

# **IFA Technical Conference**

Edmonton, Canada 12-15 September 1988

## HEMIHYDRATE OPERATING EXPERIENCE AT BRUNSWICK MINING AND SMELTING FERTILIZER FACILITIES

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#### INTRODUCTION

The fertilizer complex located in Belledune, New Brunswick, Canada, a Division of Brunswick Mining and Smelting, has the primary function of consuming the sulphuric acid produced from the lead smelting facility to produce bulk di-ammonium phosphate for markets in eastern Canada and the north-east United States.

The phosphoric acid process was converted from Prayon dihydrate to hemihydrate during the scheduled plant shutdown in 1986 and was commissioned in September of the same year. The basis for change was cost efficiency, a direct result of reduced energy requirements due to the elimination of the evaporation stage required to concentrate phosphoric acid from 30 to 40 percent  $P_2O_5$ .

Prior to conversion steam requirement was 1600 kg per tonne of  $P_2O_5$  produced, and this was attained from two 34,000 kg per hour, boilers operating on Bunker "C" fuel oil. Our studies indicated that the project would be economically favourable, and the objectives established were determined to be as follows:

- Process steam was expected to decrease from 1600 to 400 kg/tonne  $P_2 0_5$ .
- Sulphuric acid consumption would decrease by .03 tonne/tonne  $P_2O_5$ .
- Power would decrease by 10 kwh/tonne P<sub>2</sub>0<sub>5</sub>.
- Salt water would be reduced by 23000L/tonne P<sub>2</sub>0<sub>5</sub>.
- Direct labor would decrease by 6 personnel.

These benefits were expected to be somewhat offset by:

- Increased rock consumption due to lower filter recoveries of 93 percent.
- Addition of antiscalent required to prevent scale formation on filter pans, downlegs and the plant effluent line.

This paper will provide a brief description of the hemihydrate retrofit, assessment of plant performance during startup, and review of 1987 operating results as compared with the stated objectives.

#### HEMIHYDRATE PLANT CONVERSION

The original dihydrate reactor as illustrated in Figure 1, consisted of 9 compartments with phosphate rock and dilute sulphuric acid fed to C-1 and temperature control achieved by pumping the contents of C-8 through the flash cooler to C-9. Slurry from C-9 was then pumped to the Prayon Filter to separate the gypsum dihydrate and phosphoric acid  $(30 \ P_2 O_5)$ .

The dihydrate reactor was converted to hemihydrate as illustrated in Figure 2 by the following modifications:

- Reactor 1 consisted of compartments C-1 to C-6 with a slurry volume of 420 m<sup>3</sup>.
- . Reactor 2 comprised compartments  $C_7$  and  $C_8$  with a slurry volume of 260 m<sup>3</sup>.
- . Compartment C<sub>9</sub> is the filter feed tank and can hold a volume of 130m<sup>3</sup>.
- . A pump suction nozzle was added to the side of C-7 to directly feed the flash cooler pump and this flow is circulated to C-8.
- A pump suction nozzle was installed at the side of C-8 and this flow recirculates the sulphuric acid and slurry added into C-8 to digest the rock fed into C-1.
- . The filter feed pump from compartment C-9 was located at grade utilizing the existing drain nozzle.
- Agitators in compartments C-1, C-2, C-7, and C-8 were replaced based on Norsk Hydro design to ensure improved agitation in the most critical compartments. The new agitators were made of 904L and consumed less power than the original radial turbine agitators.

The sulphuric acid and recycle acid flows are introduced to compartment C-7 by a conical mixer as shown in Figure 3. In addition to 93% sulphuric acid, provisions were also made to add 70% sulphuric acid. These flows are metered separately by ratio control to ensure the combined streams do not fall below 86 percent strength, in order to minimize the adverse effect on filtration losses.

Figure 4, illustrates the attack fume scrubber, required to handle the increased load of reactor fumes generated due to the higher  $P_2 0_5$  concentration and temperatures within the hemihydrate process. The existing fume ductwork was left in place to act as a bypass and afford inspection of the attack scrubber while maintaining process ventilation.

Figure 5, illustrates the flash cooler scrubber addition used to recover heat to obtain 50 dg. C water for the filter wash and eliminate further necessity for process steam. The flash cooler vapors are prescrubbed to remove fluorides and the remaining vapor is absorbed in fresh water to produce the filter makeup water.

The filter is not illustrated; however, changes required were the addition of 317L S.S. sloped bottom pans and the filter hood was extended to cover the first strong wash area. A proprietary anti-scalent system was directed to key points of the filter to eliminate scale formation due to conversion of hemihydrate to dihydrate.

#### PLANT START-UP

The plant was started up on-schedule in September, 1986 with minimal operating problems and by September 19 had achieved 110% of capacity with filter recoveries in the order of 94 percent – a percent above the guaranteed figure. The higher purity of the product acid resulted in extremely high grade in DAP product –  $(18.2\%\ N\ and\ 48\%\ P_2O_5)$  which directed our attention to adding diluents to prevent over-formulation and subsequently further reduce operating costs. The performance run was conducted in early October and plant acceptance was achieved based on the following performance data.

<u>Criteria</u>	<u>Guaranteed</u>	<u>Actual</u>	
Production, tonnes P <sub>2</sub> 0 <sub>5</sub> per day	500	512	
Recovery, filter cake	93.0%	93.4%	
Sulphuric acid, tonnes H <sub>2</sub> SO <sub>4</sub> /tonnes P <sub>2</sub> O <sub>5</sub>	2.7	2.63	
Defoamer, kg/tonne P <sub>2</sub> O <sub>5</sub>	2.5	0.9	
Antiscalent, kg/tonne P <sub>2</sub> O <sub>5</sub>	1.2	1.0	
* P <sub>2</sub> 0 <sub>5</sub>	40.0	39.5*	

 Acceptable concentration since cloudy filtrate section of filter not required.

Plant operating conditions maintained were as follows:

- . C-8 slurry: 33.6% solids, 1.79 S.G., 41.2%  $P_2O_5$ , 2.05% free  $H_2SO_4$ .
- . C-2 Temp: 99.3 dg. C.
- . Flash Cooler: 100.6 dg. C In, 97.3 dg. C Out, 330 mHg vacuum.
- Filter: 57 dg. C wash water, 200 sec/rev., 55 mm cake, 380 mmHg vacuum.

#### HEMIHYDRATE OPERATING EXPERIENCE

After the excitement of the smooth startup in October had subsided, we started to experience some operational difficulties with processing equipment and the following discussion will highlight these problems and the measures taken to overcome them.

#### ATTACK TANK REACTOR

- . During startup the agitator motors in compartments  $\rm C_3$  to  $\rm C_6$  were overloaded and this problem was overcome by cutting 9cm off the top blades.
- The pyramid in C-9 came apart just after we had completed the transition from di to hemihydrate; however, this was found to be non-process related. The compartments were drained and inspected and we found that some of the curb bricks had come loose from C-8. There also was some minor damage to the slurry recirculating pump neoprene liner. Since it was evident that the new process conditions was dissolving old gypsum, we expected problems with other pyramids and curbs and as a precaution we installed screens at the inlets to the recirculating and flash cooler pumps.
- . The system was commissioned and the guarantee test completed using Brunswick Smelting 93%  $\rm H_2SO_4$ . The Boloeil acid (70%  $\rm H_2SO_4$ ) system was started in October resulting in lower  $\rm P_2O_5$  strengths (39%) and subsequently lower filter efficiencies. Our learning curve experience indicated that combined strength of 90% maintained efficiences at the filter above 94 percent.

#### ATTACK TANK ASSOCIATED EQUIPMENT

- The flash cooler pump, recirculating pump and filter feed pump have all functioned satisfactorily since startup with the exception that the mechanical seals failed and were replaced by conventional packing. The increased water to the process did not create problems as strengths of 42% P<sub>2</sub>O<sub>5</sub> can easily be maintained.
- The attack fume scrubber is inspected every 2 weeks with cleanup required only during the summer shutdown. During the 1987 shutdown on inspection we found that corrosion in this area had reduced expected equipment life and we had to rubber line the bottom half of the scrubber.

#### REACTOR STARTUP AND SHUTDOWNS

- The plant operates on a 5 day cycle and during shutdowns the pumps and pipelines must be drained. Startups are relatively smooth with 100% rate achieved within 12 hours provided steam is added to compartments C-2, C-4 and C-8. We have restarted the process after a weekend shutdown without steam; however, filtration losses are high for the first 12 hours due to insufficient attack temperatures and poor crystal formation.
- . One phenomena observed during the period December-March is that during our weekend shutdowns large scale will fall off the attack roof and the inlet screens have to be cleared to maintain proper process flow conditions. In order to overcome these problems we shutdown the Teller Scrubber ventilation fan and use the Booster Fan to lower heat loss and provide adequate ventilation of reactor fumes.

#### FLASH COOLER SYSTEM

- . The filter process hot water wash requirements are generated by capturing the heat from the flash cooler vapors. The flash cooler scrubber addition has proven effective in providing the process water required; however, it has also been a major problem area and many modifications have been made to overcome difficulties.
- Hot water from this source is sufficient for filter requirements when attack rate reaches 90%. During initial startup, steam is added to the filter wash water tank and the water from the flash cooler used as makeup to this tank.
- . Initially the first section of the scrubber plugged solid after 3 operating days and this problem was overcome by relocating sprays closer to the scrubber inlet duct and turning them downward. The discharge downleg was also enlarged and redirected to prevent solid blockage in this area. The system now operates as expected but has to be cleaned on a weekly basis before startup.
- The low fluoride levels in the hot water supply to the filter wash water tank and filter wash create corrosion problems and all associated equipment had to a replaced within 3 months of operation. The filter wash water tank and associated piping was rebuilt with 316L stainless steel. This piping also has deteriorated and we are now replacing it with high density polypropylene material.

#### PRAYON FILTER AND ASSOCIATED EQUIPMENT

- The Bird 24C tilting pan filter was modified by replacing the old flat bottom pans with newly designed sloped bottom 317L S.S. pans with the screen area changed to circular openings. Initially cloths used were 120 cfm to prevent scaling; however, with the improved grade at the DAP plant cloths were changed to 180 cfm to add diluents. During plant shutdowns the filter is put on hot water wash and we have not experienced any difficulties with scale formation in this area.
- The initial fume hood extension to cover half the No. 2 cut (strong acid wash) did not provide enough ventilation and this was extended to include the No. 3 cut  $(17 \text{ R P}_20_5 \text{ wash})$ . We also added a roof fan above the filter wash water trough to improve ventilation in this area.
- It should also be noted that fume ductwork from the filter must be inspected and cleaned on a regular basis to ensure adequate ventilation is maintained at the filter.
- Filtration has been excellent to date and rates up to 110 percent can easily be achieved with filter efficiences of 94.5 percent attainable. When the 70% H<sub>2</sub>SO<sub>4</sub> system is in use, efficiences will decrease in accordance with the amount used; however, at 90% combined strength and 100 percent rate the above efficiency can be maintained. The gypsum crystals achieved in the process are abnormally large with a measured specific surface area of 985 cm<sup>2</sup>/gm and this is believed to result from the use of finer rock than the process requires.

#### GYPSUM EFFLUENT SYSTEM

- This has proven to be our most difficult problem area as it became apparent in November, 1986 that the main pipeline was scaling up at the last 1000 foot section before discharge and the hemihydrate was not dissolving readily in salt water as evidenced by the large island formation at the discharge.
- In the dihydrate process, gypsum was allowed to flow by gravity into the discharge with the effluent pumps used as boosters to force the effluent to the bay. During the period November to April, 1977 we had to disconnect the effluent line in 100 foot sections and physically remove scale in order to keep the plant operable. We also had to divert the filter discharge directly to the gypsum sump and subsequently had continuous erosion problems with these pumps and associated high costs due to monthly pump replacement. Once the outside temperatures improved, scaling problems in the line became minimal; however, the plant operated at 90% rates due to the effluent pumping problems.

#### GYPSUM EFFLUENT SYSTEM CONT'D

- . The dissolution problem was researched with Norsk Hydro personnel with testwork indicating that partial reconversion to dihydrate was necessary to reduce crystal size and subsequently keep solids in suspension. At the same time a new effluent system was being built in order to reduce pipe size diameter from 60 cm to 36 cm and increase pipeline velocity from 1 to 3 m/sec. These measures would ensure a full pipeline and minimize scaling in this area.
- The new effluent system (Figure 6) was commissioned in January, 1988 and has proven to be very successful. Agitation is achieved by the tangential flow of salt water and the new velocity at the discharge is 2.9 m/sec. Samples taken at the inlet and pump discharge indicate that specific surface area has decreased from 1000 to 4300 cm²/gm and 30% conversion from hemi to dihydrate has occurred. The gypsum is now in suspension and is readily dissolved in salt water thus overcoming one of our major concerns. Maintenance costs and cleanup costs have been substantially reduced and we expect that our dissolution problems at the discharge will now be corrected.

#### ECONOMICS OF HEMIHYDRATE CONVERSION

A post audit study has recently been completed to assess 1987 operating performance as compared with the stated objectives and 1985 operating costs. Data is summarized in Table 1 and the results are as follows:

- Process steam decreased from 1600 to 500 kg/tonne P205 for a cost saving in the order of \$1,500,000. Boiler inefficiencies at the reduced load account for the deviation from expected results and the next phase is to eliminate this operation and replace them with small steam generators located within the acid plant.
- . Sulphuric acid used for digestion decreased from 2.61 to 2.53 tonnes/tonne  $P_20_5$ ; however, additional acid was used as a diluent in the DAP reactor to offset this reduction. The use of sulphuric acid for grade control resulted in an incremental increase in production with an associated cost saving in the order of \$600,000.
- . Power consumption decreased from 185 to 170 kwh/tonne  $P_2^0_5$  resulting in savings in the order of \$50,000.
- . Salt water consumption decreased by 15000L/tonne  $P_20_5$  for savings in the order of \$50,000; however, fresh water usage increased by \$90,000 negating this benefit.
- Direct production labor decreased by 4 personnel; however, the new process has proven to be more maintenance intensive with the net effect that labor costs have increased in the order of \$100,000 per annum.
- . Phosphate rock consumption has increased from 3.32 to 3.38 tonnes/tonne  $P_20_5$ ; however, efficiences are in the order of 1 percent above that expected.
- . Process antiscalent has been controlled at 1.00 kg/tonne  $P_2O_5$  as compared with initial requirements of 1.25 kg/tonne  $P_2O_5$  for an operating cost in the order of \$150,000 per annum.
- The initial capital budget was set at \$4 million with an expected annual return in the order of \$1.75 million (antiscalent not included) and had a DCF rate of return of 92 percent.
- The project was completed on schedule with costs well under budget at \$3.4 million; however, installation of the new effluent system increased project cost to \$4.1 million. Operating costs in 1987 have been reduced by \$1.5 million resulting in a DCF return of 54% as compared to a revised DCF with antiscalent included of 63%.

#### SUMMARY

- The conversion to hemihydrate has proven to be economically favourable and further improvements are expected as we progress on the learning curve and further reduce operating costs.
- Further energy savings will result with the boiler elimination and a cost reduction in the order of \$.5 million annually is expected.
- Considerable pre-study of operating plants has proven beneficial and the high emphasis given to training, resulted in a relatively smooth startup and enhanced ability to solve subsequent problems.
- . Most of the objectives of the new process were achieved; with the exception that the process was more labor intensive than expected due to more rapid deterioration of process equipment. In retrospect, some of the equipment left in place should have been changed during the plant conversion.
- The gypsum effluent system was our most serious concern and it is now apparent that we have this problem under control. This is an area that must be thoroughly studied for those operations considering similar conversions.
- Quality of diammonium phosphate has been excellent leaving us the more favourable problem of solving overformulation and subsequently reducing operating costs.

## REFERENCES

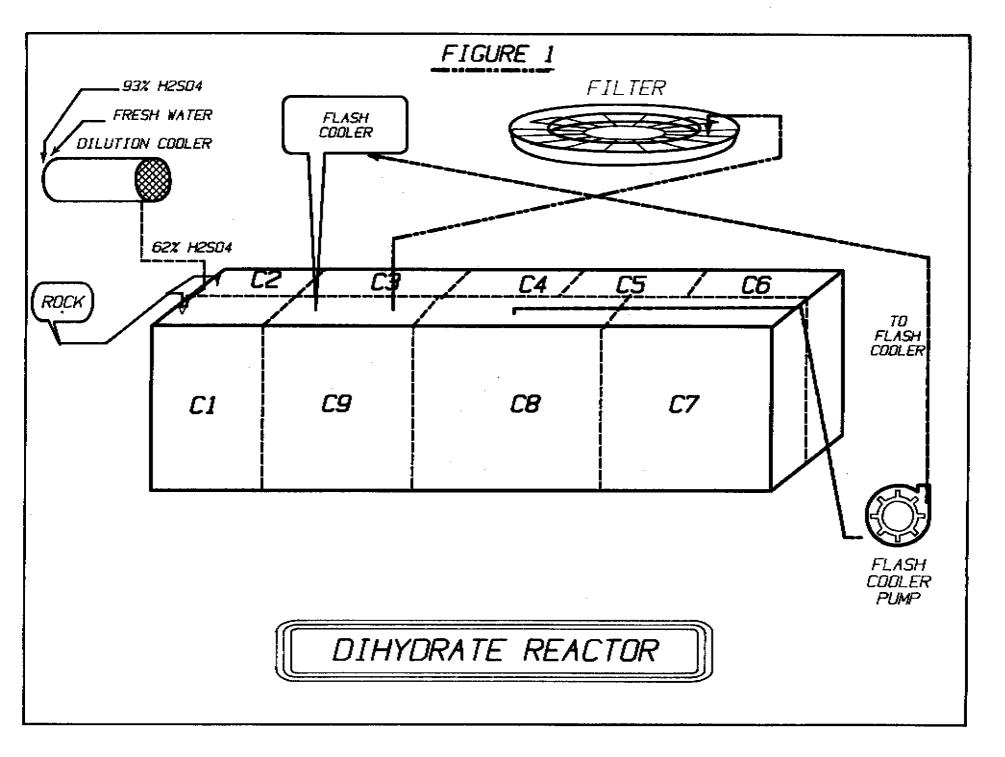
- J.H. Wing "Hemihydrate Phosphoric Acid Plant Conversion at Belledune, Canada", AI Che Meeting, Lakeland, Florida, U.S.A. October 13, 1987
- T. B. Gravestock "Hemihydrate Post-Audit Study" April, 1988

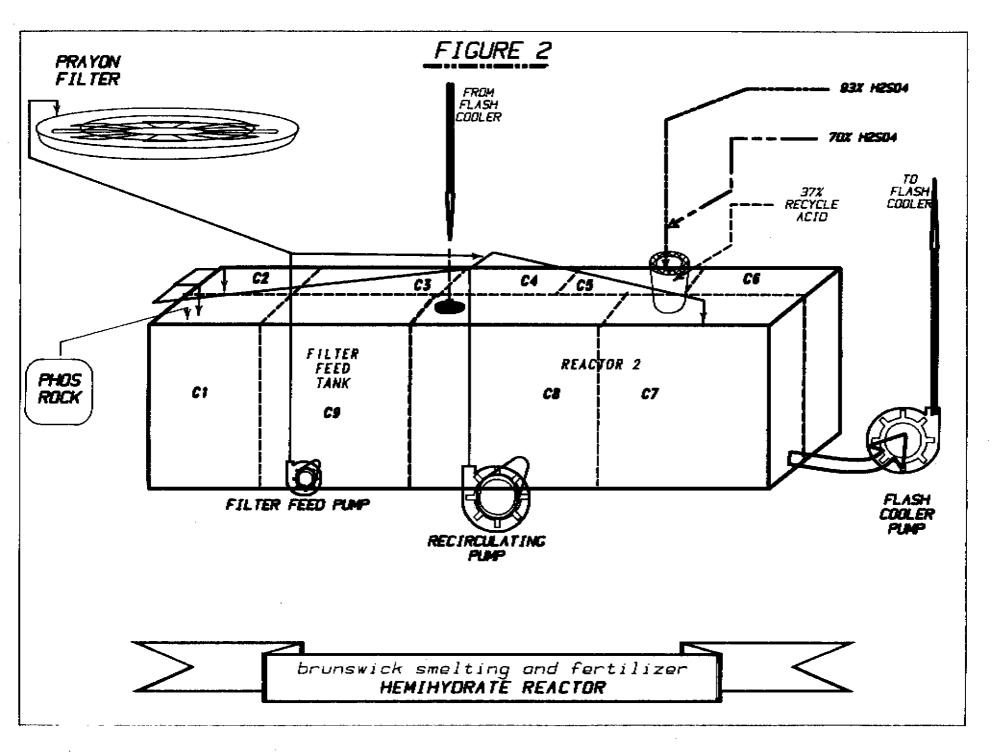
## **ACKNOWLEDGEMENT**

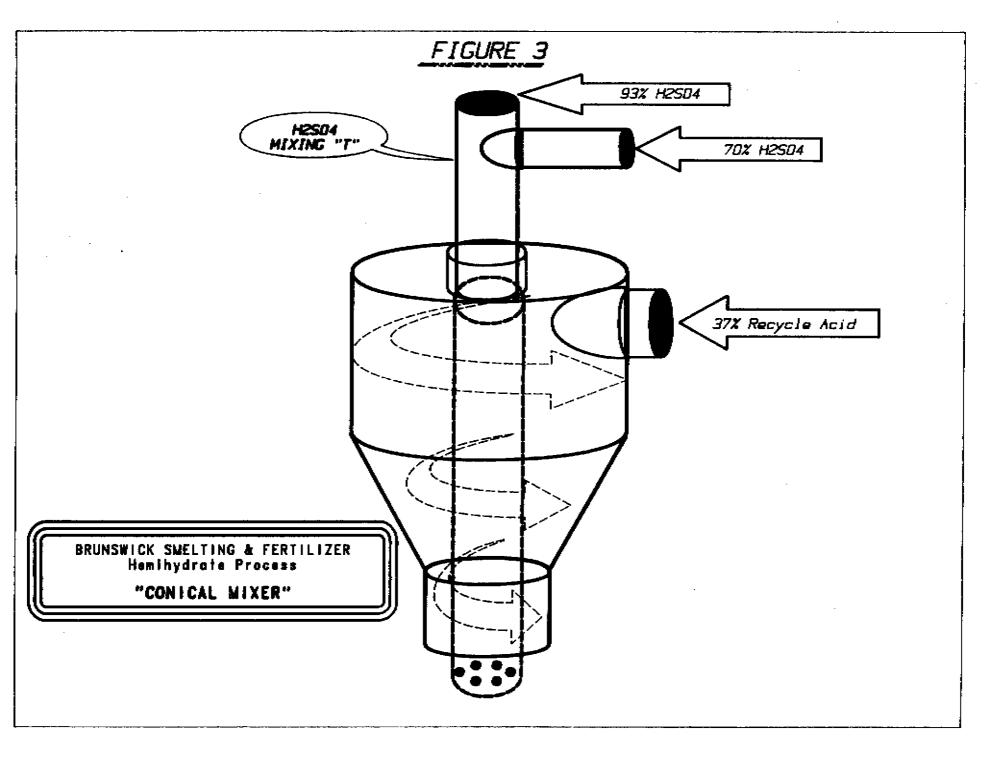
My thanks are extended to R. Stairs (Modular Training Coordinator, B.M.S.) for preparation of drawings included in this presentation.

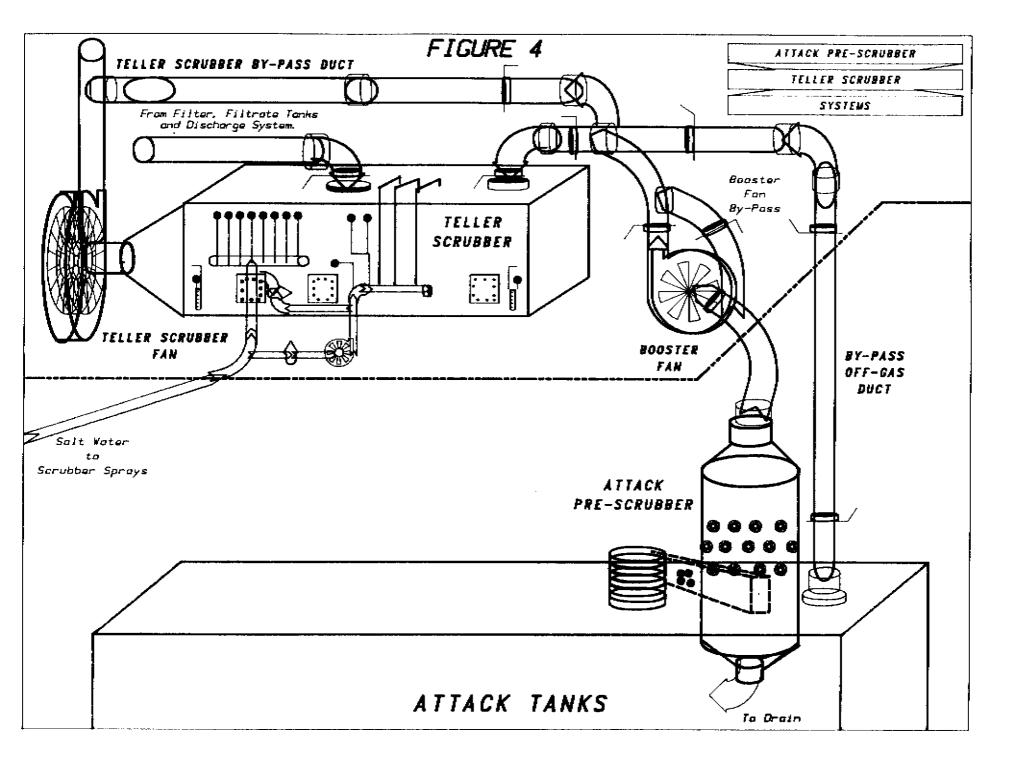
TABLE 1
SUMMARY OF PLANT DATA AND SAVINGS ACHIEVED

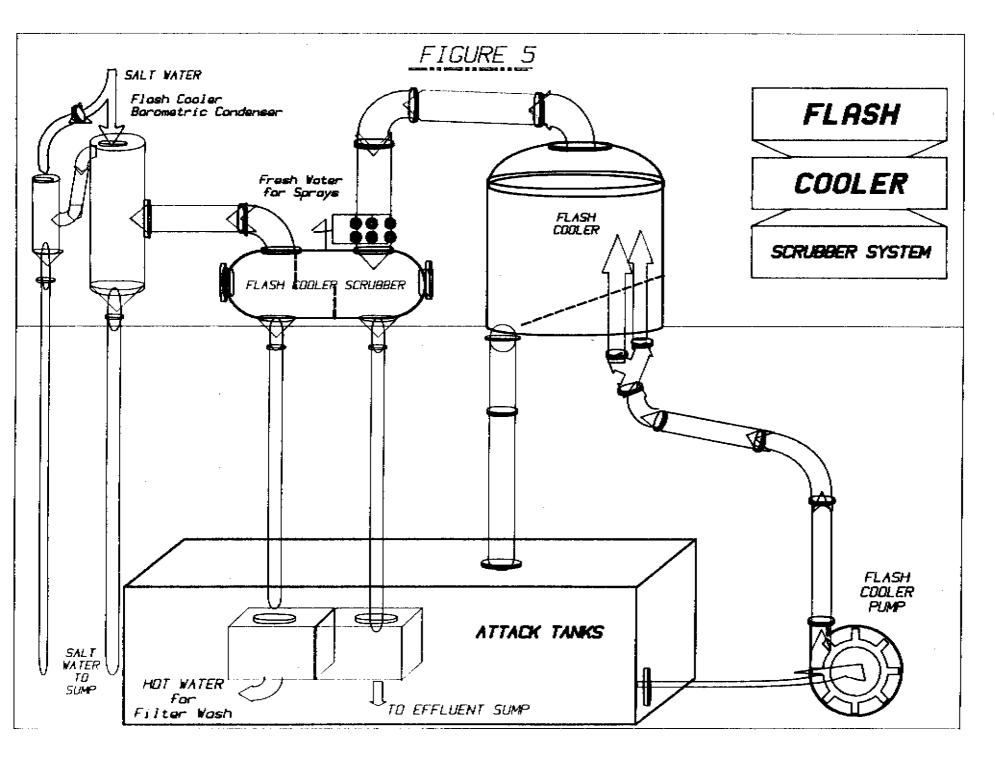
		1985	1987 S	AVINGS (\$000)
1. 0	STEAM (Kg/T. P205)	1,600	500	1.500
2. 0	SULPHURIC (T/T.P205) ACID	2. 61	2, 5 <b>3</b>	20
3 <b>.</b> D	POWER (KWH/T. P205)	185	170	50
4. D	SALT WATER (L/T.P205)	49, 000	34.000	50
5. D	FRESH WATER (L/T. P205)	11.000	22. 000	(90)
6 <b>.</b> 0	ANTISCALENT (Kg/T. P205)	NIL	1.00	(150)
7 <b>.</b> D	PHOSPHATE ROCK (T/T. P205	) 3.32	3. 38	(250)
8. O	INCREASED LABOR & MATERIAL			(250)
9. 0	INCREMENTAL PRODUCTION INCREASE			570
10. 0	TOTAL SAVINGS			1,500

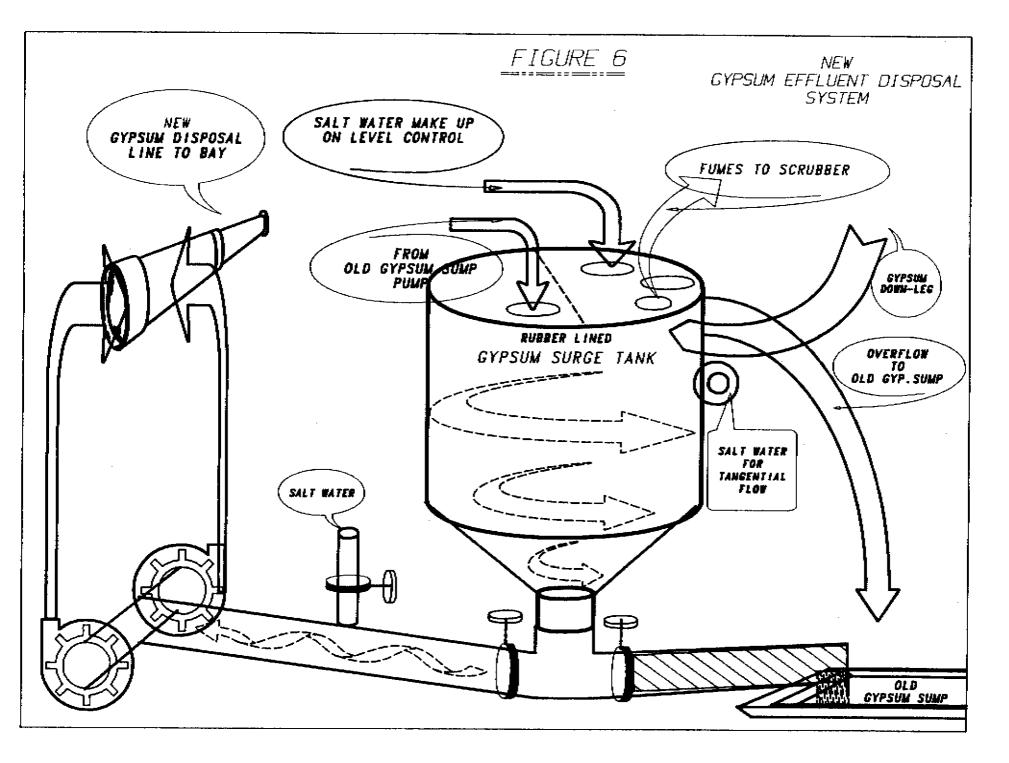












TA/88/17 Hemihydrate operating experience at Brunswick Mining and Smelting Fertilizer facilities by T. Gravestock, Brunswick Smelting and Fertilizer, Canada

DISCUSSION (Rapporteur Mr.C.M.H. Vincke, Windmill Holland, Netherlands)

- Q Mr. T. LAINTO (Kemira Oy, Finland)
  - I have two specific questions concerning your plant:
  - 1/ Do you have any limitations on the discharge of gypsum into the sea today and how do you see that situation in the future ?
  - 2/ I understand that you are running your plant five days a week. How does this affect the P2O5 losses? And do you have some special arrangements to decrease these kinds of losses?
- A The first question was the environmental impact of gypsum. With the dihydrate process, our gypsum bed was increasing at a normal rate of 11% per year. When we did convert to hemihydrate we ran into a problem. The actual rate increased to 40%. We definitely do have environmental restrictions, environmental policies in New Brunswick. We have had to do many different surveys, medical surveys, lobster studies for fluorides and this is still continuing with the participation of the government. In order to start the dredging process, we did have to get a special permit because there is lobster fishing just approximately 1,000 meters off the point of discharge.
  - 2/ We have special arrangements to decrease losses. The plant operates on a five-day basis mainly because our sulphuric acid is received from the smelter next door and our operating policy is to consume all the acid produced by the smelter. We are also pursuing other sulphuric acid sources, looking for waste acids and looking at Noranda's intention to expand in the future. There is no doubt that P205 losses are greater shutting down on this weekly basis. You lose them on start-ups, because, essentially, over the week-end, the reactor contents cool from  $100^{\circ}$  C down to about  $65^{\circ}$  C. So initially you have to restart up on steam to  $80^{\circ}$  C, start your reaction, and it takes about four hours before the filter gives you the higher efficiencies. On the shut-downs, basically, there are no particular losses.
- Q Mr. L. RASMUSSEN (Superfos Fertilizers, Denmark)
  - 1/ About process economy, in table one, you have summarized the achieved savings. What is the steam price used in your calculation?
  - 2/ Filter loss calculations can be based on filter cake samples or gypsum slurry samples. You indicate that the recovery, based on filter cake samples, is approximately 93%. Have you made loss calculations based on continuous gypsum slurry camples and, if so, what are the results? Have you any reliable figures for the overall plant P205 recovery for the two processes?
  - 3 In your table 1, you have given no credit for a better acid quality. Did you not get any ?
  - 4/ Have you any plans for the introduction of a two-stage process in your plant ?
- A 1/ The steam price, generally, when we first started off in the evaluation, was \$ 8 per thousand pounds of steam. Naturally, over

the two-year period, when we decided to go hemi, energy prices dropped. I evaluated the steam savings by taking actual 1985 operating data, correlating it to 1987 pro rata per tonnage changes and costs to form a comparative base.

- 2/ The gypsum efficiencies are based on a continuous sampling of gypsum cake. We have an automatic sampler which takes samples approximately every fifteen minutes. The sample is taken to a lab on a 24-hour basis. As an example, the P205 in cake has been running 1% versus 0.8% with the dihydrate process. We also look at overall P205 efficiency. With the dihydrate process, our normal efficiency across the plant was about 88%, and with the hemihydrate process, we are getting about 87.2%.
- 3/ I took credit for acid quality. Definitely, there is a credit for acid quality because, in this particular process, the aluminium content of the 40% acid is approximately 5%. With the lower impurities in the acid, we found that what we tried to do is to start with 120 c.f.m. plus. We found that adding sulphuric acid to control grade, we still had high results. We then went to 180 c.f.m. plus to bring the solids up. The other thing we found was that we used two different types of rock from Florida. We used an Estech rock and a Mobil rock. One particular rock we had to clarify to 1% levels. The other rock, we could let go to 2.5% and still make grade. And, naturally, you could use solids to make DAPs more economically.
- 4/ As far as our future plans go, one of the reasons we were able to, essentially, sell the idea of hemihydrate to our Board of Directors, was that we indicated that the first step of this technology was a hemi process with future plans to go into dihydrate conversion. As you know, we receive rocks from Florida, so it is expensive and there is some economic benefit to then take it a step further to the dihydrate route. So there are no plans at the moment to go that way, but there definitely could be in the future.
- 5/ Gypsum sturry loss is calculated basically on the standard calculation of cake. It is gypsum-cake efficiency based on a mature balance of P2O5 and calcium oxide.
- Q Mr. R. HUTCHINS (Texasguif inc. USA)
  - 1/ You mentioned that you use Florida rock. Do you grind the rock?
  - 2/ You mentioned in the paper that the antiscalent system that you use is proprietary, but could you tell us what you can about that system and what antiscalent agent you use ?
  - 3/ Do you use an additive to control your rehydration rate?
  - 4/ You indicated that your filtration rate had been excellent with rates up to 110% being achieved. What is that a percent of ?
  - 5/ How does that compare with the filtration rate when you are operating in the dihydrate system ?
- A 1/ Yes, we do grind the rock.
  - 2/ Unfortunately, I cannot discuss the antiscalent agent, but I can mention that, initially, we started off with the antiscalent at the

levels recommended by Norsk Hydro. Every month, we took apart the central valve at the filter, checked the down legs for scale and found there was no scaling. So what we started doing was withdrawing the use of antiscalent in these areas. The last two months before our summer shutdown of this year, we took the antiscalent off totally. We found that, basically, if you have hot water strategically located at the proper locations, you do not get scaling in the hemi process. So our antiscalent addition right now, instead of being as recommended 1.2 is in the area of 0.2 to 0.3 kg per ton.

3/ We do use an additive to control our rehydration rate, this is the purpose of it, to inhibit conversion to dihydrate.

4/ The design filtration rate is 500 tons/day P205.

5/ In the dihydrate system, we had problems at 500 short tons/day. Our previous dihydrate system was a 500 ton/day plant generally running at 95.5% efficiencies, but when we pushed the plant beyond this 500 tons/day, our efficiencies dropped off pretty quickly. With this particular process, we can get up to 600 metric tonnes and maintain the hemihydrate efficiencies. We believe that it is mainly due to the nature of the crystal that we are getting which we also feel is the result of grinding the rock.

Q - Mr. P. SMITH (Prayon, Belgium)

How much effect do you think the modification the Prayon filter has made to the number of tons/day that you can get across the filters?

A - I think that is definitely a factor, because, when we got approval to go from dihydrate to hemihydrate, we know we were going to upgrade our plans from a 316 to 317 sloped bottom. Prayon had indicated to us that basically sloped bottom plan configuration would improve efficiencies by 0.5%. So when we got the approval, we actually bought the pans about six months prior to the start-up of the hemihydrate conversion, and we put them in during one of our Christmas shutdown periods to observe the effect on the dihydrate process, and we did see some improvement in filtration. We like the sloped bottom pans. We also have not observed any build-up whatsoever in the pans. The only times you run into problems on the filtration side is if you allow holes to get into the cloth which results in scaling up of the pans.