

ISMA* Technical Conference

Edinburgh, United Kingdom

14-16 September 1965

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

RECENT DEVELOPMENTS IN PHOSPHORIC ACID MANUFACTURE

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Abstract

The present trend in fertilizers is towards high concentration complex materials, and phosphoric acid is an increasingly important intermediate. Sulphur being in short supply, sources of primary acid other than sulphuric acid are being sought for phosphate rock acidulation. While nitric acid is presently limited to the production of N-P fertilizers, the use of hydrochloric acid in phosphoric acid manufacture has become of increasing interest. The latter trend is due to progress made in the application of solvent extraction in phosphoric acid technology.

The solvent extraction method has now been extended to the purification and concentration of "wet process" phosphoric acid. This also permits "wet acid" manufacturers to diversify into the field of phosphatic materials based on high grade, high concentration acid.

Introduction

Development in the fertilizer industry has resulted in the production of fertilizers of high plant nutrient content, and the current trend of fertilizer technology is to produce concentrated products. Superphosphate, for example, has an average of 20% available P_2O_5 , whilst triple superphosphate has 46%, and by treating rock phosphate with superphosphoric acid a concentrated superphosphate of 55% P_2O_5 can be produced. The value of ammonium phosphates (11% - 21% N and 45% - 53% P_2O_5) as fertilizer has long been recognised, and the potential value of monopotassium phosphate with 52% P_2O_5 and 35% K_2O and potassium-ammonium phosphate in complete fertilizer is beyond doubt.

The advantage of concentrated materials with respect to packaging, storage, shipping and handling costs renders them competitive in certain areas with ordinary fertilizers, even though the production costs for ordinary fertilizer may be lower. In addition, it has become important for countries using large amounts of fertilizers per hectare to use fertilizers free of ballast constituents, in order to diminish the accumulation of undesirable salts in the soil which may cause damage to various crops.

The role of concentrated phosphoric acid as an intermediate in the production of multinutrient high analysis fertilizers is well established. The planned erection of a superphosphoric acid plant in Morocco expected to produce 1000 tons of acid per day and to be increased to 5000 tons/day is a testimony to the present trend towards higher concentration in phosphoric acid manufacture.

Phosphoric Acid Manufacture

In principle there are two ways to make phosphoric acid : acidulation and thermal treatment. The application of thermal acid for fertilizer purposes is a function mainly of power costs. In the thermal method the phosphate of tricalcium phosphate is reduced to elementary phosphorus at considerable expense of energy. The phosphorus obtained by reduction is then reoxidized to P_2O_5 to make phosphoric acid with the energy invested in the reduction step wasted. In acidulation, sulphuric acid is the most commonly adopted acid. However, due to the high demand for sulphur, which for its part is largely due to the considerably increasing application of phosphatic fertilizers all over the world, sulphur is in short supply in many localities with its price steadily rising. The price of sulphur a year ago for example was \$19.0 per ton; today FOB Vancouver it is \$31.5/ton. This is an increase of \$12.5 per ton which is equal to well over \$10.0 increase in the price of sulphur per ton of P_2O_5 . Further price increases are to be expected as sulphur producing areas restrict exports in order to ensure adequate supplies for the home market.

As a result of these trends, processes for manufacturing phosphoric acid with acids other than sulphuric acid are receiving increased attention. Nitric acid is usually used in making N-P fertilizers directly and not really for making phosphoric acid as such. In recent years, with the introduction of the Israel Mining Industries solvent extraction process, the use of hydrochloric acid for the manufacture of phosphoric acid became technically and economically feasible. In this process, the solvent such as n-butanol or i-amyl alcohol extracts the acid as such without the need for a special reagent as is used in metal extraction. Solvent losses are negligible and do not exceed 4 kgs. of solvent per ton of P_2O_5 produced. This low figure is achieved :

- a) by the chemical stability of the solvent in the system;
- b) by the fact that the bulk of the solvent in the process - in fact over 99% - remains in a closed cyclic liquid flow at ambient temperature, and only less than one percent of the circulating solvent is subject to recovery by distillation.

Solvent extraction in Phosphoric Acid Manufacture

Formerly, hydrochloric acid could not be used because of the difficulties in separating the calcium chloride formed during the acidulation, from the phosphoric acid, since both are soluble in the aqueous reaction medium used. This difficulty has been overcome now. In the process, which has been described in the literature (1) (2) (3), rock phosphate is dissolved in aqueous hydrochloric acid and the phosphoric acid is separated from the calcium chloride brine by solvent extraction.

While both sulphuric and nitric acids are deliberately manufactured, hydrochloric acid is almost always a by-product or waste. A direct cost comparison on acid value is therefore dependent on the use to which the hydrochloric acid is put. The graph in Fig. 1 presents break-even prices of hydrochloric acid in comparison with those of sulphuric acid when used in phosphoric acid manufacture.

When phosphoric acid is made by leaching phosphate rock with sulphuric acid the product is relatively impure, the degree of impurity being dependent on the rock composition. A typical analysis is presented in Table I.

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- (1) Brit. Chem. Eng. 4 223 (1959)
 - (2) Chemical Eng. Prog. 52 100 (1962)
 - (3) Chemical Age of India 14. 641 (1963)

Figure 1

Break-Even price of Hydrochloric Acid in comparison with Sulphuric Acid in the manufacture of Phosphoric Acid.

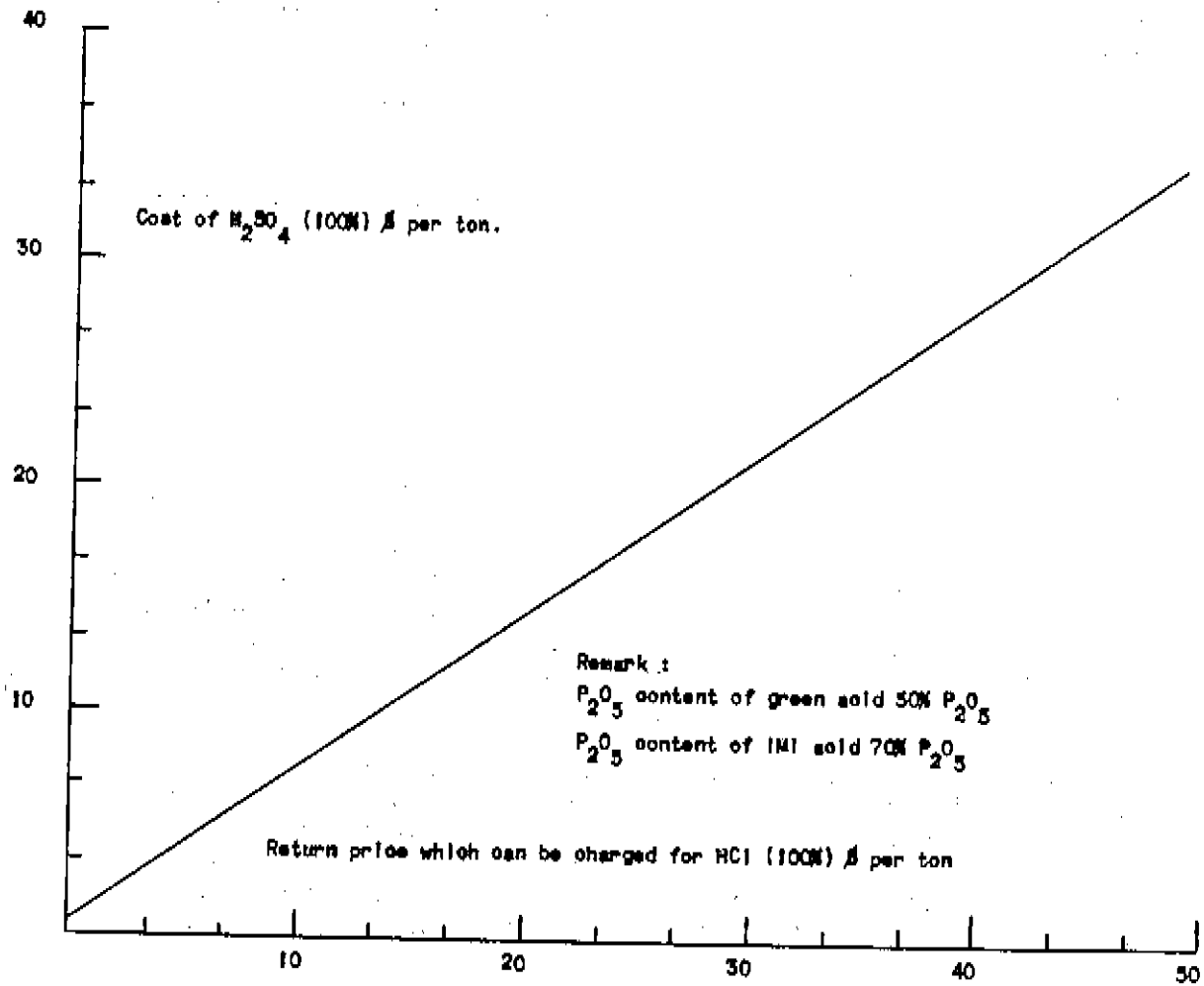


TABLE ITypical Analysis of "Wet Process" Phosphoric Acid

	<u>%</u>		<u>%</u>
H ₃ PO ₄	72	TiO ₂	0.58
SO ₄	3.0	Al	0.3
Fe	0.7	V	0.24
Ca	0.2	SiO ₂	0.14
F	0.7		

The high content of contaminating dissolved solids prevents concentration by indirect evaporation beyond about 70% H₃PO₄. Furthermore liquid fertilizers are not readily made from such an acid, because precipitates are formed. Purification of this acid by multiple precipitation methods entails considerable manipulation and expense.

The hydrochloric acid process gives a phosphoric acid of high concentration and adjustable purity. Furthermore, a plant producing fertilizer-grade acid by this method can easily meet specifications for detergent, pharmaceutical and food grade acids. Typical analyses are given in Tables II and III.

TABLE IITypical Analysis of Technical Grade Phosphoric Acid made by the IMI Solvent Extraction Process

	<u>%</u>
H ₃ PO ₄	95
Fe	0.03
Zn	0.09
Fe	0.03
As	0.005
V	0.004
Cu	0.009
Cr	0.007

T A B L E IIITypical Analysis of Food Grade Phosphoric Acid made by the
IMI Solvent Extraction Process

	<u>%</u>
H ₃ PO ₄	95
Cl max.	0.001
Heavy metals as Pb max.	0.002
Fe max.	0.002
As	0.0001

The advantage gained with regard to acid quality and concentration when based on hydrochloric acid is clear from the above. However, hydrochloric acid must be used where it is available, and this may not necessarily be where the manufacture of phosphoric acid is desired. Sulphuric acid, however, can be produced for captive use wherever it is required. Therefore a further significant step in phosphoric acid technology is the recent extension of the IMI solvent process to the purification and concentration of "wet process" (green) acid made with sulphuric acid. The procedure applies both to filter acid (25% - 30% P₂O₅) and to concentrated wet process acid of 50% P₂O₅.

Solvent Extraction in Purification of Wet Process Acid

In this newly developed purification process, wet process phosphoric acid is mixed with calcium chloride brine. A system is obtained similar to that produced by dissolving rock phosphate in hydrochloric acid. From this system pure phosphoric acid is selectively separated by solvent extraction from the calcium chloride brine, which is recycled.

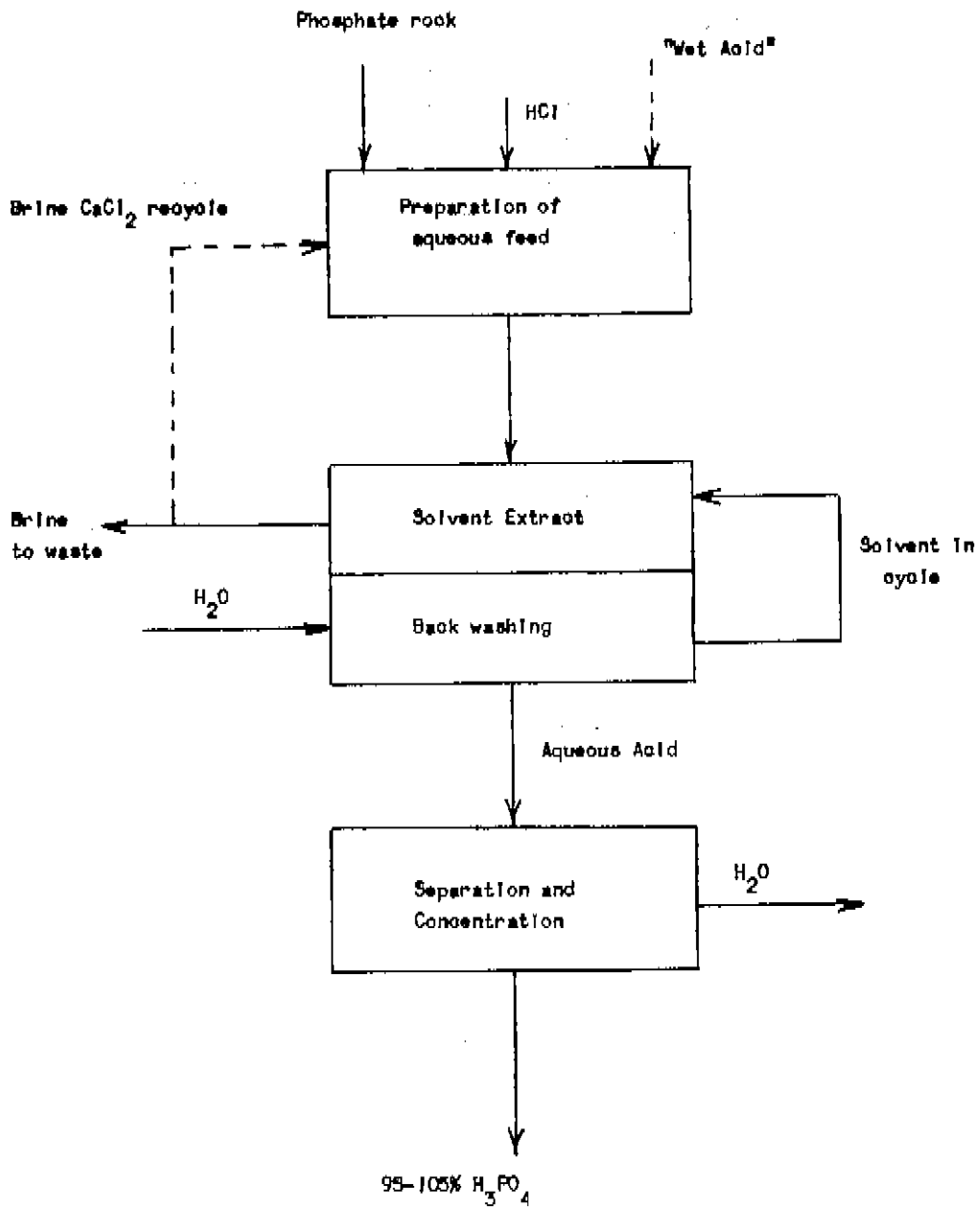
In locations where hydrochloric acid is available only in limited quantities and below the market requirements for high concentration phosphoric acid, a combination of the two variations of the solvent extraction process permits any scale of production, utilizing whatever quantity of hydrochloric acid is available.

The main steps in the solvent extraction process, as illustrated in Figure 2, are :

- a). Preparation of the aqueous phosphate feed liquor.
- b). Solvent extraction of the phosphoric acid from the aqueous liquor.
- c). Separation and concentration of the product.

Figure 2

MAIN STEPS OF THE IMI PHOSPHORIC ACID SOLVENT EXTRACTION PROCESS



Altern. A - IMI Hydrochloric Acid - Solvent Extraction Process

Altern. B - IMI Solvent Purification Process of "Wet Acid".

Step (a)

When sufficient hydrochloric acid is available, step (a) is a rock dissolution operation. Here the difference from sulphuric acid acidulation is of interest: the quantity of hydrochloric acid-insoluble residue is small, say 10% of the rock. This makes the solid-liquid separation a minor step compared with the major separation of gypsum in the sulphuric acid process. When green acid is to be used for the solvent extraction process, step (a) consists of mixing this acid with hydrochloric acid dissolution liquor or with recycle calcium chloride brine.

Step (b)

The physical chemistry of this operation has been described previously (4). Once the aqueous feed liquor has been constituted as in step (a), its behaviour is independent of its history, and this is the reason why the product is of a definite quality quite independently of how one starts. Phosphate ores which cannot be considered in the sulphuric acid method can thus be used with hydrochloric acid. An example of this is high iron and high aluminium rock.

Step (c)

Green acid (or filter acid as it is also called) will usually contain 25 - 30% P_2O_5 . The acid obtained by solvent separation will always be less concentrated than this even when filter acid is used as feed. On the other hand filter acid is rarely concentrated beyond 50% P_2O_5 because of the scaling problems in indirectly heated evaporators. There is no scaling problem with solvent acid, and direct concentration to 70% P_2O_5 or more is a straightforward operation. Multiple effect evaporation is applied, permitting efficient calorie utilization, 0.5 ton of steam per ton of water evaporated being a normal figure.

Investment and Production Costs

Comparative cost figures are given in Table IV for two types of plant, one operating wholly on hydrochloric acid, the other devoted entirely to purification and concentration of wet process (green) phosphoric acid with recycle calcium chloride brine. The estimate assumes a location similar to Western European conditions.

(4) Dechema Monographien 33. 57 - 68 (1959).

T A B L E IV

Estimated Costs of a 75 metric tons P₂O₅/day Phosphoric Acid Plant

	Hydrochloric Acid as Acid Sources	Purification and Concentration of Wet Process Acid as Feed Acid with 30% P ₂ O ₅	Purification and Concentration of Wet Process Acid as Feed Acid with 50% P ₂ O ₅
	\$	\$	\$
Cost of erected plant battery limits	2,400,000	1,450,000	1,000,000
<u>Operational costs.</u>			
Basis : 1 ton P ₂ O ₅			
<u>Materials</u> (excluding acid and rock)	<u>4.20</u>	<u>5.33</u>	<u>3.88</u>
<u>Utilities</u>			
Steam 2.2 \$/ton	4.7 tons 10.34	5.2 tons 11.44	3.2 tons 7.04
Power at 8.7 \$/1000 KWh	230 KWh 2.00	130 KWh 1.13	90 KWh 0.79
Process water at 5 c/m ³	7.5 m ³ 0.38		negligible
Cooling water at 0.3 c/m ³	150 m ³ 0.45	170 m ³ 0.51	110 m ³ 0.33
Sub Total	13.17	13.08	8.16
Labour - av. \$1.20 per man hour	2.04	2.04	2.04
Overhead (100% of labour)	2.04	2.04	2.04
Maintenance (5% on erected plant, 330 days)	4.85	2.94	2.92
Depreciation (6.7% on erected plant, 330 days)	6.50	3.93	2.71
Insurance	<u>1.33</u>	<u>0.83</u>	<u>0.56</u>
Sub Total	16.76	11.78	22.31
Total	<u>34.13</u>	<u>30.19</u>	<u>22.31</u>

not including interest on capital.

The operational costs for purification and concentration differ in accordance with the concentration of the feed acid. In evaluating the process for a specific case, the self cost of 30% - 50% P_2O_5 acid must be taken into account.

A plant which has been designed with a view to purification and concentration of wet acid only can be converted to operate on hydrochloric acid by additional investment, to incorporate a dissolution section. On the other hand, a plant which has been designed for a definite scale of operation based on the availability of a fixed quantity of hydrochloric acid can handle a considerably larger throughput of P_2O_5 without additional investment by mixing wet process acid with hydrochloric acid dissolution liquor. An increase of 30% to 50% in the production of high quality concentrated acid can be achieved in such a combination. In other words, a plant based on hydrochloric acid with a nominal production of 75 tpd P_2O_5 can accommodate an additional 30 - 40 tpd P_2O_5 from a sulphuric acid source, without increased investment.

The concentration and purity of phosphoric acid are important factors in the end use of this chemical.

The trend in phosphate fertilizers is toward high analysis materials such as ammonium phosphates, concentrated super-phosphates and high concentration compound fertilizers mainly based on phosphoric acid. The estimated consumption of phosphates in 1971 reaches 18.7 million tons P_2O_5 . If the consumption of basic slag remains at the present level of 12%, then in 1971 over 16 million tons P_2O_5 will be consumed based mainly on phosphoric acid. This, if based on sulphuric acid, would require 13.5 million tons of sulphur, and utilisation of acid sources other than sulphuric acid is becoming of increasing importance.

Some of the end products must be made from acid of high purity. The most substantial amounts of phosphoric acid used for purposes other than fertilizer is consumed in the production of sodium tripolyphosphate for use in the detergent industry. For this purpose as for other salts of phosphoric acid such as trisodium orthophosphate, baking-powder phosphates, acid for phosphating steel car bodies and components, pure phosphoric acid is required. Purified "wet process" phosphoric acid made by the IMI solvent extraction process meets required specifications at costs well within economic feasibility.

DISCUSSION

MR. A. ALON (Israel Mining Industries, Israel) : In view of the present difficult sulphur supply position, the use of acids other than sulphuric acid is becoming increasingly important. The IMI solvent extraction phosphoric acid process has been described in the scientific and technical literature and has been implemented on an industrial scale. The process uses hydrochloric acid for the dissolution of phosphate rock and separates selectively phosphoric acid from the accompanying chloride by solvent extraction. This method has now been extended to the purification and concentration of wet process phosphoric acid. In this extension, calcium chloride is used in recycle. A further possibility, not mentioned in my paper, is the combination of hydrochloric acid dissolution with wet process phosphoric acid. In this case, for each unit of P_2O_5 produced by hydrochloric acid an additional 4 - 5 units of P_2O_5 can be added from wet process phosphoric acid. Calcium chloride recycle in this case falls away and investment costs are then substantially lowered. The product acid in any of these cases can be of a high purity, complying with pharmaceutical and food-grade specifications, detergent requirements and, obviously, any fertiliser requirements. This high purity and concentration permits manufacturers of wet process phosphoric acid to diversify into the field of phosphatic materials based on high concentration and high purity.

MR. C. HEUDIER (Péchiney-St. Gobain, France) : The solubilisation of phosphate rocks by sulphuric acid combines P_2O_5 and S in well defined proportions; but these proportions are not those which best satisfy the requirements of plant growth. From this point of view, therefore, the sulphur is wasted. This is even truer in the case of phosphoric acid manufacture, since the fertilisers thus produced do not convey the sulphur to the plants.

We are gradually entering a period of relative sulphur shortage. Thus one can only congratulate Israel Mining Industries on having studied a phosphoric acid production process which is totally independent of sulphur. The interest of the process on the scientific level alone and by virtue of the direct or indirect practical developments which one can expect to flow from it, is evident.

However, we shall certainly have to pay for the HCE, whatever its origin. Figure 1, page 4, does not enable us to calculate the possible price limit, unless we suppose that all other costs relating to two plants of identical capacity, the one working with H_2SO_4 and the other with HCl, amount to the same overall total - which is certainly not the case. Can the author give us some comparative figures ?

Similarly, can the author tell us the largest plant size which is currently envisaged for the single stream operation of the IMI solvent extraction process? How do costs vary with size?

MR. ALON : With regard to the first question relating to the price comparison between hydrochloric acid and sulphuric acid, this comparison is made on an equivalent basis. For each acid equivalent of sulphuric acid, the acid equivalent of hydrochloric acid is given. Thus, one ton of sulphuric acid is compared with approximately 0.75 tons hydrochloric acid.

As to the capacity of the plant, 200 - 250 tons P_2O_5 per day can be envisaged today for a single-train plant. As to the influence of costs on varying production capacities, I should mention two figures I happen to have at hand: conversion costs per ton of P_2O_5 for a 75 t.p.d. plant are about \$22, whilst for a 30 t.p.d. plant they are \$29. The cost of erected plant, calculated according to Israeli conditions, which are approximately 15% higher than Western European conditions, amounts to \$1 million in the former case, and in the latter case to about \$500,000.

For each ton of P_2O_5 obtained in the true hydrochloric acid dissolution process, 1.8 - 2.0 tons of hydrochloric acid are required, depending on the phosphate rock used.

MR. HEUDIER : The reaction of phosphate rock with hydrochloric acid leaves an insoluble residue which must be separated. Is this separation not more or less difficult, according to the origin of the phosphate attacked? What is the influence of the clay matrix and the content of organic matter and fluorine?

MR. ALON : It is true that phosphates of different origins behave in different ways. We have examined many phosphates, including all commercially available types and a large number of commercially unavailable types, and we have encountered no difficulties which would cause unsatisfactory separation between insoluble and soluble fractions. As to the organic substances, these remain in the dissolution liquor after extraction and do not pass into the solvent extract. With regard to fluorine, when using hydrochloric acid only, part of the fluorine escapes during dissolution. Whatever remains in the dissolution liquor in the course of extraction, the distribution is such that it is retained totally in the aqueous phase and does not enter into the solvent phase. This is reflected in the fluorine content of the final product, which is of the order of 1 - 3 p.p.m.

MR. HEUDIER : Is the content of impurities in the solution obtained really independent of the phosphate used?

MR. ALON : The original impurities in the phosphate rock have no influence on the composition of the final product. The process is subject to the laws of distribution, but the flowsheet

is operated in such a way that all the impurities remain in the aqueous phase during the solvent extraction or, within the framework of the normal flowsheet, they are removed without any excessive additional treatment.

MR. HEUDIER : In particular, to extract iron from the solution, does it not have to be in divalent form ?

MR. ALON : You are right. There is more than one possibility with regard to iron. We have worked with rocks containing as much as 6 - 7% iron or as little as 0.2% iron. With low iron contents it is, or can be, advantageous to reduce the iron to its bivalent form. With very high iron contents, using the same solvent, pre-extraction is effected in a closed cycle.

MR. HEUDIER : The acid analyses quoted as examples do not give fluorine content. What is it ? And how is the fluorine separated ?

MR. ALON : In the case quoted in the paper, fluorine was in fact absent to the limit of determination, which was in this case 1 p.p.m. But, as mentioned before, the fluorine content can be in the range of 1 - 3 p.p.m.

MR. HEUDIER : Part of the organic matter in the phosphate must pass into solution. How is it subsequently separated ?

MR. ALON : Any organic material passing into solution in the solvent extract is extremely small. It may lend a certain colour to the end product. In this case a number of methods can be applied for its removal, i.e. very conventional methods of discolouration.

MR. HEUDIER : Have you observed the formation of silica gel ? How is this remedied ?

MR. ALON : With normal phosphate rocks we have not experienced the formation of silica gel. We did experience it with certain types of phosphates when the silica was solubilised, and without using any complicated procedures we were able to inhibit any effect of the silica.

MR. HEUDIER : On page 3 - b) you say that less than 1% of the solvent in circulation has to be recovered by distillation. Just before this, you say that the solvent loss does not exceed 4 Kg. per ton of P₂O₅ produced. Can you indicate what the 1% of solvent to be recovered by distillation represents by weight in relation to P₂O₅ produced ?

MR. ALON : Without going into flowsheet details, there is roughly 1% solvent in the spent brine. This is the quantity recovered by distillation. The figure of 4 Kg. per ton of P₂O₅ applies in the case of using solely hydrochloric acid.

When hydrochloric acid is combined with wet process phosphoric acid, where less calcium chloride brine is in circulation, the loss is about 2 kg. per ton. It is to a certain extent a function of the quantity of spent brine.

MR. HEUDIER : Table 4, page 9, gives steam costs for the different hypotheses considered. According to these costs, it seems that the purified acid is obtained at, or reduced to, a concentration of about 10% P₂O₅. Is this conclusion correct? If it is, is this not a serious handicap for the process? Can we not expect some improvement in this respect?

MR. ALON : When purifying wet process phosphoric acid and recycling the calcium chloride brine, certain dilution of the brine takes place. In fact, a major part of steam consumption is devoted to reconcentrating the calcium chloride brine. If we take the combined case where one unit of P₂O₅ is produced by HCl and 4 units by the addition of wet process phosphoric acid, the steam consumption per ton P₂O₅ is 1.7 tons, compared with 3.2 tons when calcium chloride is recycled.

MR. HEUDIER : Can you indicate the level of the industrial yield of P₂O₅ in the operations described in your paper?

MR. ALON : In the case of purification and recycle of calcium chloride brine, it is virtually 100% because the calcium chloride brine is recycled with any residual P₂O₅. When hydrochloric acid alone is used, recovery is between 98% and 99%.

MR. F. W. EDWARDS (Dorr-Oliver, U.K.) : Do the battery limits mentioned in Table IV include rock storage, handling or grinding or any acid storage? Does the term "erected plant" include all construction work, i.e. buildings, foundations, and civil engineering work within the battery limits?

MR. ALON : In the case of using only hydrochloric acid, the battery limits cost given includes rock storage, handling and acid storage. In the case of using wet process phosphoric acid, it does not include these items, because it is assumed that the wet process acid will come from a phosphoric acid plant. In this case, only a small day storage tank for the acid is provided. The term "erected plant" does not include buildings, foundations and civil engineering.

MR. EDWARDS : Your capital and operating costs appear to be high, and I query the need for such high purity and such a high concentration of acid for fertiliser manufacture. Even for high analysis NPK solid fertilisers, 30 - 45% P₂O₅ acid is usually adequate. Superphosphoric acid will surely be produced mainly for reasons of minimum transport costs rather than for any advantages in the direct production of NPK fertilisers.

MR. ALON : There are many uses for relatively pure

phosphoric acid. As far as superphosphoric acid is concerned, I should refer to the excellent paper presented to this conference by Mr. Powell, of International Minerals and Chemicals, U.S.A. As to the cost of processing wet phosphoric acid with 50% P_2O_5 to convert it to a 70% P_2O_5 acid equalling the quality of thermal acid, this is done for \$22 without capital charges or, say, \$25 with capital charges per ton of P_2O_5 . Is this cost too high? I think this is answered if we ask if it is too costly to convert wet acid, the price of which, at the most conservative estimate, will certainly not exceed \$100 per ton of P_2O_5 , to an acid which - again very conservatively - will not cost less than \$180 per ton P_2O_5 ? In these circumstances, is a conversion cost of \$22 high or not? This is for the manufacturers to decide.

With regard to concentration, it should be added that the farmer will eventually benefit because of the diminished transportation costs.

MR. EDWARDS : Can this process be used for separating phosphoric acid and calcium nitrate, where nitric acid is used for the attack of phosphate rock?

MR. ALON : Substantial work would be required to adapt the method to this use.

MR. EDWARDS : What commercial units are in operation using this process?

MR. ALON : Total licensed capacity using the IMI process is now 50,000 tons P_2O_5 per year. One plant has been in operation in Japan on a relatively small, though industrial, scale for the past three years. A large plant is now in course of erection in Brazil and is expected to be in operation by the end of the first half of 1966.

MR. D. KEENS (Power-Gas Corporation Ltd., U.K.) : On your table showing product quality, you do not indicate the amount of solvent remaining in the product at the p.p.m. level. This is of interest from the point of view of any unusual surface tension effects and subsequent foaming in evaporators and neutralisers in an ammonium phosphate plant.

MR. ALON : Even in the p.p.m. range, the solvent can be considered as totally absent. The product has the same surface tension figures as thermal acid.

MR. KEENS : As is well known, and as we have found in our own crystallisation work, the problem is very much easier using furnace acid rather than wet process acid in ammonium phosphate manufacture. One problem is foaming; another is impurities which precipitate and spoil the crystal growth. Do you think your process represents a breakthrough in this respect, in that your acid has properties closely resembling

those of furnace acid for the purposes of crystallisation ?

MR. ALON : In the course of the purification step in the solvent extraction, certain refinements can be made and any desired purity can be obtained; but even if none of these additional purification treatments are made, the impurities are so small that in the neutralisation with ammonia in the production of diammonium phosphate, absolutely no precipitates are present.

I should also mention that the viscosity of our acid remains low at high concentrations. Moreover, the 70% P_2O_5 acid does not contain polyphosphates. This low viscosity is, of course, a definite advantage in certain cases.

MR. P. MORAILLON (Péchinay-St. Gobain, France) : What is the P_2O_5 content of the phosphoric acid obtained before its final concentration ?

MR. ALON : It lies within the range of 15 - 35% P_2O_5 : it all depends on flowsheet details.

MR. MORAILLON : Is it possible to use phosphates high in iron and aluminium, containing relatively little lime ?

MR. ALON : Yes, it is. We have in fact used such types of phosphate rock on pilot plant work; but for each rock we have to make an examination as to its solubility in hydrochloric acid.

MR. R. BAUWENS (Ets. Kuhlmann, France.) : Has Mr. Alon found it possible to recover the calcium chloride ?

MR. ALON ; The calcium chloride can be recovered and obtained in solid form by evaporation. It can be purified : the fluorine can be precipitated as calcium fluoride. The spent calcium chloride brine is of the order of 30%.

MR. J. FROCHEN (Pierrefitte-Bassens, France) : In his paper, Mr. Alon says that the purity of the product acid can be regulated. By what means is this regulation of purity effected ?

MR. ALON : By means of the addition of one, two or more mixer-settlers; but this is all within the framework of the extraction process.

DR. R. F. McFARLIN (Armour Agricultural Chemical Co., U.S.A.)
In discussing the commercial possibilities for this process in those countries in which a solid calcium chloride might not be desired, has anyone ever raised the question of disposal of the calcium chloride brine? If so, what suggestion have you been able to make?

MR. ALON : If there is no commercial outlet for the calcium chloride, I am afraid that this remains a problem where there are also no adequate waste disposal arrangements.