

# ISMA\* Technical Meetings

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

1) A GENERAL SURVEY OF PLANT CONSTRUCTION FOR THE MANUFACTURE OF SULPHURIC ACID BY BURNING PYRITES FOR SUPERPHOSPHATE PRODUCTION.

Since before World War No. 1 we have been confronted with the problem whether it might be advisable to substitute contact process or oleum plants for chamber process plants.

The post-war period however brought war-time experience, leading to a number of technical changes, covering the improvement and renewal of equipment and methods of working so that the lead chamber process continued to be popular.

Thus, from 1920 to 1935 we witnessed a development of the leading principles by which the older traditional 3 - 4 chamber method, with its high surface volume/ratio of lead and consequent poor heat dissipation, slow reaction etc., was abandoned and much more logical and rational methods were adopted, involving a low surface volume/ratio, more intense reaction and, therefore, larger yield, the new methods being the tower-type chamber processes (Moritz, Mills Packard, Gaillard, Parrish etc.).

Yield per square metre of lead increased  $\times$  10 fold, in some cases threefold, and general expenses were reduced. At the same time the technology of equipment, namely burners, pumps, towers etc., was improved and so was the working technique.

The old lead chamber method thus succeeded in competing, also as to production costs, with its new and strong competitor, the oleum process. This vitality of the lead chamber process, however, was only secured by a maximum of effort.

The improvements brought about with lead chambers, however, showed the fundamental fault of this method: the difficulty in adjusting and proportioning the various parts to suit them to their various functions and to ensure maximum efficiency.

The great number of reactions ( $\text{SO}_2$  absorption,  $\text{NO}, \text{NO}_2$  absorption, re-oxidation of  $\text{NO}$  to  $\text{NO}, \text{NO}_2$ , dissipation of reaction heat and finally production of  $\text{H}_2\text{SO}_4$ ) cannot find conditions adequate for their developing simultaneously, but are limited by the lack of parts specially constructed for the individual reactions.

The improvements in technique naturally grew more and more refined. The result has been the adoption of tower plants, embodying the idea of having the single parts specially suited for the functions they are to perform (production tower, re-oxidation tower, heavy circulation of acids and nitrous gases etc.) and permitting yields of acid at  $53^\circ - 60^\circ \text{C}$ , at a rate of over more than 200 kilos per cubic metre of tower.

Apart from this, such plants are, so to say, interchangeable if, instead of pyrites, sulphurous waste gases from metallurgical furnaces, Gill furnaces or the like are to be used. They therefore allow the greatest flexibility since they can operate very well with gases varying considerably in their three most important features: temperature, flow quantity and  $\text{SO}_2$  contents. It thus becomes possible to utilise gases, such as Gill furnace gases, where there are temperatures not above  $100 - 150^\circ$  and very variable  $\text{SO}_2$  contents in the range between 1 and 5%. This is a case of true industrial recuperation.

Some tower plants have been erected and others are in course of construction in Italy.

These plants offer the following advantages in comparison with the lead chambers, however intensive they may be.

- (a) less space required;
- (b) acid produced is all at 58/60°Bé and can therefore be transported in iron tanks;
- (c) with one concentration tower (for pyrites burning plants) about 35% of the acid can be produced at 66° Bé, or else part of the heat of the gases can be recovered with a boiler (up to about 1/2 ton of steam per ton of pyrites);
- (d) smaller lead requirements; about 75% less than needed for the chamber method;
- (e) cost of construction of plant 7% less than of chambers;
- (f) overall production cost slightly inferior (by about 5%) to that of chambers. The lower rate of interest and depreciation offsets somewhat higher working costs (nitric acid, electric power, water) involved by the exigencies of the tower method.

Tower plants however do not represent a complete solution for industrialised zones, where the acid is used for industrial purposes besides for superphosphate production.

In this case the higher purity of the acid, monohydrate and oleum, the better transport facilities, as compared with 50/60° Bé acids, lead to a preference for erecting oleum plants, even if higher installation costs are to be faced.

The characteristic features to be considered in examining present plants are the following:

- (a) We think that the most rational plants are those for 60 tons up to 80 tons maximum. Under 40 tons working costs are too high while, beyond 80 tons, the size of the towers becomes excessive and impracticable.
- (b) Installation costs are heavy: the rate of interest and depreciation is about twice as great as for tower plants and about 30% higher than for chamber plants.
- (c) Working costs are about 8% higher than with chambers and about the same as with the tower method.
- (d) Production cost is about 15% higher than with chambers and towers owing to the rates of interest and depreciation.

In order to neutralise, partly at least, some negative factors in the economy of oleum plants, the various parts of such plants were improved during recent years, working has become more simple and the basic installation costs have become more moderate.

The most important technical changes effected may be summarised as follows:

- (1) The gas heating furnace for starting the plant has been replaced by electric resistances thus saving space, installation cost etc.
- (2) The expenses for roofed buildings have been reduced to a minimum, some parts, such as towers, now being placed in the open air.
- (3) There is a tendency to replace hot electrofilters, which are very delicate and the maintenance of which is burdensome, by cerofilters; we prefer Van Tongeren patented filters and are carrying out experiments in this field.
- (4) We are near to utilising, a great deal in temperature of the combustion gases for producing steam. We reckon that we may be able to

recover at least 1/2 ton steam per ton of pyrites, thus affecting in a favourable way the production cost of oleum by saving about 9% of its total cost which, to a great extent, makes up for its higher cost as compared with chamber and tower acids. To this end, the Americans have employed boilers with water pipes, lined with silicon iron, but they appear to recover only modest quantities of steam in this way.

As to ourselves, we utilise the jump between 550° and 300°, avoiding the dew point of the gases, and mounting simple boilers with vibrating water pipes in order to prevent cinders from precipitating, with the aid of an automatically starting naphtha burner in case the furnaces should unexpectedly cease operation or the temperature of the gas should suddenly fall off.

- (5) Catalyst cases have been simplified and are now made more practical by developing temperature regulation, heat recovery and catalyst position.

It is not possible to compare the technical requirements and trends of the various countries, which are the result particularly of the kind of raw materials used for sulphuric acid.

The United States use about 75% sulphur for the production of sulphuric acid. In 1946, of about 3,500,000 tons 100% acid produced, 5,500,000 tons were produced with oleum plants (about 67%).

In England, in 1946, about 50%, that is about 700,000 tons 100% acid were produced with oleum plants. The relatively modest percentage of 35% of acid used for superphosphate shows how widely used sulphuric acid is for industrial purposes.

In Italy, specially in Northern and Central Italy, industrialised zones are in the majority.

Hence the necessity of decidedly going in for oleum equipment with the new plants for superphosphate and for the old plants to be renewed even if, owing to the almost exclusive use of pyrites which is characteristic of Italian industry, heavier burdens are involved for purifying equipment and for equipment in general.

The facility of availability of a technically pure acid, concentrated in the form of monohydrate or oleum and, therefore, transportable even over great distances, will make up for some of the heavier burdens in installation and production costs.

Even without referring to war-time, during which oleum had a special function, and taking as a base 1938, we see that Italy, of a total of about 1,100,000 tons of acid produced in that year, produced about 190,000 tons of oleum.

The ratio is only 17%; now we are tending, with our new programmes of reconstruction and new construction, to increase this proportion.

Our raw material is almost entirely pyrites, and we have to put up with the technical complications in plants which are a consequence of this fact.

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On the other hand, it is made quite clear by the above statements that oleum production can be further improved technically, as shown by recent developments and as may be expected in the future.

2) THE RECOVERY OF FLUORINE IN THE SUPERPHOSPHATE INDUSTRY.

It is known that in the manufacture of superphosphate, besides the principal reaction between tricalcium phosphate and sulphuric acid, there is a whole series of secondary reactions due to the various components in the mineral, the most important being  $\text{CaCO}_3$ ,  $\text{NaF}_2$  and  $\text{SiO}_2$ , both free and in the form of silicates.

By decomposition with sulphuric acid, gypsum and silica are formed and gases such as  $\text{CO}_2$ ,  $\text{SiF}_4$ ,  $\text{HF}$  and aqueous vapour are evolved.

Fluorine gases, in particular, are obnoxious both to man and to vegetation: hence the necessity for thoroughly washing these gases so as to purify them before releasing them into the atmosphere.

The older and decidedly practical methods are known; the trend was not so much to recover fluorine as to disperse it in washing water through drainage and through exhaust systems.

Since 1925 our Company has been organizing its recovery plants and perfecting them more and more so as to attain almost complete recovery of fluorine from the gases, at first in the form of solutions of hydrofluosilicic acid, and then in high grade chemical products.

Nearly all the factories have small sodiumfluosilicate plants, equipped for a product of high purity (99% of  $\text{Na}_2\text{SiF}_6$ ), which could be utilised especially for the most exacting industries, enamel, glass, potteries etc.

Until 1933 this was the most important product recovered and up to 20,000 quintals a year were produced.

In 1930, a new trend arose in the field of fluorine recovery. Since 1930, the Italian aluminium industry has gradually but decidedly expanded, thus requiring more and more purchases of cryolite imported from Greenland, the sole source of supply in the world.

Owing to the high standard of purity of the cryolite, necessary for aluminium production, especially in regard to the content of  $\text{SiO}_2$  and  $\text{Fe}_2\text{O}_3$ , it appeared very doubtful, at that time, whether a synthetic fluorine compound might be obtainable at a high degree of purity so as to compete with natural cryolite. The bibliography and patents, known on the subject of manufacturing double fluorides of sodium and aluminium, showed the possibility of manufacture but did not guarantee the high degree of purity of the product, required for the aluminium industry.

After long and patient studies and experiments, beginning in 1933, our Company erected at Porto Marghera, a plant for manufacturing high purity synthetic cryolite, entirely based on superphosphate fluorine recovery, by utilising both hydrofluosilicic acids and sodium fluosilicates derived from them.

In 1938 the plant was largely extended so as to permit a production of about 2000 tons per annum which was enough to cover, not the total, but a great deal of the needs of the Italian aluminium industry.

Many plants of the Group cooperate in this cryolite production by supplying the raw material, the ones nearest to Porto Marghera by furnishing  $H_2SiF_6$  solutions, the others by supplying sodium fluosilicate.

This programme, however, required settling two kinds of technical difficulties and technological problems:

- (a) Determining the best technical conditions of manufacturing superphosphate so as to ensure maximum recovery of fluorine without impairing the high quality of the superphosphate. Unfortunately, only abt. 25% of the 3 - 4 % of fluorine, expressed as  $H_2SiF_6$ , contained in phosphorites, can be recuperated. With non-continuous dens we have nevertheless succeeded in obtaining a superphosphate with a soluble fluorine content of not more than 0.15 - 0.20%, which is a good result. As to the fluorine recovery, equivalent to about 25%, methodical trials with mixtures of phosphato in order to correct the excess or lack of  $SiO_2$ , types of more or less active  $SiO_2$ , excess or lack of  $CaF_2$  etc., gave poor results and did not permit recovery to be increased noticeably. This problem is still open to research, and we are aware of the difficulty of obtaining satisfactory results which would be of considerable interest, as will be seen hereafter.
- (b) Perfecting as much as possible plant methods for the recovery of  $SiF_4$  gas in order to recuperate completely the quota of fluorine evolving in the reaction and in the form of  $H_2SiF_6$  at high concentration (not less than 250/300 grammes/Litro) so as to obtain solutions easy to transport from the subsidiary plants in ebolite lined tank cars to Porto Marghera, which plant with its production of more than 100,000 tons of superphosphate per annum already contributes on the spot a high production of fluorine.

In the case of recovery plants simplicity of construction has been aimed at; since the extent of the recovery is modest, expensive and cumbersome technical equipment is to be avoided.

Our most recent installations comprise ebolite lined iron pipes, high efficiency absorbers of metal sheeting, lined with ebolite, equipment with atomisers of the solutions circulating till saturation (these absorbers operate in series, their number being in proportion to the size of the superphosphate plant) ebolite lined washer-ventilators and one or two final cement towers for dispersing the gases. The plant is completed by a system of vacuum filters for washing and exhausting the  $Si(OH)_4$  formed and for recuperating and re-circulating weak  $H_2SiF_6$  solutions.

The vacuum filter represents the most simple and practical means, since more complete equipments, such as rotary filters, band filters etc., are too expensive and too difficult to maintain for the small quantities of silica involved.

We are also developing a plant design which represents a further improvement in the devices worked out up to now.

The results of the operation of this plant may be characterised by the output figure of 95% recuperated fluorine compared with the fluorine available at the outlet of the dens and mixers.

The preparation of this fluosilicic acid concentrated at 25°-30° Bè

also raised problems of a certain difficulty regarding the resistance of materials suitable for treating such solutions.

The aluminium industry thus was given by the superphosphate industry a solution of the problem of high purity cryolite supply, which is the electrolyte of its electrolysis process.

The necessity, however, remained of having recourse to supplies from abroad of another chemical product needed for its electro-chemical process phase, namely aluminium fluoride in crystals.

The Italian aluminium programme required considerable supplies which up to 1942 were purchased from abroad. It is known that there are very many patents and that a great deal of research on this particularly difficult production has been carried out.

Our Company succeeded in 1942, after continued research, in erecting a small modern plant for 3,000 quintals per annum of this product, based on fluorine recovery from superphosphates. By using concentrated  $H_2SiF_6$ , the most important problems were solved, as stated below:

- (a) Conditions of attack ruled by limits of pH of the  $AlF_3$  solution, permitting the minimum possible loss of Al and F in the silicon sludge;
- (b) Conditions of filtering ruled by limits permitting minimum loss due to insolubilisation of  $AlF_3$  in the silicon sludge;
- (c) Conditions of crystallisation of  $AlF_3$  solutions permitting a minimum of  $AlF_3$  in brine to be re-circulated in the working cycle;
- (d) Conditions of working cycle avoiding passing through the various hydrates of  $AlF_3$  and the complications involved thereby, such as cooling installations, re-melting the crystals etc., which are characteristic of the processes known up to now. The only hydrate in our cycle is  $3.5 H_2O$ ;
- (e) Conditions of calcination of the hydrate developed in such a way as to obtain a product with very high  $AlF_3$  content, practically without any losses of fluorine, by splitting up and decomposition of the salt. - We succeeded in obtaining a product highly appreciated by the aluminium industry, with 87 - 90% of  $AlF_3$ , not more than 0.5 - 0.7% of combined water and minimum contents of  $SiO_2$  and  $Fe_2O_3$ , namely 0.3% of each of these two impurities. - Doubling the size of the Porto Marghera plant and putting into production another plant in Central Italy, in course of construction, will permit of better distribution of production, and above all will limit the long hauls of the concentrated 16%  $SiF_6$  solutions in ebolite lined tanks, so as to make production more practical and economical.

Research was recently concluded which is of particular interest, namely, arranging a completely closed cycle of working, thus dispensing with the burden represented in the past by the necessity of utilising about 30% of  $AlF_3$  in weak solutions for the manufacture of cryolite.

This permitted the erection of the new plant in Central Italy which will exclusively be devoted to the manufacture of aluminium fluoride.

At the same time as this production of fluorine salts, other methods have been developed of producing products of considerable interest so as to complete the range of fluorides and fluosilicates.

- Sodium fluoride.- We have a plant with an output capacity of 5,000 quintals per annum.
- Cerium fluoride.- A mordant in the dyeing and printing of wool by the Vigoreux method etc., - A modest plant is being developed.
- Barium Fluosilicate.- An insecticide. We have a plant with an output capacity of about 5,000 quintals per annum.
- Zinc and magnesium fluosilicate.- A hardener and waterproofing material for cement, as well as other industrial uses. - We have a modest plant for about 1000 quintals per annum.  
A new plant for about 5,000 quintals per annum is in course of construction.

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The industrial growth of fluorinated products and their large consumption - especially in the aluminium industry - induced us to persist in recovering more and more fluorine from the superphosphate industry so as to utilise this by-product which in the past was almost completely dispersed. There remains, however, a scarcity of fluorine for the industrial development of various products. Nowadays there is a tendency to utilise the technical equipment, developed so far for the absorption of fluorine, in conjunction with a plant for the decomposition of mineral calcium fluoride as a new source of supply of hydrofluosilicic acid. We are also working in this direction.

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### 3) CONTINUOUS SUPERPHOSPHATE MANUFACTURE - SYSTEM ELLINZONI - MONTecatini.

The large field of observation of Montecatini, with its 45 superphosphate plants, with multiple plants of various types and various capacities, has made it possible to correlate a number of observations in the operation of non-continuous plants, e.g. Wenk, Keller, Baskow etc., which may serve as a guidance in studying the realisation of a rational continuous plant.

One aim in investigating the best technical disposition of plants and working has always been to obtain a product of particular fineness and dryness which would avoid, while maturing in the pile, a number of so-called "maturation" reactions which lead to a particularly hard matured product, the withdrawal of which, besides burdensome in itself, did not permit the manufacturer to offer the customers a product with suitable physical characteristics.

With this aim in view the most modern non-continuous plants have been set up according to the following plan:

- (a) Cutting and ventilation, by means of high-speed revolving blades, of the hot superphosphate at the outlet of the fans, so as to reduce the moisture in the product to a minimum, with a strong draught, following the cutting;
- (b) Piling 200 tons and more for a maturing period of 36 - 48 hours, in order to complete the chemical reaction of the so-called maturation;
- (c) Withdrawal of the so-called "matured" product and definite piling in the storage sheds after further pulverisation and classification by means



of suitable instruments.

This working cycle will give a very soft product for shipping and assuredly a better one - as to its state of humidity and physical aspect - than the product obtained by way of the old working methods.

The solution, however, did not seem perfect for numerous reasons. The hardening of the piled product could not be completely eliminated, the cost of processing appeared to be increased by the new devices and, on the other hand, the development of modern and well realised continuous processes - Broadfield, Nordengren, Moritz-Standaert, Debaisieux, Sturtevant, Maxwell and so on abroad - instigated the search for as complete as possible a solution.

The guiding principles which have inspired the study of a continuous "Montecatini" process, may be summarised as follows:

- (a) Pushing completion of the reactions to the utmost in order to ensure the piling of a product in conditions very near to stability:
- (b) Eliminating consequently any hardening of the stocked product, securing, at the withdrawal for sale, a product of constant softness and powdery texture.

The researches, made in this respect by Nordengren, Lehrecke, Sanfourche, Maryhell, Hendricks, Stevonijs-Nielsen and others, and by our own Pratolongo, reached the conclusion that the anhydride ( $\text{CaSO}_4$ ) is the calcium sulphate predominant in the industrial superphosphate and this form seems stable, even if some views would ascribe a certain influence to the forms which, to a small extent, are derived from semi-hydrate ( $\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$ ), and from hydrate ( $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ). Further the conclusion has been reached that it is monocalcium phosphate which must be considered as the most important factor in the hardening of the superphosphate, and Lehrecke has attributed this fact to the progressive crystallisation of a gel of monocalcium phosphate (together with very minute crystals of anhydrous substance).

It has not yet been shown, however, how and when this gel is formed, and supplementary research to this end is necessary.

- (c) The above mentioned theoretical and practical studies in any event confirmed that the trend ought to be towards a group of devices which might ensure the formation of a monocalcium phosphate of the most "stable" type possible which would not be subject to a form of hydration as a consequence of a failure in the completion of the reactions:
- (d) Consequently two apparatus have been studied to this end. A large volume continuous mixer with maximum permanence of the elements in reaction and maximum completion of the reaction. A continuous den, constituted by units of small volume (500 - 600 litres per unit) where, by avoiding large masses of hardened and soaked superphosphate and long periods in the den (from 60 - 180 minutes), as in almost all present continuous processes, the completion of the reactions, so as to ensure the best physical and chemical features of the product, might be made easy and safe.

We are seeking a solution which would allow the piling of a product with a temperature of not more than 35 to 40°, that is to say, when the reaction is almost completed.

- (e) Care has also been taken that the devices might ensure constant thermic conditions, in order to permit a better exploitation of the reaction heat with the evaporation of a considerable quantity of moisture.
- (f) The final result has been a product with a high percentage of solubilisation (95-98%), considerable softness, powdery and which does not harden when returning in piles.

Our Company has patented the method of continuous working by the Italian patent No. 399796 of January 19th. 1939, (in the name of Montecatini, inventor Bollinzoni), filed also in:

Germany	on	January 13th,	1940
France	on	January 16th,	1940
Spain	on	January 13th,	1940

One plant has been in regular operation at Vercelli since 1942 and has, so far, produced more than 100,000 tons; another one, at Castolguelfo Parmense, installed in 1946, has already produced about 50,000 tons; five further plants will start work with the new equipment at the end of this year and in 1948.

As to the equipment itself, we specify below its various elements:

(A) Equipment for the regulation and the measurement of the acid and phosphate feeds for continuous mixing. The equipment consists of 3 groups:

1. A constant, but adjustable automatic feeder for weighing phosphate; it involves a band type doser of our own make. In order to ensure constant delivery, the band speed must be constant; to avoid the inconveniences in the variation of current frequency we use a direct current electric motor, i.e., a constant speed motor. A deviator at the discharge of the band permits frequent control of weighing. The error coefficient will never exceed an average of 0.5%.
2. A constant, but adjustable sulphuric acid feeder. This is composed of a constant pressure head cylinder, provided with a perfectly calibrated nozzle, so as to have exact and constant measuring. From this the acid passes through a barrel, constructed with the possibility of acting as deviator, so as to ensure the checking of delivery from time to time by means of a cylinder, mounted above a weighing device, which indicates the delivery per unit of time. The equipment also includes a heat control device, to maintain the acid at a determined temperature, which is chosen according to the type of phosphate being used.
3. An electric automatic device for cutting out the feeder group in case of irregularities of operation of the phosphate and acid dosing. Attached to the gear of the phosphate doser is a mercury ball which acts upon the locking device, in case its position shifts either upwards or downwards from its normal position, corresponding to the normal delivery levels. There is consequently an automatic interruption of current in the driving motor of the phosphate doser.

Simultaneously a system of electromagnetic interruption acts upon the acid doser by shifting the deviator and thus stopping the feeding of acid and phosphate to the mixer.

Incidentally, any other irregularity which might occur in the con-

stancy of the delivery of the acid will be signalled, by means of the stem of a float inserted in the electromagnetic circuit, putting the deviator into operation and also the locking system of the phosphate doser.

(B) Double Worm Mixer.

This is composed of a series of iron parts, (normally 4), assembled by means of flanges and bolts, which form the trough of the mixer, (lined with antiacid, refractory material which serves also to prevent the dispersion of heat) and two shafts, provided with advancing and retreating paddles of cast iron, which ensure a perfect mixture of the acid and phosphate. The capacity of the mixer, about 1000 litres, and the operation of the paddles ensure the normal development of the reaction, and the time of the material in the mixer ranges from 5 minutes to 15 minutes, according to the quality of the phosphate, and secures a rather advanced reaction which is controlled by the temperature of the mixture, and chiefly by the consistency in which the mixture leaves the mixer to fall into the buckets of the continuous den.

The regulation of the mobile overflow pipe and of the velocity of the two worm gears (by means of a variable speed motor) are the means by which it is possible to realise the best conditions of mixing and reaction in relation to the various types of phosphates used.

(C) The den is composed of open vessels of a limited capacity (which constitutes the original principle of the patent) forming a continuous mobile chain.

At a given point in their course the hot and friable superphosphate discharges itself from the vessels, the superphosphate being in such a state that it can be crushed by simple means without causing alterations in the physical properties of the product which is then conveyed to storage.

After discharge, the vessel returns to its former position to be filled again with superphosphate coming from the mixer.

The slurry being collected in containers of small capacity (about 500 litres) the completing of the reaction until solidifying can thus be realised in small layers, and the development of the reaction and the escape of the gases will thus be facilitated, the final result being a soft, powdery product which no longer shows the well-known phenomenon of hardening by maturation in the pile.

The mechanical parts of the den are:

- an iron framework which sustains the tipping bucket transporter, comprising a double chain which drives the buckets,
- the buckets of the transporter are of sheet iron, lined with refractory and antiacid material,
- the driving gear comprises a regulator which controls the speed of the buckets so as to allow the superphosphate to remain from 20 - 60 minutes in the buckets according to the phosphate being treated, the density of the acid etc.,
- above the mobile den there are wooden suction caps for absorbing the gases and for conveying them to the absorption plant;
- a metal plate belt serves to receive the superphosphate from the bucket and to convey it to the rotating blade disintegrator with a limited speed,
- a rubber belt conveyor receives the disintegrated superphosphate and takes it to the store. This rubber belt can also convey the superphosphate to the granulating plant or to the rotating dryer to obtain a dried superphosphate.

There may be various types of dens, according to the potentiality of the plant: from 20 to 32 mobile buckets and more, with distances, varying between axles, from about 9 - 16 m, with an output per hour varying from 8 - 15/20 tons, so that there are dens which permit production of 50,000 to 100,000 tons yearly. The employment of labour may be estimated at about two workmen for the whole plant.

The consumption of electric energy does not surpass 5 KW. The maintenance is not expensive: the only parts with heavy wear are the paddles of the mixer which are easy to replace.

Installation costs are assuredly inferior to those of non-continuous plants.

It is calculated that a non-continuous plant will cost from 25 to 50% more than a plant of the "Montecostini" type.

#### Quality of the Superphosphate Produced.

Physical features. We already mentioned the physical features which are the best from every point of view and by far superior to those obtainable by any other system known to us.

The product presents itself in a soft, friable and microgranular form when withdrawn for shipment without any hardness in the pile. A further characteristic, very interesting from an economical point of view, is that this quality of superphosphate can immediately be consigned to customers, without the necessity of maturing or, at any rate, for only a very few days.

Chemical features. Attached hereto are some analyses, carried out with samples of superphosphate immediately after production. These data show very high solubilisation, varying from 97/98% and this, we repeat, without the necessity of a maturing period.

Working conditions. We have obtained this quality of superphosphate, which satisfies the most exacting requirements and which is very well received by customers, without using an exceedingly fine phosphate meal, and attacking the phosphate with 52/53 B6 acid.

The final product has a moisture content varying from 12 to 14%, but as already stated above, nevertheless appears as a very dry and powdery product.

Our product has been obtained with phosphate meal with residues of about 15/20% on the screen of 1350 square cm, whilst in other similar processes the fineness of the ground phosphate was pushed to 5% residue on that screen.

In this latter case the consumption of electric energy per ton of phosphate ground was about 15 KW, but in the former case this consumption is kept below 5 KW, which represents a considerable economy.

It should also be noticed that, with our type of continuous den, we too could grind much finer and mix with stronger acid, so as to obtain a dryer superphosphate. We must, however, point out that the superphosphate, obtained under the present conditions is already, physically and chemically, in so perfect a form that there seems to be no necessity to investigate other means, save that of producing a higher grade by reducing moisture in the superphosphate as obtainable with finer meal. The latter is a method which we ourselves also, as already stated, could practise in the event of its proving more suitable.

(Signed) Ing. B. AZRIA.

STATE TESTS OF SUPERPHOSPHATE PROVED WITH PHOSPHATIC-BULLDOG BRAND.

Phosphate-type	Contents of tricalcium phosphate	Phosphate Seal residue on 1350 mesh screen used	Grade of acid used	H <sub>2</sub> O	Free	Apparatus data obtained			Attack yield	
						20% in H <sub>2</sub> O	soluble in H <sub>2</sub> O or cit-rate	Insoluble		
14/16 Superphosphate	50/63	16%	52,5 53,5 53,5	14,6 14,8 14,0	4,9 3,5 3,1 4,7	13,7 14,0 13,3 13,5	14,4 15,0 14,5 14,5	0,4 0,4 0,3 0,5	14,3 15,4 14,3 15,0	97,2 97,4 97,5 96,5
16/18 Superphosphate	65/68	16%	53,5 53,5 53,0 53,5 53,5 53,5	14,3 12,2 15,5 19,2 13,6 14,0	5,0 4,3 4,2 5,0 4,4 3,9	19,1 15,4 15,3 15,1 15,2 14,8	16,3 16,3 15,4 16,6 15,8 15,8	0,3 0,3 0,2 0,4 0,4 0,4	15,7 17,3 17,0 16,6 16,0 16,2	98,2 97,1 98,3 97,5 97,5
18/20 Superphosphate	75/78	30%	53,0	13,0 12,6 12,0 12,6 13,4 14,2	4,8 3,4 4,0 3,5 4,2 5,0	17,0 17,5 17,7 17,6 17,4 17,0	18,3 18,3 17,5 18,8 18,3 18,1	0,3 0,4 0,5 0,3 0,5 0,4	12,6 18,3 19,0 13,9 13,8 18,5	96,3 96,7 97,3 98,4 97,3 97,8