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*In 1982, the name of the International Superphosphate Manufacturers' Associations (ISMA) was changed to International Fertilizer Industry Association (IFA).

DISCUSSIONS

1st WORKING SESSION : PHOSPHATE ROCK EXTRACTION

TA/78/1 Beneficiation of South Florida High Carbonate Phosphates by J.E. Lawver, B.L. Murowchick & R.E. Snow, IMCC, U.S.A.(Rapporteur W. Berry, IMCC, USA)

Q - Mr. R. SAINT-GUILHEM , IMPHOS, Paris, France

What is the nature of the heavy media liquid ?

A - The heavy media liquid is a fine ground magnetite.

Q - Mr. R. SAINT-GUILHEM, IMPHOS, Paris, France

Are there areas in Florida where the carbonate is in the grain and separation can only be made by calcination, followed by leaching ?

A - By grinding to minus 28 mesh, a portion of the magnesium can be liberated mechanically. Inter-granular magnesium, however, cannot be liberated via flotation techniques. Calcination and leaching are not felt economic at this time and IMC has no plans to consider this type of treatment. We feel that, once the magnesium level is brought down to approximately 1 %, from a chemical processing standpoint, the rock will be acceptable.

Q - Mr. MOSTAD & Mr. AVENBERG , SUPRA-AB, Sweden

Is there any difference in the content of heavy metals, especially cadmium, between south and central Florida rock ?

A - No definite answer. Current central Florida material is approximately 4-7 parts per million Cd. We feel that it may be slightly lower in South Florida, but quantitative analysis would have to be conducted.

Q - Mr. N. ROBINSON, FISONS LTD., United Kingdom

When are the deposits in central Florida expected to be exhausted ? Bearing in mind the technical problems likely to be presented by the South Florida deposits, is it practical to stockpile rock and blend the South and Central beneficiation products, which would give some control over MgO content and extend the life of the higher quality Central deposits ?

A - There are approximately 20 years left in the Central Florida deposits without further exploration. With some exploration it would be 25-30 years from the Bone Valley. By then, we feel that the industry will shift to the South.

Q - No name given.

In 1978 dollars, what is the ratio of costs (operating costs) between South Florida and Central Florida mining ?

A - We feel that operating costs will be close to double in the South Florida region, due to the amount of additional material which must be handled, per ton of P_2O_5 recovered.

Q - Mr. T.J. PEARCE, SWIFT AGRICULTURAL CHEMICAL COMPANY, USA

What is the difference in specific gravity between the phosphate and the Hawthorne dolomite ?

A - When analyzing specific gravities, one must consider the nature of the heavy media separation. The dolomite has the specific gravity of approximately 2.4. Francolite has a gravity of approximately 2.69. The grind and velocity in the heavy media separation can be adjusted. In the IMC system, the heavy media will have a specific gravity of approximately 2.2, which provides for effective separation.

Q - Mr. A.R. TORABIAN, NPC, IRAN

What is the corrosion rate of acid prepared from South Florida rock, compared with Central Florida material ? Especially, what is the ratio of fluorine to reactive silica ?

A - At this point, we do not have information regarding this factor.

Q - Mr. D. MEYER, IMI, Israel.

What was the scale of the pilot plant operation ?

A - Heavy media work was done at approximately 20 tons per hour. Flotation tests were conducted at a rate of about one ton per hour. It was felt, since the flow sheet is quite simple and that essentially all the operations are derived from well-known technology, that the flotation tests could indeed be conducted at one ton per hour and reliable data obtained.

TA/78/2 Recovery of phosphate from Florida phosphatic clay wastes by C.E. Jordan, G.V. Sullivan, W.E. Lamont & B.E. Davis, Bureau of Mines, USA. (Rapporteur T. Pearce, SWIFT, USA)

Q - Mr. R. SAINT-GUILHEM, IMPHOS, Paris, France

Can the authors explain the spherical flotation agglomeration mentioned in the beginning of the paper ?

A - This is a selective acting process where the conditioned minerals transfer to an oil phase. The amount of oil can be low enough to form little spheres.

Q - Mr. C.H. SCHNARTIAB, Monsanto Enviro Chem. St-Louis, Mo., U.S.A.

How much will total slimes volume be reduced from a beneficiation plant after P_2O_5 recovery ?

A - Since 96 % of the volume to the cones goes out the overflow there will only be a 4 % reduction which goes to the underflow. The P_2O_5 in the underflow amounts to 15 % of the feed slimes.

Q - Mr. R. HARDEVELD, DSM, Netherlands.

Did you collect any data on cyclone erosion ?

A - No. Erosion data obtained to date.

Q - Mr. D.J.E. LAWVER, IMC.

At a typical flow rate of 85,000 gpm slimes - use of 1" cyclones would require about 40,000 cones -

What is the $\frac{I \& A}{P_2O_5}$ ratio of concentrates ?

A - Our calculations, based on 50,000 gpm of slimes at 2 % solids of 35 BPL, would be wasting 1.5 T.BPL/Min., so the potential for the BPL recovery may pay for the cones.

The Al_2O_3 was less than 2 %. The Fe_2O_3 was about 1 %. Therefore the I & A /P_2O_5 ratio would be about 0.11.

Q - Mr. N. ROBINSON, Fisons, United Kingdom.

How will the recovery of slimes affect the processing behavior of the marketed rock ?

A - Conventional acid plants grind the rock to minus 200 mesh. It should be possible to dewater the slimes to 25 % or 30 % moisture by conventional means. If the rock must be dried it will add to the drying cost.

Wet grinding acid plants should be able to handle the moisture in the feed.

Q - Mr. D . J.E. LAWVER , IMC.

A requirement of feed for Wet Process Acid is that the ratio I&A /P_2O_5 = .085. Table 4 shows Ca/P_2O_5 but no I&A data. What are the I&A/P_2O_5 values for the sink fractions ?

A - The I&A /P_2O_5 for the sink fractions are well below 0.085, because aluminium phosphate would not be in the sink fractions. The I&A /P_2O_5 ratio for the 30 % P_2O_5 flotation concentrate was 1.1.

Q - Mr. D . J.E. LAWVER

A typical slime flow for a Florida plant is about 84,000 GPM. Using 25 mm cones you stated a flow per cone of 8.5 l/m = 2.24 GPM corresponding to nearly 40,000 cones which is not a very practical approach. How do you propose to make the separation at plant level ?

A - Fine sizing of slurries at 5 µm is usually done with cyclone clusters that are operated and maintained as a single unit. The number at cyclone clusters would be lower.

Q - Mr. D . J.E. LAWVER.

What is the approximate size distribution and analysis of particles in the cyclone underflow ? Examples : -150 mesh, +44 microns, -44+20 microns -20+5 microns.

A - Since six different slime samples were studied, only a general reply can be made.

	Wt. pct.	P_2O_5
-150 mesh	90-95 %	6-18 %
+37 microns	20-60 %	4-20 %
-37/+5 microns	35-65 %	8-16 %

TA/78/3 Present trends in the design of Florida phosphate beneficiation plants by J.D. Raulerson Jr., Pridgen Engineering Co., U.S.A. (Rapporteur W. Berry, IMCC, USA).

Q - Mr. A.B. PHILLIPS, TVA, U.S.A.

Would you explain briefly the three alternatives for disposing of slime, which you mentioned in your talk.

A - The three techniques are the Brewster Sand-Spray Method, the I M C Dredge-Mix technique and the flocculated system. In the Brewster Sand-Spray method, slimes are deposited in cuts and allowed to consolidate for about one year. Tailing sand is then sprayed on this layer. This layer of tailing sand tends to press the clay layer further. This process is then repeated until the cut is completely full. In the IMC Dredge-Mix technique, slimes are deposited and allowed to stand one year. A dredge is then put into the cut and the slimes are dredged up. Thick slimes are mixed with tailings and redeposited in another cut for further consolidation. Finally, in the flocculated system, a flocculant is added to the slimes to allow for rapid thickening. The thickened slimes are then mixed with tailings and pumped to a pit for further consolidation.

Q - Mr. W.H. COATES , Humphreys & Glasgow, United Kingdom

The recovery of slimes ponds and other processed land must make Florida phosphate increasingly less competitive, compared with other parts of the world. When will the value of the real estate recovered offset the cost of the reclamation ?

A - This is difficult to say, since the real estate market can be affected by a number of factors. At this point in time, reclamation of mined land is almost mandatory. The value of the land will depend on its location and desirability from a development standpoint.

Q - Mr. LAGERHOLM , Supra-AB, Sweden.

Contrast direct matrix acidulation versus handling lower BPL beneficiated rock.

A - There are advantages to direct digestion, in that higher P_2O_5 recovery is possible. The big problem in direct digestion is the iron and aluminium content in the matrix. Also, the problem as to disposal of the sand and gyp after reaction is present. This problem will require study.

Q - Mr. P. MORAILLON , G.E.S.A., France

On page three and four, it is written that the wheel excavator is unique, in that it can dig matrix and feed it directly on a belt conveyor for low cost transport to land reclamation units. Should not this word have been overburden instead of matrix ?

A - Yes, the word matrix should be overburden.

Q - Mr. C.H. DAVIS., National Fertilizer Development Center, U.S.A.

I believe you indicated a decline in Florida production after 1990, but a continuing increase in U.S. production of phosphate rock. From where will the supplemental production originate ?

A - In all probability, the increased production will originate from the North Carolina deposits.

Q - Mr. Frank ACHORN , TVA, U.S.A.

Are slimes mostly attapulgite clay ?

A - Depending on the rock deposit, the slimes can range from high attapulgite content to high montmorillonite concentrations. Each one handles quite differently.

Q - Mr. BONGARD , Windmill Holland, Netherlands.

In which cases must other mining techniques such as those used in North Carolina, be used in the Florida deposits ?

A - If the matrix is to be transported more than four miles, other conveying techniques should be considered, e.g., belt-conveying.

Q - Mr. N. ROBINSON, Fisons, United Kingdom.

Two products are produced during the beneficiation operation, these are pebble and flotation concentrate. Are these products mixed for marketing and,if so, in what proportions ?

A - In the marketed product, the ratio can vary and will depend primarily on the product specs which must be met, e.g., P_2O_5 content, I & A ratio, etc.

TA/78/4 Flotation of carbonate and silicate minerals from partially altered phosphate rock of the Phosphoria formation, by A.R. Rule, D.C. Dahlin & A.J. Fergus (Rapporteur T. Pearce, Swift, USA).

Q - Mr. D. KELLERMAN, Negev Phosphates, Israel.

Have you tested any cations as conditioners ?

A - I assume you mean as modifying agents in the flotation circuit ; Yes, several. Fluosilicic acid is the best. The Bureau of Mines, through a Research Grant to the South Dakota School of Mines, is currently studying H_2SiF_6 absorption on phosphate minerals.

The H_2SiF_6 is chemisorbed on the phosphate mineral rendering it hydrophilic and preventing absorption of the flotation collector. Dr. M.C. Fuerstenau at South Dakota Schoold of Mines will publish this information in the near future.

Q - Mr. Dr. R. VAN HARDEVELD, DSM, Netherlands.

Can you specify in more detail the chemical formula of your flotation agents than you did in your paper ?

1. Fatty acid is ?
2. cationic amine is ?
3. fatty acid/pine oil ratio is ?

A - The fatty acids were from Arizona Chemical (FA2) and Pamac from Armour. They are mixtures of oleic, linoleic and whatever.

The amines were Armac C and T which are cocoa and tallow acetates.

The ratio of fatty acid to pine oil was about 15 to 0.2. (grams/gram).

Q - Mr. C.H. DAVIS, T.V.A., USA.

Have you tested your beneficiation technique with phosphate from South Florida ? If not, could you speculate on the applicability of your technique to the south Florida matrix ?

A - We have run only 1 or 2 tests on S.Florida matrix. In the material tested, about 80 % of the magnesium went out with the slimes. However we were not able to get below 1 % MgO in the concentrates.

Q - Mr. J. MULCKHUYSE, UKF, Netherlands.

Are there figures available about the cadmium content of the beneficiated rock and about the distribution of cadmium over the different fractions in the beneficiation ?

A - No, we do not have any figures.

Q - Mr. W.L. RODRIGUEZ, Quimbrasil, Brazil.

Could you give some more details about the selective removal of magnesium impurities by Aqueous Leaching with SO_2 ?

A - Several years ago a paper was published by the U.S. Bureau of Mines at Albany, Oregon on this approach. The work was cited in the references.

Magnesium is selectively leached by ammonium sulfate, sulfuric acid or the aqueous SO_2. The SO_2 leaching was the most selective. If the pH is lowered to 2.5 by continual addition of SO_2 to an aqueous slurry, the magnesium is selectively leached without dissolving phosphate.

SECOND WORKING SESSION : PROCESSING OF VARIOUS TYPES OF PHOSPHATE ROCK

TA/78/6 Process improvements in manufacture of wet process acid from Florida phosphate rock of various compositions by A.N. Baumann, F.T. Nielsson, J.H. Surber & J.J. Yarnell, International Minerals and Chemical Corporation, U.S.A. (Rapporteurs C.H. Davis, TVA, USA and P. Roberts, C.F. Industries, USA)

Q - Mr. C. VINKE, Windmill Holland, Netherlands.

What are the residence time and temperature of the ageing tank ?

A - About 5 to 6 hours, 180°F.

Q - Mr. J.A. BOLEY, CF CHEMICALS, INC, U.S.A.

How P_2O_5 recovery in the digestion step was measured independently from the recovery in the filtration step ?

A - The digestion loss is determined as C.I. P_2O_5 and the filter loss is W.S. P_2O_5 as analysed in the filter cake. The digestion recovery multiplied by the filtration recovery gives the overall efficiency.

Q - Mr. J.D. CRERAR, Fisons, U.K.

Why not filter the clarifier underflow on the main phosphoric acid plant filter along with the digester slurry ?

A - The clarifier underflow must be diluted to pump and handle the slurry, thus affecting the plant water balance and throughput. Also, filter rates are retarded.

Q - Mr. J. MYRICK, Occidental Chemical Company, U.S.A.

Was a belt filter used ?

A - The filter cloth on the belt filter can be separated and washed on both sides. This is helpful because of the extremely fine crystal size.

Mr. Nielsson added that a belt filter is more versatile in that it can handle a wider variety of operating conditions.

Q - Mr. BRIAN C. TITE, Omnia Fertilizers, South Africa.

Why the 30 % clarifier underflow was not returned to the filter feed tank ?

A - The clarifier underflow must be diluted for pumping and handling and reduces the filter capacity. Thirty percent solids forwarded through the evaporation reduces heat exchanger tube scaling and can be removed in the 40 % clarifier. Crystal growth in the 40 % tank is enhanced with feed crystals.

Q - Mr. H. KARINEN, Kemira Oy, Finland.

What method was used to analyse for sulfate ?

A - Barium was added for precipitation with colorimetric analyses.

Q - Mr. A.R. TORABIAN, NPC, Iran.

Were additives used for crystal growth in the aging tank, and,if so, the percentage and name of the additives ?

A - No additives were used ; various additives had been tested with no significant effect.

Q - Mr. N. KOLMEIJER, Windmill Holland, Netherlands.

Was the 30 % acid strength reported really the actual concentration or was 30 % P_2O_5 simply used for reporting purposes ?

A - The true filter acid strength was 30 % P_2O_5.

Questions not asked from the Floor

Q - Mr. J.H.SULTENFUSS, CF Industries, Inc., U.S.A.,

What improvements in chemical efficiencies do you expect to gain by eliminating the 40 % sludge recycle now going back to the attack tank ?

A - Not an increase in chemical efficiency but an increase in production rate due to elimination of recycle, savings in steam because recycle is not evaporated again, and shortening of defluorination time at the animal-feed ingredients plant because of rejection of fluorine in the filter cake.

Q - Mr. J.P. RIBIERE, Cofaz, France.

What is the exact origin of samples 66-1 and 66-2 ?

A - Hardee County.

TA/78/7 The commercial use of Kola phosphate in Nissan C-process (New Nissan phosphoric acid process) plants by M. Miyamoto & M. Kubo, Nissan Chemical Industries, Ltd.Japan (Rapporteurs C.H. Davis, TVA, USA, and P. Roberts, C.F. Industries, USP)

Q - Mr. J.D. CRERAR, Fisons, U.K.

In the first conclusion it is stated that Kola rock can be used equally or better than Florida or Moroccan rock. On page 5, it is stated that (a) the filter area required is 25 % higher, (b) gypsum moisture is 10 % higher, and (c) P_2O_5 recovery is 0.3 % lower. How do you justify the conclusion ?

A - (a) the additional filter area required is for dihydrate filtration ; (b) because of the crystal habit of Kola-produced gypsum, more water is contained ; and (c) a 97.7 % recovery is less than 98 % by 0.3 % but the recoveries are of the same relative value.

Q - Mr. J.D. CRERAR, Fisons, U. K.

How much diatomaceous earth is added to reduce the fluorine concentration in the second stage ? How many kilograms are required per ton of P_2O_5 ?

A - 0.8 % silica in the acid is adequate whether from the rock or added.

Q - Mr. P. SUPPANEN, Kemira Oy, Finland.

What was the recrystallization stage from hemihydrate to dihydrate and the required retention time ?

A - The recrystallization takes place in the second stage called the hydration stage. The retention time is 15-20 hours, which is twice or more the retention needed for Florida rock.

Q - Mr. H. KARINEN, Kemira Oy, Finland.

Does Nissan use pure or process water for gypsum washing ?

A - Ordinary process water is used.

Q - R.MONALDI, Montedison, Italy. asked about (1) the need for feeding sulfuric acid both in No.1 and No. 2 digesters, (2) the ratio between the two streams, (3) the parameters which control this ratio, and (4) the ratio of product P_2O_5:P_2O_5 recycled to premixer.

A - (1) for good filterability, (2) half to each stream, (3) free H_2SO_4 controls the ratio as measured with potentiometer, and (4) the ratio of product P_2O_5 : recycled P_2O_5 is 1.5-2.0, which is higher than for Florida or Moroccan rock.

Q - Mr. I. MINI, Montedison , Italy.

What is the power consumption in digesters and hydration tanks (kWh/M^3) ?

A - From memory, 0.5. to 1.0 kWh/M^3 is used in the digesters and 0.05 to 0.1 kWh/M^3 in the hydration tanks. This is actual horsepower as opposed to installed.

Q - Mr. F. VIERA NERY, Quimbrasil, Brazil.

Is it possible to feed phosphate rock containing 10 % water to the Nissan process ?

A - This question is not relevant to the use of Kola rock. To obtain high-strength acid, it is desirable to have dry rock. With 10 % H_2O it is barely possible to attain 45 % P_2O_5.

Questions not asked from the Floor

Q - Mr. HANNINEN, Kemira Oy, Finland reported that their experience with Kola apatite in the hemihydrate system showed that the addition of fluoride improved the rate of recrystallization and also that the effect of strontium was counterbalanced by lanthanides.

A - Mr. MIYAMOTO responded that he respected the findings of Kemira Oy. However, he did not believe that these findings would negate the findings of Nissan. He reported that Nissan will further study the effect of fluorine.

Q - Mr. J. ENRIQUEZ, Davy Powergas, U.S.A. asked for a definition of retention time in the digestion stage.

A - It is a minimum of 2 hours.

TA/78/8 Fison's experience on the effect of phosphate rock impurities on phosphoric acid plant performance by N. Robinson, Fisons Ltd, United Kingdom (Rapporteurs C.H. Davis, TVA, USA and P. Roberts, C.F. Industries, USA).

Questions asked from the Floor

Q - Mr. M. GAURON, COFAZ, France, asked for precise details about the nature of mineral compounds acting as favorable modifiers of the crystallization of gypsum outside iron. What modifier has been used with Youssoufia phosphate ?

A - This is confidential.

Q - Mr. GAURON remarked that the complex structure of the composition $Ca_3Al_3(RE)\ SO_4F_{13}.10H_2O$ and the varying chemical composition possibilies should permit action on its state of formation and probably reduce the clogging of this filter cloth. He asked if this problem had been studied and what were the inhibitions (restrictions) of that composition.

A - Fisons is studying this using laboratory equipment. Depending on the level of fluorine, octhedral crystals are obtained with subsequent filter cloth blinding.

Q - Mr. E. BARRIE WINN, International Fertilizer Development Center, U.S.A.

What is the degree of correlation between pilot-scale and industrial-scale data.

A - Pilot testing gives little information on scaling, cloth blinding and foaming. Pilot data are good indicators of P_2O_5 efficiency, filtration rate, acid composition and corrosion. Rock dissolution is more complete on full-scale plant than on pilot scale.

Q - Mr. S. ARNON, Fertilizers and Chemicals, Israel.

What is Fisons' experience relative to organic matter affecting (promoting or inhibiting) stainless steel corrosion in 30 % P_2O_5

phosphoric acid in the dihydrate process ?

A - In the case of defoamers, those are selected which control foam and are compatible from a corrosion standpoint.

Q - Mr. I. MINI, Montedison, Italy.

What is the effect of impurities in wet-process acid on viscosity of ammonium phosphate slurry at various levels of ammoniation ?

A - The question is out of context for this paper.

Q - Mr. ORMBERG, Norsk Hydro, Norway.

What is the chlorine content in the phosphate rock which gave 0.75 % chlorine in the acid ; would it be possible to use such rock in production of phosphoric acid, and if so, what would be the added cost ?

A - Most of the chlorine present in the rock goes to the acid and would cause severe corrosion problems at that chlorine level.

Q - Mr. E.J. LOWE, Albright and Wilson, United Kingdom.

What is the effect of rare earths in Florida and Moroccan rock on processing ?

A - There is very little effect on dihydrate processes. They do have some retarding effect on recrystallization of hemihydrate to dihydrate.

Q - Mr. F.V. NERY, Quimbrasil, Brazil.

What type of cations must be present in sufficient quantity to produce rhombic crystals ?

A - Cationic ions must be compared in conjunction with anionic ions and all impurities. The answer is not simple.

Questions not asked from the floor

Q - Mr. S.L. GLASS, Esso Chemical, Canada said the paper indicated that an adequate SiO_2:F ratio is needed to minimize corrosion and avoid interference with crystal growth. He asked Mr. Robinson to be more specific about the required SiO_2:F ratio. Mr. B.C. Tite, Omnia Fertilizer, South Africa, also asked about means of overcoming the effects of fluorides on dihydrate crystal growth. Mr. R. Monaldi, Montedison, Italy, asked about Fisons'experience with crystal modifiers of the salts coming from impurities.

A - The ideal SiO_2:F ratio to avoid corrosion is that which gives no excess fluoride. For best crystallization, an excess of fluoride is required. The excess needed to produce rhombic crystals depends on the concentrations of cationic impurities such as Fe and Al. Concentration levels are important in addition to ratios. Adjustment to compositions by the use of additives is possible but may not be economical.

Q - Mr. P. STEADHAM, Agrico, U.S.A.

What was the magnification of the gypsum photomicrographs.

A - The magnification was 100 times.

Q - Mr.P.BECKER,Cofaz,France :The corrosion rate for 0.75 % Cl seemed low. He asked (1) How were the corrosion tests carried out and (2) What active SiO_2:F ratio is correct for normal corrosion rates ?

A - For (1) static tests for 1 month and (2) stoechiometric ratio of SiO_2:F.

Q - Mr. MONALDI of Montedison reported that his company's experience shows strong interaction of fluorine + chlorine + sulfides on corrosion. He asked if Fisons observes and quantifies this effect with and without sulfides.

A - Reducing agents such as sulfides can cause very high corrosion rates, worse in combination with chlorides. For a given concentration, chlorides cause higher corrosion rates than fluorides. Combining all causes of corrosion will produce interactions which in some cases will be more than additive.

TA/78/9 Recent performance and innovations in Dorrco process phosphoric acid plants by D.W. Leyshon, Jacobs Engineering Company, U.S.A. (Rapporteurs C.H. Davis, TVA, U.S.A. and P. Roberts, CF Industries, USA).

Questions asked from the floor

Q - Mr. J.D. CRERAR, Fisons, U.K.

What unsatisfactory characteristics of air cooling led to adoption of vacuum cooling ?

A - Vacuum cooling has more flexibility for additional capacity with reduced scrubbing facilities and environmental problems.

Q - Mr. CRERAR also wanted to know the difference between upflow and downflow coolers.

A - Upflow coolers are fed in the bottom of the vessel, resulting in a "fountain" effect at high rates, contributing to carryover. Tangential entry or downflow has been used to overcome this.

Q - Mr. CRERAR

What is the center of the reactor in figure 5 ?

A - Figure 5 is a generalized sketch. The center section of the reactor is used to degas slurry before going to the vacuum cooler.

Q - Mr. J. MYRICK, Chemical Company, U.S.A.

Was the 950 tons per day P_2O_5 obtained on "A" or "B" train, and also what is the design of the flash cooler ?

A - It was on "A" reaction train with both filters used. The flash cooler used downflow and was about 18.5 feet in diameter.

Q - Mr. E.J. LOWE, Albright and Wilson, U.K. asked for information on particle size distribution for rock from wet versus dry grinding.

A - Plus 35 mesh is the same, 0.5 %. Wet rock is coarser in the fine fractions.

Q - Mr. LOWE asked about the minimum water content in wet grinding.

A - This ranges from 60-68 % solids as covered in previous CF Industries'papers. Additives can be added to affect the percent solids. Clay content is the critical variable in percent solids achievable.

Q - Mr. J.P. RIBIERE, COFAZ, France, asked about difficulties in uranium recovery from acid produced with the hemihydrate process.

A - The extraction coefficient for uranium decreases as you go above 30 % P_2O_5 concentration.

Q - Mr. John SURBER, New Wales Chemicals, U.S.A., referred to potential savings of $10-$20 per ton P_2O_5 for uranium recovery and $ 15 per ton P_2O_5 for the hemihydrate process. Is there technology available for recovery of uranium from strong (approximately 40 % P_2O_5) phosphoric acid ?

A - This was covered in the oral presentation. Uranium recovery would not be done in conjunction with strong acid produced by hemihydrate processes, unless some new process breakthrough were to be made.

TA/78/10 Bench-scale studies of utilization of problem rocks in wet-process phosphoric acid production, by A. Varsanyi, E.B. Winn & P.H. Peng, International Fertilizer Development Center, Muscle Shoals, Alabama, U.S.A. (Rapporteurs C.H. Davies, TVA, U.S.A. and P. Roberts, C.F. Industries, USA).

Questions asked from the floor

Q - Mr. N. KOLMEIJER, Windmill Holland, Netherlands, asked if, in filtration studies, separate batch filters were applied for each wash, or was the washing carried out in sequence on the same filter ? If the latter is the case, how do you study the possible scaling to be expected in a particular wash ?

A - They carry out the washing in succession and are not able to study scaling.

Q - Mr. F. NERY, Quimbrasil, Brazil, asked about the materials of construction of the agitator and reactor. He asked further if stainless steel agitator blades which serve as corrosion specimens will affect the material balance and consequently gypsum crystallization.

A - The agitator shaft was Kynar with polypropylene bolts. The tank was fabricated from polypropylene. Mr. Winn did not believe corrosion would significantly affect the material balance and gypsum crystallization.

Q - Mr. M.E. BANFIELD, N-ReN International, Belgium, remarked that it would be useful to have results of filtration rates, etc., from a reference rock in order to provide some form of calibration of the results presented.

A - This is important. A 72 BPL central Florida concentrate was used for this purpose. The results obtained with this concentrate agree with typical industrial results for similar concentrates from central Florida.

Q - Mr. W.L. RODRIGUEZ, Quimbrasil, Brazil.

Why Araxa rock was considered to be a problem rock ?

A - Araxa rock is known to cause corrosive conditions in wet-process phosphoric acid production and is also thought to be relatively unreactive. In addition, some workers have suspected that the high barium content would increase sulfuric acid consumption. However, the tests showed that Araxa rock performed satisfactorily.

Q - Mr. R.H. EDWARDS, Albright and Wilson, U.K. asked if there were any measurements of power consumption by the agitators and what measurements are made to recommend agitation in a full-scale plant.

A - There were no measurements of power consumption by the agitators ; they have observed the Reynolds Number only. Measurements still have to be made for scale-up to full plant.

Q - Mr. N. ROBINSON, Fisons, United Kingdom, related that intermittent operation of pilot plants can give misleading results due to distortion of crystal size and distribution.

A - Mr. WINN agreed with the need for continuous around-the-clock testing.

3rd Working Session :

TA/78/11 A route saving raw materials and energy makes P_2O_5 available through wet phosphoric acid process by A. Davister, Société de Prayon, Belgium; S.T. Houghtaling, Davy Powergas, U.S.A. (Rapporteur Mr. P.Moraillon, Générale des Engrais, France)

Q - Messrs. T. PEARCE (Swift, U.S.A.), M. BARLOY (Lebanon Chemicals, Lebanon), P. MORAILLON (Générale des Engrais, France).

A - Wet grinding of phosphate rock

Different systems were tested to determine the solids content of the phosphate slurry to the reactor : nuclear densimeters, differential pressure cells, etc... The most reliable method is the one used in the mining industry : weighing of a constant level tank with constraint gauges.

The density is not always a sufficient indication : the presence of clay affects both the density and the viscosity.

The amount of water needed depends on the characteristics of the phosphate, in particular its hardness. The harder the rock, the more phosphate is needed. More water is needed when increasing amounts of fines are produced. The solids content can then vary between 75 and 60 %.

Q - Mr. M. BARLOY (Lebanon Chemicals, Lebanon)

A - Fineness of phosphate rock needed. Possibility of avoiding grinding.

In the case of fairly soft phosphates, it is possible either to avoid grinding or to grind only the oversize beyond 0.5 mm, which result in important energy savings. From that point of view, Société de Prayon considers it is advantageous to use a mixture of two or three rocks of different origins and particle size. In the case of harder phosphates, a smaller particle size is needed. There is little difference in the fineness required between dihydrate or hemihydrate processes.

Q - Mr. P. BECKER (COFAZ, France).

A - In the phosphate feeding zone, the agitation power needed varies between 1 and 1.1 HP/m^3. Actually the agitation power per unit of volume is not a significant factor. What matters is to obtain the maximum effect with the minimum total energy.

The low grade phosphate mentioned in para III B contains 50-55 % BPL, 15 % silica, 15 % $CaCO_3$ and magnesium carbonate.

Q - Messrs. F. ACHORN (TVA, U.S.A.) and S.L. PENG (CRA, Brazil).

A - My conclusion is no criticism of the US farmers or agronomists. I quoted an American author who feels the buyers should be ready to make certain changes. The conventional formulations of some fertilisers would have to be abandoned to allow for increasing soluble impurities. We must defend the economics of lower grades and solubilities, instead of making luxury fertilisers.

TA/78/12 Experiences in phosphoric acid production with hemihydrate and dihydrate process by E. UUSITALO, M. LOIKKANEN, T. KARJALAINEN & E. HANNINEN, Kemira Oy, Finland (Rapporteur J.D. CRERAR, Fisons Ltd, U.K.).

Q - Mr. M. GAURON (COFAZ, France) asked about the P_2O_5 concentration in the recrystallisation solution.

A - The concentration was approximately 10% P2O5.

Q - Mr. M. GAURON referred to the fact that gypsum recrystallisation occurred mainly in the dump and asked whether the return of P_2O_5 to the process had been considered and in what proportion.

A - A major part of the P_2O_5 released in the gypsum dump was recycled back to the process via the water recirculation system.

Q - Mr. M. GAURON asked the author's opinion of a single filter being used to filter two slurries with different characteristics and behaviours.

A - The double-dump filter performed hemihydrate filtration on the first part and dihydrate on the second part. The use of the filter was a little complicated, but had the advantage that hemihydrate crystals did not block the filter cloth because the dihydrate slurry was fed after it.

Q - Mr. D.G. HUIJGEN (UKF, Netherlands) referred to the different quality of MAP from the hemi-di-hydrate process compared with dihydrate and noted that the moisture content was different. He asked if there would still be a difference in quality if the MAPs were dried to the same level. He also asked if there was any difference in NPK quality using the different grades of MAP.

A - Mr. HÄNNINEN felt the water content was not significant in the system, but had found granulation properties of the dihydrate MAP superior to the hemi-di-hydrate MAP.

Q - Mr. P. MORAILLON (Générale des Engrais, France) asked what process had been used for making MAP from the two types of acid.

A - Kemira's own process for neutralising phosphoric acid with ammonia which had been installed for five to six years had been used.

Q - Mr. P. MORAILLON asked what kind of packing or filtering medium was installed inside the entrainment separators following the evaporator and fluorine scrubber shown on the flowsheet.

A - Corrugated plates.

Q - Mr. A.R. TORABIAN (NPC, Iran) asked about the proportion of fluorine evolved in the hemi-di-hydrate process compared with the conventional process.

A - A larger proportion of fluorine is evolved in the hemi-di-hydrate process compared with the dihydrate reaction stage, but Mr. HANNINEN could not give a comparison after concentration in the dihydrate process. However, difficulties in fluorine recovery were greater in the hemi-di-hydrate process because residual SiF_4 hydrolyzed to SiO_2, which tended to block pipes.

Q - Mr. J. MULCKHUYSE (UKF, Netherlands) asked for the composition of the liquid in the hemihydrate recrystallisation stage.

A - H_2SO_4 : 5 % approximately, P_2O_5 : 10 % approximately.

Q - Mr. H.K.E. ITEKE (Fed. Super.Fert., Nigeria) asked for a comment on the technical and economic feasibility of sedimentation-type dewatering centrifuges compared with conventional vacuum filtration systems.

A - Mr. HÄNNINEN had no experience of using a centrifuge for dihydrate or hemihydrate slurries and could not comment.

Q - Mr. C. WEIL (Coppee-Rust, Belgium) asked for information on the modifications required on mixers to avoid damage caused by hardened gypsum particles.

A - Initially there had been serious damage and the mixers were altered, but it was difficult to explain the details. It was necessary to operate the process carefully to avoid this damage.

Q - Mr. M. BARLOY (Lebanon Chemicals, Lebanon) asked the author if he had to build a new plant, which process would he select and why.

A - From the experience with the plant discussed a dihydrate process would be selected.

After the discussion Mr. N. ROBINSON (Fisons Ltd., U.K.) submitted a comment that Fisons had found the recrystallisation rate of hemihydrate produced from Kola rock to be too slow, whereas,to obtain the full benefits from a hemi-di-hydrate process, complete and rapid transformation to dihydrate must be achieved. The conclusions of the authors are specific to Kola rock and the conclusions should not be applied to comparisons when using other rocks. He agreed with the statement in the paper that it is important when selecting the process to know the behaviour of the rock in question.

TA/78/13 Fluorine absorption and indirect condensation in wet process phosphoric acid concentration by M. Lahav & S. Arnon, Fertilizers & Chemicals, Israel. (Rapporteur Mr P. Moraillon, Générale des Engrais, France)

Q - Mr. I. MANGAT (Humphreys & Glasgow, U.K.)

In your flow diagram you show an atmospheric pressure seal tank for the vacuum flash chamber. Does this not add unnecessarily to the head on the circulation pump? What is the reason for this?

A - It is true that the existence of an atmospheric pressure seal tank in phosphoric acid recycle loop, as shown in the flow diagram, increases slightly the head of the pump, but the pump is still capable of doing the job.

Q - Mr. J. FROCHEN (COFAZ, France)

What are the materials used for heat exchanger, pumps, condensers etc...?

A - Rubber lined steel for the external walls of the fluorine absorber-scrubber and of the final condensers.

- Graphite for the shell and tube exchanger and for the condenser tubes, as well as for the fluosilicic acid recirculation pump?

Q - Mr. M. BARLOY (Lebanon Chemicals, Lebanon).

1/ What is the vacuum in the flash chamber for a dew point of atmospheric air of 25°C?.

2/ What is the quantity of recirculated water on the surface condenser compared to the amount of water through a contact condenser?

A - For an outside air dew point of 25°C, the temperature of the condensate obtained in a surface condenser is 45°C, which corresponds to an absolute pressure of 100 mm Hg inside the flash chamber.

The flowrate of cooling water through the surface condenser is 90 m^3/t P_2O_5, instead of 50 m^3 in the case of a contact barometric condenser. The atmospheric cooler is very close to the plant.

Q - Mr. T. AZADEGAN (N P C, Iran)

1/ What do you produce H_2SiF_6 for ? and where do you use it?

2/ What is the pressure in concentrators?

3/ What is P_2O_5 content in your H_2SiF_6 produced and what kind of demister pad you use in the concentrator?

A - The fluosilicic acid produced is used to manufacture Na_2SiF_6 and NaF. The P_2O_5 content of fluosilicic acid is only 0.1 %. Between the concentration and the absorber scrubber, there is a demister, the type of which cannot be revealed.

Q - Mr. P.R. ROBERTS (C.F. Chemicals, U.S.A.)

How long will the Condenser operate without cleaning and how do you clean it ?

What is the percent of time the plant operates?

/

A - If the phosphoric acid concentration in the evaporator exceeds 48 % P_2O_5, there is no tube clogging in the condenser. However up to 2 % silica is added to Israel phosphate.

After two months'operation without cleaning an incident nevertheless occurred: after a washing (boil out) of the concentration unit, the P_2O_5 content of the circulating acid was too low at start up, which resulted in a clogging of the condenser tubes. Cleaning was very easy with high pressure water. Since then the condenser is washed with water at each stop.

Q - Mr. T. PEARCE (Swift, U.S.A.)

What is the concentration of fluorine in the condensed water returning to the phosphoric acid plant ?

A - As indicated in the text, the fluorine content of the condensed water returned to the phosphoric acid plant is only 0.2 - 0.3 %.

Q - Mr. E. WINN (I F D C, U.S.A.)

Where is the process condensate recycled to in the phosphoric acid plant ?

A - The condensed water is used for the first washing of the phosphoric acid filter.

TA/78/14 Sodium silicate and calcium fluoride from sodium fluosilicate by A.F. SABRY, Sté Industrielle et Financière d'Egypte, Egypt (Rapporteur J.D. CRERAR, Fisons Ltd, UK).

Q - Mr. H.K.E. ITEKE (Fed. Super.Fert., Nigeria) had in mind a situation in which the objective was to obtain Na_2O and SiO_2 only, and enquired whether the Na_2SiF_6 - $CaCO_3$ system had been studied.

A - That system had not been studied since $CaCO_3$ is not an alkali. The objective was to obtain two products three times the value of the raw materials.

Q - Mr. N. ROBINSON (Fisons Ltd, UK) asked whether any work had been carried out reacting lime or limestone directly with H_2SiF_6.

A - Some work had been carried out to produce calcium fluosilicate, but it was impossible to get the calcium fluosilicate to precipitate - it formed a gel.

Q - Mr. SART (Prayon, Belgium) asks for the level of SiO_2 in the calcium fluoride and the level of fluorine in the sodium silicate filtrate.

A - Mr. SABRY referred to the tables in the paper which showed the calcium fluoride purity between 77.9 and 86.1 %. The 20 % (approximately) of other material is mainly hydrated silica - H_2SiO_3. The sodium silicate is free of fluorine unless insufficient $Ca(OH)_2$ has been used.

Q - Mr. E.K. DRECHSEL (Duval/Pennzoil Co., USA) asked for the concentration and disposition of the by-product HCl from the reaction of H_2SiF_6 with NaCl, and whether there is an alternative to dilute HCl.

A - Starting with a concentration of 15 % H_2SiF_6 and 30 % NaCl the HCl solution would have a concentration of 7-8 %. Mr. SABRY referred to his 1961 ISMA paper which dealt with the reaction of by- product sodium sulphate instead of sodium chloride with fluosilicic acid leading to the production of dilute sulphuric acid which was introduced to the acid process. Thus an alternative for dilute HCl existed.

Q - Mr. A.R. RULE (Bureau of Mines, USA) asked about the end use of calcium fluoride.

A - It was used in the metallurgical industry for aluminium, iron and steel production and also in the ceramic industry.

Q - Mr. R. SOMERVILLE (Landskrona International, USA) asked whether Mr. SABRY had considered counter-current washing of calcium fluoride with caustic soda to remove silica and make the product suitable for HF production.

A - Mr. SABRY pointed out that the study presented was only preliminary and there was the intention to make improvements. The 85-86 % purity of calcium fluoride was not sufficient and there is a need to get rid of the impurities particularly silica. However, caustic soda would not be effective since any free silica present would already have had the opportunity to react with the correct quantity of caustic soda, and additional quantities would have no effect.

TA/78/15 A zero discharge and water recycle system for wet process phosphoric acid plants by D.G. MERCER, Texas Gulf, U.S.A. (Rapporteur Mr. P. MORAILLON, Générale des Engrais, France).

Q - Mr. M. COPE (Borden Chemical, USA)

Have you noticed any nitrogen accumulation in your fertilizer plant reservoir ? If so,are you anticipating an air pollution problem from this pond ?

A - The DAP plant has no liquid effluent in the 60 acre reservoir. As a result, there is no nitrogen in the water.

Q - Mr. M. COPE

Why do you not include rainfall and evaporation on your 190 acres of reservoirs and why is the water added in the rock omitted from your water balance in figure 5 (p.206) ?

A - The water balance in figure 5 relates both to 120 acre cooling pond and to the gypsum disposal area, and should include rainfall on the total area. On the other hand, rainfall on the 60 acre reservoir is not taken into account.

Q - Mr. J.P. RIBIERE (Cofaz, France)

What is the present surface of the gypsum disposal area ?

A - The cooling pond must have a sufficient capacity to contain all the waters running off from the associate areas.
We should state that we were able to sell some gypsum.

Q - What is the distance between this gypsum area and the 120 acre water pond ?

A - The distance between the gypsum area and 120 acre pond was originally a few hundred feet. The new gypsum area is 1/4 mile from the pond. The water leached from the area flows by gravity to that cooling pond.

Q - Would it be possible, in view of your experience, to have only one pond for gypsum disposal and for water ?

A - We have no experience with a single pond for gypsum disposal and water cooling. It is probably feasible.

Q - Mr. J.H. SCRUGGS,(Davy Powergas, USA)

Similar questions were put by Messrs. ARNON (Fertilizers and Chemicals, Israel), S.L. PENG, (CRA, Brazil), A. SELLAMI (ICM, Tunisia).

How thick was clay liner ? To what depth did pond water affect clay liner due to chemical reactions ?

A - The bottom of the cooling reservoir is lined with compacted clay. It is not completely impervious. I do not know the depth of this clay liner which was installed before my arrival on the site. The state of the bottom will be controlled in due course. No contamination of the aquifer, which is very deep, was noticed.

The walls of the pond are also made of a local natural clay.
The pumps are made of rubber lined ordinary steel.
The experience of cleaning the ponds is limited to the 120

acre pond. It is presently cleaned for the second time only after 12 years'service. Fresh water is used for cleaning.

Q - Mr. P. BECKER (Cofaz, France)

Did you measure the fluorine concentration in the air over the ponds ?

A - The fluorine concentration over the ponds is not measured. According to EPA, it should be very low.

Q - Mr. M. BARLOY (Lebanon Chemicals, Lebanon)

What is the equilibrium fluorine content in the recirculation water ?

A - In the old system, the fluorine content was 1.2 % in the water of the 120 acre reservoir, and the P_2O_5 content 2.2 - 2.4 %. In the new system the content decreased to 0.7 % and 1.6 - 1.7 % P_2O_5 respectively. In the 60 acre reservoir we find 0.3 - 0.4 % F and 0.6 - 0.7 % P_2O_5.

Q - What is the amount of fluorine discharged in the atmosphere authorized by legislation ?
Are official controls made only at the stacks or also over the ponds ?

A - The authorized fluorine discharge to the atmosphere is 0.04 lb per short ton P_2O_5. Actually we operate much lower than that: 0.01 lb/t P_2O_5 obtained, e.g. less than 10 lb per day.

The official services only control stack emission, but not the air over the ponds.

Q - Mr. D. DIBBLE (IMC (USA)

Similar questions put by Mr M. BARLOY and Mr. I.G. HIRST (Davy Powergas, UK)

Have you noticed any difference in filtration since you changed from fresh to pond dilution water ?

A - Since fluorine containing pond waters are recirculated, no measurable differences were found in the filtration rates or in the blinding of filter cloth as compared to the use of fresh water.

Q - Mr. A. BAUMANN (IMC, USA)

What is the advantage of adding pond water through reactors instead of on filters for washing ?
Is it not better to dilute acid with 70 % recycle and add the pond water across the filter ?

A - In our Prayon plant, we need to dilute sulphuric acid before adding it to the reactors. The pond water used for that purpose can also be used on the filter. We have no experience of sulphuric dilution with 22 % P_2O_5 recycled phosphoric acid.

Q - Mr. H. LEOTTE (SAPEC, Portugal)

Have you made an energy balance before and after the installation of that system ?

A - No energy balance comparing the old and the new systems was made.

TA/78/16 Energy conservation in a fertiliser complex by W.S. Hornbeck, Agrico Chemical Company, U.S.A.
(Rapporteur : J.D. Crerar, Fisons Ltd., U.K.)

Q - Mr. P.J. BROWNE (Consolidated Fertilizer Ltd, Australia) referred to Figure 3 which shows the turbo-alternator coupled into the main incoming feeder on the site. He asked whether it would not have been better to connect the turbo-alternator into one of the acid plant 69 kV feeders so that the acid plant would continue to operate if the external power supply was lost.

A - Mr. HORNBECK pointed out that the main supply to the site was 69kV whilst the acid plant supply was 4,160 V. The problem had been that the site-generated power was greater than that required for two sub station breakers, and less than that for three. The existing circuit breakers were unsuitable for the frequent operation that this would cause, and the cost of revision was excessive.

Q - Mr. BROWNE also asked if the site power factor had improved and whether there was auto power factor correction on the turbo-alternator.

A - The original plant power factor had been 0.86 and the turbo-alternator has run at 0.85-0.87 power factor. It is intended to improve the power factor of the site which will permit operating the turbo-alternator above 0.87. No automatic power factor correction device is fitted but it is intended to install one later on.

Q - Mr. W.B. van den BERG (UKF, Netherlands) asked about wet rock grinding. Could wet ground phosphate rock be stored for a period in suspension or does it sediment and harden rapidly ?

A - A small rock slurry tank is fitted with a 25 HP agitator, but Mr. HORNBECK thought it could be shut down for a short period since settling is not rapid. Generally some agitation should be provided.

Q - Mr. W.B. van den BERG (UKF, Netherlands) asked whether wet ground phosphate rock could be shipped overseas and how vessels would be unloaded.

A - The details of this were outside the field of Mr. HORNBECK, but he pointed out that the economics would be affected by the amount of water being transported. However, it was technically feasible and there would be no corrosion problem.

Q - Mr. J.H. SURBER (IMCC New Wales, USA) enquired about the negotiations with the Electric Power Company.

A - The same basic power cost continued to apply, but the maximum demand charge changed from 50 % to 95 % of the maximum of the previous twelve months. Mr. HORNBECK pointed out that Agrico generated 45-50 % of its own power.

Q - Mr. J.J. YARNELL (IMCC New Wales, USA) requested information on the problems of interfacing the turbo-alternator with the Electric Power Company.

A - Mr. HORNBECK did not think there had been any real problems but the detailed conditions had to be agreed with the Power Company. Drawings had to be submitted and reverse current, and over and under frequency and over and under voltage relays had to be installed. A detailed procedure for phasing in with the mains has to be followed. The whole facility is well protected, but although the procedures have to be carried out rigorously there are no real problems.

Q - Mr. J.J. YARNELL (IMCC New Wales, USA) asked about recovery of condensate for boiler feed water make-up.

A - All turbines condensate is returned already and Agrico is working on a scheme to return condensate from the evaporators for boiler feed water. The latter scheme is more complicated and is taking more time.

Q - Mr. A. R. TORABIAN (NPC, Iran) asked for information about erosion rate in slurry pumps and the interior of the ball mill, and asked for a comparison between wet and dry grinding from this point of view.

A - Slurry pumps constructed of Ni-hard have given no problems neither has carbon steel piping. At South Pierce Ni-hard liners in the first ball mill failed and have been replaced by rubber liners. The rubber liners now last 1 1/2 years. On the other hand, at the Faustina plant Ni-hard liners have given satisfactory service over the last 3 1/2 years. The reason for this is not known.

4th Working Session : GRANULATION TECHNIQUES AND FERTILIZER PRODUCTION.

TA/78/17 Technical and economical evaluation of a raw phosphate rock for the production of fertilizers by R. Monaldi & C. Minghetti, Montedison SpA, Italy (Rapporteur Mr. N. Kolmeijer, Windmill Holland, Netherlands).

Q - Mr. SAINT-GUILHEM, Imphos, France.

The test-system described is remarkably complete. Has the evaluation been verified for certain cases, that is to say : has it been possible for certain "new rocks" to compare the lab.test figures with results in commercial plants, both technically and economically?

A - We have evaluated several dozens of phosphates and have been able to verify the figures with industrial scale results on at least 10 or 12 rocks. With phosphoric acid manufacture the difference in efficiency was 0.2 % max. The capacity figures have always been confirmed. The above is also valid for the preparation of superphosphates and nitric attack of rock.

Q - In what way have the tonnages of "new rock", which are indicated at the end of the lecture, been determined? Their total is the same as the reference quantity (360,000 tons).

A - The tonnage for the production of phosacid can be calculated according to following formula :

$$135{,}000 \times \frac{0.331\ (0.965 - 0.02)}{0.288\ (0.94 - 0.02)} = 159{,}000 \text{ t/year.}$$

The figures used are taken from the tables and the figure 0.02 shows the mechanical loss. For superphosphate and nitric attack of rock it is always necessary, after determination of the required quantity of rock, to adjust to the same contents of plant food. (18.2 % P205 for SSP, 12.2 + 12.2 + 12.2 for complex fertilizer) and to the same annual production capacity.

Q - Mr. A. BAUMANN, IMC, U.S.A.

Could the authors comment on what appears to be a contradiction in Table 2's empirical formula with the conclusion that the test rock has a greater reactivity than the reference rock. Chemical reactivity is related directly to the amount of substitution of Carbonate for Phosphate in the apatite structure. In order to balance the change for each mol of CO_3 substituted, a mol of F usually is also found in the structure. These substitutions lead to a lower P205 content, not higher as shown by the empirical formula. The higher insoluble P205 content in the gypsum cake also does not indicate the test rock is more reactive than the reference.

A - The empirical formula has been calculated according to the reference cited (Lehr, TVA). The presence of Dolomite must also be considered.

Q - What crystal improvement did you attain with the addition of Montaline SPCV?

A - When we used Montaline we were able to attain the same filtration rates as with the reference rock gypsum; the crystals have been shown on the photographs.

Q - Mr. BARLOY, Lebanon Chemicals, Lebanon.

I believe that if you would, in the case of phosacid, make corrections for corrosion rate and plant capacity, the price correction would be different.

A - You are right, but if we test a rock giving a corrosion rate which is greater than the limit acceptable for the Montedison-plants, we will not buy it. In case that plant capacity is not the same and we would have to buy acid, the extra cost of this is also taken into account.

Q - You consider the same rock for several uses. Very often the best quality for phosacid is not suitable for SSP and/or TSP-manufacture. Do you suggest to use two different qualities to obtain minimum final product cost?

A - Yes I do. When two rocks can be stored separately, it can be profitable.

Q - Mr. A. BAUMANN, IMC, U.S.A.

Could you comment on why the apparently more reactive test-rock gave inferior yield in the phosacid test results than the reference rock?

A - In our plants we have the experience that the more reactive a rock the higher the insoluble P2O5-loss. We explain this phenomenon by assuming that the rock particles are more easily covered with a layer of gypsum. A high crystallization rate normally leads to unfavourable morphology of the crystals.

Q - Mr. P. BECKER, Cofaz, France.

In table no. 6 you indicate a filtration rate of 14 t P2O5/m^2/day for the reference rock and 13.8 t/m^2/day for the tested rock. I should be glad to learn how one can reach such good figures, as commercial plants usually work in the range of 3-9 t/m^2/day.

A - Usually we attain 130t/day P2O5 and more on a 12m^2 vacuum filter. The figure of 13.8 t/m^2/day refers to lab.tests and does not take into account the necessary correction (conservative figure 80%) for non-constant efficiency or differences in porosity of filter cloth. That means 12.1 t/m^2/day. This is the value we attain during operation time (about 22 hours in one day).

Q - Mr. N. ROBINSON, Fisons, U.K.

In your phosacid production test what is the total residence time based on the 3 reaction vessels ?

A - About 3 to 3.5 hours.

Q - How long do you operate before the results are assessed?

A - Usually to have a reliable assessment a ten days max test is needed (see also questions by Messrs Edwards and Somerville).

Q - From the P2O5-losses and efficiency figures given in table 7 it appears that the P2O5 to CaO ratios in the rocks tested are very low. Can you reveal which rocks were tested?

A - Djebel Onk, Algeria.

Q - How was the cocrystallized P2O5 determined?

A - By dissolving the gypsum in ammonium citrate solution.

Q - Mr. E.B. WINN, IFDC, U.S.A.

How do you evaluate the reduction in grinding maintenance cost as given in table 12?

A - The reduction in cost results from the difference in grindability which in this case was very favourable.

Q - Why did you not include a variation in maintenance costs of the phosacid plant itself?

A - There is no difference in capacity and maintenance in the phosacid plant.

Q - Mr. P. BECKER, Cofaz, France

What is the upper limit of chloride which you accept in a rock used for phosacid production?

A - We use 316 L materials in our plant and consequently our limit of acceptance is 0.020-0.025% Cl.

Q - Mr. A. HöRKKö, Kemira Oy, Finland

Do you have any real evidence from tests that self-sustaining decomposition increased at higher level of organic matter in the rock?

A - We have a lot of experience in this field and can confirm that this is the case.

Q - Mr. R.H. EDWARDS, Albright & Wilson, U.K.

Referring to phosacid production test with H_2SO_4, for how many hours should the test run for reliable results and how much rock is used in this test?

A - The test usually takes 160 hours and the feedrate is 1 kg rock/hour.

Q - Mr. R. VAN HARDEVELD, D.S.M., Netherlands

On page 4 of your paper you state that excess F is causing difficulties in the purification of phosacid.

A - We wrote about an excess of fluorine against the SiO_2 content. In the acid the fluorine must be present completely as H_2SiF_6. Silica is added if necessary.

Q - Mr. R.L. SOMERVILLE, Somerville & Associates, U.S.A.

Do you think that operation during the full 24 hours is necessary? We operate a similar plant for rock evaluation. We consider that operator-technique is so critical that we prefer to operate only in day-service for 8 to 12 hours to avoid changing operators. We have not found it necessary to make long continuous runs.

A - We operate 16 hours per day during 10 days max. The operator cannot influence the process in the bench scale plant. He only attends its operation. Analysis is automatic. The length of the run depends on how much time is required to establish the process parameters and it obviously is a function of the rock.

TA/78/18 Pipe reactor : an innovation for improvement of granulation plant performances by I. Mangat, Humphreys & Glasgow Ltd, United Kingdom and J.M. Toral, Sociedad Anonima Cros, Spain (Rapporteur O.H. Lie, Norsk Hydro, Norway)

Speaker : Mr. I.S. Mangat, Humphreys & Glasgow

Q - Mr. W.P. RONAN,(Albatros Fertilisers Ltd, Ireland)

Is there a difference in the capacity required per cross section for co-neutralised acids compared with the direct Amm. Phosphate Slurry?

A - Yes. Cross section depends on the rate of heat release.

Q - Does the use of the pipe reactor have any advantage over the conventional slurry system in terms of granulation characteristics?

A - Yes. Granulation efficiency is better enabling low recycle ratio. Physical characteristics of the product, in particular the hardness, are superior.

Q - Apart from the total water balance requirements is there any special reason why

a/ You use sulphuric acid to scrub the NPK gases and use either sulphuric or phosphoric acid to scrub the NP gases?

b/ You feed the scrubber solution to the granulator for NPK products and to the reactor for NP products?

A - One of the major reasons is the amount of NH_3 to be recovered in the scrubber. When this is small, there is no reason why it cannot be scrubbed with the H_2SO_4 available. If the amount of ammonia to the scrubber is relatively high, then a mixture of acid may be required.

The main reason is that we wish to keep chloride out of the reactor.

Q - Mr. I. PODILCHUK, Ind. Luchsinger, Brazil

Referring to table 1, page 242, and assuming MAP and DAP were made on the same plant, what limited the MAP production to 20 TPH?

A - Shortage of phosphoric acid.

Q - What is the molar ratio in scrubbing liquid sent back to the pipe-reactor when DAP is made?

A - Between 0.2 to 0.3.

Q - What are the limitations of the pipe-reactor? Production rate, operation time, phosphoric acid or anything else?

A - There is no limitation on the production rate. Operating time should be better than conventional systems. We are not aware of any limitations arising from the source of the acid. With regard to concentration of the acid it is a matter of the heat balance.

Q - Is the gypsum, used for MAP, wet or dry? How is the gypsum transported to the granulator?

A - The gypsum used is substantially dry, in the sense it must not cake. In our case the moisture is about 5%. The gypsum is transported by a lorry from an associated company about 40 km away.

Q - Mr. G.R.G. COULTHARD,(Haldor Topsoe, Denmark).

Do Cros operate a granulation plant specifically designed to their Pipe-Reactor process or is the present experience limited to modified plant which still has the security of a large drier to fall back on? Please try to define the risks a new fertiliser producer would be taking in adopting this process without the large drier security on a plant in a developing country in a remote location of the world.

A - S.A. Cros plants were built to operate conventional processes where drying was required. We do not see the need to remove the drier system. In fact for the manufacture of compound fertiliser we have indicated that hot air drying is necessary. As most of the plants built, particularly in Europe, are multi product plants, it is unlikely that the drier system will be omitted. This would be risky. We believe that for a straight MAP or DAP plant the drier is not necessary.

Q - Mr. J.E. LEONARD (Grassland Fertilizers Ltd., Ireland)

How is the production of polyphosphate avoided in the process? (Page 240 of preprint).

A - By control of the concentration of feeds.

Q - How do maintenance costs of this system compare with those of a conventional preneutraliser process. For example-how often would the unit require replacement and are increased costs of maintenance likely to offset some of the savings mentioned?

A - The maintenance costs for the Cros pipe reactor system are negligible compared to the tank, agitator, pumps and piping system in a conventional plant.

Q - Mr. A. VERDASCA, (Quimigal, Portugal).

Is it possible to know what kind of material, in terms of corrosion resistance, you are utilising on the granulator gas scrubbing system, when you work with phosphoric and sulphuric acids simultaneously?

A - Polypropylene and reinforced fibre glass.

Q - Mr. W.C. LAVERS (British Sulphur Corporation, U.K.).

I understood from operators at TVA Demonstration that they had found sulphuric acid beneficial in controlling scale-up within the pipe. Could the authors comment on their own experience with scaling and the means by which it is controlled?

A - In our experience there is a complete absence of scaling in the reactor. We do not, therefore, have to introduce any scale controlling procedures.

Q - In the literature of pipe-reactor slurries, there are many references to the benefits of moderate levels of polyphosphate in the slurry during subsequent granulation. Could the authors comment further upon the remark in the paper that in their experience the presence of polyphosphate does not offer advantage in granulation?

A - Not very much more can be said about the subject, other than what has been said in the paper. There have to be reasons for our different experience in this matter. We can suggest one or two, but this would be no more than a mere suggestion.

Q - Mr. I.A. BROWNLIE (Scottish Agric. Ind.,U.K.)

With reference to co-neutralisation mentioned on page 253 could the speaker please give details of: the moisture content of the ammonium nitrate/phosphate liquor leaving the Cros type reactor, and typical NPK formulations which can be produced e.g. the maximum N content of the NPK formulation possible by this method?

A - Pilot plant scale tests only have been conducted so far. Within the limits of our experience we are unable to present any meaningful information on moisture content at this stage. Further development work is in progress and we may be able to provide the information at a future date. We produce 20-10-10 and 15-15-15. We have not had the need to explore above these N contents. We do not, however, see any reason why there would be any grade limitations compared with a conventional ammonium phosphate slurry system.

Q - Mr. I. MINI (Montedison, Italy)

The pipe reactor, differently from the reactor described in the paper 11 presented at ISMA Conference in the Hague, presents low ammoniation degree in DAP production and therefore requires a high quantity of sulphuric acid which costs more than 2 $ per ton of DAP. We ask : can Cros make a revamping also in DAP plants where the ammoniation of solids is a bottleneck?

A - We cannot discuss the Hague paper referred to as we do not have ready access to it now. However, the answer to your question is "yes , the design and size of the granulator can be reviewed for debottlenecking .

Q - What is the time between two cleaning operations if the pipe reactor must ammoniate acid rich in impurities and chiefly Mg (we remember the paper 11 by Davister and Houghtaling where high-Mg containing phosphoric acid production is described)?

A - To answer this question it must be appreciated that Cros' normal experience is not with acid containing a lot of MgO. In Cros' plants cleaning is very rare. Operation in the U.S.A. with Florida black rock acid, which has a high impurity content, has so far not revealed any significant difficulty.

Q - Mr. R. MONALDI, (Montedison, Italy).

No data about ecological problems are given. Montedison tested since 1971 a pipe reactor and we preferred for ecological and other reasons the ammoniator described in paper 11 presented at ISMA Conference at the Hague (high efficiency ammoniator). We ask : What is the NH_3 concentration in gases at the exit of the cooler stage? What is the total weight of ammonia in the exit gases referred to 1000 kg of 18-46-0 ?

A - Once again we cannot recall the contents of the paper referred to by Mr. Monaldi, and most certainly not the details of the ammoniator. Our answer, consequently, has to be within the context of the paper presented by us here today. The total ammonia going to the scrubber is 7 to 10 percent of the ammonia fed to the plant. With the use of any one of a number of appropriate scrubbers, most of this ammonia is recovered so that overall ammonia efficiency is as good as that in a conventional plant, i.e. 98-98. 5%. The pipe reactor does not directly add on to the normal ecological problems arising from any granulation plant.

Q - Mr. N. LAGERHOLM, (Supra AB, Sweden).

Would it not be preferable from an energy point of view to use gaseous ammonia instead of liquid ammonia? Liquid ammonia could be vaporised by low grade heat and a less concentrated phosphoric acid could be used.

A - A good comment and the overall implication is accepted without reservation. In an operation confined to an isolated granulation plant, a suitable low temperature heat source may not be available and the likelihood of it being available at zero cost is remote. Such a plant would however import phosphoric acid and the concentration of merchant grade acid is likely to be above 50 % P_2O_5 regardless of the state of process ammonia.

Q - Mr. S.L. PENG (CRA, Brazil).

I wish to know whether the addition of sulphuric acid in your pipe reactor, which seems to be a general practice, is justified by technical or economical reasons. If sulfur in the product is treated as a secondary nutrient, there may be cheaper ways of supplying it. If sulfuric acid is used to generate heat in order to save fuel, there may be some economic problems involved. If it is added to prevent scaling of the pipe, well, it is then the price you pay for using the pipe reactor. Am I right?

A - We do not add sulphuric acid for the purpose of preventing scale formation. We make large tonnages of MAP without the addition of sulphuric acid. The use of sulphuric acid in our formulations reflects economic considerations peculiar to our group of companies. The agronomic and thermal advantages have been coincidental and totally supportive to our commercial objectives. However owing to recent price changes we are now using progressively decreasing amounts of sulphuric acid, and more often than not this is the bare minimum required by the formulation.

Q - Mr. S.L. GLASS (Esso Chemical, Canada)

What materials are being used in the construction of the S.A. Cros reactor?

A - Normally for MAP and other grades not requiring sulphuric acid feed to the reactor, the material is 317L. For sulphuric acid Hastelloy C is used.

Q - Will the S.A. Cros reactor also effectively utilise 42% acid.

A - Yes, the reactor can utilise 42% P_2O_5 acid. The additional water introduced with the acid however requires to be removed at the expense of heat energy. Higher recycle ratios may also be required. The slurry from the reactor, however, can have a higher solids concentration than that of slurry from conventional system using 42% P_2O_5 acid, when slurry solids have to be maintained at 85 to 88% maximum for it to be fluid enough for pumping.

Q - Mr. S. LIIMATAINEN (Kemira Oy, Finland)

Do you think there is enough experience from the full-scale plants to recommend which is the better way of granulation with pipe-reactor : pugmill or drum granulator? If one method is better than the other - what are the main reasons?

A - We believe that the factors determining the choice between a drum granulator and a pugmill are not influenced by the pipe reactor. The choice may be economical i.e. equipment cost. If ammoniation is carried out in the granulator, then the drum is preferred. This also applies to the case of most of the slurry process plants.

Q - Mr. S.V. HOUGHTALING (Davy Powergas, U.S.A).

What rock was used to manufacture the acid used to make the DAP discussed in the paper. What was the Fe_2O_3, Al_2O_3 and MgO content of the rock?

A - Moroccan phosphate, low in R_2O_3 and MgO was used.

Q - Mr. J. ENRIQUEZ (Davy Powergas, U.S.A.).

Does Cros have any experience handling Central Florida rock in the pipe reactor ?If so, how long does it run before it has to be cleaned out?

A - Yes. A reactor is in operation in the U.S.A. No scaling was observed after one month operation during commissioning. We expect to receive information relating to subsequent operation, including use of other rocks from Florida.

Q. Mr. P. SUPPANEN, (Kemira Oy, Finland).

The retention time in pipe reactor is very short and changes in the feed of raw materials affect directly changes in the feed to granulator. Does this disturb the granulation? What is the control system you use to secure the exact raw material feed to pipe reactor?

A - The reactor operates under stable conditions. The only comment we can make is that it is necessary to employ suitable feeding instruments and systems to exercise tight control.

Q - Mr. VINKE (Windmill-Holland, Netherlands)

Do you experience any scaling in the Cros pipe reactor and, if so, how frequently has this to be cleaned out? What types of acid (rock source) has been used so far.

A - We have not experienced any scaling . Experience in our plants has been with acid from Morocco rock. However, recently a plant fitted with Cros pipe reactor has been using Florida black rock acid without any signs of scale build-up in the reactor.

Q - Mr. A.R. TORABIAN (N.P.C., Iran)

Don't you have any nozzles plugging with slurry containing 4% water to granulator?

A - No, we dont have any plugging.

TA/78/19 Pilot and demonstration scale developments in production of ammonium phosphate-based fertilizers using the pipe and pipe cross reactors by B.R. Parker, M.N. Norton, J.W. McCamy & D.G. Salladay, Tennessee Valley Authority, U.S.A. (Rapporteur Mr. O.H. Lie, Norsk Hydro, Norway).

Q - Mr. F. MOSTAD, (Supra AB, Sweden)

According to Cros' experience they emphasize the importance of avoiding polyphosphate formation in the pipe-cross reactor, because this is not good for either granulation (higher recycle ratios) or for the product quality. How does this experience fit within TVA's experience?

A - TVA's experience shows that polyphosphates are more mobile in the soil, more readily soluble when the solid product is converted to fluid fertilizers, and provide greater plasticity for granulating UAP grades. In addition, polyphosphates improve storage characteristics and improve utilization of micronutrients (notably zinc oxide) by the soil.

Q - Mr. I. PODILCHUK, (Ind. Luchsinger, Brazil).

What is the highest production rate of MAP possible and achieved with one pipe-reactor?

A - We have produced 30 tph of 11-55-0 MAP (containing 25% poly) in our Demonstration Plant (TVA) with a 6 inch pipe. A commercial plant in the US has produced 30 tph of 12-48-0 in a pipe cross reactor.

Q - Mr. R. LOSTE (S.A. Cros, Spain).

Is the scaling buildup in the pipe related with the water content in the slurry?

A - Scale buildup is related to water content of slurry as well as impurities in feed and temperature in the pipe.

Q - Do the distribution pattern of the slurry and internal back pressure have any influence on the scaling problems? About scaling, which pattern is better: open-end, turned-down elbow or spaced drilled holes?

A - We have not related scaling with pipe back-pressure or distribution patterns. We prefer drilled or slotted pipes for pilot-plant work. The turned down elbow is satisfactory for large applications.

Q - In your opinion, the buidup rate is strongly related to the acid source. Which impurities and level of solids content have more influence ?

A - I believe that magnesium impurities are very detrimental, but other solids (Fe and Al) as well as gypsum may contribute. We have operated our pilot plant on acid with more that 5% solids. Sludge acid containing as much as 12% solids has been used in commercial pipe-Cross reactors.

Q - Mr. W.H. COATES (Humphreys & Glasgow, U.K.).

In making NPK is urea added as solid or melt - and if melt, how is this added to the system - and does it overcome problems of urea decomposition?

A - We have both solid and molten urea for NPK grades. Solid urea is added to the recycle material. Molten urea is added downstream from the pipe-cross reactor effluent. Little urea decomposition occurred when operating in this manner.

Q - What is TVA's experience in adding ammonium nitrate to the T-reactor system. Do they use solid AN or melt? How is AN added and what formulations have been achieved?

A - We have not added ammonium nitrate to the T-reactor system. We have added ammonium nitrate as a separate stream to pilot-plant pugmill producing 24-24-0 grade.

Q - Mr. F.G. MEMBRILLERA (S.A. Cros, Spain).

Have you any explanation to the fact described in your paper about the influence of the reaction temperature on the scaling inside the reactor? In other words, do you know why scaling increases between 300°F and 400°F?

A - Scaling is less when pipe is below 300°F because of sufficient moisture (8-15%) in the slurry to prevent crystallization (same result as scaling). Between 300°F and 400°F, the pipe-cross effluent may be a combination of thick slurry and melt which may readily crystallize and cause stoppages (not necessarily scaling, per se.). Above 400°F, the pipe-cross effluent is essentially a fluid melt that flows readily.

Q - Mr. G.R.G. COULTHARD (Haldor Topsøe, Denmark)

The Cros Pipe Reactor process seems to be a direct development from early TVA work. Do you agree? To what extent does the TVA Pipe-Cross Reactor process differ from the Cros Pipe Reactor process?

A - I believe that the TVA Pipe or Pipe-Cross is of simpler design than the S.A. Cros-Reactor. S.A. Cros premixes the acid before feeding; TVA does not.

Q - "Currently 15 TVA Pipe-Cross Reactors are being used in granulation plants" (Ref. page 276). Is there any information feedback from these plants generally available to industry, if so, please give examples.

A - TVA Pipe-Cross reactors are used in more that 15 plants. Results from operation of these plants are available through TVA.

Q - Do you think that your Pipe-Cross Technology is sufficiently proven that a new large plant in a developing country could use it without undue risk? Please define the risks.

A - I believe that a Pipe-Cross system can be installed anywhere with no greater risks than for conventional plants. However, limited drying capability would be advisable to allow for production of some grades from inferior raw materials.

Q - Mr. I. MINI (Montedison, Italy)

We notice that in your tests with fertilizers other than DAP (excluding 33-11-0) the ammoniation degree of P205 in pipe reactor is always less than 1. What is the true reason, considering that with higher ammoniation the cost is lower? Perhaps ecological reasons?

A - Operation of the pipe-cross with N/P ratio less than about 1.0 drastically reduces NH_3 evolution from the reactor. In some cases, a scrubber may not be required for ammonia recovery when operation in low N/P range.

Q - Mr. A. DAVISTER (Stê de Prayon, Belgium).

What are the acceptable solid, Fe_2O_3, Al_2O_3 and MgO contents in the acid?

A - The question is partly covered in the answer to Mr. Loste. Between 3 % and 5 % R_2O_3 in acid did not significantly hinder operation.

Q - Mr. S. HOUGHTALING (Davy Powergas, U.S.A.).

Does TVA or anyone present have experience producing DAP (18-46-0) in a pipe reactor using run of plant acid produced from Central Florida rock? If so is there a scale problem in the pipe reactor?

A - TVA has used "run of the plant" acid from Central Florida in pipe-cross pilot-plant work with good results. We have not had a significant scale problem.

Q - Mr. J. LE PAGE (Cie Sén. Phos.Taiba, Senegal).

In Table VII you mention a production test of 13-13-13 without urea with a N/P ratio of 16.0/12.9 in the pipe reactor. What is the material used in the pipe reactor in that test?

A - Hastelloy C-276 has been used in pipe cross reactors used in most tests. I assume it was used in production of the 13-13-13 grade.

Q - How long can that reactor work in industrial use?

A - Some Hastelloy C-276 reactors have operated for more that 3 000 hours without failure. Proper purging and flushing during shutdown procedure is essential for long life of pipe cross reactor.

Q - How far can we go in increasing the N/P ratio without using urea ?

A - The maximum N content achieved without supplemental nitrogen is 18 % in 18-46-0.

Q - Mr. I.S. MANGAT (Humphreys & Glasgow Ltd., U.K.)

Do you not feel that in the pilot plant your recycle cooler is oversized thus giving very cold recycle which overcomes granulation problems which you otherwise might face?

A - The TVA Pilot Plant - recycle cooler may be oversized for some grades and rates. However, we compensated by varying air rates and temperature to give recycle temperatures we consider best for granulation. Granulation is hindered by having recycle material too cool as well as having it too hot. This fact should be kept in mind when either modifying an old plant or designing a new plant.

Q - Mr. L.H. BLATCH (Triomf Fertilizers, South-Africa).

You state an optimum heat flux of 0.6 x 10^6 BTU/hr in^2 in pipe reactor. What disadvantages occur if you operate at much higher heat flux, say 1.5 x 10^6 BTU/hr in^2 and higher back pressure?

A - Operation at too high heat flux in the pipe cross (ie. 1.5 x 10^6 BTU/hr in^2) may decrease ammonia absorption efficiency. Also, operating at high back pressures may make precise feeding of raw materials to the pipe difficult with conventional feed systems.

TA/78/20 The effect of impurities on the product qualities of MAP by J.A. Brownlie, E. Davidson, T.R. Dick & I.S.E. Martin, Scottish Agricultural Industries, United Kingdom (Rapporteur Mr. O.H. Lie, Norsk Hydro, Norway).

Speaker : Mr. I.S.E. Martin, SAI.

Q - Mr. I. PODILCHUK, (Ind. Luchsinger, Brazil).

Has phosphoric acid from hemihydrate process been used to make MAP by SAI process? If so, did you observe any difference in granulation between MAP produced with acid from dihydrate and hemi-hydrate process as reported by Kemira Oy Finland in paper N° 12?

A - SAI has no experience of acid from hemi-hydrate process. It is being used by one of our licensees and there is no difference in granulation properties of the MAP produced.

Q - From what phosphate rocks phosphoric acid was used so far to make MAP by SAI process?

A - "PhoSAI" has been made from phosphoric acid derived from Morocco, Florida, Nauru, Senegal, Togo, Gafsa, Kola and Jordan phosphate rocks and has always shown good storage and granulation characteristics when used in all types of granulation plant.

Q - Mr. N. ROBINSON, (Fisons, U.K.).

What are the residence times of the primary and secondary reaction vessels? Are the optimum times dependent on the amount of acid fed to the secondary vessel? What type of equipment is used to disengage moisture?

A - 1½ hours and 10 min. Residence time does not appear to be very critical. We have only limited experience with full scale modification. Moisture disengaged is vented through main moisture disengagement unit.

Q - Mr. D.W. LEYSHON (Jacobs Engineering, U.S.A.)

It is generally accepted, at least in the U.S., that shrinkage losses in front end loader handling, in truck, rail or ship handling amount to at least 2 to 3% for "run-of-pile" (powdered) TSP. What is the general level of shrinkage experienced with handling powdered MAP? Does the particle size, crystal size or drying affect the amount of shrinkage?

A - Particle size and crystal size do not affect shrinkage. We do not dry "PhoSAI". 2-3 % shrinkage seems very high, we would expect no more than 1 %.

Q - Mr. R. LOSTE (S.A. Cros, Spain).

In the NPK production, usually powder MAP is ammoniated inside the drum to a N/P ratio between 1.3/1.6. Does the fixation of NH_3 improve when "PhoSAI" MAP is used instead of flash MAP?

A - We did not try to ammoniate flash-dried "PhoSAI", mainly because we do not ammoniate in our granulator. We have ammoniated "PhoSAI" for a short period in one of our ammonium nitrate based plants and had no problems. One of our licensees doubled his production rate for granular DAP by replacing part of the phosphoric acid feed by "PhoSAI", so ammoniation of "PhoSAI" is easy.

Q - Mr. M. GAURON, (Cofaz, France).

To explain the good storage behaviour of MAP you consider that impurities coat crystals and constitute a physical barrier between crystals. In addition, to explain the granulation properties you consider that impurities constitute a favourable binding. Is there no contradiction between these two statements?

A - This does appear to be a contradiction, but both effects take place. "PhoSAI" does tend to stick together when in a granulator, but the particles remain separated when under normal storage conditions.

TA/78/21 The use of MAP in large capacity granulation plants by R.F. Barut, Heurtey Industries, France; I. Kotlarevsky, Gardinier S.A., France (Rapporteur Mr. N. Kolmeijer, Windmill Holland, Netherlands).

Presented by Messrs. R.F. BARUT and J. CARIOU.

Q - Mr. F. MOSTAD, (Supra, Sweden).

It is surprising to see that you have reached in your plants a recycle ratio of less than 1.2 : 1 for a 17 + 17 + 17 product based on MAP and

AN. Could you please give us complementary information on conditions with regard to granulator, dryer, recycle and raw material feeds?

A - Average figures obtained in the GARDINIER-plants are as follows :

- Granulator : temp. 80-90° C
  - moisture content : 2.5 %
  - granulation efficiency : 60 % between 1.6 & 4 mm.
- Dryer : inlet gas temp. : 200-220°C
  - outlet gas temp. : 110-115°C
  - product out : 95°C
  - retention time : less than 30 min.
- Recycle : temp. : 70-75°C
  - moisture : 0.4-0.5 %
- Raw materials : AN-solution : 95-97 %
  - temp. of the MAP-powder : 40-60°C

Q - Mr. N. KOLMEIJER (Windmill Holland, Netherlands)

What is the N/P-atomic ratio of the Ammoniumphosphate component in your 17 + 17 + 17?

A - 1.5 to 1.6.

Q - What is the granule size of the 17 + 17 + 17 product at the quoted low recycle ratio ?

A - 95 % between 2.2 and 4 mm.
100 % is larger than 2 mm.

Q - Mr. J.L. TORAL (S.A. Cros, Spain).

The flowsheet on page 312 shows that you produce MAP in a reactor which is located outside the granulator. In my opinion this is a disadvantage from the energy point of view as you loose part of the reaction heat in the spraytower, so that you must burn fuel oil in order to dry the granulated MAP. Please enlarge on the advantages of your method.

A - The process described has not been designed for the manufacture of granular MAP but primarily for DAP and NPK-fertilizers. However, it is still possible to use the plant for the production of granular MAP out of MAP-powder. In that case small extra quantities of phosacid and ammonia are used in the granulator, such that the heat of reaction suffices to dry out the humidity which is brought in with the MAP-powder. The dryer is only used to start the plant; once the unit is running the flame in the combustion chamber can be extinguished.

Q - Mr. I.A. BROWNLIE (Scottish Agricultural Industries, U.K.).

With reference to page 303 of the preprint :
What moisture % and temperature is necessary to avoid caking of the MAP?

A - The conditions which are required to avoid caking of the MAP depend on the quality of the phosacid which is used for its manufacture. With good quality acids (such as obtained from Morocco- or Taiba-rock) a moisture content of 5% can be tolerated. When low grade Florida-acid is used (which has high Fe- and Al-contents), the moisture content must not be over 4% and the temp. 50° C max.

Q - Mr. F. ACHORN (Tennessee Valley Authority, U.S.A.)

Why use MAP in granulation plants? Why not use phosacid and ammonia which react to MAP in the granulator? The heat of reaction can then be used for drying the product.

A - I think I have partially dealt with this question when I answered Mr. TORAL about the manufacture of granular MAP. For other products it is easier to react liquid or gaseous components on a bed of finely divided particles with a large active surface than when the reaction must take place directly between e.g. a gas and liquid-droplets with a relatively small specific surface.

CORRIGENDA

TA/78/10 Page 10-6 Second paragraph, fifth line, should read : "The main conclusion of the characterization study ..."

Page 10-11 Table 5 B

The residence time in hours for the Araxa and North Carolina runs should be "7 hours" and "5-7 hours", respectively, not "5-6 hours" as stated in both cases. The mean solids content of the slurry for the Araxa run should be "33.2 %", not "33.1 %" as stated.

Page 10-11 First paragraph, last sentence, should read : "The small silica particles in the cake resulted in <u>a</u> relatively low average ..."

Page 10-12 Table 6A
The weight of sludge after clarification of concentrated Hazara acid for 3 days at 60°C should be "117 kg/ton of crude acid", not "96 kg/ton" as stated. The weight of water-insoluble solids in the sludge after clarification of concentrated Hazara acid for 7 days at 60°C should be "3 %", not "2.9", as stated.

Page 10-13 Table 6B
The weight of sludge after clarification of concentrated North Carolina acid for 3 days at 60°C should be "60 kg/ton of crude acid", not "149 kg/ton", as stated.

TA/78/17 Page 17-12 (228)
Delete the following sentence in the middle of the page :

"keeping unaltered the amounts produced during a year and the grades of finished products".

Page 17-13 (229)

Add the following paragraph :
"Referring this figure to the consumption of the two different rocks in one year, we obtain the price ratio referred to one ton of rocks :

$$K_1 = \frac{135,000 + 115,000 + 100,000}{159,000 + 113,000 + 97,000} = 0.74$$

and the relative value of the tested rock :

$V = K_1 \times 38.6 = 28.56$ doll/t. "

Page 17-21 (237)
Add the following formulae :

"Price ratio referred to one ton of rocks :

$$K_1 = k \times \frac{350,000}{159,000 + 113,000 + 97,000} = 0.74$$

Relative value of tested rock : $V = K_1 \times 38.6 = 28.56$ doll/t.