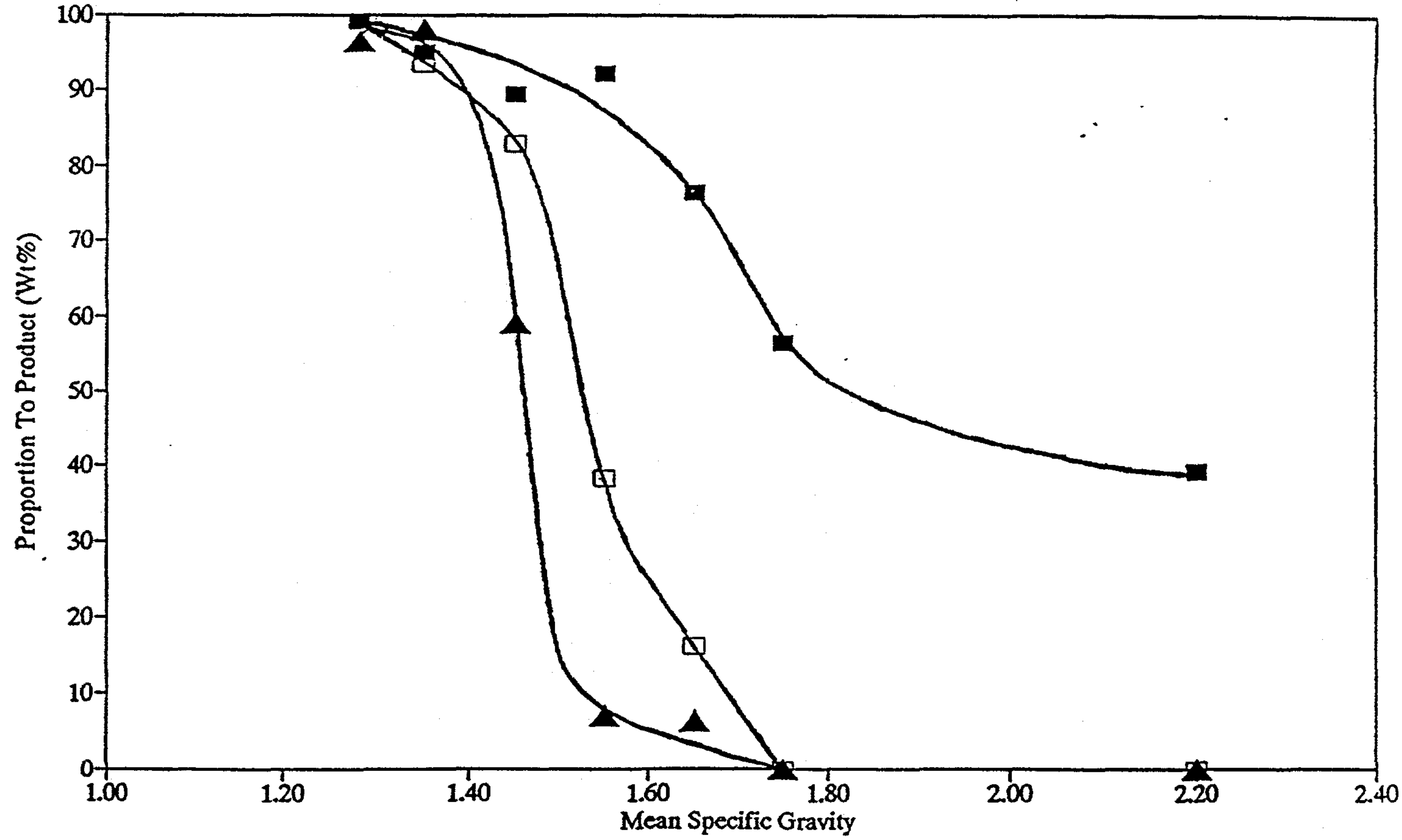


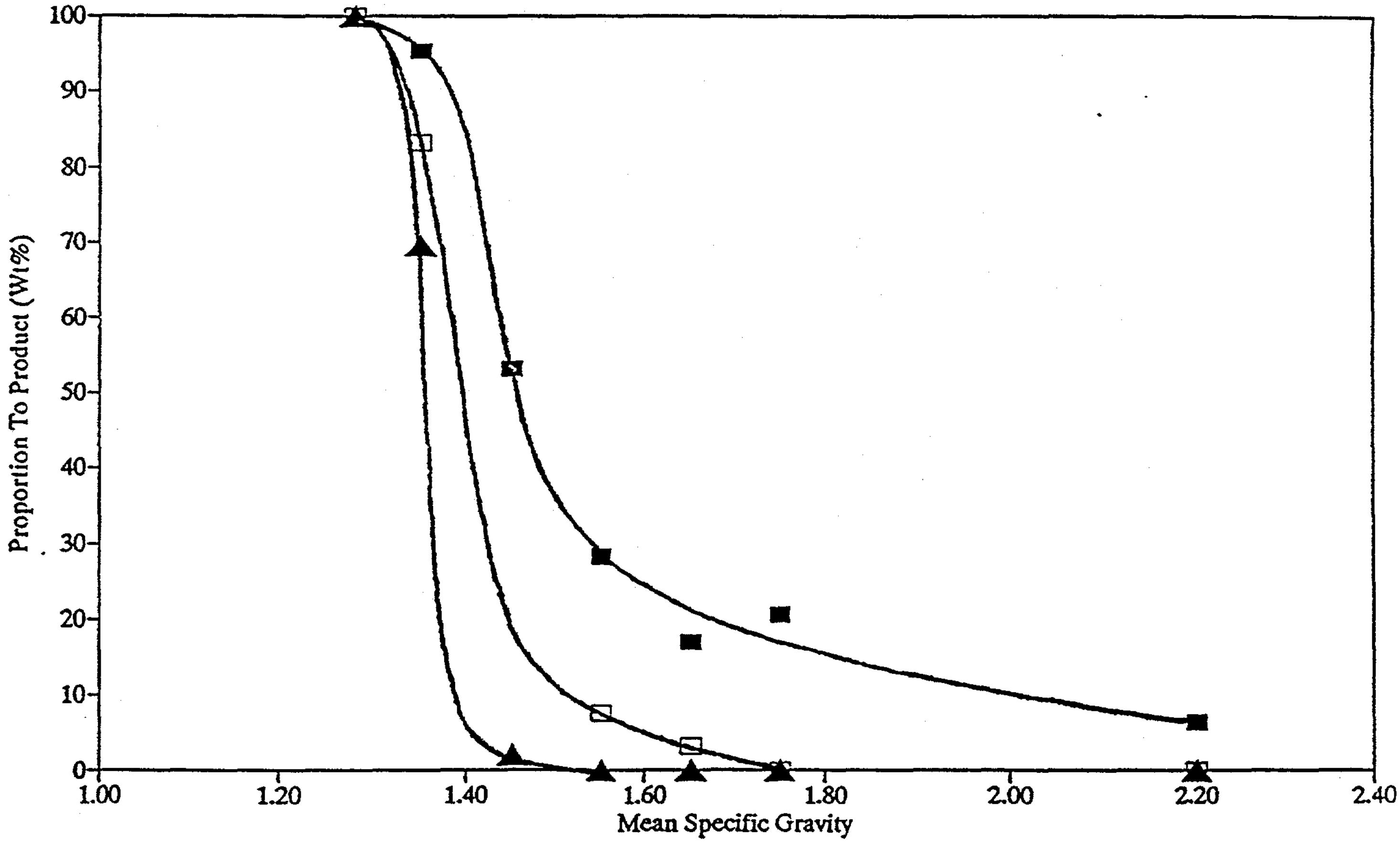
Figure 1B

Figure 2
OPTIMUM MCL/PERC MEDIA RESULTS
2" KREBS CYCLONE
(100M x 0, Illinois No. 6 Seam Precleaned Coal)



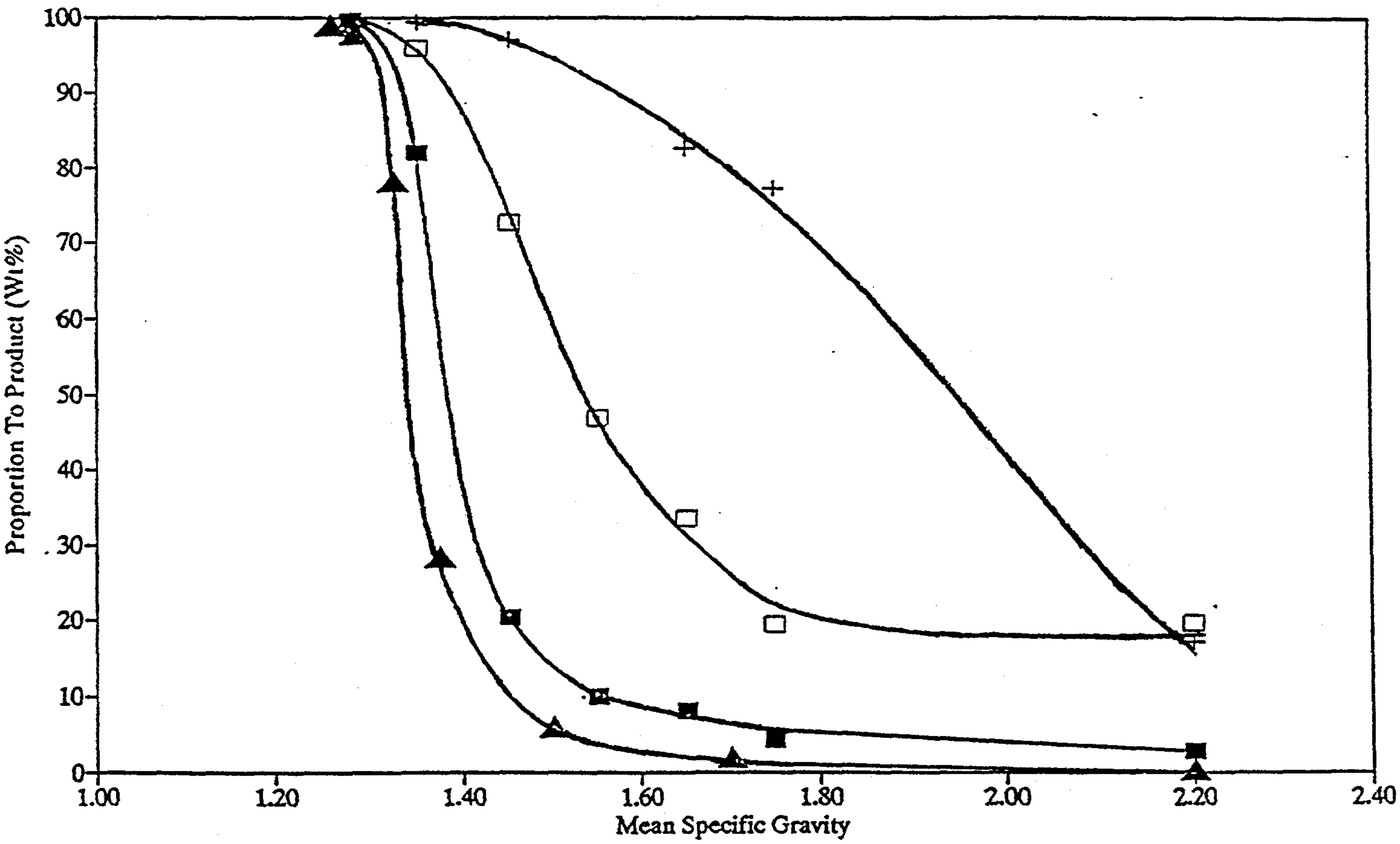
Legend	Size	Sep'n SG	Ep
▲	100M x 200M	1.46	0.030
□	200M x 400M	1.53	0.055
■	400M x 0	1.80	NA

Figure 3
OPTIMUM Ca(NO3)2 MEDIA RESULTS
ALFA-LAVAL HORIZONTAL CENTRIFUGE
(100M x 0, Illinois No. 6 Seam Precleaned Coal)



Legend	Size	Sep'n SG	Ep
▲	100M x 200M	1.36	0.020
□	200M x 400M	1.44	0.050
■	400M x 0	1.46	0.095

Figure 4
COMPARISON OF OPTIMUM OVERALL SEPARATIONS FOR VARIOUS MEDIA, COAL FEED SIZES, AND SEPARATOR OPTIONS TESTED



Legend	Media	Separator	Size	Media SG	Sep'n SG	Ep
▲	Micromag	3" Cyclone	28M x 400M	1.30	1.35	0.025
□	MCL/PERC	2" Cyclone	100M x 0	1.45	1.53	0.135
■	Ca(NO3)2	A-L Centrifuge	100M x 0	1.35	1.39	0.035
+	Ca(NO3)2	A-L Centrifuge	500M x 0	1.40	1.92	0.195

Table 2
SIMULATION RESULTS
(Advanced Cycloning Project)

	Final Product Quality					Performance Relative to Raw Coal					
	SG of Sep'n	Ash (Wt%)	Sulfur (Wt %)	Pyritic Sulfur (Wt%)	Heating Valve (BTU/lb.)	Yield (Wt%)	Energy Recovery (%)	Ash Removal (Wt%)	Sulfur Removal (Wt%)	Pyritic Sulfur Removal (Wt%)	Hancock Efficiency (%)
MEIG No. 9 Seam											
Raw Coal (Phase I)	---	20.55	6.05	3.13	11315	100.0	100.0	0.0	0.0	0.0	0.0
Precleaned Coal (Phase I)	1.75	12.65	5.21	2.14	12323	84.8	93.4	47.8	27.0	42.0	35.4
Micromag 3" Cyclone (28M Grind)	1.60	9.13	4.51	1.27	13020	69.7	80.5	69.0	47.6	71.8	52.3
2" Krebs MCL/PERC (100M Grind)	1.60	7.64	4.13	0.75	13140	71.5	84.0	73.4	51.2	82.9	66.9
A-L Cent. Ca(NO3)2 (100M Grind)	1.60	7.24	4.08	0.69	13207	73.2	86.4	74.2	50.7	83.9	70.3
Illinois No. 6 Seam											
Raw Coal (Phase I)	---	21.51	4.51	2.24	11197	100.0	100.0	0.0	0.0	0.0	0.0
Precleaned Coal (Phase I)	1.65	10.45	3.58	1.44	12393	77.0	86.5	62.6	38.8	50.4	36.9
Micromag 3" Cyclone (28M Grind)	1.70	7.04	2.97	0.68	13002	65.9	77.6	78.8	56.5	79.4	57.0
2" Krebs MCL/PERC (100M Grind)	1.70	6.56	2.73	0.49	13022	70.7	83.5	78.4	57.2	84.5	68.0
A-L Cent. Ca(NO3)2 (100M Grind)	1.70	6.20	2.69	0.44	13079	71.1	84.3	79.5	57.5	86.0	70.3
Upper Freeport Seam											
Raw Coal (Phase I)	---	36.99	3.62	3.08	9423	100.0	100.0	0.0	0.0	0.0	0.0
Precleaned Coal (Phase I)	2.20	23.34	2.77	2.21	11619	74.7	92.7	52.9	42.9	46.3	39.0
Micromag 3" Cyclone (28M Grind)	1.70	7.42	1.23	0.54	14382	46.7	71.7	90.7	85.4	92.7	66.5
2" Krebs MCL/PERC (100M Grind)	1.70	9.50	1.20	0.51	14017	58.3	87.3	85.0	80.7	90.3	77.6
A-L Cent. Ca(NO3)2 (100M Grind)	1.70	7.62	1.06	0.36	14337	57.6	88.2	88.1	83.2	93.3	81.5
Pittsburgh No.8 Seam											
Raw Coal (Phase I)	---	33.60	4.91	3.18	9552	100.0	100.0	0.0	0.0	0.0	0.0
Precleaned Coal (Phase I)	1.75	10.02	4.78	2.31	13098	67.3	93.5	79.9	34.4	51.1	44.6
Micromag 3" Cyclone (28M Grind)	1.60	6.33	3.97	1.32	13651	55.2	80.3	89.6	55.2	76.9	57.2
2" Krebs MCL/PERC (100M Grind)	1.60	5.30	3.30	0.66	13900	59.3	87.4	90.6	60.1	87.7	75.1
A-L Cent. Ca(NO3)2 (100M Grind)	1.60	5.15	3.34	0.65	13921	60.9	89.9	90.7	58.5	87.5	77.4

NOTE:
- –Precleaned Coal listed is feed to 100M Grnd Washability
- –Hancock Efficiency = Pyritic Sulfur Removal + Energy Recovery – 100
- –Yield is lower for Micromag because 400M x 0 is assumed to be rejected in the Micromag Process

performance and filter cake washing effectiveness to enable them to size their equipment for quotation purposes. From these tests the filter cakes were retained and analyzed for residual amounts of calcium nitrate. The two ion species of calcium nitrate, calcium and nitrate, were analyzed separately at the MTU IMP; but it was reported that nitrate tends to be very reactive, making it less detectable with the analytical method utilized. MTU investigators indicated that the calcium results were much more reliable than the nitrate results. Therefore, the amount of calcium was multiplied by the stoichiometric ratio of calcium to calcium nitrate to arrive at the total amount of calcium nitrate. For every unit weight of calcium, one would expect that 3.1 times as much would be attributed to nitrate. Therefore, 4.1 times the amount of calcium was used to represent the amount of calcium nitrate.

Both vendor systems used a form of product stream pre-thickening because of its low solids content, around 5%. Outomec used a tube filter while Eimco used a Shriver thickener. Both the tube and Shriver filters are once-through devices that remove a portion of the mother liquor from a feed stream while yielding a higher concentration of solids in a product slurry.

The amount of calcium nitrate remaining on filter cake is largely a function of the amount of wash water used to rinse it; but, other factors influencing the residual amount of calcium nitrate include the thickness of the filter cake, and the initial amount of medium in the cake. Thin cakes enhance washing effectiveness but reduce filter capacity (more filter area required) and thick cakes, while increasing filter capacity leave a correspondingly higher moisture content on the cake and inhibit cake washing. Filter cake moistures (before washing) ranged between 15% and 28% on all tests. CTC relied on the judgment of the filter vendors in determining the best balance between filter cake thickness, dryness, and capacity. However, the amount of wash water used does have a direct effect on the degree of dilution of the medium and thermal costs associated with evaporating excess water and regenerating the medium.

Cake washing need not take place exclusively on the filter; cake can be premixed with fresh water in a separate vessel prior to filtering. This can be a very effective means to rinse the solids prior to filtering where further washing can occur. When this is done, no mother liquor is obtained from the filter system.

In the comparison of the filter tests, individual tests were not penalized or rewarded for producing a particularly wet or dry cake. All met the project goals of less than 30% moisture. Examined were the amount of wash water used and the residual calcium nitrate left on the cake, the primary determinants of the process operating costs. The amount of fresh water used and the amount of dry coal filtered were reported by each vendor and CTC converted it to a "wash rate." Wash rate is defined as the weight of fresh water with respect to the amount of dry solids. The residual amount of medium remaining on the cakes in relation to wash rate is listed in Tables 3 and 4 for Outomec and Eimco, respectively. An "unwashed product" is not listed for Outomec because the upstream tube filter initiates washing by introducing fresh water to discharge its cake as a thickened slurry. In the Outomec case it was assumed that calcium nitrate on the product was identical to that on the unwashed refuse, which is not subjected to tube filtration. In the Eimco case, Shriver thickener product was filtered again on their normal filter material to obtain a discrete product for determining calcium content.

In order to compare the cost tradeoffs between calcium nitrate replacement and water evaporation costs, it was assumed that an uncoated grade of calcium nitrate would be used, which costs \$250/ton at 79% purity. Water removal costs were based on a multiple effect evaporator requiring 184.84 Btu/pound of water and a thermal energy cost of \$3.00/million Btu. Using the data in Tables 3 and 4, and the cost assumptions stated above for medium replacement and heating costs, Figures 5 and 6 were developed. Each shows the summation of costs to replace lost calcium nitrate and thermal energy to evaporate excess water for a given wash ratio in a plant of 200 tph capacity. A wash water ratio of 0.0 in Figures 5 and 6 represents no washing or unrinsed material. No washing is one of the more expensive options and is likely to add an appreciable amount of ash to the product. As wash water is added, more calcium nitrate is recovered than that which would be expected from filtering alone but at the cost of diluting the medium. Generally, wash ratios in the 1.0 to 2.0 range provide the least costly operating condition, minimizing the sum of calcium nitrate replacement and thermal energy costs. At higher wash ratios calcium nitrate replacement costs tend to be lower and thermal costs higher.

<u>Organic Circuit</u>

The organic circuit tests consisted of a filtering step followed by a hot water flash simulation. The organic medium is first pre-filtered for primary recovery of the mother liquor to approximately 50 to 55% solids. The two streams, clean coal and refuse, report to separate hot water flash baths as recommended by Dow Chemical Company at 205°F and 20% solids. The hot water flashes the organic medium off of the solids to a plenum where vapors are collected and condensed. Water and the organic medium form an azeotrope with the approximate properties of a diluted organic.

Some data are available from the Inner Coal Company, which utilized a perc and hot water flash process in recovering

TABLE 3

CLEAN COAL CIRCUIT COSTS - OUTOMEC
200 TPH BASIS: 140 TPH CLEAN COAL PRODUCT, 60 TPH REJECT

Sample	Wash Rate[1]	Remaining on Cake After Wash		$Ca(NO_3)_2$ Loss TPH	$Ca(NO_3)_2$ Cost $/HR	Evap[3] Cost $/HR	Total Cost $/HR
		Wt% Sol Calcuim	Wt%[2] $Ca(NO_3)_2$				
No $Ca(NO_3)_2$ Exposure							
Clean Coal	-	0.057	-	-	-	-	-
Refuse	-	0.400	-	-	-	-	-
Outomec Cakes							
Unwashed Product[4]	0.00	-	14.096	19.73	6245	0	6245
7 Washed Product	1.24	0.748	2.833	3.97	1255	193	1448
8 Washed Product	2.07	0.174	0.480	0.67	213	321	534
9 Washed Product	2.13	0.177	0.492	0.69	218	331	549
10 Washed Product	2.29	0.205	0.607	0.85	269	356	624
11 Washed Product	2.80	0.369	1.279	1.79	567	435	1001
17 Unwashed Refuse	0.00	3.838	14.096	8.46	2676	0	2676
22 Washed Refuse	0.36	3.024	10.758	6.46	2043	24	2067
23 Washed Refuse	0.42	3.143	11.246	6.75	2135	28	2163
24 Washed Refuse	1.41	0.881	1.972	1.18	374	94	468
25 Washed Refuse	1.37	1.312	3.739	2.24	710	91	801
26 Washed Refuse	1.32	1.296	3.674	2.20	698	88	785

The least cost operating point based on the indicated data is test 8 Product and test 24 Refuse for a cumulative cost of $1002/hr or $5.01 per feed ton.

Notes:

(1) Wash rate is based on weight of clean fresh water with respect to bone dry solids.

(2) Both calcium and nitrate analyses were performed on the filter cake samples. The investigators at Michigan Technological University (MTU) indicated that the nitrate analysis is not as reliable as the calcium analysis due to its reactive nature and tendency to bind with other chemical species. One would expect a 3.1 ratio to exist between that of nitrate and calcium given the molecular formula for calcium nitrate. It was decided to use the calcium percentages only, and multiply by a factor of 4.1 (the elemental ratio between calcium and a molecule of calcium nitrate) to obtain a total calcium nitrate result. Coal has naturally occurring calcium as indicated for the non-exposed samples of clean and refuse material (0.057 and 0.400 respectively). These values were subtracted from those of the exposed samples before calculating the calcium nitrate percentages. Medium loss costs were based on salt at $0.125/lb and 79% purity, or $250/ton. Resulting $CA(NO_3)_2$ cost is $0.1582/lb.

(3) 184.84 BTU per pound of water and $3.00/million BTU were assumed. The evaporator is seven-effect providing a steam economy of 5.41.

(4) An unwashed product was not available. It was assumed that calcium nitrate percentage was the same as in the unwashed refuse.

TABLE 4

CLEAN COAL CIRCUIT COSTS - EIMCO
200 TPH BASIS: 140 TPH CLEAN COAL PRODUCT, 60 TPH REJECT

Sample	Wash Rate[1]	Remaining on Cake After Wash		$Ca(NO_3)_2$ Loss TPH	$Ca(NO_3)_2$ Cost $/HR	Evap[3] Cost $/HR	Total Cost $/HR
		Wt% Sol Calcuim	Wt%[2] $Ca(NO_3)_2$				
No $Ca(NO_3)_2$ Exposure							
Clean Coal	-	0.057	-	-	-	-	-
Refuse	-	0.400	-	-	-	-	-
Eimco Cakes							
Unwashed Product	0.00	5.620	22.808	31.93	10105	0	10105
1 Washed Product	0.67	0.981	3.788	5.30	1678	104	1782
2 Washed Product	1.33	0.685	2.575	3.60	1141	207	1347
3 Washed Product	2.00	0.619	2.304	3.23	1021	311	1331
4 Washed Product	3.33	0.412	1.456	2.04	645	517	1162
5 Washed Product	6.67	0.365	1.263	1.77	559	1036	1595
Unwashed Refuse	0.00	5.230	19.803	11.88	3760	0	3760
1 Washed Refuse	1.00	0.903	2.062	1.24	392	67	458
2 Washed Refuse	2.00	0.621	0.906	0.54	172	133	305
3 Washed Refuse	4.00	0.520	0.492	0.30	93	266	360
4 Washed Refuse	5.67	0.467	0.275	0.16	52	377	429
5 Washed Refuse	10.00	0.458	0.238	0.14	45	665	711

The least cost operating point based on the indicated data is test 4 Product and test 2 Refuse for a cumulative cost of $1467/hr or $7.34 per feed ton.

Notes:

(1) Wash rate is based on weight of clean fresh water with respect to bone dry solids.

(2) Both calcium and nitrate analyses were performed on the filter cake samples. The investigators at Michigan Technological University (MTU) indicated that the nitrate analysis is not as reliable as the calcium analysis due to its reactive nature and tendency to bind with other chemical species. One would expect a 3.1 ratio to exist between that of nitrate and calcium given the molecular formula for calcium nitrate. It was decided to use the calcium percentages only, and multiply by a factor of 4.1 (the elemental ratio between calcium and a molecule of calcium nitrate) to obtain a total calcium nitrate result. Coal has naturally occurring calcium as indicated for the non-exposed samples of clean and refuse material (0.057 and 0.400 respectively). These values were subtracted from those of the exposed samples before calculating the calcium nitrate percentages. Medium loss costs were based on salt at $0.125/lb and 79% purity, or $250/ton. Resulting $CA(NO_3)_2$ cost is $0.1582/lb.

(3) 184.84 BTU per pound of water and $3.00/million BTU were assumed. The evaporator is seven-effect providing a steam economy of 5.41.

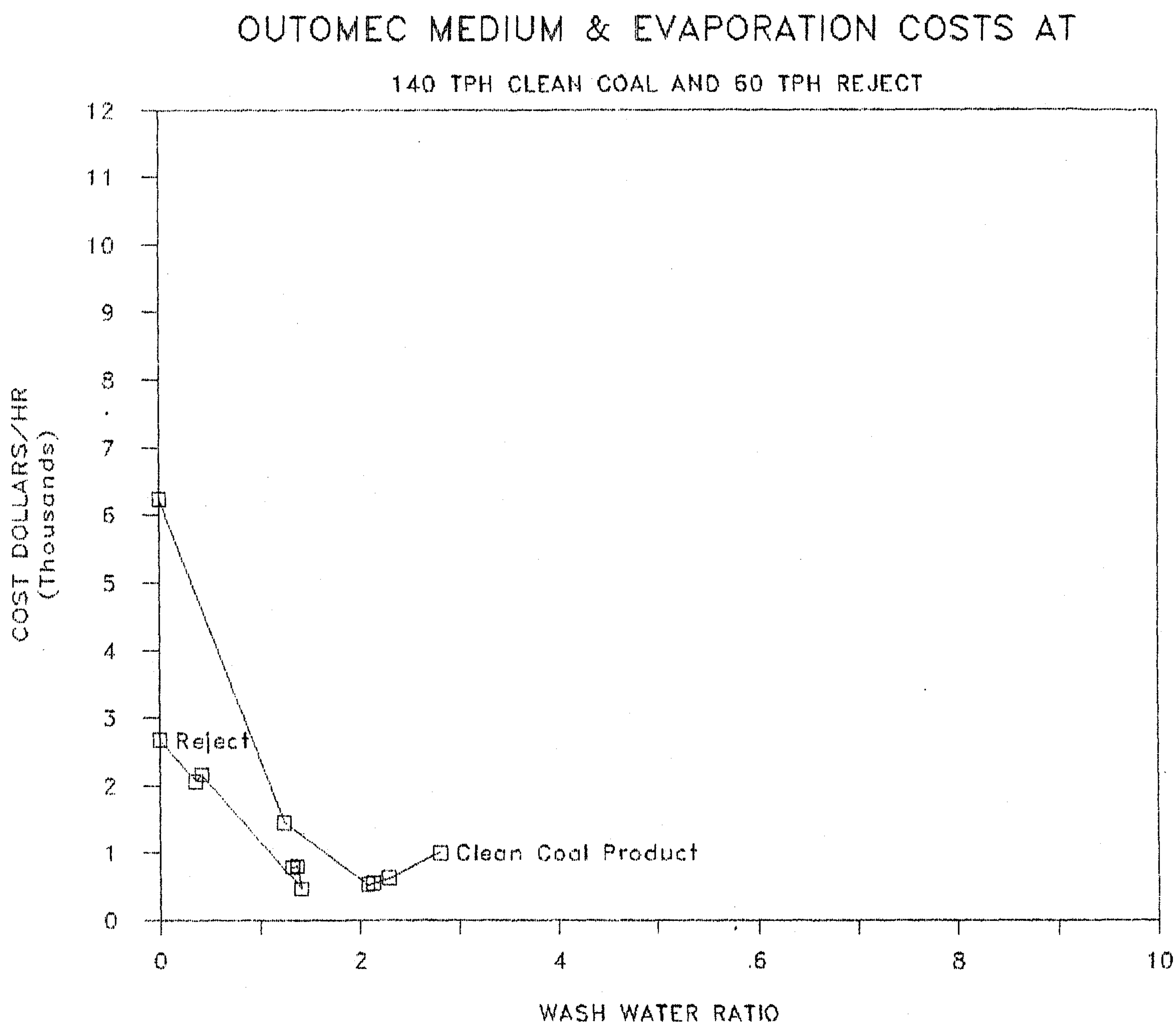
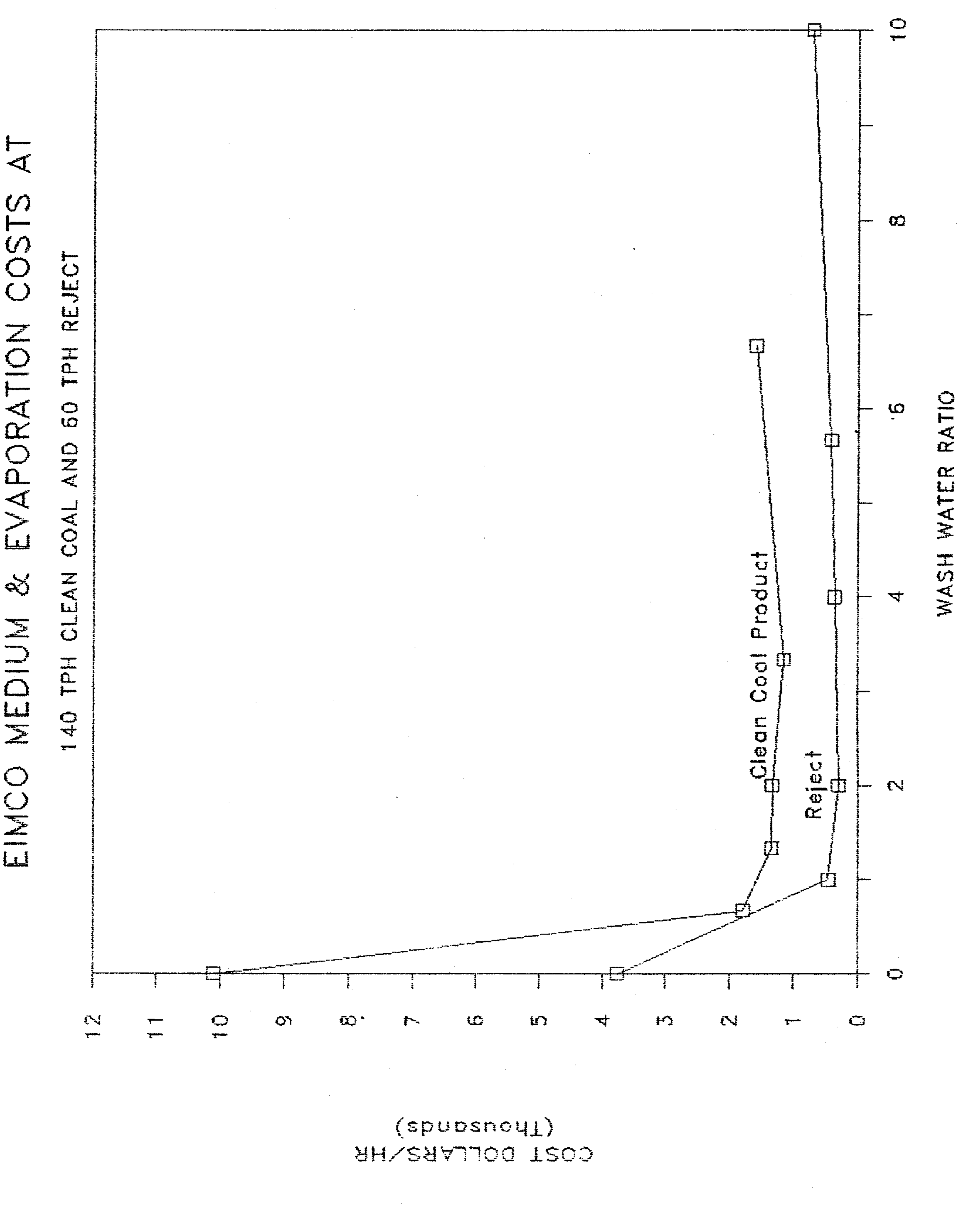

OUTOMEC MEDIUM & EVAPORATION COSTS AT
140 TPH CLEAN COAL AND 60 TPH REJECT
COST DOLLARS/HR
(Thousands)
12
11
10
9
8
7
6
5
4
3
2
1
0
Reject
Clean Coal Product
0
2
4
.6
8
10
WASH WATER RATIO

FIGURE 5



EIMCO MEDIUM & EVAPORATION COSTS AT
140 TPH CLEAN COAL AND 60 TPH REJECT
Clean Coal Product
Reject
COST DOLLARS/HR
(Thousands)
WASH WATER RATIO
0
2
4
6
8
10
12
11
9
7
5
3
1

FIGURE 6

anthracite from culm in Eastern Pennsylvania during the 1980s. The hot water flash bath portion of the circuit was operated as a joint venture with Dow Chemical Company, who provided the system design. Operational data from Inner Coal Company indicate that losses on the coal from a hot water flash were fairly low. A layer of water was maintained above the organic liquid to help control evaporative losses. Even so, vapor losses due to system leakage were estimated by the operators to have been about 1/4 pound per ton of feed. Methylene chloride's vapor pressure exceeds that of perchloroethylene.

Data concerning solvent-on-coal losses, specifically for the organic medium under consideration, have been measured by Dow and reported in Table 21 of the Preliminary Separating Media Evaluation Report (PSMER). Each of the four project test coals was exposed to organic solvent at 1.35 sg, filtered, treated in a hot water flash bath, filtered again, dried, and analyzed to determine the amount of residual organic medium. Solvent losses appear to be coal specific. For methylene chloride, values of 146, 37, 103 and 38 ppm were reported respectively for the Meigs, Illinois, Upper Freeport, and Pittsburgh seam coals. The average is 81 ppm and standard deviation is 53.2. Similarly for perchloroethylene, values of 769, 1146, 288, and 119 ppm were reported for the four test coals. The perchloroethylene average is 581 ppm and standard deviation 466.9.

4.0 COSTS OF CIRCUITS

Comparison of Aqueous and Organic Circuits

A cost comparison of two hypothetical coal processing plants is presented in this section. Two 200 tph, 7,500 hour per year, 10 year life plants are compared. Cost quotes for essential equipment were obtained from vendors and are tabulated in Table 5. Operating and maintenance costs for both separating/filtering scenarios were also provided by vendors. Energy costs are calculated for the aqueous system evaporator and the organic hot water flash baths. Common circuit component costs, such as pumping and material handling, are ignored. Media replacement costs from the previous section are used.

Power costs are calculated as a function of motor horsepower and summarized as operating costs. The organic circuit carries capital costs for double filtering (medium and water), hot water flash, vapor containment, and secondary recovery systems. The aqueous circuit carries capital and operating costs for water evaporation during regeneration of the medium when water is used to rinse the filter cakes. The aqueous circuit also requires a higher cost separator, i.e., a centrifuge as opposed to a cyclone. Operating personnel and control of the operation are

Table 5
Selected Component Costs[1]
200 TPH Plant, 75,000 Operating Hours

Calcium Nitrate Circuit $ in 000's

Equipment	Make/Model	Qty	Capital $	Maintenance $	Operation $	Medium[2] $
Centrifuge	Sharples PM 95000	1	850	64	1,846	--
Clean Coal Tube Filter	Goretex 900 Sq Ft	5	750	950	1,757	--
Clean Coal Filter	Outomec Ceramic 1155 Sq Ft	5	5,500	2,325	1,547	16,128
Refuse Filter	EIMCO Extractor 1130 Sq Ft	5	4,950	5,300	3,847	13,046
Evaporator/ Condenser	Swenson 572,000 #/Hr Evaporative Capacity	1	7,000	5,500	33,000	--
Subtotal			19,050	14,139	41,997	29,174

Total: $104,360

MCL/PERC Circuit $ in 000's

Equipment	Make/Model	Qty	Capital $	Maintenance $	Operation $	Medium[2] $
Cyclone	Krebs 10"	10	225	563	3,180	--
Clean Coal Tube Filter	Goretex 1000 Sq Ft	43	6,880	7,870	1,493	--
Refuse Tube Filter	Goretex 1000 Sq Ft	14	2,240	2,562	263	--
Clean Coal Hot Water Bath	Dow-Swenson 20,000 Gal	3	1,846	1,458	16,696	--
Refuse Hot Water Bath	Dow-Swenson 10,000 Gal	3	1,034	817	7,155	--
Clean Coal Filter	Outomec Ceramic 1155 Sq Ft	8	8,800	3,720	2,475	--
Refuse Filter	Outomec Ceramic 1155 Sq Ft	5	5,500	2,325	1,548	--
Vapor Containment	Durr Industries Regenerative Thermal Oxidizer 58,300 SCFM	1	1,700	1,228	6,095	6,900
Subtotal			28,225	20,543	38,905	6,900

Total: $94,573

$\Delta\$ = 104,360 - 94,573 = \$9,787$

$\Delta\$/Ton^1$, Calcium Nitrate vs. MCL/PERC = $9,787 \div 15,000 = \$0.65/Ton$

Table 5 (Continued)

Notes:

(1) Components which are considered relatively comparable between the two circuits (labor, materials handling, etc.) are ignored in this analysis. Comparison is of dissimilar components only and is intended to indicate an order of magnitude difference in cost/feed ton processed by the two circuits. This comparison does not reflect total cost. It is assumed equipment components common to both circuits generate common costs and are not considered. Cost of money is also not considered.

(2) Medium consumption calculation based on 70% plant yield.

November 30, 1994

not considered; the main differences are taken to be in the separators, media recovery schemes and attendant media losses.

5.0 PROPERTIES OF CLEAN COAL AND REFUSE

This section discusses the handling characteristics of the respective plant products. Handling properties are a function of the medium, solids content, moisture content and size consist. Based on experiments conducted in Phases I and II, the consistency of the product and refuse will be quite wet and sticky. A 30% maximum moisture is allowable on the material products though it is doubtful that the refuse will be easily handleable at that moisture. Clean coal when dry would likely form a hard cake. This is a characteristic of wetted fine coal no matter what the medium. Refuse may be difficult to handle, particularly after it is allowed to set. Care must be taken in the selection of weighing systems for this material.

Material contacted by the calcium nitrate medium has a soapy feel. Organic medium would provide a "drier" feel; but, in the Project Team's experience, the only handling difference between the organic and aqueous media is that wetting of solids with calcium nitrate solution is slightly more difficult. Agitation, mixing, or wet grinding in the aqueous medium will negate this difference; it is not a cause for concern. No handling, foaming, or pumping difficulties were experienced with either medium. Pumpability is a function of the percent solids and is not so dependent on the type of media; coal slurry is generally pumpable at a low percent solids. However, medium consisting of calcium nitrate could improve slurry rheology over that resulting from ordinary water by keeping solids in mechanical suspension. Once processed and the medium removed, the clean coal and refuse product characteristics from either process are identical. Both cakes tend to form agglomerated hard pack cakes when dry, but this would be expected from most any process with coal in this size range.

This project requires intermittent steps to gather weight, levels, flowrate, etc. that actually interfere with the handling of the material in the plant by interrupting the flow of material for weighing and sample collection. Handling would not be so difficult in a commercial facility because such data acquisition would not be required.

In order to obtain the clean coal yield and perform a material balance at pilot scale, it is necessary that a cumulative weight of clean coal and refuse be obtained periodically. Weighing of the plant's products on weigh belts is one method of determining yield; bin weighing may be more accurate. The main concern with bins is that material will sit for an extended period of time before the weight is recorded and the material discharged. For example, consider an 8-hour run at 1,000 lb/hr. Provided the product bins have the capacity and are not discharged during a run, a total of 4 tons of material will be in the clean coal and refuse bins at the conclusion of the run. This material will have set in the bin for an extended period of time and discharge may be difficult due to material compaction, drying, and adhesion to the bin surfaces. Lumps would congeal and harden upon drying; no data on the angle of repose were obtained.

6.0 HEALTH AND SAFETY CONCERNS FOR MEDIA

The safety, health, and environmental aspects of each medium are of major concern. Organic solvents are precursors of smog. Both perchloroethylene and methylene chloride are considered toxic and are suspected to be carcinogenic. Both are coming under increased scrutiny and control. Environmental regulations from cities, states, and the Federal government can be expected to proliferate. Calcium nitrate is classified as an oxidizer and care should be taken in its handling; protection from combustible dust is required. However, calcium nitrate is not as threatening to the worker or environment as are organic solvents.

The main governing bodies that have jurisdiction over materials in transit, use, and disposal are: DOT, OSHA, and EPA, respectively. Other Federal statutes are also applicable.

OSHA limits the exposure of workers to perchloroethylene at 25 ppm and to methylene chloride at 500 ppm on a time weighted average. The methylene chloride limit is under review and may be dropped to 25 ppm, pending appeal. More detailed information concerning the current worker exposure limits is shown in Table 6, Summary of Responses and Limits for Inhalation of Chlorinated Solvents.

The Resource Conservation and Recovery Act (RCRA) regulates all hazardous waste and treats material contacted with organic solvents as hazardous waste. A spill of more than 100 pounds of any chlorinated solvent requires the notification of the Coast Guard and the National Response Center. Material Safety Data Sheets and the listing from Dangerous Properties of Industrial Materials are included in Appendix A.

The Superfund Amendments and Reauthorization Act (SARA) Title III outline requirements for providing chemical hazard information, inventory data, and emission data to local, state and Federal emergency planning and response groups. SARA requires industrial plants across the country to report inventories of chemicals used on-site and to estimate annual releases of these chemicals into the environment. It also contains the Emergency Planning and Community Right to Know Act. The handler of regulated chemicals must have plans in place to deal with the eventuality of a spill

Table 6[10]

SUMMARY OF RESPONSES AND LIMITS FOR INHALATION OF CHLORINATED SOLVENTS (PPM)[1,2]

	Solvent Odor[3]		
	Odor Threshold	Slight, Not Unpleasant	Strong, Unpleasant
Perchloroethylene	5-70	100-200	> 280
Trichloroethylene	20-50	100-400	>1000
1,1,1-Trichloroethane	100	350-500	> 900
Methylene Chloride	150-600	250-1000	>1000

	Vapor Inhalation Subjective Response[3]			
	None	Eye Irritation	Nose, Throat Irritation	Slight Anesthetic Effects (lightheaded, dizzy, etc.)
Perchloroethylene	<75	75-200	100, transient 600, severe	100, 7 hrs 200, >20 min 600, 10 min
Trichloroethylene	100, 7 hrs	200, slight transient	200	300-400, 80 min 1000, ≥28 min
1,1,1-Trichloroethane	<500	500, transient 900-1000, mild	2000	900-1000, ≥20 min 2000, ≥5 min
Methylene Chloride	<500	500, transient	500, transient	850-1150, ≥20 min 2300, ≥5 min

	OSHA Permissible Exposure Limits[4]	
	Time-Weighted Average[5]	Short-Term Exposure Limit[5]
Perchloroethylene	25	—
Trichloroethylene	50	200
1,1,1-Trichloroethane	350	450
Methylene Chloride[6]	500	1000[7] 2000, 5 min (peak) in any 2 hrs

	ACGIH TLVs[8]	
	Time-Weighted Average (TWA)	Short-Term Exposure Limit (STEL)
Perchloroethylene	50	200
Trichloroethylene	50	200
1,1,1-Trichloroethane	350	450
Methylene Chloride	50[9]	—

[1]All values in the table are in parts per million by volume in air.

[2]For further information on a specific solvent and its effects, consult the appropriate Material Safety Data sheet from The Dow Chemical Company.

[3]Values based on published literature and Dow unpublished data.

[4]The 8-Hour Time-Weighted Averages and Short-Term Exposure Limits for perchloroethylene, trichloroethylene, and 1,1,1-trichloroethane were effective September 1, 1989 (Federal Register, Vol. 54, No. 12, January 19, 1989) using any form of controls. During the time period between September 1, 1989 and December 31, 1992, the previous limits (TWA, ceiling, peak) continue to apply.

[5]The 8-Hour Time-Weighted Average (TWA) is an employee's permissible average exposure in any 8-hour shift of a 40-hour work week. The Short-Term Exposure Limit (STEL) is an employee's 15-minute time-weighted average exposure which shall not be exceeded at any time during a work day.

[6]OSHA has issued advanced notice of proposed rulemaking to revise the permissible exposure limit for methylene chloride (Federal Register, Vol. 51, No. 226, November 24, 1986, pp. 42257-42267). Consult current Material Safety Data Sheet.

[7]For methylene chloride, the acceptable ceiling concentration is 1000 ppm and the maximum acceptable peak is 2000 ppm (5 min in any 2 hrs).

[8]TLV-TWA is an employee's permissible average exposure for any 8-hour work day and 40-hour work week as established by the American Conference of Governmental Industrial Hygienists (ACGIH). TLV-STEL is a 15-minute time-weighted average exposure which should not be exceeded at any time during a work day (ACGIH Threshold Limit Values and Biological Exposure Indices for 1989-90).

[9]ACGIH also placed methylene chloride on the list of suspect carcinogens (A2), and stated that "where a TLV has been assigned . . . if exposures are controlled to this level, we would not expect to see a measurable increase in cancer incidence or mortality." These changes were adopted in May 1988.

[10]Specialty Chlorinated Solvents Product Stewardship Manual, 1991, Courtesy, The Dow Chemical Company, 2020 DOW Center, Midland, Michigan 48674

so that local authorities can know what they may be dealing with in such an event. Methylene chloride and perchloroethylene are Section 313 chemicals and subject to most reporting requirements. Calcium nitrate is not listed, but ammonia (its contaminant) is. Federal SARA regulations become applicable at a level of 10,000 lbs transferred or on site per year each for perchloroethylene and methylene chloride.

The 1990 Clean Air Act classifies mcl and perc as hazardous air pollutants. The local Air Pollution Control Board (APCB) was contacted with respect to the allowable limits of pollutant release to the atmosphere. Perchloroethylene and methylene chloride are classified as air toxins and have limits listed below:

Perchloroethylene	22.8 lb/hr	no more than 49.2 tons/yr
Methylene chloride	11.5 lb/hr	no more than 25 tons/yr

Though perchloroethylene and methylene chloride are technically volatile organic carbons the APCB does not classify them as such. The limit on the total amount of VOCs (no matter their species) released to the atmosphere is 25 tons/yr. These limits would weigh against plant operations in some locations on a commercial basis.

Ammonia also has a limit of 0.8 lb/hr and no more than 2.5 tons/yr. Ammonia will evolve upon boiling the dilute aqueous solutions for medium regeneration; however, it is not expected to evaporate and pass to the atmosphere as the organic mixtures would.

Dow's Specialty Chlorinated Solvents Product Stewardship Manual indicates that when disposing of any quantity of chlorinated solvent, the preferred option is to send waste via a permitted waste hauler to a licensed reclaimer or permitted incinerator. The Toxicity Characteristic Leaching Procedure (TCLP) determines the fitness of material for landfilling. The limit for organic liquids is 0.7 ppm. CTC requested a local analytical laboratory accustomed to characterizing soil samples contaminated by underground storage tanks to perform a leaching test on a coal refuse sample. The refuse sample had been separated in the organic medium during Phase II and subjected to a hot water flash process. The leachate contained 0.001 ppm perchloroethylene indicating the effectiveness of the hot water flash in disengaging the chlorinated solvent from the coal. Presumably this material would be suitable for landfilling unless there is something generally non-permissible about coal refuse. Methylene chloride is not measured in the TCLP leaching tests because it is considered so volatile that it would evaporate to the atmosphere before leaching into the ground water.

7.0 CHEMICAL AND PHYSICAL PROPERTIES OF MEDIA

These data are presented to facilitate and enhance the reader's knowledge of the candidate media. The characteristics, properties, and form of the two media are discussed. These characteristics determine appropriate handling methods. The industrial grade calcium nitrate is a white granular solid containing less than 2% ammonia and melts at 105°F. Methylene chloride and perchloroethylene are liquids at room temperatures. The vapor pressure of methylene chloride and perchloroethylene is 240 mm and 18 mm of Hg, respectively, at room temperature. More detailed information concerning the organic solvents is shown in Table 7, Physical Properties of Chlorinated Solvents. Surface tension of aqueous calcium nitrate solutions is unknown; however, the viscosity has been measured during this study. At 20°C the viscosity of a 1.35 sg aqueous solution is 3.4 cp. The measured viscosity of the organic liquid mixture at room temperature is less than 0.5 cp.

Physical Characteristics of Candidate Media

	Methylene Chloride	Perchloroethylene	Calcium nitrate
Boiling Point, °C (°F)	40.1 (104)	121 (250)	NA
Melting Point, °C (°F)	NA	-22.3	42 (108)
Specific Gravity	1.32	1.6	1.82
Appearance	Clear Colorless Liquid	Clear Colorless Liquid	White Crystals
Solubility in Water (grams/100g water)	1.32	0.015	121

Temperature dependence of the physical properties of the media is important since some temperature variation is expected during the operation of the plant. The tendency is toward elevated temperatures because of the expected media recovery methods. Freezing temperatures for either medium are not anticipated. The expected temperature range during storage of calcium nitrate chemical allow it to remain a solid; if in solution, at elevated temperatures, water and ammonia would boil off.

Excess water in the aqueous regeneration scheme is created by rinsing the filter cakes with fresh water. Medium recovery will entail boiling dilute solutions in order to remove this excess water and regenerate the original medium specific gravity. Water boils at approximately 212°F, though salt tends to elevate this somewhat. Experiments by an evaporator supplier have indicated a boiling point elevation of about 8°F (to 220°F). Swenson's report on their experiments is included in Appendix B.

Table 7*

PHYSICAL PROPERTIES OF CHLORINATED SOLVENTS.

PROPERTIES	SOLVENTS: METHYLENE CHLORIDE	INHIBITED 1,1,1-TRICHLOROETHANE	TRICHLORO-ETHYLENE	PERCHLORO-ETHYLENE
Chemical Formula	CH_2Cl_2	$C_2H_3Cl_3$	C_2HCl_3	C_2Cl_4
Molecular Weight	84.9	133.4	131.4	165.8
Boiling Pt. @ 760 mm Hg	103.5°F (39.7°C)	165°F (74°C)	189°F (87°C)	250°F (121.1°C)
Freezing Point	-139°F (-95°C)	-34°F (-37°C)	-124°F (-86.7°C)	-9°F (-22.8°C)
Specific Gravity @ 25/25°C	1.32	1.32	1.456	1.619
Pounds per Gallon @ 25°C	10.98	10.97	12.11	13.47
Vapor Density (air = 1.00)	2.93	4.60	4.53	5.76
Specific Heat @ 25°C cal/g°C	0.283	0.259	0.226	0.209
Heat of Vaporization @ Boiling Point				
cal/g	78.9	56.7	56.4	50.1
BTU/lb	142	102	101.6	90.2
Refractive Index @ 25°C	1.421	1.434	1.474	1.503
Viscosity @ 25°C centipoises	0.41	0.79	0.54	0.84
Flash Point Tag Open Cup ASTM, Method D-1310	none	none	none	none
Tag Closed Cup ASTM, Method D-56	none	none	none	none
Solubility (g/100g) @ 25°C				
H_2O in solvent	0.17	0.05	0.04	0.0105
solvent in H_2O	1.70	0.07	0.10	0.015
Surface Tension (dynes/cm @ 25°C)	27.1	25.1	28.7	31.8
Kauri Butanol Value	136	124	129	90
Solvent-Water Azeotropic Boiling Point	100.6°F (38.1°C)	149°F (65°C)	164°F (73.3°C)	190°F (87.8°C)
Flammable Limits (volume % of solvent in air) @ 25°C				
Lower Limit	14	7.5	8.0	none
Upper Limit	22	12.5	9.2 (saturation)	none

*Specialty Chlorinated Solvents Product Stewardship Manual, 1991, Courtesy, The Dow Chemical Company, 2020 DOW Center, Midland, Michigan 48674

In the laboratory experiments, water and some of the ammonia were removed (boiled) from the dilute solution in the process of regenerating and concentrating the working media. This caused the pH of the solution to drop. The ammonia behaves as a pH modifier, which, when removed from the mother solution, causes the pH of the remaining portion to drop into the acidic range. Actually very little of the ammonia is boiled off (about 4%), but this is enough to significantly lower the pH. Special acid-resistant-lined tanks, pipes, and process equipment would be cost prohibitive. A report by Dr. Jim Hwang who assisted CTC in studying this phenomenon is included as Appendix C. Dr. Hwang suggests treating the calcium nitrate solution with lime. In a 1,000 lb/hr pilot plant facility, without condensing the evaporative emissions, (i.e., venting the steam to atmosphere), ammonia releases are estimated to be 0.01 lb/hr. Ammonia emissions below 0.8 lb/hr are exempt. At commercial scale, the evaporated water would be condensed and recycled. By recycling condensate, water makeup would be minimized, there would be little or no ammonia emissions to the air, and the elevated temperature of the returning water could improve the efficiency of the filter wash.

The organic recovery operation consists of filtering followed by hot water flash. The hot water bath is used to vaporize the organic liquids remaining on the coal after an initial filtering to recover as much neat organic liquid as possible. Organic vapors from the hot water bath are recovered, condensed, and recycled to the process. Hot water flash would elevate the circulating medium temperature since the condensed portion of the returning medium would be hot from flashing off. Even though perc boils at 250°F, a water/perc eutectic forms which boils near 200°F. The hot water bath would therefore be maintained around 205°F. Since mcl boils at 104°F, the condenser would have to be maintained below this temperature.

The hot water flash method of disengaging the organic from the clean coal and refuse is the most thorough method of recovering chlorinated solvents and provides a cost saving compared to straight thermal drying in spite of the double filtering required. Immersion in hot water does a better job at volatilizing and disengaging the non-coal organic phase. This is supported by data from Dow Chemical Company. Hot water flash also presents a way to pre-leach the products, effectively removing water soluble components from the solids. In the event of outdoor storage and exposure to the weather, rain water would otherwise leach the solids and the runoff could contain organic material.

It is likely that in either the aqueous or organic circuit, circulating media would be at an elevated temperature. This is because both processes need thermal treatment steps to recover or otherwise regenerate the media. From a safety standpoint caution around scalding liquids would be necessary. Care around hot process pipes would also be in order.

8.0 FINAL RECOMMENDATION FOR PHASE III - MEDIA AND SEPARATOR

Organic media were considered throughout Phases I and II because of considerable prior work, whereas the success and capability of any other media (aqueous included) were less comprehensively documented. However, based on the understanding developed in Phases I and II, the Project Team recommends proceeding to the next phase of the project using calcium nitrate medium.

The organic circuit represents a significant risk to the worker and environment, while calcium nitrate does not. While the economic benefits of the organic circuit are significant, a single large spill could require a massive cleanup effort and bring operation of a plant of this type to a halt. The economic scale tips toward the organic circuit only as long as no large spills or atmospheric emissions occur. A significant rupture in containment would result in the serious involvement of government regulatory agencies, loss of production capability, and the expenditure of funds for environmental cleanup. Large spills are very likely to occur over a 10-20 year operating life. These spills may impact on not only the organic circuit facility but any facility or mine which may be near enough to share the contamination through air, surface water, ground water, etc.

Even a small spill would be significant in terms of response requirements, mitigation measures, containment, fines and reporting. For example, a spill of 100 pounds of perchloroethylene or methylene chloride requires the notification of the Coast Guard and local authorities for hazardous material response and control measures. The National Response Center is to be notified also. Spills of perc that contact the ground require the contaminated earth to be removed and transported to an approved reclamation site. Fines for spills would also be levied.

A commercial scale organic plant would require at the very least what is termed the Best Available Control Technology (BACT) required by the state. Usually this technology is what is commonly used by others in the same industry. The Source Category applicable to a coal washing plant has not yet been determined in accordance with the 1990 Clean Air Act Amendments; however, the chemical process manufacturers and the dry cleaning industries are required to install MACT. Given the time, money and effort needed to comply with environmental laws now in place, and anticipating the implementation of Maximum Achievable Control Technology (MACT), CTC feels that the cost advantages of the organic circuit are outweighed by ES&H considerations.

ES&H problems are not as grave for an aqueous or calcium nitrate circuit. In an aqueous salt circuit, solution (medium) tanks can be left open to the atmosphere without fear of loss of chemical or emission to the environment. A conceptual flowsheet for calcium nitrate is shown in Figure 7. This salt-based, coal cleaning plant would be less complex than an organic circuit and containment costs would also be less. The conceivable benefit of residual calcium on the clean coal product in capturing sulfur in a power plant boiler is unquantified at this writing. While admittedly less economic, it is the intent of the Project Team to explore methods of improving the recovery and regeneration of the aqueous medium as the project proceeds. Operating costs of the aqueous circuit can be reduced with accumulation of operating experience to judge design requirements.

Due to the tendency of the separating gravity cutpoint (d_{50}) to be higher than that of the actual medium specific gravity, plant operators set their medium gravity a measure below that of the desired cutpoint. The difference (offset) between the cutpoint and the actual medium specific gravity increases with decreasing particle size and requires that the actual operating medium gravity be correspondingly lower in order to achieve the same product specifications, i.e., cutpoint throughout the range of particle sizes. Due to the trend by advanced processes toward finer sizes, and in order to effect reasonable cutpoints, very low media gravities may be desirable for fine particle gravimetric processes. While gravities below 1.30 may be required, the organic media cannot obtain a gravity below 1.33 (that of mcl, the lightest component). Naphtha or gasoline could possibly be used to adjust the gravity below 1.33 but has been rejected because of flammability. Magnetite suspensions, too, have difficulty in going to lower gravities and are not used in practice below about 1.25 sg. Below this, magnetite suspensions are said to collapse. On the other hand, salt solutions can achieve low medium gravities (below 1.25) quite easily.

Decanter centrifuges have not previously been utilized for gravimetric separations. CTC believes the high-g decanter is the best available technology to meet the requirements of the Advanced Cyclone Project. This area is rife with opportunity for further investigation and development.

Since the utilization of calcium nitrate as a heavy medium for coal beneficiation would create a new demand, it was important to verify that there would be sufficient supply in the future. Norsk-Hydro, a major manufacturer of calcium nitrate, sells 600,000 metric tons per year of calcium nitrate and has the capacity to manufacture 800,000 metric tons. Norsk-Hydro has excess manufacturing capacity and is seeking additional markets and uses for this product, one of which is to increase the specific gravity of drilling mud. The largest market for calcium

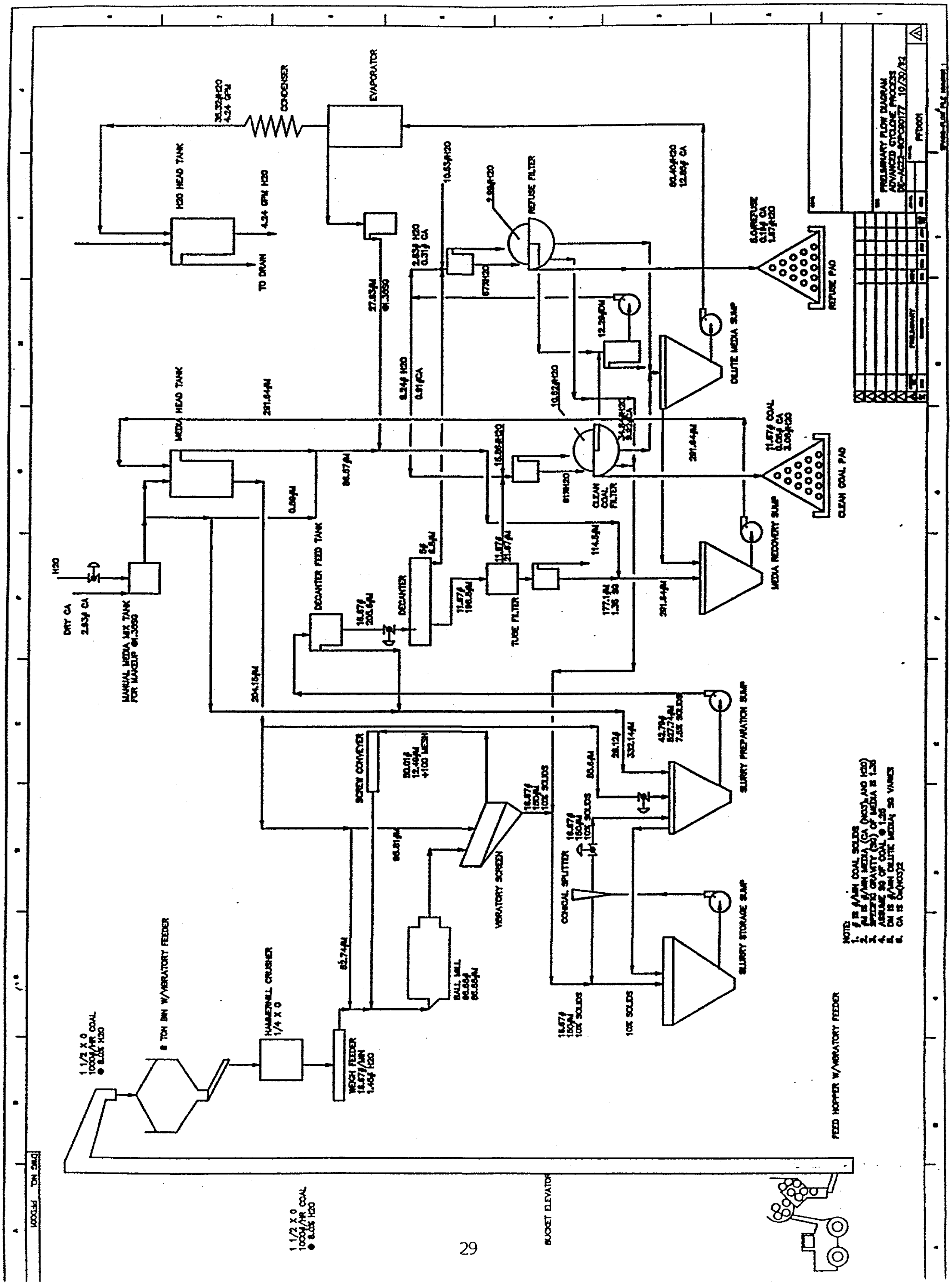

PRELIMINARY FLOW DIAGRAM
ADVANCED CYCLONE PROCESS
EVAPORATOR
CONDENSER
H2O HEAD TANK
TO DRAIN
REFUSE FILTER
REFUSE PAD
DILUTE MEDIA SUMP
MEDIA HEAD TANK
CLEAN COAL FILTER
CLEAN COAL PAD
MEDIA RECOVERY SUMP
DECANTER FEED TANK
DECANTER
TUBE FILTER
MANUAL MEDIA MIX TANK FOR MAKEUP
SCREW CONVEYOR
VIBRATORY SCREEN
SLURRY PREPARATION SUMP
CONICAL SPLITTER
SLURRY STORAGE SUMP
BALL MILL
HAMMERMILL CRUSHER
1/4 X 0
8 TON BIN W/VIBRATORY FEEDER
WEIGH FEEDER
FEED HOPPER W/VIBRATORY FEEDER
BUCKET ELEVATOR

Figure 7

nitrate is fertilizer. Calcium nitrate is a by-product of the manufacturing process used to manufacture other NPK (nitrogen, phosphorous, potassium) fertilizers for which there is a larger demand. CTC has estimated that one 500 tph commercial scale coal cleaning plant would utilize 67,000 metric tons per year of calcium nitrate. Based on these figures, Norsk-Hydro would be able to supply several calcium-nitrate-based coal cleaning plants. The contact at Norsk-Hydro indicated if there were a demand, they could supply it.

9.0 PHASE III TEST MATRIX - PROCESS OPTIMIZATION TEST PLAN (POTP)

The Process Optimization Test Plan will be presented in a separate document. Variables of interest in the calcium nitrate/centrifuge-based process include:

- coal (Illinois, Pittsburgh, Upper Freeport, Meigs)
- coal size
- medium gravity
- percent solids
- g force
- feed rate (separator may handle up to 2-3 tph)
- wash rate on filter cake (not expected to affect separation performance but will influence media recovery, circuit operation, and process economics)

The significance and interrelationships of these and other variables will be systemically investigated in Phase III.

10.0 IGC AND HPM'S ACCOMPLISHMENTS AND CONCLUSIONS

<u>IGC - MODIFIED CYCLONE, DYNAWHIRLPOOL</u>
Magnetically enhanced media (MEM) had advanced to Phase II; however, there was difficulty in developing a suitable separator to use it. Prior to the start of this program, Intermagnetics General Corporation (IGC) had developed and commercialized a laboratory scale separator called the 1000E. The 1000E uses MEM and performs very accurate float and sink separations. It has a capacity of about 100 liters/hr (0.44 gpm) and costs about $100,000. Multiple laboratory-scale separators to obtain the capacity planned for Phase III would be cost prohibitive. Equipment for a 1,000 lb/hr circuit (7.5% solids, 20 gpm) would cost about $4.5 million. The device needed to be scaled up to provide a large throughput at a low price. It was the plan of the Project Team to develop a larger capacity separator (tons/hour) using the MEM.

The developmental approach consisted of housing a Dynawhirlpool or elongated cylindrical-section cyclone within a magnetic field. Diagrams of these two separators appear in Quarterly Report #03.

Placing a cyclonic separator in a permanent magnet and using the MEM was thought to be a very simple and elegant process. Both designs were built and tested. Many experimental runs were completed and the IGC data are attached as Appendix D. From the data it can be seen that different operational parameters and configurations were tried without acceptable separation. The elongated cylindrical section cyclonic separator worked with true heavy liquids but not with MEM. Dr. Kameswara Upadrashta was consulted to analyze the design and data. His notes are also included in Appendix D. The problem is one of matching the magnetic force profile (density) to the centrifugal force in the separator. It was determined that the solution to these problems would require study too complex, costly and time consuming for the project. IGC voluntarily dropped out of consideration but continues to be interested in other gravimetric-type separators using MEM.

HPM - CENTRIFLOAT

As it became evident that IGC would not succeed, an alternative separator candidate (and media) was considered. When IGC dropped out, the Hydro Processing & Mining (HPM) Centrifloat was evaluated instead.

The HPM Centrifloat uses water (with surfactant additive) as medium and represents a hybrid of flotation and cycloning. The Centrifloat technology was tested during Phase II by HPM using one of the project coals, Illinois No. 6. Unfortunately, its performance in rejecting pyritic sulfur was unacceptable. The reader may refer to Quarterly Reports #05 and #06 for details on the device and test results. The Centrifloat medium was the least expensive and most environmentally benign of those under consideration, and it was regretful that it did not perform as well as the others. While project goals were not met by this technology, continued work in this area is recommended.