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MOLTEN-CAUSTIC-LEACHING (GRAVIMELT)
SYSTEM INTEGRATION PROJECT

TOPICAL REPORT FOR TEST CIRCUIT
PROCUREMENT, FABRICATION, INSTALLATION,
SHAKEDOWN AND TEST PLAN FORMULATION

CONTRACT NUMBER DE-AC22-86-PC91257

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ABSTRACT

The objectives of the tasks covered in this document are to design, construct, and shakedown a 20 pounds of coal per hour integrated MCL test circuit to demonstrate the feasibility of the technology for producing a demineralized and desulfurized coal that meets New Source Performance Standards (NSPS). These objectives were met with the construction and shakedown of the integrated test circuit. Although mild kiln conditions (340°C and 2 hours residence time) and a low caustic to coal ratio (1 to 1) were used, the combination of continuous operation and rigorous exclusion of air from the system allowed the production of MCL coal, from high sulfur, high ash coal, which has virtually no carbonate and volatiles loss, which does not form excessively wet cakes in during washing, and which has low alkali retention by the product MCL coal. Equipment performance was generally consistent with design requirements.

1. INTRODUCTION AND SUMMARY

This is a report of the procurement, fabrication, and installation of equipment, shakedown testing, and test plan formulation for an integrated test circuit for the Molten-Caustic-Leaching (MCL or Gravimelt) process for the desulfurization and demineralization of coal. The project is sponsored by the Pittsburgh Energy Technology Center of the U.S. Department of Energy under Contract No. DE-AC22-86-PC91257.

The objectives of this project are to design, construct, shakedown and operate a 20 pounds of coal per hour integrated MCL test circuit to demonstrate the feasibility of the technology for producing a demineralized and desulfurized coal that meets New Source Performance Standards (NSPS), to prove process economics assumptions, to deliver product coal, and to test process conditions aimed at significantly lowering costs. This is to be accomplished while processing two coals representative of the Eastern United States coal production. The coal seams selected were Pittsburgh No. 8 and Kentucky No. 9.

The procurement, installation and shakedown objectives were met by the construction of the integrated test circuit and shakedown testing which produced coal with sulfur content well under the New Source Performance Standards and with equipment performance consistent with commercial design requirements. An assessment of the results indicate that the circuit is ready for operational testing of the MCL process. An operational test plan was formulated that includes the proposed test matrix using the two selected coals at representative process conditions.

1.1 PROCESS DESCRIPTION

The test circuit consists of six unit operations which together provide a continuous system for leaching coal with caustic and regenerating the reactant. These units are a) a kiln for reacting molten caustic with coal, b) a seven stage water washing section for recovering

caustic from the coal, c) a three stage acid washing section for removing the last traces of metals and alkali and providing an ultra-pure coal product, d) a regeneration section to provide purified aqueous caustic, e) an evaporation section to provide molten caustic for recycle to the kiln reactor, and f) a water treatment section to provide either dischargeable or recyclable water.

The integrated test circuit facility that was constructed contains about 150 pieces of equipment including filters, centrifuges, tanks, reactors, feeders, a kiln, and a rising film evaporator. It occupies 3700 square feet and is fitted with more than 6000 feet of piping, 425 valves, 80 instruments and controls as well as a control room with computer control and a data acquisition and reduction system.

A schematic of the MCL process is shown in Figure 1-1. Feed coal is first combined with an approximately equal weight of anhydrous sodium hydroxide or mixed sodium and potassium hydroxides in a rotary kiln reactor where the mixture is heated to reaction temperatures of 325 to 415°C causing the caustic to 1) become sorbed in the coal matrix, 2) react with the coal sulfur and mineral matter, and then 3) dissolve the reaction products containing sulfur and inorganic components.

The reaction mixture exits the rotary kiln in the form of pellets which are next washed with water in a seven stage countercurrent separation system of two filters and five centrifuges. As an example, four weights of wash water per weight of coal may be used resulting in a 50% aqueous reacted caustic solution exiting the first stage of filtration. Nearly all of the coal-derived iron (usually 2-3% of the weight of the coal) and a small amount of sodium and/or potassium (0.1-2% of the weight of the coal) remains with the coal cake exiting this countercurrent filtration system.

The coal cake is next washed to remove the residual iron and caustic in a countercurrent three stage centrifuge system. Sulfuric acid is added to the first stage to produce a pH of 2 in order to dissolve the residual

Figure 1-1. Schematic of the MCL Process

alkali and iron hydroxide. As an example, two weights of wash water per weight of coal may be used resulting in an acid extract leaving the first stage containing about 2,000 ppm of mixed sodium and potassium sulfates and about 17,000 ppm of iron sulfate. The product coal exiting this acid washing system contains about one-half weight of sorbed water.

All of the iron sulfate and a major portion of the alkali sulfates can be removed by a sequence of heating and lime treatment to form insoluble minerals such as gypsum. The water can be recycled. Under some (very carefully controlled) conditions, the precipitate formed can be a jarosite-like double salt which can be easily disposed of or land-filled.

The aqueous caustic recovered from the first countercurrent washing circuit is limed at 180 to 250°F to remove the coal-derived mineral matter, sulfur compounds, and carbonates. The mixture is allowed to stand to permit settling out of the impurities, and the purified liquid is sent to a caustic evaporator where the wash water is recovered for recycle to the first wash train, while producing anhydrous caustic (as either a molten liquid or as flakes) for reuse in the initial leaching of the coal.

1.2 TEST COALS

A major goal of the integrated MCL project is to demonstrate reduction in sulfur to meet New Source Performance Standards. These standards are based on the sulfur content per million Btu, which is expressed in the form of $\text{lbs SO}_2/10^6 \text{ Btu}$. This removal applies to the run-of-mine (ROM) coal analysis. The two coals to be tested in this program (Table 1-1) contain 8-11 $\text{lbs SO}_2/10^6 \text{ Btu}$, which is the range that represents a majority of coal mines east of the Mississippi, and falls within Category 3 of the New Source Performance Standards which requires 90 percent reduction from ROM to 0.8 to 1.1 $\text{lbs SO}_2/10^6 \text{ Btu}$.

The data within the table show that each coal is reduced in sulfur content per unit heat content by about 40-45 percent by physical cleaning at the mine, but the remaining sulfur content is about five times that

TABLE 1-1. COAL FEEDS TO MCL CIRCUIT

Coal	%, w/w Dry Basis			Heat Content, Btu/Lb		Lbs SO ₂ /10 ⁶ Btu	
	<u>Sulfur</u>	<u>Ash</u>	<u>Vol%</u>	<u>MF</u>	<u>MAF</u>	<u>Coal</u>	<u>NSPS</u>
Pittsburgh No. 8 ^a							
Run of Mine	5.08	34.90	--	9144	--	11.10	
Cleaned	4.27	11.57	38.60	13016	14719	6.55	1.11
Kentucky No. 9 ^b							
Run of Mine	4.70	17.00	--	10525	--	8.93	
Cleaned	3.41	9.25	40.53	13259	14610	5.14	0.89

^a Powhatan No. 6 Mine (Powhatan Point, Ohio) Nacco Division, North American Coal Company

^b Pittsburgh & Midway Coal Company

required by the New Source Performance Standards. There are two options for meeting New Source Performance Standards, either post-combustion removal (flue gas desulfurization) or front-end cleaning as represented by the Gravimelt or MCL process.

1.3 SHAKEDOWN TESTS RESULTS

The results of shakedown testing can be summarized as follows: 1) sulfur and ash removals of 46 and 76 percent, respectively, were near normal for a kiln wall temperature of 340°C, despite the fact that the kiln reactor residence time was short (approximately two hours versus the previous 2.5 to 5 hours) and the caustic to coal ratio was low (only one weight of caustic per weight of coal rather than the usual 2 to 2.5 weights of caustic), 2) cake moisture in the caustic water washing train of approximately one weight of liquid per weight of coal is significantly lower than the three weights of liquid per weight of coal observed in previous noncontinuous testing, 3) the final MCL coal product moisture level, which averaged about 23 percent moisture and was as low as 10 percent moisture, is much lower than the one weight of moisture per weight of product coal observed in previous noncontinuous testing, 4) the product coal volatile content, which averaged 40 percent as compared with 38 percent for untreated feed Pittsburgh No. 8 coal, corresponds to virtually zero loss of volatiles due to MCL treatment; the small volatiles increase can be accounted for by the removal of the ash from the coal, 5) the efficient action of the countercurrent water washing system resulted in the acid wash solution containing less than 26 ppm of sodium and potassium which corresponds to residual alkali on the coal of only 6 ppm, 6) the single stage lime treatment vessel in the waste water treatment system produced a regenerated waste stream containing less than 25 ppm of coal derived sodium, potassium, silicon, aluminum and iron, and 7) very little conversion of caustic to carbonate (about 5%) occurred during reaction of caustic with the coal in contrast to all previous laboratory, benchscale and modular kiln operations; even this conversion might be further lowered by tightening any possible exposure of hot coal to air.

The following initial process conclusions were drawn from the shakedown information: 1) the combination of continuous operation, rigorous exclusion of air from the system, and mild conditions allows the production of MCL coal, for the first time, which has virtually no carbonate and volatiles loss and which does not have the propensity to form excessively wet cakes in countercurrent washing, 2) the low moisture content of the cakes can significantly lower processing costs through the elimination of a number of stages of washing and the utilization of less wash water, 3) very low alkali retention by MCL coal obtained from the water washing section indicates that alkali losses may be reduced to an insignificant level resulting in large cost savings to the process, 4) the use of one weight of caustic per weight of coal instead of 2 or 2-1/2 weights of caustic would result in additional savings of wash water and evaporation energy costs, and 5) carbonate formation may not be a problem in continuous test circuit operation, thereby saving lime costs and enhancing operation.

The following sections of this report contain a description of the test circuit, a description of the equipment installed, the shakedown test plans, the shakedown test results, and the operational test plan.

2. TEST CIRCUIT DESCRIPTION

One of the objectives of this project was to construct an integrated MCL or Gravimelt test circuit which incorporates as much as possible the already existing Government-owned modular test circuit from the previous Gravimelt program (U.S. Department of Energy Contract No. DE-AC22-83PC63032). That is, the current program involves modification of the existing facility and the procurement and installation of additional equipment to upgrade the test circuit.

In addition to the existing Government-owned modular test circuit, TRW had constructed and installed special TRW-owned facilities and equipment on the previous Gravimelt program that are available to the current project. These include: 1) a high bay building especially constructed to house the kiln reactor and aqueous caustic regeneration sections of the test circuit, 2) an 800 square foot cement process pad with 400 KVA of electrical utilities as well as water and sewage service, 3) analytical equipment purchased specifically for performance on the modular and integrated projects which includes a Fisher sulfur analyzer, an ion chromatograph and a coal analyzer for determination of volatiles, fixed carbon and ash, 4) a 24 cubic foot tray type electrical drying oven for coal with nitrogen blanket capability, and 5) two instrumentation data logger systems with video screen for operation of the aqueous regeneration and evaporation modules.

TRW installed the following capital improvements at the test plant site specifically for this system integration project: 1) an additional 500 KVA electrical power substation with motor control center to operate the new filtration/centrifugation train, 2) 1400 square feet of covered concrete process area adjacent to the kiln to house the filtration/centrifugation train, 3) a 400 square foot cement process pad for the evaporator, 4) a larger water treatment facility for disposal of

wash water and waste from the process meeting all county, state and federal regulations for disposal of solid and liquid waste.

The new integrated MCL installation allows for the continuous and simultaneous operation of all the MCL unit operations enabling the collection of operating data and equipment performance data that could not be obtained during batch or intermittent operation. The MCL process involves the treatment of coal with molten caustic (NaOH or KOH) followed by washing with water and acid to remove the sulfur and mineral matter from the coal. The coal product obtained from the MCL process meets New Source Performance Standards (NSPS) for sulfur and contains very low levels of coal derived mineral matter. The MCL process also involves the regeneration and reuse of the caustic media to minimize the costs associated with make-up chemicals, and the treatment of process waste water to allow for potential reuse in the washing operations.

The following six unit operations make up the MCL process (Figure 2-1):

- Rotary Kiln Reactor System (Section 100)
- Water Washing System (Section 200)
- Acid Washing System (Section 300)
- Caustic Regeneration System (Section 400)
- Caustic Concentration System (Section 500)
- Water Treatment System (Section 600)

All systems in the integrated test circuit were designed to operate continuously to produce 20 pounds of MCL coal per hour. The material balance used to design the integrated test circuit for a 20 lb/hr plant is presented in Table 2-1 along with design assumptions. The design was based on TRW's knowledge and experience acquired from the previous modular Gravimelt program, as well as information provided by equipment vendors. The six unit operations are described in detail in the following sections.

Figure 2-1. Block Diagram of the MCL Process

TABLE 2-1. MCL MATERIAL BALANCE

16-Mar-90

INPUT PARAMETERS FOR 20 POUND/HR COA! PRODUCT RATE (BASE CASE)

	FRACTION OF COAL AS FINES IN STREAM 202	0	
	IONICALLY BOUND CAUSTIC, LB/HR, STREAM 6	0.2	
	LIQUID/FILTERED COAL, LB/LB, STREAM 6	2	
	FINAL WASH WATER/COAL, LB/LB, STREAM 17	3	
	LIQUID/FILTERED PRODUCT COAL, LB/LB, STREAM 15	1	
	LIQUID/CENTRIFUGED SOLIDS, LB/LB, STREAM 12	0.5	
	K ₂ S, NA ₂ S REMOVED WITH MINERAL MATTER PER PASS, %	75	
	MINERAL MATTER REMOVED PER PASS, %	80	
	EXCESS CAO, CAUSTIC REGENERATION, %, STREAM 9	25	
	EXCESS CAO, WASTE WATER TREATMENT, %, STREAM 19	33	
	LIQUID/FILTERED SOLIDS, LB/LB, STREAM 22	5	
	PERCENT WATER IN CAUSTIC RECYCLE, STREAM 4	7.5	
	K ₂ CO ₃ , NA ₂ CO ₃ /KOH, NAOH IN MAKEUP CAUSTIC, STREAM 3	5.92/86.58	
	pH OF STREAM 18	2	
20			
87	MAF		
3	S		
10	ASH		
99	MAF		
0.5	S		
0.5	ASH		
2	CAUSTIC/COAL IN REACTOR EFFLUENT, LB/LB		
60	DISSOLVED MINERAL MATTER, % SI02		
25	AL2O3		
15	FE2O3		
1.61	H ₂ IN VENT, PERCENT OF COAL, STREAM 5		
0.42	CO ₃ /COAL FROM REACTOR, LB/LB, STREAM 2		
4	HUMIC ACID & PHENOLICS,% OF COAL,STREAM 2		
0.05	K,K,NA2CO3/K,NAOH FROM CENTRIFUGE,STREAM 11		
4	WASH WATER/COAL LB/LB, STREAM 7		

CALCULATED VALUES

FE2(SO4)3, K2SO4, NA2SO4 IN PRODUCT COAL, % OF COAL, STREAM 15	0.141
RESIDUAL CAUSTIC AFTER WASH, LB/HR, STREAM 6,	0.097
BOUND	0.200
TOTAL	0.297

TABLE 2-1. MCL MATERIAL BALANCE (CONTINUED)

STREAM	1	2	3	4	5	6	7	8	9	10	11
DRY FEED		REACTOR	MAKEUP	CAUSTIC	REACTOR	WASHED	WASH	CAUSTIC	CAO	LIMING	LIMING
COAL		EFFLUENT	CAUSTIC	RECYCLE	VENT	COAL	WATER	FILTRATE		REACTOR	CENTRATE
LB/HR						CAKE				EFFLUENT	
M.W.											
<hr/>											
COAL, MAF	24.890	19.800				19.800					
S	32.060	0.100				0.100					
ASH	2.861	0.100				0.100					
TOTAL	28.610	20.000				20.000		0.000			
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NAOH, KOH	48.055	40.000	7.436	53.513		0.297		39.703		51.112	46.077
NA2S, K2S	94.150	2.967		0.740		0.007		2.960		2.960	0.740
H2O	18.020		7.945	4.674	7.068	39.646	80.000	40.354		38.215	34.450
NA2CO3, K2CO3	122.100	17.091	0.508	2.812		0.041		17.050		2.556	2.304
AL2O3	101.960	0.862		0.172		0.002		0.860		0.860	0.172
SiO2	60.080	2.069		0.413		0.005		2.064		2.064	0.413
CAC03	100.090									11.881	
FE2O3	159.690										
FE(OH)3	106.875	0.554				0.554		0.000			
H2	2.020				0.322						
H2SO4	98.080										
CAO	56.080								8.321	1.664	
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HUMIC ACID/ PHENOLICS		0.800		0.798		0.002		0.798		0.798	0.798
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TOTAL, NON-COAL	0.000	64.344	15.889	63.122	7.390	40.554	80.000	103.790	8.321	112.111	84.953
TOTAL	28.610	84.344	15.889	63.122	7.390	60.554	80.000	103.790	8.321	112.111	84.953

TABLE 2-1. HCL MATERIAL BALANCE (CONTINUED)

STREAM	12	13	14	15	16	17	18	19	20	21	22
MINERAL RECYCLE TO											
WATER FROM											
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2.1 ROTARY KILN REACTOR SYSTEM (SECTION 100)

The rotary kiln reactor system is used for the reaction of the coal/caustic mixture at a temperature of 325 to 415°C and a residence time of 1 to 5 hours. The kiln system was installed and successfully operated on the previous modular Gravimelt program but required some modifications to the kiln feed and exit to allow for continuous steady state operation and integration with the other unit operations. The reactor system consists of the following subsystems:

- A feed subsystem to deliver a mixture of caustic and coal to the reactor.
- An electrically heated rotary kiln reactor.
- A quench and dissolution subsystem to convert the leached solid mixture exiting the kiln to a slurry for treatment in the subsequent water washing system.

The reactor unit accommodates a coal feed rate of 20 lb/hr and a caustic (potassium and sodium hydroxides) feed rate of 20-50 lbs/hr (caustic to coal ratios of 1 to 2 1/2 parts caustic to 1 part coal). The entire system (including feed, reactor, and quench subsystems) is blanketed with nitrogen to prevent oxidation of coal or contamination of the caustic by contact with the water vapor and CO₂ present in ambient air. A schematic of the reactor system is shown in Figure 2-2.

2.1.1 Reactor Feed Subsystem

Flaked recycle caustic is admixed with fresh make-up caustic and coal in a stainless steel drum. The drum is then attached to a tumbling device to promote intimate mixing of caustic and coal, which will be prepared in a ratio of 1 to 2 1/2 parts caustic to 1 part coal. From the drum tumbling device the coal/caustic feed is transferred to one of two coal/caustic feed bins (BN-101 and BN-102). The 10 ft³ capacity feed bins have been sized to store up to 12 hours of feed mixture at the nominal flow rate of 20 lb/hr coal with 20 to 50 lb/hr caustic. The feed mixture is pneumatically conveyed by nitrogen from the feed bins to a bag house filter, F-101. The dust free nitrogen from the filter is

Figure 2-2. Rotary Kiln Reaction System

SECTION 100 REACTION SECTION CONCEPTUAL FLOW DIAGRAM	CDF-1
DRAWN JLA	DATE 2-98
SCALE	REV SHEET 1 OF

recirculated by blower, BL-101. The solids are periodically shaken off the filter bags and discharged into the coal/caustic feed hopper/screw feeder of the kiln. The screw feeder to the kiln has a variable speed driver which allows for varying the feed rate from 30 lb/hr to 120 lb/hr.

2.1.2 Rotary Kiln Subsystem

The rotary kiln, supplied by C.E. Raymond, consists of a feed section, a 12 foot long furnace section, a cooling zone and a discharge area. The overall length of the kiln is about 32 feet. The feed section contains the variable speed screw feeder.

The main part of the kiln contains a 14-inch diameter tube which is 26 feet long. The tube is constructed of Inconel 601 which had high corrosion resistance to caustic at elevated temperatures. Part of the tube is enclosed in a 12 foot long furnace section which is electrically heated in four separate zones. The wall temperature of each zone is measured and controlled separately. The kiln will normally operate in the temperature range of 325° - 415°C.

The coal-caustic mixture will reside in the reactor for one to five hours. A residence time of 2.5 hours and temperatures of 370-390°C have been shown to provide coal meeting NSPS standards during operations of the kiln on the previous Gravimelt program. It is anticipated that a lower temperature range (e.g. 350-360°C) and longer residence time (4 hours) as well as a higher temperature range (380-390°C) and shorter residence time (1-2 hours) combinations will meet NSPS standards for most coals. The residence time is controlled by setting the rotational speed of the kiln, the angle of inclination and the solids level. The rotational speed can be varied from 0.98 to 9.84 rpm.

The kiln has been modified for this program to have the capability of operating with either an all solid feed stream consisting of coal and caustic flake or with separate feed streams consisting of coal and recycled molten caustic. The coal or coal and caustic flake pneumatic

solids feed system is described above in Section 2.1.1. The 500 to 600°F recycled molten caustic will be pumped directly into the kiln tube through a heat traced pipe.

The kiln is equipped with a rotating scraper to keep the internal walls of the kiln tube free of sticky solid deposits. The wall scraper is an auger-like device, 6 inches in diameter, that runs the length of the kiln tube and is in contact with the tube wall 15 degrees offset from the kiln bottom. It has a variable rotary speed driver (up to 20 RPM) and is rotated in an automatically timed sequence in alternate clockwise and counterclockwise directions. The use of a variable kiln slope, kiln rotational speeds, and the rotation settings of the wall scraper provide for differing volume loadings and retention times in the kiln.

The solids are transported from the feeding zone into the furnace section and then into the cooling zone. This cooling zone consists of the Inconel tube enclosed in a cooling jacket where cooling water is sprayed directly on the hot kiln tube. The jacket has the capability of cooling the coal-caustic mixture to 100°F.

Following the cooling zone, the solids exit the kiln through a bellows seal breaching arrangement and a rotary airlock valve. The bellows seal is capable of maintaining pressures up to ± 2 inches W.G. in the kiln tube without leakage. The reacted solids from the rotary discharge valve drop into a grinder for size reduction. The grinder is a new piece of equipment needed to break up the occasional large size coal/caustic balls which sometimes exit the kiln. A constant, small particle size ensures a constant and fast rate of caustic dissolution in the reactor product dissolver/quench tank.

From the grinder, the solids flow through a series of two gate valves which are operated sequentially by a timer, such that one of the valves is always closed. This "lock hopping" protects the kiln from any in-leakage of steam from the downstream quench subsystem.

Nitrogen is fed to the kiln to maintain a slightly positive pressure on the system and to sweep away the small amount of hydrogen gas and phenolic-type vapors produced as a by-product. The gas exhaust is swept through a venturi scrubber system prior to venting (Figure 2-2).

2.1.3 Reactor Product Quench and Dissolution Subsystem

The 40-70 pounds per hour of solids exiting the second gate valve drop into a stirred 12 gallon Inconel tank, T-101, where they are dissolved in an aqueous caustic filtrate solution that flows into this tank from the second vacuum filter in the coal washing system (stream 205). T-101 is sized for a nominal residence time of 30 minutes. However, the residence time can be varied from 15 to 45 minutes (1/4 to 3/4 full). The solid caustic (as well as sulfur compounds and ash leached from the coal) dissolve in the filtrate to produce an approximately 50 percent caustic solution. Due to the propensity of this solution to precipitate dissolved mineral matter and sulfur compounds on standing for extended periods of time, the residence time in T-101 is kept at a minimum.

The temperature of the resulting slurry is controlled at the desired value (180°F maximum) by adding or removing heat using a slurry heater, H-101, or a water cooled slurry cooler, E-101, located in a pump-around loop external to the dissolver/quench tank (Figure 2-2).

The approximately 0.2 gpm of 50 percent caustic solution produced in T-101 (stream 206) is pumped to the first vacuum filter in the water washing system by progressive cavity pump, P-102 (Inconel wetted parts). Excess filter feed will spill back to T-101.

2.2 WATER WASHING SYSTEM (SECTION 200)

In the water washing (or coal-caustic separation) system, the coal/caustic slurry from the reactor product dissolver/quench tank is water washed to remove the caustic, sulfur forms, and dissolved silica and alumina mineral matter. A block flow diagram of the multistage countercurrent water washing system is depicted in Figure 2-3 and a

typical design material balance is given in Table 2-2. All new equipment was installed in this section. The coal/caustic slurry is sequentially washed and dewatered in two vacuum filters, F-201 and F-202, five centrifuges, C-201 through C-205, and six interstage slurry tanks, T-203, T-206, and T-208 through T-211, in a countercurrent manner producing a washed coal virtually free of dissolved caustic (stream 6) and a concentrated aqueous caustic filtrate stream requiring regeneration (stream 8).

Two vacuum filtration stages are used prior to the five centrifuge stages because at caustic concentrations of greater than 20%, the aqueous caustic solution has a greater specific gravity than the coal cake and thus the liquor cannot be separated from the cake using centrifuges. Only after two vacuum filtration stages is the liquor dilute enough for separation from the cake using centrifuges.

A design wash water to coal ratio of about four to one will be used. The 0.15 gpm of wash water (stream 7) is fed to the final slurry tank, T-211. As the wash water streams move in a countercurrent direction with respect to the solids flow, they become more concentrated in caustic and salts while the residual liquid on the solids becomes more dilute in caustic and salts.

The main objectives of installing a continuous countercurrent washing system with a sufficient number of stages are to: 1) verify alkali recovery from coal utilizing water washing, 2) determine the minimum amount of water necessary to accomplish this recovery, 3) obtain filtration rate and cake moisture data sufficient to forecast the economics and operability of the process at full scale, 4) assess process chemistry and rates by providing the essential connecting unit operations of filtration and centrifugation so that the process can be operated on a real-time continuous basis with recycle of caustic and build-up of coal derived impurities to steady state levels, and 5) determine the minimum stages of filtration, centrifugation, and washing needed to adequately remove the caustic from the coal.

TABLE 2-2. WATER WASHING MATERIAL BALANCE

INPUT PARAMETERS

CAUSTIC/COAL IN FEED, LB/LB	2
WASH WATER/COAL, LB/LB	4
FIRST VACUUM FILTER DEWATERING (LIQ/SOLIDS, LB/LB)	3
SECOND VACUUM FILTER DEWATERING (LIQ/SOLIDS, LB/LB)	2
SECOND VACUUM FILTER WASHING EFFICIENCY	0.5
FRACTION OF WASH WATER USED FOR VACUUM FILTER WASH	1
CENTRIFUGES DEWATERING (LIQ/SOLIDS, LB/LB)	2
NUMBER OF CENTRIFUGES	5
IONICALLY BOUND CAUSTIC, LB/HR	0.2
FRACTION OF COAL AS FINES IN FILTRATE STREAM 202	0
BAG FILTER DEWATERING (LIQUID/COAL FINES, LB/LB)	5
COAL FINES WASH WATER (WATER/FINES, LB/LB)	1

STREAM NO.	201(2)**	202	203(7)**	204(6)**	205	206	207	208	209	209A	210
COAL/	1ST VACUUM	TOTAL	WASHED	2ND VAC	1ST VAC	1ST VACUUM	2ND VACUUM	VAC FILT	INTERMED.	CENTRIFUGE	
CAUSTIC	FILTRATE	WATER	COAL*	FILTRATE	FILTRATE	CAKE	FILTRATE	FEED	CAKE	CENTRATE	
LB/HR	FEED										
COAL	20.000	0.000	0.000	0.000	20.000	20.000	0.000	20.000	20.000	0.000	0.000
NaOH, KOH	40.000	39.703	0.000	0.297	22.971	23.268	9.092	32.359	9.388	5.997	5.997
Na2S, K2S	2.967	2.960	0.000	0.007	1.704	1.711	0.674	2.385	0.681	0.445	0.445
H2O	0.000	40.354	80.000	39.646	63.367	23.013	65.501	88.514	25.147	70.437	70.437
Na2CO3, K2CO3	17.091	17.050	0.000	0.041	9.815	9.856	3.885	13.741	3.926	2.562	2.562
AL2O3	0.862	0.860	0.000	0.002	0.495	0.497	0.196	0.693	0.198	0.129	0.129
SiO2	2.069	2.064	0.000	0.005	1.188	1.193	0.470	1.664	0.475	0.310	0.310
Fe(OH)3	0.554	0.000	0.000	0.554	0.000	0.554	0.000	0.554	0.554	0.000	0.000
HUMIC ACID/ PHENOLICS	0.800	0.798	0.000	0.002	0.459	0.461	0.182	0.643	0.184	0.120	0.120
TOTAL	84.344	103.790	80.000	60.554	100.000	184.344	80.554	160.554	60.554	80.000	80.000

* INCLUDES IONICALLY BOUND CAUSTIC IN ADDITION TO THE DISSOLVED CAUSTIC.

**STREAM NUMBERS IN PARENTHESES REFER TO TABLE 2-1.

TABLE 2-2. WATER WASHING MATERIAL BALANCE (CONTINUED)

STREAM NO.	210A	210B	211	212	213	214	215	216	217	218	219
	2ND VACUUM	2ND VACUUM	2ND VACUUM	FILTER	CENTRIFUG	CENTRIFUG	CENTRIFUG	CENTRIFUG	CENTRIFUG	CENTRIFUG	CENTRIFUG
	FILT WASH	VAC FILT	CAKE	CENTRATE	FEED	CAKE	CENTRATE	FEED	CAKE	CENTRATE	FEED
LB/HR	LIQUOR	BY-PASS									
COAL	0.000	0.000	20.000	0.000	20.000	20.000	0.000	20.000	20.000	0.000	20.000
NAOH, KOH	5.997	0.000	6.293	2.902	9.195	3.198	1.354	4.552	1.651	0.580	2.231
NA2S, K2S	0.445	0.000	0.452	0.215	0.667	0.222	0.100	0.323	0.108	0.043	0.151
H2O	70.437	0.000	30.083	75.373	105.455	35.018	77.841	112.859	37.486	79.075	116.561
NA2CO3, K2CO3	2.562	0.000	2.604	1.240	3.843	1.281	0.579	1.860	0.620	0.248	0.868
AL2O3	0.129	0.000	0.131	0.063	0.194	0.065	0.029	0.094	0.031	0.013	0.044
SiO2	0.310	0.000	0.315	0.150	0.465	0.155	0.070	0.225	0.075	0.030	0.105
FE(OH)3	0.000	0.000	0.554	0.000	0.554	0.554	0.000	0.554	0.554	0.000	0.554
HUMIC ACID/ PHENOLICS	0.120	0.000	0.122	0.058	0.180	0.060	0.027	0.087	0.029	0.012	0.041
TOTAL	80.000	0.000	60.554	80.000	140.554	60.554	80.000	140.554	60.554	80.000	140.554

**STREAM NUMBERS IN PARENTHESES REFER TO TABLE 2-1.

TABLE 2-2. WATER WASHING MATERIAL BALANCE (CONTINUED)

STREAM NO.	220	221	222	223	224	225(8)**	226	227	228	229(14)**	230
	THIRD	FIFTH	FOURTH	FOURTH	FIFTH	BAG	BAG	FINES	COAL	CONDENSATE	MAKEUP
LB/HR	CENTRIFUG CAKE	CENTRIFUG CENTRATE	CENTRIFUG FEED	CENTRIFUG CAKE	CENTRIFUG FEED	FILTRATE	FILTER CAKE	WASH WATER	FINES RECYCLE	FROM EVAPORATOR	DI WATER
COAL	20.000	0.000	20.000	20.000	20.000	0.000	0.000	0.000	0.000	0.000	0.000
NAOH, KOH	0.877	0.193	1.070	0.490	0.490	39.703	0.000	0.000	0.000	0.000	0.000
NA2S, K2S	0.050	0.014	0.065	0.022	0.022	2.960	0.000	0.000	0.000	0.000	0.000
H2O	38.720	79.692	118.412	39.337	119.337	40.354	0.000	0.000	0.000	37.720	42.280
NA2CO3, K2CO3	0.289	0.083	0.372	0.124	0.124	17.050	0.000	0.000	0.000	0.000	0.000
AL2O3	0.015	0.004	0.019	0.006	0.006	0.860	0.000	0.000	0.000	0.000	0.000
SiO2	0.035	0.010	0.045	0.015	0.015	2.064	0.000	0.000	0.000	0.000	0.000
FE(OH)3	0.554	0.000	0.554	0.554	0.554	0.000	0.000	0.000	0.000	0.000	0.000
HUMIC ACID/ PHENOLICS	0.014	0.004	0.017	0.006	0.006	0.798	0.000	0.000	0.000	0.000	0.000
TOTAL	60.554	80.000	140.554	60.554	140.554	103.790	6.000	0.000	0.000	37.720	42.280

**STREAM NUMBERS IN PARENTHESES REFER TO TABLE 2-1.

There are a number of advantages in using centrifuges wherever possible in place of filters in the integrated 20 lb/hr plant. First and foremost, modular equipment testing on the previous Gravimelt Program has shown that cake moisture levels for dewatering Gravimelt coal are considerably lower for centrifuge operation than for filters. Other advantages are the relative simplicity of operation and control in a multistage system and a lower power requirement for centrifuges due to elimination of the vacuum pump systems.

The design feed to the water washing system from the reactor system contains approximately two parts caustic per part coal (stream 2). The first vacuum filter, F-201, is of Inconel construction and is an enclosed system in an inert atmosphere. The filtrate from this vacuum filter is an approximately 50% aqueous caustic solution containing most of the dissolved sulfur compounds and silica and alumina mineral matter leached from the coal (stream 202). This filtrate stream is filtered in bag filter F-203 to remove any coal fines before being sent to the caustic regeneration system (stream 8). The coal fines recovered in bag filter F-203 are manually transferred to tank T-212, reslurried with water, and reintroduced into the water washing system ahead of the first centrifuge.

Iron hydroxide does not dissolve in the aqueous caustic but remains on the coal cake. The solids from the first vacuum filter, containing about 3 parts liquid per part coal (stream 207), is reslurried with the wash filtrate from the second vacuum filter in the first interstage slurry tank, T-203.

The six slurry tanks, T-203, T-206, and T-208 through T-211, are all 12 gallon 316L stainless steel vessels sized for a nominal 30 minutes residence each time. However, the residence time for each tank can be varied from 15 to 45 minutes. All tanks in the water washing section are provided with mixers to aid the repulping operations. All tanks are also provided with electric band heaters to maintain the slurries at temperatures up to 150°F to facilitate the liquid-solids separation in

the filters and centrifuges. The temperatures in the two vacuum filters will never be higher than 180°F to avoid boiling in the filters when a vacuum is drawn.

The second vacuum filter, F-202, is of 316 stainless steel construction and is an enclosed system in an inert atmosphere. Slurry tanks T-203 and T-206 are located next to and slightly below the two vacuum filters in order that the cakes produced by the filters can drop by gravity directly into the repulping tanks, eliminating the need for manually or mechanically transferring the cakes to the tanks. The solids from the second vacuum filter contain about 2 parts liquid per part coal on a design basis (stream 211). Progressive cavity pumps are used throughout the water wash system to provide a steady feed stream to the filters and centrifuges. Diaphragm pumps are used to recirculate the filtrate and centrate streams to the repulping tanks.

After the second vacuum filter, the solids are reslurried and dewatered in a series of five slurry tanks (T-206 and T-208 through T-211) and five centrifuges (C-201 through C-205). The liquid content of the solids from each centrifuge has a design level of about 2 parts liquid per part of coal. Test data on a similarly sized centrifuge used in the previous modular Gravimelt project indicates that actual weight ratios will be near 1.5 parts of liquid per part of coal. The solid bowl centrifuges are 6 inches in diameter and of 316 stainless steel construction. The slurry tanks for all centrifuges are located next to and slightly below the centrifuges in order that the cake can drop by gravity directly into the repulping tanks.

The coal cake from the final centrifuge, C-205 (stream 6), is further washed with acid and water in the acid washing system to remove the dissolved and ionically bound caustic along with the iron-containing mineral matter remaining on the coal cake.

The entire water washing system (filters, centrifuges, and tanks) is blanketed with nitrogen in order to prevent air (carbon dioxide) from entering the system.

2.3 ACID WASHING SYSTEM (SECTION 300)

In the acid washing system, the coal cake from the final centrifuge in the water washing system is treated with sulfuric acid to neutralize and remove the iron hydroxides and the residual bound and dissolved caustic forming sulfate salts. The resulting slurry is then centrifuged and washed to remove the sulfate salts and excess acid producing the final coal product. A block flow diagram of the acid washing system is depicted in Figure 2-4 and a typical design material balance is given in Table 2-3. All new equipment was installed in this system. Three stages of centrifuges are used, C-301 through C-303, along with three interstage slurry tanks, T-301 through T-303, to remove the sulfate salts and excess acid, producing the final coal product (stream 15). The coal product is low in sulfur, ash, and sulfate salts. The centrate from the first centrifuge, C-301 (stream 18) containing the excess acid and the sulfate salts is sent to the waste water treatment system. This 0.15 gpm centrate stream has a pH of about two.

About three parts wash water per part coal is used (design basis). The 0.12 gpm of wash water (stream 17) is feed to the final slurry tank, T-303. As the wash water streams move in a countercurrent direction with respect to the solids flow, they become more concentrated in acid and sulfate salts while the residual liquid on the coal solids becomes more dilute in acid and salts. This countercurrent washing arrangement is very similar to that used in the water washing system. The design liquid content of the solids from each centrifuge is about one part liquid per part coal.

The coal cake is treated with sulfuric acid in a 12 gallon Alloy 20 acid contacting vessel, T-301, sized to provide a nominal 30 minutes of residence time but which can be varied from 15 to 45 minutes. The sulfuric acid is metered into the acid contactor from a storage drum. The first two solid bowl centrifuges, C-301 and C-302, and slurry tank T-302 are of Alloy 20 construction. The final solid bowl centrifuge, C-303, and the final tank, T-303, are of 316 stainless steel construction. All three

Figure 2-4. Acid Washing System

TABLE 2-3. ACID WASHING MATERIAL BALANCE

INPUT PARAMETERS

LIQUIDS/COAL IN FEED, LB/LB 2
 WASH WATER/COAL, LB/LB 3
 CENTRIFUGES DEWATERING (LIQ/SOLIDS, LB/L 1
 NUMBER OF CENTRIFUGES 3
 pH OF CENTRATE TO WATER TREATMENT 2

STREAM NO.	301(6)*	302(16)*	303	304	305(18)*	306
	COAL		SECOND	FIRST	CENTRATE	FIRST
LB/HR	M.W.	CAKE	SULFURIC CENTRIFUG CENTRATE	FEE	TREATMENT	CAKE
COAL	20.000	0.000	0.000	20.000	0.000	20.000
NAOH, KOH	48.055	0.297	0.000	9.000	0.000	0.000
NA2S, K2S	94.150	0.007	0.000	0.009	0.007	0.002
H2O	18.020	39.646	0.000	99.677	80.067	19.610
NA2CO3, K2CO3	122.100	0.041	0.000	0.050	0.041	0.010
AL2O3	101.100	0.002	0.000	0.003	0.002	0.001
SiO2	60.080	0.005	0.000	0.006	0.005	0.001
FE(OH)3	106.875	0.554	0.000	0.000	0.000	0.000
H2SO4	98.080	0.000	1.107	0.009	0.040	0.010
NA2SO4, K2SO4	158.150	0.000	0.000	0.108	0.479	0.117
FE2(SO4)3	399.870	0.000	0.000	1.267	1.018	0.249
HUMIC ACID/ PHENOLICS	0.002	0.000	0.000	0.002	0.002	0.000
TOTAL	60.554	1.107	60.000	121.661	81.661	40.000

*STREAM NUMBERS IN PARENTHESES REFER TO TABLE 2-1.

TABLE 2-3. ACID WASHING MATERIAL BALANCE

STREAM NO.	LB/HR	M.W.	307	308	309	310(17)*	311	312(15)*
			THIRD CENTRIFUG CENTRATE	SECOND CENTRIFUG FEED	SECOND CENTRIFUG CAKE	WASH WATER	THIRD CENTRIFUG FEED	THIRD CENTRIFUG PRODUCT COAL
COAL			0.000	20.000	20.000	0.000	20.000	20.000
NaOH, KOH	48.055		0.000	0.000	0.000	0.000	0.000	0.000
Na2S, K2S	94.150		0.000	0.002	0.001	0.000	0.001	0.000
H2O	18.020		59.910	79.520	19.880	60.000	79.880	19.970
Na2CO3, K2CO3	122.100		0.002	0.012	0.003	0.000	0.003	0.001
AL2O3	101.100		0.000	0.001	0.000	0.000	0.000	0.000
SiO2	60.080		0.000	0.001	0.000	0.000	0.000	0.000
Fe(OH)3	106.880		0.030	0.000	0.000	0.000	0.000	0.000
H2SO4	98.080		0.002	0.012	0.003	0.000	0.003	0.001
Na2SO4, K2SO4	158.150		0.027	0.144	0.036	0.000	0.036	0.009
Fe2(SO4)3	399.870		0.058	0.307	0.077	0.000	0.077	0.019
HUMIC ACID/ PHENOLICS			0.000	0.001	0.000	0.000	0.000	0.000
TOTAL			60.000	100.000	40.000	60.000	100.000	40.000

*STREAM NUMBERS IN PARENTHESES REFER TO TABLE 2-1.

centrifuges are 6 inches in diameter. Slurry tanks T-302 and T-303 are 12 gallons each in order to provide a nominal 30 minutes of residence time which can also be varied from 15 to 45 minutes. The acid contactor and the two tanks are provided with mixers and with electric band heaters to maintain the slurries at temperatures up to 150°F to facilitate the liquid-solids separation in the centrifuges.

In a full scale plant, the slurry tanks in both the water washing and acid washing systems will be sized for about five minutes residence time. In this integrated 20 lb/hr plant, a five minute residence time corresponds to only 0.75 gallons or 0.1 cubic feet (based on a flow rate of 0.15 gpm). Tanks designed for only 5 minutes residence time at this scale are simply too small to be practical. For this reason, the nominal slurry tank residence time was increased to 30 minutes.

Progressive cavity pumps are used to provide a steady slurry feed stream to the centrifuges. Diaphragm pumps are used to recirculate the centrate streams to the repulping tanks. Tanks T-302 and T-303 are located next to and slightly below centrifuges C-301 and C-302 in order that the cake produced by the centrifuges can drop by gravity directly into the repulping tanks. In a similar manner, the acid contractor, T-301, is located next to and slightly below the final centrifuge, C-205, of the water washing system.

The relatively sulfur and ash-free coal product from centrifuge C-303 should contain less than 50% moisture and is collect in 55 gallon drums, T-304. Samples of the final coal cake are dried in ovens off-line. The centrate stream from the first centrifuge, C-301 (stream 18) containing the excess acid and most of the sodium sulfate and ferric sulfate salts formed by the neutralization reactions is sent to the waste water treatment system.

The entire acid washing system (centrifuges and tanks) is blanketed with nitrogen in order to prevent air (carbon dioxide) from entering the system.

2.4 CAUSTIC REGENERATION SYSTEM

The caustic regeneration system has been designed to remove the dissolved coal mineral matter and sulfur compounds from the caustic before the caustic is sent to the evaporator and recycled to the kiln. Caustic is recovered and recycled for economic and environmental reasons; caustic is expensive and difficult to dispose of. The feed to the caustic regeneration section is the filtrate from the first vacuum filter in the water washing section (stream 8, Figure 2-1). This filtrate consists of approximately 50% caustic (sodium hydroxide and potassium hydroxide) and 50% water, with associated dissolved mineral matter and sulfur compounds. These dissolved materials, together with water, must be removed in order to recover pure anhydrous caustic for recycle. The dissolved mineral matter and sulfur compounds are precipitated in the caustic regeneration system by the addition of lime. The precipitated solids are then mechanically separated from the caustic in a centrifuge and bag filters (Figure 2-5). The water is later removed from the mineral matter-free and sulfur-free caustic in the caustic concentration system (Section 500, Figure 2-1). The caustic regeneration operations are being conducted as much as possible in existing equipment from the previous Gravimelt program.

The feed to the caustic regeneration system corresponding to a product coal rate of 20 lb/hr, is approximately 0.11 gpm (stream 8, Figure 2-1). The feed is continuously pumped to an insulated 100 gallon 316 stainless steel precipitation tank, T-401. This existing tank provides a residence time range of 4 to 8 hours (1/4 to 3/4 full). The tank is provided with band heaters to heat and maintain the caustic solution at reaction temperatures between 180° and 250°F.

Lime, CaO, is added continuously by a solids feeder, FD-401, to tank T-401. The required amount of CaO (stream 9) is about 0.3 pound CaO/gallon of caustic. The lime forms insoluble complexes with the mineral matter and also causes the precipitation of the carbonates and sulfur compounds present. The caustic with the precipitated solids and

excess lime is continuously transferred using progressive cavity pump P-401 from tank T-401 to a second heated, existing 100 gallon 316 SS precipitation tank, T-402. Tank T-402 can provide up to an additional 8 hours of residence time to facilitate the further precipitation of mineral matter, carbonates, and sulfur-containing solids if needed. Such relatively long residence times may be required as the precipitation reactions involve potentially slow crystallizations. Tank T-402 may be bypassed if it is determined that adequate residence time is provided by tank T-401 alone. Tanks T-401 and T-402 are provided with mixers to prevent the solids from settling in the tanks and plugging the tank outlet lines.

The caustic along with the precipitated solids and excess lime is transferred using pump P-402 from tank T-402 to centrifuge C-401. P-402 is a progressive cavity pump to provide a steady feed stream to the centrifuge. In centrifuge C-401 most of the precipitated solids and excess lime are removed from the caustic. The centrifuge is an existing unit from the previous Gravimelt program. The centrifuge is 6 inches in diameter, of 316 stainless steel construction, and is virtually identical to the centrifuges in the water washing (Section 200) and acid washing (Section 300) systems. The remaining solids are removed downstream in bag filters F-401 through F-404.

The cake produced by centrifuge C-401 (stream 12) is collected in 55 gallon drums (D-401). These cake solids are wet with caustic solution (about 1 part solution per 2 parts solids). In order to recover this caustic solution for recycle and to make the solids more environmentally suitable for disposal, the solids are manually transferred to an existing 65 gallon 316 SS solids wash tank, T-403, reslurried with DI water, and pumped using P-402 back through the centrifuge. This solids reslurrying and recentrifuging operation is performed off-line at a later time when centrifuge C-401 is not in use removing mineral matter and sulfur compounds from 50 percent caustic solutions. After being centrifuged the second time, the solids are drummed and sent to disposal.

The centrate (stream 11) from centrifuge C-401 is collected in a 10 gallon heated centrate receiver, T-404, before being pumped through polishing bag filters, F-401 and F-402. A progressive cavity pump, P-403, is used to provide a steady feed to the bag filters. Bag filters F-401 and F-402 remove the remaining precipitated solids and excess lime not removed by centrifuge C-401. The filtrate from the bag filters is collected in an existing, heated 100 gallon 316 SS caustic storage tank, T-405, which also serves as the evaporator feed tank. Tank T-405 can hold up to 8 hours of caustic and provides a surge volume for caustic storage in case of a temporary shutdown of the evaporator system.

Since some caustic will be lost due to hang-ups on the sides of vessels, to unwanted side reactions, to caustic remaining on solids sent to disposal, and to the neutralization reaction with sulfuric acid in the acid washing system, a small amount of makeup caustic is continually added to the MCL system. Sodium hydroxide and potassium hydroxide is purchased as 50% (by weight) solutions in drums. The caustic drums are placed on a scale, and the caustic is pumped using drum pumps to a 100 gallon fiberglass caustic feed tank, T-406. The sodium and potassium hydroxide are mixed in tank T-406 using a mixer and/or an external pump-around loop before being transferred to the caustic storage tank, T-405, using pump P-405. This makeup caustic along with the mineral matter-free and sulfur-free caustic from the caustic regeneration system is pumped by progressive cavity pump P-404 from tank T-405, through polishing bag filters F-403 and F-404, and on to the evaporator where the remaining water is removed to produce an anhydrous caustic for recycle to the kiln. A second stage of polishing filtration is performed in filters F-403 and F-404 because some additional solids precipitation may occur after the caustic was filtered in F-401 and F-402 during the 8 hours (maximum) that the caustic may be held in caustic storage tank T-405. Polishing filters F-403 and F-404 are present to protect the evaporation system from plugs caused by additional solids precipitation.

All equipment (tanks, mixers, pumps, bag filters, centrifuge) in the caustic regeneration system are of 316 stainless steel or fiberglass construction. Most tanks are provided with mixers to provide agitation during the precipitation reactions. The four on-line caustic tanks (T-401, T-402, T-404, and T-405) are provided with electrical band heaters to maintain the caustic solutions at temperatures up to 250°F to facilitate the precipitation reactions and the liquid-solids separation in the centrifuge and bag filters. The entire caustic regeneration system (bag filters, centrifuge, and tanks) is blanketed with nitrogen in order to prevent air (carbon dioxide) from entering the system.

2.5 CAUSTIC CONCENTRATION SYSTEM (SECTION 500)

The feed to the caustic concentration or evaporation system is the mineral matter and sulfur-free 50 percent caustic solution from the caustic regeneration system (Figure 2-1). Approximately 20 to 50 pounds per hour of anhydrous caustic for recycle to the kiln is recovered by the evaporation step. The existing batch evaporation unit from the previous Gravimelt program is inadequate for the integrated system both in terms of size and in terms of mode of operation (batch). A new continuous rising film evaporator was installed along with a caustic flaking unit (Figure 2-6).

The caustic evaporator purchased for the integrated MCL process is a skid mounted package unit designed and built by the Buflovak Division of the Blaw Knox Food and Chemical Company. The aqueous caustic feed, at an anticipated concentration of 50% caustic, is stored and kept at temperature in a 100 gallon storage tank, T-405, in the caustic regeneration system. The hot caustic is pumped through two ten micron bag filters, F-403 and F-404, by a progressive cavity pump into the evaporator unit. This unit consists of a shell and tube exchanger in which hot oil circulates through the shell and the caustic flows through the tubes. The exchanger is vertical and the caustic is carried up the walls as a thin film by the evaporated steam. The hot oil and caustic flows are cocurrent. The exchanger has a total heat transfer surface of 11.65 ft² (provided by three 15 feet long, one inch Inconel tubes).

REVISIONS

LTR	DESCRIPTION	DATE

EQUIPMENT ENCLOSED WITHIN DASHED LINES IS INCLUDED IN CAUSTIC EVAPORATION PACKAGE UNIT

EV-501
CAUSTIC EVAPORATION UNIT (PACKAGE)

E-501
OVERHEAD CONDENSER

V-501
CONDENSATE DRUM

E-502
COLD TRAP

P-501
ANHYDROUS CAUSTIC METERING PUMP

E-503
WATER COOLER

V-502
CONDENSATE DRUM

U-501
SCALE

P-502
CONDENSATE PUMP

REGENERATED AQUEOUS CAUSTIC (SECTION 400)

HOT OIL

EXPANSION TANK

CIRCULATING PUMP

ELECTRIC CIRCULATION HOT OIL HEATER

EVAPORATOR HEAT EXCHANGER

VAPOR-LIQUID SEPARATOR

MOLTEN CAUSTIC HOLDING TANK

FLAKER

55 GALLON DRUM (MANUAL TRANSFER)

U-501

P-501 STROKE CONTROL

P-502

RECYCLE WATER TO WATER WASHING SYSTEM (SECTION 200)

FLAKED CAUSTIC TO KILN FEED SYSTEM (SECTION 100)

MOLTEN CAUSTIC TO KILN (SECTION 100)

SECTION 500

CFD-S

EVAPORATION SYSTEM

CONCEPTUAL FLOW DIAGRAM

SCALE

REV

SHEET 1 OF

Figure 2-6. Caustic Concentration System

Heat input to the evaporation unit is provided by electric resistance heaters in a 60 kW hot oil unit supplied as part of the evaporator package. Syltherm 800 is heated in this unit and is circulated through the shell of the evaporating exchanger (and the jacket of the flaker pan) by a centrifugal pump. The oil exiting this equipment passes through an expansion tank, to allow for any necessary degassing, before returning to the heating unit. Syltherm 800 is a silicone type oil which is extremely stable even at temperatures as high as 750°F. All Syltherm containing lines are insulated and electrically traced.

The evaporator unit was designed to process 180 lbs/hr of 30 percent aqueous caustic (50:50 NaOH:KOH) or 130 lbs/hr of 50 percent caustic (50:50 NaOH:KOH) and dry these feeds to give a caustic product containing about eight percent moisture. The overhead product temperature from the evaporator at this product moisture level is about 600°F.

The vapor-liquid stream exiting the top of the heat exchanger tubes consists of steam and molten anhydrous (8% moisture) caustic. The steam is separated from the molten caustic in a vapor-liquid separating vessel. The steam flowing out the top of this vessel is desuperheated with a small stream of injected water and then condensed in a shell and tube water cooled condenser, E-501. The resulting condensate is collected in a separation drum, V-501, downstream of the condenser. The vapor from this separation drum flows through a double pipe exchanger, E-502, that utilizes chilled water. Any resulting condensate is collected in a second separation drum, V-502. The uncondensed vapor is vented to the atmosphere. The condensate collected in the separation drums is recycled to the water washing system (Section 200).

The molten caustic product leaving the evaporator separator vessel drains by gravity through electrically traced piping into a heated holding tank, T-501, located directly below the vapor-liquid separator. The molten caustic is then directed to either a metering pump for injection into the rotary kiln reactor as a liquid or to the pan of a caustic

flaking unit for solidification. In the latter case, the caustic is fed to the kiln as a solid with the feed coal.

The flaker consists of a six inch diameter rotating drum (nine inches long) cooled internally with a water spray. The drum rotates through the pan collecting the molten caustic. Molten caustic picked up by the drum solidifies as it is cooled on the drum surface. The solid caustic is scraped off the drum as a thin flake by a knife and collected in 55 gallon drums. These 55 gallon drums are manually transferred to the rotary kiln reactor feed system where the recycled flaked caustic is mixed with fresh makeup caustic and feed coal. (The kiln feed system is described above in Section 2.1.1.)

In an alternative mode of operation, hot molten caustic from the evaporator is fed directly into the kiln reactor, by-passing the flaking system entirely. When operated in this mode, holding tank T-501 acts as a molten caustic feed tank. Molten caustic is transferred from tank T-501 through a heated traced insulated line to a specially designed kiln feed inlet. The molten caustic is transferred by diaphragm pump, P-501, which has remotely mounted nickel diaphragms.

In laboratory tests performed during the previous modular Gravimelt program, humic acids, derived from coal and soluble in the aqueous caustic feed, appear to convert to a coal-like insoluble solid during the evaporation cycle at 617°F (325°C) by a process of decarboxylation and float to the surface of the molten caustic. If this conversion is verified in integrated test operation, the decarboxylated humic acids will float to the top of holding tank T-501 and enter the flaker at the end of a run and will be separated in flake form, or will be recycled to the kiln and become a part of the coal filter cake, recovered in the washing section.

The caustic concentration system including the molten caustic holding tank, the enclosure for the flaker drum, and the hot oil expansion tank is blanketed with nitrogen to prevent air from entering the system.

2.6 WASTE WATER TREATMENT SYSTEM (SECTION 600)

The waste treatment system has been designed to treat the acid containing centrate stream from the acid washing system (stream 18, Figure 2-1) to make this stream suitable for disposal as waste water. If analysis indicates that this waste treatment system does not make the acid washing centrate stream suitable for disposal, this stream will be further treated in the site waste treatment facility. The 0.15 gpm centrate stream from the acid washing section contains potassium sulfate, sodium sulfate, and ferric sulfate salts formed by the neutralization reactions between iron hydroxide, the residual caustic, and sulfuric acid. The sulfate salts and any residual acid in this centrate stream are contacted with ferric sulfate, $(\text{Fe}_2[\text{SO}_4]_3)$ and lime (CaO) in the waste water treatment system to precipitate gypsum (CaSO_4) , ferric hydroxide, $\text{Fe}(\text{OH})_3$, and double salts of sodium and iron such as sideronatrite $(\text{NaSO}_4)_2\text{FeOH}\cdot 3\text{H}_2\text{O}$, ferrinatrite, $(\text{NaSO}_4)_3\text{Fe}\cdot 3\text{H}_2\text{O}$, and natrojarosite, $\text{NaFe}_3(\text{SO}_4)_2(\text{OH})_6$.

The water treatment system contains two 300 gallon tanks each followed by a bag filter system to remove the precipitated solids. A flow diagram of the water treatment section is depicted in Figure 2-7. All new equipment was installed in this system. The tanks were sized to provide about 24 hours residence time each. Such relatively long residence times may be required as the reactions involve potentially slow crystallizations. The tanks are provided with immersion heaters, mixers, and screw feeders.

The first tank, T-601, is of fiberglass construction and has internal immersion heaters to facilitate the iron complex mineral precipitation reactions. Solutions containing ferric sulfate react with sodium and potassium sulfates to form the highly insoluble sideronatrite, ferrinatrite, and natrojarosite in sulfuric acid solutions. Typical reaction conditions for these reactions are at a pH less than 4, temperatures from 100 to 180°F, and long residence times (20 hours). In some of the testing, ferric sulfate, $\text{Fe}_2(\text{SO}_4)_3$, will be added to

Figure 2-7. Water Treatment System

tank T-601 by means of a screw feeder in order to adjust the sodium to iron ratio to 1:3 to facilitate formation of natrojarosite. The precipitated salts are removed from the weak acid solution in an Alloy 20 bag filter, F-601, before the acid stream is sent to tank T-602. Progressive cavity pumps are used throughout the waste water treatment system to provide a steady feed stream to the bag filters.

If the sodium to iron ratio of the centrate stream from the acid washing section (stream 18) is found to be already near 1:3 due to exceptionally efficient washing in the water and acid washing systems (Sections 200 and 300), the centrate stream may be sent directly to the liming tank, T-602, thus eliminating the $\text{Fe}_2(\text{SO}_4)_3$ addition step in tank T-601. In this configuration, the iron complex minerals are precipitated in tank T-602 in the same step as the gypsum (CaSO_4) and other calcium salts and removed along with these solids from the waste water in bag filter F-602. Tank T-602 is a lined fiberglass vessel, and bag filter F-602 is of 316 stainless steel construction.

In the second tank, T-602, the acid stream is contacted with lime to neutralize the excess acid and precipitate gypsum, CaSO_4 , and ferric hydroxide, $\text{Fe}(\text{OH})_3$. A pH monitor/controller is used to control the speed of the lime screw feeder. The liming of alkali metal sulfates sometimes results in the formation of highly insoluble syngenite minerals which are intersalts of calcium and alkali metal sulfates. These salts are thus likely to be environmentally sound disposal products. The resulting sludge (stream 22) will be removed in a bag filter, F-602, before the waste water (stream 21) is sent to disposal. The 0.14 gpm waste water stream should only contain about 2000 ppm of dissolved solids (sodium, potassium, and calcium sulfates) which may qualify this stream for disposal as waste water. If this stream does not qualify, it will be further treated in the site waste treatment facility where it is subjected to the following: settling tank to remove solids, treatment to adjust the pH to ten, filtration, neutralization, and a second filtration. The final solids-free, neutralized filtrate is collected in a holding tank to be hauled away by a disposal company.

The waste water treatment system (bag filters and tanks) is not blanketed with nitrogen as are the other systems of the MCL plant since there is no need to prevent air from entering this system.

Samples are taken as needed from all gas, solid and liquid streams throughout in MCL plant in order to obtain sufficient data to assess the efficiency of the MCL system.

2.7 DATA ACQUISITION AND CONTROL SYSTEM

The operation of the integrated MCL test circuit will result in the generation of an extensive data base. Included in this data base will be pressure (psig), vacuum (inches H₂O or inches Hg), differential pressure (inches H₂O), liquid flow (gpm), air or nitrogen flow (SCFM), temperature (°F), and tank level (percent). This information, being voluminous, is most easily handled by a data acquisition and control system which collects and stores the process measurements in real time and utilize a portion of it for feedback control.

The microprocessor based data acquisition and control system installed at the test site is comprised of modular components as much as possible including process interface equipment, process control units and operator's display stations. The two operator's display stations incorporate CRT's displaying loop and alarm status and real time plant graphics. Keyboards allow configuration and control function adjustments to be entered into the system. The system was designed so that the failure of any single component will not disable the entire system.

The data acquisition and control system was designed to perform the following functions:

- To measure and collect data necessary to assess the technical performance of the process over the specified ranges of operating conditions.

- To evaluate the performance and reliability of selected instruments and controls in MCL service.
- To control and monitor the operation of the continuous MCL test plant over a wide range of operating conditions.

There are approximately 80 instruments and controls in the integrated MCL plant.

3. PROCUREMENT AND FABRICATION

Task 3 of the integrated MCL program involves the procurement and fabrication of all process equipment necessary to convert the existing modular test circuit from the previous Gravimelt program into an integrated, continuous, real-time test circuit. TRW actively solicited equipment design, performance, specification, availability and cost information from vendors and evaluated the information received. Long lead items and potential problem areas were identified and a timely procurement schedule was set up. TRW subcontracted the manufacturing or fabrication of equipment components as needed.

TRW integrated as much as possible the existing government-owned modular process equipment and accessories into the integrated test circuit. TRW also procured wear plugs, test coupons, and electronic monitoring probes necessary to acquire corrosion/erosion data. The materials to be studied for corrosion/erosion include stainless steel, nickel, Inconel, Alloy 20, and carbon steel.

The final integrated test circuit contains about 150 pieces of equipment including filters, centrifuges, tanks, reactors, feeders, the kiln, and the rising film evaporator. The major items of equipment are listed in Table 3-1.

Table 3-1. Major Equipment

ITEM	NAME
BL-101	PNEUMATIC CONVEYING SYSTEM BLOWER
BN-101	COAL FEED BINS
BN-102	CAUSTIC FLAKE FEED BINS
CV-101	PNEUMATIC COAL/CAUSTIC FEED SYSTEM
C-201	WATER WASHING SOLID BOWL CENTRIFUGE
C-202	WATER WASHING SOLID BOWL CENTRIFUGE
C-203	WATER WASHING SOLID BOWL CENTRIFUGE
C-204	WATER WASHING SOLID BOWL CENTRIFUGE
C-205	WATER WASHING SOLID BOWL CENTRIFUGE
C-301	ACID WASHING SOLID BOWL CENTRIFUGE
C-302	ACID WASHING SOLID BOWL CENTRIFUGE
C-303	ACID WASHING SOLID BOWL CENTRIFUGE
C-401	MINERAL MATTER CENTRIFUGE
D-401	MINERAL MATTER RECEIVER
E-101	SLURRY COOLER
E-102	SCRUBBER WATER COOLER
E-501	OVERHEAD CONDENSER
E-502	COLD TRAP EXCHANGER
E-503	WATER COOLER
EV-501	CAUSTIC EVAPORATION SYSTEM
FD-401	LIME FEEDER/HOPPER
FD-601	IRON OXIDE FEEDER/HOPPER
FD-602	LIME FEEDER/HOPPER
F-101	DUST FILTER
F-201	FIRST ROTARY DRUM VACUUM FILTER
F-202	SECOND ROTARY DRUM VACUUM FILTER
F-203AB	BAG FILTERS (2)
F-204AB	BAG FILTERS (2)
F-401,2	BAG FILTERS (2)
F-403,4	BAG FILTERS (2)
F-601AB	BAG FILTERS (2)
F-602AB	BAG FILTERS (2)
GR-101	KILN PRODUCT GRINDER
H-101	PIPE HEATER (BAND)
H-202	SLURRY TANK HEATER (BAND)
H-203	SLURRY TANK HEATER (BAND)
H-204	SLURRY TANK HEATER (BAND)
H-205	SLURRY TANK HEATER (BAND)
H-206	SLURRY TANK HEATER (BAND)
H-207	SLURRY TANK HEATER (BAND)
H-301	SLURRY TANK HEATER (BAND)
H-302	SLURRY TANK HEATER (BAND)
H-303	SLURRY TANK HEATER (BAND)
H-401	PRECIPITATION TANK HEATER (BAND)
H-402	PRECIPITATION TANK HEATER (BAND)
H-403	TANK HEATER (BAND)
H-404	TANK HEATER (BAND)
H-601	TANK HEATER (IMMERSION)

Table 3-1. Major Equipment (Continued)

ITEM	NAME
H-602	TANK HEATER (IMMERSION)
H-XXX	CONTROL PANEL, CONTACTORS, ETC.
KN-101	ROTARY KILN SYSTEM
MX-101	DISSOLVE TANK MIXER
MX-202	SLURRY TANK MIXER
MX-203	SLURRY TANK MIXER
MX-204	SLURRY TANK MIXER
MX-205	SLURRY TANK MIXER
MX-206	SLURRY TANK MIXER
MX-207	SLURRY TANK MIXER
MX-208	COAL FINES MIXER
MX-301	ACID CONTACTOR MIXER
MX-302	SLURRY TANK MIXER
MX-303	SLURRY TANK MIXER
MX-401	PRECIPITATION TANK MIXER
MX-402	PRECIPITATION TANK MIXER
MX-403	TANK MIXER
MX-404	TANK MIXER
MX-601	TANK MIXER
MX-602	TANK MIXER
P-101	CIRCULATING PUMP (CENTRIFUGAL)
P-102	CAUSTIC/COAL SLURRY PUMP (PROG CAV)
P-102A	CAUSTIC/COAL SLURRY PUMP (PROG CAV)
P-103	SCRUBBER CIRCULATING PUMP (CENTRIFUGAL)
P-202	FILTRATE PUMP (CENTRIFUGAL)
P-203	VAC FILTER FEED SLURRY PUMP (PROG CAV)
P-204	FILTRATE PUMP (CENTRIFUGAL)
P-205	WASH FILTRATE PUMP (CENTRIFUGAL)
P-206	VACUUM PUMP
P-207	CENTRIFUGE FEED SLURRY PUMP (PROG CAV)
P-208	CENTRATE PUMP (DIAPHRAGM)
P-209	CENTRIFUGE FEED SLURRY PUMP (PROG CAV)
P-210	CENTRATE PUMP (DIAPHRAGM)
P-211	CENTRIFUGE FEED SLURRY PUMP (PROG CAV)
P-212	CENTRATE PUMP (DIAPHRAGM)
P-213	CENTRIFUGE FEED SLURRY PUMP (PROG CAV)
P-214	CENTRATE PUMP (DIAPHRAGM)
P-215	CENTRIFUGE FEED SLURRY PUMP (PROG CAV)
P-216	CENTRATE PUMP (DIAPHRAGM)
P-217	COAL FINES PUMP (PROG CAV)
P-218	VACUUM PUMP
P-219	FILTRATE TRANSFER PUMP (DIAPHRAGM)
P-302	CENTRIFUGE FEED SLURRY PUMP (PROG CAV)
P-303	CENTRATE PUMP (DIAPHRAGM)
P-304	CENTRIFUGE FEED SLURRY PUMP (PROG CAV)
P-305	CENTRATE PUMP (DIAPHRAGM)
P-306	CENTRIFUGE FEED SLURRY PUMP (PROG CAV)
P-307	CENTRATE PUMP (DIAPHRAGM)

Table 3-1. Major Equipment (Continued)

ITEM	NAME
P-401	CAUSTIC TRANSFER PUMP (PROG CAV)
P-402	CAUSTIC TRANSFER PUMP (PROG CAV)
P-403	FILTER FEED PUMP (PROG CAV)
P-404	EVAPORATOR FEED PUMP (PROG CAV)
P-405	CAUSTIC METERING PUMP (FLEX-LINER)
P-406	CAUSTIC DRUM PUMP
P-407	CAUSTIC DRUM PUMP
P-501	ANHYDROUS CAUSTIC METERING PUMP
P-502	EVAPORATOR CONDENSATE PUMP (FLEX-LINER)
P-601	SLURRY TRANSFER PUMP (PROG CAV)
P-602	SLURRY TRANSFER PUMP (PROG CAV)
P-XXX	SPARE PUMP
T-101	KILN PRODUCT DISSOLVE TANK
T-102	QUENCH WATER TANK
T-202	FILTRATE TANK
T-203	SLURRY TANK
T-204	FILTRATE RECEIVER
T-205	WASH FILTRATE RECEIVER
T-206	SLURRY TANK
T-207	SEPARATOR/ SILENCER
T-208	SLURRY TANK
T-209	SLURRY TANK
T-210	SLURRY TANK
T-211	SLURRY TANK
T-212	COAL FINES SLURRY TANK
T-213	SEPARATOR/ SILENCER
T-301	ACID CONTACTOR TANK
T-302	SLURRY TANK
T-303	SLURRY TANK
T-304	PRODUCT COAL RECEIVER (200 DRUMS)
T-401	REGENERATION REACTOR
T-402	REGENERATION REACTOR
T-403	SOLIDS WASH TANK
T-404	CENTRATE RECEIVER
T-405	REGENERATED CAUSTIC STORAGE TANK
T-406	CAUSTIC MIXING TANK
T-601	IRON OXIDE CONTACTOR
T-602	LIME CONTACTOR
V-101	VENTURI SCRUBBER, SEPARATOR
V-501	CONDENSATE DRUM
V-502	CONDENSATE DRUM
W-101	WEIGH SCALE
W-102	WEIGH SCALE
W-301	WEIGH SCALE
W-501	WEIGH SCALE

4. INSTALLATION

Task 4 of the integrated MCL program involves preparing the test circuit for continuous process operation. The major efforts in this task include: installation of the process equipment, preparation of the shakedown test plan, checkout of the individual equipment units, verification of the sampling and analysis procedures and the corrosion/erosion procedures, test readiness review and performance of the shakedown tests. The installation of process equipment is covered in this section of this topical report. The shakedown test plan, the sampling and analysis procedures, and the corrosion/erosion procedures are covered in Section 5 of this topical report. Checkout of the individual equipment units and the performance of the shakedown tests are covered in Section 6.

In support of this integrated project, TRW spent its own funds to provide facilities for installation of equipment and to provide utilities to operate the plant. The major facility improvements included:

- Design and construction of concrete foundations for equipment mounting.
- Design and installation of a major electrical substation supplying 480/208V 3-phase and 110V single phase for all motors, pumps, heaters and instrumentation (motor control center and electrical distribution system).
- Design and installation of rain/weather covering for equipment and instrumentation.
- Design and installation of water and compressed air service to the facility.
- Refurbishment of the existing temperature controlled test area for use as a project control room to house the data acquisition and control system, and to provide desk facilities for the engineering staff during installation, shakedown and operation of the plant.
- Design and construction of a waste materials treatment system to interface with TRW waste treatment equipment allowing legal disposal of by-product solids and liquids generated during the project.

Following the construction of these facility and utilities installations, the major equipment for the plant was installed. When the larger equipment was in place, piping, electrical and instrumentation installations began. Finally, the instrumentation and controls were interfaced with the data acquisition and control system, and the system was calibrated.

Altogether, the integrated test circuit facility contains about 150 major pieces of equipment including filters, centrifuges, tanks, reactors, feeders, the kiln, and the rising film evaporator. It occupies 3700 square feet and is fitted with more than 6000 feet of piping, 425 valves, 80 instruments and controls as well as a computer control room with a data acquisition and reduction system.

4.1 REACTION SYSTEM

The rotary kiln is the major piece of equipment for the reaction system. The twelve-foot heated reaction section surrounded by a four-zone electric furnace can be seen at the far end of Figure 4-1. One of two riding rings for rotating the kiln, resting on a pair of trunnion rolls, can be seen on the near side of the electric furnace. To the left of this assembly is the cooler jacket housing the water spray cooler zone of the revolving cylinder. The bellows seal assembly is the next section to the left of the cooler jacket housing. The final feature (foreground) is the discharge breaching assembly with a hopper bottom and discharge airlock.

The feed end of the rotary kiln, prior to the installation of the pneumatic feed system, is shown in Figure 4-2. One of four cylinder knockers can be seen to the left in this photo. To the right of this is the second riding ring resting on trunnion rolls followed by the cylinder drive assembly which houses the drive chain and drive sprocket. To the right and below of this is the kiln drive motor. Above the motor is the bellows seal assembly followed by a flanged vent. To the right of this is the feed hopper which encloses the screw feed conveyor. The emergency hand crank for rotation of the kiln cylinder in the event of a power

Figure 4-1. Discharge End of Rotary Kiln

Figure 4-2. Feed End of Rotary Kiln

interruption or motor failure can be seen at the far lower right of the kiln structure.

The kiln control panel can be seen in Figure 4-3. The unit includes a Honeywell 10-point strip chart recorder (center) and a Barber-Coleman 560 series digital indicating-proportioning three-mode controller with deviation alarm and bar graph. The power supply to electric motors, the furnace and instrument panel can be seen at the left.

The pneumatic feed system for the kiln is depicted in Figure 4-4. Shown in the middle of this photo are the two feed bins BN-101 and BN-102. When fifty-five gallon drums containing coal and caustic are attached to the top of these bins, the feed materials flow by gravity into these bins. Shown at the bottom of this photo is the pneumatic nitrogen line. The dust bin is shown in Figure 4-5. The dust bin is located on a mezzanine above the feed end of the kiln. The kiln feed hopper can be seen in the lower left of this photo.

The 12-gallon dissolver/quench tank, which is located in a trench at the outlet of the kiln, is shown in Figure 4-6. The trench was dug especially for this program in order that the solids discharged from the kiln can fall by gravity into the quench tank. The frame of the kiln can be seen in the upper right of the photo. The grinder, slide valves, and mixer can be seen above the quench tank.

4.2 WATER WASHING AND ACID WASHING SYSTEMS

TRW provided a 1400 square foot covered concrete process area adjacent to the kiln to house the water washing and acid washing systems. This area, during groundbreaking, is depicted in Figure 4-7. The building housing the kiln is shown in the left rear of this photo. The same area is depicted in Figure 4-8 after the installation of the equipment. The water washing train of five centrifuges is shown on the left side of this photo and the acid washing train of three centrifuges is shown on the right side of Figure 4-8. An individual centrifuge is depicted in Figure 4-9 with its 12-gallon reslurry tank with mixer to the right and a

Figure 4-3. Kiln Control Panel and Power Supply

Figure 4-4. Pneumatic Feed System

Figure 4-5. Feed Dust Bin Above Kiln

Figure 4-6. Dissolver/Quench Tank

Figure 4-7. Water and Acid Washing Process Area During Groundbreaking

Figure 4-8. Water and Acid Washing Process Equipment

Figure 4-9. Water Washing Centrifuge on Stand

progressive cavity pump and a diaphragm pump on the ground underneath. One of the two vacuum filters can be seen in Figure 4-10. The filter is elevated above its 12-gallon reslurry tank which is shown on the right. To the left are the two filtrate receiving tanks.

4.3 CAUSTIC REGENERATION SYSTEM

Most of the tanks and the centrifuge in the caustic regeneration system were existing equipment from the previous modular Gravimelt program that were repiped and refurbished for this program. Figure 4-11 shows the refurbished regeneration system. In the middle of the photo is the existing centrifuge with a small solids collection drum underneath. In the top of the photo can be seen the bottoms of the existing 100 and 65 gallon tanks which are located on a mezzanine. The tops of two of the tanks can be seen on the right in Figure 4-5.

4.4 CAUSTIC CONCENTRATION SYSTEM

The caustic evaporator module is located on a specially constructed cement pad provided by TRW and equipped with electrical supply and cooling water utilities. The evaporator was bought as a packaged unit and is shown in Figure 4-12 as it was delivered to TRW. The top of the evaporator unit is shown to the right. The horizontal Syltherm expansion tank is on the top in this photo with the vapor-liquid separator below. The evaporator after it was installed and insulated is shown in Figure 4-13. The 15 foot high rising film exchanger can be seen in the middle of the photo. The vapor-liquid separator can be seen at the very top of the photo, extending above the roof. The horizontal Syltherm expansion tank which is also located at the top of the evaporator is hidden from view by the roof. The flaker is shown in Figure 4-14. The evaporator control panel is depicted in Figure 4-15. The Syltherm pump is shown at the bottom of this photo and the Syltherm heater is at the left.

4.5 WASTE WATER TREATMENT SYSTEM

The waste water treatment system consists of two 300-gallon fiberglass precipitation tanks, each with a set of bag filters. One of the tanks,

Figure 4-10. Vacuum Filter on Stand

Figure 4-11. Caustic Regeneration System

Figure 4-12. Evaporator During Delivery to Site

Figure 4-13. Installed Evaporator System

Figure 4-14. Flaker

Figure 4-15. Evaporator Control Panel

the liming tank, is shown in Figure 4-16. The lime feeder with feed hopper is above the tank, and the two bag filters downstream of the tank are standing on the pad to the right.

4.6 DATA ACQUISITION AND CONTROL SYSTEM

The data acquisition and control (DAC) system is shown during installation in Figure 4-17. One of the control monitors is shown at the left. Figures 4-18 through 4-32 present the major output screens of the DAC system and a description of the utilization of process data for control of unit operations.

Figure 4-16. Liming Tank in Water Treatment System

Figure 4-17. Data Acquisition System During Installation

Bar control chart for the Section 300 (acid wash) and Section 400 (regeneration system) liquid levels and controls. The top line shows the tank designation (T301) and the second line shows the pump (P302) being controlled by the tank level. The values indicated are all as a % of full scale (full tank or full pump speed). The line marked "P" is the actual tank level reading (% of full). The line marked "S" is the desired set point in % full for each tank. The line marked "M" indicates the % of full pump speed required to bring the actual "P" reading equal to the "S" set point. These functions are also graphically displayed: "P" is the height of the bar on the graph, "S" is the triangle to the right of each bar, and "M" is the triangle to the left of each bar. In this example the Section 400 tanks were not yet operating. Also note, that levels may be controlled either automatically (AUT) or manually (MAN).

Figure 4-18.

"1101" Jun.13 13:09 LI-207 T208 LVL (P209) PV= 25.70 INH20 IOP
 "1101" Jun.13 13:10 LIC-207 T208 LVL (P209) PV= 100.0 % HI
 "1102" Jun.13 13:10 LI-207 T208 LVL (P209) PV= 22.49 INH20 NR FROM HI
 "1102" Jun.13 13:10 LIC-207 T208 LVL (P209) PV= 69.4 % NR FROM HI

Bar control chart showing functions of the kiln reactor including sweep gas flow and pressure, and kiln external and internal temperatures. Channel 8 shows the logic indicators for the computer-timed lock-hopper valves at the kiln solids discharge.

Figure 4-19.

"1401" Jun.13 10:56 LIC-302 T302 LVL (P304) AUT
 "1401" Jun.13 10:56 LIC-303 T303 LVL (P306) AUT

Bar control chart showing the dissolver tank (Section 100) and the water wash train (Section 200) tank levels. Two tanks (T-203 and T-206) were not in use.

Figure 4-20.

Bar control chart for the tanks in both the water and acid wash sections. In this example, only the first (dissolver tank) temperature is controlled by the computer. The rest of the tanks were controlled by external controllers; however, all temperatures are displayed by the computer for data acquisition.

Figure 4-21.

Bar control chart showing kiln quench tank (dissolver tank) system including level; pump around loop heating, cooling, flow rate and pump pressure; and, various temperatures. Only the first three data channels are controlled by the computer - the others are read-out data only. Note, at the top of each bar is the maximum value (in engineering units) for the particular channel displayed.

Figure 4-22.

This is a piping diagram for the rotary kiln and the vent gas scrubber associated with it. Values for measured parameters are given directly below the instrument designation. For instance, TR-102 (zone 2 temperature in the kiln furnace) was 803.6 deg.F; venturi scrubber liquid flow was 4.70 gpm (FR-102); and GN2 input flow rate (sweep gas) was 13.58 scfm (FR-107).

Figure 4-23.

Piping diagram showing kiln dissolve tank and pump around loop showing temperatures, flow rates and pump pressure.

Figure 4-24.

WATER WASHING

89.Jun.13 13:42
GRAPH007 007/020

Piping diagram of the second and third centrifuges in the water wash train showing temperatures and liquid levels.

Figure 4-25.

ACID WASHING

89.Jun.13 13:44
GRAPH010 010/020

Typical piping diagram of the last part of the acid wash system showing temperatures, liquid levels and flow rates.

Figure 4-26.

General Explanation

TREND PANEL - Historical graph of up to 8 channels of data usually grouped together functionally.

TIME - 10 hours back from present time to present time is shown. By moving cursor (vertical dash immediately above the top right edge of the graph border) it is possible to display data in 10-hr increments (as far back as 5 days) for each period of recording (5 minutes per point here).

VALUES - Printed values under the ID for each channel at the top of the page provide the most current engineering unit reading at the time relative to the cursor; in this case, it is the current time shown to the left of the graph border.

SCALE - Values are displayed on the Y-axis in % of full scale for each channel. It is necessary to refer to other displays to determine the full scale value for each channel. For instance, Channel 1 shows the GN₂ pressure in the kiln in inches of water column on a scale of 0-2.0. The actual value shown (0.549) is approximately 25% of 2. The smooth curve at about 25% of full scale on the graph is a plot of this channel.

IDENTIFICATION - Displays on the data and control screen are in four colors which allow identification of each channel's trace. This cannot be seen in the black and white printout here presented.

Figure 4-27.

Trend Panel showing start-up of kiln dissolver tank system and the associated pump-around loop.

IDENTIFICATION - Displays on the data and control screen are in four colors which allow identification of each channel's trace. This cannot be seen in the black and white printout here presented.

Figure 4-28.

Trend Panel showing the result of an increase in the set point of temperatures in the wash train slurry tanks.

IDENTIFICATION - Displays on the data and control screen are in four colors which allow identification of each channel's trace. This cannot be seen in the black and white printout here presented.

Figure 4-29.

Trend Panel showing typical start-up of the kiln together with calibration of the internal thermocouple (boxy trace).

IDENTIFICATION - Displays on the data and control screen are in four colors which allow identification of each channel's trace. This cannot be seen in the black and white printout here presented.

Figure 4-30.

This Trend Panel shows the start-up of a run on the evaporator caustic flaker. Note the point at which the first hot, molten caustic is delivered (at about 0915) and the subsequent, relatively steady operation beginning at about 1000.

IDENTIFICATION - Displays on the data and control screen are in four colors which allow identification of each channel's trace. This cannot be seen in the black and white printout here presented.

Figure 4-31.

This Trend Panel shows the liquid level in tanks associated with the acid wash train (Section 300) and the regeneration system (Section 400). The tanks in Section 400 were being pumped out in a batch mode during this run and the cyclic nature of this operation contrasts with the automatic level controls in the Section 300 tanks (flat curve).

IDENTIFICATION - Displays on the data and control screen are in four colors which allow identification of each channel's trace. This cannot be seen in the black and white printout here presented.

Figure 4-32.

5. TEST PLANS

Prior to the performance of the shakedown tests, a Shakedown Test Plan defining coals to be tested, consumable materials, sampling, analysis, data collection, health and safety precautions, test schedule, and test conditions was prepared and submitted to the DOE for approval. The Shakedown Test Plan is included as Appendix A of this topical report. In addition to the above, the Shakedown Test Plan also includes an instrumentation list and sample Engineering Test Directives.

TRW also prepared a Sampling and Analysis Plan and a Corrosion/Erosion Plan. These two plans are included as Appendices B and C of this topical report, respectively. During shakedown, TRW performed tests to verify that the sampling and analysis procedures met the specific objectives of the program. All of the procedures identified for this integrated program are standard approved procedures. These procedures have been used repeatedly by the assigned TRW or vendor personnel on the previous Gravimelt modular circuit program. Therefore, the verification task during shakedown in most cases was of a refamiliarization effort for these personnel.

6. SHAKEDOWN

The purpose of shakedown testing is to identify and correct operating problems, to demonstrate the capability to properly operate the installed equipment, and to confirm sampling and analysis procedures.

6.1 CHECKOUT OF INDIVIDUAL EQUIPMENT UNITS

After the proper installation of process equipment, but prior to shakedown testing, TRW conducted a mechanical, structural, and electrical checkout of all installed equipment and instrumentation with emphasis on new equipment to insure the proper and satisfactory initiation of shakedown operation. All mechanical equipment was inspected for completeness of installation according to vendor specifications. Process equipment and piping were either pneumatically or hydrostatically tested for leaks. Valves were inspected and tested to verify operability. Motor connections were checked to verify correct rotational direction to preclude mechanical damage during start-up. Additionally, all instruments and controls were activated and checked for electrical continuity and proper response. Finally, the system was charged with water to check the operability of the pumps, heater systems, mixers, wash systems, and associated instruments and controls prior to shakedown testing.

6.2 SHAKEDOWN OPERATION

The MCL Integrated Test Circuit was operated in a shakedown mode for approximately 100 hours with the kiln reactor temperature at approximately 340°C and at a residence time of approximately 2 hours utilizing only one weight of caustic per weight of coal. The caustic feed composition was 50/50 sodium hydroxide to potassium hydroxide. The coal/caustic pellets exiting the kiln were washed with water in a countercurrent mode utilizing the five centrifuge stages. The water washed coal was next contacted with dilute sulfuric acid and washed free of the sulfuric acid utilizing the three stage countercurrent acid washing system. Product MCL

coal was obtained from the last stage of this system as a dewatered cake. The acid wash centrate stream was sent to a single stage lime treatment vessel for precipitation of impurities. Spent caustic was sent to the caustic regeneration system where mineral matter and sulfur compounds were allowed to precipitate at ambient temperature.

The package evaporator unit was not used during shakedown testing as the previous equipment checkout and evaluation had shown that the manufacturer has miscalculated heat losses and thereby underpowered the hot oil thermal system (60 kW versus a necessary 120 kW) for plant load following and had also failed to provide adequate heat tracing and insulation of the molten caustic collection and exit system. These changes were ordered.

The first two separators in the caustic water washing system are rotary drum vacuum filters. These units were not operated during shakedown testing as the previous equipment checkout had shown that the level control instrumentation supplied by the vendor needed to be replaced. This change was ordered. The caustic washing system has functioned reasonably well, utilizing only the five stages of centrifugation, but with elevated wash water flow rate to caustic ratio, to provide a first stage liquid specific gravity under 1.2 as is required for centrifuge coal separation.

The kiln reactor, newly fitted with an auger scrapper device, functioned exceedingly well providing for smooth feed and discharge of product coal-caustic, heating to reaction temperature, and a constant residence time.

6.3 SUMMARY OF SHAKEDOWN RESULTS

The results of shakedown testing can be summarized as follows: 1) sulfur and ash removals of 46 and 76 percent, respectively, were near normal for a kiln wall temperature of 340°C, despite the fact that the kiln reactor residence time was short (approximately 2 hours versus the previous 2.5 to 5 hours), and the caustic to coal ratio was low (only one

weight of caustic per weight of coal rather than the usual 2 to 2.5 weights of caustic), 2) cake moisture in the caustic water washing train of approximately one weight of liquid per weight of coal is significantly lower than the three weights of liquid per weight of coal observed in previous noncontinuous testing, 3) the final MCL coal product moisture level, which averaged about 23 percent moisture and was as low as 10 percent moisture, is much lower than the one weight of moisture per weight of product coal observed in previous noncontinuous testing, 4) the product coal volatile content, which averaged 40 percent as compared with 38 percent for untreated feed Pittsburgh No. 8 coal, corresponds to virtually zero loss of volatiles due to MCL treatment; the small volatiles increase can be accounted for by the removal of the ash from the coal, 5) the efficient action of the countercurrent water washing system resulted in the acid wash solution containing less than 26 ppm of sodium and potassium which corresponds to residual alkali on the coal of only 6 ppm, 6) the single stage lime treatment vessel in the waste water treatment system produced a regenerated waste stream containing less than 25 ppm of coal derived sodium, potassium, silicon, aluminum and iron, and 7) very little conversion of caustic to carbonate (about 5%) occurred during reaction of caustic with the coal in contrast to all previous laboratory, benchscale and modular kiln operations; even this conversion might be further lowered by tightening any possible exposure of hot coal to air.

The following initial process conclusions were drawn from the shakedown information: 1) the combination of continuous operation, rigorous exclusion of air from the system, and mild conditions allows the production of MCL coal, for the first time, which has virtually no carbonate and volatiles loss and which does not have the propensity to form excessively wet cakes in countercurrent washing; if these features can be maintained under more severe conditions, it may be possible to produce compliance MCL coal which has excellent combustion properties because of near zero loss of carbonates and hydrocarbon volatiles, 2) the low moisture content of the cakes, if maintained while producing compliance coal or near compliance coal, can significantly lower processing costs through elimination of a number of stages of washing and

the utilization of less wash water, 3) very low alkali retention by MCL coal obtained from the water washing section indicates that alkali losses may be reduced to an insignificant level resulting in large cost savings to the process and eliminating the necessity to precipitate sodium and potassium from waste water prior to recycle, 4) since one weight of caustic per weight of coal appears to operate as efficiently as 2 or 2-1/2 weights of caustic, it may be possible to further cut the use of caustic to 0.75 or 0.5 weights per weight of coal which would result in additional savings of wash water and evaporation energy costs, and 5) carbonate formation may prove not to be a problem in continuous test circuit operation, thereby saving lime costs and enhancing operation.

6.4 DETAILED SHAKEDOWN TEST RESULTS

6.4.1 Coal Product Quality

The shakedown test was run with a 1:1 caustic/coal feed, using 50:50 KOH/NaOH and 14 mesh Pittsburgh No. 8 coal. During the shakedown test on 12/15/1988, the rotary kiln was operated with wall temperatures of 346-350°C in Zone 1, 336-346°C in Zone 2, and 331-338°C in Zones 3 and 4. The coal product samples analyzed for product quality were all processed by the rotary kiln during this time period, even though some of the kiln processed coal was later fed to the wash train on 12/16/1988. The residence time of the caustic/coal mixture in the heated zones of the rotary kiln was approximately two hours.

Coal product samples were obtained during the shakedown test for analysis. The analysis results presented in Table 6-1 show an average residual sulfur content of 2.52 percent (dry basis) in the coal product, which corresponds to 46.19 percent sulfur dioxide (per unit heat content) removal based on coal feed to the rotary kiln, or 68.22 percent sulfur dioxide removal based on run-of-mine coal. The average residual ash content of the coal product was 2.76 percent, which corresponds to 76.10 percent ash removal. The substantial, but relatively modest sulfur and ash removals for MCL processing were due to the mild operating temperatures in the rotary kiln, which were necessary during the shakedown

test. As shown in Figure 6-1, there is good correlation between the sulfur content and ash content of the coal product:

$$\% \text{ sulfur} = 2.1941 + 0.1165 \% \text{ ash}$$

with a correlation coefficient of 0.923. This good correlation indicates that the operating conditions that favor sulfur removal also facilitate ash removal.

In addition to analysis of total sulfur, two coal product samples were also analyzed for sulfur forms. The sulfur forms for these two samples are compared with those for the feed coal as follows, all on a dry basis:

<u>Sample</u>	<u>% Pyritic S</u>	<u>% Sulfate S</u>	<u>% Organic S</u>
Feed	1.69	0.04	2.54
No. 1	0.20	0.02	2.15
No. 2	0.85	0.02	1.43

The sulfur forms analysis results show that under the mild operating temperatures, insoluble sulfate is not formed in the MCL process, and that some removal of organic sulfur takes place before the complete removal of pyritic sulfur. The two coal product samples were taken from two time periods (1 hour and 15 minutes apart) with the same operating conditions. The difference in the pyritic and organic sulfur contents for these two samples was probably caused by the difference in local operating parameters. For example, Sample No.1 might be processed with a higher local caustic/coal ratio and shorter residence time as compared to Sample No. 2.

The heating values of the coal product samples in Table 6-1 were computed based on the 14664 Btu/lb (MAF basis) determined from the analysis of two samples, and the ash content of the individual coal product samples. On a MAF basis, the heating value of the feed coal is 14719 Btu/lb. Thus, there is only 0.37 percent loss in heating value due to MCL processing.

Figure 6-1. Correction Between Product Coal Sulfur and Ash Content

The average volatile content of the coal product was 40.83 percent on dry basis, or 41.99 percent on MAF basis. The volatile content of the feed coal is 38.6 percent on dry basis, or 43.65 percent on MAF basis. The average loss in volatiles is therefore only 1.66 percent, or 3.8 percent relative to the feed coal.

The moisture content of the coal product was very low, ranging from 10.69 percent to 36.04 percent. The average moisture content was 23.15 percent. This low moisture content was the result of the mild operating conditions in the shakedown test. As shown in Figure 6-2, there is good correlation between the moisture content and ash content of the coal product:

$$\% \text{ moisture} = 48.975 - 9.34 \% \text{ ash}$$

with a correlation coefficient of -0.920. This good correlation suggests that the mild operating conditions which lead to more modest ash removal would also lead to lower moisture content for the coal product, probably because the resulting product would be associated with fairly low porosity and surface area. The moisture content can also be correlated with particle size of the coal product. As shown in Figure 6-3, the moisture content of the coal product increases with increasing percentage of coal product passing through 200 mesh, because the greater surface area of the finer coal particles would lead to higher moisture retention.

The changes in the carbon, hydrogen, nitrogen and oxygen content of the coal after MCL processing are presented in Table 6-2 on a moisture, ash, and sulfur-free basis (MASF). For comparison, the analysis results from the two operational runs conducted in 1986 for Pittsburgh No. 8 are also included in this table. These results show that under the mild operating conditions of the shakedown test, there were only small changes in the elemental makeup of the MCL coal from the feed coal, and the hydrogen loss was only about 0.6 percent. For Runs 1 and 6 conducted in 1986, the changes in the hydrogen and oxygen content of the product coal were significantly greater.

Figure 6-2. Relationship Between Product Coal Moisture Content and Ash Content

Figure 6-3. Relationship Between Product Coal Moisture Content and Particle Size

TABLE 6-2. CARBON, HYDROGEN, NITROGEN AND OXYGEN CONTENT CHANGE

<u>Sample</u>	<u>Carbon (MASF)</u>		<u>Hydrogen (MASF)</u>		<u>Nitrogen (MASF)</u>		<u>Oxygen (MASF)</u>	
	<u>%, w/w</u>	<u>% Change</u>	<u>%, w/w</u>	<u>% Change</u>	<u>%, w/w</u>	<u>% Change</u>	<u>%, w/w</u>	<u>% Change</u>
Feed	85.46	-	6.17	-	1.57	-	6.80	-
No.1	85.63	0.17	5.56	0.61	1.46	0.11	7.35	0.55
No.2	85.32	0.14	5.51	0.66	1.45	0.12	7.72	0.92
Run 1*	81.68	3.78	4.74	1.43	1.51	0.06	11.27	4.47
Run 6*	81.43	4.03	3.05	3.12	2.73	1.16	12.45	5.65

* Runs 1 and 6 were tests conducted in 1986. The reaction temperatures for Runs 1 and 6 were 320°C and 400°C, respectively.

6.4.2 Carbonate Formation

Carbonate is formed by the reaction of molten caustic with coal carbon in the presence of oxygen. During the shakedown run, selected kiln discharge (KD) samples and spent caustic solution (C-201 centrate) samples were collected and analyzed for carbonate and caustic compositions. These composition data were used to compute the fraction of caustic converted to carbonate, as well as the fraction of coal or coal carbon converted to carbonate. The computations were based on the following equations:

$$\text{KOH+NaOH feed} = \text{KOH+NaOH in product} + \text{KOH+NaOH used for sulfur and ash removal}$$
$$\text{Carbonate formed} = \text{K}_2\text{CO}_3 + \text{Na}_2\text{CO}_3 \text{ in product} - \text{K}_2\text{CO}_3 + \text{Na}_2\text{CO}_3 \text{ in caustic feed}$$

For 50:50 KOH/NaOH feed, one pound of KOH+NaOH converted to carbonate would result in 1.2783 lb of potassium and sodium carbonate. Therefore,

$$\% \text{ of caustic converted to carbonate} = \frac{100 \times \text{carbonate formed}}{1.2783 \times \text{KOH+NaOH feed}}$$

In reducing the data, the following compositions of the solid caustic mixture were used: 92.42 percent sodium and potassium hydroxide, 3.37 percent sodium and potassium carbonate, and 4.21 percent water. These are the average compositions of the mixed caustic fed to the rotary kiln, as determined from the analysis of nine samples.

It was also assumed that the carbon in the carbonate formed was derived from the carbon in the coal feed. For 50:50 KOH/NaOH feed, one pound of KOH+NaOH reacts with 0.12859 lb of coal carbon to form carbonate. Since the caustic feed contains only 92.42 percent sodium and potassium hydroxide and only part of this hydroxide is converted to carbonate and the caustic to coal ratio was 1.0, the percentage of coal feed converted to carbonate was calculated as follows:

$$\% \text{ coal converted to carbonate} = \frac{0.12859 \times 92.42 \times \text{carbonate formed}}{1.2783 \times \text{KOH+NaOH feed}}$$

In Table 6-3, data on carbonate formation from four KD samples and four C-201 centrate samples are presented. For the KD samples, the average conversion of caustic to carbonate was 5.01 percent, which corresponds to an average conversion of 0.60 percent of the feed coal to carbonate. For the C-201 centrate samples, data from one non-representative sample were not used in computing average values. On this basis, the average conversion of caustic to carbonate was 5.05 percent, which also corresponds to an average conversion of 0.60 percent of the feed coal to carbonate. Therefore, the KD and the spent caustic samples both indicate identical, low conversions of caustic and coal carbon to carbonate.

The low conversions of caustic and coal carbon to carbonate were due to the relatively low operating temperatures in the rotary kiln. In addition, operating with a low caustic/coal ratio of 1.0 might also lead to low conversion to carbonates.

6.4.3 Humic Acid

Humic acid analysis was performed on two of the liquid samples collected during the shakedown test. The concentration of humic acid in the C-301 (the first centrifuge in the acid wash section) centrate sample was 0.26 mg/g, which corresponds to 0.35 percent of the feed coal converted to humic acid. For the caustic regenerator effluent sample, the humic acid concentration was determined to be 3.1 mg/g, which corresponds to 4.1 percent of the feed coal converted to humic acid. These data show that most of humic acid would be found in the spent caustic and not in the acid wash. The total 4.5 percent conversion of feed coal to humic acid is indicative of the mild kiln operating temperatures during the shakedown run. Humics formation is known to decrease with increasing temperature. It is thought that the humics, which are polycarboxy organics, dissolve in the caustic even under mild conditions used in shakedown. Under more severe operating conditions, the humics would decarboxylate to insoluble coal-like particles and exit the plant with the coal product.

TABLE 6-3. CARBONATE FORMATION

<u>Sample</u>	<u>Date/Time</u>	<u>% Hydroxide</u>	<u>% Carbonate</u>	<u>% Caustic to Carbonate</u>	<u>% Coal to Carbonate</u>
KD	1212/1640	44.09	7.74	7.93	0.942
KD	1213/1530	40.57	1.98	0.52	0.062
KD	1215/0130	50.58	4.27	2.74	0.326
KD	1215/0215	36.87	7.10	8.83	1.049
			Mean	5.01	0.595
			Std Dev.	4.02	0.477
C-201	1215/0400	7.60	0.85	4.38	0.521
C-201	1215/0700	2.39	0.25	3.95	0.469
C-201	1216/0700	0.30	0.13	19.63	2.333
C-201	1216/1229	11.18	1.73	6.81	0.809
			Mean*	5.05	0.600
			Std Dev.	1.54	0.183

* The C-201, 1216/0700 sample contained unusually low hydroxide concentration, and was not representative of the shakedown operation. Small variations in this concentration could lead to large changes in computed carbonate conversion values. Therefore, data from this sample were not used in computing the mean carbonate conversion.

6.4.4 Particle Size Distribution

Particle size distribution data for the feed coal and processed coal are presented in Table 6-4. Representative data from Table 6-4 are plotted in Figure 6-4 to provide better illustration of the effects of MCL processing on coal particle size. The data plotted are derived from the experimental data, assuming that the coal particles follow a logarithmic-probability distribution of the form:

$$\text{Cumulative \% by Weight Undersize} = 50 [1 + \text{erf}(\log (X/X')/s)]$$

where X = particle size, X' = parameter with dimension of size, s = standard deviation parameter, and erf = normal probability function or error function. The parameters X' and s were determined from linear regression of the experimental data. In all cases, it was found that the experimental data can be well represented by the logarithmic-probability function. This indicates that the logarithm of the coal particle size is normally distributed.

The data presented in Table 6-4 and Figure 6-4 show that there is little or no size reduction for the coal particles after processing in the rotary kiln, based on examination of the kiln discharge (KD) coal particle size data. However, there is significant size reduction for the coal particles from the downstream operations, including: the dissolver loop pump around and initial centrifuge, as indicated by the T-101 (dissolver tank) data and C-201 (first centrifuge in caustic wash section) data; and subsequent washing, centrifuge and pump around, as indicated by the coal product (CP) data.

The C-201 data show that in the dissolver loop pump around and after the first centrifuge, only the larger coal particles have been reduced in size, and there is no significant size reduction for coal particles less than 0.075 mm (200 mesh). About 47 percent of the feed coal particles were greater than 0.425 mm (40 mesh). By comparison, only 13.9 to 28.9 percent of the C-201 coal particles were greater than this size.

TABLE 6-4. COAL PARTICLE SIZE DISTRIBUTION

<u>Sample</u>	<u>Date/Time</u>	Cumulative % Retained on Screen*					
		40 mesh <u>0.425mm</u>	60 mesh <u>0.250mm</u>	80 mesh <u>0.180mm</u>	100 mesh <u>0.150mm</u>	200 mesh <u>0.075mm</u>	Pan <u>0</u>
Feed	1212	47.0	64.0	71.4	75.2	85.3	100
KD	1214/0130	37.9	56.0	63.8	68.1	80.3	100
KD	1214/0830	39.7	56.7	63.8	68.1	79.7	100
T-101	1214/0805	8.5	33.0	47.1	55.7	77.1	100
C-201	1214/0550	19.5	41.4	53.3	60.0	75.9	100
C-201	1214/0805	13.9	35.3	50.3	58.8	80.4	100
C-201	1214/0815	28.9	53.6	65.9	72.5	87.6	100
CP	1216/0115	1.5	2.3	3.3	5.0	12.2	100
CP	1216/0200	1.0	2.1	3.3	6.2	16.0	100
CP	1216/0400	1.3	2.1	3.3	4.8	20.5	100
CP	1216/0700	1.8	9.4	19.9	28.3	56.7	100
CP	1216/1130	1.3	6.1	12.9	18.7	44.3	100
CP	1216/1600	3.7	19.6	40.4	54.7	85.3	100

* The screen sizes are given in both mesh size (40 mesh to pan) and in the size of the corresponding screen opening (0.425 mm to zero)

Figure 6-4. Particle Size Distribution for Feed Coal and Coal Product

The washing, centrifuge and pump around operations downstream of C-201 appear to further reduce the size of coal particles in all size ranges, as illustrated in Figure 6-4. In the C-201 coal product, only about 20 percent of the coal particles were smaller than 0.075 mm (200 mesh). In the final coal product, an average of 61 percent of the coal particles were smaller than this size, and an average of only 1.8 percent of the coal particles were greater than 0.425 mm (40 mesh).

With the additional operation of the two vacuum filters to remove most of the spent caustic from MCL coal before centrifuge separation, it may be possible to lessen coal particle size reduction by reducing the number of centrifuge stages, or by reducing centrifuge speed and minimizing shear generated in pump around loops and stirring of the coal slurry.

6.4.5 Process Liquid Analysis

Three liquid samples were collected on 12/16/1988, 12:40 and analyzed to determine the partition of aluminum, silicon, iron, sodium and potassium in the wash sections. These liquid samples included an effluent sample from the caustic regenerator (T-401), a centrate sample from the first centrifuge in the acid wash section (C-301), and an effluent sample from the lime contactor (T-602). The analysis results for these samples are presented in the following table, along with computed concentration levels in the spent caustic, assuming that all the mineral matter removed (an average of 76.1 percent) are in solution:

<u>Sample</u>	<u>Al(mg/L)</u>	<u>Si(mg/L)</u>	<u>Fe(mg/L)</u>	<u>Na(mg/L)</u>	<u>K(mg/L)</u>	<u>pH</u>
T-401 Effluent	2090	3190	1100	No Data	No Data	12.9
C-301 Centrate	14	<20	160	21.1	4.2	1.7
T-602 Effluent	<3	<3	<0.2	17.8	3.3	No Data
Spent Caustic (Computed)	707	1261	1233	20647	25033	---

These analysis results show that almost all the aluminum and silicon in the coal feed were found in the caustic regenerator effluent. For this

shakedown run, only about 13 percent of the iron in the coal feed was partitioned to the acid wash section. This result is atypical of the findings from later operational runs, where most of the iron was found in the acid wash section.

The sodium and potassium concentrations in the C-301 centrate totaled 25.3 mg/w, as compared to 45680 mg/w estimated for the spent caustic. This indicates that most of the caustic was effectively removed from the coal product in the caustic wash section. This also implies that the estimated caustic loss through the acid wash section would be only $25.3/45680$, or 0.055 percent of the input caustic. The final coal product contained an average of 23.15 percent moisture. Using this moisture content and the 25.3 mg/w alkali concentration in the C-301 centrate, it is estimated that the final coal product would only contain $25.3 \times 0.2315 = 5.9$ ppm alkali from the MCL process.

The T-602 effluent data show that lime treatment of the waste water from acid wash would effect almost total removal of the iron, but little or no additional removal of sodium and potassium salts. The treated water also contained very low levels of aluminum and silicon, and may be suitable for recycle as wash water.

The flow rates for the T-401 effluent, C-301 centrate, and the spent caustic stream were all the same, and constituents of the spent caustic were essentially split between the T-401 effluent and C-301 centrate. Therefore, the total concentrations of a constituent in the T-401 effluent and the C-301 centrate should be equal to its estimated concentration in the spent caustic. Comparison of the process liquid and estimated spent caustic concentrations show that there is excellent agreement between the measured and computed iron concentrations. The total measured iron concentration is 1260 mg/w versus the computed iron concentration of 1233 mg/w in the spent caustic. The total measured concentration levels of aluminum and silicon, however, are each about 2.5 to 3.0 times higher than the corresponding computed concentration values. This could be the result

of inadequate mixing in the caustic regenerator during the shakedown test, so that the measured aluminum and silicon concentrations in the T-401 effluent sample were not representative.

6.4.6 Wash Train Performance

The wash train during the shakedown test includes five centrifuge stages in the caustic wash section and three centrifuge stages in the acid wash section. The performance of the centrifuges can be evaluated from measurements of coal particles in the centrates and cake moisture in the coal product. The effectiveness of the wash train can be determined from measurements of alkalinity and pH as the washing progresses downstream.

The average pH, coal particle concentration, and the concentrations of sodium and potassium hydroxide and sodium and potassium carbonate in the centrate (C) and centrifuge feed slurry (S) at various centrifuge stages are presented in Table 6-5. As expected, the pH in both the centrate and the slurry decreased from C-201 to C-205, as washing progressed in the caustic wash section. In the acid wash section, the pH in both the centrate and the slurry increased from C-301 (where sulfuric acid solution is separated) to C-303 (where the fresh water is separated), again as expected. The pH in the centrate from C-303 was almost neutral. Similarly, Table 6-5 also shows the general decrease in both hydroxide and carbonate concentrations from C-201 to C-205. The hydroxide concentration decreased from 5.60 percent in the C-201 centrate to 6.7 ppm in the C-205 centrate. In Figure 6-5, the hydroxide and carbonate concentration is plotted against the pH for the caustic wash water. The plot shows that there is good correlation between alkalinity concentration and pH. Both the measured alkalinity concentration (sum of the hydroxide and carbonate concentrations) and the pH suggest the effectiveness of the wash stations in removing caustic from coal in the caustic wash section, and in removing acid from coal in the acid wash section.

Except in the C-201 centrate, the concentrations of coal particles in the centrifuge centrates were very low. Separation in the first centrifuge was not as good because of the smaller density difference

TABLE 6-5. CENTRIFUGE CENTRATE AND SLURRY CHARACTERISTICS

<u>Sample</u>	<u>pH</u>	<u>% Coal</u>	<u>% NaOH+KOH</u>	<u>% Na₂CO₃+K₂CO₃</u>
C-201/C	14.0	0.38	5.60	0.73
C-202/C	11.5	0.01	0.073	0.032
C-203/C	11.0	0.081	0.13	0.21
C-204/C	10.8	0.022	0.031	0.093
C-205/C	10.0	0.038	0.00067	0.042
C-201/S	13.8	1.93	3.09	0.54
C-202/S	12.3	2.62	0.31	0.057
C-203/S	11.0	1.45	0.062	0.33
C-204/S	10.6	0.33	0.029	0.097
C-205/S	9.7	1.26	0.032	0.029
C-301/C	1.7	0.04	--	--
C-302/C	4.9	0.05	--	--
C-303/C	6.7	0.015	--	--
C-301/S	2.1	1.78	--	--
C-302/S	3.4	2.09	--	--
C-303/S	6.4	1.52	--	--

* C indicates centrate and S indicates slurry

Figure 6-5. Relationship Between Alkalinity Concentration and pH in Caustic Wash Water

between the concentrated caustic solution and the coal product. With the additional operation of the two vacuum filters, the caustic concentration in the first centrifuge will be significantly lower and separation in this centrifuge is expected to improve accordingly. The average concentration of coal particles in the C-202 to C-205 centrate was 0.038 percent, as compared to the average coal particle concentration of 1.42 percent in the C-202 to C-205 slurry. For the acid wash section, the average concentration of coal particles in the centrates was 0.035 percent, as compared to the average coal particle concentration of 1.80 percent in the slurry. Thus, these centrifuges performed well and showed good separation. In addition, the average cake moisture of only 23.15 percent in the final coal product is also an indication of the good performance of the centrifuges in removing liquid from the solid product.

Excellent liquid removal was attained even for C-201, where the moisture to coal ratio in the two cake samples collected was only 0.51 and 0.96. The moisture content of this coal cake was significantly lower than the expected 2:1 moisture to coal ratio for the coal cake exiting the caustic wash section, and even lower than the expected 1:1 moisture to coal ratio for the final washed coal product.

7. TEST PLAN FORMULATION

Task 5 of the MCL Integrated Test Circuit program involves the formulation of a detailed operating test plan to provide sufficient data to permit a thorough technical and economic assessment of the integrated process. The test plan (which is attached as Appendix D) includes the proposed test matrix using two coals. Due to the multiplicity of processing factors desired for evaluation of the reaction, washing, and caustic regeneration/concentration systems, fractional factorial experimental designs will be used, where necessary, to evaluate the effect of changing process parameters.

A detailed Sampling and Analysis Plan is attached as Appendix B. Sampling of each of the process systems during simultaneous operation of the integrated circuit for analyses will entail the collection of solid, liquid, and gaseous samples. Sufficient sample sizes will be taken at various intervals to insure an adequate and representative portion of each sampled process stream. Also, sufficient sampling will be performed to obtain complete material balances of dry coal, organics, ash, sulfur, caustic, acid, water, and gaseous effluents. Where applicable, sampling/analysis will conform to procedures contained in ASTM Standards.

A detailed Corrosion/Erosion Plan is attached as Appendix C. TRW will acquire adequate corrosion/erosion data to provide the basis for proper selection of materials of construction for future plants.

APPENDIX A
SHAKEDOWN TEST PLAN

TRW Inc.
Electronics &
Defense Sector

One Space Park
Redondo Beach, CA 90278

FSCM NO. 11982

TITLE

MOLTEN - CAUSTIC - LEACHING
SYSTEM INTEGRATION
SHAKEDOWN TEST PLAN

DATE October 11, 1988

NO. K534.3.88-015

REV. 1

SUPERSEDING: _____

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1.0 INTRODUCTION

This test plan defines the objectives, requirements, and schedule for the shakedown of the Molten Caustic Leaching (MCL) plant that was constructed at TRW's Capistrano Chemical Facility. The test program is being conducted for the Department of Energy (DOE), Pittsburgh Energy Technology Center under contract number DE-AC22-86-PC91257. The new MCL installation allows for the continuous and simultaneous operation of all MCL unit operations enabling the collection of operating data and equipment performance data.

The MCL process involves the treatment of coal with molten caustic (NaOH and KOH) followed by washing with water and acid to remove the sulfur and mineral matter from the coal. The coal product obtained from the MCL process meets New Source Performance Standards (NSPS) for sulfur and contains very low levels of coal derived mineral matter. The MCL process also involves the regeneration and reuse of the caustic media to minimize the costs associated with make up chemicals, and the treatment of process waste water to allow for potential reuse in the washing operations.

The following unit operations make up the MCL process:

- Rotary Kiln Reactor System
- Water Washing System
- Acid Washing System
- Caustic Regeneration System
- Caustic Evaporation/Flaking System
- Water Treatment System

This document presents the shakedown plan which includes sections describing the hardware and instrumentation required by the six unit operations listed above, the consumable materials, the sampling, analysis

and data collection procedures and the safety precautions. Finally, the overall system shakedown tests are described.

2.0 OBJECTIVES

The overall program objectives are to design, install, and operate a steady state molten-caustic-leaching system to gather the data necessary for assessing the operability, economics and environmental acceptability of the MCL process for desulfurizing and demineralizing coal in the presence of caustic. During shakedown of the recently constructed/refurbished 20 lb/hr of coal unit, each unit operation will first be operated individually without heat. Finally, the entire system will be operated on an integrated, continuous basis with heat (in a manner that simulates commercial practice) to provide operational information for various caustic, acid, and coal streams. Product and by-product data will be acquired to evaluate sulfur and ash removal, to perform material balances, and to determine the environmental acceptability of the process.

Prior to the start of shakedown testing, a Test Readiness Review will be conducted to confirm that the processing equipment has been installed properly, that all necessary operating procedures have been prepared and approved, and that all safety precautions necessary to protect personnel and equipment have been implemented.

Shakedown testing will begin with sequential testing of individual unit operations. Initial tests of the system will be performed without applying heat to determine the capability of feeding, transporting, separating, and collecting coal and caustic using the system. Once the capability of transporting coal and caustic through the reactor has been demonstrated, the power to the rotary kiln and the various tank heaters will be turned on. As the test conditions for each unit operation reaches the levels required by the test directive, in-process and product materials samples will be collected and analyzed using the techniques and procedures planned.

3.0 TEST HARDWARE CONFIGURATION

The MCL system is depicted in Figure 1. The 20 lb/hr of coal facility consists of six major subsystems as follows:

- a rotary kiln reactor section
- a multi-stage filtration/centrifugation water wash section for separating caustic from coal
- a multi-stage centrifugation acid wash section for removing the residual caustic
- a caustic regeneration section for removing sulfide and mineral matter from spent caustic
- an evaporation section for concentrating the regenerated caustic
- a water treatment section for removing ions and salts from process water for recycle or discharge.

The rotary kiln reactor system is used for the reaction of the coal/caustic mixture at a temperature of 325-400°C and a residence time of 2 to 5 hours. The kiln system was installed and successfully operated on the previous MCL program but required some modification to allow for the continuous steady state operation and integration with the other operations. The water washing system is a new installation that will provide continuous countercurrent washing and dewatering of the kiln product to remove the caustic with the dissolved sulfur and mineral matter that has been leached from the coal. The acid washing system, also new, provides for the further washing and dewatering of the coal with acid to remove residual caustic and mineral matter that cannot be removed with reasonable amounts of wash water. The caustic regeneration system processes the aqueous caustic stream produced from the coal washing system to remove and recover the sulfur and mineral matter that has been leached from the coal. This system was installed and operated during the previous MCL program and has now been modified to allow for continuous steady state operation. The evaporation/flaking system consists of a new continuous caustic evaporator that produces anhydrous caustic from the regenerated aqueous caustic and enables the recycle of either molten liquid or flaked solid caustic to the kiln reactor. The water treatment system is a new

REVISIONS			
LTR	DESCRIPTION	DATE	APPROVED

Figure 1

TRW			
ORIGINATOR	DATE	MCL Conceptual Flow Diagram	
J.L. Amstutz	11/20/76		
Rev 1	12/1/76		
SIZE	PAGE NO.	CFD - 1	SHEET 1 OF
B	11982		

installation that will process the acid centrate stream from the acid washing operation to remove iron and sulfate salts as precipitates. The resultant effluent from this system can then be reused as process water or further treated for safe disposal.

4.0 REQUIREMENTS

The MCL test circuit is required, as a minimum, to demonstrate the ability to:

- Process 14 mesh x 0 bituminous coal at a nominal rate of 25 pounds per hour. The coal will contain approximately 10 percent ash and 2.5 to 5 percent sulfur. Only one coal will be used during shakedown testing.
- Produce a clean coal product with a total sulfur content that is significantly reduced to meet the NSPS criteria.
- Produce a clean coal product with an ash content less than 0.5 percent and a final moisture content less than 50 percent.
- Store the clean coal product in sealed drums after blanketing with nitrogen.
- Segregate, store, remove, and dispose of any toxic, hazardous, or waste material (solid, liquid, or gas) in a lawful and safe manner.
- Provide for the sampling of all feed, product, and process streams in order to perform heat and material balances.
- Provide for the weighing, drumming, and shipment of clean coal as directed by the Department of Energy.
- Provide corrosion and erosion data.

4.1 CONSUMABLE MATERIALS

All of the process chemicals are very commonly used in industry, as are the types of operations and the hardware to be used. Although there are no particularly unusual health and safety issues to be addressed, the process chemicals must be used with caution due to the potentially hazardous nature of hot caustic, acidic, and Syltherm solutions and caustic containing cake solids. These are many of the same issues which have been dealt with successfully on earlier Gravimelt programs.

The consumable materials required for shakedown testing are listed in Table 1. Handling and storage requirements for all chemicals (including waste streams) are listed in Table 2. See the Safety Hazard Analysis Report (K534.3.87-037) for a more complete description of chemical handling and storage requirements and for all Material Safety Data Sheets (MSDS).

Table 1. MCL System Consumable Materials

<u>Item</u>	<u>Grade/ Specification</u>	<u>Supplier</u>
Sodium Hydroxide	Commercial, 50% wt solution	
Potassium Hydroxide	Commercial, 50% wt solution	
Sulfuric Acid	Concentrated (98% wt)	
Coal	14 mesh x 0, dry	
Syltherm	Syltherm 800	Dow
Nitrogen	Commercial	
Lime	Commercial	
Iron Sulfate	Commercial	

Table 2. Chemical Handling and Storage Requirements
(note: lab coats, safety glasses, and safety shoes are to be worn at all times).

● Caustic (Sodium Hydroxide/Potassium Hydroxide)	Use/store per site requirements. Wear face shields and rubber gloves. Verify commercial quality as required.
● Sulfuric Acid	Use/store per site requirements. Wear face shields and rubber gloves. Verify commercial quality as required.
● Coal	<ul style="list-style-type: none"> - A dry nitrogen blanket is to be maintained over the ground coal at all times. - Maintain a positive pressure in the coal storage vessels to keep out moisture and air. - Wear dust masks and rubber gloves.
● Syltherm 800	Use/store per manufacturers requirements. Wear rubber gloves and face shields.
● Nitrogen	Use/store per applicable test site requirements.
● Lime	Use/store per site requirements. Wear dust masks and rubber gloves.
● Iron Salt (Ferric Sulfate)	Use/store per site requirements. Wear dust masks and rubber gloves.
● Gypsum/Sulfur Compounds/Mineral Matter	Store per site requirements. Wear face shields and rubber gloves.
● Cooling Water	<p>The rotary kiln and various heat exchangers will be cooled with water. Keep the coolant system as clean as possible. Probable sources of contamination are:</p> <ul style="list-style-type: none"> - Pipe scale/rust - Thread lubricants

4.2 INSTRUMENTATION/CONTROLS/DATA ACQUISITION

A list of all instruments including those which are computer/data logger functions is enclosed as Appendix A. A new on-line/off-line data acquisition/control/reduction system is being installed to interface with the instruments.

Instruments and process controls are being installed to perform the following:

- to measure and collect the data necessary to meet the data acquisition requirements of the Statement of Work to permit an evaluation and assessment of the technical and economic performance of the process over a range of operating conditions.
- to evaluate the performance and reliability of selected instruments and controls in MCL service.
- to provide a means of controlling and monitoring the operation of the continuous MCL system over a wide range of operating conditions.

Instruments and controls are being installed to allow for safe, reliable, unattended operation and data collection. The control system will also shut down the operation of the plant and notify security personnel as a result of equipment failures.

All magnetic tapes or disks of data will be retained and will be available for analysis on off-line data systems. Copies will be made available to DOE.

4.3 SAMPLING AND ANALYSIS REQUIREMENTS

The MCL process equipment has been designed to provide for taking representative samples of feed coal, processed coal, makeup and recycled caustic, wash filtrates, cakes, waste streams, and exhaust gases for chemical analysis. Also, wear plugs, test coupons, and electronic monitoring probes necessary to acquire corrosion/erosion data have been provided.

Particular attention will be given to those analyses necessary to obtain the following:

- The characteristics of the clean coal.
- Heat, mass, and material balances.
- The composition of non-coal waste streams.
- Contaminants present in the clean coal (potassium, sodium, acid, etc.).
- Compliance to local, state and Federal environmental regulations and requirements.
- Inertness, stability, and suitability of solid waste for use as a land fill material.
- The corrosion/erosion behavior of process equipment components (stainless steel, nickel, Inconel, Alloy 20).
- The correct operating range for the molten-caustic leaching, caustic-coal separation, caustic regeneration, and water treatment sections.
- The sulfur and ash removal efficiency as a function of the key process variables.
- Sufficient operating condition data to enable a technical/economic evaluation of the process.

4.4 QUICK LOOK DATA REQUIREMENTS

A new on-line/off-line data acquisition/control/reduction system is being installed for the MCL process. The on-line capabilities are as follows:

- Two process color CRT's with operator keyboards. The operators will be able to access the data base and process graphics as well as issue control commands.
- The operators upon request will be able to call up all information necessary to run the process units including plant overview displays, group displays, individual loop displays, trend displays, process flow sheet displays, and alarm displays.
- The process data handled by the system will include pressure, differential pressure, flow, temperature and level.

- The operators will be able to perform the following control functions: loop set point adjustments, alarm set point adjustment, mode switching, manual output adjustment, and remote/local set point switching.

These on-line capabilities will be used during shakedown to determine the ability of the MCL plant to meet the design and test requirements.

4.5 DATA ACCURACY REQUIREMENTS

In addition to the on-line data, the data base will include the physical and chemical characteristics of the feed coal, caustic, selected gas, liquid, and solid process streams, and equipment, piping, and instrument wear and performance data. This information will be stored in operating and maintenance logs or on space provided on the Test Directives. Preliminary Test Directives are enclosed in Appendix B.

During shakedown, the data collected, the design of the test directives, and the CRT information will be evaluated to determine whether adequate information is being collected to assess the capability of the MCL process to meet the design and test requirements. Mass balances will be performed to determine the adequacy of data collection and data reduction methods. Data gaps and errors in sampling or analysis techniques will be identified and correctional steps taken.

4.6 SPECIAL TECHNICAL AND ENGINEERING SUPPORT REQUIREMENTS

Flexibility of the TRW management system enables the project manager to draw upon resources of specialized technical and administrative groups throughout the company. While performing project tasks, supporting specialists take direction and control from the project manager. When their specific task is completed, they return to their respective groups. The project is not burdened by unnecessary personnel.

Data analysis is one area that will require the support of technicians and engineers other than the operating crew. For example, various solid, liquid, and gaseous streams after being sampled, will be analyzed either by in-house technicians or by an outside laboratory.

In addition, TRW will utilize a recognized expert on solid-liquid separation as a technical consultant to the project. This consultant will assist TRW in the evaluation of solid-liquid separation test data, and in the development of the process model for solid-liquid separation equipment.

4.7 DATA REPORTING REQUIREMENTS

All work will be documented and summarized for DOE. The starting coal, the product coal, and various centrifuge and filter cake samples will be analyzed for particle size distribution, moisture, total sulfur, sulfur forms, ash, and Btu content. Various aqueous centrate and filtrate streams will be sampled and analyzed for pH, total solids, carbonates, total organic carbon, total sulfur, and metal ions such as Na, K, Ca, Fe, Si, and Al. The nitrogen purge gas from the kiln will be analyzed for hydrogen, carbon dioxide, sulfur compounds, methane, and light hydrocarbons. During shakedown, the sampling and analysis techniques may be verified by the use of spiked samples. This data will be used to determine the effectiveness of the centrifuge and filters to separate liquids and solids and the effectiveness of the MCL process for removing sulfur and ash from coal.

4.8 RETENTION/SAMPLING REQUIREMENTS

During shakedown, tests will be performed to verify the sampling and analysis procedures and the corrosion/erosion procedures. Sufficient sampling will be performed to obtain complete material balances of dry coal, organics, ash, sulfur, caustic, reagents, water, and gaseous effluents. In addition, the on-line data collected by the data acquisition system such as flow rates, temperatures, pressure drops, and tank levels will be converted to a format such that it can be transferred to an IBM PC for storage and subsequent off-line analysis.

4.9 QUALITY ASSURANCE REQUIREMENTS

Quality assurance and reliability support will not be required beyond the level of activity necessary to maintain properly calibrated and controlled instrumentation and flow control devices. There is no deliverable hardware. However, there are deliverable representative clean coal samples to DOE.

4.10 SPECIAL HEALTH/SAFETY REQUIREMENTS

Potential hazards exist from chemical, thermal, and mechanical operations of the equipment. Open atmosphere exposure of the chemicals has been minimized. Operating personnel must wear standard laboratory protective gear such as safety glasses, laboratory coats, and safety shoes at all times to avoid skin or eye contact or breathing of any process chemicals. Emergency shower/eyewash equipment meeting Health and Safety requirements are provided. Special protective gear such as face shields, rubber gloves, rubber aprons, and/or dust masks shall be worn in addition to safety glasses, lab coats, and safety shoes during the following special operations:

- when centrifuges, vacuum filters, and bag filters are open
- during sampling or draining of liquids
- during transfer or sampling of solids
- during pre-operation preparation of solutions
- during handling of feed materials

See the "Safety Hazard Analysis Report, MCL Process", K534.3.87-037, for a more detailed discussion of special health and safety requirements.

5.0 TEST PROCEDURE REQUIREMENTS

5.1 OPERATING PROCEDURES

The MCL process is divided into six major unit operations:

- o Rotary Kiln Reactor System
- o Water Washing System
- o Acid Washing System
- o Caustic Regeneration System
- o Caustic Evaporation/Flaking System
- o Water Treatment System

Operating procedures have been written for each of the above six unit operations. The personnel conducting the shakedown tests must have a working knowledge of the components and function of each of the unit operations within the overall MCL system, have read and be familiar with each of the six operating procedures and with the Safety Hazard Analysis Report and all other pertinent documents (see list of references, Appendix C), and be properly attired at all times.

5.2 TEST SCHEDULE

A six week shakedown test schedule is planned for the MCL plant. The labor distribution for these six weeks is as follows:

Week 1 - 5 men (1 shift) for 8 hour days (200 man-hours total)

Week 2 - 10 men (1 shift) for 8 hour days (400 man-hours total)

Week 3 - 10 men (1 shift) for 8 hour days, (400 man-hours total)

Week 4 - 10 men (2 shifts) for 8 hour days each shift, (400 man-hours total)

Week 5 - 5-man crews (2 shifts) for 12 hour days each shift, 24-hour around the clock operation (600 man-hours total)

Week 6 - 5-man crews (2 shifts) for 8 hour days, 2-1/2 days, (200 man-hours total)

During the first four weeks, the various unit operations (rotary kiln, water washing, caustic regeneration, etc.) will be shaken down individually and finally integrated on 24-hour, 2 shift days. During week five the entire integrated process will be run just as if a test run was being made.

During week six, there will be two 5-man crews working for 2-1/2 8-hr days each. During this shortened week, repairs, maintenance, adjustments, etc., will be made to the MCL plant to assure smoother operation in the future. That is, any problems encountered during around-the-clock operation will be corrected. Also, during this shortened week, the analyses of any samples taken during around-the-clock operation will be completed. Based on the analysis results, additional adjustments will be made to the MCL plant. During this week, any final adjustments, repairs, and maintenance will be made to the MCL plant in preparation for regular operation of the plant during the next phase of the program.

The sampling and analysis procedure and analytical techniques will be verified during shakedown testing. That is, samples will be taken of representative solid, liquid, and gas streams whenever the plant is operated. Some samples will be analyzed in-house, while other samples will be sent to an outside laboratory for analysis. The results of these analyses will be used to guide future shakedown and process testing.

5.3 COAL TYPES

Only one bituminous coal (Pittsburgh No. 8) will be used during shakedown testing.

5.4 TEST CONDITIONS

The early shakedown tests will be performed using cold coal-water and cold coal-caustic-water solutions. However, during week five the system will be operated under actual commercial heated conditions.

6.0 SHAKEDOWN TESTS

6.1 TEST SEQUENCE

Shakedown testing will last for about six weeks. Shakedown testing will begin with the sequential testing of individual unit operations. Initial tests of the unit operations will be performed without applying heat to determine the capability of feeding, transporting, separating, and collecting coal and caustic. Once the capability of transporting coal and caustic through the system has been demonstrated, the power to the rotary kiln and the various heaters will be turned on. Test Directives (see Appendix B) will be distributed at the beginning of each shakedown test. The Test Directives will specify flow rates, instrumentation setting, temperatures, location of samples, etc.

6.2 INITIAL SHAKEDOWN TESTS

The initial shakedown tests will be performed on a cold system in order to determine the capability of feeding, transporting, separating and collecting coal and caustic using unreacted coal and caustic.

Shakedown testing will begin with the rotary kiln reactor section and will progressively add the caustic-coal separation water washing section, the acid washing section, the caustic regeneration section, the caustic evaporation section, and the wastewater treatment section. During the shakedown tests, only one coal will be tested. The shakedown tests will initially define bounds of smooth operation. For example, whether the kiln can process coal at caustic to coal ratios of 1-1/2:1 as well as the already demonstrated 2 to 2-1/2:1 as well as assessing the operation of the new kiln scraping device.

6.2 1 Rotary Kiln Cold Flow Tests

In the initial shakedown test, dry coal will be fed to the rotary kiln through the screw feeder. The primary focus in this initial test will be to make appropriate adjustments in the feeding, scraping and discharge systems where necessary. Although the rotary kiln is the same reactor used in the previous Gravimelt program and can be considered as a thoroughly tested processing device, direct delivery of reacted product through a size reduction device into the quench tank will require evaluation as will testing of the new kiln scraper device. The kiln can be disconnected at the bellows ahead of the grinder if needed to check on the kiln operation.

6.2.2 Other Subsystem Shakedown Tests

After achieving satisfactory operation of the reactor section, shakedown testing of the caustic-coal separation and water washing section will begin. The caustic-coal separation section contains multiple stages of filters, centrifuges and reslurrying tanks. Wash water will be added to the system in a countercurrent manner starting with the final reslurrying tank. Cold unreacted coal from the kiln will be contacted with cold water in the quench tank and then repeatedly separated and reslurried in the filters, centrifuges, and tanks in this subsystem until it is demonstrated that the whole train can operate simultaneously in a countercurrent wash mode on a cold coal-water slurry.

Shakedown testing of the acid washing section will next begin. The coal from the last centrifuge stage of the caustic separation section will be fed to the acid wash tank. The shakedown tests for this section will proceed in a similar way as for the water washing section, using only water (not acid) to reslurry the coal. "Product" coal will be obtained from the last centrifuge.

Shakedown of the caustic regeneration section will involve lime addition to the water "filtrate" stream from the water washing section and the centrifuge separation of the lime from the water solution. There

should be no coal mineral matter present in this filtrate since no heat has been applied to the coal. Shakedown testing in this section will begin after quantities of "filtrate" are obtainable from the first filter of the caustic-coal separation section. Shakedown testing of the caustic regeneration system is expected to be straightforward because of operating experience with the equipment in the previous Gravimelt program.

The wastewater treatment section will be tested in a similar manner. That is, a ferric salt and lime will be added to the "wastewater" stream from the "acid" washing section and subsequently removed in bag filters. Testing of this system will begin as soon as a "wastewater" stream is generated.

Shakedown testing of the caustic evaporator will commence using a clean caustic solution. The evaporator/flaker section will be the only section to be tested hot (at operating temperatures) during the first two weeks of shakedown testing. This is because the evaporator/flaker can be tested independently of the rest of the MCL plant. Barrels of purchased or prepared 50% aqueous caustic solution will be used as the feed to the evaporator. As anhydrous caustic is formed, it will be flaked and stored. This flaked caustic will be used as feed to the kiln during around-the-clock operation.

6.3 OVERALL SYSTEM SHAKEDOWN

With the exception of the evaporation unit, the flow and separation of liquid and solids in all subsystems will have been tested at this point using cold coal and water. The next step will be to introduce caustic into the system. That is, caustic flakes will be added to the kiln feed system and allowed to work their way through the kiln and the water washing, acid washing, and caustic regeneration systems. Since the kiln heaters have not been turned on as yet, there should not be any coal-caustic reactions taking place or mineral matter being produced. However, the tank heaters may be on to facilitate the separation of coal from caustic and water. The flow to any system should not be interrupted

by the addition of caustic. Only after the kiln and all filters, centrifuges, and pumps have been checked and have demonstrated their capability of handling a coal-caustic-water solution, will the kiln heaters be turned on.

The heaters on the kiln, and on the tanks in the water washing, acid washing, caustic generation, and waste treatment systems will next be turned on and the overall system will be allowed to come to temperature. The flow to any section should not be interrupted by the addition of heat and the subsequent chemical reactions and the production and separation of mineral matter.

The around-the-clock test in week five will involve only one coal feed rate, one caustic/coal ratio and one set of temperature/ residence time reaction conditions for coal desulfurization, demineralization and caustic regeneration. The process operating variables for the other sections, such as the amount of lime addition and the reaction temperature and residence time to precipitate the coal mineral matter in caustic regeneration, will also be fixed. All the test conditions will be selected on the basis of successful operation in the previous Gravimelt program and the initial shakedown tests and will be specified in the Test Directives.

6.4 SAMPLING AND ANALYSIS REQUIREMENTS

The MCL process equipment has been designed to provide for taking representative samples of feed coal, processed coal, makeup and recycled caustic, wash filtrates, cakes, waste streams, and exhaust gases for chemical analysis. Also, wear plugs, test coupons, and electronic monitoring probes necessary to acquire corrosion/erosion data have been provided.

During shakedown, tests will be performed to verify the sampling and analysis procedures and the corrosion/erosion procedures. Sufficient sampling will be performed to obtain complete material balances of dry

coal, organics, ash, sulfur, caustic, reagents, water, and gaseous effluents. Spiked samples may be used. The time, size, and location of samples will be specified in the shakedown Test Directives.

6.5 DATA ACQUISITION

The on-line data collected by the data acquisition system such as flow rates, temperatures, pressure drops, and tank levels will be analyzed. Also, during shakedown, the data collected, the design of the Test Directives, and the CRT information will be evaluated to determine whether adequate information is being collected to assess the capability of the MCL process to meet the design and test requirements. Data gaps and errors in sampling or analysis techniques will be identified and correctional steps taken.

APPENDIX A

SECTION 100: ROTARY KILN REACTOR SYSTEM

<u>Instrument No.</u>	<u>Description</u>
<u>Corrosion/Erosion Probes</u>	
CC-101	Corrosion Coupon on P-101 Discharge
CC-102	Corrosion Coupon in Tank T-101
<u>Control Valves</u>	
PV-101	Pressure Control Valve on Rotary Kiln Sweep Gas
TCV-110	Temperature Control Valve on CW to E-101
<u>Flow Instruments</u>	
FE-102	Flow Measuring Element on Caustic to V-101
FT-102	Flow Transmitter on Caustic to V-101
FR-102*	Flow Recorder on Caustic to V-101
FAL-102*	Flow Alarm (Low) on Caustic to V-101
FI-103	Flow Indicator for CW to E-102
FE-104	Flow Measuring Element on Slurry from P-101
FT-104	Flow Transmitter on Slurry from P-101
FR-104*	Flow Recorder on Slurry from P-101
FAL-104*	Flow Alarm (Low) on Slurry from P-101
FT-105	Flow Transmitter on CW to E-101
FR-105*	Flow Recorder on CW to E-101
FT-106	Flow Transmitter on CW to Kiln Quench Section
FR-106*	Flow Recorder on CW to Kiln Quench Section
FAL-106*	Flow Alarm (Low) on CW to Kiln Quench Section
FT-107	Flow Transmitter on N ₂ to Kiln
FI-107	Flow Indicator on N ₂ to Kiln
FR-107*	Flow Recorder on N ₂ to Kiln
FAL-107*	Flow Alarm (Low) on N ₂ to Kiln

* Computer/Data Logger Function

Instrument No.DescriptionLevel Instruments

LT-101	Tank T-101 Level Transmitter
LIC-101*	Tank T-101 Level Indicator Controller
LSH-101*	Tank T-101 Level Switch (High)
LAH-101*	Tank T-101 Level Alarm (High)
LSL-101*	Tank T-101 Level Switch (Low)
LAL-101*	Tank T-101 Level Alarm (Low)
LG-102	Tank T-102 Level Gage
LT-103	Rotary Kiln Feed Bin Level Transmitter
LSH-103	Rotary Kiln Feed Bin Level Switch (High)
LAH-103	Rotary Kiln Feed Bin Level Alarm (High)
LSL-103	Rotary Kiln Feed Bin Level Switch (Low)
LAL-103	Rotary Kiln Feed Bin Level Alarm (Low)

Pressure Instruments

PT-101	Pressure Transmitter on Sweep Gas from Kiln
PIC-101*	Pressure Indicator Controller on Sweep Gas from Kiln
PR-101*	Pressure Recorder on Sweep Gas from Kiln
PAH-101*	Pressure Alarm (High) on Sweep Gas from Kiln
PAL-101*	Pressure Alarm (Low) on Sweep Gas from Kiln
PT-102	Pump P-101 Discharge Pressure Transmitter
PR-102*	Pump P-101 Discharge Pressure Recorder
PI-103	Pump P-102 Discharge Pressure Indicator
PI-104	Pump P-103 Discharge Pressure Indicator
PI-106	N ₂ Make-up to BL-101 Pressure Indicator
PI-107	Pressure Indicator, N ₂ Purge to BN-102
PI-108	Pressure Indicator, N ₂ Purge to BN-101
PI-110	Pressure Indicator, N ₂ to Kiln
PI-111	Differential Pressure Indicator, Baghouse Filters
PRV-101	N ₂ Make-up to BL-101 Pressure Regulator
PRV-102	Baghouse Shaker N ₂ to Pressure Regulator
PRV-103	N ₂ to Kiln Pressure Regulator
PRV-103A	N ₂ to Kiln Pressure Regulator

* Computer/Data Logger Function

Instrument No.DescriptionRelief Valves

RV-101	Quench Tank T-101 Relief Valve
RV-102	Slurry Pump P-102 Relief Valve
RV-103	Caustic Flake Bin, BN-102
RV-104	Coal Bin, BN-101
RV-105	Baghouse Filter, F-101

Speed Control

SC-101	Speed Control on Pump P-102
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Temperature Instruments

TE-101	Temperature Measuring Element, Kiln Zone #1 Heater
TIC-101	Temperature Indicator Controller, Kiln Zone #1 Heater
TR-101*	Temperature Recorder, Kiln Zone #1 Heater
TE-102	Temperature Measuring Element, Kiln Zone #2 Heater
TIC-102	Temperature Indicator Controller, Kiln Zone #2 Heater
TR-102*	Temperature Recorder, Kiln Zone #2 Heater
TE-103	Temperature Measuring Element, Kiln Zone #3 Heater
TIC-103	Temperature Indicator Controller, Kiln Zone #3 Heater
TR-103*	Temperature Recorder, Kiln Zone #3 Heater
TE-104	Temperature Measuring Element, Kiln Zone #4 Heater
TIC-104	Temperature Indicator Controller, Kiln Zone #4 Heater
TR-104*	Temperature Recorder, Kiln Zone #4 Heater
TE-105	Temperature Measuring Element, Kiln Contents
TI-105	Temperature Indicator, Kiln Contents
TR-105*	Temperature Recorder, Kiln Contents
TE-106	Temperature Measuring Element, Kiln Contents in Quench Section
TI-106	Temperature Indicator, Kiln Contents in Quench Section

* Computer/Data Logger Function

Instrument No.DescriptionTemperature Instruments (Continued)

TE-107	Temperature Measuring Element, Caustic from E-102
TR-107*	Temperature Recorder, Caustic from E-102
TE-108	Temperature Measuring Element, Vent Gas from T-102
TR-108*	Temperature Recorder, Vent Gas from T-102
TE-109	Temperature Measuring Element, CW to E-101
TR-109*	Temperature Recorder, CW to E-101
TE-110	Temperature Measuring Element, T-101 Contents
TIC-110*	Temperature Indicator Controller, T-101 Contents
TR-110*	Temperature Recorder, T-101 Contents
TAH-110*	Temperature Alarm (High), T-101 Contents
TAL-110*	Temperature Alarm (Low), T-101 Contents
TE-111	Temperature Measuring Element, Slurry Return to T-101
TR-111*	Temperature Recorder, Slurry Return to T-101
TE-112	Temperature Measuring Element, CW from E-101
TR-112*	Temperature Recorder, CW from E-101
TE-113	Kiln Sweep Gas to V-101 Temp. Element
TR-113*	Kiln Sweep Gas to V-101 Temp. Recorder
TE-114	CWR from Cooler E-102 Temp. Element
TR-114*	CWR from Cooler E-102 Temp. Recorder
TE-115	Temperature Measuring Element, T-102 Contents
TR-115*	Temperature Recorder, T-102 Contents

* Computer/Data Logger Function

SECTION 200: WATER WASHING

Instrument No.

Description

Corrosion/Erosion Probes

CC-201	Not Used
CC-202	Not Used
CC-203	Corrosion Coupon or Probe in Tank T-203
CC-204	Corrosion Coupon on F-202 Inlet
CC-205	Corrosion Coupon on C-201 Inlet
CC-206	Corrosion Coupon on C-202 Inlet
CC-207	Corrosion Coupon on C-203 Inlet
CC-208	Corrosion Coupon on C-204 Inlet
CC-209	Corrosion Coupon on C-205 Inlet

Flow Instruments

FE-201	Flow Measuring Element on Caustic Filtrate
FT-201	Flow Transmitter on Caustic Filtrate
FI-201	Flow Indicator for Caustic Filtrate
FR-201*	Flow Recorder for Caustic Filtrate
FAL-201*	Flow Alarm (Low) for Caustic Filtrate
FE-202	Flow Measuring Element on Wash Water
FT-202	Flow Transmitter on Wash Water
FR-202*	Flow Recorder for Wash Water
FAL-202*	Flow Alarm (Low) for Wash Water
FV-202	Flow Control Valve for Wash Water
FE-203	Flow Measuring Element on F-202 Wash Liquor
FIC-203*	Flow Indicator Controller on F-202 Wash Liquor
FR-203*	Flow Recorder for F-202 Wash Liquor
FT-203	Flow Transmitter on F-202 Wash Liquor
FV-203	Flow Control Valve on F-202 Wash Liquor
FE-204	Flow Measuring Element on C-201 Centrate
FI-204	Flow Indicator for C-201 Centrate
FR-204*	Flow Recorder for C-201 Centrate
FT-204	Flow Transmitter for C-201 Centrate

* Computer/Data Logger Function

Instrument No.DescriptionLevel Instruments

LT-201	Not Used
LSH-202	Tank T-202 Level Switch (High)
LCH-202	Tank T-202 Level Control (High)
LCL-202	Tank T-202 Level Control (Low)
LT-203	Tank T-203 Level Transmitter
LIC-203*	Tank T-203 Level Indicator Controller
LSH-203*	Tank T-203 Level Switch (High)
LAH-203*	Tank T-203 Level Alarm (High)
LSL-203*	Tank T-203 Level Switch (Low)
LAL-203*	Tank T-203 Level Alarm (Low)
LSH-204	Tank T-204 Level Switch (High)
LCH-204	Tank T-204 Level Control (High)
LCL-204	Tank T-204 Level Control (Low)
LSHH-205	Tank T-205 Level Switch (High-High)
LSH-205	Tank T-205 Level Switch (High)
LSL-205	Tank T-205 Level Switch (Low)
LAHH-205	Tank T-205 Level Alarm (High-High)
LT-206	Tank T-206 Level Transmitter
LIC-206*	Tank T-206 Level Indicator Controller
LSH-206*	Tank T-206 Level Switch (High)
LAH-206*	Tank T-206 Level Alarm (High)
LSL-206*	Tank T-206 Level Switch (Low)
LAL-206*	Tank T-206 Level Alarm (Low)

* Computer/Data Logger Function

Instrument No.DescriptionLevel Instruments (Continued)

LT-207	Tank T-208 Level Transmitter
LIC-207*	Tank T-208 Level Indicator Controller
LSH-207*	Tank T-208 Level Switch (High)
LAH-207*	Tank T-208 Level Alarm (High)
LSL-207*	Tank T-208 Level Switch (Low)
LAL-207*	Tank T-208 Level Alarm (Low)
LT-208	Tank T-209 Level Transmitter
LIC-208*	Tank T-209 Level Indicator Controller
LSH-208*	Tank T-209 Level Switch (High)
LAH-208*	Tank T-209 Level Alarm (High)
LSL-208*	Tank T-209 Level Switch (Low)
LAL-208*	Tank T-209 Level Alarm (Low)
LT-209	Tank T-210 Level Transmitter
LIC-209*	Tank T-210 Level Indicator Controller
LSH-209*	Tank T-210 Level Switch (High)
LAH-209*	Tank T-210 Level Alarm (High)
LSL-209*	Tank T-210 Level Switch (Low)
LAL-209*	Tank T-210 Level Alarm (Low)
LT-210	Tank T-211 Level Transmitter
LIC-210*	Tank T-211 Level Indicator Controller
LSH-210*	Tank T-211 Level Switch (High)
LAH-210*	Tank T-211 Level Alarm (High)
LSL-210*	Tank T-211 Level Switch (Low)
LAL-210*	Tank T-211 Level Alarm (Low)
LT-211	Not Used
LCV-212	Separator/Silencer T-213 Continuous Drainer
LCV-213	Separator/Silencer T-207 Continuous Drainer
LSL-214	Tank T-212 Low Level Switch/P-217 Shutdown
LAL-214*	Tank T-212 Low Level Alarm

* Computer/Data Logger Function

Instrument No.DescriptionPressure Instruments

PV-201	Pulsair on F-201 Blowback
PV-202	Pulsair on F-202 Blowback
PDT-202	F-203 Differential Pressure Transmitter
PDIR-202*	F-203 Differential Pressure Indicator Recorder
PDAH-202*	F-203 Differential Pressure Alarm (High)
PT-203	Pressure Transmitter for Tank T-202
PI-203	Pressure Indicator for Tank T-202
PR-203*	Pressure Recorder for Tank T-202
PT-204	Pressure Transmitter for Tank T-204
PI-204	Pressure Indicator for Tank T-204
PR-204*	Pressure Recorder for Tank T-204
PI-205	Pump P-202 Discharge Pressure Indicator
PI-206	Tank T-203 Pressure Indicator
PI-207	Pump P-203 Discharge Pressure Indicator
PI-208	Pump P-204 Discharge Pressure Indicator
PI-209	Pump P-205 Discharge Pressure Indicator
PI-210	Tank T-206 Pressure Indicator
PI-211	Pump P-207 Discharge Pressure Indicator
PI-212	Pump P-208 Discharge Pressure Indicator
PI-213	Tank T-208 Pressure Indicator
PI-214	Pump P-209 Discharge Pressure Indicator
PI-215	Pump P-210 Discharge Pressure Indicator
PI-216	Tank T-209 Pressure Indicator
PI-217	Pump P-211 Discharge Pressure Indicator
PI-218	Pump P-212 Discharge Pressure Indicator
PI-219	Tank T-210 Pressure Indicator
PI-220	Pump P-213 Discharge Pressure Indicator
PI-221	Pump P-214 Discharge Pressure Indicator
PI-222	Tank T-211 Pressure Indicator
PI-223	Pump P-215 Discharge Pressure Indicator
PI-224	Pump P-216 Discharge Pressure Indicator
PI-225	Bag Filter F-203A Pressure Indicator
PI-226	Bag Filter F-203B Pressure Indicator
PI-227	Not Used
PI-228	Pump P-217 Pressure Indicator
PI-229	Tank T-202 Pressure Indicator
PI-230	Tank T-204 Pressure Indicator
PI-231	Tank T-205 Pressure Indicator
PI-232	Separator/Silencer T-213 Outlet Press. Indicator

* Computer/Data Logger Function

Instrument No.DescriptionPressure Instruments (Continued)

PT-232	Separator/Silencer T-213 Outlet Press. Transmitter
PR-232*	Separator/Silencer T-213 Outlet Press. Recorder
PI-233	Vacuum Filter F-201 Pressure Indicator
PI-234	Separator/Silencer T-207 Outlet Press. Indicator
PT-235	Separator/Silencer T-207 Outlet Press. Transmitter
PR-235*	Separator/Silencer T-207 Outlet Press. Recorder
PI-236	Vacuum Filter F-202 Pressure Indicator
PI-237	Vacuum Filter F-201 Blowback Press. Indicator
PI-238	Vacuum Filter F-202 Blowback Press. Indicator
PI-239	Filter F-204 Inlet Pressure Indicator

Relief Valves

RV-201	Not Used
RV-202	Not Used
RV-203	Not Used
RV-204	Relief Valve on Tank T-203
RV-205	Relief Valve on Pump P-203 Discharge
RV-206	Not Used
RV-207	Not Used
RV-208	Relief Valve on Tank T-206
RV-209	Relief Valve on Pump P-207 Discharge
RV-210	Not Used
RV-211	Relief Valve on Tank T-208
RV-212	Relief Valve on Pump P-209 Discharge
RV-213	Relief Valve on Pump P-210 Discharge
RV-214	Relief Valve on Tank T-209
RV-215	Relief Valve on Pump P-211 Discharge
RV-216	Relief Valve on Pump P-212 Discharge
RV-217	Relief Valve on Tank T-210
RV-218	Relief Valve on Pump P-213 Discharge
RV-219	Relief Valve on Pump P-214 Discharge
RV-220	Relief Valve on Tank T-211
RV-221	Relief Valve on Pump P-215 Discharge
RV-222	Relief Valve on Pump P-216 Discharge
RV-223	Relief Valve on Pump P-217 Discharge
RV-224	Relief Valve on Pump P-219 Discharge
RV-225	Relief Valve on Centrifuge C-201
RV-226	Relief Valve on Centrifuge C-202

Instrument No.DescriptionRelief Valves (Continued)

RV-227	Relief Valve on Centrifuge C-203
RV-228	Relief Valve on Centrifuge C-204
RV-229	Relief Valve on Centrifuge C-205
RV-230	Relief Valve on Separator/Silencer T-213
RV-231	Relief Valve on Vacuum Filter F-201
RV-232	Relief Valve on Vacuum Filter F-202
RV-233	Relief Valve on Separator/Silencer T-207

Speed Control

SC-201	Not Used
SC-202	Not Used
SC-203	Speed Control on Pump P-203
SC-204	Not Used
SC-205	Not Used
SC-206	Speed Control on Pump P-207
SC-207	Speed Control on Pump P-209
SC-208	Speed Control on Pump P-211
SC-209	Speed Control on Pump P-213
SC-210	Speed Control on Pump P-215
SC-211	Speed Control on Pump P-217

Temperature Instruments

TE-201	Not Used
TE-202	Tank T-203 Temperature Measuring Element
TIC-202	Tank T-203 Temperature Indicator Controller
TE-203	Tank T-206 Temperature Measuring Element
TIC-203	Tank T-206 Temperature Indicator Controller
TE-204	Tank T-208 Temperature Measuring Element
TIC-204	Tank T-208 Temperature Indicator Controller
TE-205	Tank T-209 Temperature Measuring Element
TIC-205	Tank T-209 Temperature Indicator Controller
TE-206	Tank T-210 Temperature Measuring Element
TIC-206	Tank T-210 Temperature Indicator Controller
TE-207	Tank T-211 Temperature Measuring Element
TIC-207	Tank T-211 Temperature Indicator Controller

Instrument No.DescriptionTemperature Instruments (Continued)

TE-208	Tank T-203 Temperature Measuring Element
TR-208*	Tank T-203 Temperature Recorder
TE-209	Tank T-206 Temperature Measuring Element
TR-209*	Tank T-206 Temperature Recorder
TE-210	Tank T-208 Temperature Measuring Element
TR-210*	Tank T-208 Temperature Recorder
TE-211	Tank T-209 Temperature Measuring Element
TR-211*	Tank T-209 Temperature Recorder
TE-212	Tank T-210 Temperature Measuring Element
TR-212*	Tank T-210 Temperature Recorder
TE-213	Tank T-211 Temperature Measuring Element
TR-213*	Tank T-211 Temperature Recorder
TE-214	Tank T-202 Temperature Measuring Element
TR-214*	Tank T-202 Temperature Recorder
TE-215	Tank T-204 Temperature Measuring Element
TR-215*	Tank T-204 Temperature Recorder
TE-216	Tank T-205 Temperature Measuring Element
TR-216*	Tank T-205 Temperature Recorder

* Computer/Data Logger Function

SECTION 300: ACID WASHING

Instrument No.

Description

Analytical Instruments

AE/AT-301	Acid Contactor T-301 pH Monitor
AIC-301	Acid Contactor T-301 pH Controller
AR-301*	Acid Contactor T-301 pH Recorder

Corrosion/Erosion Probes

CC-301	Corrosion Coupon on C-301 Inlet
CC-302	Corrosion Coupon on C-302 Inlet
CC-303	Corrosion Coupon on C-303 Inlet

Flow Instruments

FT-301	Flow Transmitter on Acid Centrate
FI-301	Flow Indicator on Acid Centrate
FR-301*	Flow Recorder on Acid Centrate
FAL-301*	Flow Alarm (Low) on Acid Centrate
FT-302	Flow Transmitter on Wash Water
FR-302*	Flow Recorder on Wash Water
FAL-302*	Flow Alarm (Low) on Wash Water
FIC-302*	Flow Indicator Controller on Wash Water
FV-302	Flow Control Valve on Wash Water

Level Instruments

LT-301	Tank T-301 Level Transmitter
LIC-301*	Tank T-301 Level Indicator Controller
LSH-301*	Tank T-301 Level Switch (High)
LAH-301*	Tank T-301 Level Alarm (High)
LSL-301*	Tank T-301 Level Switch (Low)
LAL-301*	Tank T-301 Level Alarm (Low)

* Computer/Data Logger Function

Instrument No.DescriptionLevel Instruments (Continued)

LT-302	Tank T-302 Level Transmitter
LIC-302*	Tank T-302 Level Indicator Controller
LSH-302*	Tank T-302 Level Switch (High)
LAH-302*	Tank T-302 Level Alarm (High)
LSL-302*	Tank T-302 Level Switch (Low)
LAL-302*	Tank T-302 Level Alarm (Low)
LT-303	Tank T-303 Level Transmitter
LIC-303*	Tank T-303 Level Indicator Controller
LSH-303*	Tank T-303 Level Switch (High)
LAH-303*	Tank T-303 Level Alarm (High)
LSL-303*	Tank T-303 Level Switch (Low)
LAL-303*	Tank T-303 Level Alarm (Low)

Pressure Instruments

PI-301	Tank T-301 Pressure Indicator
PI-302	Pump P-302 Discharge Pressure Indicator
PI-303	Pump P-303 Discharge Pressure Indicator
PI-304	Tank T-302 Pressure Indicator
PI-305	Pump P-304 Discharge Pressure Indicator
PI-306	Pump P-305 Discharge Pressure Indicator
PI-307	Tank T-303 Pressure Indicator
PI-308	Pump P-306 Discharge Pressure Indicator
PI-309	Pump P-307 Discharge Pressure Indicator

* Computer/Data Logger Function

Instrument No.DescriptionRelief Valves

RV-301	Relief Valve on Tank T-301
RV-302	Relief Valve on Pump P-302 Discharge
RV-303	Relief Valve on Pump P-303 Discharge
RV-304	Relief Valve on Tank T-302
RV-305	Relief Valve on Pump P-304 Discharge
RV-306	Relief Valve on Pump P-305 Discharge
RV-307	Relief Valve on Tank T-303
RV-308	Relief Valve on Pump P-306 Discharge
RV-309	Relief Valve on Pump P-307 Discharge
RV-310	Relief Valve on Centrifuge C-301
RV-311	Relief Valve on Centrifuge C-302
RV-312	Relief Valve on Centrifuge C-303
RV-313	Relief Valve on Tank T-304

Speed Control

SC-301	Speed Control on Pump P-302
SC-302	Speed Control on Pump P-304
SC-303	Speed Control on Pump P-306

Temperature Instruments

TE-301	Tank T-301 Temperature Measuring Element
TIC-301	Tank T-301 Temperature Indicator Controller
TE-302	Tank T-302 Temperature Measuring Element
TIC-302	Tank T-302 Temperature Indicator Controller
TE-303	Tank T-303 Temperature Measuring Element
TIC-303	Tank T-303 Temperature Indicator Controller
TE-304	Tank T-301 Temperature Measuring Element
TR-304*	Tank T-301 Temperature Recorder
TE-305	Tank T-302 Temperature Measuring Element
TR-305*	Tank T-302 Temperature Recorder
TE-306	Tank T-303 Temperature Measuring Element
TR-306*	Tank T-303 Temperature Recorder

* Computer/Data Logger Function

SECTION 400: CAUSTIC REGENERATION

<u>Instrument No.</u>	<u>Description</u>
<u>Corrosion/Erosion Coupons</u>	
CC-401	Tank T-401 Inlet
CC-402	Tank T-402 Inlet
CC-403	Centrifuge C-401 Inlet
CC-404	Inlet to Filters F-401, F-402
CC-405	Tank T-405 Inlet
CC-406	Filters F-403, F-404 Outlet

Level Instruments

LT-401	Tank T-401 Level Transmitter
LIC-401*	Tank T-401 Level Indicator/Controller
LAH-401*	Tank T-401 Level Alarm (High)
LAL-401*	Tank T-401 Level Alarm (Low)
LSH-401*	Tank T-401 Level Switch (High)
LSL-401*	Tank T-401 Level Switch (Low)
LT-402	Tank T-402 Level Transmitter
LIC-402*	Tank T-402 Level Indicator/Controller
LAH-402*	Tank T-402 Level Alarm (High)
LAL-402*	Tank T-402 Level Alarm (Low)
LSH-402*	Tank T-402 Level Switch (High)
LSL-402*	Tank T-402 Level Switch (Low)
LT-403	Tank T-403 Level Transmitter
LAH-403*	Tank T-403 Level Alarm (High)
LAL-403*	Tank T-403 Level Alarm (Low)
LT-404	Tank T-404 Level Transmitter
LIC-404*	Tank T-404 Level Indicator/Controller
LAH-404*	Tank T-404 Level Alarm (High)
LAL-404*	Tank T-404 Level Alarm (Low)

* Computer/Data Logger Function

Instrument No.DescriptionLevel Instruments (Continued)

LSH-404*	Tank T-404 Level Switch (High)
LSL-404*	Tank T-404 Level Switch (Low)
LT-405	Tank T-405 Level Transmitter
LIC-405*	Tank T-405 Level Indicator/Controller
LAH-405 (setting 1)*	Tank T-405 Level Alarm (High)
LAH-405 (setting 2)*	Tank T-405 Level Alarm (High-High)
LAL-405 (setting 1)*	Tank T-405 Level Alarm (Low)
LAL-405 (setting 2)*	Tank T-405 Level Alarm (Low-Low)
LV-405	Tank T-405 Level Control Valve
LT-406	Tank T-406 Level Transmitter
LI-406*	Tank T-406 Level Indicator
LAL-406*	Tank T-406 Level Alarm (Low)

Pressure Instruments

PI-401	Pump P-401 Discharge Pressure Indicator
PI-402	Pump P-402 Discharge Pressure Indicator
PI-403	Pump P-403 Discharge Pressure Indicator
PI-404	Pump P-404 Discharge Pressure Indicator
PI-405	Bag Filter F-403 Pressure Indicator
PI-406	Bag Filter F-404 Pressure Indicator
PI-407	Bag Filter F-401 Pressure Indicator
PI-408	Bag Filter F-402 Pressure Indicator
PDT-401	F-401, F-402 Differential Pressure Transmitter
PDIR-401*	F-401, F-402 Differential Pressure Indicator/ Recorder
PDAH-401*	F-401, F-402 Differential Pressure Alarm (High)
PDT-402	F-403, F-404 Differential Pressure Transmitter
PDIR-402*	F-403, F-404 Differential Pressure Indicator/ Recorder
PDAH-402*	F-403, F-404 Differential Pressure Alarm (High)

Relief Valves

RV-401	Relief Valve on Tank T-401
RV-402	Relief Valve on Pump P-401
RV-403	Relief Valve on Tank T-402
RV-404	Relief Valve on Pump P-402
RV-405	Relief Valve on Tank T-404
RV-406	Relief Valve on Pump P-403

* Computer/Data Logger Function

Instrument No.DescriptionRelief Valves (Continued)

RV-407	Relief Valve on Tank T-403
RV-408	Relief Valve on Tank T-405
RV-409	Relief Valve on Pump P-404
RV-410	Relief Valve on Tank T-406
RV-411	Relief Valve on Drum D-401
RV-412	Relief Valve on Drum D-402

Speed Control

SC-401	Speed Control on Pump P-401
SC-402	Speed Control on Pump P-402
SC-403	Speed Control on Pump P-403
SC-404	Speed Control on Pump P-404

Temperature Instruments

TE-401	Thermocouple in Tank T-401
TIC-401	Temperature Indicator/Controller for Tank T-401
TE-402	Thermocouple in Tank T-402
TIC-402	Temperature Indicator/Controller for Tank T-402
TE-403	Not Used
TE-404	Thermocouple in Tank T-404
TIC-404	Temperature Indicator/Controller for Tank T-404
TE-405	Thermocouple in Tank T-405
TIC-405	Temperature Indicator/Controller for Tank T-405
TE-406	Thermocouple in Tank T-406
TE-407	Tank T-401 Temperature Measuring Element
TR-407*	Tank T-401 Temperature Recorder
TE-408	Tank T-402 Temperature Measuring Element
TR-408*	Tank T-402 Temperature Recorder
TE-409	Tank T-404 Temperature Measuring Element
TR-409*	Tank T-404 Temperature Recorder

* Computer/Data Logger Function

Instrument No.

Description

Temperature Instruments (Continued)

TE-410	Tank T-403 Temperature Measuring Element
TR-410*	Tank T-403 Temperature Recorder
TE-411	Tank T-405 Temperature Measuring Element
TR-411*	Tank T-405 Temperature Recorder

* Computer/Data Logger Function

SECTION 500: CAUSTIC EVAPORATION SYSTEM

<u>Instrument No.</u>	<u>Description</u>
<u>Corrosion/Erosion Probes</u>	
CC-501	Corrosion Coupon in Molten Caustic
<u>Control Valves</u>	
TCV-504	Evaporator Overhead Temperature Control Valve (on water addition inlet)
<u>Flow Instruments</u>	
FE-501	Flow Measuring Element on Aqueous Caustic Feed
FT-501	Flow Transmitter on Aqueous Caustic Feed
FIC-501*	Flow Indicator Controller on Aqueous Caustic Feed
FR-501*	Flow Recorder on Aqueous Caustic Feed
FT-502	Flow Transmitter on Water Addition
FR-502*	Flow Recorder on Water Addition
FI-503	Flow Indicator on CW to E-501
FI-504	Flow Indicator on CW to E-502
FI-505	Flow Indicator on CW to Flaker
FT-506	Flow Transmitter on Evaporator Condensate
FR-506*	Flow Recorder on Evaporator Condensate
FI-507	Flow Indicator on GN ₂ to Hot Oil Relief System Catch Drum

* Computer/Data Logger Function

Instrument No.DescriptionLevel Instruments

LT-501	Molten Caustic Storage Drum Level Transmitter
LIC-501*	Molten Caustic Storage Drum Level Indicator Controller
LR-501*	Molten Caustic Storage Drum Level Recorder
LIC-502*	V-501, Level Indicator Controller
LT-502	V-501, Level Transmitter
LR-502*	V-501, Level Recorder
LAHH-502*	V-501, Level Alarm (High High)
LSH-502*	V-501, Level Switch (High)
LSL-502*	V-501, Level Switch (Low)
LV-502	V-501, Level Control Valve

Pressure Instruments

PI-501	Aqueous Caustic to Evaporator Pressure Indicator
PI-502	N ₂ to Flaker Pressure Indicator
PI-503	Flaker Enclosure Pressure Indicator
PI-505	P-502 Discharge Pressure Indicator
PI-506	Hot Oil Circulation Pump Suction Pressure
PI-507	Hot Oil Circulation Pump Discharge Pressure
PRV-501	Hot Oil Expansion Tank Back Pressure Regulator
PRV-502	GN ₂ to Expansion Tank Pressure Regulator

Relief Valves

RV-501	Flaker Enclosure Relief Valve
RV-503	Hot Oil Liquid Circulation Line Relief Valve
RV-504	Hot Oil Expansion Tank Relief Valve
RV-505	GN ₂ to Expansion Tank Relief Valve
RV-506	Hot Oil Circulation Pump Suction Bypass Relief Valve

Speed Control

SC-501*	P-501 Speed (stroke) Controller
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* Computer/Data Logger Function

Instrument No.DescriptionTemperature Instruments

TE-501	Oil Heater Outlet Temperature Measuring Element
TIC-501	Oil Heater Outlet Temperature Indicator Controller
TE-502	Not Used
TT-503	Oil from Evaporator Temperature Transmitter
TR-503*	Oil from Evaporator Temperature Recorder
TE-504A	Evaporator Overhead Temperature Measuring Element
TE-504B	Evaporator Overhead Temperature Measuring Element
TIC-504	Evaporator Overhead Temperature Indicator Controller
TR-504*	Evaporator Overhead Temperature Recorder
TT-504	Evaporator Overhead Temperature Transmitter
TE-505	CW to E-501 Temperature Measuring Element
TR-505*	CW to E-501 Temperature Recorder
TE-506	Vapor/Liquid from E-501 Temperature Measuring Element
TR-506*	Vapor/Liquid from E-501 Temperature Recorder
TE-507	CW from E-501 Temperature Measuring Element
TR-507*	CW from E-501 Temperature Recorder
TE-508	CW from E-502 Temperature Measuring Element
TR-508*	CW from E-502 Temperature Recorder
TE-509	Vapor from E-502 Temperature Measuring Element
TR-509*	Vapor from E-502 Temperature Recorder
TE-510	CW to E-502 Temperature Measuring Element
TR-510*	CW to E-502 Temperature Recorder
TE-511	CW from Flaker Temperature Measuring Element
TR-511*	CW from Flaker Temperature Recorder

* Computer/Data Logger Function

Instrument No.DescriptionTemperature Instruments (Cont'd)

TE-512	Molten Caustic Storage Drum Temperature Measuring Element
TR-512*	Molten Caustic Storage Drum Temperature Recorder
TAL-512*	Molten Caustic Storage Drum Temperature Alarm (Low)
TE-513	Overhead Condenser E-501 Inlet Temperature Element
TR-513*	Overhead Condenser E-501 Inlet Temperature Recorder
TT-514	Hot Oil to Evaporator Temperature Transmitter
TR-514*	Hot Oil to Evaporator Temperature Recorder
TT-515	Aqueous Caustic to Evaporator Temperature Transmitter
TR-515*	Aqueous Caustic to Evaporator Temperature Recorder
TE-516	P-501 Pump Head Hot Oil Bath Temperature Measuring Element
TSH-516	Hot Oil System High Temperature Switch
TE-517	Molten Caustic Drum Discharge Line Skin Temperature Measuring Element
TE-518	Molten Caustic Line to Flaker Skin Temperature Measuring Element
TE-519	Molten Caustic Line to Metering Pump Skin Temperature Measuring Element
TE-521	Molten Caustic Line to Kiln Skin Temperature Measuring Element

* Computer/Data Logger Function

SECTION 600: WASTE WATER TREATMENT

<u>Instrument No.</u>	<u>Description</u>
<u>Analytical Instruments</u>	
AE/AT-601	Lime Contactor T-602 Outlet pH Monitor
AIC-601	Lime Contactor T-602 Outlet pH Controller
AR-601*	Lime Contactor T-602 Outlet pH Recorder
<u>Corrosion/Erosion Coupons</u>	
CC-601	Inlet to Filter F-601
CC-602	Inlet to Filter F-602
CC-603	Tank T-601
CC-604	Tank T-602
<u>Level Instruments</u>	
LT-601	Tank T-601 Level Transmitter
LIC-601*	Tank T-601 Level Indicator/Transmitter
LAH-601*	Tank T-601 Level Alarm (High)
LAL-601*	Tank T-601 Level Alarm (Low)
LSH-601*	Tank T-601 Level Switch (High)
LSL-601*	Tank T-601 Level Switch (Low)
LT-602	Tank T-602 Level Transmitter
LIC-602*	Tank T-602 Level Indicator/Transmitter
LAH-602*	Tank T-602 Level Alarm (High)
LAL-602*	Tank T-602 Level Alarm (Low)
LSH-602*	Tank T-602 Level Switch (High)
LSL-602*	Tank T-602 Level Switch (Low)
<u>Pressure Instruments</u>	
PI-601	Pump P-601 Discharge Pressure Indicator
PI-602	Bag Filter F-601A Pressure Indicator
PI-603	Bag Filter F-601B Pressure Indicator
PI-604	Pump P-602 Discharge Pressure Indicator
PI-605	Bag Filter F-602A Pressure Indicator
PI-606	Bag Filter F-602B Pressure Indicator
PDIR-601*	Filter F-601 Differential Pressure Indicator/ Recorder
PDAH-601*	Filter F-601 Differential Pressure Alarm (High)
PDT-601	Filter F-601 Differential Pressure Transmitter

* Computer/Data Logger Function

Instrument No.DescriptionPressure Instruments (Continued)

PDIR-602*	Filter F-602 Differential Pressure Indicator/ Recorder
PDAH-602*	Filter F-602 Differential Pressure Indicator/ Recorder
PDT-602	Filter F-602 Differential Pressure Transmitter

Relief Valves

RV-601	Relief Valve on Pump P-601
RV-602	Relief Valve on Pump P-602

Speed Control

SC-601	Speed Control on Pump P-601
SC-602	Speed Control on Pump P-602

Temperature Instruments

TE-601	Thermocouple in Tank T-601
TIC-601	Temperature Indicator/Controller for Tank T-601
TE-602	Thermocouple in Tank T-602
TIC-602	Temperature Indicator/Controller for Tank T-602
TE-603	Tank T-601 Temperature Element
TR-603*	Tank T-601 Temperature Recorder
TE-604	Tank T-602 Temperature Element
TR-604*	Tank T-602 Temperature Recorder

* Computer/Data Logger Function

APPENDIX B

Engineering Test Directive

MCL Process
Rotary Kiln Section

Page 1 of 4

Test Run Number

Coal Type

Date/Time Run Commenced

Scheduled Run Duration, Hrs

Feed Caustic/Coal Ratio

Caustic Makeup, NaOH/KOH Ratio

Caustic Feed, Flake _____ or Molten _____

Coal Feed Rate to Kiln, Lbs/Hr

Kiln Hopper Pneumatic Batch Loading System:

Coal Transfer

<u>Date</u>	<u>Time</u>	<u>Weight of</u> <u>BN-101</u>	<u>Coal</u> <u>Transferred</u>
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Caustic Flake Transfer

<u>Date</u>	<u>Time</u>	<u>Weight of</u> <u>BN-102</u>	<u>Caustic</u> <u>Transferred</u>
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Kiln Control Settings

Page 2 of 4

Rotational Speed, RPM

Inclination, Inches/Ft

Auger Speed, RPM Clockwise/Counterclockwise

Furnace Temperatures

Zone #1, TIC 101, °F

Zone #2, TIC 102, °F

Zone #3, TIC 103, °F

Zone #4, TIC 104, °F

CW Flow to Quench Section, FR106, GPM

Pressure, PIC 101, in H₂O

Blanket Nitrogen Flow, FR107, SCFM

Auxiliary Equipment Settings

Kiln Offgas Scrubbing System:

CW Flow to E-102, FI103, GPM

Solution Circulation Rate, FR102, GPM

Solution Level in T-102, LG102, Inches(or Gallons)

Kiln Product Discharge Valves:

Timer Setting (Cycles/Minute)

Kiln Product Dissolve System:

Level in T-101, LIC101, % Full

CW Flow to E-101, FR105, GPM

Solution Circulation Flow, FR104, GPM

Tank Temperature, TIC110, °F

Rotary Kiln Section

Page 3 of 4

Auxiliary Equipment Settings (Cont'd)

Pneumatic Conveying System:

ΔP for Bag Shaking _____

Sampling Schedule

Sample Size/Frequency

Coal _____

Caustic Flake _____

Kiln Product _____

Kiln Offgas _____

Kiln Vent Gas _____

T-101 Circulating Solution _____

T-102 Circulating Solution _____

Data to be Logged Manually:

Instrument No.

Location

Recording Frequency

TI106 Kiln Solids from Cooling Section _____

FI103 CW to E-102 _____

PI103 P-102 Discharge _____

PI104 P-103 Discharge _____

PI106 GN₂ Makeup _____

PI107 N₂ to BN-102 _____

PI108 N₂ to BN-101 _____

PI109 Baghouse Outlet _____

PI111 Baghouse P _____

Rotary Kiln Section

Page 4 of 4

Log of Events:

Date

Time

Comments/Observations

Engineering Test Directive

MCL Process
Water Washing Section

Page 1 of 5

Test Run Number

Date/Time Run Commenced

Scheduled Run Durations, Hrs

Coal in Feed, Lbs/Hr

Wash Water/Coal Ratio

Feed Caustic Concentration

Wash Water Flow, FIC 202, Lbs/Hr

Separation Equipment Settings:

Vacuum Filter, F-201
Vacuum Level, In. Hg Vacuum
Drum Speed, RPM

Vacuum Filter, F-202
Vacuum Level, In. Hg Vacuum
Drum Speed, RPM

Centrifuge, C-201
Bowl Speed, RPM
Backdrive Setting, % of Full Range

Centrifuge, C-202
Bowl Speed, RPM
Backdrive Setting, % of Full Range

Centrifuge, C-203
Bowl Speed, RPM
Backdrive Setting, % of Full Range

Centrifuge, C-204
Bowl Speed, RPM
Backdrive Setting, % of Full Range

Centrifuge, C-205
Bowl Speed, RPM
Backdrive Setting, % of Full Range

Tank Settings:

Page 2 of 5

Slurry Tank, T-206
Level, LIC 206, % Full
Temperature, TIC 203, °F

Slurry Tank, T-208
Level, LIC 207, % Full
Temperature, TIC 204, °F

Slurry Tank, T-209
Level, LIC 208, % Full
Temperature, TIC 205, °F

Slurry Tank, T-210
Level, LIC 209, % Full
Temperature, TIC 206, °F

Slurry Tank, T-211
Level, LIC 210, % Full
Temperature, TIC 207, °F

Water Washing Section

Page 3 of 5

Sampling Schedule:

Sampling Size/Frequency

F-201 Feed

F-201 Filtrate Upstream of Polishing Filter

F-201 Filtrate Downstream of Polishing Filter

F-201 Cake

F-202 Feed

F-202 Filtrate

F-202 Wash Filtrate

F-202 Cake

C-201 Feed

C-201 Centrate Upstream of Polishing Filter

C-201 Centrate Downstream of Polishing Filter

C-202 Feed

C-202 Centrate

C-202 Cake

C-203 Feed

C-203 Centrate

C-203 Cake

C-204 Feed

C-204 Centrate

C-204 Cake

C-205 Feed

C-205 Centrate

C-205 Cake

Water Washing Section

Page 4 of 5

Data to be Logged Manually:

<u>Instrument No.</u>	<u>Location</u>	<u>Recording Frequency</u>
PI233	F-201 Case	_____
PI237	N ₂ Blowback to F-201	_____
PI205	P-202 Discharge	_____
PI225	F-203A Inlet	_____
PI226	F-203B Inlet	_____
PI207	P-203 Discharge	_____
PI236	F-202 Case	_____
PI238	N ₂ Blowback to F-202	_____
PI208	P-204 Discharge	_____
PI209	P-205 Discharge	_____
PI211	P-207 Discharge	_____
PI228	P-217 Discharge	_____
PI212	P-208 Discharge	_____
PI239	F-204 Inlet	_____
PI214	P-209 Discharge	_____
PI215	P-210 Discharge	_____
PI217	P-211 Discharge	_____
PI218	P-212 Discharge	_____
PI220	P-213 Discharge	_____
PI221	P-214 Discharge	_____
PI223	P-215 Discharge	_____
PI224	P-216 Discharge	_____

Water Washing Section

Page 5 of 5

Log of Events:

Date

Time

Comments/Observations

Engineering Test Directive

MCL Process
Acid Washing System

Page 1 Of 3

Test Run Number

Date/Time Run Commenced

Scheduled Run Duration

Coal in Feed, Lbs/Hr

Wash Water/Coal Ratio

Wash Water Flow, FIC 302, Lbs/Hr

Centrifuge C-301

Bowl Speed, RPM

Backdrive Setting, % of Full Range

Centrifuge C-302

Bowl Speed, RPM

Backdrive Setting, % of Full Range

Centrifuge C-303

Bowl Speed, RPM

Backdrive Setting, % of Full Range

Tank, T-301

Level, LIC 301, % Full

Temperature, TIC 301, °F

pH, AIC 301

Tank, T-302

Level, LIC 302, % Full

Temperature, TIC 302, °F

Tank, T-303

Level, LIC 302, % Full

Temperature, TIC 303, °F

Acid Washing Section

Page 2 of 3

Sample Schedule:

Coal/Caustic to T-301

Feed to C-301

Centrate from C-301

Cake from C-301

Feed to C-302

Centrate from C-302

Cake from C-302

Feed to C-303

Centrate from C-303

Cake from C-303

Sample Size/Frequency

Data to be Logged Manually:

Instrument No.

Location

Recording Frequency

PI302	P-302 Discharge
PI303	P-303 Discharge
PI305	P-304 Discharge
PI306	P-305 Discharge
PI308	P-306 Discharge
PI309	P-307 Discharge
W301	Weight of Coal Product

Acid Washing Section

Page 3 of 3

Log of Events:

Date

Time

Comments/Observations

Engineering Test Directive
MCL Process
Caustic Regeneration Section

Page 1 Of 4

Test Run Number

Date/Time Run Commenced

Scheduled Run Duration, Hrs

Aqueous Caustic Feed Concentration, Nominal, %

Aqueous Caustic Feed Rate, FR 201, GPH

Regeneration Reactor #1, T-401:

Temperature, TIC 401, °F

Level, LIC 401, % Full

Lime Feeder, FD 401, Flow, Lbs/Hr

Regeneration Reactor #2, T-402:

Temperature, TIC 402, °F

Level, LIC 402, % Full

Centrifuge, C-401:

Bowl Speed, RPM

Backdrive Setting, % of Full Range

Centrate Tank, T-404:

Level, LIC 404, % Full

Temperature, TIC 404, °F

Regenerated Caustic Storage Tank, T-405:

Level, LIC 405, % Full

Temperature, TIC 405, F

Caustic Feed Tank:

Level, LI 406, % Full

NaOH/KOH Ratio

Sampling Schedule:Sample Size/Frequency

Caustic Feed

T-401 Effluent

T-402 Effluent

Centrate

Centrifuge Cake

Filtered Centrate

Bag Filter F-401, F-402 Cake

T-405 Effluent

Bag Filter F-403, F-404 Cake

Evaporator Feed

Data to be Logged Manually:Instrument No.LocationRecording Frequency

PI401

P-401 Discharge

PI402

P-402 Discharge

Lime Feeder, FD-401, Hopper Level

Mineral Matter Storage Drum, D-401, Level

Caustic Regeneration Section

Page 3 of 4

Data to be Logged Manually: (Cont'd)

<u>Instrument No.</u>	<u>Location</u>	<u>Recording Frequency</u>
PI403	P-403 Discharge	_____
PI407	F-401 Inlet	_____
PI408	F-402 Inlet	_____
PI404	P-404 Discharge	_____
PI405	F-403 Inlet	_____
PI406	F-404 Inlet	_____

Caustic Regeneration Section

Page 4 of 4

Log of Events:

Date

Time

Comments/Observations

Engineering Test Directive

MCL Process
Caustic Evaporation Section

Page 1 of 4

Test Run Number _____

Date/Time Run Commenced _____

Aqueous Caustic Feed Concentration, Wt% Caustic _____

Scheduled Run Duration, Hrs _____

NaOH/KOH Ratio in Feed _____

Anhydrous Caustic Product Recycled to Kiln: Flake _____ or Molten _____

For Flaked Product, Caustic Feed Rate, FIC 501, Lb/Hr _____

For Molten Product, Caustic Product Rate, SC 501, Lb/Hr _____

Aqueous Caustic Feed Temperature, TR 515, °F _____

Anhydrous Caustic Product Temperature, TIC 504, °F _____

Hot Oil System:

Oil Temperature to Evaporator, TIC 501, °F _____

Oil Temperature from Evaporator (50°F T minimum >TIC 504), TR 503, °F _____

For Molten Caustic Product:

Molten Storage Tank Level, LIC 501, % Full _____

Molten Caustic Storage Temperature, TR 512, °F _____

Hot Oil in Metering Pump, P-501, Head Reservoir, TE 516, °F _____

For Flaked Caustic Product:

Flaker Drum Speed, RPM _____

CW Flow to Drum, FI 505, GPM _____

Evaporator Overhead Condensing System:

CW to E-501, FI 503, GPM

Chilled Water to E-502, FI 504, GPM

Level in V-501, LIC 502, % Full

Caustic Line Heaters Controller Settings, °F

TC 6

TC 7

TC 8

TC 9

TC 10

Sampling Schedule

Sample Size/Frequency

Aqueous Caustic Feed

Flaked Caustic Product

or

Molten Caustic Product

Condensate

Vent Gas

Flaked Product Log

<u>Date</u>	<u>Time</u>	<u>Weight of Collection Drum</u>	<u>Flake Collected</u>
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Caustic Evaporation Section

Page 3 of 4

Data to be Logged Manually:

<u>Instrument No.</u>	<u>Location</u>	<u>Recording Frequency</u>
PI506	Hot Oil Circulating Pump Suction Pressure	_____
PI507	Hot Oil Circulating Pump Discharge Pressure	_____
LG504	Hot Oil Expansion Tank Level	_____
PI501	Evaporator Inlet Pressure	_____
FI503	CW to E-501	_____
FI504	CW to E-502	_____
FI505	CW to Flaker Drum	_____
PI502	GN ₂ to Flaker Enclosure	_____
PI503	Flaker Enclosure	_____
PI505	P-502 Discharge Pressure	_____
TE516	Hot Oil Reservoir for P-501 Pump Head	_____
TE517	Skin Temp. of Molten Caustic Line from T-501	_____
TE518	Skin Temp. of Molten Caustic Line to Flaker	_____
TE519	Skin Temp. of Molten Caustic Line to P-501	_____
TE521	Skin Temp. of Molten Caustic Line to Kiln	_____

Caustic Evaporation Section

Page 4 of 4

Log of Events:

Date

Time

Comments/Observations

Engineering Test Directive

MCL Process
Waste Water Treating Section

Page 1 Of 3

Test Run Number

Date/Time Run Commenced

Scheduled Run Duration, Hrs

Waste Water Flow to T-610, FR 301, Lbs/Hr

Fe₂O₃ Addition to T-601, FD 601, Lbs/Hr

Level in T-601, LIC 601, % Full (Residence Time = _____ Hrs)

Temperature in T-601, TIC 601, °F

pH in T-602

Level in T-602, LIC 602, % Full (Residence Time = _____ Hrs)

Temperature in T-602, TIC 602, °F

Sampling Schedule

Sample Size/Frequency

Waste Water to T-601

Effluent from T-601

Effluent from F-601

Effluent from T-602

Cake from F-601

Cake from F-602

Waste Water Treatment Section

Page 2 of 3

Data to be Logged Manually:

<u>Instrument No.</u>	<u>Location</u>	<u>Recording Frequency</u>
PI601	P-601 Discharge	_____
PI602	F-601A Inlet	_____
PI603	F-601B Inlet	_____
PI604	P-602 Discharge	_____
PI605	F-602A Inlet	_____
PI606	F-602B Inlet	_____
	Fe ₂ O ₃ Level in FD-601 Hopper	_____
	CaO Level in FD-602 Hopper	_____

Waste Water Treatment Section

Page 3 of 3

Log of Events:

Date

Time

Comments/Observations

Appendix C - References

A.1 DRAWINGS

- Six line schematics (PID-101 through PID-601) of the piping and instrumentation

A.2 MANUALS

- Capistrano Test Site Manual
- Six MCL operating procedures (for Sections 100 through 600)
- Final Design Package, Molten-Caustic-Leaching System Integration, Rev. 2, October 16, 1987
- Molten-Caustic-Leaching System Integration Test Plan, 26 September 1988, K534.3.88-038
- Molten-Caustic-Leaching System Integration Corrosion/Erosion Plan, 26 September 1988, K534.3.88-037
- Safety Hazard Analysis Report, Molten Caustic Leaching Process, 9 September 1988, K534.3.87-037
- Vendor's manuals supplied with equipment
- Health and Safety COR Manual

APPENDIX B
SAMPLING AND ANALYSIS PLAN

MCL SAMPLING AND ANALYSIS PLAN

During operation of the MCL Plant, it is necessary to sample the various operating modules (filters, centrifuges, etc.) to assist in on-line decisions for optimizing processing parameters (temperature, flow, etc.) and to subsequently perform analyses on process gas, liquid and solid streams to: 1) assess the effects of operating conditions on coal sulfur and ash removal and on equipment performance; 2) evaluate the validity of the preliminary process models and to develop improved process models. Although many sampling points are available in the MCL plant (Tables 1 and 2), it is not practical to attempt to take samples at all locations during the course of an MCL run. As such, sampling will be done in accordance with the requirements for a particular MCL run. The sampling frequency as shown in Tables 1 and 2 is suggested when samples are taken at the respective locations.

1. SAMPLING

Three types of samples will be taken during MCL operation, i.e., gas, liquid and solid. Sampling will be conducted by a designated test technician cognizant of the unit operation. Required personnel protection during sampling of liquids and solids is rubber gloves, laboratory coat and face shield. Gas sampling does not require rubber gloves or face shield, however safety glasses (goggles) should be worn. A description of the gas, liquid, and solid sampling procedures is as follows.

1.1 GAS SAMPLES

Gas samples will be taken at valve VT-1026 on the kiln exhaust line. Since the system does not have a by-pass stream such that gas cylinders can be used for pass-through sampling, samples will be taken by attaching an evacuated gas cylinder to a "T" in the exhaust line. After the cylinder is attached to the "T", the cylinder inlet valve will be opened,

TABLE 1. SOLID SAMPLES

RUN IDENTIFICATION _____ DATE _____ START TIME _____

Solid Samples				RUN TIME-----) (HOURS)											
	Location	Frequency	Valve No.												
Feed Coal		Once/Coal (Dup anal)													
Caustic Flake	Evaporator	8hrs		8	16	24	32	40	48	56	64	72	80	88	96
Kiln Output	Kiln	2hrs	SP 102	2	4	6	8	10	12	14	16	18	20	22	24
				62	64	66	68	70	72	74	76	78	80	82	84
										86	88	90	92	94	96
										98	100	102	104	106	108
										110	112	114	116	118	120
(Water Wash System)															
Filter Cake F201	1st Vac Filter	4hrs	SC F1	4	8	12	16	20	24	28	32	36	40	44	48
										52	56	60	64	68	72
										76	80	84	88	92	96
										100	104	108	112	116	120
Filter Cake F202	2nd Vac Filter	Once/run	SC F2												
Cake C201	Centrifuge	4hrs Composite	SC 201	4	8	12	16	20	24	28	32	36	40	44	48
										52	56	60	64	68	72
										76	80	84	88	92	96
										100	104	108	112	116	120
Cake C202	Centrifuge	4hrs Composite	SC 202	4	8	12	16	20	24	28	32	36	40	44	48
										52	56	60	64	68	72
										76	80	84	88	92	96
										100	104	108	112	116	120
Cake C203	Centrifuge	4hrs Composite	SC 203	4	8	12	16	20	24	28	32	36	40	44	48
										52	56	60	64	68	72
										76	80	84	88	92	96
										100	104	108	112	116	120
Cake C204	Centrifuge	4hrs Composite	SC 204	4	8	12	16	20	24	28	32	36	40	44	48
										52	56	60	64	68	72
										76	80	84	88	92	96
										100	104	108	112	116	120
Cake C205	Centrifuge	4hrs Composite	SC 205	4	8	12	16	20	24	28	32	36	40	44	48
										52	56	60	64	68	72
										76	80	84	88	92	96
										100	104	108	112	116	120
(Acid Wash System)															
Cake Composite C301	Centrifuge	8hrs Composite	SC 301	8	16	24	32	40	48	56	64	72	80	88	96
Cake Composite C302	Centrifuge	8hrs Composite	SC 302	8	16	24	32	40	48	56	64	72	80	88	96
Cake Composite C303	Centrifuge	8hrs Composite	SC 303	8	16	24	32	40	48	56	64	72	80	88	96
Product Coal	1hr Bags	Sample each bag (8 hr composite)		8	16	24	32	40	48	56	64	72	80	88	96
Caustic Solid Reject C401	Centrifuge	16hrs 48hrs	SC 401	16			32	40	48	64			80	96	
								48						96	
Bag Filter		Once/run	(Send to Ames)												
Waste Water Bag Filter	F602A/602B	Once/run	(Send to Ames)												

TABLE 2. LIQUID SAMPLES

RUN IDENTIFICATION _____				DATE _____				START TIME _____																									
LIQUID SAMPLES				RUN TIME -----) (HOURS)																													
	Location	Frequency	Valve No.																														
Scrubber Water	Kiln	8hrs		8	16	24	32	40	48	56	64	72	80	88	96	104	112	120															
1st Vac Filter	F201	4hrs	VF 2037	4	8	12	16	20	24	28	32	36	40	44	48	52	56	60	64	68	72	76	80	84	88	92	96	100	104	108	112	116	120
Before Filter	Polishing	4hrs	VP 2024	4	8	12	16	20	24	28	32	36	40	44	48	52	56	60	64	68	72	76	80	84	88	92	96	100	104	108	112	116	120
2nd Vac Filter	F202	4hrs	VP 2042	4	8	12	16	20	24	28	32	36	40	44	48	52	56	60	64	68	72	76	80	84	88	92	96	100	104	108	112	116	120
Centrate	C201	4hrs	VP 2006	4	8	12	16	20	24	28	32	36	40	44	48	52	56	60	64	68	72	76	80	84	88	92	96	100	104	108	112	116	120
Centrate	C202	4hrs	VP 2103	4	8	12	16	20	24	28	32	36	40	44	48	52	56	60	64	68	72	76	80	84	88	92	96	100	104	108	112	116	120
Centrate	C203	4hrs	VP 2122	4	8	12	16	20	24	28	32	36	40	44	48	52	56	60	64	68	72	76	80	84	88	92	96	100	104	108	112	116	120
Centrate	C204	4hrs	VP 2142	4	8	12	16	20	24	28	32	36	40	44	48	52	56	60	64	68	72	76	80	84	88	92	96	100	104	108	112	116	120
Centrate	C205	4hrs	VP 2162	4	8	12	16	20	24	28	32	36	40	44	48	52	56	60	64	68	72	76	80	84	88	92	96	100	104	108	112	116	120
(Acid Washing System)																																	
Centrate	C301	8hrs	VP 3033 or VP 3041	8	16	24				32	40	48	56	64	72	80	88	96	104	112	120												
Centrate	C302	8hrs	VP 3052	8	16	24				32	40	48	56	64	72	80	88	96	104	112	120												
Centrate	C303	8hrs	VP 3072	8	16	24				32	40	48	56	64	72	80	88	96	104	112	120												
(Caustic Regeneration System)																																	
Effluent Filtered	Reactor T 401	Once/run	VP 4011																														
Effluent Filtered	Reactor T 402	8hrs	VP 4021	8	16	24				32	40	48	56	64	72	80	88	96	104	112	120												
(Waste Water Treatment System)																																	
Effluent after Bag Filters	Iron oxide Contactor	24	VF 6017			24						48			72			96														120	
Effluent	Lime Contactor	24	VF 6027			24						48			72			96														120	

followed by opening the valve on the "T". The cylinder inlet valve should be closed immediately thereafter. The valve on the "T" should be closed next, before removing the gas sampling cylinder from the line.

1.2 LIQUID SAMPLES

Liquid samples consist of several types: 1) wash water effluent; 2) coal slurries; 3) caustic regeneration media; and 4) process water for disposal. Certain of these streams are classified as "live" in that they must be specially prepared to ensure proper measurements of the ionic species. In particular, those streams containing concentrated caustic from both the wash train and regeneration system must be specially treated. Standard liquid or slurry samples (non-live) may be stored without special handling except that caustic samples must be taken rapidly and sealed in their respective containers to prevent exposure to atmospheric carbon dioxide.

All of the liquid samples will be taken through the lever actuated ball valves so designated at each unit operation by valve number. Samples will be obtained by opening the valve and allowing about 25 mL of liquid to pass into a sample container. A 250 mL plastic bottle with screw lid will be used to collect approximately 200 mL of liquid (filled to about 3/4 full). The valve should be closed immediately, and the bottle capped and labeled to identify the sample.

1.3 SOLID SAMPLES

Four types of solid samples are to be taken: 1) coal; 2) regeneration sludge; 3) waste water treatment residue; and 4) caustic flake. These solids require no special handling except for those which need to be analyzed for caustic and carbonate concentration. Such samples should be immediately sealed in their respective containers until analysis.

Solid coal samples are taken by entrapping falling wet solids from the centrifuges and vacuum filters in a sample cavity contained in a half-open

tubular rod which extends through the enclosed section above the various reslurry tanks. A sample bottle is affixed to the end of the rod. The rod contains a solid bar (O.D. slightly smaller than rod I.D.) which fills the inner volume. In normal operation the tubular rod is rotated such that the cavity opening is downward, and the sample bottle is inverted. When samples are desired, the bottle is rotated upright with the tubular rod opening facing upward. The solid rod (plunger) is withdrawn exposing an open cavity to solids flow. After a predetermined time (collection time TBD during shakedown), the rod is pushed into the tube which forces the collected solids into the sample bottle. The procedure is repeated until sufficient sample is obtained. The bottle is removed and immediately capped and labeled. The sample bottle is replaced on the unit with another bottle for use at the next sampling period. The sampling cavity and the empty bottle are rotated to an inverted position.

Regeneration sludge and waste water treatment residue must be removed from bag filters. The bag filters are installed in pairs, with one filter on-line and one off-line at all times. The bag containing the solids is removed from the off-line filter, and 50 grams of wet solids should be sampled and placed in plastic bottles with screw lids. Rubber gloves must be worn when handling these residues. Caustic flake samples will be obtained from the flaker catch drum, bottled and labeled.

2. SAMPLE IDENTIFICATION

The test technician will mark each designated sampling time on the sampling frequency sheet to verify that the sample was taken (Table 3). Identification of the sample will be written on the furnished label (Figure 1) and affixed to the sample container with clear tape. Sample identification will include run identification, valve number or sample location, plus the date and time the sample was taken (Figure 1).

Table 3 Designation of Sampling Frequency (Example)

RUN IDENTIFICATION SD-1 DATE XX CCT START TIME CCC

Solid Samples	Location	Frequency	Valve No.	RUN TIME-----> (HOURS)

Feed Coal		Once/Coal (Dup anal)		2	4	6	8	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40	42	44	46					
Caustic Flake	Evaporator	8hrs		62	64	66	68	70	72	74	76	78	80	82	84	86	88	90	92	94	96	98	100	102	104	106					
Kiln Output	Kiln	2hrs	SP 102																												

Table 3b Verification of Sampling

Caustic Flake	Evaporator	8hrs																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																	
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Figure 1 Sample Identification

RUN I.D.	SD-1		
DATE	XX Oct	TIME	0400
RUN TIME	4		
VALVE/LOCATION	SP 102 (Kiln)		

3. SAMPLE ANALYSIS

A variety of analyses is required to satisfy the requirements for in-process information and provide data for material balances and model studies. Table 4 lists those analyses which may be considered for each process module. Selection of the specific analyses will be made by the test engineer. The analytical schemes for liquids and cake solids are shown in Figures 2 and 3.

4. SAMPLE SELECTION

The designated sampling locations (Tables 1 and 2) will be determined at the direction of the test engineer. The test planning directive will include facsimiles of Tables 1 and 2 with the sampling frequency designated on the respective sampling time lines (e.g., Table 3). The sampling locations and frequency described below are based on the need to assess plant performance and to perform material balances.

4.1 ROTARY KILN REACTOR

- a) Sample feed coal and analyze for ash content, sulfur content (total and organic sulfur), moisture content, mineral matter, volatiles, heat content and size fraction (duplicate analysis for each coal type).
- b) Sample and analyze caustic flake from caustic evaporator for CO_3 , humics, water,, Na and K (every eight hours). Also, analyze for mineral matter (once per week).
- c) Sample and analyze kiln gas (VT-1026) for H_2 and CH_4 (every eight hours), and for CO_2 and H_2O (once per run).
- d) Sample and analyze scrubber water (VP-1031) for TOC (every eight hours). Also, need to know the total amount of scrubber water to compute the amount of TOC generated per unit time.
- e) Sample kiln output (SP-102) and analyze for coal sulfur, ash/volatiles and CO_3 (every two hours).

4.2 WATER WASHING SYSTEM

- a) Sample and analyze filtrate from first vacuum filter (VF-2037, collected after the polishing filters and representative of the feed to the caustic regeneration system) for coal fines, Na, K,

TABLE 4. LIQUID AND SOLID SAMPLES ANALYSES

LIQUIDS

LOCATION	Frequency	%S	TOC	Alk	CO3	Na	K	Ult Coal		Humic	Density	Visc	Si	Al	Fe	Ca	pH	SO4
								Min	Fine									
Kiln	8hr		X															
1st Vac Filter F-201 VP-2037	4hr	X						X	X	X								
Before Polishing VP-2024	4hr								X		X	X						
2nd Vac Filter F-202	once per run	X		X	X			X	X	X	X	X						
Centrifuge (C-201)	once per run			X	X				X		X	X						
Centrifuge (C-202)	once per run			X	X				X		X	X						
Centrifuge (C-203)	once per run			X	X				X		X	X						
Centrifuge (C-204)	once per run			X	X				X		X	X						
Centrifuge (C-205)	once per run			X	X				X		X	X						
Centrifuge (C-301)	once per run					X	X		X		X	X	X	X	X		X	X
Centrifuge (C-302)	once per run					X	X		X		X	X						
Centrifuge (C-303)	once per run					X	X				X							
Reactor T-401	once per run	X		X	X								X	X				
Reactor T-402	8hr	X		X	X			X		X								
Iron Contactor	8hr	X				X	X								X		X	X
Lime Contactor	8hr	X				X	X								X	X	X	X

TABLE 4. LIQUID AND SOLID SAMPLES ANALYSES (CONTINUED)

SOLIDS

LOCATION	Frequency	Short Prox	Ult Min	%S	Ash	Alk	CO3	Part Size	Na	K	Ult Ash	Moist	Humics	Density	Dry Solid	Volatiles
-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
Feed Coal	2 for each coal type	X	X	X				X			X	X				
Evaporator	once per week					X	X		X	X	X	X	X			
Kiln	2hr			X	X	X	X					X			X	X
1st Vac Filter F-201	4hr					X	X					X			X	
2nd Vac Filter F-202	once per run					X	X					X			X	
Centrifuge (C-201)	once per run							X				X		X		
Centrifuge (C-202)	once per run							X				X		X		
Centrifuge (C-203)	once per run							X				X		X		
Centrifuge (C-204)	once per run							X				X		X		
Centrifuge (C-205)	once per run							X				X		X		
Centrifuge (C-301)	once per run							X				X		X		
Centrifuge (C-302)	once per run							X				X		X		
Centrifuge (C-303)	once per run							X				X		X		
1hr bags	once per run	X	X	X	X							X				
Centrifuge (C-401)	10hr											X				
Centrifuge (C-401)	48hr					X	X				X					
Iron Contactor (F-601AB)	send to Ames															
Lime Contactor (F-602AB)	send to Ames															

FIGURE 2

ANALYTICAL SCHEME FOR LIQUID SAMPLES

FIGURE 3
ANALYTICAL SCHEME FOR KILN SAMPLES

*TBD

Al, Si, Fe, CO₃, S and humic acid/phenolics (every four hours during run and eight hours between runs). Also, determine the density and viscosity (using the Brookfield viscometer) of the filtrate at the operating temperature (once per run).

- b) Sample and analyze filter cake from first vacuum filter for cake moisture and caustic content (every four hours during run and eight hours between runs). Also, determine the average mass of dry suspended solids per unit mass of cake (once per run).
- c) Sample and analyze filtrate from second vacuum filter (VP-2041) for coal fines, Na, K, Al, Si, Fe, CO₃, S and humic acid/phenolics, and determine the filtrate density and viscosity at the operating temperature (once per run).
- d) Sample and analyze filter cake from second vacuum filter (PV-202) for cake moisture and caustic content, and determine the average mass of dry solids per unit mass of cake (once per run).
- e) Sample centrates from the five centrifuges (VP-2086, VP-2103, VP-2122, VP-2142, VP-2162), every four hours during run and eight hours between runs. Analyze the composite sample from each run for fines and caustic content, and determine the viscosity (Brookfield) and density at the operating temperatures (once per run).
- f) Sample cakes from the five centrifuges, every four hours during run and eight hours between runs. Analyze the composite sample from each run for moisture content. Also, determine the density of the cake solid (particles containing interstitial liquid) and the size distribution of these particles (once per run).

4.3 ACID WASHING SYSTEM

- a) Sample and analyze centrate from the first centrifuge (VP-3033 or VP-3041) for fines, Na, K, Fe, pH, SO₄ (every eight hours) and Al and Si (once per run), and determine the viscosity (Brookfield) and density at the operating temperature (once per run). This centrate is the feed to waste water treatment system.
- b) Sample and analyze centrates from the second and third centrifuges (VP-3052 and VP-3072) for fines, Na, K (every eight hours), and determine the viscosity (Brookfield) and density at the operating temperature (once per run).
- c) Sample cakes from the three centrifuges (once every eight hours). Analyze the composite sample from each run for moisture content. Also, determine the density of the cake solid (particles containing interstitial liquid) and the size distribution of these particles (once per run).

- d) Place product coal in one hour sacks. Analyze each sack for S, ash, volatiles, moisture. Perform ultimate and proximate analysis on product coal samples (once per run).

4.4 CAUSTIC REGENERATION SYSTEM

- a) Sample effluent from reactor tank T-401 (VP-4011, once per run). Filter the sample and analyze the filtrate for Si, Al, S and CO_3 .
- b) Sample the effluent from reactor tank T-402 (VP-4021, every eight hours). Filter the sample. Analyze the filtrate for Si, Al, Fe, S, CO_3 , and humic acid/phenolics. Alternately, sample the centrate from the centrifuge C-401 (VP-4031) and analyze for fines, Si, Al, Fe, S, CO_3 , and humic acid/phenolics. The two samples should provide essentially the same information concerning reactor performance.
- c) Sample and analyze cake from centrifuge C-401 for moisture content (every 16 hours). Analyze sample for mineral matter and CO_3 (every 48 hours). This sample represents the caustic solid reject.

4.5 CAUSTIC EVAPORATION SYSTEM

- a) Sample and analyze caustic flake produced (refer to rotary kiln reactor).

4.6 WASTE WATER TREATMENT SYSTEM

- a) Sample and analyze effluent from the iron oxide contactor (VF-6017, collected after the bag filters) for Na, K, Fe, SO_4 , and pH.
- b) Sample and analyze effluent from the lime contractor (VF-6027, collected after the bag filters) for Na, K, Fe, Ca, SO_4 , and pH.
- c) Collect samples from bag filters F601A/601B and F602A/602B (once per run). Send these samples to Ames Laboratory for analysis to determine the composition of the double salts and other compounds formed.

APPENDIX C
CORROSION/EROSION PLAN

FSCM NO. 11982

TITLE

MOLTEN - CAUSTIC - LEACHING
SYSTEM INTEGRATION
CORROSION/EROSION PLAN

DATE 26 September 1988

NO. K534.3.88-037

REV. 1

SUPERSEDING: _____

PREPARED BY: J.L. Anastasi / E.M. Barrish

J.L. Anastasi/E.M. Barrish

APPROVAL SIGNATURES:

John F. Jones 11-7-88
DATE

John F. Jones, Manager
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DATE

DATE

Robert A. Meyers 11-8-88
DATE

Robert A. Meyers
Program Manager

DATE

DATE

1.0 INTRODUCTION

This plan defines the objectives, requirements, and schedule for the corrosion/erosion monitoring of the Molten Caustic Leaching (MCL) plant that was constructed at TRW's Capistrano Chemical Facility. The test program is being conducted for the Department of Energy (DOE), Pittsburgh Energy Technology Center under contract number DE-AC22-86-PC91257. The new MCL installation allows for the continuous and simultaneous operation of all MCL unit operations enabling the collection of operating data and equipment performance data.

The MCL process involves the treatment of coal with molten caustic (NaOH and KOH) followed by washing with water and acid to remove the sulfur and mineral matter from the coal. The coal product obtained from the MCL process meets New Source Performance Standards (NSPS) for sulfur and contains very low levels of coal derived mineral matter. The MCL process also involves the regeneration and reuse of the caustic media to minimize the costs associated with make up chemicals, and the treatment of process waste water to allow for potential reuse in the washing operations.

The following unit operations make up the MCL process:

- Rotary Kiln Reactor System
- Water Washing System
- Acid Washing System
- Caustic Regeneration System
- Caustic Evaporation/Flaking System
- Water Treatment System

This document presents the corrosion/erosion monitoring plan for the MCL plant. The metallurgy specified for the evaporator, filters, centrifuges, pumps, and tanks (nickel, Inconel, Alloy 20, 316 stainless

steel, plastic) should be suitable for the corrosive/erosive nature of caustic, sulfuric acid, dissolved sulfides, carbonates, organics, mineral matter, precipitated solids, and suspended, abrasive coal solids. The equipment should be capable of operating (without significant performance degradation) for the duration of testing. Wear plugs, test coupons, and electronic probes will be used to collect dynamic test data on corrosion and erosion in the plant.

2.0 OBJECTIVES

The overall program objectives are to design, install, and operate a steady state molten-caustic-leaching system to gather the data necessary for assessing the operability, economics and environmental acceptability of the MCL process for desulfurizing and demineralizing coal in the presence of caustic. The 20 lb/hr of coal MCL unit will be operated on an integrated, continuous basis with heat (in a manner that simulates commercial practice) to provide operational information for various caustic, acid, and coal streams, and data on process parameters such as reaction rates, filtration rates, cake moisture, caustic losses, and corrosion/erosion rates.

Due to the corrosive nature of the caustic and acid streams present in the MCL plant as well as the abrasive nature of the suspended solids, expensive alloys (nickel, Inconel, Alloy 20) were used in several key areas of the plant (evaporator, kiln, acid contactor) in order to assure the operation of the plant for the duration of testing and the safety of the operators. Stainless steel and plastic were used in the less critical areas of the MCL plant. In key areas, wear plugs, coupons and electronic probes were placed to gather corrosion/erosion data that will be useful in future scale-ups to commercial sized equipment. The data collected will provide the basis for proper selection of materials of constructions for future plants.

3.0 TEST HARDWARE CONFIGURATION

The MCL system is depicted in Figure 1. The 20 lb/hr of coal facility consists of six major subsystems as follows:

- a rotary kiln reactor section (Section 100)
- a multi-stage filtration/centrifugation water wash section for separating caustic from coal (Section 200)
- a multi-stage centrifugation acid wash section for removing the residual caustic (Section 300)
- a caustic regeneration section for removing sulfide and mineral matter from spent caustic (Section 400)
- an evaporation section for concentrating the regenerated caustic (Section 500)
- a water treatment section for removing ions and salts from process water for recycle or discharge (Section 600).

Wear plugs, coupons and/or electronic probes were installed in all six major plant subsystems. Table 1 lists the instrument number, the location, the material, and the type (wear plug, coupon or electronic probe) of each of the 23 corrosion/erosion instruments installed in the MCL plant. Table 2 presents a numerical breakdown of the instruments according to type and material. Most of the instruments are of 316 stainless steel since most of the major equipment in the MCL plant are of 316 stainless steel. Expensive alloy materials (nickel, Inconel, and Alloy 20) are used only in those areas of the plant (evaporator, kiln, acid contactor) where high temperatures or other conditions warrant it. Generally, corrosion/erosion for process streams at temperatures below 250°F are of less concern because suitable plastics materials with good chemical resistance are available for use in process equipment.

3.1 WEAR PLUGS AND TEST COUPONS

The wear plugs and test coupons are 1/4 inch wide by 1/16 inch thick by 2 inches long inserts of nickel, Inconel, Alloy 20, and stainless steel fastened to alloy holders which are screwed into the lines at elbows.

REVISIONS			
LTR	DESCRIPTION	DATE	APPROVED

Figure 1

ORIGINATOR		DATE	MCL Conceptual Flow Diagram	
J. L. Amstutz		11/20/86		
Rev 1		12/10/86		
SIZE		PSCH NO.	CFD-1	
B		11982		
SCALE			SHEET 1 OF	
MLO				

TABLE 1. CORROSION/EROSION COUPONS AND PROBES

INSTRUMENT NO.	LOCATION	MATERIAL	COUPON/PROBE
----------------	----------	----------	--------------

SECTION 100 - KILN REACTOR SECTION

CC-101	P-101 DISCHARGE	INCONEL	COUPON
CC-102	TANK T-101	INCONEL	PROBE

SECTION 200 - WATER WASHING SECTION

CC-203	TANK T-203	316 SS	PROBE
CC-204	F-202 INLET	316 SS	WEAR PLUG
CC-205	C-201 INLET	316 SS	WEAR PLUG
CC-206	C-202 INLET	316 SS	WEAR PLUG
CC-207	C-203 INLET	316 SS	WEAR PLUG
CC-208	C-204 INLET	316 SS	WEAR PLUG
CC-209	C-205 INLET	316 SS	WEAR PLUG

SECTION 300 - ACID WASHING SECTION

CC-301	C-301 INLET	ALLOY 20	WEAR PLUG
CC-302	C-302 INLET	ALLOY 20	WEAR PLUG
CC-303	C-303 INLET	316 SS	WEAR PLUG

SECTION 400 - CAUSTIC REGENERATION SECTION

CC-401	T-401 INLET	316 SS	COUPON
CC-402	T-402 INLET	316 SS	WEAR PLUG
CC-403	C-401 INLET	316 SS	WEAR PLUG
CC-404	F-401/2 INLET	316 SS	COUPON
CC-405	T-405 INLET	316 SS	COUPON
CC-406	F-403/4 OUTLET	316 SS	COUPON

SECTION 500 - EVAPORATION SECTION

CC-501	P-501 OUTLET	NICKEL	WEAR PLUG
--------	--------------	--------	-----------

SECTION 600 - WASTE TREATMENT SECTION

CC-601	F-601 INLET	ALLOY 20	COUPON
CC-602	F-602 INLET	316 SS	COUPON
CC-603	TANK T-601	ALLOY 20	PROBE
CC-604	TANK T-602	316 SS	PROBE

TABLE 2. CORROSION/EROSION COUPON/PROBE DISTRIBUTION

	INCONEL	NICKEL	ALLOY 20	316 SS	TOTAL
PROBES	1	-	1	2	4
COUPONS	1	-	1	5	7
WEAR PLUGS	-	1	2	9	14
TOTAL	2	1	4	16	23

Wear plugs will be placed in lines downstream of pumps where wear (erosion) will most likely occur. Test coupons will be placed in such locations as tank inlets where, due to the low flow rates, only corrosion (not erosion) will occur. These wear plugs and test coupons will be weighed and measured before they are inserted into the lines. At the end of the test program, these wear plugs and coupons will be removed and examined. The rates of corrosion/erosion will be determined by weight and dimensional losses. Additional information on the form of localized attack (e.g., pitting, stress corrosion cracking, crevice corrosion, and intergranular corrosion) will be obtained by metallographic examination of the wear plugs and test coupons.

3.2 ELECTRONIC PROBES

TRW has an electronic corrosometer for which several probes were purchased for the MCL plant. The probes have about 1 1/2 inch long sensing elements exposed to the process fluid and are inserted through a 1/2 inch threaded opening. Because of the size of the electronic probes, they can only be inserted into tanks. (Most of the lines in the MCL plant are 1/2 inch tubing.) The sensing element is a hairpin loop of alloy surrounded by a 1/2 inch diameter cylindrical sheath. The probe is welded to a hermetically sealed electrical connector. The probe is installed so that the alloy sensing element is exposed to the corrosive environment. As corrosion of the sensing element occurs, its cross section is reduced, increasing the electric resistance. Periodically, the corrosometer will be connected to the probe to take a reading. The corrosometer converts the resistance increases into known amounts of metal loss. Instantaneous readings of corrosion/erosion rates will be determined from the changes in resistance. These monitoring devices will provide information on changes in corrosion/erosion rates as process operating conditions vary. Readings of electronic corrosion monitoring devices will be taken at least once during each test run, or more frequently if there are significant changes in process operating conditions.

4.0 REQUIREMENTS

The MCL process equipment has been designed to provide for taking representative samples of the process streams, process data (pressure, flow, temperature, level), and corrosion/erosion data. Wear plugs, test coupons and electronic monitoring probes necessary to acquire corrosion/erosion data have been provided.

TRW has an electronic corrosometer for which several alloy probes were purchased for the MCL plant. Periodically, the corrosometer will be attached to the probes to take readings. The frequency of readings will be specified in the test directives. The data from the corrosometer will be recorded on the places provided on the test directives. Before and after weights of the wear plugs and test coupons will also be recorded.

5.0 TEST REQUIREMENTS

5.1 OPERATING PROCEDURES

The MCL proces is divided into six major unit operations:

- Rotary Kiln Reactor System
- Water Washing System
- Acid Washing System
- Caustic Regeneration System
- Caustic Evaporation/Flaking System
- Water Treatment System

Operating procedures have been written for each of the above six unit operations. The personnel conducting the tests must have a working knowledge of the components and function of each of the unit operations within the overall MCL system, have read and be familiar with each of the six operating procedures and with the Safety Hazard Analysis Report, the Shakedown Test Plan, the Test Plan, and all other pertinent documents (see list of references, Appendix A), and be properly attired at all times.

5.2 TEST SCHEDULES

The MCL shakedown test schedule is presented in the Shakedown Test Plan, K534.3.88-015, and the MCL process test schedule is presented in the Test Plan, K534.3.88-038.

5.3 TEST SEQUENCE/MATRIX

Shakedown testing is planned for 6 weeks and process testing is planned for 10 weeks. The system will not be shutdown between the end of shakedown testing and the beginning of actual test operation, one operation will lead into the other.

5.4 CORROSION/EROSION MONITORING

The wear plugs and test coupons will be weighed and inserted into the lines before shakedown testing begins. There is no way in which the wear plugs and test coupons can be removed from the process circuit and weighed during testing without disrupting operation. Thus the wear plugs and test coupons will not be removed, weighed, or examined until completion of all test operations.

The electronic corrosion monitoring probes, however, do not require removal from the system for readings. Periodically, the corrosometer will be attached to the probes for readings. The frequency of the readings will be specified in the test directives. As a minimum, at least one reading of each probe will be made during each test. If the data indicates that rapid corrosion is taking place, readings will be taken more frequently. The data will be recorded on the places provided on the test directives.

Appendix A - References

A.1 DRAWINGS

- Six line schematics (PID-101 through PID-601) of the piping and instrumentation

A.2 MANUALS

- Capistrano Test Site Manual
- Six MCL operating procedures (for Sections 100 through 600)
- Final Design Package, Molten-Caustic-Leaching System Integration, Rev. 2, October 16, 1987
- Molten-Caustic-Leaching System Integration Shakedown Test Plan, 26 September 1988, K534.3.88-015
- Molten-Caustic-Leaching System Integration Test Plan, 26 September 1988, K534.3.88-038
- Safety Hazard Analysis Report, Molten Caustic Leaching Process, 9 September 1988, K534.3.87-037
- Vendor's manuals supplied with equipment
- Health and Safety COR Manual

APPENDIX D
OPERATING PLAN

OPERATING PLAN, OPERATING SCHEDULE AND TEST MATRIX

The operating plan table shows five potential modes of test circuit operations, 1) a five day week 12-hour shift start-up mode, 2) a seven day week start-up mode, 3) a five day week transition mode, 4) a five day week debugged mode, and 5) a seven day week debugged mode. The forecast efficiencies of these modes are compared, in terms of manpower hours per plant run hour, in column eight of the table.

The basic assumption is that a test plant requires certain amounts of downtime for maintenance or due to malfunction and the amount of downtime can, in time, be reduced to levels approaching that of a production plant with 90% operation factor. In industrial practice, it would be normal to take at least a year of operation in a commercial plant to reach a 90% operation factor. Therefore, it seems apparent that the plant test circuit operation should proceed through phases toward this goal. It is clear that start-up is likely to be most efficient in a five day week 12-hour shift mode rather than the seven day 8-hour shift option. This is due to the large operating crew, required for the later option, which charges even when the plant is shutdown.

Thus, the following sequence is recommended for the current project: 1) a five day week start-up, 2) a five day week transition, and 3) a five day week debugged 90% operation mode. This would complete the current project. Subsequently, TRW can anticipate that the plant would move into a seven day week validated mode which will maximize the data per unit operator hour.

The operations schedule table shows application of this transition series to shakedown and then to operation of the test circuit providing a potential for 950 hours of test circuit operation. At the conclusion of this testing and initiation of further operation, it is likely that the plant can begin to move into the seven day week validated mode of

operation which could be utilized for both data gathering and production of Gravimelt coal for larger scale testing. The dollar figures shown in the table are presented for comparison purposes only, and do not necessarily represent actual dollar costs as in a proposal or project plan.

The test matrix table contains a series of tests designed to fit the 950 hours of potential available operational time. The test matrix shown includes conditions for the reactor, regenerator and caustic wash sections as well as a complete specification of the data base to be obtained during operations. Many of the tests shown will have test matrix subsets which will be performed concurrently in the acid wash section for removal of dissolved materials from the acid wash water or in the regeneration section. The analyses were assigned an average dollar value of \$30 and the costs totalled. Once again, the costs are for comparative purposes only and may not represent actual costs.

This test matrix is aimed at providing: 1) the highest grade compliance coal containing both ultra low sulfur and ultra low ash, and 2) evaluation of the most economic processing conditions as well as production of coal samples which will show the minimum by-product formation. Generally speaking, the conditions shown in the table begin with mild conditions moving quickly to conditions which will give the lowest sulfur and ash product coal and culminating in testing of conditions, which while possibly not providing compliance coal, will provide low sulfur and low ash coal under very economic conditions. These conditions are at significantly lower residence time than specified in the "optimistic" engineering studies performed by SAIC. The product and the process design data obtained under these conditions can be compared directly to other processes now being considered by DOE which also do not provide compliance coal but do provide significant reductions in sulfur.

Code:Opschd

OPERATING SCHEDULE

TASK 4. SHAKEDOWN

	Week					
	1	2	3	4	5	6
Crew, hrs	200	200	300	400	600	200
Supr, hrs	100	100	100	100	100	100
Total	300	300	400	500	700	300
Cum, hrs	300	600	1000	1500	2200	2500
Cum \$, k	19.5	39	65	97.5	143	162.5
Cum Op hrs						

5-man crew single 8 hr shift wks 1 and 2, 10-man crew alternating wks of two 12-hr shifts with wks of two 4-hr shifts. Supervision is a constant 80-hrs per week (Jones full time, sec 1/2 and engineer 1) for rest of shakedown and all of presently funded operation.

TASK 6. OPERATION

Five day week startup mode

Week	1	2	3	4	5	6
	600	200	600	200	600	200
	100	100	100	100	100	100
	700	300	700	300	700	300
	3200	3500	4200	4500	5200	5500
	208	227.5	273	292.5	338	357.5
	96		192		288	

Five day week transition mode

Five day week plant debugged

1	2	3	4	5	6	7
600	600	600	600	600	600	600
100	100	100	100	100	100	100
700	700	700	700	700	700	700
6200	6900	7600	8300	9000	9700	10400
403	448.5	494	539.5	585	630.5	676
376	464	552	652	752	852	952

Code: testhrs

OPERATING PLAN

OPERATIONAL TEST HOURS/COST

Type per 2-wks	Startup hrs	Test hrs	Shutdown hrs	Manpower hrs ops	Manpwr Maint hrs	Total Manpwr hrs	Manpwr hr per run hr	@curr 2500 hr budg
5-day week - (startup mode)	8	96	16	480	320	800	8	300
7-day week - (startup mode)	24	184	32	920	760	1680	9	274
5-day week - (transition mode)	16	176	48	880	320	1200	7	NA
5-day week - (plant debugged)	16	200	24	1000	200	1200	6	NA
7-day week - (plant debugged)	4	288	8	1440	240	1680	6	NA

=====

5-day week (startup mode) = 10 operators (5/shift) 5 days 12-hr shifts followed by one week of half days of maintenance

7-day week (startup mode) = 21 operators (5/shift) 3-shift 8-hr days with sched or unsched maintenance downtime once per week for 2-days for a 70% uptime.

5-day week (transition mode) = 15 operators (5/shift) 8-hr shifts for 5 day weeks with sched maintenance downtime once per week for 24 hrs while shutting down

5-day week (plant debugged) = as above but downtime is once per 2-weeks for an 83% uptime

7-day week(plant debugged) = 21 operators (5/shift) 3-shift 8-hr days with mainly scheduled maintenance downtime once per month for 3-days for a 90% uptime.

@budg
wks

=====

6 Efficient in plant startup

3 Not efficient until expd personnel and debugged plant

Potentially efficient

Potentially most Efficient operation

TEST MATRIX - PAGE 1

MCL CIRCUIT TEST MATRIX AND SCHEDULE

Objectives: 1) test two coals, 2) meet standards with for at least one run with each, 3) make at least two long duration runs at steady state and deliver 500 lbs of each to DOE, 4) perform as many variable evaluations as possible and in conformance with a test matrix, 5) corrosion evaluation, and 6) maximize ratio of product coal to total coal throughput.

TEST	C/COAL	RES TM	REACT T	COAL	Na/K	CAUST INJCT	WASH H2O/COAL	EQUIP CHANGE HRS
1	2/1	2.5	320	Pit	50/50	Flake	5/1	0
2	2/1	2.5	320	Pit	50/50	Flake	5/1	1.5
3	2/1	2.5	390	Pit	50/50	Flake	5/1	4
4	2/1	2.5	390	Pit	50/50	Moltn	5/1	4
5	2/1	2.5	365	Ky	50/50	Flake	5/1	4
6	2/1	2.5	365	Pit	50/50	Flake	5/1	4
7	1/1	5	365	Pit	50/50	Flake	5/1	4
8	1/1	5	365	Pit	50/50	Flake	3/1	4
9	1/1	5	365	Pit	90/10	Flake	3/1	4
10	1/1	5	365	Pit	90/10	Flake	5/1	4
11	1/1	5	320	Pit	90/10	Flake	5/1	4
12	1/1	5	390	Pit	90/10	Flake	5/1	4
13	1/1	2.5	390	Pit	90/10	Flake	5/1	4
14	1/1	2.5	365	Pit	90/10	Flake	5/1	4
15	1/1	2.5	365	Ky	90/10	Flake	5/1	4
16	1/1	5	365	Ky	90/10	Flake	5/1	4
17	2/1	2.5	390	Ky	90/10	Flake	5/1	4
18	2/1	2	390	Pit	90/10	Flake	5/1	4
19	2/1	1.5	390	Pit	90/10	Flake	5/1	4
20	2/1	1	390	Pit	90/10	Flake	5/1	4
21	2/1	1	365	Pit	90/10	Flake	5/1	4
22	2/1	1	340	Pit	90/10	Flake	5/1	4
23	1/1	1	340	Pit	90/10	Flake	2/1	4
24	2/1	1	390	Ky	90/10	Flake	5/1	4
25	2/1	1	365	Ky	90/10	Flake	5/1	4
26	2/1	1	340	Ky	90/10	Flake	5/1	4
27	1/1	1	340	ky	90/10	Flake	2/1	4
28	2/1	2.5	390	Ky	90/10	Flake	5/1	4
29	1/1	5	365	Pit	90/10	Flake	3/1	4

Assumptions 1) steady state for whole system is closely approached at 2X res time of unit chnged as regen ops unaffected by feed, 2) reqs 4 hrs for any eqmt change - temp, res tm, water ratio, caustic ratio, 3) take run samples over 5 hrs at steady state and delivery samples over 20 hrs

** End of current project funding

Notes: 1) each test listed below can include a series of additional tests around the acid wash section and the water treatment section with no additional crew labor

TEST	EQUIP CHANGE	1st COAL		REACH STDY S HRS	RUN PERIOD HRS	CUM COAL		CUM OP TIME HRS	Wash Sect* Analyses Cum No.	Entrl Lab Kiln Opt*	Entrl Lab Prod Coal*	Entrl Lab Regen Sys* CO ₂ , HmMS	Entrl Lab Flakes* CO ₂ , HmMW	Entrl Lab Caus Solid CO ₂ , alk, min	Entrl Lab Kiln Gas H ₂ -CH ₄	Entrl Lab Acid Solnt* Na & Fe
		HRS	OUT			LBS NOT IN RUN	CUM COAL LBS COLLECTED									
1	0	11		22	25	660	500	58	156	63	58	44	22	4	7	7
2	1.5	1.5		3	25	780	1000	89	261	96	89	67	33	6	11	11
3	4	5		10	25	1160	1500	133	390	144	133	100	50	8	17	17
4	4	5		10	5	1540	1600	157	444	170	157	118	59	10	20	20
5	4	10		20	25	2220	2100	216	602	234	216	162	81	14	27	27
6	4	5		10	25	2600	2600	260	649	282	260	195	98	16	33	33
7	4	10		20	5	3280	2700	299	658	324	299	224	112	19	37	37
8	4	3.5		7	5	3570	2800	319	668	345	319	239	119	20	40	40
9	4	10		20	5	4250	2900	358	677	387	358	268	134	22	45	45
10	4	3.5		7	25	4540	3400	397	724	430	397	298	149	25	50	50
11	4	10		20	5	5220	3500	436	733	445	436	327	164	27	55	55
12	4	10		20	5	5900	3600	475	743	485	475	356	178	30	59	59
13	4	5		10	5	6280	3700	499	752	509	499	374	187	31	62	62
14	4	5		10	5	6660	3800	523	761	534	523	392	196	33	65	65
15	4	5		10	5	7040	3900	547	771	558	547	410	205	34	68	68
16	4	10		20	25	7720	4400	606	818	619	606	455	227	38	76	76
17	4	5		10	25	8100	4900	650	864	664	650	488	244	41	81	81
18	4	4		8	5	8420	5000	671	874	685	671	503	252	42	84	84
19	4	3		6	5	8680	5100	689	883	703	689	517	258	43	86	86
20	4	2		4	25	8880	5600	724	930	739	724	543	272	45	91	91
21	4	2		4	5	9080	5700	739	939	754	739	554	277	46	92	92
22	4	2		4	5	9280	5800	754	949	770	754	566	283	47	94	94
23	4	5.5		11	5	9690	5900	780	958	796	780	585	292	49	97	97
24	4	2		4	5	9890	6000	795	968	811	795	596	298	50	99	99
25	4	2		4	5	10090	6100	810	977	826	810	607	304	51	101	101
26	4	2		4	25	10290	6600	845	1024	862	845	633	317	53	106	106
27	4	5.5		11	5	10700	6700	870	1033	888	870	653	326	54	109	109
28	4	5		10	25	11080	7200	914	1080	933	914	686	343	57	114	114
29	4	10		20	5	11760	7300	953	1089	973	953	715	357	60	119	119

Analyses:

- 1) sample 5 separator cakes and liquids each 4 hrs during run and 8 hrs between runs - for first 5 runs, then 1/2 this amount and only during run thereafter
 - 2) analyze for cake moisture*, caustic content*, fines in liquid*, 2) sample kiln output every 2 hrs first 10 runs, then every 8 hrs - analyze for coal S*, ash-vols* and ave once per shift CO3* as check, 3) product coal placed in 1 hr sacks - analyze composite samples for each 2 hrs for S*, ash/vols/moist* and once each run obtain an ultimate and prox anal,
 - 4) sample caustic regen system (in and out pairs) each 8 hrs - analyze for CO3*, Huminics*, Si, Al and S*, 5) sample caustic flakes each 8 hrs - analyze for CO3*, Huminics* and H2O*,
 - 6) sample caustic solid reject every 16 hrs - obtain coal mineral analysis total alk and CO3 each 48 hours,
 - 7) sample kiln gas and scrubber every 8 hrs - analyze H2, CH4, TOC,
 - 8) sample two tanks and two solids in acid water treatment section each 8 hrs
- analyze Na*, Fe* in soln each 8 hrs and obtain coal mineral anal on solid each 48 hrs, obtain ICP elemental suite on section H2O effluent each 24 hrs.

Occasional analyses to support modeling - cake density and viscosity of filtrate in wash sections

TEST MATRIX - PAGE 3

TEST	Prch Anal Prod Coal	Prch Anal Caus Regen	Prch Anal Caus Solid	Prch Anal Kiln Trap	Prch Anal TOC	Prch Anal ICP	Total ALL Anal	Total Cost, \$	REMARKS
	Cum No.	Cum No.	Cum No.	Cum No.	Cum No.	Cum No.	Cum No.	Cum @ \$30	
1	30	15	1	7	2	415	12456		For deliv. H2O only, Shakedown
2	46	22	2	11	4	658	19753		For deliv. W. Ac wsh
3	68	33	3	17	6	984	29519		For deliv. Best SOx
4	80	39	3	20	7	1145	34360		First test of injector, if works, continue to use
5	109	54	5	27	9	1566	46976		Provisional for delivery of sec coal
6	131	65	5	33	11	1809	54268		Provisional for deliv.
7	151	75	6	37	12	1992	59765		Matrix test, long res. time
8	160	80	7	40	13	2088	62654		Matrix test, low wash water
9	180	89	7	45	15	2272	68152		Matrix test, high NaOH caustic
10	200	99	8	50	17	2495	74841		For delivery, complete factorial 5,6,7,8
11	219	109	9	55	18	2651	79521		Matrix test, low temp./long res.
12	239	119	10	59	20	2832	84946		Matrix test, complete temp. parametric set
13	251	125	10	62	21	2946	88392		Matrix test, short res. time
14	263	131	11	65	22	3061	91838		Matrix test, complete factorial 8,10,11,12
15	275	137	11	68	23	3176	95284		Matrix test, duplicates Pgh coal, test 12
16	304	152	13	76	25	3482	104471		For deliv., duplicates Pgh coal, test 8
17	326	163	14	81	27	3723	111680		For deliv., Low SOx, like Pitts test 3
18	337	168	14	84	28	3824	114731		Shorten res time in kiln to min sideprods
19	346	172	14	86	29	3913	117386		Shorten res time in kiln to min sideprods
20	363	181	15	91	30	4114	123407		Short res time for delivery of coal 1
21	371	185	15	92	31	4189	125667		Short res time and low T for coal 1, min sideprods
22	378	189	16	94	31	4264	127926		Mildest conditions for coal 1
23	391	195	16	97	32	4386	131570		Most economic conditions for coal 1
24	398	199	17	99	33	4461	133830		Short residence time coal 2
25	406	202	17	101	34	4536	136089		Short res time and low T coal 2, min sideprods
26	423	211	18	106	35	4737	142111		Mildest conditions for delivery of coal 2
27	436	218	18	109	36	4859	145755		Most economic conditions for coal 2
28	458	229	19	114	38	5099	152964		Repeat of run 17, to verify precision
29	478	238	20	119	40	5280	158388		Repeat of run 9, to verify precision

END

DATE FILMED

10 / 22 / 90