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DEFINING THE MgO PROBLEM AND ITS ECONOMIC IMPACT ON PHOSPHORIC ACID PRODUCTION

Prepared By

Jacobs Engineering Group, Inc
and
A. N. Baumann

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Florida Institute of Phosphate Research
1855 West Main Street
Bartow, Florida 33830
(863) 534-7160
Fax:(863) 534-7165

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FIPR No. 92-01-102

JACOBS ENGINEERING GROUP, INC.
and
A.N. BAUMANN, CONSULTANT

Contract Manager
G. Michael Lloyd, Jr.

FLORIDA INSTITUTE OF PHOSPHATE RESEARCH

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Perspective

Magnesium has been recognized as a problem in phosphoric acid manufacture for a number of years now, but little information has been published that defines the economic penalties associated with the use of high magnesium rock for phosphoric acid production. This report attempts to define the "cost of magnesium."

Since it is well known that future mining in Florida will produce phosphate rock with magnesium contents above those experienced today, the magnesium problem greatly influences both mining and chemical operations. Either processing the rock to remove or reduce the magnesium content and/or learning how to operate the phosphoric acid plants economically when using high magnesium phosphate rock would be acceptable solutions to this problem. Successful utilization of high magnesium phosphate rock would result in a more efficient utilization of a vital Florida mineral resource.

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SECTION 1

1.0 INTRODUCTION

Jacobs Engineering carried out a series of phosphoric acid pilot plant tests to define the MgO problem in phosphate feed rock for the Florida institute of Phosphate Research.

A test matrix of variables was set up by A. N. Baumann, project consultant for the program, as shown in Table 1. In addition, two "experimental" runs 'E' and 'F' were done at the start to establish workable sulfate levels. The tests were run at three levels of MgO in the feeds, 0.65% MgO, 1.23% MgO, and 1.80% MgO on an "as is" basis. Retention was varied at 3, 4 and 5 hours and reactor acid strength was run at 24.5% P_2O_5, 26% P_2O_5, and 27.5% P_2O_5.

The pilot plant tests began in May 1993 and were concluded in January 1994. An addendum run was performed after the 11th test in which the reactor configuration was altered to a two stage system using a post treatment addition of H_2SO_4. This test was an attempt to improve the P_2O_5 recovery and filtration for 1.23% MgO feed up to the performance level of 0.65% MgO rock. The tests, however, suffered mechanical and control problems and indicated only marginal benefits for the sulfuric acid post treatment scheme.

TABLE 1

Also TABLE 3.2.6

Test Design
2 by 3 for MgO Digestion Project

CONSTANTS: Digestion
Soluble Sulfate Range
% Solids: 35% by Weight
Temperature: 80°C

Variable Levels		–	o	+
X1	Rock MgO Content %(1)	**0.6**	**1.2**	**1.8**
X2	Filtrate P_2O_5 Conc.	**24.5**	**26.0**	**27.5**
X3	Retention Time - Hours	**3.0**	**4.0**	**5.0**

Test Designation	Test Run	X1	X2	X3
A	1	-	-	-
B	8	+	-	-
C	6	-	+	-
D	10	+	+	-
E	5	-	-	+
F	11	+	-	+
G	4	-	+	+
H	3	+	+	+
I	12	o	o	o
J	9	o	o	o
K	7	o	o	o
L	2	o	o	o

TEST ORDER

Test Designation	Test Run Order	% MgO(1)	% P_2O_5	Detention Hours
A	1	0.6	24.5	3.0
L	2	1.2	26.0	4.0
H	3	1.8	27.5	5.0
G	4	0.6	27.5	5.0
E	5	0.6	24.5	5.0
C	6	0.6	27.5	3.0
K	7	1.2	26.0	4.0
B	8	1.8	24.5	3.0
J	9	1.2	26.0	4.0
D	10	1.8	27.5	3.0
F	11	1.8	24.5	5.0
I	12 (Deleted)	1.2	26.0	4.0

Determine: Sulfuric Acid Consumption
Filtration Rates
Digestion and Filtration Efficiencies

(1) Actual MgO in rock mixtures was 0.66%, 1.24%, and 1.83% on a dry basis (see Table 3.2.1) and 0.65%, 1.23% and 1.80% on an "as is" basis.

SECTION 2

2.0 SUMMARY

This report covers a series of phosphoric acid pilot plant tests performed to determine the major effects of treating phosphate rocks containing relatively high quantities of MgO, up to 1.8%.

Run Averages are given in Table 2. Average Analyses - USAGES & PRODUCTS, are given in Table 3. Rock analyses are summarized in Table 4.

The tests show, as expected, increased losses and reduced filtration rates as the MgO is increased. Figures 1, 2 and 3 plot total losses, filtration rates and water soluble losses as a function of reactor acid strength at parameters of 0.65%, 1.23% and 1.80% MgO in the phosphate rock.

The higher MgO affects filtration rate, as summarized in Table 5 which is derived from the test data. It shows that for 1.23 MgO, the filtration rates are not appreciably different to a base case of 100 for 0.55% MgO and 27% reactor acid. Probably the effect would not be noticed in terms of capacity but 'the water soluble losses are higher.

The plant would suffer substantially, however, in filter capacity if 1.8% MgO rock were treated. A significant increase in rate is experienced, dropping from 27% acid to 25% reactor acid. This would be about 24% P_2O_5 after dilution across the filter.

TABLE 2

RUN AVERAGES

(Also Table 4.2.1)

	RUN F	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6A	RUN 6B	RUN 7	RUN 8	RUN 9	RUN 10A	RUN 10B	RUN 11A	RUN 11B	ADDENDUM REACTOR1	RUN NO. 1 REACTOR2
TEST CONDITIONS																	
DURATION OF RUN (hours)	95	96	96	96	96	96	108	108	96	96	96	108	108	108	108	110	110
PERIOD AVERAGED (hours)	56-94	62-95	50-95	50-95	50-95	50-95	38-84	86-107	50-95	50-95	50-95	32-86	86-107	49-81	83-107	98-109	98-109
FEED ROCK ANALYSES (1):																	
%P2O5	29.36	31.79	30.58	29.36	31.79	31.79	31.79	31.79	30.58	29.36	30.58	29.36	29.36	29.36	29.36	30.58	30.58
%CaO	45.58	47.40	46.50	45.58	47.40	47.40	47.40	47.40	46.50	45.58	46.50	45.58	45.58	45.58	45.58	46.50	46.50
%MgO	1.80	.65	1.23	1.80	.65	.65	.65	.65	1.23	1.80	1.23	1.80	1.80	1.80	1.80	1.23	1.23
ROCK FEED RATE (gms/hr.)	1084	1048	805	662	646	638	1062	1062	805	1082	805	1106	1106	653	653	1061	1061
OPERATING CONDITIONS																	
RETENTION (hours)	3.03	3.14	4.04	5.13	4.99	4.89	3.04	3.02	4.02	3.00	4.06	2.99	2.88	4.96	4.98	3.06	.97
REACTION VOLUME (m^3/stpd P2O5)	1.13	1.06	1.43	1.83	1.71	1.71	1.04	1.06	1.42	1.10	1.42	1.13	1.11	1.82	1.84	1.10	1.09
PLANT ANALYSES:																	
%H2SO4	2.78	2.72	2.53	2.54	2.49	2.48	2.20	2.74	2.50	2.50	2.60	2.60	2.20	2.53	2.14	2.19	3.09
ACID S.G.	1.322	1.301	1.324	1.355	1.332	1.292	1.328	1.330	1.324	1.315	1.325	1.361	1.361	1.318	1.314	1.319	1.340
%SOLIDS IN SLURRY	34.9	36.1	35.2	35.1	35.0	34.8	35.2	34.9	34.8	35.6	35.5	34.9	33.9	35.0	35.2	34.6	34.6
LAB ANALYSES:																	
%H2SO4	2.67	2.22	2.25	2.49	2.25	2.15	1.65	2.47	2.25	2.23	2.22	2.14	1.64	2.17	1.50	1.70	2.66
%P2O5	24.63	25.27	26.43	27.93	27.59	24.94	27.47	27.52	26.39	24.57	26.05	27.31	27.74	24.79	24.64	25.90	27.18
FILTER TESTS																	
%SOLIDS IN SLURRY	33.5	33.6	33.4	32.9	33.6	32.8	32.7	33.0	32.8	32.7	33.2	32.4	31.0	32.8	32.9		28.9
%SOLIDS IN CAKE	73.5	72.8	75.3	74.3	73.8	76.0	71.8	71.5	74.7	75.1	74.9	71.5	69.0	76.8	75.6		72.7
t GYPSUM/ft^2/day	4.53	4.44	4.64	4.18	4.34	4.63	4.33	4.17	4.47	4.13	4.47	3.47	3.43	4.51	4.51		3.86
t P2O5/ft^2/day	.84	.89	.90	.79	.88	.94	.87	.82	.88	.79	.87	.63	.63	.85	.85		.74
EXTRACTION LOSSES (% of P2O5 fed)																	
CITRATE SOLUBLE	2.93	2.22	2.41	2.53	1.77	1.34	2.64	3.21	2.13	2.67	2.05	3.84	3.86	2.25	3.12	2,82	2.82
CITRATE INSOLUBLE	2.02	1.28	.60	1.55	.75	.20	.26	1.65	.47	.26	.48	2.61	.77	.22	.20	1.03	.38
WATER SOLUBLE	.48	.31	.23	.45	.35	.11	.16	.40	.22	.15	.19	.74	.61	.15	.13	.51	.51
TOTAL	5.43	3.81	3.24	4.53	2.87	1.65	3.06	5.26	2.82	3.08	2.72	7.19	5.24	2.62	3.45	4.36	3.71
P2O5 RECOVERY	94.57%	96.19%	96.76%	95.47%	97.13%	98.35%	96.94%	94.74%	97.18%	96.92%	97.28%	92.81%	94.76%	97.38%	96.55%	95.64%	96.29%

NOTES:

(1) feed rock analyses calculated from analyses of the components
(2) (9000 x 2000)/(646 x 24 x 0.3179 x 0.9713 x 2205)
(3) (9000 x 2000)/(638 x 24 x 0.3179 x 0.9836 x 2205)

TABLE 3

AVERAGE ANALYSES (1)
USAGES AND PRODUCTS

(Also Table 4.2.2)

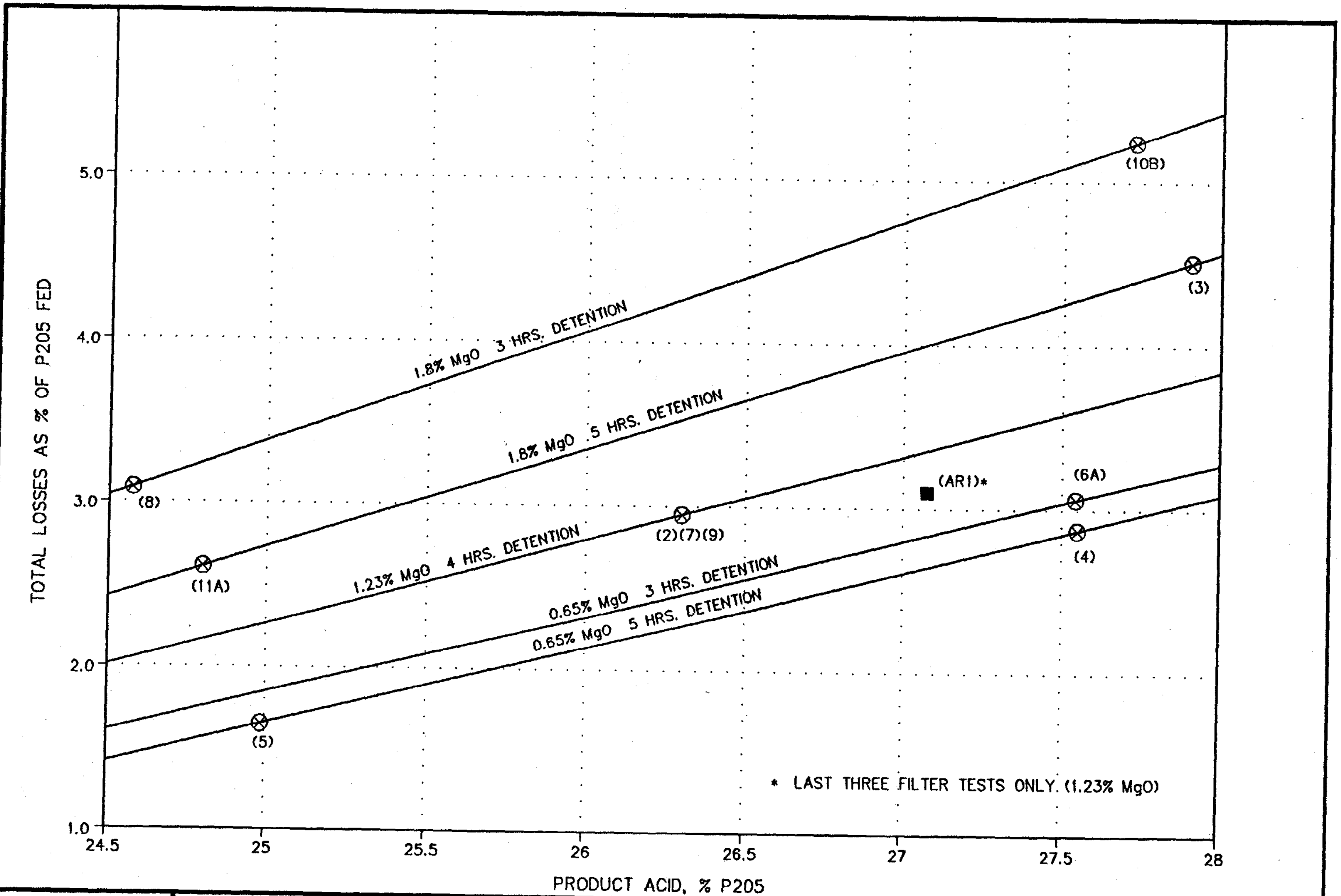
	RUN F	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6A	RUN 6B	RUN 7	RUN 8	RUN 9	RUN 10A	RUN 10B	RUN 11A	RUN 11B	ADDENDUM REACTOR1	RUN NO. 1 REACTOR2
ROCK																	
%P2O5	29.36	31.79	30.58	29.36	31.79	31.79	31.79	31.79	30.58	29.36	30.58	29.36	29.36	29.36	29.36	30.58	30.58
%CaO	45.58	47.40	46.50	45.59	47.40	47.40	47.40	47.40	46.50	45.58	46.50	45.58	45.58	45.58	45.58	46.50	46.50
%SO3	1.18	1.20	1.19	1.18	1.20	1.20	1.20	1.20	1.19	1.18	1.19	1.18	1.18	1.18	1.18	1.19	1.19
%MgO	1.80	.65	1.23	1.80	.65	.65	.65	.65	1.23	1.80	1.23	1.80	1.80	1.80	1.80	1.23	1.23
ACID																	
%P2O5	24.63	25.27	26.43	27.93	27.59	24.94	27.47	27.52	25.39	24.57	26.05	27.31	27.74	24.79	24.64	25.90	27.18
%CaO	.05	.19	.40	.10	.04	.25	.08	.06	.11	.04	.04	.04	.07	.10	.16	.06	.03
%SO3	2.27	2.22	2.07	2.07	2.03	2.02	1.80	2.24	2.04	2.04	2.12	2.12	1.80	2.07	1.75	1.39	2.17
%MgO	1.70	.53	1.10	1.80	.55	.49	.63	.65	1.04	1.52	1.12	1.80	1.82	1.55	1.55	1.13	1.18
GYPSUM																	
%P2O5	1.076	.795	.657	.899	.592	.342	.634	.954	.574	.599	.548	1.405	1.017	.510	.676	.700	.650
%CaO	30.64	31.03	30.58	30.87	30.97	30.91	30.90	30.93	30.88	30.50	30.46	30.18	30.08	30.20	30.19	30.69	31.03
%SO3	43.12	43.62	43.67	43.65	44.43	45.14	44.73	44.61	44.16	43.91	44.31	42.40	42.69	43.48	43.55	43.26	44.42
%MgO	.010	.002	.005	.006	.006	.005	.003	.004	.004	.040	.007	.014	.012	.004	.003	.002	.006
MATERIAL USAGE(2)																	
t H2SO4/t rock fed	.802	.830	.819	.799	.845	.859	.849	.852	.826	.818	.843	.795	.799	.817	.812	.806	.829
t H2SO4/t P2O5 produced	2.888	2.716	2.769	2.850	2.737	2.746	2.755	2.808	2.779	2.872	2.834	2.919	2.871	2.856	2.866	2.743	2.801
t rock/t P2O5 produced	3.602	3.270	3.379	3.567	3.238	3.198	3.245	3.297	3.365	3.513	3.362	3.671	3.594	3.497	3.528	3.401	3.378
kg defoamer/t P2O5 produced(3)	9.177	5.376	5.231	8.714	6.437	5.437	4.714	4.575	6.638	8.474	5.223	8.197	8.141	5.652	6.362	5.634	5.595
PRODUCTS(2)																	
t acid/t P2O5 produced	4.060	3.957	3.784	3.580	3.625	4.010	3.640	3.634	3.789	4.070	3.839	3.662	3.605	4.034	4.058	3.861	3.679
t gypsum/t P2O5 produced	5.352	4.971	5.089	5.256	4.951	4.872	4.968	5.046	5.054	5.245	5.127	5.539	5.438	5.265	5.305	5.146	5.058
MgO DISTRIBUTION (%)																	
ROCK	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
PRODUCT ACID	99.23	99.63	99.39	99.51	98.53	98.78	99.35	99.15	99.49	96.72	99.17	98.84	99.02	99.66	99.75	99.76	99.31
GYPSUM	.77	.37	.61	.49	1.47	1.22	.65	.85	.51	3.28	.83	1.16	.98	.34	.25	.24	.69

NOTES:

(1) averaged over the time period shown in Table 1
(2) calculated from the analyses of the rock, acid, and gypsum by mass balance
(3) calculated from the actual usage

Figure 1

(Also Figure 4.2.3)



PN:	29-H387-00
Prepared By:	DWL/CMP
Date:	12-1-93
Ref:	H387002A

( ) DENOTES TEST RUN NO.

NOTE: ALL TESTS EXCEPT E, F & No.1.

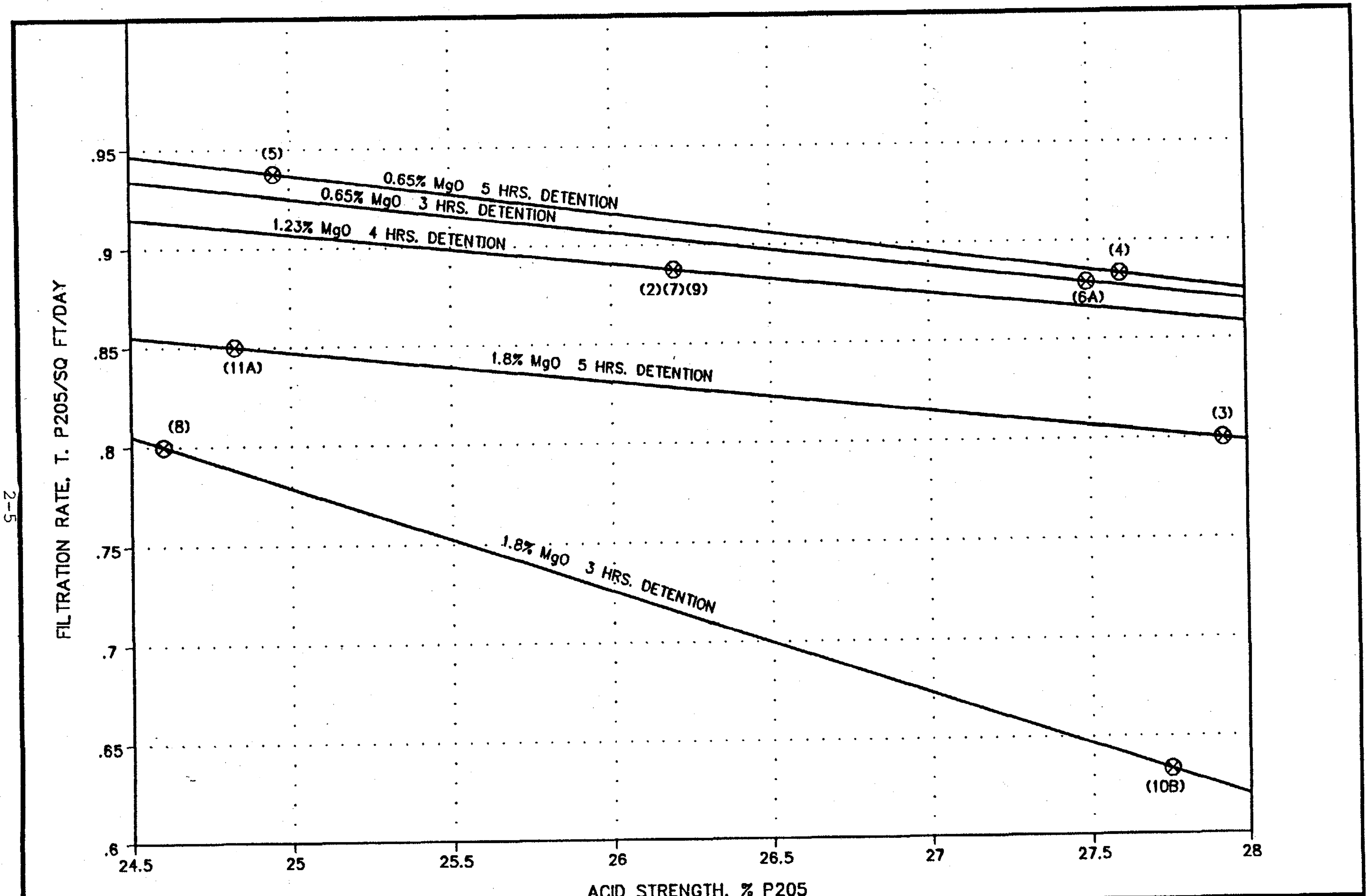
JACOBS ENGINEERING GROUP INC.

LAKELAND FLORIDA

TOTAL LOSSES VS % P205 IN ACID

Figure 2

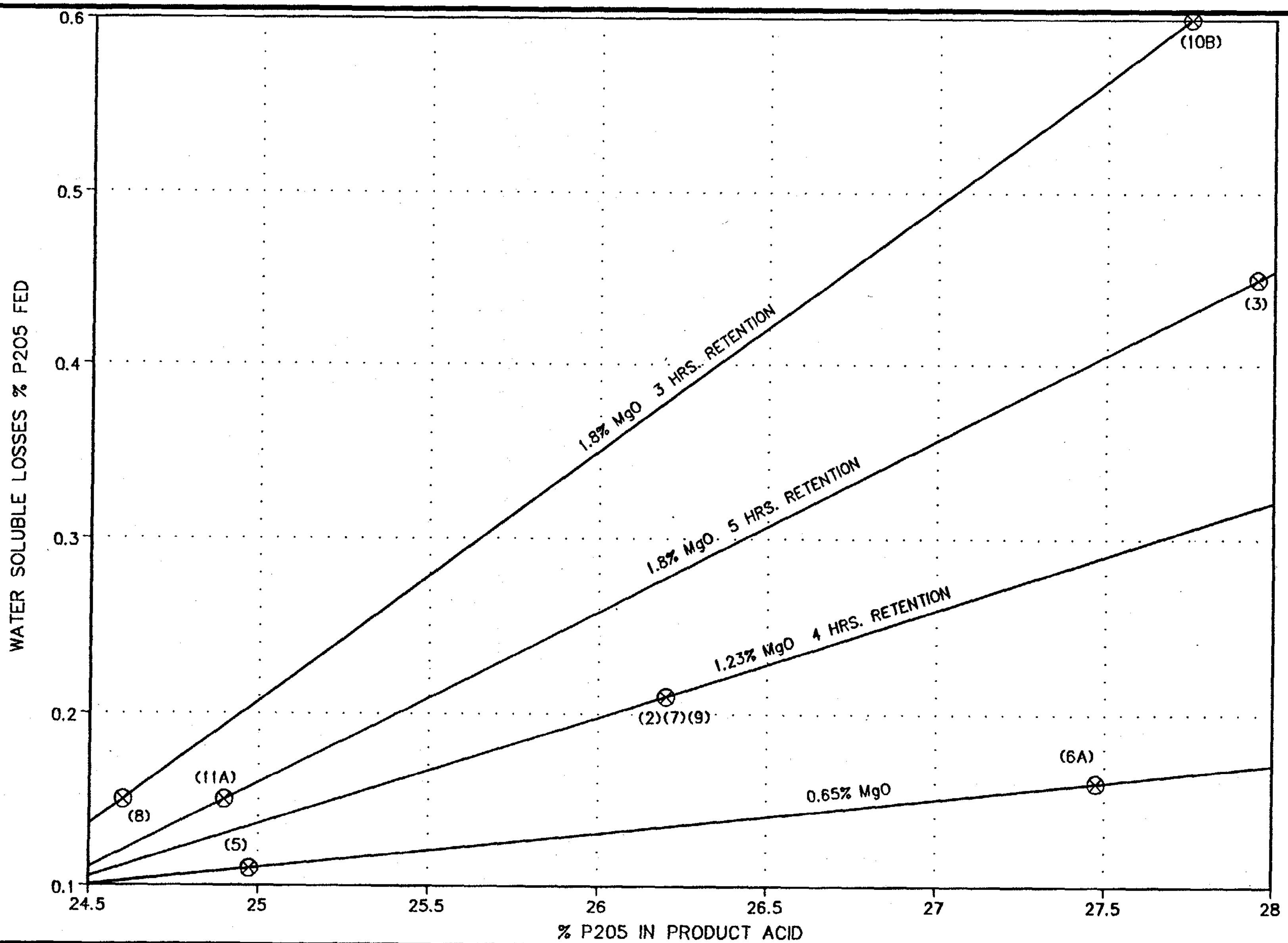
(Also Figure 4.2.4)



PN: 29-H387-00	( ) DENOTES TEST RUN NO.	JE	JACOBS ENGINEERING GROUP INC.	FILTRATION RATES VS ACID % P205
Prepared By: DWL/CMP	NOTE: ALL TESTS EXCEPT E, F & No.1.		LAKELAND FLORIDA	
Date: 12-1-93				
Ref: H387003A				

Figure 3

(Also Figure 4.2.5)



PN: 29-H387-00 Prepared By: DWL/CMP Date: 12-1-93 Ref: H387004A	( ) DENOTES TEST RUN NO.	JE JACOBS ENGINEERING GROUP INC. LAKELAND FLORIDA	WATER SOLUBLE LOSSES VS % P205 IN PRODUCT ACID

TABLE 4

Phosphate Rock Analyses[1]

	Composition, % Dry Basis			
	Low MgO Concentrate	High MgO Pebble	Medium MgO Feed	High MgO Feed
P_2O_5	32.32	23.46	31.06	29.79
CaO	48.13	41.57	47.23	46.26
MgO	0.66	4.75	1.24	1.83
Fe_2O_3	1.35	1.31	1.35	1.34
Al_2O_3	0.95	0.69	9.90	0.87
CaO/P_2O_5	1.49	1.77	1.52	1.55
$I\&A/P_2O_5$	0.071	0.085	0.072	0.074
MER[2]	0.092	0.288	0.112	0.136
Particle Size, %				
+35 mesh	2.39	2.72	2.39	2.44
-200	40.3	59.1	43.8	45.7

(1) Also see Table 3.2.1.

(2) Minor Element Ratio: $\frac{\%I\&A + \%MgO}{\%P_2O_5}$

TABLE 5

Relative Filtration Rates

Reactor Acid, % P_2O_5	Filtration Rate Index 27%	26.0%	25.0%
MgO, %			
0.65	100	102	104
1.23	97	99	101
1.80	76	82	88

Sulfuric acid consumption is slightly higher for the higher MgO phosphates, as expected. H_2SO_4 consumption was monitored in several ways. The weights of H_2SO_4 inputs to the pilot plant gave the best correlation with current plant practice and these indicate about 0.75% and 3.0% increase in H_2SO_4 consumption for 1.2% MgO and 1.8% MgO, respectively.

The general conclusion of this study is that 12% MgO rock is marginally poorer in performance than 0.65% MgO rock but can probably be handled in existing plants with only a slight loss in yield and/or product acid strength. The product acid, however, is above 0.11 MER and even with extreme efforts in clarification and the addition of 3.0% to 4.0% urea will probably just barely make an 18-46-0 product. This evaluation is outside the scope of this study.

The 1.8% MgO level increases losses and reduces plant (filter) capacity to such an extent that it is outside the. range of acceptable feed to plants currently operating in Central Florida.

We believe the 1.8% MgO rock could make an acceptable feed to a plant tailored to produce a lower grade of DAP, 15-45-0, for example. However, the economics of treating this high MgO rock may not warrant its use unless the low grade DAP can be made at lower cost than 18-46-0. Again, this evaluation is outside the scope of this work.

The test series using a single reactor covered two experimental Runs 'E' and 'F' and eleven formal tests each with preliminary day runs followed by continuous tests of 96 to 109 hours. The results of these tests correlated well and are reported above in tables and graphs. An addendum test run (AR-1) was performed after the 11th test, but control and mechanical problems plagued the run and only data from the very end of the run is included in the correlations above. The results of the two reactor configuration indicated only marginal benefits, process-wise. However, in commercial plant operation, the two stage system might well reduce filter and line scaling which cannot be studied in the pilot plant tests.

Selected Data was subject to statistical analysis. Based on this analysis the controlled variables caused significant differences in digestion recoveries, sulfuric acid requirements, filtration rate and filter capacities.

These differences were used to estimate the production costs of using high MgO rock for wet process acid manufacture.

SECTION 3

3.0 PILOT PLANT DIGESTION AND FILTRATION TESTS

3.1 Objective of Tests

The objective of this test work is to develop the data needed to evaluate the economic impact of using higher MgO rock. Factorial designed tests were run in a continuous small scale phosphoric acid pilot plant, varying detention time (throughput) and product acid strength, and using three levels of MgO in the feed rock. Evaluation of optimum sulfate level was done in the initial tests and again in several later runs where two sulfate levels were investigated.

The tests produced various P_2O_5 recoveries, broken down into water soluble, citrate soluble and citrate insoluble losses, and filtration area requirements as the MgO level in the feed rock, the throughput, and product acid P_2O_5 concentration were varied.

The tests were also monitored for H_2SO_4 consumption by measuring actual consumption and by a mass balance based on input and product analyses.

3.2 Test Plan and Methodology

3.2.1 Rock Samples

The tests employed three levels of MgO in the feed rock, 0.65%, 1.23% and 1.8% MgO. These feeds were produced from two rock samples coming from IMC/Agrico Four Corners mine and beneficiation facility. The low MgO feed came directly from a sample of currently produced flotation concentrate. The high MgO feed was the pebble waste tailings from the heavy media circuit. The 1.23% MgO and 1.8% MgO pilot plant feeds were produced by blending the low MgO concentrate with the high magnesium pebble tailing sample after grinding. The high MgO pebble analyzed 4.75% MgO.

The chemical analyses of the low MgO concentrate and the high MgO pebble samples are given in Table 3.2.1. Each rock was ground separately and each hour's test feed was combined from each of the two feed rocks to ensure that each hour received the proper composition of feed.

For the medium level of MgO, 1.23%, a blend of 85.8% low magnesium concentrate and 14.2% of high magnesium pebble was used. For the 1.80% MgO level, 71.8% of the mix was low magnesium concentrate and 28.4% was high magnesium pebble. The blending was done to achieve a 1.225% MgO and a 1.80% MgO on an "as is" basis including free moisture. Hence, the analysis on a dry basis in Table 3.2.1 are slightly higher.

The metal analyses were carried out on an HCI extract of the rock, so that they do not include any iron which might be present as pyrites. This extraction method is usual in Central Florida, and is based on the belief that iron pyrites do not dissolve in phosphoric acid manufacture and are, therefore, best included in the acid insoluble part of the analysis.

The "acid soluble SiO_2" was determined by the standard AFPC method, as the difference between total SiO_2 and the silica content of acid insolubles from HCI digestion. This results in the "acid soluble SiO_2" being reported as much higher than occurred in the pilot plant where about 15% to 20% of total SiO_2 was soluble. See Material Balances, Section 4.4.

The standard AFPC method was also used in the determination of organic carbon. This method measures any oxidizable materials present. The results are consistent with the assumption that no inorganic oxidizable material was present.

The low magnesium concentrate and the high MgO pebble were ground separately and the wet-dry screen analyses are shown in Table 3.2.2, and plotted in Figure 3.2.3.

Samples of the medium MgO feed blend and high MgO feed blend were screened and are also reported in Table 3.2.2.

TABLE 3.2.1

Chemical Analysis of Feed Rocks PN 29-H387-00

	Low MgO Concentrate	High MgO Pebble	Medium MgO Feed	High MgO Feed
		Composition, % Dry Basis		
BPL	70.61	51.27	67.90	65.1
P_2O_5	32.32	23.46	31.06	29.79
CaO	48.19	41.57	47.23	46.26
MgO	0.66	4.75	1.24	1.83
Fe_2O_3	1.35	1.31	1.35	1.34
Al_2O_3	0.95	0.69	0.90	0.87
Na_2O	0.60	0.54	0.59	0.58
K_2O	0.12	0.08	0.12	0.11
F	3.33	2.82	3.21	3.07
Cl	-	0.03	-	-
SO_3	1.22	1.06	1.21	1.18
CO_2	4.34	12.52	5.50	6.68
Organic C	0.30	0.32	-	-
Other Volatiles(1)	0.84	0.41	-	-
Soluble SiO_2 (HCl)	1.17	3.11	-	-
Acid Insoluble	4.07	6.75	-	-
Total	100.01	99.42	-	-
Minus O = F + Cl(2)	1.41	1.19	-	-
% Accounted for	98.60	98.23	-	-
Total SiO_2	5.24	9.86	5.89	6.55
Acid Insoluble SiO_2 (HCl)	4.07	6.75	-	-
Moisture(3)	1.63	1.00	-	-
LOI at 1000°C	5.48	13.25	-	-
CaO/P_2O_5	1.491	1.772	1.52	1.55
$(I\&A)/P_2O_5$	0.071	0.085	0.072	0.074
$(MgO + I\&A)/P_2O_5$	0.092	0.288	0.112	0.136
F/Acid Soluble SiO_2(4)	1.959	0.418	-	-
Pilot Plant feed %, wet basis Low MgO concentrate/high MgO Pebble	100/0.0	-	85.8/14.2	71.6/28.4

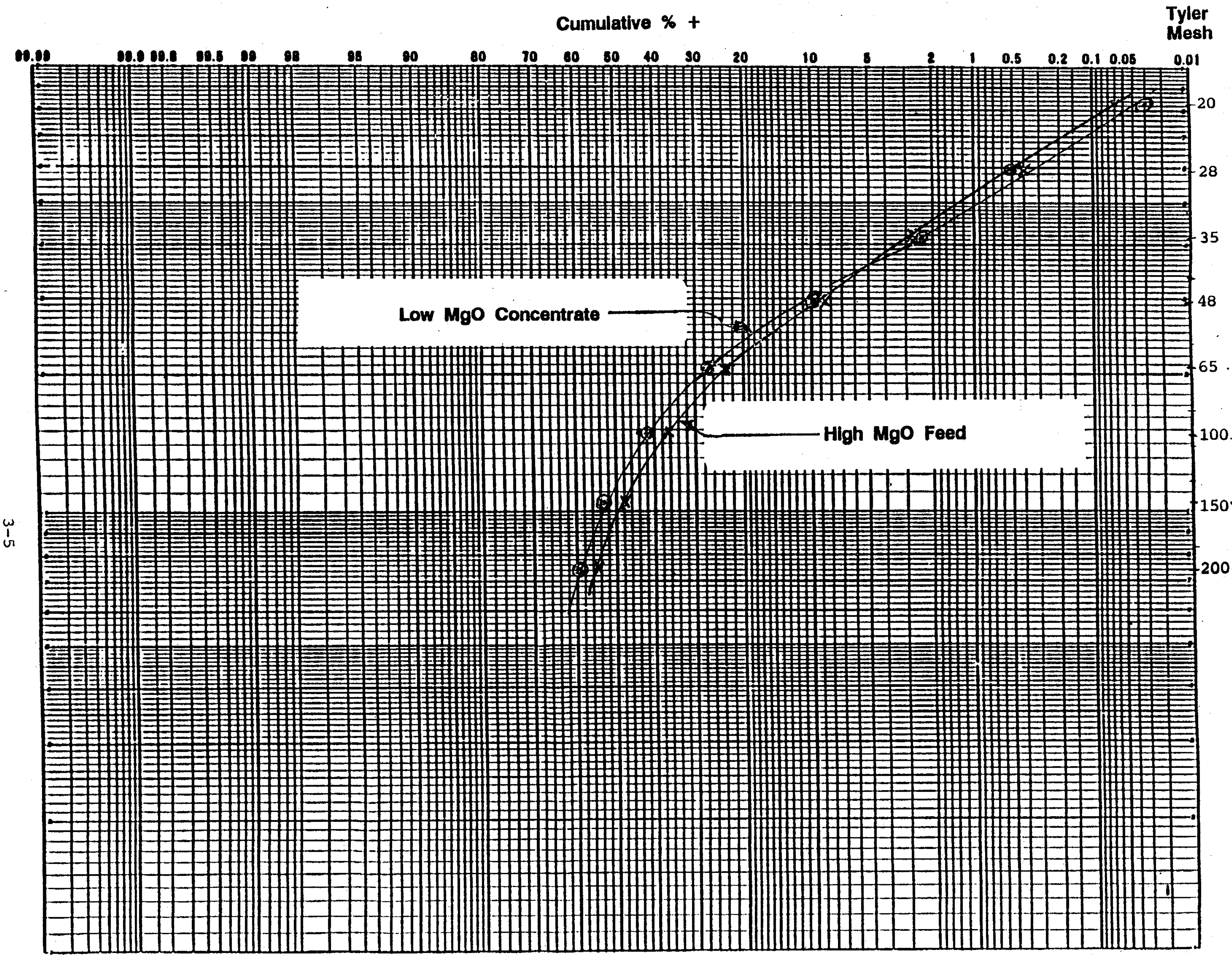
Notes: 1. Other volatiles = LOI - CO_2 - Organic C
2. Oxygen equivalent to F (F x 16/38) plus Cl (Cl x 16/70.9)
3. Moisture on "as is" basis. All other analyses on dry basis.
4. The ratio equivalent of H_2SiF_6 is 1.90

TABLE 3.2.2

Size Analyses of Ground Feed Rocks

Cumulative % Retained on Screen

Tyler Screen Number	Low MgO Concentrate	High MgO Pebble	Medium MgO Feed	High MgO Feed
20	0.03	0.12	0.06	0.06
28	0.51	0.90	0.41	0.42
35	2.39	2.72	2.39	2.44
48	9.93	5.48	8.98	8.66
65	26.30	10.69	24.76	22.99
100	42.08	23.71	38.59	36.43
150	53.27	31.55	49.76	47.85
200	59.64	40.89	56.24	54.28
-200	40.30	59.11	43.76	45.72
Total	99.94	100.00	100.00	100.00



3-5

Pilot Plant Feed Screen Analysis

Figure 3.2.3

3.2.2 Methodology

Table 3.2.4 lists the test conditions that were held constant during the studies. Some experimentation in optimum sulfate level was conducted at the beginning of the tests. This is discussed below under tests E and F. Later, some segments of tests 6, 10 and 11 were run at about 2.2% H_2SO_4, whereas the tests generally employed a sulfate level of approximately 2.5%.

Test variables are given in Table 3.2.5, listing both the objectives and the levels actually achieved.

Test constants and test variables appear to have been controlled well within normal limits for these tests.

Table 3.2.6 gives the test design as set out by A. N. Baumann. in the actual execution, test run 12 was deleted and two experimental runs E and F were done instead at the outset of the tests. Test run 12 was to have been a repeat of runs 2, 7 and 9 and was deemed of less importance than tests E and F which were used to set the sulfate level for the whole series of tests.

At the conclusion of the 11 test run, an addendum run, AR-1, was done. Run AR-l employed a two reactor system and is described later in Section 4.5.

Tests 3.2.4

Test Constants

- **Soluble Sulfate**

  Goal: 2.5% ±0.1 (Pilot Plant Determinations)

  Actual: Tests E & F were run at higher sulfate levels to determine feasibility of operation.

  Tests 6, 10 and 11 had short periods of lower sulfate levels.

  In process tests were run by a rhodizonate titration, checked by a centrifuge method. Gravimetric checks were also run on aged lab samples and gave figures about 0.3% lower.

- **Temperature**

  Goal: 80°C ±1°C

  Actual: 79°C - 81°C

- **Rock Grind**

  Goal: 3% +35 mesh max. and 40% to 50% -200 mesh

  Actual: 2.4% to 2.5% + 35 mesh and 43% to 46% -200 mesh

- **H_2SO_4**

  Goal: 80% by weight H_2SO_4

Table 3.2.5

Test Variables

- **MgO in Rock Feed**

  Set at 0.65% MgO as received in the low magnesium concentrate and 1.80% in high magnesium feed. The intermediate MgO feed was 1.225% MgO (rounded to 1.23%).

- **Retention**

  Goal: 3, 4 and 5 hours

  Actual: 3.0 + 0.1, 4.0, and 5.0 ±0.2 hours

  The retentions above are based on in-process measurement of slurry density. Filter test samples indicate slightly lower slurry percent solids so that actual retentions are about 0.2 hours less on the latter basis.

- **Product acid Strength, % P_2O_5**

Goal	Actual (Average)
27.5	27.2 - 27.9
26.0	26.0 - 26.4
24.5	24.6 - 25.2

TABLE 3.2.6

Test Design
2 by 3 for MgO Digestion Project

CONSTANTS: Digestion
Soluble Sulfate Range
% Solids: 35% by Weight
Temperature: 80°C

Variable Levels		–	o	+
X1	Rock MgO Content %	**0.6**	**1.2**	**1.8**
X2	Filtrate P_2O_5 Conc.	**24.5**	**26.0**	**27.5**
X3	Retention Time - Hours	**3.0**	**4.0**	**5.0**

Test Designation	Test Run	X1	X2	X3
A	1	-	-	-
B	8	+	-	-
C	6	-	+	-
D	10	+	+	-
E	5	-	-	+
F	11	+	-	+
G	4	-	+	+
H	3	+	+	+
I	12	o	o	o
J	9	o	o	o
K	7	o	o	o
L	2	o	o	o

TEST ORDER

Test Designation	Test Run Order	% MgO	% P_2O_5	Detention Hours
A	1	0.6	24.5	3.0
L	2	1.2	26.0	4.0
H	3	1.8	27.5	5.0
G	4	0.6	27.5	5.0
E	5	0.6	24.5	5.0
C	6	0.6	27.5	3.0
K	7	1.2	26.0	4.0
B	8	1.8	24.5	3.0
J	9	1.2	26.0	4.0
D	10	1.8	27.5	3.0
F	11	1.8	24.5	5.0
I	12 (Deleted)	1.2	26.0	4.0

Determine: Sulfuric Acid Consumption
Filtration Rates
Digestion and Filtration Efficiencies

3.3 Digestion and Filtration Test Procedure

3.3.1 Digestion

The pilot plant used for the digestion and filtration tests is shown diagramatically in the illustration, Figure 3.3.1. A single stirred tank reactor made of 316L stainless steel was used. The liquid volume in the reactor was approximately 9.0 liters. The reactor was fed with 60% sulfuric acid, phosphate rock and return acid. Liquid feeds were by means of peristaltic pumps. The rock was fed, pre-weighed, by screw feeder and dispersed into the reactor by the rock wetting agitator (corrosion test agitator). No corrosion test data were collected as it was not deemed pertinent. The feeding of exact hourly amounts was ensured as discussed below:

- The rock was pre-weighed in hourly amounts. The speed of the screw was adjusted to feed these amounts in just under one hour, running out ail the contents of the feeder every hour.

- The return acid flow - which is less critical than the rock flow or the sulfuric acid flow - was monitored by hourly checks of delivery rate by a graduated cylinder, and adjusted to maintain the proper percent solids in the reactor.

- Defoamer feed was by gravity from a burette, the flow being monitored by visual observation of the dropping rate and adjusted by the operator to keep foam at an acceptable level.

The temperature in the reaction vessel was maintained by means of a Variac controlled electrical hot plate, based on continuous temperature measurements by a probe.

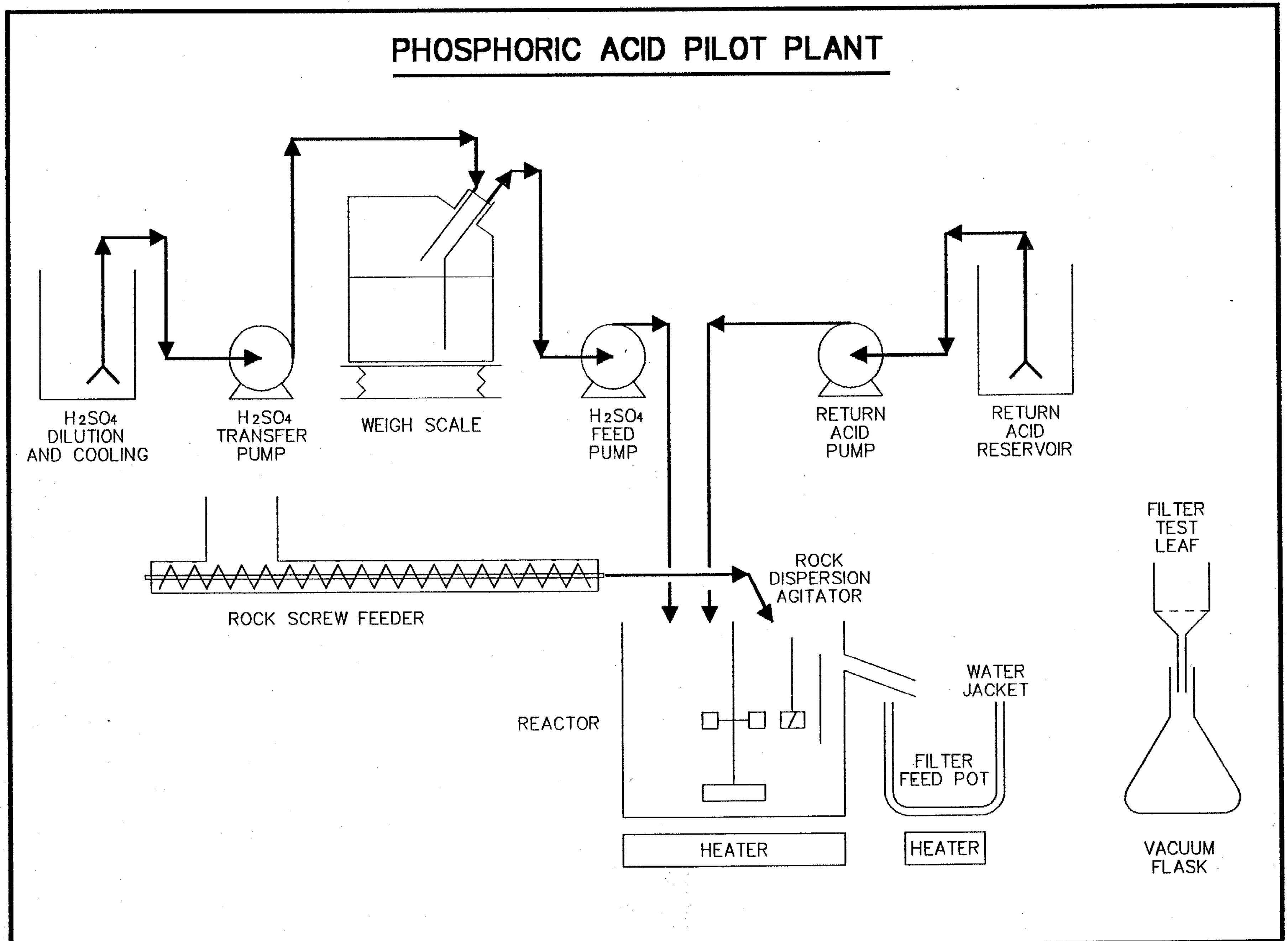


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Figure 3.3.1

The sulfuric acid was diluted to 80%, cooled and transferred to a container on a scale. A peristaltic pump was used to feed the reactor at a constant rate. The pump was turned off for about one minute each hour for taring the scale.

Filtration of the slurry for the production of product acid and return acid was done using a Buchner filter which was moved from vacuum flask to vacuum flask so as to simulate the countercurrent operation of a full scale phosphoric acid plant filter.

For the quantitative determination of filtrate rates, a test leaf filter of 3.03" diameter, 0.050 sq.ft. area was used. The test filter was fitted with polypropylene cloth No. 224-047-25 supplied by National Filter Media Corporation. This cloth is similar to those in general use for the manufacture of phosphoric acid. Data collected during the filter tests were used to calculate the filtration area requirements. The test results were adjusted to represent the area required for a filter turning at three minutes per revolution. These formal filter tests were run every six hours.

Operating conditions, filtration rates, and P_2O_5 losses were plotted on a graphical Chronological Log of the tests. Test data for each test is given in the Appendix.

The pilot plant was initially operated on day shift only for a week on each test as a shakedown run. The reactor contents were allowed to cool after each day, and were re-heated to the operating temperature before the feeds were re-started on the following day. This initial intermittent operation is not reported here. The formal tests reported here consisted of continuous round-the-clock operation. The day tests started operation using water, or a previous slurry of appropriate MgO content, and part of the purpose of these tests was to produce a gypsum slurry representative of the particular rock for each continuous test.

Control of pilot plant operation was by specific gravity measurements of the liquid feeds, product slurry, and product acid, and by analysis of the reaction liquor for sulfate. The sulfate control analysis was by precipitation with barium chloride using rhodizonate as the end point indicator. Some centrifuge method checks were made during Run 6, and gravimetric sulfates were run later on each product acid sample (every six hours), for all the tests.

Table 3.3.2

Digestion Conditions

The test runs were carried out under the following conditions:

	Retention in Hours		
Rock Feed:	3	4	5
High MgO Feed, g/h	1082-1106	--	662
Medium MgO Feed, g/h	--	805	--
Low MgO Feed, g/h	1048-1062	--	638-642
Reactor size, liters:	9.0		
Sulfuric acid feed:	80% H_2SO_4 at room temperature		
Reactor temperature:	79-81°C		

The other controlled variables are shown on the graphical data logs for each run.

3.3.2 Phosphoric Acid Filter Test Procedure

The filtration test conditions are given in Table 3.3.3.

The quantity of feed slurry, 900 grams in all cases, produced about a 3.0" cake.

The pulp is poured in the filter, the vacuum adjusted, and the ball valve opened. When the liquid disappears from the cake surface, the time is noted and called the form time. Five seconds of delay is allowed, then the wash acid is added. After the cake appears dry, the time is recorded and after five seconds delay, the water wash is added. After the liquid drains from the surface of the cake, another 30 seconds dry time is allowed to complete the cycle. The cake is weighed, repulped in 500 ml. of water and washed with 500 ml. more water in increments. These washes are combined and are sampled for W.S. loss. The cake is then washed with alcohol to facilitate drying, then dried at 65°C to avoid loss of water of crystallization. The dried solids are weighed and the weight recorded on the various log sheets and used on the extraction calculation sheet.

Table 3.3.3

Filtration Conditions

The conditions of the filtration tests were standardized as follows:

Test filter:	3.03" diameter, 1/20th of a sq.ft. area
Vacuum:	20" of mercury
Weight of Slurry:	900 g
Wash Acid:	
Volume	252 ml
Specific gravity	1.05
Temperature	60°C
Wash Water (tap water):	
Volume	210 ml
Specific gravity	1.00
Temperature	60°C
Intervals between washes:	5 seconds (included in total cycle time)
Drying time after last wash:	30 seconds
Cake Thickness:	2-3/4" - 3"

Calculation of Filtration Rates

Raw Filtrate Rate =

Tons Gyp/sq./ft./24 hrs =

$$\frac{(1440)\ (60)\ (\text{Dry Wt. Gyp in Grams})}{(454)\ (\text{Area in sq.ft.})\ (2000)\ (\text{Total Cycle time in seconds})}$$

- where the test filter is 0.05 sq.ft.

Example: Filter Tests No. 13, Run 11.

Raw filtration rate is:

$$\frac{1440\ (60)\ \ (192.9)}{454(0.05)(2000)(102)} = 5.45 \text{ tons gyp/sq.ft./24 hrs}$$

All test results are adjusted to a 3 min/revolution filter speed, which at 85% active area, gives a cycle time of 153 seconds.

Adjusting the cycle to 153 seconds for the commercial filter with the test cycle in seconds of 102, we have an adjustment factor F, based on the square root of the two cycle times, as follows:

$$F = \sqrt{\frac{102}{153}} = 0.816$$

Adjusted Filtration Rate = 5.45 x 0.816 = 4.45 tons gyp/sq.ft./24 hrs

To convert the capacity to t. P_2O_5/sq. ft/day:

Tons gyp/t P_2O_5 produced =

$$\frac{\text{gyp/rock ratio}}{(\text{Wt fraction } P_2O_5 \text{ in rock})\ (\text{Wt fraction } P_2O_5 \text{ recovered})}$$

- where gyp/rock = $\frac{\text{\% CaO in Rock}}{\text{\% CaO in Gyp}}$ = 1.52

$$= \frac{1.52}{(0.2936)\ (.9726)} = 5.32 \text{ t gyp/t } P_2O_5 \text{ produced}$$

$$\text{Tons } P_2O_5\text{/sq. ft./day} = \frac{4.45 \text{ t gyp/sq.ft./day}}{5.32 \text{ t gyp/t P2O5 produced}} = 0.84$$

3.3.3 Addendum Test Run (AR-1)

After the test program described above, a further test run comprising a day test plus a 109 hour continuous test was completed using a two reactor configuration. This test, AR-1, is described in Section 4.5.

SECTION 4

4.0 TEST RESULTS

4.1 Experimental Runs

Two experimental runs E and F were made beginning in late May to establish optimum H_2SO_4 levels for the digestion tests. One theory for treating high MgO rocks is to use a higher total sulfate level so as to have a higher free acid present and a higher driving force for dissolving the rock. This is intended to counteract the effect of the Mg^{++} ions and increase total acidity. On this basis, testing was begun in Run E, the first test, attempting to hold sulfate levels above 3.0% H_2SO_4 at 1.8% MgO, 24.5% P_2O_5 and 3.0 hours residence time.

Run E is reported only as the numerical data log, the extraction calculation sheet, and the chronological log. It was readily apparent that this level of sulfate could not be run in the Jacobs' pilot plant configuration. It is also likely that no commercial phosphoric acid plant could sustain 3.0% total H_2SO_4 in the liquid phase on high MgO rock at normal retentions where appreciable unattacked rock is present.

Run F was also run at 1.30% MgO, 24.5% P_2O_5 and 3.0 hours residence time, but at a goal of 2.8% total sulfate. After some difficulty in control, the run was completed but the citrate insoluble losses were generally high (2.02% of P_2O_5 fed) and the overall losses were 5.43%, over 2% higher than later run No. 8, run at the same conditions but at a lower sulfate level, about 2.5% or slightly less. Part of the difference in recoveries may have been due to a more experienced crew by the time test No. 8 was run in early October. However, the results of Run 8 correlate well with other tests while Run F does not and Run F results, therefore, were not included in the graphs of test results shown later in Section 4.4.

4.2 Test Run Averages, Usages and Products

4.2.1 Discussion of Test Results

The test run averages, usages and products are given in Tables 4.2.1 and 4.2.2. These are the final Tables 1 and 2 from the last monthly report.

Test run No. 1, along with Tests E and F, was not included in Jacobs' correlations of the tests. Test No. 1, attempting a 2.8% H_2SO_4 level, had control difficulties. The rock was the low magnesium concentrate, 0.55% MgO at 3 hours retention and 24.5% P_2O_5 product acid. The total losses for test No. 1 were in the range of 4.0% of the P_2O_5 fed. Based on Test 6A results at 27.5% P_2O_5 and a sulfate around 2.2%, and the correlations shown in Figure 4.2.3, the losses should have been a little above 2.0% of the P_2O_5 fed.

After test run No. 1, the sulfate objective was set at 2.5% and the H_2SO_4 level was run at about that value for tests 2, 3, 4 and 5.

In Test 6, as the run progressed, it was noticed that the gap between the plant rhodizonate sulfate and the gravimetric lab determination was larger than usual. It was established that the pilot plant had been running at a sulfate lower than intended due to a reagent error. The sulfate was then raised, perhaps a little too much, for the remainder of the run. The sulfates for the first part of the run 6A are shown as corrected values and run about 2.2%. The second part of the run at higher sulfate was labelled 6B.

The No. 6 run at 3 hours retention, 27.5% P_2O_5 and 0.65 MgO showed much lower losses at the lower sulfate (2.2% in pilot plant, 1.65% in the lab gravimetric).

Run No. 10 was also divided into two portions and again the lower sulfate (2.2% in pilot plant) showed lower losses. In both cases, the citrate insoluble losses were high in the high sulfate operation. Run 10 was 3 hours, high magnesium - 1.8% MgO, and 27.5% P_2O_5 in the product acid.

Table 4.2.1

RUN AVERAGES

	RUN F	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6A	RUN 6B	RUN 7	RUN 8	RUN 9	RUN 10A	RUN 10B	RUN 11A	RUN 11B	ADDENDUM REACTOR1	RUN NO. 1 REACTOR2
TEST CONDITIONS																	
DURATION OF RUN (hours)	95	96	96	96	96	96	108	108	96	96	96	108	108	108	108	110	110
PERIOD AVERAGED (hours)	56-94	62-95	50-95	50-95	50-95	50-95	38-84	86-107	50-95	50-95	50-95	32-86	86-107	49-81	83-107	98-109	98-109
FEED ROCK ANALYSES (1):																	
%P205	29.36	31.79	30.58	29.36	31.79	31.79	31.79	31.79	30.58	29.36	30.58	29.36	29.36	29.36	29.36	30.58	30.58
%CaO	45.58	47.40	46.50	45.58	47.40	47.40	47.40	47.40	46.50	45.58	46.50	45.58	45.58	45.58	45.58	46.50	46.50
%MgO	1.80	.65	1.23	1.80	.65	.65	.65	.65	1.23	1.80	1.23	1.80	1.80	1.80	1.80	1.23	1.23
ROCK FEED RATE (gms/hr.)	1084	1048	805	662	646	638	1062	1062	805	1082	805	1106	1106	653	653	1061	1061
OPERATING CONDITIONS																	
RETENTION (hours)	3.03	3.14	4.04	5.13	4.99	4.89	3.04	3.02	4.02	3.08	4.06	2.99	2.88	4.96	4.98	3.06	.97
REACTION VOLUME (m^3/stpd P205)	1.13	1.06	1.43	1.83	1.71	1.71	1.04	1.06	1.42	1.10	1.42	1.13	1.11	1.82	1.84	1.10	1.09
PLANT ANALYSES:																	
%H2SO4	2.78	2.72	2.53	2.54	2.49	2.48	2.20	2.74	2.50	2.50	2.60	2.60	2.20	2.53	2.14	2.19	3.09
ACID S.G.	1.322	1.301	1.324	1.355	1.332	1.292	1.328	1.330	1.324	1.315	1.325	1.361	1.361	1.318	1.314	1.319	1.348
%SOLIDS IN SLURRY	34.9	36.1	35.2	35.1	35.0	34.8	35.2	34.9	34.8	35.6	35.5	34.9	33.9	35.0	35.2	34.6	34.6
LAB ANALYSES:																	
%H2SO4	2.67	2.22	2.25	2.49	2.25	2.15	1.65	2.47	2.25	2.23	2.22	2.14	1.64	2.17	1.50	1.70	2.66
%P205	24.63	25.27	26.43	27.93	27.59	24.94	27.47	27.52	26.39	24.57	26.05	27.31	27.74	24.79	24.64	25.90	27.18
FILTER TESTS																	
%SOLIDS IN SLURRY	33.5	33.6	33.4	32.9	33.6	32.8	32.7	33.0	32.8	32.7	33.2	32.4	31.0	32.8	32.9		28.9
%SOLIDS IN CAKE	73.5	72.8	75.3	74.3	73.8	76.0	71.8	71.5	74.7	75.1	74.9	71.5	69.0	76.8	75.6		72.7
t GYPSUM/ft^2/day	4.53	4.44	4.64	4.18	4.34	4.63	4.33	4.17	4.47	4.13	4.47	3.47	3.43	4.51	4.51		3.86
t P205/ft^2/day	.84	.89	.90	.79	.88	.94	.87	.82	.88	.79	.87	.63	.63	.85	.85		.74
EXTRACTION LOSSES (% of P205 fed)																	
CITRATE SOLUBLE	2.93	2.22	2.41	2.53	1.77	1.34	2.64	3.21	2.13	2.67	2.05	3.84	3.86	2.25	3.12	2.82	2.82
CITRATE INSOLUBLE	2.02	1.28	.60	1.55	.75	.20	.26	1.65	.47	.26	.48	2.61	.77	.22	.20	1.03	.38
WATER SOLUBLE	.48	.31	.23	.45	.35	.11	.16	.40	.22	.15	.19	.74	.61	.15	.13	.51	.51
TOTAL	5.43	3.81	3.24	4.53	2.87	1.65	3.06	5.26	2.82	3.08	2.72	7.19	5.24	2.62	3.45	4.36	3.71
P2O5 RECOVERY	94.57%	96.19%	96.76%	95.47%	97.13%	98.35%	96.94%	94.74%	97.18%	96.92%	97.28%	92.81%	94.76%	97.38%	96.55%	95.64%	96.29%

NOTES:

(1) feed rock analyses calculated from analyses of the components

(2) (9000 x 2000)/(646 x 24 x 0.3179 x 0.9713 x 2205)

(3) (9000 x 2000)/(638 x 24 x 0.3179 x 0.9836 x 2205)

Table 4.2.2

AVERAGE ANALYSES (1)
USAGES AND PRODUCTS

	RUN F	RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6A	RUN 6B	RUN 7	RUN 8	RUN 9	RUN 10A	RUN 10B	RUN 11A	RUN 11B	ADDENDUM REACTOR1	RUN NO. 1 REACTOR2
ROCK																	
%P205	29.36	31.79	30.58	29.36	31.79	31.79	31.79	31.79	30.58	29.36	30.58	29.36	29.36	29.36	29.36	30.58	30.58
%CaO	45.58	47.40	46.50	45.59	47.40	47.40	47.40	47.40	46.50	45.58	46.50	45.58	45.58	45.58	45.58	46.50	46.50
%SO3	1.18	1.20	1.19	1.18	1.20	1.20	1.20	1.20	1.19	1.18	1.19	1.18	1.18	1.18	1.18	1.19	1.19
%MgO	1.80	.65	1.23	1.80	.65	.65	.65	.65	1.23	1.80	1.23	1.80	1.80	1.80	1.80	1.23	1.23
ACID																	
%P205	24.63	25.27	26.43	27.93	27.59	24.34	27.47	27.52	25.39	24.57	26.05	27.31	27.74	24.79	24.64	25.90	27.18
%CaO	.05	.19	.40	.10	.04	.25	.08	.06	.11	.04	.04	.04	.07	.10	.16	.06	.03
%SO3	2.27	2.22	2.07	2.07	2.03	2.02	1.80	2.24	2.04	2.04	2.12	2.12	1.80	2.07	1.75	1.39	2.17
%MgO	1.70	.53	1.10	1.80	.55	.49	.63	.65	1.04	1.52	1.12	1.80	1.82	1.55	1.55	1.13	1.18
GYPSUM																	
%P205	1.076	.795	.657	.899	.592	.342	.634	.954	.574	.599	.548	1.405	1.017	.510	.676	.700	.650
%CaO	30.64	31.03	30.58	30.87	30.97	30.91	30.90	30.93	30.88	30.50	30.46	30.18	30.08	30.20	30.19	30.69	31.03
%SO3	43.12	43.62	43.67	43.65	44.43	45.14	44.73	44.61	44.16	43.91	44.31	42.40	42.69	43.48	43.55	43.26	44.42
%MgO	.010	.002	.005	.006	.006	.005	.003	.004	.004	.040	.007	.014	.012	.004	.003	.002	.006
MATERIAL USAGE(2)																	
t H2SO4/t rock fed	.802	.830	.819	.799	.845	.859	.849	.852	.826	.818	.843	.795	.799	.817	.812	.806	.829
t H2SO4/t P205 produced	2.888	2.716	2.769	2.850	2.737	2.746	2.755	2.808	2.779	2.872	2.834	2.919	2.871	2.856	2.866	2.743	2.801
t rock/t P205 produced	3.602	3.270	3.379	3.567	3.238	3.198	3.245	3.297	3.365	3.513	3.362	3.671	3.594	3.497	3.528	3.401	3.378
kg defoamer/t P205 produced(3)	9.177	5.376	5.231	8.714	6:437	5.437	4.714	4.575	6.638	8.474	5.223	8.197	8.141	5.652	6.362	5.634	5.595
PRODUCTS(2)																	
t acid/t P205 produced	4.060	3.957	3.784	3.580	3.625	4.010	3.640	3.634	3.789	4.070	3.839	3.662	3.605	4.034	4.058	3.861	3.679
t gypsum/t P205 produced	5.352	4.971	5.089	5.256	4.951	4.872	4.968	5.046	5.054	5.245	5.127	5.539	5.438	5.265	5.385	5.146	5.058
MgO DISTRIBUTION (%)																	
ROCK	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
PRODUCT ACID	99.23	99.63	99.39	99.51	98.53	98.78	99.35	99.15	99.49	96.72	99.17	98.84	99.02	99.66	99.75	99.76	99.31
GYPSUM	.77	.37	.61	.49	1.47	1.22	.65	.85	.51	3.28	.83	1.16	.98	.34	.25	.24	.69

NOTES:
(1) averaged over the time period shown in Table 1
(2) calculated from the analyses of the rock, acid, and gypsum by mass balance
(3) calculated from the actual usage

Run No, 11, again divided into two portions, showed better recoveries this time at the higher sulfate level. This test used 5 hours retention and low, 24.5% P_2O_5, product acid strength. These latter conditions were more favorable to high sulfate, in that the rock and acid were more easily dispersed at low P_2O_5 strength and effective dilution of feeds was greater because of the lower feed rates at 5 hours retention. The optimum sulfates appear to be dynamic; they change with retention and acid strength.

We believe that, at 3 hours detention, there is more supersaturation occurring and that the true sulfate in the digester may be somewhat higher than the measured sulfate which changes as the sample is collected. At 5 hours detention, conditions are more at equilibrium, supersaturation is less, contributing to a higher optimum measured sulfate.

On the basis of these tests, it appears that the 1.80% MgO rock can be treated best at low acid strengths and longer retentions (lower feed rates). The higher MgO rock is more sensitive to coating at higher sulfates and higher P_2O_5 in the acid.

It also appears to us that the regime is responding typically and that 1.8% MgO rock is dissolving and precipitating gypsum at normal or near normal rates, and that there is adequate driving force present. In fact, you might consider there was too much driving force in several instances where high citrate insolubles were encountered. A microscopic examination should establish whether the citrate insoluble loss is gypsum coating of rock or unattacked rock caused by a reduced dissolution rate.

Higher solid solution loss (citrate soluble loss) is likely due to the presence of Mg^{++} ions in solution. Unfortunately, raising the H_2SO_4 to reduce citrate soluble loss does not seem to work because the 1.8% MgO digestion conditions are, if anything, more likely to be sensitive to rock coating than is the case when running lower MgO rocks.

As retention goes up, it becomes possible to run at higher sulfate levels. Perhaps at 8 or 10 hours, it might be possible to digest the high MgO rock at high enough sulfate level to counteract the effect of Mg^{++} ions and get lower or normal c.s. losses. This is obviously not a good solution economically.

4.2.2 Test Correlations

Figures 4.2.3, 4.2.4 and 4.2.5 plot most of the test results. In Figures 4.2.3 and 4.2.4, only Tests E, F and No. 1 are left out. We believe these correlations are remarkably consistent. They show the expected benefits for lower acid strength and longer detentions and the relationships between these.

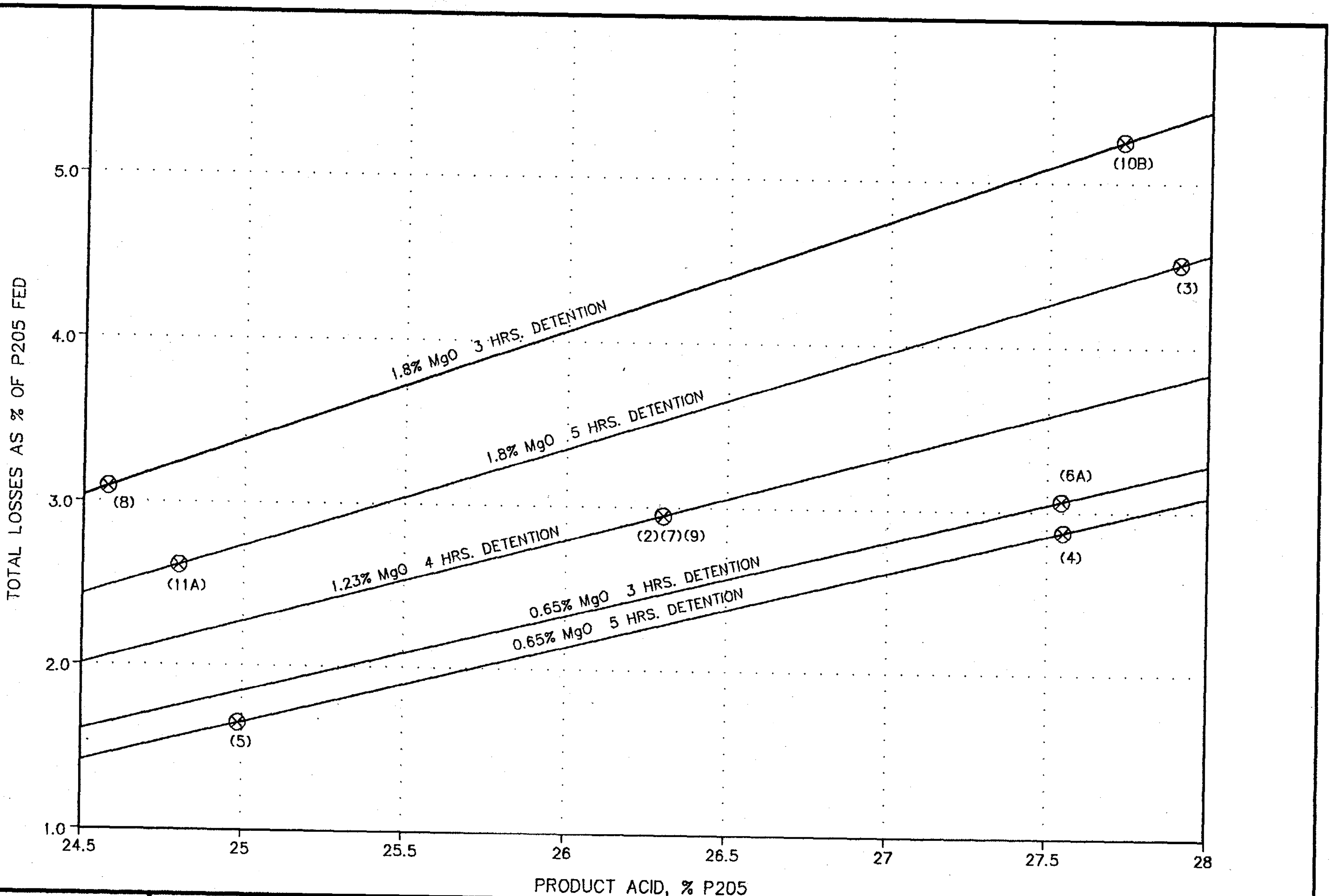
Enough data was collected to plot major effects. Interpolation could be done for the intermediate level MgO (1.23% MgO) with reasonable accuracy, and for intermediate detentions and product acid strength.

According to Figure 4.2.3, a plant using 1.8 MgO rock would need to sacrifice about 3% P_2O_5 in product acid strength to maintain recovery at 3 hours retention comparable to that from 0.65 MgO rock. This comparison is using "ideal" filter test water soluble losses, so that in commercial practice where filters are less than perfect, the differential losses at a given acid strength are likely to be greater than shown on Figure 4.2.3.

Figure 4.2.4 plots filtration rates as T. P_2O_5/sq ft/day vs product acid strength. The data is remarkably consistent. The leaf tests at 0.65% MgO correlate well with plant practice in general. In some cases, plant rates are a little higher than lab rates where the commercial reactor configurations provide a concentration gradient through the digestion system, as in the Prayon or Jacobs' reactors. Therefore, we feel the rates shown in Figure 4.2.4 are conservative and contain a safety factor of 10%, or so, for most commercial installations. They do indicate that for the 1.23% MgO it is possible to maintain a plant filtration rate equivalent to a 0.65% MgO feed if the acid strength is dropped by 1% P_2O_5, or so. However, for 1.8% MgO, even lowering the acid strength would not sustain a comparable filtration rate.

Figure 4.2.5 plots water soluble losses vs % P_2O_5 in product acid at the three MgO parameters. Results plotted include 9 of the 11 tests that were run.

Table 4.2.6 compares the performance of three identical tests run as check tests. The replication appears to be well within expectations.



PN:	29-H387-00
Prepared By:	DWL/CMP
Date:	12-1-93
Ref:	H387002A

( ) DENOTES TEST RUN NO.

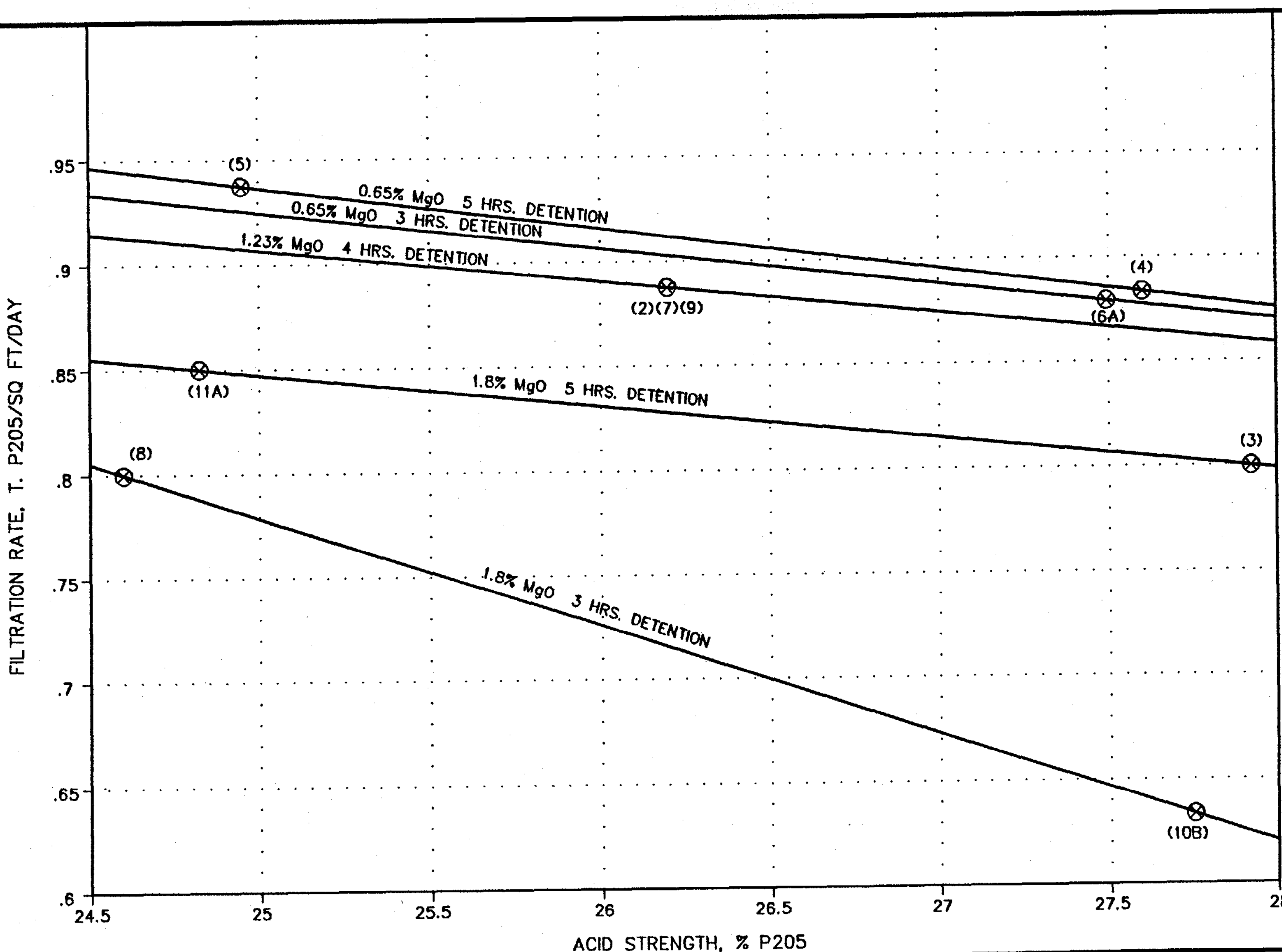
NOTE: ALL TESTS EXCEPT E, F & No.1.

JE **JACOBS ENGINEERING GROUP INC.**

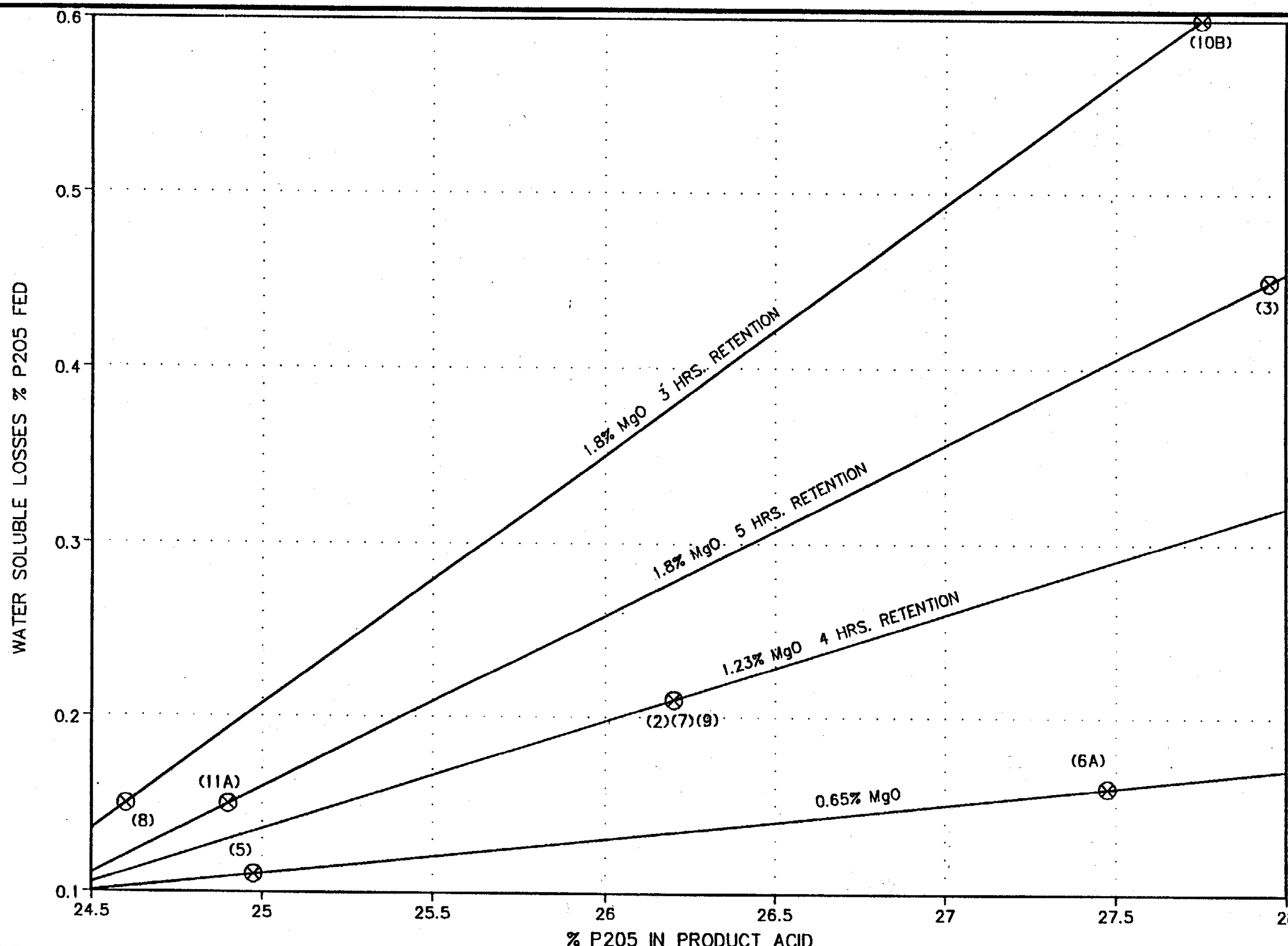
LAKELAND FLORIDA

Figure 4.2.3

TOTAL LOSSES VS % P205 IN ACID



PN: 29-H387-00	( ) DENOTES TEST RUN NO.	JE	JACOBS ENGINEERING GROUP INC.	Figure 4.2.4
Prepared By: DWL/CMP	NOTE: ALL TESTS EXCEPT E, F & No.1.		LAKELAND FLORIDA	FILTRATION RATES VS ACID % P205
Date: 12-1-93				
Ref: H387003A				



PN: 29-H387-00 Prepared By: DWL/CMP Date: 12-1-93 Ref: H387004A	( ) DENOTES TEST RUN NO.	**JACOBS ENGINEERING GROUP INC.** LAKELAND FLORIDA	**FIGURE 4.2.5** WATER SOLUBLE LOSSES VS % P205 IN PRODUCT ACID

TABLE 4.2.6

Duplicate Runs - 1.23% MgO

Run No.	Detention Hrs.	% P_2O_5	H_2SO_4 Content %	H_2SO_4 Consumption T/T P_2O_5 (1)	Losses c.s.	Losses c.i.	Losses w.s.	Total	Filt. Cap. T Gyp sq/ft/D (2)
2	4.00	26.26	2.53	2.72	2.41	0.60	0.23	3.25	0.90
7	4.00	26.39	2.50	2.695	2.13	0.47	0.22	2.82	0.88
9	4.08	26.05	2.60	2.709	2.05	0.48	0.19	2.72	0.87
Avg.	4.03	26.23						2.93	0.88

(1) Inputs

(2) Last 45 hours

4.2.3 Commercial Plant Performance

If we look at the results of the pilot plant run No. 6A, operating at conditions of P_2O_5 strength and retention (27.5% P_2O_5 and 3 hours) similar to many commercial plants, the water soluble loss by test leaf is only 0.16% of the P_2O_5 fed. See Figure 4.2.5. This is a much lower loss than would be encountered with commercial filters where the cake loss might be 0.4% to 1.0% of P_2O_5 fed as pan samples but because of P_2O_5 nestled in the heel and cloth and piping and because of splash and spillage, the normal commercial w.s. loss, including these "mechanical" losses is something like 1.5% to 3.0%, depending on the condition of the filter and the skill of the operators.

Based on the insoluble losses in these tests, and adjustments to the water soluble losses to include a reasonable commercial level of water soluble loss consisting of cake plus repulp losses, the following Table 4.2.7 can be predicted using 3 hour retention data. It should be remembered that the higher losses in Table 4.2.7 are based on a lower filtration rate as well, as the tests produced.

The table is based on the base case of 0.65% MgO in the rock and 26% and 27% P_2O_5 reactor acid, probably 1% less as No. 1 filtrate. If a given plant now experiences losses more or less than shown opposite 0.65% MgO on Table 4.2.7, then the expected losses for higher MgO's would vary accordingly, compared to the losses shown in this table. To arrive at an overall inventory recovery of P_2O_5, a figure for miscellaneous losses, spills sludge losses and shrinkage would need to be added to the losses of Table 4.2.7.

TABLE 4.2.7

Expected Commercial Plant Losses

Reactor Acid Strength % P_2O_5	27%			26%		
MgO in Rock, %	Insoluble	Water Soluble(1)	Total	Insoluble	Water Soluble(1)	Total
0.65	2.65	2.0	4.65	2.4	1.5	3.9
1.20	3.10	2.5	5.60	2.6	2.0	4.6
1.80	4.25	3.5	7.75	3.75	2.5	6.25

(1) Includes cake and repulp losses; all losses as % of P_2O_5 fed.

4.3 H_2SO_4 Consumption

4.3.1 H_2SO_4 Consumption Increase for Higher MgO

Sulfuric acid consumption was monitored by two methods, measured inputs and by a material balance based on laboratory analysis of the gypsum, product acid, and feed rock. In addition, H_2SO_4 consumption was checked by a stoichiometric calculation based on the rock analysis and P_2O_5 recovery. Further, a simple check was made of the change in the CaO/P_2O_5 ratio.

A summary of the averages of the increase in H_2SO_4 consumption for each of these methods is shown in Table 4.3.1.

4.3.2 Measured H_2SO_4 Consumption Average

The actual consumption figures for four high MgO runs (3, 8, 10B and 11) three medium MgO runs (2, 7, and 9) and four low MgO runs (1, 4, 5, and 6) are given in Table 4.3.2.

The sulfuric acid consumption figures are in line in terms of the stoichiometry in comparing one test with another. However, the absolute values appear high compared to industry reported averages, considering the high recovery in the pilot plant tests:

Year	Tons H_2SO_4/T P_2O_2 (TFI)	
	>400,000 TPY	All Plants
1993	2.695	2.684
1992	2.725	2.724
1991	2.651	2.717
1990	2.645	2.730
1989	2.688	2.721

Considering that the pilot plant configuration and filter test methods result in relatively high P_2O_5 recovery, the test results for 0.65 MgO concentrate should be well below industry averages in H_2SO_4 consumption. The industry no doubt does not give credit for the H_2SO_4 in the pond water but this calculates only to about 0.02 T H_2SO_4/T P_2O_5 produced.

TABLE 4.3.1

Increase in H_2SO_4 Consumption Over 0.65% MgO Rock

Basis	High MgO	Medium MgO
Measured Inputs	3.0%	0.75%
Material Balance	4.75%	1.8%
Stoichiometric*	4.5% .	1.9%
CaO/P_2O_5 Ratio	4.0%	2.0%

* Based on the rock analysis and recovery of P_2O_5

TABLE 4.3.2

H_2SO_4 Requirements, Average, T 100% H_2SO_4/T P_2O_5 Produced

Basis:	Wts. of Inputs	Material Balance By Analyses	Stoichiometric(1)
High MgO Rock	2.77	2.86	2.81
Medium MgO Rock	2.71	2.79	2.74
Low MgO Rock	2.69	2.74	2.69

(1) Based on all CaO in rock converted to gypsum, less CaO equivalent to SO_3 in rock, and based on test P_2O_5 recovery.

There is also a significant discrepancy between the material balance H_2SO_4 consumption and the measured inputs. On the basis of industry figures, measured inputs apparently give the most accurate consumption. We believe there may be a consistent bias in the SO_3 analyses of the gypsum which could account for the high consumption reported in the material balance method. However, there are some very low H_2SO_4 consumption figures reported by plants running less than 400,000 TPY P_2O_5 that would seem to be out of line.

4.3.3 Sulfate Control

Sulfate control improved as the tests progressed as evidenced by the following table of standard deviations. However, this table shows a wider deviation for high MgO and low detention (Test No. 10) even toward the end of the program.

Run	% MgO	% P_2O_5	Retention, Hours	Standard Deviation % H_2SO_4
2	1.2	26.0	4	0.113
3	1.8	27.5	5	0.129
4	0.6	27.5	5	0.13
5	0.6	24.5	5	0.08
6	0.6	27.5	3	0.21
7	1.2	26.0	4	0.07
8	1.8	24.5	3	0.09
9	1.2	26.0	4	0.07
10	1.8	27.5	3	0.17
11	1.8	24.5	5	0.09

4.4 Material Balances

Mass balances were run for the following tests:

Test	% MgO	% P_2O_5	Retention Hours
3	1.8	27.5	5.0
4	0.6	27.5	5.0
5	0.6	24.5	5.0
9	1.2	26.0	4.0
11	1.8	24.5	5.0

These balances were developed from averaging the analyses of four sets of samples taken during steady operation at near average conditions.

Certain assumptions have been made in these balances. The fluorine content of the acid is taken as a base figure. The Na_2O analyzed in the filter acid is taken as correct. The remainder of the Na_2O coming from the rock is calculated to be in the gypsum. The reactor off-gases are assumed to contain 5% of the fluorine in the rock. The remainder of the fluorine is calculated to be in the gypsum.

The fluorine is assumed to evolve from the reactor as SiF_4, which is the basis for the SiO_2 in the off gas.

The SiO_2 in the gypsum is taken as the analyzed SiO_2 in rock minus SiO_2 in off-gases minus SiO_2 in acid.

The general conclusion of these analyses is that the cation impurities are relatively soluble. See Table 4.4.

MASS BALANCE CALCULATIONS

4 CORNERS - HIGH MgO FILE: FIPR3R3
ACIDULATION RUN3 11/13/93

ROCK			GRAMS ROCK =	100.00
%P205	29.79	.30		
%CaO	46.26	.46		
%SO3	1.20	.01		

PROD. ACID			GRAMS ACID =	102.30
%P205	28.03	.28		
%CaO	.11	.00		
%SO3	1.95	.02		

GYPSUM			GRAMS GYP. =	149.74
%P205	.75	.01		
%CaO	30.82	.31		
%SO3	43.89	.44		

SULFURIC ACID GRAMS H2SO4 = 81.48

T H2SO4/T P205 (PRODUCED) 2.8

P205 IN THE GYPSUM INCLUDES BOTH SOL. AND INSOL. P205 LOSSES

COMPONENT BALANCE

	IN	OUT
P205	29.79	29.79
SO3	67.71	67.71
CO2	6.68	6.68
Fe2O3	1.34	1.34
Al2O3	.87	.87
CaO	46.26	46.26
MgO	1.83	1.83
Na2O	.58	.58
K2O	.11	.11
F	3.07	3.07
SiO2	6.55	6.55
OequivF	-1.29	-1.29
OTHER	185.15	185.15

MATERIAL BALANCE

------ IN ------ 4 CORNERS RUN No.3 ----- OUT -----

-----------PHOSPHATE ROCK---------

	WEIGHT %	WEIGHT UNITS	% OF TOTAL
P205	29.79	29.79	100
SO3	1.20	1.20	1.77
CO2	6.68	6.68	100
Fe2O3	1.34	1.34	100
Al2O3	.87	.87	100
CaO	46.26	46.26	100
MgO	1.83	1.83	100
Na2O	.58	.58	100
K2O	.11	.11	100
F	3.07	3.07	100
SiO2	6.55	6.55	100
OequivF	-1.29	-1.29	
OTHER	3.01	3.01	
TOTAL	100.00	100.00	
FREE H2O	1.45	1.45	.87
TOTAL	101.45	101.45	

--------------SULFURIC ACID-----------

	WEIGHT %	WT. UNITS	% OF TOTAL
SO3		66.51	98.23
COMB H2O		14.97	
H2SO4	79.86		
FREE H2O	20.14	20.37	12.18
TOTAL	100.00	101.85	

----------------WATER-----------------

	WEIGHT %	WT. UNITS	% OF TOTAL
H2O	100.00	145.36	86.95
TOTAL	100.00	145.36	

* CALCULATED VALUE

--------------GAS--------------

	CALC WT %	WT. UNITS	% REPORTING
STEAM*	80.07	27.68	16.56
CO2*	19.32	6.68	100.00
F*	.44	.15	5.00
SiO2*	.35	.12	1.85
OequvF	-.19	-.06	
TOTAL	100.00	34.57	

-------------GYPSUM-------------

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P205	.75	1.12	3.74	
SO3	43.89	65.72	97.06	
Fe2O3	.03	.05	3.47	
Al2O3	.04	.07	7.55	
CaO	30.82	46.15	99.76	
MgO	.01	.01	.51	
Na2O*	.30	.45	77.57	.033
K2O*	.03	.04	39.91	.006
F*	.29	.43	13.98	.058
SiO2*	3.82	5.73	87.38	3.78
OequvF	-.12	-.18		
OTHER	20.14	30.16	16.29	
TOTAL	100.00	149.74		
FREE H2O	41.44	62.05	37.12	
TOTAL	141.44	211.79		

---------PRODUCT ACID----------

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P205	28.03	28.68	96.26	
SO3	1.95	1.99	2.94	
Fe2O3*	1.26	1.29	96.53	1.34
Al2O3*	.79	.81	92.45	.75
CaO	.11	.11	.24	
MgO*	1.78	1.82	99.49	1.80
Na2O	.13	.13	22.43	
K2O	.06	.07	60.09	
F	2.44	2.49	81.02	
SiO2	.69	.71	10.77	
OequvF	-1.03	-1.05	-.57	
OTHER	63.79	65.26	35.24	
TOTAL	100.00	102.30		

FIPR　　　　FILE: FIPR3R3
ACIDULATION RUN3　　　　11/13/93

29-H387-00　　　　CHEMICAL ANALYSIS

SAMPLE NO. PRODUCT		%P2O5	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O	CONTROL %H2SO4	A. LAB %H2SO4
ACID #1		28.17	1.93									2.47	2.37
2		27.97	1.83									2.35	2.24
3		28.31	1.58									2.60	1.93
4		26.81	1.84									2.33	2.26
5		28.15	2.04									2.60	2.50
6		28.20	1.80									2.40	2.20
7		27.34	2.00									2.30	2.45
8		27.85	1.88									2.34	2.30
9		28.17	2.20									2.70	2.70
10	*	28.02	2.06	.14	1.35	.80	1.79	2.51	.69	.15	.065	2.50	2.52
11	*	28.02	1.94	.10	1.33	.76	1.79	2.38	.69	.14	.064	2.45	2.38
12		27.18	2.27									2.73	2.78
13		27.88	1.80									2.52	2.21
14	*	28.26	1.84	.10	1.33	.73	1.81	2.47	.70	.12	.067	2.47	2.26
15	*	27.83	1.94	.10	1.34	.71	1.79	2.38	.68	.10	.063	2.40	2.38
16		27.62	2.20									2.73	2.70
17		28.39	2.06									2.54	2.52
AVERAGE		28.03	1.95	.11	1.34	.75	1.80	2.44	.69	.13	.065	2.46	2.39

SAMPLE NO.	%P2O5	% C.I. P2O5	% W.S. P2O5	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O
GYPSUM #1	.64	.130	.130	43.51	30.73							
2	.57	.070	.080	44.42	30.73							
3	.71	.120	.063	43.77	30.82							
4	.71	.110	.055	43.68	30.73							
5	.71	.180	.072	43.13	30.90							
6	.81	.270	.096	43.36	30.73							
7	.76	.170	.066	43.44	30.82							
8	.71	.170	.067	43.68	30.82							
9	.88	.330	.096	43.94	30.73							
10	.71	.180	.079	43.78	30.82	.032	.042	.006	.082	3.50	.032	.006
11	.64	.150	.067	43.73	30.82	.032	.044	.007	.050	3.87	.032	.007
12	1.27	.710	.136	43.36	30.98							
13	.83	.340	.108	43.49	30.98							
14	.73	.210	.079	43.80	30.82	.030	.040	.006	.051	3.87	.032	.006
15	.61	.210	.065	44.25	30.82	.030	.050	.006	.047	3.88	.034	.006
16	.83	.340	.094	43.37	30.98							
17	.80	.300	.076	43.12	30.90							
AVERAGE	.67	.188	.073	43.89	30.82	.031	.044	.006	.058	3.78	.033	.006

ROCK COMPOSITE

	AS RECD	DRY BASIS
%BPL	64.15	65.10
%P2O5	29.36	29.79
%CaO	45.59	46.26
%MgO	1.80	1.83
%Fe2O3	1.32	1.34
%Al2O3	.86	.87
%Na2O	.57	.58
%K2O	.11	.11
%F	3.03	3.07
%Cl	.07	.07
%SO3	1.18	1.20
%CO2	6.58	6.68
%INSOL	5.23	5.31
% LOI	7.59	7.70
T SiO2	6.46	6.55
% H2O	1.45	

* SAMPLES AVERAGED AND USED FOR MATERIAL BALANCE

MASS BALANCE CALCULATIONS

4 CORNERS - LOW MgO FILE: FIPR3R4
ACIDULATION RUN4 11/13/93

ROCK			GRAMS ROCK =	100.00
%P205	32.32	.32		
%CaO	48.19	.48		
%SO3	1.22	.01		
PROD. ACID			GRAMS ACID =	113.22
%P205	27.77	.28		
%CaO	.05	.00		
%SO3	1.82	.02		
GYPSUM			GRAMS GYP. =	155.56
%P205	.56	.01		
%CaO	30.94	.31		
%SO3	44.48	.44		
SULFURIC ACID			GRAMS H2SO4 =	85.79

T H2SO4/T P205 (PRODUCED) 2.7

P205 IN THE GYPSUM INCLUDES BOTH SOL. AND INSOL. P205 LOSSES

COMPONENT BALANCE

	IN	OUT
P205	32.32	32.32
SO3	71.26	71.26
CO2	4.34	4.34
Fe2O3	1.35	1.35
Al2O3	.95	.95
CaO	48.19	48.19
MgO	.66	.66
Na2O	.60	.60
K2O	.12	.12
F	3.33	3.33
SiO2	5.24	5.24
OequivF	-1.40	-1.40
OTHER	200.02	200.02

MATERIAL BALANCE

------ I N ------ 4 CORNERS RUN No.4 ----- O U T -----

PHOSPHATE ROCK

	WEIGHT %	WEIGHT UNITS	% OF TOTAL
P205	32.32	32.32	100
SO3	1.22	1.22	1.71
CO2	4.34	4.34	100
Fe2O3	1.35	1.35	100
Al2O3	.95	.95	100
CaO	48.19	48.19	100
MgO	.66	.66	100
Na2O	.60	.60	100
K2O	.12	.12	100
F	3.33	3.33	100
SiO2	5.24	5.24	100
OequivF	-1.40	-1.40	
OTHER	3.09	3.09	
TOTAL	100.00	100.00	
FREE H2O	1.63	1.63	.90
TOTAL	101.63	101.63	

SULFURIC ACID

	WEIGHT %	WT. UNITS	% OF TOTAL
SO3		70.04	98.29
COMB H2O		15.76	
H2SO4	80.07		
FREE H2O	19.93	21.45	11.84
TOTAL	100.00	107.24	

WATER

	WEIGHT %	WT. UNITS	% OF TOTAL
H2O	100.00	158.09	87.26
TOTAL	100.00	158.09	

* CALCULATED VALUE

GAS

	CALC WT %	WT. UNITS	% REPORTING
STEAM*	86.45	29.14	16.09
CO2*	12.88	4.34	100.00
F*	.49	.17	5.00
SiO2*	.39	.13	2.51
OequvF	-.21	-.07	
TOTAL	100.00	33.71	

GYPSUM

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P205	.56	.87	2.70	
SO3	44.48	69.20	97.11	
Fe2O3	.03	.04	3.11	
Al2O3	.04	.06	6.25	
CaO	30.94	48.13	99.89	
MgO	.01	.01	1.29	
Na2O*	.28	.44	72.63	.049
K2O*	.03	.05	42.22	.006
F*	.32	.50	15.03	.058
SiO2*	2.77	4.31	82.35	2.45
OequvF	-.14	-.21		
OTHER	20.67	32.16	16.08	
TOTAL	100.00	155.56		
FREE H2O	41.44	64.47	35.58	
TOTAL	141.44	220.03		

PRODUCT ACID

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P205	27.77	31.45	97.30	
SO3	1.82	2.06	2.89	
Fe2O3*	1.16	1.31	96.89	1.21
Al2O3*	.78	.89	93.75	.80
CaO	.05	.05	.11	
MgO*	.58	.65	98.71	.55
Na2O	.15	.16	27.37	
K2O	.06	.07	57.78	
F	2.36	2.67	79.97	
SiO2	.70	.79	15.14	
OequvF	-.99	-1.12	-.56	
OTHER	65.58	74.25	37.12	
TOTAL	100.00	113.22		

FIPR ACIDULATION RUN4

FILE: FIPR3R4 11/13/93

29-H387-00

CHEMICAL ANALYSIS

SAMPLE NO. PRODUCT		%P2O5	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O	CONTROL %H2SO4	A. LAB %H2SO4
ACID #1		27.89	2.29									2.35	2.81
2		28.12	1.98									2.78	2.43
3		28.76	1.66									2.47	2.03
4		28.08	1.59									2.40	1.95
5		27.84	1.95									2.35	2.39
6		27.98	1.95									2.54	2.39
7		28.00	2.03									2.57	2.49
8		27.81	1.83									2.45	2.24
9	*	28.11	1.94	.03	1.19	.82	.55	2.28	.68	.12	.060	2.55	2.38
10		27.10	2.13									2.58	2.61
11		27.69	1.76									2.62	2.15
12		27.31	1.96									2.40	2.40
13	*	27.70	1.87	.05	1.19	.79	.55	2.28	.69	.16	.059	2.45	2.29
14	*	27.65	1.82	.05	1.20	.79	.54	2.38	.69	.15	.064	2.48	2.23
15	*	27.63	1.64	.05	1.24	.79	.54	2.48	.74	.15	.066	2.52	2.01
16		27.39	1.69									2.53	2.07
17		27.74	1.70									2.44	2.08
AVERAGE		27.77	1.82	.05	1.21	.80	.55	2.36	.70	.15	.062	2.50	2.23

SAMPLE NO.	%P2O5	% C.I. P2O5	% W.S. P2O5	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O
GYPSUM #1	.66	.330	.096	44.48	30.91							
2	1.30	.840	.175	44.23	31.24							
3	.69	.350	.144	44.64	31.07							
4	.55	.170	.095	44.07	31.07							
5	.53	.120	.073	44.52	30.74							
6	.67	.290	.102	44.45	30.91							
7	.71	.340	.114	44.35	31.07							
8	.53	.190	.089	44.38	31.07							
9	.50	.170	.068	44.59	31.07	.026	.038	.006	.062	2.50	.050	.006
10	.64	.280	.077	44.59	30.91							
11	.64	.280	.104	44.42	30.98							
12	.51	.190	.086	44.76	31.07							
13	.53	.140	.085	44.30	30.74	.026	.040	.006	.058	2.46	.050	.006
14	.49	.130	.071	44.50	30.98	.028	.038	.006	.054	2.41	.046	.006
15	.44	.100	.057	44.54	30.98	.028	.036	.004	.057	2.43	.048	.007
16	.51	.060	.051	44.98	30.98							
17	.46	.060	.053	43.54	30.98							
AVERAGE	.49	.135	.070	44.48	30.94	.027	.038	.006	.058	2.45	.049	.006

ROCK COMPOSITE

	AS RECD	DRY BASIS
%BPL	69.46	70.61
%P2O5	31.79	32.32
%CaO	47.40	48.19
%MgO	.65	.66
%Fe2O3	1.33	1.35
%Al2O3	.93	.95
%Na2O	.59	.60
%K2O	.12	.12
%F	3.28	3.33
%Cl	.08	.08
%SO3	1.20	1.22
%CO2	4.27	4.34
%INSOL	4.00	4.07
% LOI	5.39	5.48
% SiO2	5.15	5.24
% H2O	1.63	

* SAMPLES AVERAGED AND USED FOR MATERIAL BALANCE

MASS BALANCE CALCULATIONS

4 CORNERS - LOW MgO FILE: FIPR3R5
ACIDULATION RUN5 11/13/93

ROCK			GRAMS ROCK = 100.00
%P2O5	32.32	.32	
%CaO	48.19	.48	
%SO3	1.22	.01	
PROD. ACID			GRAMS ACID = 128.66
%P2O5	24.72	.25	
%CaO	.21	.00	
%SO3	1.81	.02	
GYPSUM			GRAMS GYP. = 155.22
%P2O5	.33	.00	
%CaO	30.87	.31	
%SO3	45.15	.45	
SULFURIC ACID			GRAMS H2SO4 = 87.21

T H2SO4/T P2O5 (PRODUCED) 2.7

P2O5 IN THE GYPSUM INCLUDES BOTH SOL. AND INSOL. P2O5 LOSSES

COMPONENT BALANCE

	IN	OUT
P2O5	32.32	32.32
SO3	72.41	72.41
CO2	4.34	4.34
Fe2O3	1.35	1.35
Al2O3	.95	.95
CaO	48.19	48.19
MgO	.66	.66
Na2O	.60	.60
K2O	.12	.12
F	3.33	3.33
SiO2	5.24	5.24
OequivF	-1.40	-1.40
OTHER	214.30	214.30

MATERIAL BALANCE

4 CORNERS RUN No.5

------ I N ------

-----------PHOSPHATE ROCK---------

	WEIGHT %	WEIGHT UNITS	% OF TOTAL
P2O5	32.32	32.32	100
SO3	1.22	1.22	1.68
CO2	4.34	4.34	100
Fe2O3	1.35	1.35	100
Al2O3	.95	.95	100
CaO	48.19	48.19	100
MgO	.66	.66	100
Na2O	.60	.60	100
K2O	.12	.12	100
F	3.33	3.33	100
SiO2	5.24	5.24	100
OequivF	-1.40	-1.40	
OTHER	3.09	3.09	
TOTAL	100.00	100.00	
FREE H2O	1.63	1.63	.84
TOTAL	101.63	101.63	

--------------SULFURIC ACID-----------

	WEIGHT %	WT. UNITS	% OF TOTAL
SO3		71.19	98.32
COMB H2O		16.02	
H2SO4	80.10		
FREE H2O	19.90	21.80	11.17
TOTAL	100.00	109.01	

----------------WATER-----------------

	WEIGHT %	WT. UNITS	% OF TOTAL
H2O	100.00	171.76	88.00
TOTAL	100.00	171.76	

* CALCULATED VALUE

----- O U T -----

--------------GAS--------------

	CALC WT %	WT. UNITS	% REPORTING
STEAM*	86.64	29.62	15.18
CO2*	12.69	4.34	100.00
F*	.49	.17	5.00
SiO2*	.38	.13	2.51
OequvF	-.21	-.07	
TOTAL	100.00	34.19	

-------------GYPSUM-------------

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P2O5	.33	.52	1.60	
SO3	45.15	70.08	96.78	
Fe2O3	.03	.04	3.16	
Al2O3	.04	.06	5.83	
CaO	30.87	47.91	99.43	
MgO	.01	.01	1.23	
Na2O*	.29	.45	74.79	.052
K2O*	.03	.04	35.66	.005
F*	.13	.20	5.96	.021
SiO2*	2.72	4.22	80.53	2.43
OequvF	-.05	-.08		
OTHER	20.47	31.78	14.83	
TOTAL	100.00	155.22		
FREE H2O	41.44	64.33	32.96	
TOTAL	141.44	219.55		

---------PRODUCT ACID----------

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P2O5	24.72	31.80	98.40	
SO3	1.81	2.33	3.22	
Fe2O3*	1.02	1.31	96.84	1.09
Al2O3*	.69	.89	94.17	.71
CaO	.21	.27	.57	
MgO*	.51	.65	98.77	.47
Na2O	.12	.15	25.21	
K2O	.06	.08	64.34	
F	2.31	2.97	89.04	
SiO2	.69	.89	16.96	
OequvF	-.97	-1.25	-.58	
OTHER	68.84	88.57	41.33	
TOTAL	100.00	128.66		

FIPR
ACIDULATION RUNS

FILE: FIPR3R5
11/13/93

29-H387-00

CHEMICAL ANALYSIS

SAMPLE NO. PRODUCT		%P205	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O	CONTROL %H2SO4	A. LAB %H2SO4
ACID #1		24.08	2.09									2.47	2.56
2		24.40	1.65									2.35	2.02
3		24.62	2.21									2.60	2.71
4		25.27	1.59									2.05	1.95
5		24.11	1.92									2.45	2.35
6		24.49	2.06									2.48	2.52
7		25.16	1.81									2.43	2.22
8		24.74	1.92									2.45	2.35
9		25.04	1.91									2.48	2.34
10	*	24.47	1.94	.25	.96	.70	.47	2.25	.66	.13	.059	2.50	2.38
11		24.88	1.85									2.41	2.02
12		25.09	1.80									2.45	2.20
13	*	24.32	2.07	.26	1.00	.70	.46	2.27	.69	.18	.070	2.52	2.53
14	*	25.28	1.62	.14	1.21	.73	.47	2.25	.67	.08	.060	2.40	1.99
15		25.40	1.63									2.45	2.00
16	*	24.79	1.61	.20	1.19	.72	.46	2.46	.74	.08	.055	2.45	1.97
17		25.07	1.58									2.40	1.93
AVERAGE		24.72	1.81	.21	1.09	.71	.47	2.31	.69	.12	.061	2.47	2.22

SAMPLE NO.	%P205	% C.I. P205	% W.S. P205	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O
GYPSUM #1	.39	.082	.039	44.88	31.01							
2	.36	.086	.033	44.95	31.01							
3	.34	.068	.034	45.46	31.01							
4	.36	.077	.038	44.85	31.10							
5	.36	.075	.031	44.90	30.85							
6	.40	.097	.035	44.97	31.18							
7	.34	.062	.032	45.07	30.68							
8	.31	.040	.026	45.28	31.01							
9	.31	.038	.030	45.26	31.10							
10	.33	.050	.018	45.04	30.93	.028	.040	.005	.023	2.40	.048	.005
11	.35	.048	.024	45.34	31.01							
12	.33	.042	.023	45.00	31.10							
13	.32	.033	.025	44.93	30.85	.028	.038	.004	.020	2.44	.054	.005
14	.30	.042	.022	45.17	30.68	.026	.036	.004	.015	2.49	.054	.005
15	.30	.051	.020	45.33	30.68							
16	.30	.028	.021	45.46	31.01	.028	.028	.008	.024	2.39	.050	.005
17	.32	.037	.017	45.57	30.85							
AVERAGE	.31	.038	.022	45.15	30.87	.028	.036	.005	.021	2.43	.052	.005

ROCK COMPOSITE

	AS RECD	DRY BASIS
%BPL	69.46	70.61
%P2O5	31.79	32.32
%CaO	47.40	48.19
%MgO	.65	.66
%Fe2O3	1.33	1.35
%Al2O3	.93	.95
%Na2O	.59	.60
%K2O	.12	.12
%F	3.28	3.33
%Cl	.08	.08
%SO3	1.20	1.22
%CO2	4.27	4.34
%INSOL	4.00	4.07
% LOI	5.39	5.48
T SiO2	5.15	5.24
% H2O	1.63	

* SAMPLES AVERAGED AND USED FOR MATERIAL BALANCE

M A S S B A L A N C E C A L C U L A T I O N S

4 CORNERS - MID MgO FILE: FIPR3R9
ACIDULATION RUN9 11/13/93

ROCK			GRAMS ROCK = 100.00
%P205	31.06	.31	
%CaO	47.23	.47	
%SO3	1.21	.01	
PROD. ACID			GRAMS ACID = 116.21
%P205	26.01	.26	
%CaO	.03	.00	
%SO3	1.81	.02	
GYPSUM			GRAMS GYP. = 154.92
%P205	.54	.01	
%CaO	30.46	.30	
%SO3	44.28	.44	
SULFURIC ACID			GRAMS H2SO4 = 85.14

T H2SO4/T P205 (PRODUCED) 2.8

P205 IN THE GYPSUM INCLUDES BOTH SOL. AND INSOL. P205 LOSSES

C O M P O N E N T B A L A N C E

	IN	OUT
P205	31.06	31.06
SO3	70.71	70.71
CO2	5.50	5.50
Fe2O3	1.35	1.35
Al2O3	.90	.90
CaO	47.23	47.23
MgO	1.24	1.24
Na2O	.59	.59
K2O	.12	.12
F	3.21	3.21
SiO2	5.89	5.89
OequivF	-1.35	-1.35
OTHER	203.51	203.51

M A T E R I A L B A L A N C E

------ I N ------ 4 CORNERS RUN No.9 ----- O U T -----

-----------PHOSPHATE ROCK---------

	WEIGHT %	WEIGHT UNITS	% OF TOTAL	ANALYZED %
P205	31.06	31.06	100	
SO3	1.21	1.21	1.71	
CO2	5.50	5.50	100	
Fe2O3	1.35	1.35	100	
Al2O3	.90	.90	100	
CaO	47.23	47.23	100	
MgO	1.24	1.24	100	
Na2O	.59	.59	100	
K2O	.12	.12	100	
F	3.21	3.21	100	
SiO2	5.89	5.89	100	5.89
OequivF	-1.35	-1.35		
OTHER	3.04	3.04		
TOTAL	100.00	100.00		
FREE H2O	1.54	1.54	.83	
TOTAL	101.54	101.54		

--------------SULFURIC ACID-----------

	WEIGHT %	WT. UNITS	% OF TOTAL
SO3		69.50	98.29
COMB H2O		15.64	
H2SO4	80.84		
FREE H2O	19.16	21.28	11.52
TOTAL	100.00	106.42	

----------------WATER-----------------

	WEIGHT %	WT. UNITS	% OF TOTAL
H2O	100.00	162.01	87.65
TOTAL	100.00	162.01	

* CALCULATED VALUE

--------------GAS--------------

	CALC WT %	WT. UNITS	% REPORTING
STEAM*	83.48	28.92	15.65
CO2*	15.89	5.50	100.00
F*	.46	.16	5.00
SiO2*	.37	.13	2.15
OequvF	-.20	-.07	
TOTAL	100.00	34.65	

-------------GYPSUM-------------

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P205	.54	.83	2.67	
SO3	44.28	68.60	97.02	
Fe2O3	.01	.02	1.51	
Al2O3	.03	.05	5.69	
CaO	30.46	47.19	99.92	
MgO	.01	.01	.85	
Na2O*	.24	.37	62.62	.031
K2O*	.04	.06	49.56	.008
F*	.33	.51	15.92	.065
SiO2*	3.25	5.03	85.35	3.392
OequvF	-.14	-.22		
OTHER	20.95	32.46	15.95	
TOTAL	100.00	154.92		
FREE H2O	41.44	64.20	34.73	
TOTAL	141.44	219.12		

---------PRODUCT ACID----------

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P205	26.01	30.23	97.33	
SO3	1.81	2.11	2.98	
Fe2O3*	1.14	1.33	98.49	1.13
Al2O3*	.73	.85	94.31	.71
CaO	.03	.04	.08	
MgO*	1.06	1.23	99.15	1.12
Na2O	.19	.22	37.38	
K2O	.05	.06	50.44	
F	2.18	2.54	79.08	
SiO2	.63	.74	12.50	
OequvF	-.92	-1.07	-.53	
OTHER	67.06	77.93	38.29	
TOTAL	100.00	116.21		

FIPR　　　　FILE: FIPR3R9
ACIDULATION RUN9　　　　11/13/93

29-H387-00　　　　CHEMICAL ANALYSIS

SAMPLE NO. PRODUCT		%P2O5	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O	CONTROL %H2SO4	A. LAB %H2SO4
ACID #1		25.80	1.94									2.65	2.38
2		25.98	2.14									2.85	2.62
3		26.03	1.80									2.48	2.20
4		25.80	1.68									2.53	2.06
5		26.11	1.72									2.50	2.11
6		26.15	1.44									2.40	1.77
7		25.70	1.66									2.52	2.03
8		25.70	1.56									2.45	1.91
9		26.33	1.53									2.39	1.88
10		26.38	1.86									2.55	2.28
11	*	26.36	1.93	.040	1.17	.73	1.11	2.20	.65	.23	.053	2.57	2.36
12	*	26.56	1.77	.020	1.15	.71	1.13	2.20	.63	.16	.052	2.55	2.17
13	*	25.89	1.76	.030	1.12	.72	1.13	2.18	.61	.18	.048	2.55	2.16
14	*	25.25	1.72	.040	1.08	.70	1.09	2.16	.65	.20	.052	2.60	2.11
15	*	25.98	1.89	.030	1.11	.70	1.12	2.18	.63	.18	.047	2.56	2.31
16		25.71	1.88									2.56	2.30
17		25.62	1.93									2.56	2.37
AVERAGE		26.01	1.81	.032	1.13	.71	1.12	2.18	.63	.19	.050	2.57	2.22

SAMPLE NO.	%P2O5	% C.I. P2O5	% W.S. P2O5	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O
GYPSUM #1	.46	.082	.033	45.05	30.51							
2	.57	.141	.057	44.73	30.85							
3	.50	.055	.042	44.76	30.68							
4	.57	.064	.030	44.52	30.43							
5	.52	.073	.028	44.49	30.51							
6	.50	.050	.029	44.59	30.51							
7	.50	.045	.026	44.92	30.51							
8	.50	.045	.023	44.56	30.43							
9	.55	.045	.022	44.71	30.51							
10	.48	.055	.028	44.20	30.43							
11	.52	.091	.038	44.08	30.43	.020	.040	.008	.059	3.44	.040	.008
12	.50	.132	.043	44.38	30.43	.012	.030	.008	.068	3.41	.030	.006
13	.50	.114	.042	44.42	30.43	.010	.030	.008	.068	3.40	.030	.014
14	.50	.105	.044	44.01	30.51	.012	.030	.005	.068	3.41	.027	.008
15	.46	.110	.034	44.52	30.51	.012	.036	.005	.060	3.30	.026	.006
16	.52	.110	.039	44.38	30.43							
17	.52	.100	.048	44.06	30.43							
AVERAGE	.50	.110	.040	44.28	30.46	.013	.033	.007	.065	3.39	.031	.008

ROCK COMPOSITE

	AS RECD	DRY BASIS
%BPL	66.82	67.86
%P2O5	30.58	31.06
%CaO	46.50	47.23
%MgO	1.23	1.24
%Fe2O3	1.33	1.35
%Al2O3	.89	.90
%Na2O	.58	.59
%K2O	.11	.12
%F	3.16	3.21
%Cl	.07	.07
%SO3	1.19	1.21
%CO2	5.42	5.50
%INSOL	4.63	4.70
% LOI	6.48	6.59
T SiO2	5.80	5.89
% H2O	1.54	

* SAMPLES AVERAGED AND USED FOR MATERIAL BALANCE

MASS BALANCE CALCULATIONS
4 CORNERS - HIGH MgO FILE: IPR3R11
ACIDULATION RUN3 11/13/93

ROCK GRAMS ROCK = 100.00

%P205	29.79	.30
%CaO	46.26	.46
%SO3	1.20	.01

PROD. ACID GRAMS ACID = 117.46

%P205	24.71	.25
%CaO	.10	.00
%SO3	1.84	.02

GYPSUM GRAMS GYP. = 152.91

%P205	.50	.01
%CaO	30.18	.30
%SO3	43.79	.44

SULFURIC ACID GRAMS H2SO4 = 83.21

T H2SO4/T P205 (PRODUCED) 2.9

P205 IN THE GYPSUM INCLUDES BOTH SOL. AND INSOL. P205 LOSSES

COMPONENT BALANCE

	IN	OUT
P205	29.79	29.79
SO3	69.12	69.12
CO2	6.68	6.68
Fe203	1.34	1.34
Al203	.87	.87
CaO	46.26	46.26
MgO	1.83	1.83
Na20	.58	.58
K20	.11	.11
F	3.07	3.07
SiO2	6.55	6.55
OequivF	-1.29	-1.29

MATERIAL BALANCE

------ I N ------ 4 CORNERS RUN No.11 ----- O U T -----

-----------PHOSPHATE ROCK---------

	WEIGHT %	WEIGHT UNITS	% OF TOTAL
P205	29.79	29.79	100
SO3	1.20	1.20	1.73
CO2	6.68	6.68	100
Fe203	1.34	1.34	100
Al203	.87	.87	100
CaO	46.26	46.26	100
MgO	1.83	1.83	100
Na20	.58	.58	100
K20	.11	.11	100
F	3.07	3.07	100
SiO2	6.55	6.55	100
OequivF	-1.29	-1.29	
OTHER	3.01	3.01	
TOTAL	100.00	100.00	
FREE H20	1.45	1.45	.78
TOTAL	101.45	101.45	

--------------SULFURIC ACID-----------

	WEIGHT %	WT. UNITS	% OF TOTAL
SO3		67.92	98.27
COMB H20		15.28	
H2SO4	79.86		
FREE H20	20.14	20.80	11.20
TOTAL	100.00	104.01	

----------------WATER-----------------

	WEIGHT %	WT. UNITS	% OF TOTAL
H20	100.00	163.43	88.02
TOTAL	100.00	163.43	

* CALCULATED VALUE

--------------GAS--------------

	CALC WT %	WT. UNITS	% REPORTING
STEAM*	80.41	28.27	15.22
CO2*	18.99	6.68	100.00
F*	.44	.15	5.00
SiO2*	.35	.12	1.85
OequvF	-.18	-.06	
TOTAL	100.00	35.15	

-------------GYPSUM-------------

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P205	.50	.77	2.57	
SO3	43.79	66.96	96.88	
Fe203	.01	.02	1.48	
Al203	.02	.03	3.59	
CaO	30.18	46.14	99.74	
MgO	.004	.01	.33	
Na20*	.28	.43	73.59	.038
K20*	.03	.04	35.01	.003
F*	.21	.32	10.38	.015
SiO2*	3.34	5.11	77.99	3.78
OequvF	-.09	-.13		
OTHER	21.73	33.22	16.29	
TOTAL	100.00	152.91		
FREE H20	41.44	63.37	34.13	
TOTAL	141.44	216.28		

---------PRODUCT ACID----------

	WEIGHT %	WT. UNITS	% REPORTING	ANALYZED %
P205	24.71	29.03	97.43	
SO3	1.84	2.16	3.12	
Fe203*	1.12	1.32	98.52	1.15
Al203*	.72	.84	96.41	.76
CaO	.10	.12	.26	
MgO*	1.55	1.82	99.67	1.55
Na20	.13	.15	26.41	
K20	.06	.07	64.99	
F	2.22	2.60	84.62	
SiO2	1.13	1.32	20.16	
OequvF	-.93	-1.10	-.54	
OTHER	67.36	79.12	38.79	

FIPR FILE: FIPR3R11
ACIDULATION RUN 11 11/13/93

29-H387-00 CHEMICAL ANALYSIS

SAMPLE NO. PRODUCT		%P2O5	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O	CONTROL %H2SO4	A. LAB %H2SO4
ACID #1		24.38	1.17									1.80	1.43
2		24.03	2.02									2.70	2.48
3		23.80	1.68									2.51	2.06
4		24.37	1.70									2.59	2.08
5		24.33	1.79									2.55	2.19
6		24.51	1.58									2.30	1.93
7		24.37	1.61									2.45	1.97
8		25.04	1.75									2.43	2.14
9	*	24.52	1.87	.10	1.15	.76	1.55	2.19	1.30	.133	.060	2.50	2.29
10	*	24.81	1.83	.11	1.15	.75	1.55	2.24	.84	.125	.060	2.44	2.24
11	*	24.95	1.93	.10	1.15	.77	1.55	2.26	1.34	.147	.064	2.53	2.36
12	*	24.57	1.72	.10	1.14	.75	1.55	2.17	1.02	.118	.060	2.56	2.11
13		25.20	1.71									2.65	2.09
14		24.71	1.56									2.46	1.91
15		25.20	1.25									2.00	1.53
16		24.49	1.44									2.01	1.76
17		24.31	1.30									2.13	1.59
18		24.53	1.22									2.27	1.50
19		24.67	1.23									2.21	1.51
AVERAGE		24.71	1.84	.10	1.15	.76	1.55	2.22	1.13	.131	.061	2.51	2.25

SAMPLE NO.	%P2O5	% C.I. P2O5	% W.S. P2O5	%SO3	%CAO	%Fe2O3	%Al2O3	%MgO	%F	%SiO2	%Na2O	%K2O
GYPSUM #1	.61	.032	.035	44.01	30.26							
2	.52	.041	.029	44.28	30.26							
3	.50	.064	.029	44.01	30.09							
4	.52	.045	.026	43.41	30.09							
5	.48	.032	.026	43.96	30.26							
6	.43	.023	.025	44.37	30.42							
7	.46	.036	.026	44.21	30.26							
8	.48	.029	.023	43.77	30.42							
9	.46	.032	.028	44.26	30.26	.012	.020	.003	.005	3.80	.034	.003
10	.46	.033	.030	43.77	30.26	.016	.020	.004	.012	3.70	.041	.003
11	.48	.058	.035	43.70	30.09	.012	.022	.004	.024	3.82	.043	.003
12	.48	.050	.030	43.44	30.09	.012	.020	.004	.019	3.78	.034	.003
13	.50	.042	.031	42.79	30.09							
14	.50	.038	.025	42.89	30.09							
15	.60	.033	.026	43.96	30.09							
16	.71	.033	.022	43.66	30.26							
17	.62	.033	.022	43.06	30.09							
18	.65	.046	.029	43.63	30.26							
19	.65	.050	.031	43.46	30.26							
AVERAGE	.47	.043	.031	43.79	30.18	.013	.021	.004	.015	3.78	.038	.003

ROCK COMPOSITE

	AS RECD	DRY BASIS
%BPL	64.15	65.10
%P2O5	29.36	29.79
%CaO	45.59	46.26
%MgO	1.80	1.83
%Fe2O3	1.32	1.34
%Al2O3	.86	.87
%Na2O	.57	.58
%K2O	.11	.11
%F	3.03	3.07
%Cl	.07	.07
%SO3	1.18	1.20
%CO2	6.58	6.68
%INSOL	5.23	5.31
% LOI	7.59	7.70
T SiO2	6.46	6.55
% H2O	1.45	

* SAMPLES AVERAGED AND USED FOR MATERIAL BALANCE

TABLE 4.4
Solubility of Impurities

Test	Fe_2O_3	Al_2O_3	MgO
3	96.5	92.5	99.5
4	96.9	93.8	98.7
5	96.8	94.2	98.8
9	98.5	94.3	99.2
11	98.5	96.4	99.7

The acid MER ratios are shown in the following table. It is doubtful that acid at 0.113 MER would make 18-46-0 or 11-52-0 since this ratio is well above the 0.10 MER limit normally considered a maximum level for impurities.

TABLE 4.5
Acid MER Ratios

Run	Rock MgO	P_2O_5	Fe_2O_3	Al_2O_3	MgO	MER
3	1.83	28.03	1.26	0.79	1.78	0.137
4	0.66	27.77	1.16	0.78	0.58	0.091
5	0.66	24.72	1.02	0.69	0.51	0.090
9	1.24	26.01	1.14	0.73	1.06	0.113
11	1.83	24.71	1.12	0.72	1.55	0.137

4.5 Addendum Test Run (AR-1)

The results of the first 11 phosphoric acid pilot plant runs showed that high levels of MgO penalized both recovery of P_2O_5 and filtration rates. These effects are depicted in Figures 4.2.3, 4.2.4 and 4.2.5.

A plan was developed to reduce losses and improve filtration by using a two stage attack system where a normal to high sulfate was to be used in Reactor

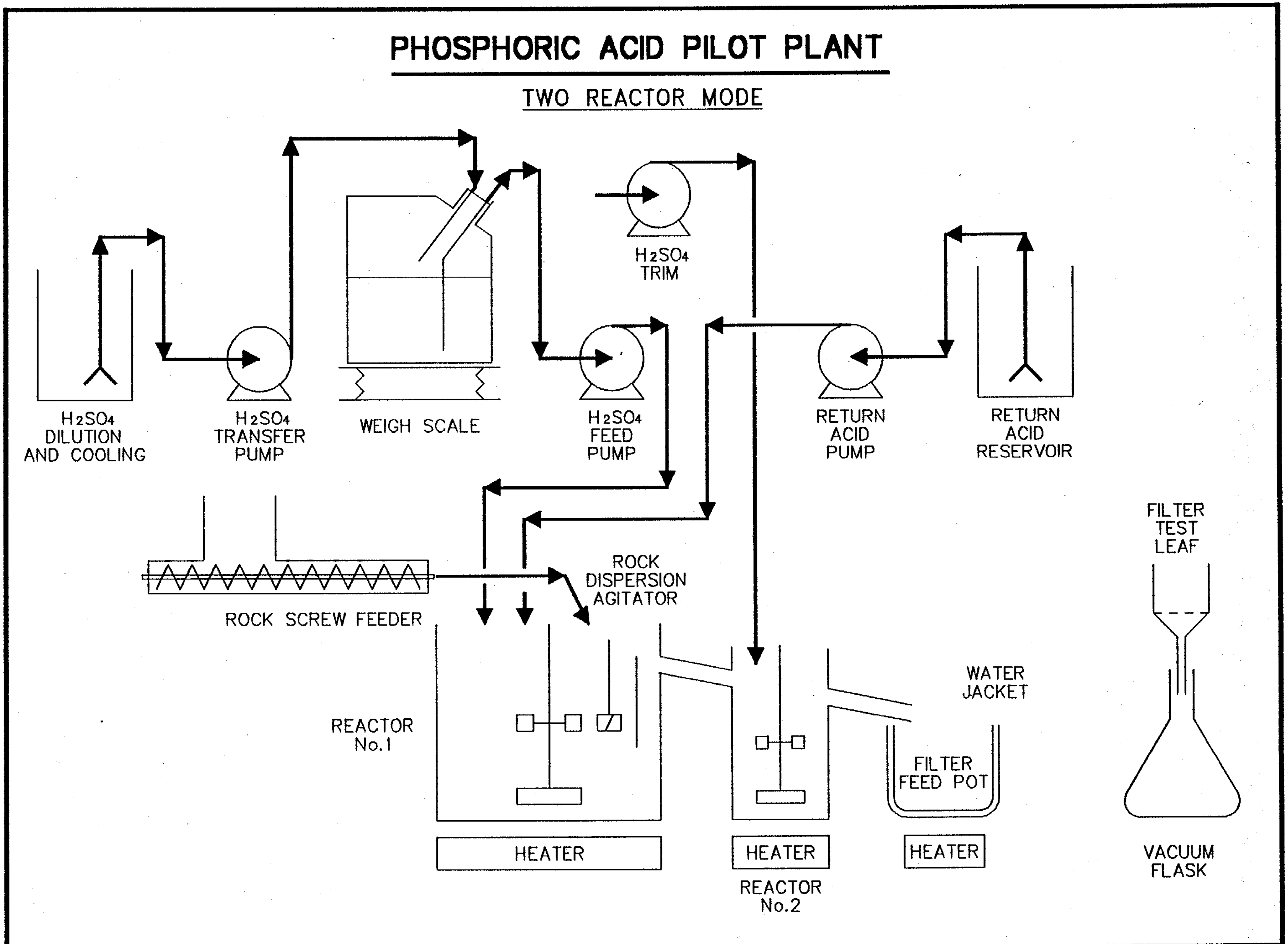
No. 1 and a higher yet sulfate in Reactor No. 2. Initial goals were set at 2.5% in Reactor No. 1 and 3.0% in Reactor No. 2. The theory here is that maintaining as high a sulfate driving force as possible through the system would dissolve the high magnesium mineral and reduce losses. A pilot plant run designated as Addendum Run No. 1 (AR-1) was carried out to see if benefits could be achieved.

Test Run AR-1 employed a modified reactor configuration consisting of a post treatment vessel downstream of the main reactor. This is shown in Figure 4.5.1. The test was set up to duplicate Runs 2, 7 and 9, using intermediate level MgO in the rock (1.2%), intermediate acid strength, 26.0% P_2O_5, and 4 hours detention. In the AR-1 configuration, the first reactor provided 3 hours detention, the second vessel 1.0 hour, The run was split into several target sulfates. The first 56 hours used a target of 2.5% H_2SO_4 in the first reactor, 3.0% in the second. From hour 56 to hour 80, the target sulfates were 2.5% and 3.5%. This was followed by 2.2% and 3.0% at hour 82 to the end of the test at hour 109.

However, conditions did not steady out at this latter level until about hour 100. From hour 89 to the end of the run at hour 109, the goal was 2.2% in Reactor 1, but several hours were above this so that only the samples from 100 hours on proved to be optimum.

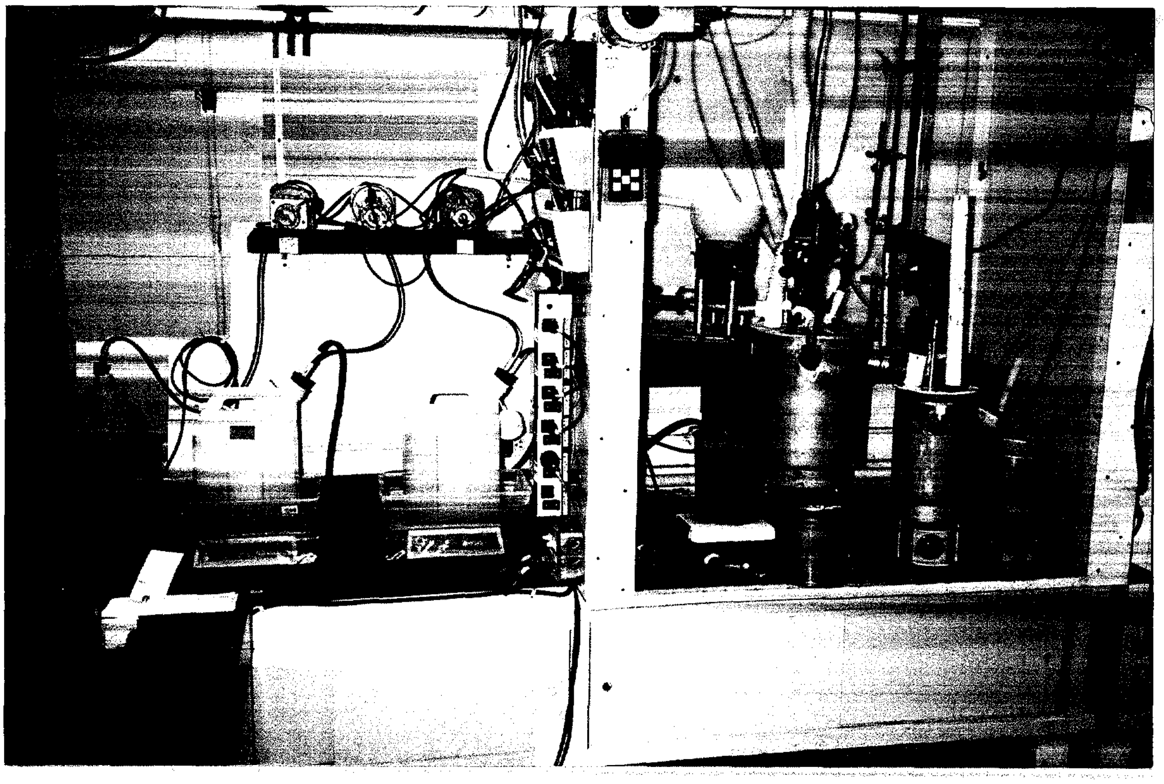
4.5.2 Test Results

The 1.2% MgO rock continued to show sensitivity to coating and most of the run proved to be a struggle to maintain control in the first pot at 2.5% H_2SO_4. Both citrate insolubles and citrate soluble losses were higher than control tests 2,7 and 9 for most of the run at the target sulfates. Only at the very end when the lower sulfates in NO. 1 Reactor were stabilized did the system return to the performance demonstrated in Runs 2, 7 and 9 where the whole 4 hours of retention were provided in the single reactor vessel. This is shown by the square located on Figure 4.5.2.



H387001B

Figure 4.5.1



Phosphoric Acid Pilot Plant
Two Reactor Configuration

Figure 4.5.1.1

There are indications that the citrate insoluble P_2O_5 for the end of Test AR-1 was slightly lower than for Test 9. In Tests 2, 7 and 9, there were no results as low as 0.13% citrate insoluble P_2O_5 as % of P_2O_5 fed. The range was more like 0.25% to 0.50% c.i. P_2O_5 loss.

The small improvement indicated in citrate insoluble loss is the only benefit that can be seen for the second reactor addition. While there may well be benefits to gypsum crystallization and filtration, they were not demonstrated in these tests, primarily due to control difficulties described in the next section.

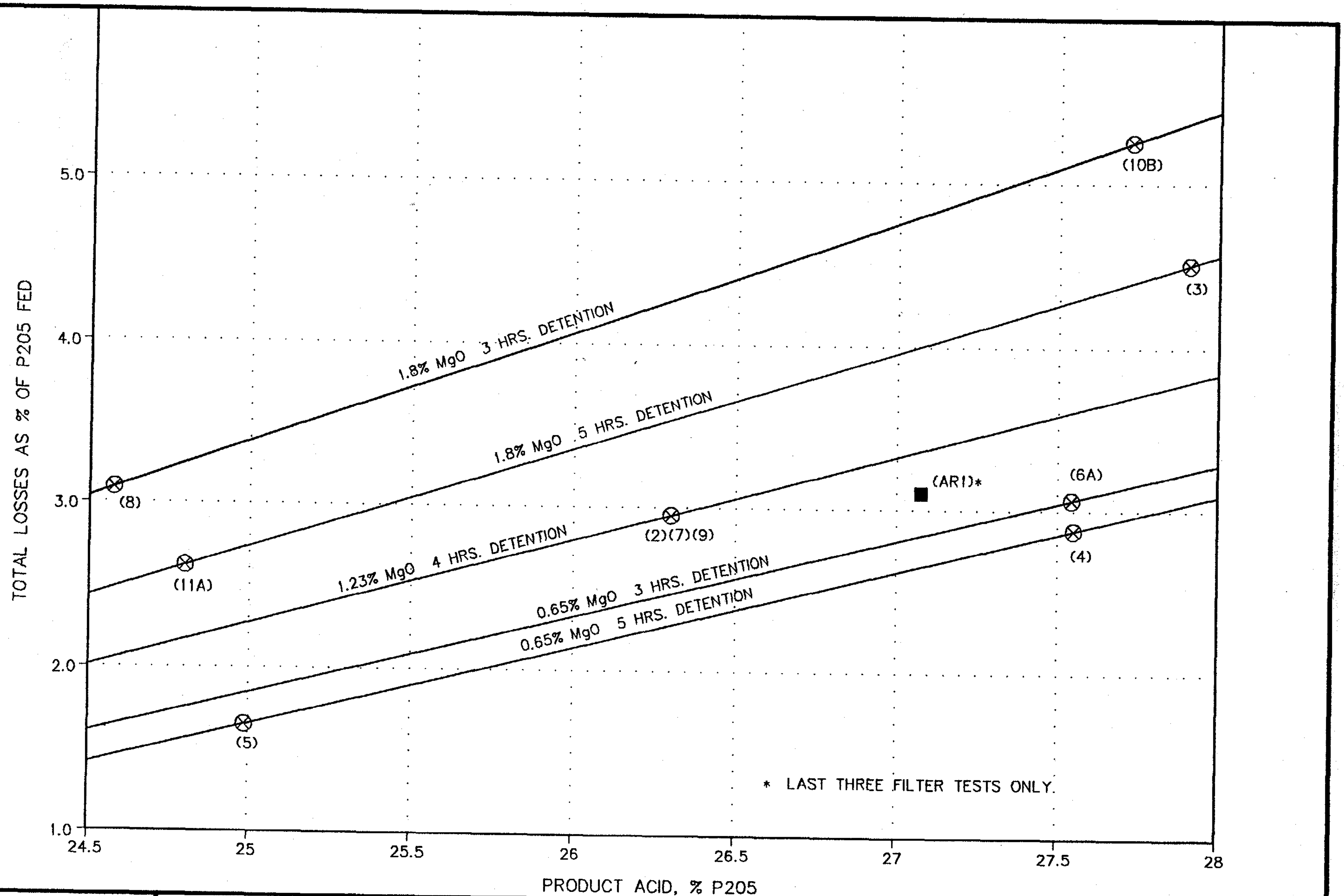
The chronological phosphoric acid pilot plant log, Figure 4.5.3, shows the two sulfate levels in Reactors No. 1 and No. 2 on the same graph. In the first 40 hours, two high sulfates occurred in the first reactor. In many of the previous runs, we were able to handle a 2.5% H_2SO_4 as the target at 3 hours retention. We didn't manage it in this test. It appears that the target for Reactor 1 should have been between 2.0% and 2.2% H_2SO_4.

The results of the last three filter test analyses are shown in Table 4.5.4. This table also gives the analysis of the gypsum in Reactor No. 1 for the last three samples.

The citrate insolubles indicate a level of sulfate sensitivity intermediate between the 0.65 MgO rock and the 1.6 MgO rock at 3 hours retention in the previous tests.

Table 4.5.4
Run AR-1 - Losses at End of Test

Filter Test No.	Hour	Acid % P_2O_5	Losses % of P_2O_5 Fed: Reactor No. 2 c.s.	Reactor No. 2 c.i.	Reactor No. 2 w.s.	Reactor No. 2 Total	Reactor No. 1 c.s.	Reactor No. 1 c.i.
16	102	27.9	2.88	0.30	0.40	3.58	2.82	0.84
17	105	27.1	2.71	0.13	0.33	3.17	2.83	0.59
18	109	26.4	2.13	0.13	0.33	2.59	2.55	0.44
Average		27.2	2.57	0.19	0.35	3.11	2.73	0.62



PN:	29-H387-00
Prepared By:	DWL/CMP
Date:	12-1-93
Ref:	H387002A

( ) DENOTES TEST RUN NO.
NOTE: ALL TESTS EXCEPT E, F & No.1.

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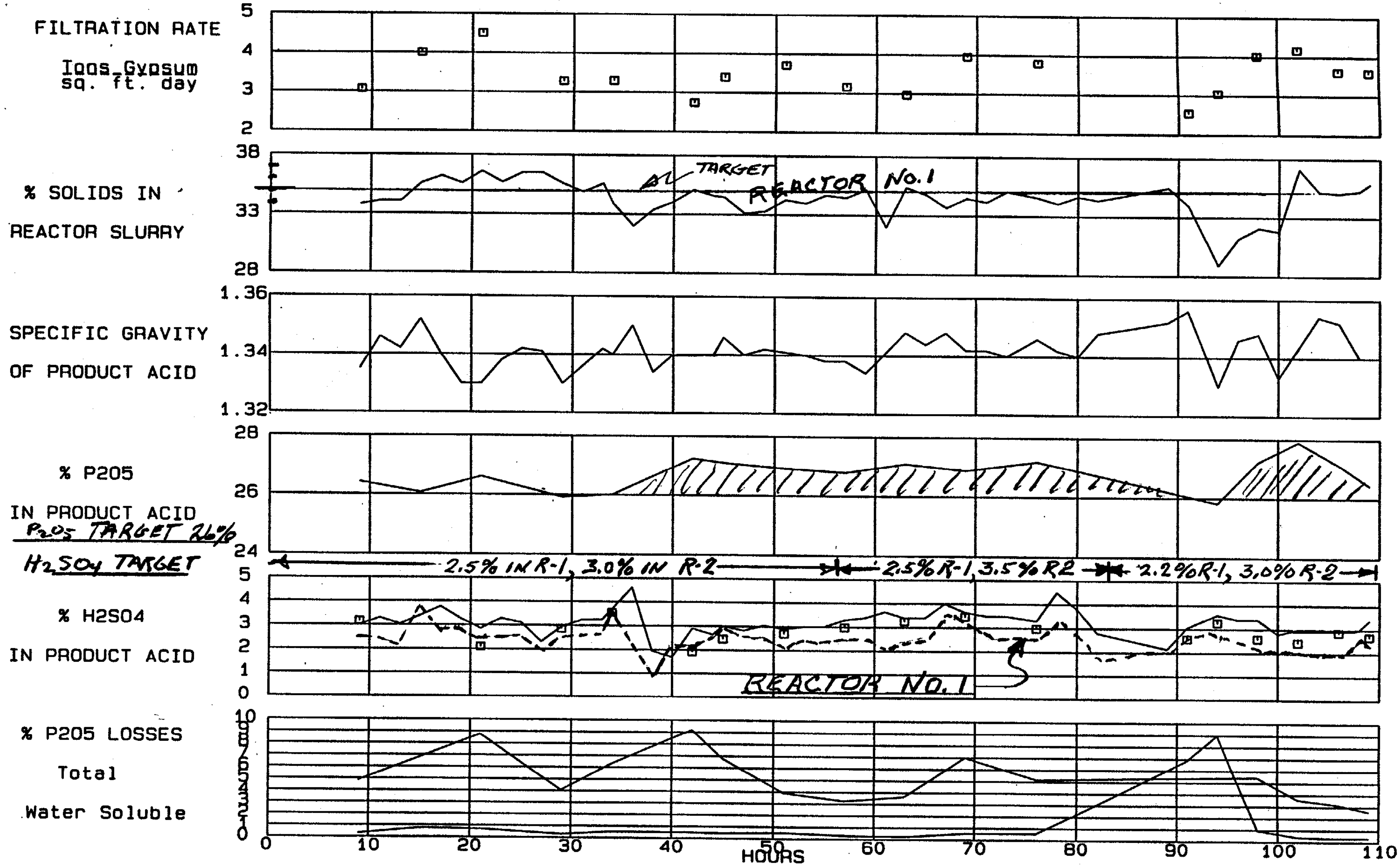
LAKELAND FLORIDA

Figure 4.5.2
TOTAL LOSSES VS % P205 IN ACID

PHOSPHORIC ACID PILOT PLANT DATA LOG ROCK TESTED: FIPR AR1: REACTOR 2



A further factor which contributed to poor control was the relatively high product acid strength, as shown in the shaded area of Figure 4.5.3. The strength during the last three tests was closer to 27.5% P_2O_5 than it was to the target 26.0% P_2O_5 as shown in Figure 4.5.2.

4.5.3 Filter Tests - Run AR-1

Filter test results for AR-1 were well below expectations and below the control tests Runs 2, 7 and 9 which gave consistent results between themselves and correlated well with the other tests. Test 2 averaged 0.9 T P_2O_5/sq. ft/day, Test 7,0.88 T P_2O_5/sq. ft/day and Test 9, 0.87 T P_2O_5/sq. ft/day.

Test AR-1 ran 0.65 to 0.74 T P_2O_5 per sq. ft/day. The gypsum appeared normal and the routine filtration appeared adequate but two mechanical problems may have existed. It was recognized about halfway through the run that the weights of filter cake from the normal 900 grams of slurry were yielding only 28% or 29% solids feed to the tests. Some samples were even less than 27% solids.

One apparent cause for this difficulty was the chimney baffle in the overflow of No. 1 Reactor. Apparently it allowed a settling of solids to take place such that the first reactor had the correct percent solids or nearly so, about 35% solids, while the second reactor had only 28% or 29% solids. The No. 1 Reactor agitator was sped up but this allowed some splashover from the first reactor. There was not time or means available to shorten the baffle which probably would have solved the problem.

In addition, there appears to be a sampling problem which may also be contributing to the lesser solids in the feed to the filter test. This was compounded by the fact that the operators tended to run a low percent solids in this test, generally under 35% solids versus 35% to 40% solids, a more normal figure for Florida rock.

A further factor in the execution of the test was the extra burden on the operators imposed by running two sulfate controls. Also, the higher strength of product acid during most of the run certainly contributed to lower filtration rates. As a result of these difficulties, the filtration rates are reported only in the Appendix, and not included in the data plotted in Figure 4.2.4.

SECTION 5.0

5.0 STATISTICAL EVALUATION OF DATA

5.1 TEST DESIGN VARIABLES

The term variables here covers feed, digestions conditions and recoveries. The three types of variables and target levels for this design were:

- FIXED VARIABLES
  - -80% Sulfuric acid to be used as feed.
  - -Tap water to be used as process water.
  - -35% solids in digestion slurry.
  - -2.5% soluble sulfate in digestion slurry.
  - -Agitation levels to be constant for tests.

- CONTROLLED VARIABLES
  - -MgO content of feed rock was varied from 0.65, 1.23, and 1.8.
  - -Concentration of P2O5 in filtrate ie. digestion liquor was varied in the tests from 24.5 to 26.0 and 27.5.
  - -Digestion time was controlled at 3, 4, and 5 hours. The reaction volume per ton of P2O5 produced per day represented by these digestion times are approximately 1.1, 1.4 and 1.8 cubic Meters. Pierre Becker in his book "Phosphates and Phosphoric Acid" states that it is common for di-Hydrate plants to be designed with reaction volumes of 1.5-1.8 cubic Meters per ton of P2O5.

- INDEPENDENT VARIABLES
  - -These are the chemical Recoveries, Sulfuric acid consumption, Filter capacity for tons of acid P2O5/day/ft2 and tons Gypsum/day/ft2 as well as tons of gyp produced per ton of acid P2O5.

5.2 CHOOSING THE DATA FOR EVALUATION

The total test data developed by Jacobs for each run had too much variation as is normally the case with plant data for direct use. This body of data however when separated in individual filtration periods had long periods of operation suitable for statistical evaluation. It was reasoned that the filtration characteristics displayed ie. recoveries and rate were the result of the digestion conditions prior to the filtration. Digestion data for a period four times the test retention time prior to the filtration was chosen. About 93-94% of the gypsum being filtered would have been formed in this period.

Stability of operation during this period as evidenced by the soluble sulfate levels standard of deviation and proximity to the target level of 2.5 (log sheet data) was used as the criteria for use as evaluation data. Contiguous filtration periods displaying close to the target levels of soluble sulfate and low deviations were then average to obtain the data used for the evaluations.

Figure V-l shows Test 2 data summarized prior to choosing the 20 hour to 56 hour period data as representative for that set of controlled variables.

All the Tests were summarized like this. Copies of the summaries for all tests are contained in Appendix B of this report. The data selected for the evaluations of the Digestion variables and the calculated major effects are given in Table V-l.

The table is arranged with the controlled variables listed as in the test design. The Test-Period column shows the order in which they were run and the hours of operation chosen for the data evaluation.

TABLE V-1

DIGESTION DATA EVALUATED

		VARIABLES								
		Controlled			Independent					
Test		Filt		Reten	Ov'll	Gypsum			T H2SO4	
#	Period	% MgO	P2O5	time	Recov	CI	CS	WS	/T P2O5	
1	26/32 h	0.65	25.33	3	97.74	0.34	1.80	0.16	2.68	
8	14/86 h	1.80	24.63	3	96.82	0.36	2.71	0.15	2.76	
6	44/92 h	0.65	27.38	3	96.87	0.35	2.61	0.18	2.61	
10	32/50 h	1.80	27.09	3	93.51	2.31	3.63	0.60	2.83	
5	20/95 h	0.65	24.78	5	98.28	0.25	1.35	0.12	2.61	
11	25/67 h	1.80	24.64	5	97.47	0.18	2.24	0.14	2.70	
4	74/95 h	0.65	27.62	5	97.36	0.47	1.87	0.30	2.61	
3	62/95 h	1.80	27.92	5	95.47	1.47	2.48	0.43	2.78	
2	20/56 h	1.23	25.90	4	97.20	0.46	2.12	0.23	2.71	
7	15/75 h	1.23	26.11	4	97.27	0.41	2.13	0.19	2.69	
9	26/62 h	1.23	26.02	4	97.33	0.27	2.34	0.14	2.69	

5.3 CALCULATED MAJOR EFFECTS-DIGESTION

	FILTRATE		RETEN	VARIANCE
	% MgO	%P2O5	TIME	REPLICATES
OVERALL RECOVERY	-1.75	-1.78	0.91	0.0028
LOSSES-GYPSUM CI	0.73	0.87	-0.25	0.0064
GYPSUM CS	0.86	0.62	-0.70	0.0101
GYPSUM WS	0.14	0.23	-0.02	0.0014
Tons H2SO4/Ton Acid P2O5	0.14	0.03	-0.05	0.0001

This data applies to the variations of the controlled variables within the range tested and of course to the single reactor configuration. The effects will probable be present in any multi-compartment reactors but the magnitude of the effects will differ. The influence of fluorine containing process water normally used by commercial operations on filtration recoveries was not investigated in this series of tests. Test filtration recoveries therefore are significantly better than one would expect in normal plant operation. Calculation of the above major effects by assuming normal filtration recoveries would reduce overall recoveries more, increase water soluble P2O5 losses and sulfuric acid consumption.

The variances obtained in the replicated tests are small when compared with the major effects and indicate that the effects shown are significant and real.

OVERALL RECOVERY - The data shows that increasing MgO content and the filtrate P2O5 concentration will result in lower recoveries. Increasing retention time educing the production rate in a continuous system will increase recovery.

The recovery data from Table V-l is plotted graphically in the Figure V-2. The scatter of points is caused by the attempt to plot the actual Filtrate concentrations not the targets. The increase in recovery by increasing digestion time from three to five hours is evident.

A recovery contour was calculated for the seven tests that had 4 and 5 hours digestion times. The result is presented in Figure V-3. This figure shows the curvature of the data caused by the effect of the Filtrate concentration. The filtrate scale is not accurate but the actual spacing of the data is therefore the surface calculated is believed accurate.

GYPSUM CAKE LOSSES - The data shows increasing MgO content and the filtrate P2O5 concentration increases CI, CS and WS P2O5 losses.

As one might expect increasing the filtrate P2O5 content results in higher WS losses. The increased recovery shown by the increased retention time is primarily because of the decrease in the gyp cake's CS P2O5 content.

SULFURIC ACID CONSUMPTION - Could be increased by as much as 8.4% from 2.61 t/t of acid P2O5 for the .065% MgO rock to 2.83 t/t for the 1.8% MgO rock. This would occur with a 3 hour digestion time producing a 27.5% Filtrate.

Data selected for the evaluation of filtration and filter capacity are presented in the following Table IV-2. The data is arranged as in the previous Table V-l.

FILTER CAPACITY - The increased MgO content (0.65-1.8) results in a 14.5% drop in acid filtration rate. The filter rate per foot square would drop from 0.90 to 0.77 tons of acid P205. The Gyp cake capacity would also be reduced from 4.41 to 4.09 tons of gyp per square foot per day a drop of 7.3%. Increasing filtrate strength causes a similar drop in filter capacity. Increasing retention time would negate some of the lost capacity but would cause a greater economic effect because of the decrease in the production rate.

GYPSUM STACK REQUIREMENTS-The higher MgO containing rock on average would increase the production of gypsum by 8.7%. An additional 1-2% increase in gypsum production is indicated if the filtrate in maintained at the normal industry target of 27.5% P2O5.

TABLE V-2

FILTRATION DATA EVALUATED

VARIABLES

Test #	Time Period	Controlled %MgO	P2O5	Retent Time	Independent t Acid P2O5 /day/ft2	t Gyp/ day/ft2	t Gyp/ t P2O5
1	26/32 h	0.65	25.33	3	0.89	4.35	4.89
8	14/86 h	1.80	24.63	3	0.79	4.16	5.26
6	44/92 h	0.65	27.38	3	0.86	4.28	5.00
10	32/50 h	1.80	27.09	3	0.63	3.49	5.58
5	20/95 h	0.65	24.78	5	0.95	4.64	4.91
11	25/67 h	1.80	24.64	5	0.86	4.51	5.36
4	74/95 h	0.65	27.62	5	0.88	4.35	4.95
3	62/95 h	1.80	27.92	5	0.79	4.19	5.28
2	20/56 h	1.23	25.90	4	0.86	4.40	5.11
7	15/75 h	1.23	26.11	4	0.88	4.47	5.07
9	26/62 h	1.23	26.02	4	0.86	4.40	5.11

5.4 CALCULATED MAJOR EFFECTS-FILTRATION

	% MgO	FILTRATE %P2O5	RETEN TIME	VARIANCE REPLICATES
t Acid P2O5/day/ft2	-0.13	-0.08	0.08	0.0001
Gyp t/day/ft2	-0.32	-0.34	0.35	0.0011
GYP t/t Acid P2O5	0.43	0.10	-0.06	0.0004

5.5 THEORETICAL SPECULATION

The data in Table V-l indicates that increasing feed MgO and filtrate concentration decreases recovery. Lets look at analytical data which might give us some insight on why the recovery is less when this happens.

Jacobs reported ‘Material Balances’(Sec 4.4) on the 4 five hour tests run. They analyzed the filtrates from Tests 3, 4, 5 and 11.

These analyses were used to calculate the ionic composition of the acids. That is the pluses and minuses of the cations and anions.

This data is listed in TABLE V-3. Included are the recoveries from the tests which were used for the statistical evaluation.

> The ionic equivalents of the constituents show little variation between the different tests except for the MgO content and the filtrate concentration. These were controlled variations.
>
> Comparing tests 5 & 4 and also 11 & 3 we show the drop in recovery due to the filtrate concentrate changing with no change in the MgO content. The principle and most probable cause for this decrease is the change in the SO3/P2O5 ratio in the acid because of the change in concentration. We attempted quite successfully to maintain a 2.50% soluble SO4 in the digestion tank (2.08 SO3) for all the tests and this level apparently drops while waiting to be analyzed to approximately the 1.8% level (0.05 equivalents) shown by Jacobs analyses. The ratios, %SO3 to %P2O5, in both 24.72% ac-acids tests are 0.073 while the ratios for the 27.77% acids are 0.066 and 0.069.

TABLE V-3

IONIC EQUIVALENTS OF TEST ACIDS

Test Run	5	4	11	3
Digestion time	5	5	5	5
Feed Rock MgO%	0.65	0.65	1.80	1.80
Filtrate Conc	24.72	27.77	24.71	27.77*
Anions				
P2O5	1.04	1.17	1.04	1.17
F	0.02	0.02	0.02	0.02
SO3	0.05	0.05	0.05	0.05
Total	1.11	1.24	1.11	1.24
Cations				
CaO	0.01	0.00	0.00	0.00
MgO	0.04	0.05	0.13	0.15
Fe2O3	0.04	0.05	0.04	0.05
Al2O3	0.04	0.05	0.04	0.04
Total	0.13	0.15	0.21	0.24
O'vr Recoveries	98.28	97.36	97.47	95.47

*-All values in this column adjusted to reflect change in concentration of the filtrate from 28.03 to 27.77

We know that increasing the sulfate level in the digestion slurry increases the rock reaction and solubility rate. Here we have increased the soluble sulfate level in the dilute acid tests by 6 to 10%. This type of increase could increase the reaction rate of the rock enough to account for the higher recoveries shown by both dilute acid tests when compared to the recoveries of the more concentrated acid digestions.

Now the answer to why the increasing MgO content of the rock decreases recovery lies in the equivalent calculations. The major soluble cations in these tests increased from 0.13-0.15 per 100 grams of acid for the Lo-Mag rock to 0.21-0.24 for the Hi-Mag rock. Since the acid equivalents (P2O5, F, and SO3) remain constant at 1.11 and 1.24 respectively the 0.08-0.09 more cationic equivalents present in the Hi-Mag tests reduce the Hydrogen ion content of the digestion slurry decreasing the reaction rate.

It is concluded that the above reasoning can explain the lower recoveries shown by the Hi-mag rock and the more concentrated filtrate tests.

SECTION 6.0

6.0 ECONOMIC EFFECTS OF USING HIGHER MgO ROCK

The comparative costs of producing phosphoric acid using the data developed by the test design was estimated using the following reasoning. Test 6 data from the 0.65% MgO rock was used in this comparison with the average test results obtained in the replicated tests using 1.23% MgO rock.

The plant designed for 1000 tons of acid P2O5 with 4 hours of retention (1.4 M3 digestion volume/t P2O5) time is producing 1284 tons and now has a the retention time of 3+ hours. The plant is limited by the filter capacity. It is operating with a rock similar to the 0.65 MgO rock and is producing a 27.5% filtrate.

Two estimates were made using the test data to develop operating costs. One used the four hour test retention time of the 1.23% MgO rock as the limiting factor while the second used the filter capacity as the limiting factor. In both the production rate is reduced. The second in addition to the small rate reduction the overall recovery will be less than that experienced in the test data.

6.1 RAW MATERIALS AND CONDITIONS

The feed rocks' analyses and the data developed by our testing used for the economic evaluations are listed below.

	%P2O5	%MgO	%CaO	%Fe2O3	%Al2O3	%SO3	%CO2
ROCK	**31.79**	**0.65**	**47.4**	**1.33**	**0.93**	**1.2**	**4.27**
	30.57	**1.23**	**46.49**	**1.33**	**0.89**	**1.19**	**5.42**

ROCK/HR	**Filt P2O5**	**Recoveries O'vall**	**Dig**	**tons H2SO4 /ton P2O5**	**t P2O5 /ft2/d**	**Gyp t /ft2/d**	**Gyp t/ t P2O5**
0.65/3	**27.38**	**96.87**	**97.05**	**2.63**	**.86**	**4.28**	**5.00**
1.23/4*	**26.01**	**97.27**	**97.43**	**2.69**	**.87**	**4.42**	**5.10**

***Average of three replicas**

PRODUCTION RATES

The production rate of 1284 ton P2O5 with the 0.65 MgO rock would produce 6420 tons of gypsum requiring a Prayon 30C filter operating on a 3 minute cycle. Filtering the same amount of gypsum produced using the 1.23% MgO rock would reduce the production rate to I258 tons of P2O5 due to the increased gypsum/ton of P2O5.

The analysis of variance of the test data indicates that a 0.91 increase in recovery can be expected if the retention time is increased from 3 to 5 hours. Using this data it can then be calculated that reducing the retention time from 4 to 3 hours the expected drop in recovery would be .46%. Recovery therefore one might expect using the 1.23% rock at the production rate of 1258 *tons/day is 96.8% while producing a 26% filtrate,

The test data as developed ie. 4 hours retention time, 26% filtrate and overall recovery of 97.27% was used to make the second estimate of operating cost using the 1.23% rock. Under these conditions the production rate would be limited to 1000 tons/day of acid P2O5.

FEED ROCK

Using the overall recoveries shown on the previous page we can see that the normal rock will require 3.25 tons of rock/ton of acid P2O5. The 1.23% rock with the filter limiting production would require 3.38 tons while using 4 hours of retention time as mandatory requires 3.36 tons of rock/ton of acid P2O5.

The transfer price of the 0.65% MgO rock to the phosphoric acid plant is assumed to be $21.50 per ton. The price of the 1.23% MgO rock would be at same P2O5 unit cost. The 30.57% P2O5 rock would be transferred at $20.68 per ton.

SULFURIC ACID FEED

A sulfur price of $73 per short ton delivered to the plant is assumed. The steam and electricity credits are all applied in the Sulfuric acid plant and allow the 100% acid to be transferred to the phosphoric acid plant at $28. per ton.

TABLE VI-1

ESTIMATED 1993 OPERATING COSTS

PRODUCTION RATE t/d P2O5	**1284**	**1258**	**1000**
RAW MATERIALS	**CONTROL**	**FILTER**	**TIME**
0.65%	**LIMITING**	**LIMITING**	
PHOSPHATE ROCK @$21.50/t	**$69.87**		
@ $20.68/t		**$69.90**	**$69.49**
Sulfuric Acid @$28.00/t	**$73.64**	**$75.32**	**$75.32**
Reagents(def. etc)	**1.00**	**1.22**	**1.22**
CONVERSIONS COSTS			
Labor, supplies & plant overheads	**28.96**	**29.56**	**37.19**
Maintenance	**11.23**	**11.46**	**14.42**
Ins., Taxes, & Depr.	**4.30**	**4.39**	**5.52**
Plant Costs/t P2O5	**$189.00**	**$191.85**	**$202.90**

These costs are for filter product acid. The 1.23% MgO acid produced at the 26% concentration would require about 10% more steam for evaporating acid that the plant uses or sells at the 5254% P205 concentration. These additional costs are site specific and therefore are not quantified here.

The 1.23% MgO rock was produced for our tests by blending a pebble reject rock containing 23.23% P205 and 4.7% MgO. We used 14% of this rock and 86% of the 0.65% MgO rock in order to produce the 1.23 % MgO feed. Since this 4.7% MgO rock is not now a saleable product and has no value it can be argued that the P205 derived in the blend from this rock does not cost anything and the 1.23% MgO rock is not worth the same unit price as the concentrate.

Unfortunately there are some costs that cannot be ignored even on this unsalable product. These costs are reasoned to be transportation, and depletion/royalties. These would total $2.50, assuming the acid plant is 10 miles from the mine and shipping costs are $0.10 per ton mile with the other costs totaling $0.70. The transfer price of the 1.23% MgO rock can then be calculated at $18.84 reflecting the $2.50 cost of the reject pebble used in the blend.

Cost at this transfer price of this rock to produce a ton of acid P2O5 would be reduced from $ 69.90 in Table VI-l to $ 63.49. This reduced raw material cost would make the cost of the filter limited plant product $185.44 per ton acid P2O5 instead of the $191.85 shown in the above table. Using this transfer price in the other alternate-the four hour digestion time- would reduce the cost from $202.90 to $196.72 per ton of Acid P2O5.

SECTION 5.0

5.0 STATISTICAL EVALUATION OF DATA

5.1 TEST DESIGN VARIABLES

The term variables here covers feed, digestions conditions and recoveries. The three types of variables and target levels for this design were:

- o FIXED VARIABLES
    - -80% Sulfuric acid to be used as feed.
    - -Tap water to be used as process water.
    - -35% solids in digestion slurry.
    - -2.5% soluble sulfate in digestion slurry.
    - -Agitation levels to be constant for tests.

- CONTROLLED VARIABLES

  -MgO content of feed rock was varied from 0.65, 1.23, and 1.8.

  -Concentration of P2O5 in filtrate ie. digestion liquor was varied in the tests from 24.5 to 26.0 and 27.5.

  -Digestion time was controlled at 3, 4, and 5 hours. The reaction volume per ton of P2O5 produced per day represented by these digestion times are approximately 1.1, 1.4 and 1.8 cubic Meters. Pierre Becker in his book “Phosphates and Phosphoric Acid” states that it is common for di-Hydrate plants to be designed with reaction volumes of 1.5-1.8 cubic Meters per ton of P2O5.

- INDEPENDENT VARIABLES

  -These are the chemical Recoveries, Sulfuric acid consumption, Filter capacity for tons of acid P2O5/day/ft2 and tons Gypsum/day/ft2 as well as tons of gyp produced per ton of acid P2O5.

5.2 CHOOSING THE DATA FOR EVALUATION

The total test data developed by Jacobs for each run had too much variation as is normally the case with plant data for direct use. This body of data however when separated in individual filtration periods had long periods of operation suitable for statistical evaluation. It was reasoned that the filtration characteristics displayed ie. recoveries and rate were the result of the digestion conditions prior to the filtration. Digestion data for a period four times the test retention time prior to the filtration was chosen. About 93-94% of the gypsum being filtered would have been formed in this period.

Stability of operation during this period as evidenced by the soluble sulfate levels standard of deviation and proximity to the target level of 2.5 (log sheet data) was used as the criteria for use as evaluation data. Contiguous filtration periods displaying close to the target levels of soluble sulfate and low deviations were then average to obtain the data used for the evaluations.

Figure V-1 shows Test 2 data summarized prior to choosing the 20 hour to 56 hour period data as representative for that set of controlled variables.

All the Tests were summarized like this. Copies of the summaries for all tests are contained in Appendix B of this report. The data selected for the evaluations of the Digestion variables and the calculated major effects are given in Table V-1.

The table is arranged with the controlled variables listed as in the test design. The Test-Period column shows the order in which they were run and the hours of operation chosen for the data evaluation.

TABLE V-1

DIGESTION DATA EVALUATED

VARIABLES

		Controlled			Independent				
Test		Filt		Reten	Ov'll	Gypsum			T H2SO4
#	Period	% MgO	P2O5	time	Recov	CI	CS	WS	/T P2O5
1	26/32 h	0.65	25.33	3	97.74	0.34	1.80	0.16	2.68
8	14/86 h	1.80	24.63	3	96.82	0.36	2.71	0.15	2.76
6	44/92 h	0.65	27.38	3	96.87	0.35	2.61	0.18	2.61
10	32/50 h	1.80	27.09	3	93.51	2.31	3.63	0.60	2.83
5	20/95 h	0.65	24.78	5	98.28	0.25	1.35	0.12	2.61
11	25/67 h	1.80	24.64	5	97.47	0.18	2.24	0.14	2.70
4	74/95 h	0.65	27.62	5	97.36	0.47	1.87	0.30	2.61
3	62/95 h	1.80	27.92	5	95.47	1.47	2.48	0.43	2.78
2	20/56 h	1.23	25.90	4	97.20	0.46	2.12	0.23	2.71
7	15/75 h	1.23	26.11	4	97.27	0.41	2.13	0.19	2.69
9	26/62 h	1.23	26.02	4	97.33	0.27	2.34	0.14	2.69

5.3 CALCULATED MAJOR EFFECTS-DIGESTION

	FILTRATE % MgO	%P2O5	RETEN TIME	VARIANCE REPLICATES
OVERALL RECOVERY	-1.75	-1.78	0.91	0.0028
LOSSES-GYPSUM CI	0.73	0.87	-0.25	0.0064
GYPSUM CS	0.86	0.62	-0.70	0.0101
GYPSUM WS	0.14	0.23	-0.02	0.0014
Tons H2SO4/Ton Acid P2O5	0.14	0.03	-0.05	0.0001

This data applies to the variations of the controlled variables within the range tested and of course to the single reactor configuration. The effects will probable be present in any multi-compartment reactors but the magnitude of the effects will differ. The influence of fluorine containing process water normally used by commercial operations on filtration recoveries was not investigated in this series of tests. Test filtration recoveries therefore are significantly better than one would expect in normal plant operation. Calculation of the above major effects by assuming normal filtration recoveries would reduce overall recoveries more, increase water soluble P2O5 losses and sulfuric acid consumption.

The variances obtained in the replicated tests are small when compared with the major effects and indicate that the effects shown are significant and real.

OVERALL RECOVERY - The data shows that increasing MgO content and the filtrate P2O5 concentration will result in lower recoveries. Increasing retention time ie. reducing the production rate in a continuous system will increase recovery.

The recovery data from Table V-1 is plotted graphically in the Figure V-2. The scatter of points is caused by the attempt to plot the actual Filtrate concentrations not the targets. The increase in recovery by increasing digestion time from three to five hours is evident.

A recovery contour was calculated for the seven tests that had 4 and 5 hours digestion times. The result is presented in Figure V-3. This figure shows the curvature of the data caused by the effect of the Filtrate concentration. The filtrate scale is not accurate but the actual spacing of the data is therefore the surface calculated is believed accurate.

GYPSUM CARE LOSSES - The data shows increasing MgO content and the filtrate P2O5 concentration increases CI, CS and WS P2O5 losses. As one might expect increasing the filtrate P2O5 content results in higher WS losses.

The increased recovery shown by the increased retention time is primarily because of the decrease in the gyp cake's CS P2O5 content.

SULFURIC ACID CONSUMPTION-Could be increased by as much as 8.4% from 2.61 t/t of acid P205 for the .065% MgO rock to 2.83 t/t for the 1.8% MgO rock. This would occur with a 3 hour digestion time producing a 27.5% Filtrate,

Data selected for the evaluation of filtration and filter capacity are presented in the following Table IV-2. The data is arranged as in the previous Table V-1.

FILTER CAPACITY - The increased MgO content (0.65-1.8) results in a 14.5% drop in acid filtration rate. The filter rate per foot square would drop from 0.90 to 0.77 tons of acid P2O5. The Gyp cake capacity would also be reduced from 4.41 to 4.09 tons of gyp per square foot per day a drop of 7.3%. Increasing filtrate strength causes a similar drop in filter capacity. Increasing retention time would negate some of the lost capacity but would cause a greater economic effect because of the decrease in the production rate.

GYPSUM STACK REQUIREMENTS-The higher MgO containing rock on average would increase the production of gypsum by 8.7%. An additional 1-2% increase in gypsum production is indicated if the filtrate in maintained at the normal industry target of 27.5% P2O5.

TABLE V-2

FILTRATION DATA EVALUATED

VARIABLES

		Controlled			Independent		
Test #	Time Period	%MgO	P2O5	Retent Time	t Acid P2O5 /day/ft2	t Gyp/ day/ft2	t Gyp/ t P2O5
1	26/32 h	0.65	25.33	3	0.89	4.35	4.89
8	14/86 h	1.80	24.63	3	0.79	4.16	5.26
6	44/92 h	0.65	27.38	3	0.86	4.28	5.00
10	32/50 h	1.80	27.09	3	0.63	3.49	5.58
5	20/95 h	0.65	24.78	5	0.95	4.64	4.91
11	25/67 h	1.80	24.64	5	0.86	4.51	5.36
4	74/95 h	0.65	27.62	5	0.88	4.35	4.95
3	62/95 h	1.80	27.92	5	0.79	4.19	5.28
2	20/56 h	1.23	25.90	4	0.86	4.40	5.11
7	15/75 h	1.23	26.11	4	0.88	4.47	5.07
9	26/62 h	1.23	26.02	4	0.86	4.40	5.11

5.4 CALCULATED MAJOR EFFECTS-FILTRATION

	FILTRATE % MgO	%P2O5	RETEN TIME	VARIANCE REPLICATES
t Acid P2O5/day/ft2	-0.13	-0.08	0.08	0.0001
Gyp t/day/ft2	-0.32	-0.34	0.35	0.0011
GYP t/t Acid P2O5	0.43	0.10	-0.06	0.0004

5.5 THEORETICAL SPECULATION

The data in Table V-1 indicates that increasing feed MgO and filtrate concentration decreases recovery. Lets look at analytical data which might give us some insight on why the recovery is less when this happens.

Jacobs reported 'Material Balances'(Sec 4.4) on the 4 five hour tests run. They analyzed the filtrates from Tests 3, 4, 5 and 11. These analyses were used to calculate the ionic composition of the acids. That is the pluses and minuses of the cations and anions.

This data is listed in TABLE V-3. Included are the recoveries from the tests which were used for the statistical evaluation.

The ionic equivalents of the constituents show little variation between the different tests except for the MgO content and the filtrate concentration. These were controlled variations.

Comparing tests 5 & 4 and also 11 & 3 we show the drop in recovery due to the filtrate concentrate changing with no change in the MgO content. The principle and most probable cause for this decrease is the change in the SO3/P2O5 ratio in the acid because of the change in concentration. We attempted quite successfully to maintain a 2.50% soluble SO4 in the digestion tank (2.08 SO3) for all the tests and this level apparently drops while waiting to be analyzed to approximately the 1.8% level (0.05 equivalents) shown by Jacobs analyses. The ratios, %SO3 to %P2O5, in both 24.72% acids tests are 0.073 while the ratios for the 27.77% acids are 0.066 and 0.069.

TABLE V-3

IONIC EQUIVALENTS OF TEST ACIDS

Test Run	5	4	11	3
Digestion time	5	5	5	5
Feed Rock MgO%	0.65	0.65	1.80	1.80
Filtrate Conc	24.72	27.77	24.71	27.77*
Anions				
P2O5	1.04	1.17	1.04	1.17
F	0.02	0.02	0.02	0.02
SO3	0.05	0.05	0.05	0.05
Total	1.11	1.24	1.11	1.24
Cations				
CaO	0.01	0.00	0.00	0.00
MgO	0.04	0.05	0.13	0.15
Fe2O3	0.04	0.05	0.04	0.05
Al2O3	0.04	0.05	0.04	0.04
Total	0.13	0.15	0.21	0.24
O'vr Recoveries	98.28	97.36	97.47	95.47

*-All values in this column adjusted to reflect change in concentration of the filtrate from 28.03 to 27.77

We know that increasing the sulfate level in the digestion slurry increases the rock reaction and solubility rate. Here we have increased the soluble sulfate level in the dilute acid tests by 6 to 10%. This type of increase could increase the reaction rate of the rock enough to account for the higher recoveries shown by both dilute acid tests when compared to the recoveries of the more concentrated acid digestions.

Now the answer to why the increasing MgO content of the rock decreases recovery lies in the equivalent calculations. The major soluble cations in these tests increased from 0.13-0.15 per 100 grams of acid for the Lo-Mag rock to 0.21-0.24 for the Hi-Mag rock. Since the acid equivalents (P2O5, F, and SO3) remain constant at 1.11 and 1.24 respectively the 0.08-0.09 more cationic equivalents present in the Hi-Mag tests reduce the Hydrogen ion content of the digestion slurry decreasing the reaction rate.

It is concluded that the above reasoning can explain the lower recoveries shown by the Hi-mag rock and the more concentrated filtrate tests.

SECTION 6.0

6.0 ECONOMIC EFFECTS OF USING HIGHER MgO ROCK

The comparative costs of producing phosphoric acid using the data developed by the test design was estimated using the following reasoning. Test 6 data from the 0.65% MgO rock was used in this comparison with the average test results obtained in the replicated tests using 1.23% MgO rock.

The plant designed for 1000 tons of acid P2O5 with 4 hours of retention (1.4 M3 digestion volume/t P2O5) time is producing 1284 tons and now has a the retention time of 3+ hours.

The plant is limited by the filter capacity. It is operating with a rock similar to the 0.65 MgO rock and is producing a 27.5% filtrate.

Two estimates were made using the test data to develop operating costs. One used the four hour test retention time of the 1.23% MgO rock as the limiting factor while the second used the filter capacity as the limiting factor. In both the production rate is reduced. The second in addition to the small rate reduction the overall recovery will be less than that experienced in the test data.

6.1 RAW MATERIALS AND CONDITIONS

The feed rocks' analyses and the data developed by our testing used for the economic evaluations are listed below.

	%P2O5	%MgO	%CaO	%Fe2O3	%Al2O3	%SO3	%CO2
ROCK	31.79	0.65	47.4	1.33	0.93	1.2	4.27
	30.57	1.23	46.49	1.33	0.89	1.19	5.42

ROCK/HR	Filt P2O5	Recoveries O'vall	Dig	tons H2SO4 /ton P2O5	t P2O5 /ft2/d	Gyp t /ft2/d	Gyp t/ t P2O5
0.65/3	27.38	96.87	97.05	2.63	.86	4.28	5.00
1.23/4*	26.01	97.27	97.43	2.69	.87	4.42	5.10

***Average of three replicas**

PRODUCTION RATES

The production rate of 1284 ton P2O5 with the 0.65 MgO rock would produce 6420 tons of gypsum requiring a Prayon 30C filter operating on a 3 minute cycle. Filtering the same amount of gypsum produced using the 1.23% MgO rock would reduce the production rate to. 1258 tons of P2O5 due to the increased gypsum/ton of P2O5.

The analysis of variance of the test data indicates that a 0.91 increase in recovery can be expected if the retention time is increased from 3 to 5 hours. Using this data it can then be calculated that reducing the retention time from 4 to 3 hours the expected drop in recovery would be .46%. Recovery therefore one might expect using the 1.23% rock at the production rate of 1258 *tons/day is 96.8% while producing a 26% filtrate.

The test data as developed ie. 4 hours retention time, 26% filtrate and overall recovery of 97.27% was used to make the second estimate of operating cost using the 1.23% rock. Under these conditions the production rate would be limited to

1000 tons/day of acid P2O5.

FEED ROCK

Using the overall recoveries shown on the previous page we can see that the normal rock will require 3.25 tons of rock/ton of acid P2O5. The 1.23% rock with the filter limiting production would require 3.38 tons while using 4 hours of retention time as mandatory requires 3.36 tons of rock/ton of acid P2O5.

The transfer price of the 0.65% MgO rock to the phosphoric acid plant is assumed to be $21.50 per ton. The price of the 1.23% MgO rock would be at same P2O5 unit cost. The 30.57% P2O5 rock would be transferred at $20.68 per ton,

SULFURIC ACID FEED

A sulfur price of $73 per short ton delivered to the plant is assumed. The steam and electricity credits are all applied in the Sulfuric acid plant and allow the 100% acid to be transferred to the phosphoric acid plant at $28. per ton.

TABLE VI-1

ESTIMATED 1993 OPERATING COSTS

PRODUCTION RATE t/d P2O5		**1284**	**1258**	**1000**
RAW MATERIALS		**CONTROL**	**FILTER**	**TIME**
		0.65%	**LIMITING**	**LIMITING**
PHOSPHATE ROCK	**@$21.50/t**	**$69.87**		
	@ $20.68/t		**$69.90**	**$69.49**
Sulfuric Acid	**@$28.00/t**	**$73.64**	**$75.32**	**$75.32**
Reagents(def. etc)		**1.00**	**1.22**	**1.22**

CONVERSIONS COSTS			
Labor, supplies & plant overheads	28.96	29.56	37.19
Maintenance	11.23	11.46	14.42
Ins., Taxes, & Depr.	4.30	4.39	5.52
Plant Costs/t P2O5	$189.00	$191.85	$202.90

These costs are for filter product acid. The 1.23% MgO acid produced at the 26% concentration would require about 10% more steam for evaporating acid that the plant uses or sells at the 52-54% P2O5 concentration. These additional costs are site specific and therefore are not quantified here.

The 1.23% MgO rock was produced for our tests by blending a pebble reject rock containing 23.23% P2O5 and 4.7% MgO. We used 14% of this rock and 86% of the 0.65% MgO rock in order to produce the 1.23 % MgO feed. Since this 4.7% MgO rock is not now a saleable product and has no value it can be argued that the P2O5 derived in the blend from this rock does not cost anything and the 1.23% MgO rock is not worth the same unit price as the concentrate.

Unfortunately there are some costs that cannot be ignored even on this unsalable product. These costs are reasoned to be transportation, and depletion/royalties. These would total $2.50, assuming the acid plant is 10 miles from the mine and shipping costs are $0.10 per ton mile with the other costs totaling $0.70. The transfer price of the 1.23% MgO rock can then be calculated at $18.84 reflecting the $2.50 cost of the reject pebble used in the blend.

Cost at this transfer price of this rock to produce a ton of acid P205 would be reduced from $ 69.90 in Table VI-1 to $63.49.

This reduced raw material cost would make the cost of the filter limited plant product $185.44 per ton acid P205 instead of the $191.85 shown in the above table. Using this transfer price in the other alternate-the four hour digestion time- would reduce the cost from $202.90 to $196.72 per ton of Acid P2O5.

Paragraph for addition to the summary section 2

Selected Data was subject to statistical analysis. Based on this analysis the controlled variables caused significant differences in digestion recoveries, sulfuric acid requirements, filtration rate and filter capacities.

These differences were used to estimate the production costs of using higher MgO rock for wet process acid manufacture.