

THE PRODUCTION OF PHOSPHORIC ACID FROM PHOSPHATE SLIMES
WITH HIGH CHLORINE CONTENT

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ABSTRACT

In order to produce 2×10^6 tons/year of Zin washed phosphate containing 32.4% P_2O_5 and 0.04% Cl, approximately 1.5×10^6 tons of fine phosphate slime tailings are discarded. These phosphate slimes have a P_2O_5 content of about 24% and are rejected in a recycle wash water stream containing 10,000 ppm. Cl.

A research and development programme has been carried out to provide design data for a 250,000 tpy P_2O_5 wet process phosphoric acid plant, using treated fine reject as a raw feed material.

The emphasis of the programme was directed towards the problem of dewatering the phosphate slimes, in order to reduce the chlorine content of the feed as far as possible and improve the water balance of the phosphoric acid plant. Design data has also been obtained on the construction of a plant capable of operating with a 30% weak acid containing up to 5,000 ppm. Cl.

A block diagram is provided, together with an estimate of the investment required.

INTRODUCTION

Negev Phosphates produce 2×10^6 tons/year washed phosphate containing 32.4% P_2O_5 and 0.04% Cl at their Zin mine. Due to the characteristics of the soft sedimentary-type ore, approximately 30% of the run of mine material is smaller than 74 microns and is discarded during the washing process which is used to leach out the chlorine from the ore.

After thickening, the fine material is pumped to large tailing dams as a 35% slurry using a recycle wash water stream which contains approximately 10g/l Cl as NaCl. These tailings, which contain between 22-26% P_2O_5 , represent a P_2O_5 potential of more than 500,000 tons/year.

Direct use of this material as a saleable product is limited due to its low quality and fine dusty nature. Because of the increasing cost of phosphate production, especially the mining costs, and the need to increase overall P_2O_5 recovery, Negev Phosphates decided to initiate a full-scale research and development programme whose main aim was the production of merchant grade 54% phosphoric acid from slime rejects.

Preliminary mass balance figures indicated that a phosphoric acid plant producing 250,000 t P_2O_5 could be considered, i.e. recovery of approximately 50% of the rejected P_2O_5 . All mass balance and subsequent decisions on size of equipment have been based on this target.

Before any start could be made on the detailed design of this project, it was necessary to undertake a large-scale research and development programme whose main aim was the investigation of the following:

- a. Definition of the fine rejects and, especially, any relationship between the main ore body, product, and the fine rejects.
- b. Preparation of material as a feed for phosphoric acid manufacture with emphasis on:
 - P_2O_5 level
 - Moisture content
 - Cl content
- c. Basic design of a phosphoric acid plant with respect to:
 - wet feed
 - high Cl content of 30% weak acid solution due to utilization of saline water in acid plant
- d. Economic feasibility study to determine the main areas where improvements could be made to the proposed process and investment programme.

WATER SUPPLY

Any plant design for operation in the Negev region of Israel must take into consideration that only brackish water will be available as the process water. This water, containing approximately 700 ppm. Cl, will affect the Cl balance in any phosphoric acid plant.

Table 1: Analysis of Negev Saline Water

	ppm
Cl-	671
HCO_3^-	314
$SO_4^{=}$	378
Na+	435
K+	81
Ca++	150
Mg++	66
pH	66
Total hardness	642.5 as $CaCO_3$

DEFINITION OF ZIN PHOSPHATE TAILINGS

A systematic analysis was carried out of the slimes obtained during operation of the plant. The material was sampled before the scavenger system. A block diagram of the main areas of the Zin plant are given in figure 1.

Table 2: Fine Rejects, Zin Plant

Mesh Size	Microns	Wt. %	Cumulative wt. %	% P ₂ O ₅	Ave. P ₂ O ₅
+200	74	9.4	9.4	29.4	27.2
-200+270	74/54	6.8	16.2	28.0	
-270+325	54/44	8.4	24.6	27.1	
-325+400	44/37	0.7	25.3	26.5	
-400+500	37/25	11.9	37.2	26.4	
-500+600	25/20	7.0	44.2	25.4	24.2
-600	-20	56.8	100	21.4	

Analysis of many plant and geological samples has shown that a relationship exists between the P₂O₅ quality in the 200/600 fraction and the run of mine ore. In general, this layer is 1.4% higher than the P₂O₅ layer of the run of mine material, and 5.6% higher than the -600 mesh fraction.

The chlorine level of the recycle wash water system ranges from 5-8g/l and has a maximum level of 10g/l (10,000 ppm).

PHOSPHATE FEED

Beneficiation of the fine reject stream

The overflow stream from the hydrocyclones at the Zin plant contains approximately 4.5% solids. The obvious method of upgrading this stream, containing solids with a size distribution as given in Table 2, is to use hydrocyclones. Thus, an investigation was carried out to determine the configuration of hydrocyclones which would give an optimum of:

- sharpness of cut at 25 microns diameter;
- high per cent solids in underflow;
- efficiency and recoveries at various operating conditions with respect to abrasion;
- investment/operating costs;
- single stage vs. double stage operation.

Results have shown that a single stage 6" diameter hydrocyclone operating at an inlet pressure of 20 psi using a ½" apex is the optimum. A typical set of results is given in figure 2. Approximately 170 cyclones will be required to produce 134 tons of deslimed material on a dry basis. A two-stage system would contain 170 cyclones in the first stage and 70 in the second.

Dewatering

The dewatering of the 55% hydrocyclone underflow obtained from the previous beneficiation stage is of utmost importance. The results of this stage will determine the water balance of the phosphoric acid plant, affecting possible P_2O_5 recoveries in the gypsum filtration and the Cl level in the phosphoric acid reaction stage. As all the chlorine is present as dissolved NaCl, reduction of the moisture content will lower the chlorine content of the phosphate feed.

Tests were carried out on various commercial equipment at both a laboratory and a pilot stage. Typical results obtained in these experiments are given in Tables 8 and 9 and figure 3. A summary of the results is given below.

Table 3: Filtration tests on 200/600 mesh fraction

Apparatus	Final Moist. % H ₂ O	Test Method	Cl content of cake %	Cl content of washed cake	No. of units requ'd for 100 tph dry solids
<u>Vacuum filter</u>					
Top feed	32	Lab.	0.41	0.1	150m ² (3 units)
Bottom feed	28	Pilot	0.39	0.1	150m ² (3 units)
<u>Pressure filter</u>	22	Pilot	0.28	0.1	2 x 2m x 150 plates (3 units)
<u>Centrifuge</u>	18	Lab.	0.23	N.T.	(2-5 units)

(N.T. = not tested)

Results

The high moisture contents obtained with the vacuum filter tests indicate that further experimental work should be carried out before this equipment can be considered suitable for our purpose. The results have been confirmed by tests carried out on each fraction which show that the residual water in the cake is a function of the large surface area of the material associated with the porous nature of the phosphate. Additional experimentation with the use of heated wash water and/or surface-active agents has been initiated. The preliminary results:

have been encouraging, reducing the moisture content to 22%. A study is now being made on the investment/operating costs of the various types of equipment.

Settling test

The Zin plant has two 104 m diameter thickeners to increase the percent solids of tailings from 4.5 to 35% solids. Sedimentation tests were carried out to determine the area required to thicken the -600 mesh material. The results show that the same area is required, as the increase in thickening area required is offset by the reduction in the quantity of material which will be pumped to the thickeners.

Table 4: Settling test for -600 mesh material

% solids feed	% solids +600 mesh	Conc. of settled solids after 48 hours	Height of column (cm.)	<u>Req'd area settler</u>	
				<u>m²/ton, day</u> for final conc. of 27% solids	for conc. after 48 hours
1.6	3.2	44.6	34	5.2	13.0
1.6	3.2	44.6	150	4.1	13.0
1.6	3.2	44.6	250	4.1	13.0
2.4	6.8	39.3	34	8.1	8.2
3.6	6.8	39.3	34	6.7	7.4
3.5	4.0	34.8	34	6.8	7.1

PHOSPHORIC ACID PRODUCTION

Since 1979 Negev Phosphates has been operating a phosphoric acid pilot plant (5 Kg/hr) which has been utilized to provide technical assistance and knowledge for the production of phosphoric acid from Zin phosphate. The pilot has been calibrated on the basis of feed-back from our clients' operation and we consider it a reliable instrument for determining the necessary operating parameters and design data for full-scale manufacture. A comprehensive corrosion testing system, incorporating weighed impeller coupons, interchangeable impellers and electro-chemical corrosion-erosion measurements, has been installed (see figures 5 and 6).

Two long-term tests were carried out over a period of three months to determine the effects of the fine material on the normal operating parameters and, in particular, phosphoric acid slurry characteristics, gypsum formation and crystal habit, and corrosion characteristics of various CrNi alloys.

A large amount of data was collected on corrosion at levels of Cl up to 7500 ppm, a range about which there exists little experimental data and no full-scale operating experience.

<u>Table 5A: Feed to Phosphoric Acid Plant</u>		
<u>Size Analysis</u>	<u>Mesh</u>	<u>%</u>
	+200	22
	-200+270	21
	-270+325	15
	-325+500	24
	-500+600	9
	-600	9
<u>Chemical Analysis</u>	P_2O_5	28.8
	SiO_2 (+ clay addition)	1.8
	Al_2O_3 (+ clay addition)	0.45
	CaO	51.9
	Cl	0.5
	Fe_2O_3	0.07
	F	3.6
	SO_3	3.7
	CO_2	6.1
	Na_2O	1.0
	K_2O	0.02
	O.M	0.38
	Acid consumption (lab.)	3.05

Results of WPA Production on Pilot PlantTable 5B: Comparison of Chemical Analysis of Product Acids

	<u>Zin Fine</u>		<u>Zin Regular</u>
	Expt. 37	Expt. 40	
P ₂ O ₅	24.9	27.5	27.8
SO ₄	3.9	2.1	2.8
F	1.2	1.7	1.7
SiO ₂	0.49	0.68	0.61
Al ₂ O ₃	0.56	0.50	0.37
Fe ₂ O ₃	0.116	0.13	0.135
Cl	0.41	0.30	0.085
6F/SiO ₂	1.34	1.13	1.17
Molar Ratio			

Table 5C: Comparison of Chemical Analysis of Gypsum

	Zin Fine		<u>Zin Regular</u>
	Expt. 37	Expt. 40	
Moisture	21.6		23.3
Total P ₂ O ₅	0.32	0.42	0.48
Water-soluble P ₂ O ₅	0.07	0.07	0.20
Insoluble P ₂ O ₅	0.25	0.35	0.28
F	0.16	0.22	0.45
SiO ₂	0.92	0.85	0.70
Al ₂ O ₃	0.12	0.10	0.22
Fe ₂ O ₃			0.026
Crystal size (u)	75/44	92/48	48/25
Length:width ratio	1.7:1	1.9:1	1.8:1
Shape of crystal	Rhombic	Twins/ Rhombic	Rhombic
Total efficiency	98.1		97.5
Reaction efficiency	98.6		98.5
Wash losses	0.5		1.0

Phosphate Feed

Phosphate feed is similar to ordinary Zin rock except for the higher Cl content (0.5) in the fine reject. The level of SiO_2 and Al_2O_3 is similar to our normal product and, therefore, 2.5% of active clay was added to convert any free HF present to H_2SiF_6 . The levels of Fe_2O_3 and MgO are at the same characteristically low levels (Table 5A). Theoretical acid consumption was 3.10 vs 2.8 ton H_2SO_4 /ton P_2O_5 for normal Zin phosphate, an increase of 10%.

Slurry characteristics

No difference in slurry characteristics was noticed in mixing agitation or pumping. The pilot operated at its normal 32-36% solids, 25-30% P_2O_5 and 2-3% SO_4 (Table 5B).

Gypsum formation

Due to the reactive nature of the phosphate, we considered that the main problem would be the formation of small gypsum crystals. However, the opposite was true. Gypsum crystals are approximately twice the size of those usually obtained. The L/B ratio of the gypsum set by the $\text{Al}_2\text{O}_3/\text{SiO}_2$ ratio and SO_3 for Zin rock remained the same. The efficiency was increased (Table 5C).

Corrosion test

Tables 6A and 6B give a summary of the corrosion tests carried out during the experimental period. The addition of 2.5% clay containing 55% SiO_2 and 25% Al_2O_3 was used to remove the free HF present in the absence of SiO_2 . The effect of the clay addition can be seen in the enhanced corrosion figures obtained when testing the phosphate rock with 0.7% Cl without clay addition. Of the many metals tested only the HASTELLOY C4 impeller and the AVESTA SMO, SANICRO 28 and HASTELLOY G and 285 agitator blades remain relatively uncorroded. The impeller tests being more vigorous provide a better testing method, as can be seen when comparing the relative results of HASTELLOY 285 and HASTELLOY G. Long term testing with pumps fabricated from suitable materials is now being carried out.

Concentration Section

The acid has been concentrated in a pilot unit to obtain specifications, Cl balance and corrosion results. (Tables 7 and 8).

CONCLUSION

As a result of the programme, we have drawn up a basic flow sheet to produce 250,000 tons P_2O_5 /year of 54% P_2O_5 merchant acid from the fine rejects of the Zin beneficiated process (figure 4).

In Table 9 an estimate has been made of the total investment required, as well as the extra investment which must be made in order to adapt the phosphoric acid technology for high Cl (up to 5000 ppm) weak acid solutions.

Because of the low cost of the phosphate raw material, the profitability of the project is dependent on the price of sulphur, the consumption of sulphuric acid per unit P_2O_5 produced, and the optimum utilization of the energy produced as a by-product of the sulphuric acid plant. In general, about 1.2 tons of by-product steam is produced with each ton of H_2SO_4 while steam consumption in a normal dihydrate plant is about 2 tons steam/ton P_2O_5 or 500,000 tons/year steam.

The placing of the phosphoric acid plant at the mine site, situated 40 km. from the nearest industrial site, creates a problem of steam utilization. The production of electricity is a partial solution to this problem, but use of a back pressure turbine provides the pressure needed to utilize the steam still further. Due to the economic importance of full utilization of the energy source, the scope of this project has been enlarged to include possible solutions to this problem:

- Upgrading of phosphate fines using a novel flotation technique, thus lowering the required sulphuric acid consumption/ton P_2O_5 produced.
- Utilization of steam instead of liquid fuels to dry phosphate in suitable equipment.

These two programmes should be completed by the end of 1982.

CORROSION TESTS ON PILOT PLANT

Table 6A: Tests carried out on agitator blades

Feed Phosphates	Corrosion rates (mm/year)									
	AVESTA			URANUS		HASTELLOY		SAS	SS	SANICRO
	SLX	SMO	SKR	UB6	UB6P	285	G	20	316	28
Zin + 2.5% clay	0.22	0.25	0.57	0.17	0.25	0.37	0.14	0.21	68	0.10
Zin + 2.5% Clay + 0.2% Cl	0.22	0.31	119	0.17	0.12	0.34	0.08	0.19	165	0.05
Zin + 2.5% clay + 0.5% Cl	0.18	0.18	91	25		0.21	0.10	0.19	194	0.06
Zin + 2.5% clay + 0.7% Cl	0.18	0.15	137	20	0.2	0.17	0.12	0.16	256	0.10
Zin + 0.7% Cl	145	0.40	163	108	6	0.38	0.19	21	130	0.14
Zin + 2.5% clay	0.32		0.5	1.4				0.27		

Table 6B: Tests carried out on interchangeable pump impeller

Feed Phosphate	Corrosion rates (mm/year)					
	HASTELLOY			DURION CD4	URANUS UB6	SS 316
	G	C4	285			
Zin + 2.5% clay	0.9	0.57		0.8	0.4	
Zin + 2.5% clay + 0.2% Cl		1.35		0.6	1.25	117
Zin + 2.5% clay + 0.5% Cl	1.01	2.60	0.81	0.64	20.6	
Zin + 2.5% clay + 0.7% Cl	0.9	1.10	2.47	1.25	13.5	
Zin + 0.7% Cl	20.3	1.30	2.48	8.5	140	
Zin + 2.5% clay	1.1		1.1	1.2	70	153

Table 7: Analysis of Feed and Concentrated Acid

	Dilute feed acid %	Concentrate product acid %
P_2O_5	28.1	49.4
Al_2O_3	0.47	1.0
Fe_2O_3	0.11	0.34
F	1.0	0.30
Cl	0.36	0.03
SO_4	4.1	8.9
Total C	0.1	0.19
SiO_2	0.33	0.006
MgO	0.28	0.6

Table 8: Centrifuge Test

Feed

52.5% solids

23.93% P₂O₅

		I	II	III
Speed - Cake build-up	RPM	2000	2000 1000	1000
Dewatering	RPM	3000	3000	4000
Time of dewatering	Sec.	30	30	30
% Moisture cake		17.6	18	14.7
P ₂ O ₅ (dry basis)		23.4		
Cl (in cake or dry basis)		0.23	0.25	0.21
Cl (in mother liquor)		11.5g/l	11.5g/l	11.5

Test carried out - bowl type laboratory centrifuge

Carl Padberg Type LS

Diameter 200 mm.

Max. RPM 4,500

Volume 2 litres

Granulometric Analysis:

+200	13%
-200+325	38%
-325+600	36%
-600	13%

Table 9: Summary of Pressure Filter Tests (Pilot Tests)

Test No.	Dry Solids	Cake Moisture	Cake Density	Size & No. of Cakes	Cloths	Pressure	Filtration Parameters			Chloride in Cake	Cake Condition	Remarks
1	-	34.73	-	2 x 25mm	Rilsan	15 bar	15 min. pressing			0.1988%	Poor	No air blow
2	-	8.83	-	2 x 25mm	WWPP810	7 bar	4½	6	-	0.1778	Good	Cake moisture content susp. Good cake release
3	-	27.79	1359kg/m ³	2 x 25mm	Rilsan	7 bar	4	6	-	0.2130	Good	Good cake release
4	-	24.6	1406 "	3 x 38mm	Courlene	15 bar	8	10	-	0.144	Good	" " "
5	-	25.54	1542 "	3 x 38mm	Rilsan	10 bar	5	15	-	0.2234	Good	" " "
6	-	25.4	1728 "	3 x 38mm	Rilsan	10 bar	5	10	-	0.1753	Good	" " "
7	57.6	25.7	1814 "	3 x 45mm	Rilsan	10 bar	5	12	-	0.2099	Good	" " "
8	56.54	23.41	1473 "	3 x 32mm	Rilsan	10 bar	5	10	-	0.2243	Good	" " "
9	58.47	22.99	1525 "	3 x 38mm	Rilsan	10 bar	5	10	-	0.0603	Good	" " "
10	-	25.24	1474 "	3 x 38mm	Rilsan	10 bar	5	10	-	0.1373	Good	" " "
11	-	25.57	1520 "	3 x 38mm	Rilsan	10 bar	5	10	-	0.2600	Good	" " "
12	-	22.9	1355 "	2 x 38mm	Rilsan	7 bar	7	10	3	0.221	Good	Pumping problems Good release
							P	AB	M			

P = Pressing

AB = Air Blow

M = Membrane

Table 10: Cost Estimate

Estimate of additional investment required in attack and filtration section of H_5PO_4 plant for high chlorine operation (basis 120,000 tpy plant, Prayon type dehydrate plant).

<u>Equipment</u>	<u>Cost (\$)</u>	<u>Additional cost (\$)</u> for high chlorine <u>(0.5% Cl in feed)</u>
Attack tank	650,000	
Pumps Flash Cooler x 2	42,000	15,300 Hast C Rubber lined impellers
- Filter feed	87,000	40,000 " " " "
- Slurry recirculation x 2	73,000	25,500 " " " "
- Filter cake & cloth	7,000	
- Water return	175,000	50,000
- Filtrate x 4	40,000	10,000 Rubber lined
- Acid drain	6,500	3,000 Hast C + Rubber lined
- Scrubber recycle	3,000	
- Gypsum slurry	42,000	20,000 UB6P impeller
Defoaming	10,000	
Agitators attack 1 + 2	100,000	44,000
3 + 4	50,000	22,000
5	48,000	20,000
6	30,000	15,000
9	1,500	1,000
- Filtrate seal	20,000	10,000
Mixing tanks	30,000	-
Gypsum slurry tank	5,000	-
Fume scrubber foam	20,000	
Compressor instrument + plant	50,000	-
Filter	1,400,000	650,000 Uranus 6P or rubber lined Prayon unit
Condensor Flash Cooler	30,000	-
Scrubber	111,300	-

Table 10 (cont.)

<u>Equipment</u>	<u>Cost (\$)</u>	<u>Additional cost (\$)</u> for high chlorine <u>(0.5% Cl in feed)</u>
Monorail + Hoist	20,000	-
Flash Cooler	110,000	-
Tanks - Barom. seal	20,000	-
- Filtrate seal	45,000	-
- Scrubber seal	1,500	-
- Defoamer	25,000	-
- Gypsum slurry	15,000	-
Duct works and chutes	60,000	-
	<u>3,228,900</u>	<u>925,800</u>

Evaporation Section (Assume Karbate Heat Exchangers)

Total Equipment Cost - 1,500,000

Additional Costs for high Cl

- Pumps & Transfers	10,000
- Circulation pumps	100,000
- Condensate pumps	10,000
	<u>\$120,000</u>
Total extra additional cost	\$1,045,800

Cost of supply and installation
of phosphoric acid plant

(Israeli conditions) \$29,500,000

Table 11: Total cost for phosphate slime acid complex (Israel 1980)120,000 tpy

(\$1,000)

Sulphuric acid	20,500
Phosphate preparation	7,000
Phosphoric acid	29,500
Service and utilities	14,000
	<u>71,000</u>

Additional cost for high chlorine utilization 1,045

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Figure 1: Zin Plant flow sheet

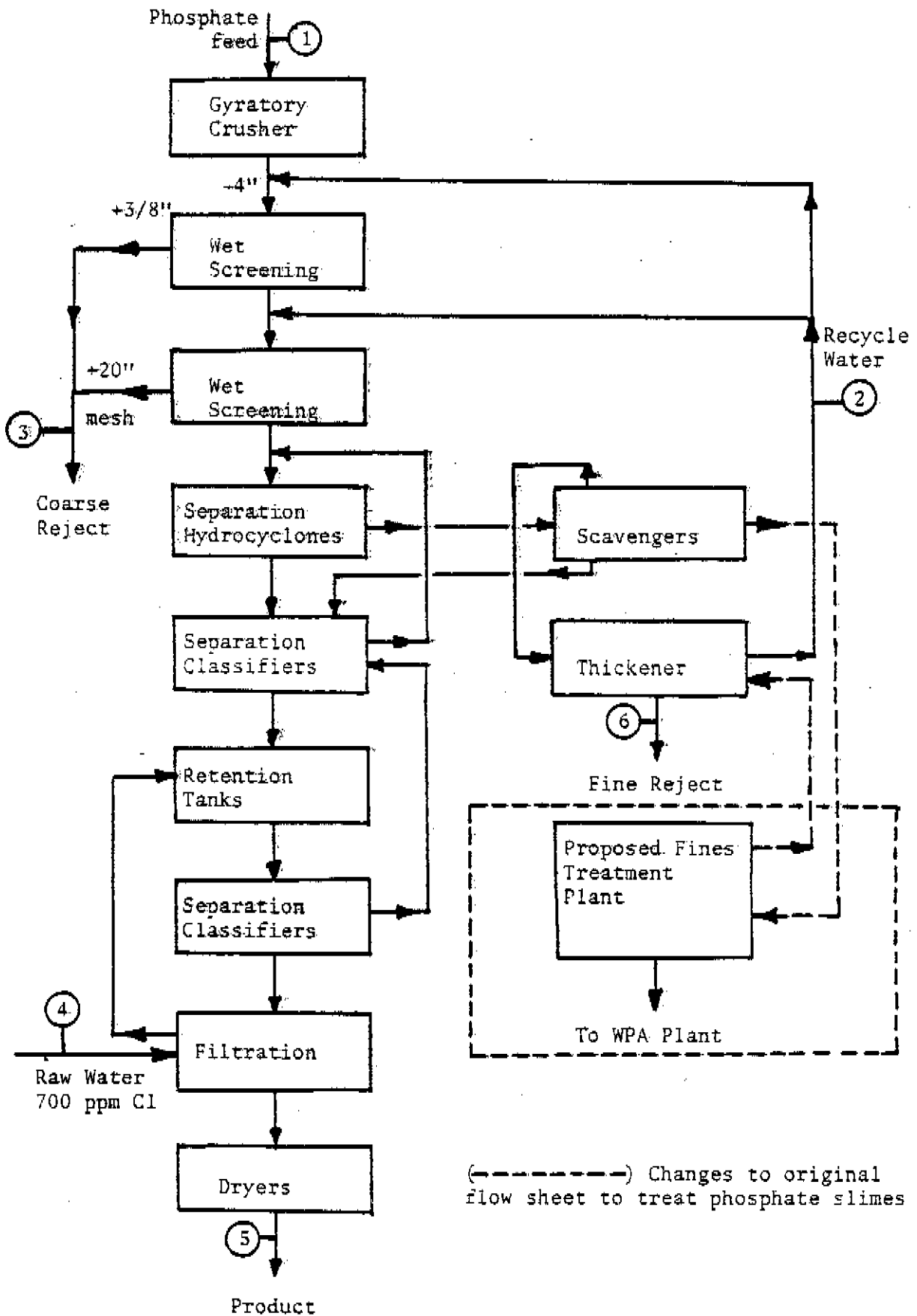
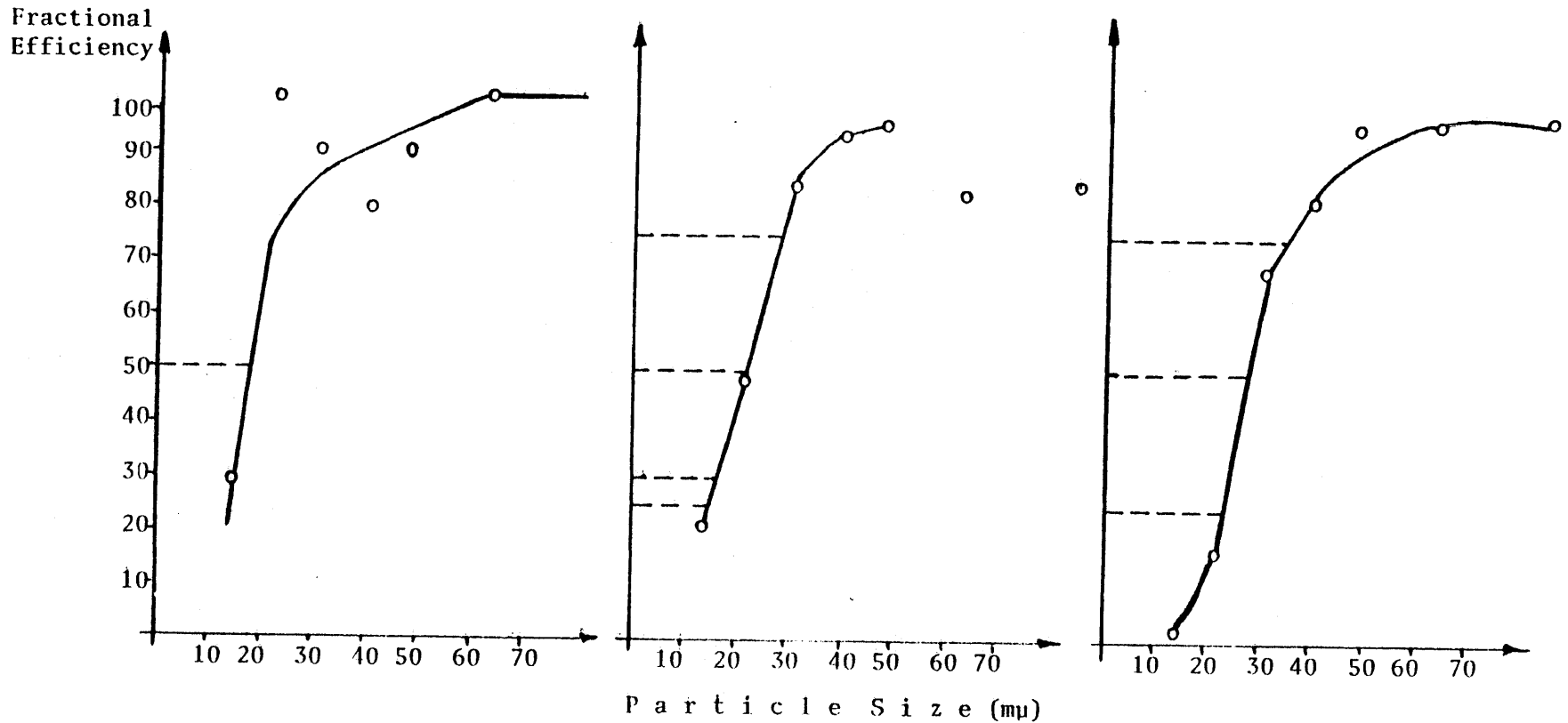


Figure 1A: Zin Plant flow sheet

	1		2		3		4		5		6	
	tph	%	tph	%	tph	%	tph	%	tph	%	tph	%
P ₂ O ₅	257	26.5	-	-	67.6	20	-	-	125.7	32.4		24
H ₂ O	90	8.5	1366	-	80	19	494				1456	65
Cl	4.85	0.5		1	1.69	0.5		0.07		0.04		1
Solids	970	91.5	-	-	338	81			387	99	246	35

Figure 2: Separation by 6" H.C. Efficiency Curves



Du = 1" ΔP = 6 PSI

μ = 55.9%

μ+20 = 91.1%

μ-20 = 72.0%

d₅₀ = 18.5μ

Sharpness of Sep. = 0.22

Du = 3/4" ΔP = 8 PSI

μ = 50.7%

μ+20 = 87.6%

μ-20 = 79.3%

d₅₀ = 23.0μ

Sharpness of Sep. = 0.26

Du = 1/2" ΔP = 6 PSI

μ = 30.4%

μ+20 = 75.8%

μ-20 = 97.9%

d₅₀ = 27μ

Sharpness of Sep. = 0.20

Figure 2A: Separation by 6" H.C. Efficiency Curves

Particle Size (mm)	Feed		Du = 1" ΔP = 6 PSI				Feed		Du = 3/4" ΔP = 8 PSI				Feed		Du = 1/2" ΔP = 8 PSI			
			O.F.		U.F.				O.F.		U.F.				O.F.		U.F.	
	wt. %	P ₂ O ₅ %	wt. %	P ₂ O ₅ %	wt. %	P ₂ O ₅ %	wt. %	P ₂ O ₅ %	wt. %	P ₂ O ₅ %	wt. %	P ₂ O ₅ %	wt. %	P ₂ O ₅ %	wt. %	P ₂ O ₅ %	wt. %	P ₂ O ₅ %
+74	9.4	29.4	-	-	12.9	30.2	8.6	29.3	1.8		9.7		11.7	29.6	0.3		23.9	30.0
53/74	6.8	27.7	-	-	9.2	28.1	7.4	26.4	2.2		10.4		3.9	28.6	0.3		17.8	24.2
44/53	8.4	26.8	2.1	-	15.1	27.7	10.7	25.4	0.9		19.3		11.0	27.8	0.6		29.3	27.6
37/44	0.7	26.5	3.7	23.7	11.5	26.6	5.0	25.4	0.9		13.6		3.9	26.7	0.6		6.7	27.3
25/37	11.9	26.1	3.2	22.0	23.0	25.8	13.8	24.3	4.5		23.7		6.5	26.6	2.8		14.2	26.4
20/25	7.0	25.2	0	-	0.4	24.2	1.2	24.1	0.9		0.8		6.8	26.0	8.7		3.9	23.1
-20	53.8	21.4	91.1	21.5	28.0	22.5	53.3	21.1	88.2		22.5		59.1	22.0	86.7		4.3	22.7
Total	100		100		100		100		100		100		100		100		100	
Solids %	4.9		2.8		12.0		4.8		2.7		19.7		5.3		3.8		56.4	
Flow Rate (m ³ /hr)	11.7		9.1		2.6		12.6		11.1		1.5		11.5		11.24		0.26	

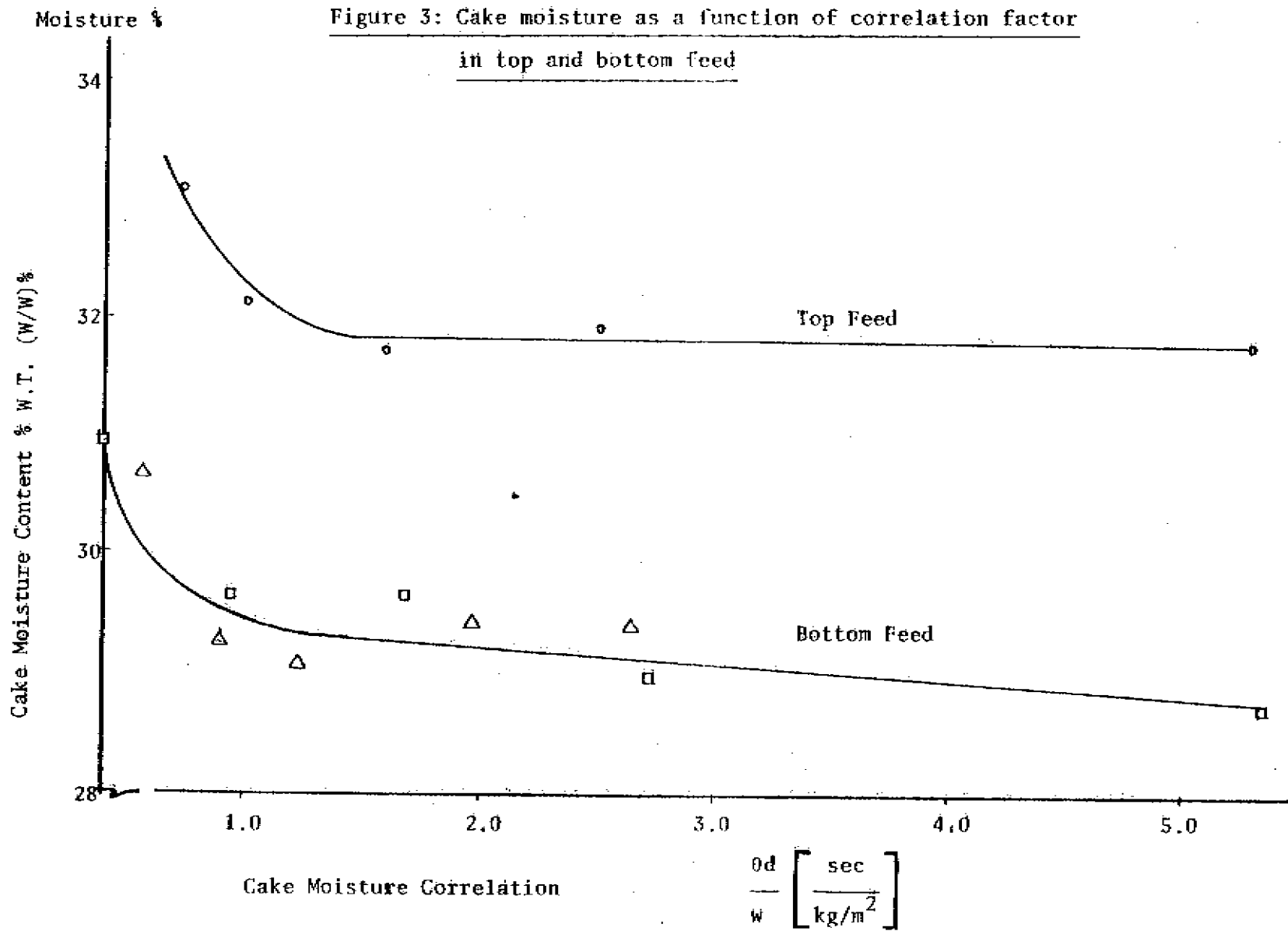


Figure 4: Block diagram for utilization of phosphate slimes for phosphoric acid production

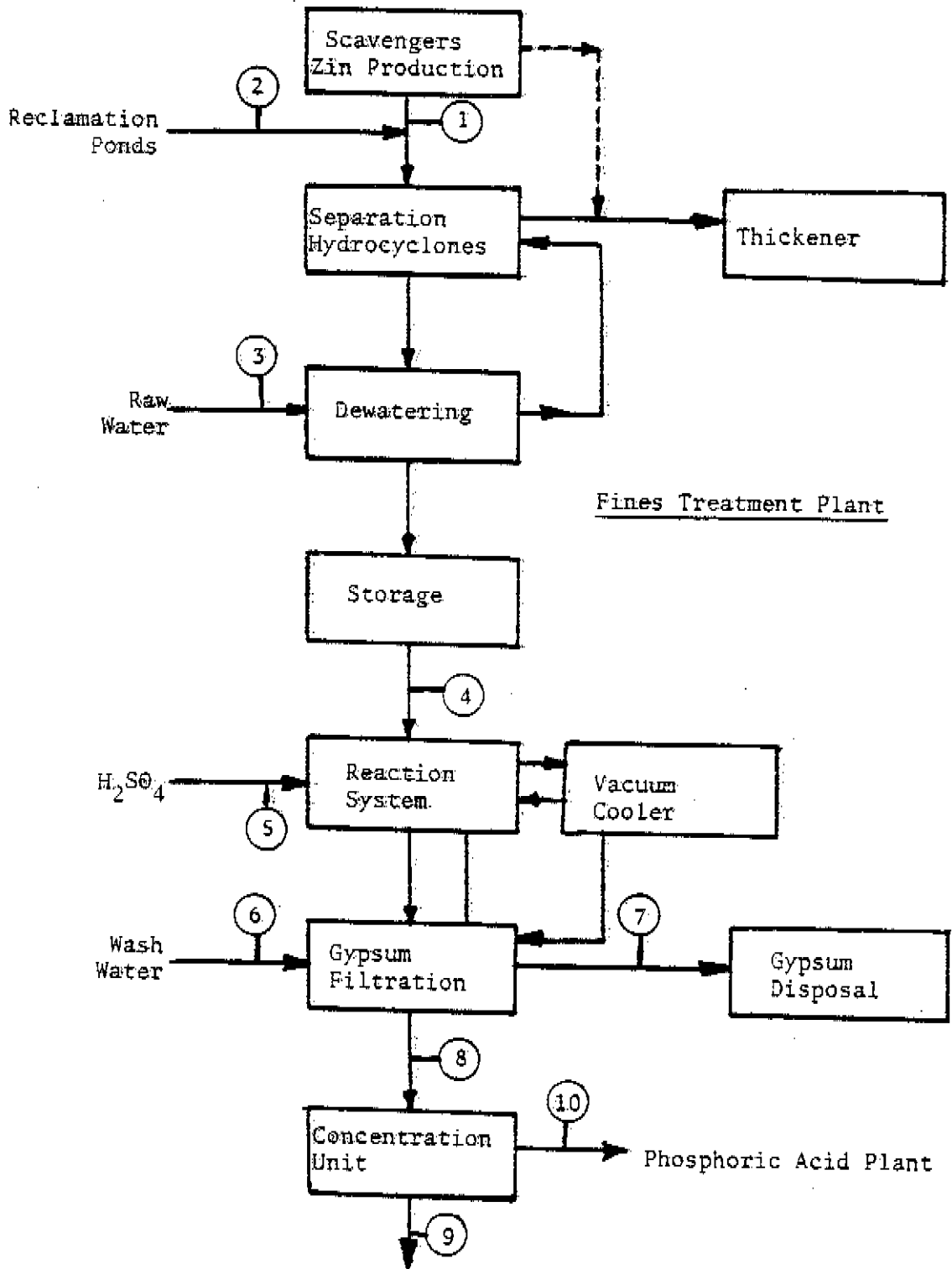


Figure 4A: Block diagram for utilization of phosphate slimes for phosphoric acid production

	1		2*		3		4		5		6		7		8		9		10	
	tph	%	tph	%	tph	%	tph	%	tph	%	tph	%	tph	%	tph	%	tph	%	tph	%
Flow Rate	7025	-	-	-	53.6	-	189	-	120	98	155	-	266.2	-	125.2	-	63.0	-	62.2	-
P ₂ O ₅	73	26	-	-	-	-	36.2	28	-	-	-	-	1.5	0.75	34.6	27.7	34.2	54.0	0.4	0.64
Cl	1.124	0.4	-	-	700 ppm	0.07	0.134	0.1	-	-	0.11	0.07	-	0.017	0.264	0.227	0.006	0.01	0.23	0.37
Solids	281	5	-	-	-	-	134	71	-	-	-	-	191.7	75	-	<1	-	<1	-	-

* as required

Figure 5: Interchangeable impeller of centrifugal mix pump for testing erosion-corrosion effects

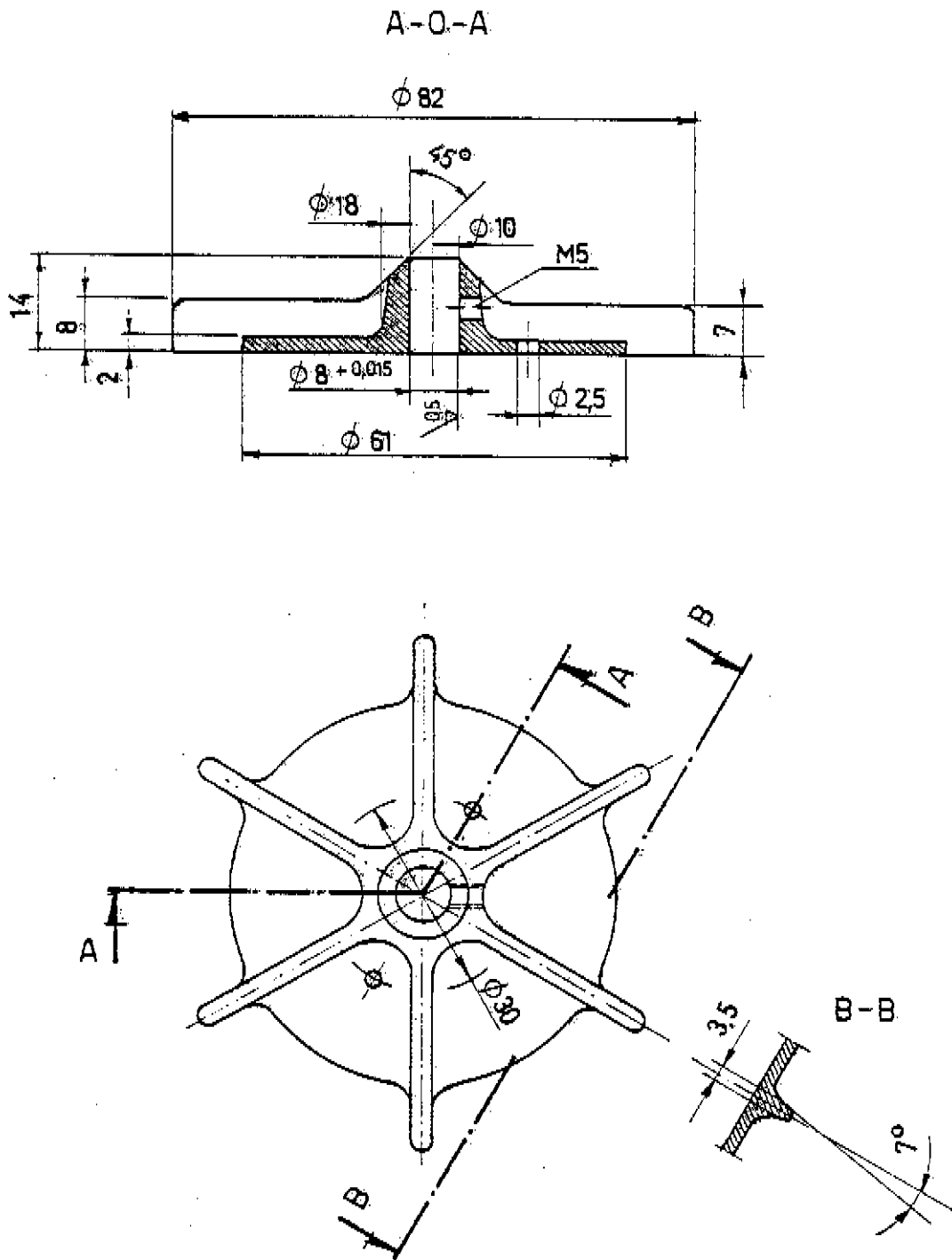


Figure 6: Negev Phosphates pilot plant for WPA

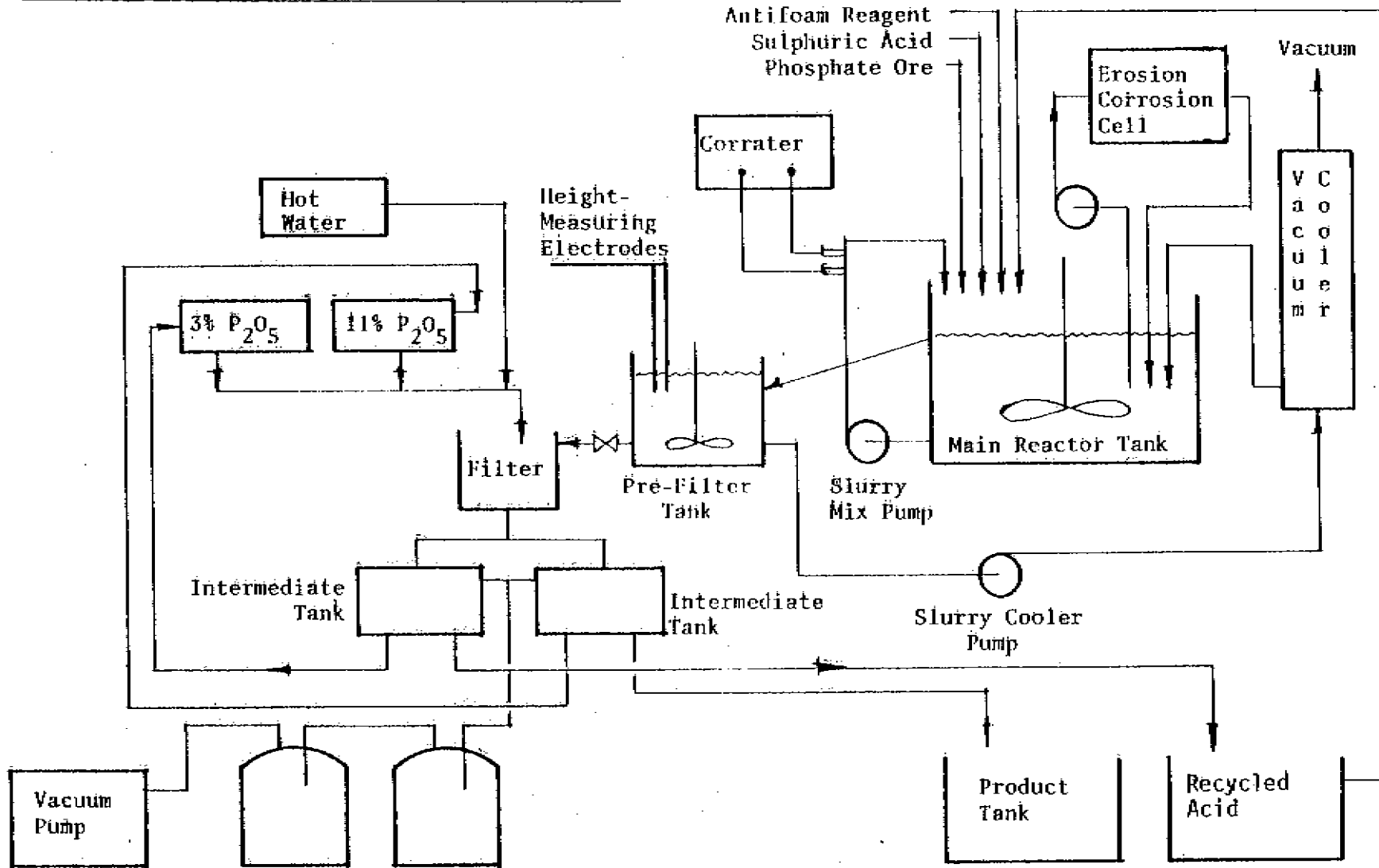
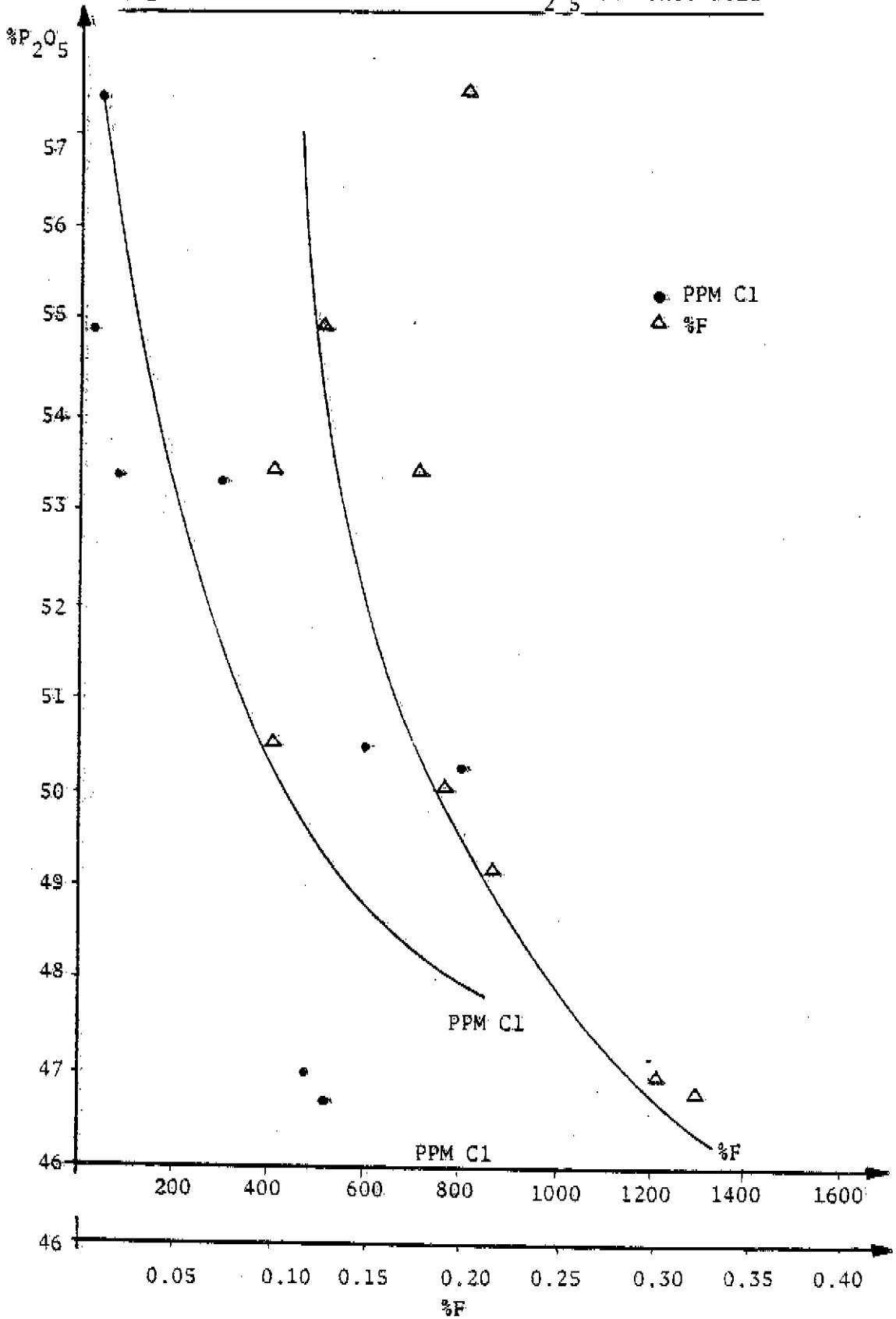


Figure 7: Conc. of Cl and F vs. P_2O_5 in conc. acid



TA/82/10 The production of phosphoric acid from phosphate slimes with high chlorine content, by J.A. Rabinowitz, M. Silberstein, R. Schlezinger, D. Fride and Y. Yagil, (Technical Division, Negev Phosphates Ltd, Israel)

(DISCUSSION : Rapporteur Mr. J.H. Markham, ICI Ltd, United Kingdom)

Q - Mr. A. NICOTRA, ANIC SpA, Italy

We can see (Table 7 and Fig. 4A) that chlorine is eliminated during the concentration of phosphoric acid with an efficiency greater than 90%. If the chlorine goes out with the vapours, how do you explain this high volatility?

A - It is a physical fact. It might be explained by the mechanism of azeotropic distillation of the mixture of acids present, including the HCl.

Q - Mr. P. BECKER, COFAZ SA, France

In Fig. 7 - at what temperature was the evaporation conducted?

A - Normal concentration temperatures under vacuum conditions as in standard phosphoric acid plant operation i.e. 80-85° C.

Q - Mr. J. LE PAGE, SCPA, France

a) What is the water content of the 6 "Hydrocyclone underflow"?

b) What types of vacuum filters did you try?

c) How do you handle the filtered slimes (at 30% H₂O) between filter discharge and the reaction tanks?

A - a) Between 45 and 50% water.

b) Top and bottom Pilot vacuum filters.

c) On a normal conveyor belt.

Q - Dr. T.R. BOULOS, Misr Phosphate Company, Egypt

a) Table 2 - The wt% of the -20 microns should be 55.8% instead of 56.8%. By rejecting this fraction during beneficiation you will be making use of just 13.3% by weight of the total feed of the Zin plant. Do you think it is worth acidulating such a high Cl and CO₂ containing product?

b) Table 5A shows that the CO₂ content of the fines is 6.1% and the CaO/P₂O₅ ratio is 1.8, which means too much calcite in the acidulation feed. Do you think that you can tolerate that commercially?

c) Table 7 indicates that you obtain 49.4% P₂O₅ concentration acid with 0.6% MgO. Do you think that you will be able to reach 54% P₂O₅ acid and what will be the MgO content of this product?

A - a) The result of many tests show that by cutting the fine material at 500 mesh about 50% of the coarser material can be recovered (200-500 mesh) with a P₂O₅ content in the solids of around 28%. We think that it will be very economical to use this material due to the fact that its cost is almost zero. As far as the chlorine content is concerned there are two possibilities:

i. The chlorine could be washed out

ii. Even without washing the material could still be used provided that the correct materials of construction are used in the plant.

b) The answer given in (a) covers this question.

c) We are sure that we can achieve the 54% P_2O_5 concentration in the product acid and the magnesium concentration will be changed accordingly.