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A NEW PROCESS FOR THE DIRECT PRODUCTION

OF

CONCENTRATED GRANULAR COMPLEX FERTILISERS

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## 1. INTRODUCTION AND SUMMARY

This paper describes a new technique wherein slurries containing calcium phosphates, ammonium phosphates, ammonium sulfate and ammonium nitrate can be used in a unique granulating system together with dry potassium chloride or sulfate, ammonium sulfate and large quantities of recirculated material acting as seed or nuclei to build up three-component complex fertilizers in the form of closely sized smooth granules. The process is being practised in several large installations.

The three essential components of a complete fertilizer require differing degrees of preparation :-

Phosphorus, derived principally from naturally occurring phosphorites. These are insoluble as mined and must be chemically treated to convert the P<sub>2</sub>O<sub>5</sub> into an available or soluble form.

Nitrogen, aside from the use of minor quantities of natural sodium nitrate, is derived from ammonia and nitric acid both of which must be bound or converted into solid compounds.

Potassium, on the other hand, as delivered to the fertilizer factory in the form of potassium chloride or sulfate, is already in an available and useable form.

In contradistinction to the conventional granulating systems which start with mixing and granulation of dry processed components such as superphosphate, ammonium sulfate and potassium chloride, this process starts from the basic raw materials phosphate rock, phosphoric acid, sulfuric acid, ammonia and potassium chloride or sulfate. The processing of the phosphates and nitrogen is done as part of the overall process which produces the finished granular compounds.

The process comprises a "wet system" and a "dry system". In the "wet system", where the phosphate - nitrogen processing occurs, a slurry is produced which must have a well defined ratio of solution volume to suspended solid volume. This slurry can be adjusted to any desired N-P ratio over a broad range. In the "dry system" a potash salt can be added in any reasonable quantity so that the range of possible compounds is almost infinite. The processing requirements do not limit the possible compounds as in the ammoniation type of granulating system or in the various nitrophosphate processes.

In addition, the process has remarkable flexibility in producing a broad range of fertilizer materials. Essentially the same plant can be used for producing, in practically identical granular form:-

- A. Double or triple superphosphate,
- B. Monoammonium or mixtures of monoammonium and diammonium phosphates,
- C. Ammonium sulfate,
- D. Mixtures of A and C or B and C,
- E. Nitrophosphates,
- F. N-P or N-P-K or P-K compounds.

Typical products that have been produced by this process are: 11-48-0, 16-20-0, 16-48-0, 19-19-0, 19-38-0, 0-47-0, 14-14-14, 12-36-14, 14-28-14, 12-12-18.

The granulating system can be operably adjusted to practise a layering or nucleation technique or an agglomerating and coating technique or a combination of the two. The choice depends on many factors such as the ratio of slurry constituents to dry constituents and the nature of the basic raw materials.

The constituents added as dry salts and the crushed oversize fragments act as nuclei in the layering technique. The dry constituents are stuck together by the slurry and coated with a slurry envelope when practicing the agglomerating technique. These techniques produce an outstandingly uniform, closely sized, and in most cases smooth, spherical and hard granule.

2. GRANULAR AND CONCENTRATED FERTILIZERS

Aside from improvements in processing, of which this process and the economical production of phosphoric acid are outstanding examples, the two most significant recent developments in the fertilizer industry have been the trend towards granular products and towards high analysis complex fertilizers.

These are both significant and logical developments and, interestingly enough, they are peculiarly interrelated.

A granular free flowing product aids in handling all the way from the producer to the soil. It stores and bags readily, works better in the fertilizer drill and eliminates dust nuisance and loss. But most important are its agronomic advantages. It permits better placement and the plant foods dissolve more slowly and are available for a longer period. In this respect it should be pointed out that the response can be varied considerably to suit the solubility of the material and the needs of the crop. For instance, a 1.2 to .8 mm spherical granule has an approximate surface area of 22 000 cm<sup>2</sup> per kilogram of product, a 1.8 to 2.7 mm product 14 700 cm<sup>2</sup> and a 2.7 to 4.0 mm product 9800 cm<sup>2</sup>. Such a range and closeness of sizing is possible with the process under discussion.

Higher analyses permit significant savings in raw materials, as evidenced by the following very approximate and typical comparison of a 1-2-0 fertilizer:-

Low Analysis: 6.75% N<sub>2</sub> - 13.5% P<sub>2</sub>O<sub>5</sub>

100 kg P <sub>2</sub> O <sub>5</sub>	= 500 kg 20% superphosphate requiring	200 kg H <sub>2</sub> SO <sub>4</sub>
50 kg N <sub>2</sub>	= 240 kg ammonium sulfate requiring	180 kg H <sub>2</sub> SO <sub>4</sub>
<hr/>		
Gives	740 kg of product requiring	380 kg H <sub>2</sub> SO <sub>4</sub>

High Analysis: 19.25% N<sub>2</sub> - 38.5% P<sub>2</sub>O<sub>5</sub>

100 kg P <sub>2</sub> O <sub>5</sub>	} = 218 kg of 19 - 46 ammonium phosphate	
and		
41 kg N <sub>2</sub>	} = 43 kg ammonium sulfate requiring	265 kg H <sub>2</sub> SO <sub>4</sub>
9 kg N <sub>2</sub>		
<hr/>		
Gives	261 kg of product requiring	297 kg H <sub>2</sub> SO <sub>4</sub>

However, the principal advantage of high analysis is the saving in handling costs. This saving starts immediately, when the product is put into seasonal bulk storage, which can be much smaller, and then continues on through the bagging (including cost of the bags), loading, transportation and ultimately the farmer must store and handle fewer bags.

This trend to granulation and higher analysis is undoubtedly going to continue and accelerate as the producer, the agricultural authorities and the farmer get to appreciate the advantages and learn to use the products. There is no obstacle or disadvantage to their use given an understanding of necessary changes in practice.

Another interesting trend is the thinking on desirable degree of solubility. In Great Britain and the Commonwealth water solubility of the P2O5 has been the criterion of availability. In many other countries, including the United States, empiric methods have been adopted which allegedly more nearly duplicate the dissolving value of the average soil solution, and citric acid or ammonium citrate solubility has been accepted as the criterion of availability.

In Great Britain there has developed a desire to get away from the water soluble straightjacket and interestingly enough in the United States more and more importance is being attached to water solubility for which reason nitrophosphates have not been generally accepted. Of course, there are many modifying factors such as the pH of the soil but the industry, worldwide, seems to be moving towards a meeting of the minds. This is an interesting angle in the study of processes for producing granular concentrated products. With coarse, uniform granules greater water solubility can be tolerated and is probably desirable.

### 3. PRESENT METHODS OF GRANULATION

The most usual method of granulating compounds in Europe is to dry mix the ingredients, agglomerate the mixture with a small amount of added water and/or steam in a pan, inclined disc or drum granulator and then dry and screen the resultant product. The relatively small amount of fines and crushed oversize are returned. This method has the basic disadvantage that it does not start from the primary raw materials but the processing must be done separately and preliminarily and considerable rehandling and storage of intermediates is involved. It has the advantage that it can turn out a wide variety of formulations and can be added readily to existing conventional plants. It is for these reasons primarily that it has achieved such widespread acceptance.

In the United States, the so-called TVA or ammoniator type of granulation system has been very generally adopted. With this method the superphosphate and other ingredients together with ammonia or nitrogen solutions and in some cases phosphoric and sulfuric acids, are fed into a specially designed rotary drum or pug mill "ammoniator" and subsequently dried, cooled and screened.

A reasonably good granular product is obtained. The reversion which occurs when superphosphate is ammoniated is acceptable in the States thanks to the citrate soluble criterion adopted there. The ammoniation assists granulation and conditioning. This method has the advantage that it permits the production of higher analysis goods and at least partially starts from the basic raw materials. It suffers from the disadvantage that the possible range of compounds and formulations are limited by processing requirements.

In the authors' opinion, either of these two granulation

approaches constitutes a transitional phase in the trend to granular and high analysis compounds and a more fundamental approach to the problem is desirable and will probably be the main reliance of the industry in the future. It is believed that this will take the general form of a "slurry process" wherein starting from the basic phosphato and nitrogen raw materials a slurry is formed which is then, together with an added source of potash, used in a granulating system. A plant will then consist of salts, ammonia and acid storage, a granulating plant and product storage and shipping. No intermediate processing, curing or rehandling will be required. To all practical purposes an unlimited variety of compounds and formulations will be possible. The products will all be in identical physical form and be stable, granular, non-hygroscopic and free flowing.

The various nitrophos processes only partially meet these specifications. These are serious formulation limitations and the product characteristics are not ideal.

The process which will now be described represents, it is believed, a long step forward in the search for the "process of the future".

#### 4. DESCRIPTION OF A TYPICAL PLANT

The Flowsheet, Fig. 6, is a simplified diagrammatic representation of a typical plant employing the new process. Many variations, not shown, are possible and will be described in a later section. This flexibility - the process is a system for producing a variety of granular one, two or three element plant foods, and not a process for producing a particular product - is a fundamental feature.

##### A. "Wet System"

If double or triple superphosphate is to be produced or complexes are to be made based on double or triple superphosphate as the P<sub>2</sub>O<sub>5</sub> source, then the "wet system" comprises a phosphate rock bin 11, a gravimetric rock feeder 12, flow controllers 13A and 13B, reaction tanks 14A, 14B and 14C, an exhaust fan 15 and a scrubber 16. The phosphate rock and phosphoric acid (and/or sulfuric acid) are reacted to form a slurry of solid mono and diacalcium phosphate suspended in a monocalcium phosphate - phosphoric acid solution phase. The evolved fluorine, carbon dioxide and water vapor are exhausted by a fan 15 and scrubbed in an atmospheric pollution abatement scrubber 16. The scrubber solution goes to waste or to fluorine recovery. The reaction temperature is maintained at 90 to 95°C primarily by the exothermic heat of reaction and dilution, and secondarily by the introduction of live steam to the last reaction tank. The slurry overflows to the "Blunger" 41 of the "dry system".

If ammonium phosphate or complexes based on ammonium phosphate as the P<sub>2</sub>O<sub>5</sub> source are to be produced, then the "wet system" comprises the flow controllers 13A, 13B and 13C, a dissolver 17, an exhaust fan 15 and an ammonia recovery scrubber 16. If the desired N:P<sub>2</sub>O<sub>5</sub> ratio can be obtained by a combination of monoammonium and diammonium phosphate alone (1:2.5 to 1:4.3) then phosphoric acid and ammonia are fed into the reactors. This is a stagewise operation, the reasons for which will be explained later. Ammoniation to substantially monoammonium phosphate is accomplished in the first reactor 14A, and the ammoniation is completed to the desired N-P ratio in reactor 14B and, depending on the phase conditions, the slurry may be cooled and stabilized in the third reactor 14C. If ammoniation is carried only to a 1:1 mole ratio the partial pressure of ammonia in the vapors exhausted by fan 15 are so low that scrubbing can be omitted. The

partial pressure of ammonia over diammonium phosphate bearing solution is sufficiently high so that the vapors are passed through a wet impingement scrubber for recovery of  $\text{NH}_3$ . The slurry from the last reactor overflows to the "Blunger" of the "dry system".

If the desired N:P2O5 ratio requires additional nitrogen over that which can be absorbed in the phosphoric acid then ammonium sulfate is usually employed although urea as salt or ammonium nitrate as solution, are alternate possible sources. The ammonium sulfate may be in the form of the dry salt in which case the salt is dissolved in the phosphoric acid and the returned scrubber liquor in dissolver 17 with the addition of a small quantity of steam. The solution then goes on to ammoniation. If the ammonium sulfate is to be derived from sulfuric acid, then the sulfuric acid from flow controller 13A is introduced into the reactors and neutralized along with the phosphoric acid. In some cases part of the ammonium sulfate salt may be introduced directly into the "dry system" via conveyor 28.

If granular ammonium sulfate is to be produced the "wet system" comprises the flow controllers 13A and 13C, reactor 14C, exhaust fan 15 and scrubber 16. The sulfuric acid is substantially neutralized in the reactor and the final neutralization is accomplished in Blunger 41. The water boiled off due to the exothermic heat of reaction and dilution carries a small amount of ammonia which is recovered in scrubber 16. Either a crystallization inhibitor is added or the slurry from 14C is ground to give the very fine crystals necessary for good granulation.

### B. Filter System

It is usually necessary to adjust the grade of the product. A formulation can be chosen that will come to the desired analysis but it is well, for operating flexibility and to avoid the costs of over-formulation, to add small quantities of an inert material. One very good source, where a phosphoric acid plant is part of the works, is the unfiltered phosphoric acid-gypsum slurry. This can be stored and adjusted for concentration in an agitated tank 31 and then introduced into the reactor by a pump 32 through a flow control device, 33. Alternatively, other materials either dry or in slurry form can be utilized.

### C. Salts System

The ingredients which are introduced into the process as dry salts are usually stored in a shed or silos and conveyed to a recovery hopper 21 which is part of the granulating plant. They are delivered by elevator 22 and conveyor 23 to surge bins 24A, 24B, 24C, 27, etc., since this part of the plant is usually operated intermittently or on one shift only.

The potash salt, potassium chloride or potassium sulfate is fed into the "dry-system" from surge bin 24A by a continuous gravimetric feeder 25A which delivers to conveyor 28. Depending on the screen analysis of the particular potash and the type of granulating technique being followed (see later description) it may or may not be necessary to grind the potash in a mill 26A.

The ammonium sulfate salt, if used, is continuously measured out of the surge bin 24C by gravimetric feeder 25C to a mill 26C and then via elevator 29 to the dissolver 17 previously described.

If a phosphoric acid-gypsum slurry is not available or is not used to adjust the grade as previously described, then a small amount of a dry filler can be used. This can be sand, dolomite, or any available inert material. This is stored in bin 24B and continuously added to the "dry system" by a gravimetric feeder 25B with or without grinding in 26B and via conveyor 28.

When making radical grade changes it is necessary to empty the "dry system" to storage (this is automatic) and to recharge the dry system with some previously produced material of the same grade. This material is placed in surge bin 27 and then introduced into the "dry system" via conveyor 28. This change-over from one grade to another can be done in two to four hours.

#### D. Dry System

This is a cyclic operation so that a description can be started almost anywhere, but the most logical point is the Blunger 41.

Returned undersized granules, crushed oversized granules, recovered dust and the salts, filler and charge stock fed to conveyor 28 as described previously, are all delivered by secondary elevator 56 to the Blunger 41. Here they meet and are mixed with the slurry from reactor 14C of the "wet system". This is a vital part of the process and will be described in more detail.

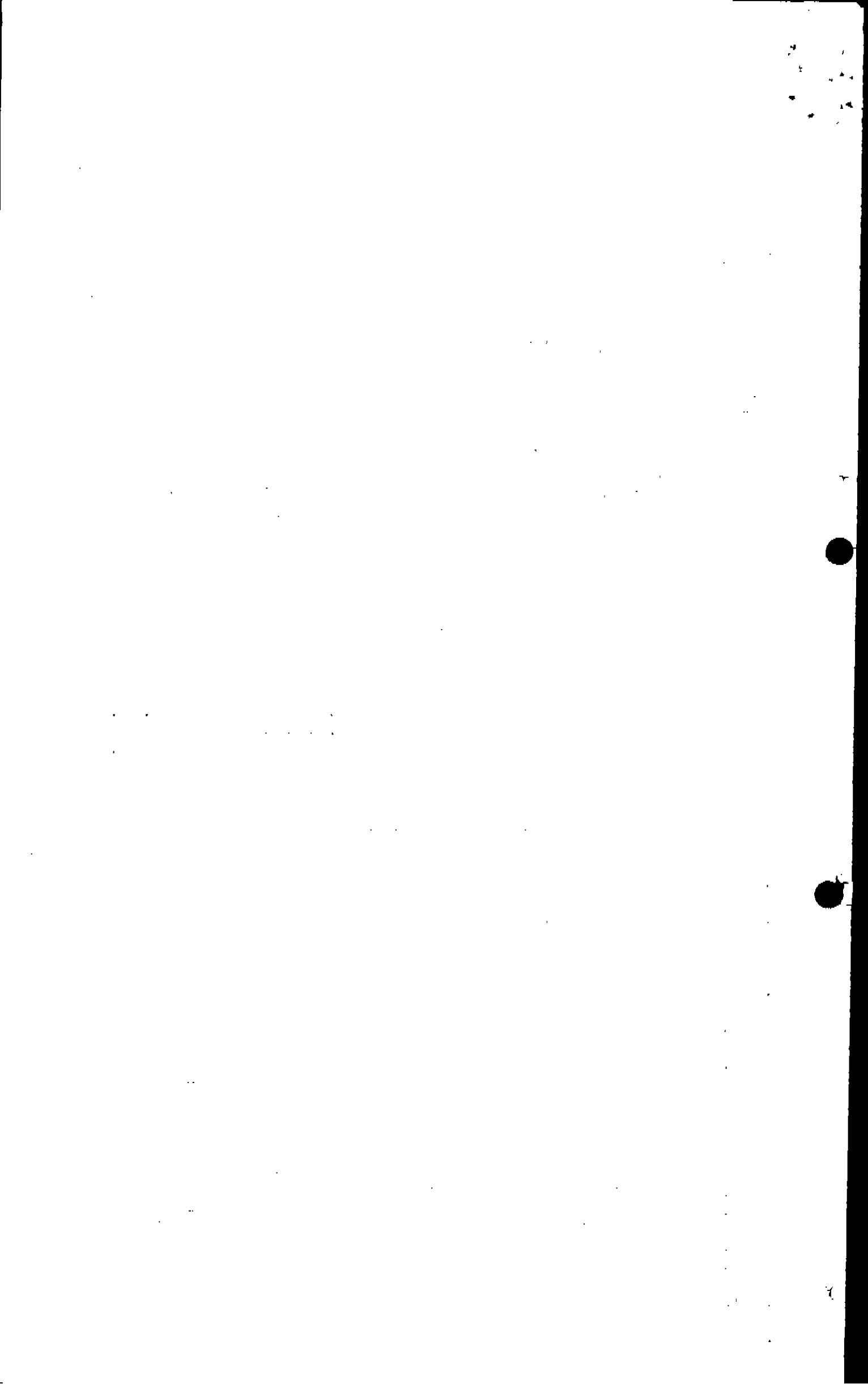
The moist granules gravitate by a steep chute to the drier 42. This is a direct heat con-current unit and must be specially designed for the unusually large through-put. In the case of the production or use of double or triple superphosphate, the drier also completes the reaction. As the water is removed, the increased concentration of the liquid phase forces the reaction toward completion. The combustion chamber 43 which supplies the tempered hot air can be designed to burn gas, oil or pulverized coal. The drier gases pass through a multiple cyclone 44 and an exhaust fan 45 to a scrubber 46. When triple superphosphate is being produced, the scrubber is provided to eliminate atmospheric pollution from the fluorine evolved in the drier. The dust is relatively coarse and the cyclone efficiency high. The scrubbing liquor may be sent to waste. When producing ammonium phosphates, the fine dust escaping the cyclones and any ammonia evolved during drying is recovered in the scrubber and returned to the reactors. The drier is provided with a 1" opening rotating grizzly at the discharge end which removes any casual, coarse material which might obstruct the subsequent equipment. This is crushed and returned to the system.

The drier discharge is carried by primary elevator 47 to the double-dock vibrating screen or screens 48. The undersized granules drop into a surge bin 49 and are then re-introduced into the circulating system by a remotely controlled vibrating feeder 52. The oversize, together with a portion of the on-size material if necessary, is stored in a surge bin 50 and then re-introduced into the circulating system via a remotely controlled vibrating feeder 53 and a hammer mill 54.

The undersize, ground oversize and the dust from the cyclones gravitate to a fines conveyor 55 delivering to the secondary elevator 56 previously described.

The product size material from between the screens overflows a separate compartment of the surge bin 49 to a product conveyor 51 which delivers it to the bulk storage. The surge bin 49 is so arranged





that surplus on-size material is automatically available to supplement the undersize material and maintain the recirculation rate at any value selected by the operator.

A general dust collecting system is provided evacuating from all conveyor transfer points and other points of dust production such as the screens and hammer mill. This system includes a multiple high-efficiency cyclone 57 and exhaust fan 58 discharging to a wet scrubber 46. The recovered dust is delivered to the fines conveyor 55. The scrubbing solution is returned to the "wet system".

## 5. TECHNOLOGY OF THE PROCESS

### A. The "Blunger"

The heart of the process is the so-called "Blunger" which operates according to a fluidization technique. The design of this unit has been developed to meet the unique requirements of the process.

It is a double shaft trough paddle mixer, but beyond this all resemblance to anything conventional ceases. It is a very powerful and rugged unit. The trough or tank and paddle shafts are set at a steep downward slope. The angular paddle blades are interlocking and self-cleaning. It operates at rather high speed and has a large through-put capacity. The tank sides open up to permit easy cleaning. The shafts operate in opposite directions so that the blades descend at the sides and rise intermeshing in the centre.

The novelty is mainly in the operation and what goes on in the machine. The dry salts and granules are fed into the upper part of the machine and between the shafts. The intermeshing blades rising rapidly through this mass tend to keep the solids in very active suspension so that in effect a fluidized bed of granules flows down the centre of the machine.

At the same time the granules overflow the shafts laterally and are shoved into the bottom of the bed by the blades. Thus, there is a continuous turnover from top to bottom as the bed progresses down the sloping tank. The angularity of the blades is not depended upon to convey the material. They provide the agitation to fluidize the bed which then mass flows due to the slope of the unit.

The slurry is fed on to the top of the fluidized bed. The granules as discharged from the Blunger carry 4 to 5% moisture. A thorough appreciation of the unique action in the Blunger is necessary to an understanding of the ability of the process to effect the types of granulation which will be described.

### B. Granulation by Layering and Nucleation

This is the preferred method of operation where the slurry to dry salts ratio, the physical characteristics of the slurry and the drying capacity permit. It can and does produce a spherical, smooth, uniform granule. Conditions are maintained that discourage agglomeration.

The objective is to take the undersize granules, broken oversize fragments and added dry salts crystals, and coat them individually with slurry and to do this over and over again until they reach the desired size and have attained a laminated form or are onionlike in

character. By this gradual building up, starting from a nucleus a dense and much smoother and more spherical finished granule is obtained than by conventional granulating techniques.

This method of operation requires the maintenance of a very large circulating load and a relatively thin slurry. To obtain the desirable smooth round granule, it is necessary to circulate the particles many times through the Blunger and to apply a relatively thin coat each time. If a thick slurry is used and an attempt made to apply a thick coat at each passage through the Blunger, the granules will tend to stick together or gather up small particles and become rough and irregular.

With this type of granulation, the circulating load, i.e. ratio of dry material to the Blunger to solids in the slurry, is usually in the range of 8 to 12:1 and the percentage of solution by volume in the slurry 68 to 70%. There are limitations on the size of added dry salts since too much surface area can not be tolerated without requiring excessive slurry dilution. In a true nucleation process the number of nuclei introduced as salt crystals can not exceed the number of particles in the final product.

#### C. Granulation by Agglomeration and Coating

With certain formulations and where there are limitations on circulating load, drier capacity or salts size, it may be necessary or more desirable to operate under conditions that favor agglomeration.

The objective is then to use a slurry of such a character that it will tend, in the Blunger, to stick together or agglomerate the undersize granules, broken oversize fragments and added dry salt crystals. For this purpose more grinding and finer salts are required. However, by judicious choice of slurry dilution and circulating load, it is possible to coat the agglomerates to produce a granule resembling a seed pod. This tends towards a smoother and more spherical product although never as good as that produced by true layering.

It should be evident that the two operating techniques, layering and agglomeration, represent only the extremes and that in practice both will be operative to varying degrees, depending on the conditions chosen.

This method of operation requires lower circulating loads and a relatively thick slurry. The components must, however, be finer. Circulating loads are usually of the order of 5 to 8:1 and the percentage of solution by volume in the slurry 65 to 68%.

#### D. Preparation of a Proper Slurry

The slurry used is dependent on the sources of phosphates and nitrogen that are most economical for the particular plant location or complex fertilizer to be produced. For instance, the P<sub>2</sub>O<sub>5</sub> may be derived from the treatment of phosphate rock with phosphoric acid to give a superphosphate slurry or the treatment of phosphate rock with nitric acid or the neutralization of phosphoric acid with ammonia. Similarly the nitrogen may be derived from the neutralization of sulphuric acid or nitric acid with ammonia or from ammonium nitrate solutions.

It is useful to develop a triangular diagram of the phosphate-

nitrogen system from which the desired slurry composition can be ascertained corresponding to the N-P ratio and concentration desired. For purposes of illustration, Fig. 1 is a plot of the system ammonium sulfate - monoammonium phosphate - diammonium phosphate. Reading along the sloping solid lines for the desired N-P ratio one can determine the possible composition. The sloping dotted lines give the total N + P<sub>2</sub>O<sub>5</sub> analyses of the mixtures. Thus, if a 1:3 N-P ratio were desired it is seen that a total plant food of 60% is possible at the point of intersection corresponding to a composition of 60% monoammonium phosphate, 30% diammonium phosphate and 10% ammonium sulfate.

Now, having the desired chemical composition it is necessary to determine the amount of water which must be present to give a slurry with the proper granulating characteristics.

The triangular diagram, Fig. 2, is an empirical diagram of the type which can be developed for this purpose. This is for the same system as Fig. 1.

The curves show the amount of water required to obtain a slurry having the required volume ratio of solution to solids. It is of course, essentially, a three component solubility diagram. Having the necessary water content of the slurry, and by arithmetical computation for the particular formulation, the amount of slurry solids, it is possible to compute the required drier evaporative capacity.

It is necessary to learn considerably on experience in the light of the relative quantities of dry system salts and slurry to determine the type of granulation technique to be recommended and the optimum recirculation ratio.

The optimum slurry solution to solids volume ratio is also dependent on many other factors which must be weighed such as impurities in the acid, ammoniation procedures and temperatures, source of ammonium sulfate, etc.

It is apparent from this discussion that the use, for producing the slurry, of highly soluble materials e.g. ammonium nitrate, ammonium phosphate and ammonium sulfate or combinations is more advantageous than use of less soluble or insoluble salts such as dicalcium phosphate.

#### E. Producing Diammonium Phosphate

One corollary to the development of this process has been a practical method of producing a stable diammonium phosphate from wet process phosphoric acid. Heretofore, it has been considered necessary to filter off the impurities precipitated on neutralization, to saturate at low temperature to minimize ammonia losses, to crystallize the di salt and to dry at low temperature. This is a cumbersome and expensive operation and the diammonium phosphate produced is relatively unstable at elevated temperatures and humidities.

The method of producing diammonium phosphate which has been developed will be clearer if the characteristics of the ammonium phosphate salts and their solutions are studied.

Figure 3 gives the partial pressure of ammonia in the vapors over solutions of ammonium phosphate of varying mole ratios. It will be noted that the partial pressure increases by 10 times between a

1.43 and a 1.78 mole ratio.

Figure 4 is a phase diagram for the system  $\text{NH}_3\text{-H}_3\text{PO}_4\text{-H}_2\text{O}$  at varying temperatures. The mono salt, line O-B, has the lowest solubility. The di salt, line O-A, is considerably more soluble. Maximum solubilities are obtained with more or less equal weight percentages of the two salts at point C. Within the limits of triangle ABC three phases are present, solid diammonium phosphate, solid monoammonium phosphate and a mother liquor having the composition indicated at the triple point C. As long as the slurry composition remains within the triangle the composition of the solution (at  $75^\circ\text{C}$ ) will be:-

$$\text{Mol. ratio of } \text{NH}_3/\text{H}_3\text{PO}_4 = \frac{15/17}{60/98} = 1.43$$

This ratio is largely independent of temperature. Upon moving out of this triangle into ACD the mono salt will disappear and the solution composition will increase in ammonia content by moving along the line C-D.

Figure 5 shows the approximate characteristics of the system  $\text{NH}_3\text{-H}_3\text{PO}_4\text{-H}_2\text{O}$  at the "triple point" or where the solution  $\text{NH}_3\text{-H}_3\text{PO}_4$  ratio is about 1.41 to 1.45.

It has been found that as long as there is monoammonium phosphate present to the extent of about 20% (a mole ratio of 1.8:1.0) the ammonia losses during drying, if the material temperature is kept under 85 to  $90^\circ\text{C}$ , are not significant. The ammonia evolution during drying follows the curve for partial pressure (right hand) in Fig. 5.

The neutralization is carried to monoammonium phosphate or slightly above in a first stage. The ammonia carried off in the water evaporated is unappreciable as will be seen from Fig. 3. The neutralization to 80% diammonium phosphate is effected in a second stage where the composition of solution is maintained approximately at or near the "triple point".

Also, since maximum solubility is obtained at the "triple point", a slurry can be obtained with minimum water content.

This method obviates any necessity for filtering or separately handling the precipitated iron and aluminium phosphates and other impurities which actually assist the granulation. No crystallization is required and the diammonium phosphate is obtained in granular form either alone or as a constituent of complexes.

#### F. Control

An outstanding feature of the process and its commercial application is its controlability. This is a serious drawback in many conventional granulating plants. A great deal of thought has been given to instrumentation, and while the process does not lend itself to full automation, it does require a minimum of operating labour.

The formulation and maintenance of product grade within close tolerances is possible with the accurate feed controllers provided for the basic ingredients. The "wet system", in the case of ammonium phosphates, is controlled by periodic titration to hold the desired N-P mole ratio and regular moisture determination to maintain the slurry characteristics for optimum granulation.

The basic control of the "dry system" is at the Blunger discharge. Here a certain amount of art and "feel" is required, but this is something that can be learned by a reasonably intelligent operator in a short time. By observing the wet granules leaving the Blunger, the operator can adjust the rate of recirculation, production of nucleoli (amount pulverized), drying conditions and slurry dilution to maintain uniform and optimum conditions. All adjustments are by remote control and the operator has these at his finger tips, together with indicating and recording instruments conveniently accessible on a graphic panel board. The surge bins for recirculated undersize and oversize are necessary to provide the flexibility and lag which are essential if adjustments for unusual conditions are to be possible without the system spiralling out of control. Since the process is cyclic and large flows are used, a very complete system of electrical interlocks is essential.

Control of the drying is very important. It has been found that if the drier exit gases are maintained at low relative humidities or partial pressures of water vapor, most of the products can be dried down to 0.3% moisture or less, and that such products will exhibit little, if any, tendency to pile or bag set.

All vented gases are wet scrubbed and in most cases the scrubber liquors are returned to the process so that appreciable losses are practically impossible. The scrubbing system is nearly automatic, the only operating control required is to insure that sufficient water is returned to the wet system to carry the recovered solids at a concentration not exceeding about 40%.

Equipment which is closed and vented through a dust collection system will insure a clean, dust- and fume-free plant.

#### G. Variations

The process has been used to date particularly to produce granular triple superphosphate, ammonium phosphates or phosphate-sulfates and complex fertilizers based on ammonium phosphates.

One plant has experimentally produced complete complex fertilizers based on triple superphosphate slurry.

If high nitrogen or higher analysis products are wanted, there appears to be no reason why ammonium nitrate can not be introduced into the "wet system" or urea into the "dry system".

Also, if the use of nitric acid appears economically attractive, laboratory tests have shown that phosphate rock can be attacked with nitric and sulfuric or phosphoric acid and the resulting slurry introduced into the reaction tanks (14A, 14B and 14C) for neutralization with ammonia.

The important point is that the granulation in the "dry system" is entirely independent of the reaction occurring in the "wet system" provided a slurry can be produced with the proper ratio of solution volume to solids volume to ensure effective blunging. This can be accomplished over a wide range. Pilot plant tests have indicated that almost any soluble salt, for instance ammonium sulfate, potassium chloride or sodium nitrate, can be granulated by this technique.

#### 6. INSTALLATIONS

The basic concepts of this process, originally developed by

M. Giorgini and H. Tiedemann (Canadian patent N° 350714, Gorman patent N° 627273 and others) were first applied in 1931 for the production of granular monoammonium phosphate at the plant of Consolidated Mining & Smelting Co. of Canada at Trail, British Columbia. Shortly afterwards the Hellenic Company of Chemical Products & Fertilizers Ltd. installed at Piraeus a similar plant for the production of granular triple superphosphate, which functioned for almost 20 years until it was replaced in 1951 by a new plant designed after the same basic concepts, but obviously taking into consideration the experience gathered in other plants of the same type built in the meantime in the United States, Japan, India and other countries. Large plants of the same type to produce granular triple superphosphate were installed, amongst others, by Pisons' Ltd. at Immingham, U.K., and Davison Chemical Co. at Bartow, Florida, U.S.A.

The first plant operating the process as described in this paper to produce complex fertilizers and also diammonium phosphate was built at Joplin, Missouri, U.S.A., for the Missouri Farmer's Association (now Farmers' Chemical Co.) in 1955.

Since then, similar plants to produce complete granular fertilizers have been installed at Loith, Scotland, for Scottish Agricultural Industries Ltd., for Imperial Chemical Industries at Billingham, U.K., and for two fertilizer factories in Japan.

#### 7. ACKNOWLEDGMENTS

The authors wish to thank the International Superphosphate Manufacturers' Association for the opportunity to present this paper and to outline a new approach to the common problem of making high-grade fertilizer at the least cost.

The assistance of our associates, William A. Lutz and Christopher J. Pratt, without whose valuable help the paper could not have been prepared, and permission by the Management of Dorr-Oliver to publish the data, are similarly appreciated.

Fig. 2

SYSTEM AMMONIUM SULFATE-PHOSPHATE

Required slurry moistures

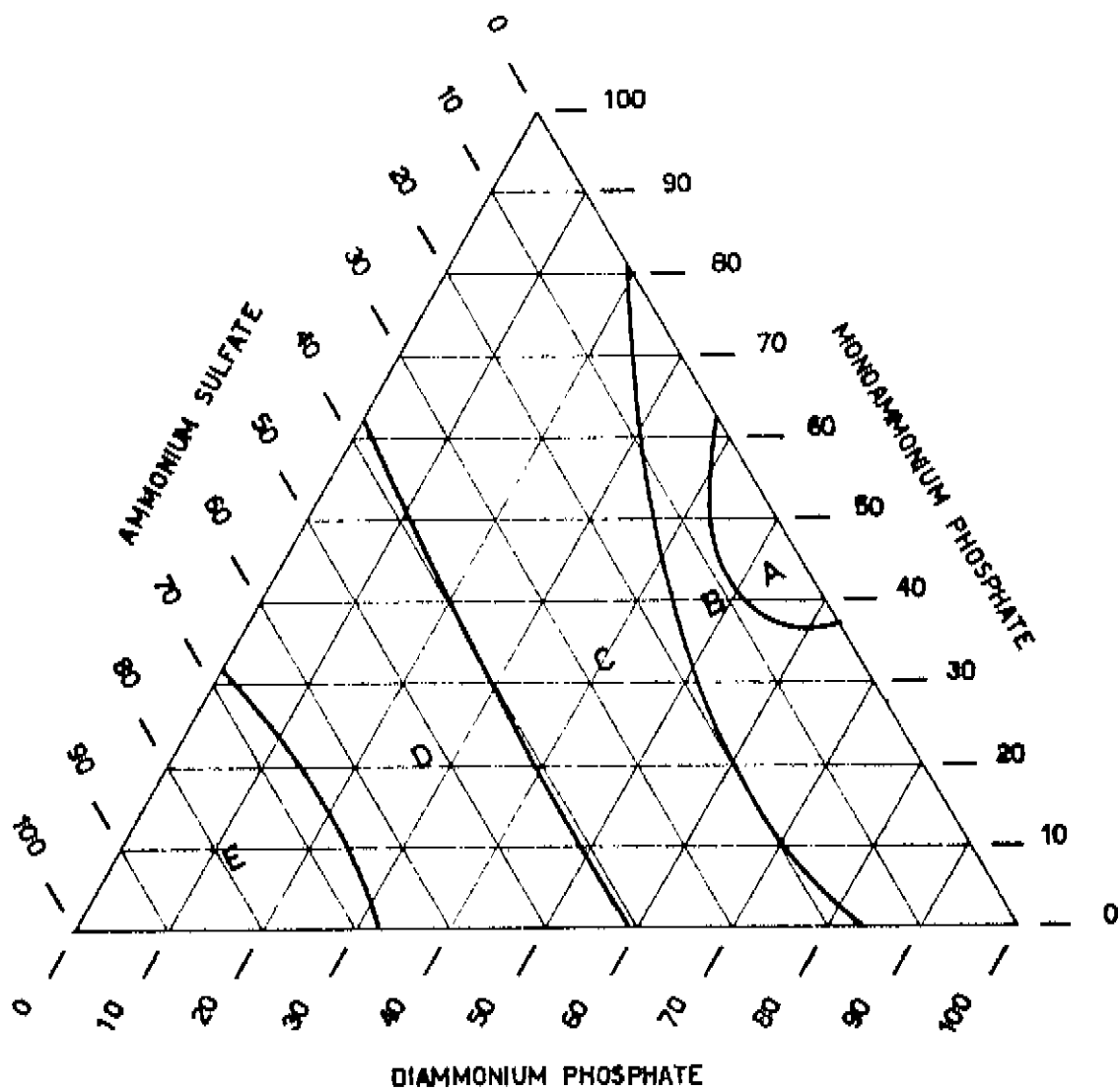
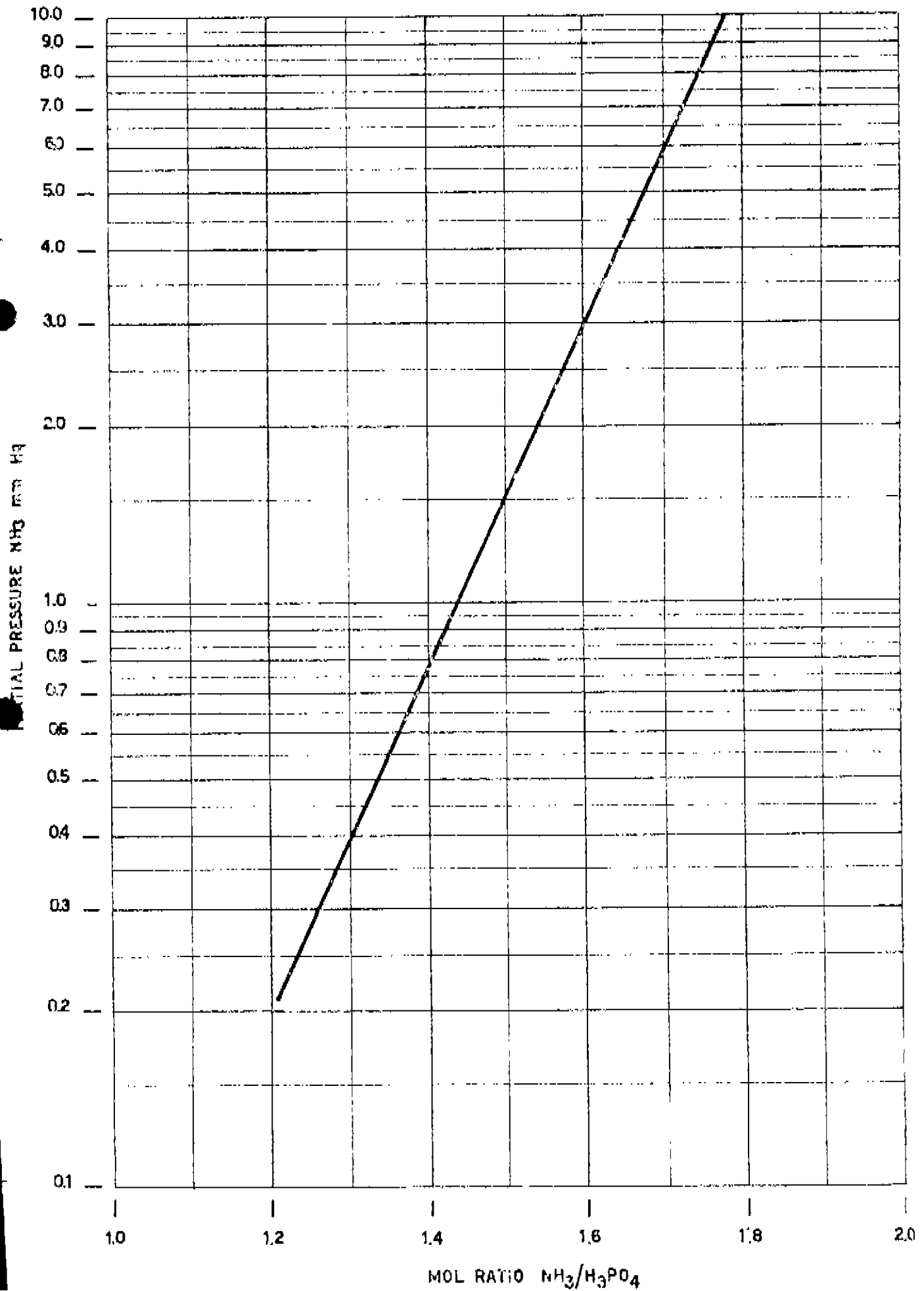




Fig. 3

PARTIAL PRESSURE OF AMMONIA OVER SATURATED  
SOLUTIONS 75°C



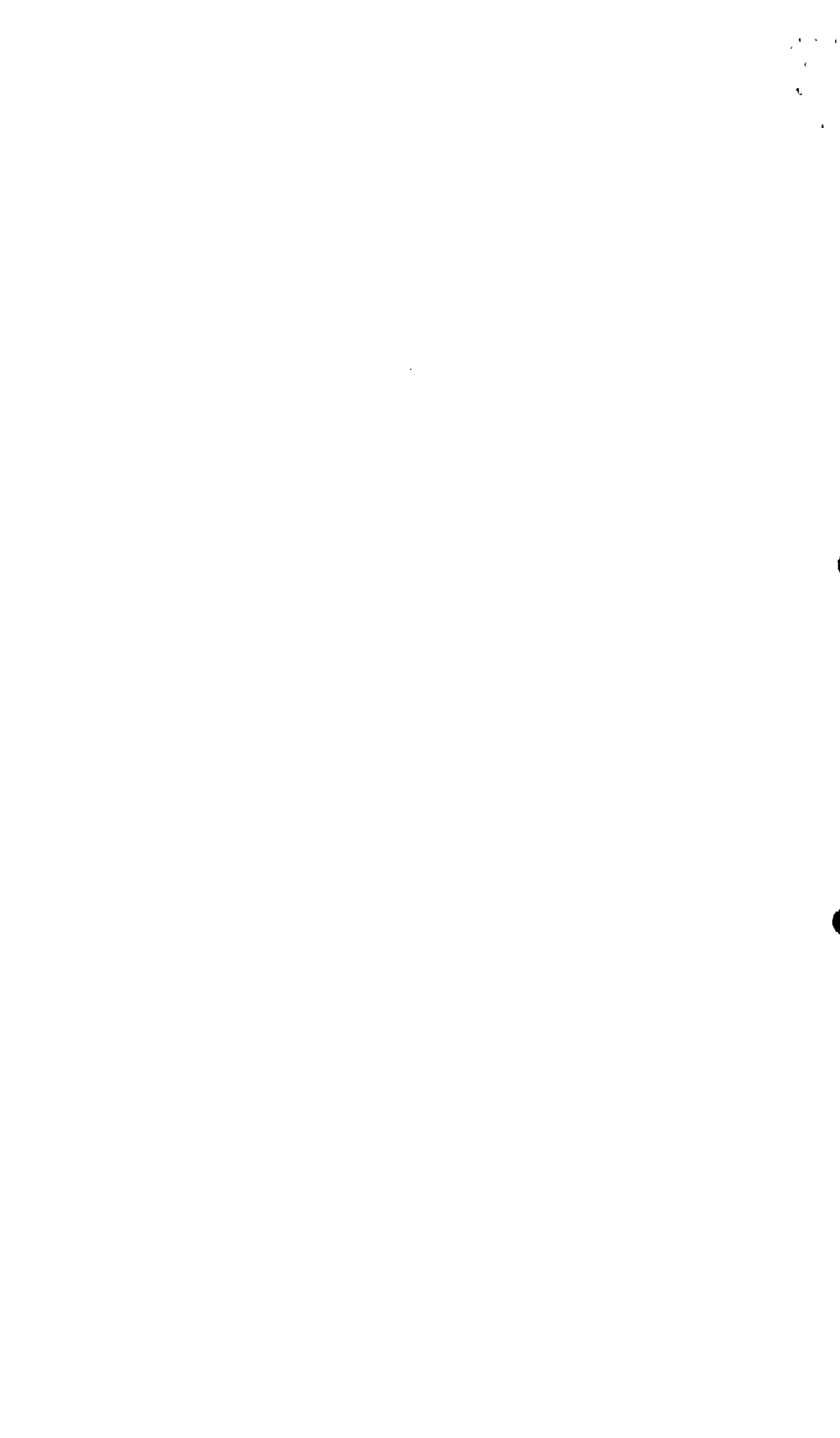


FIG. IV

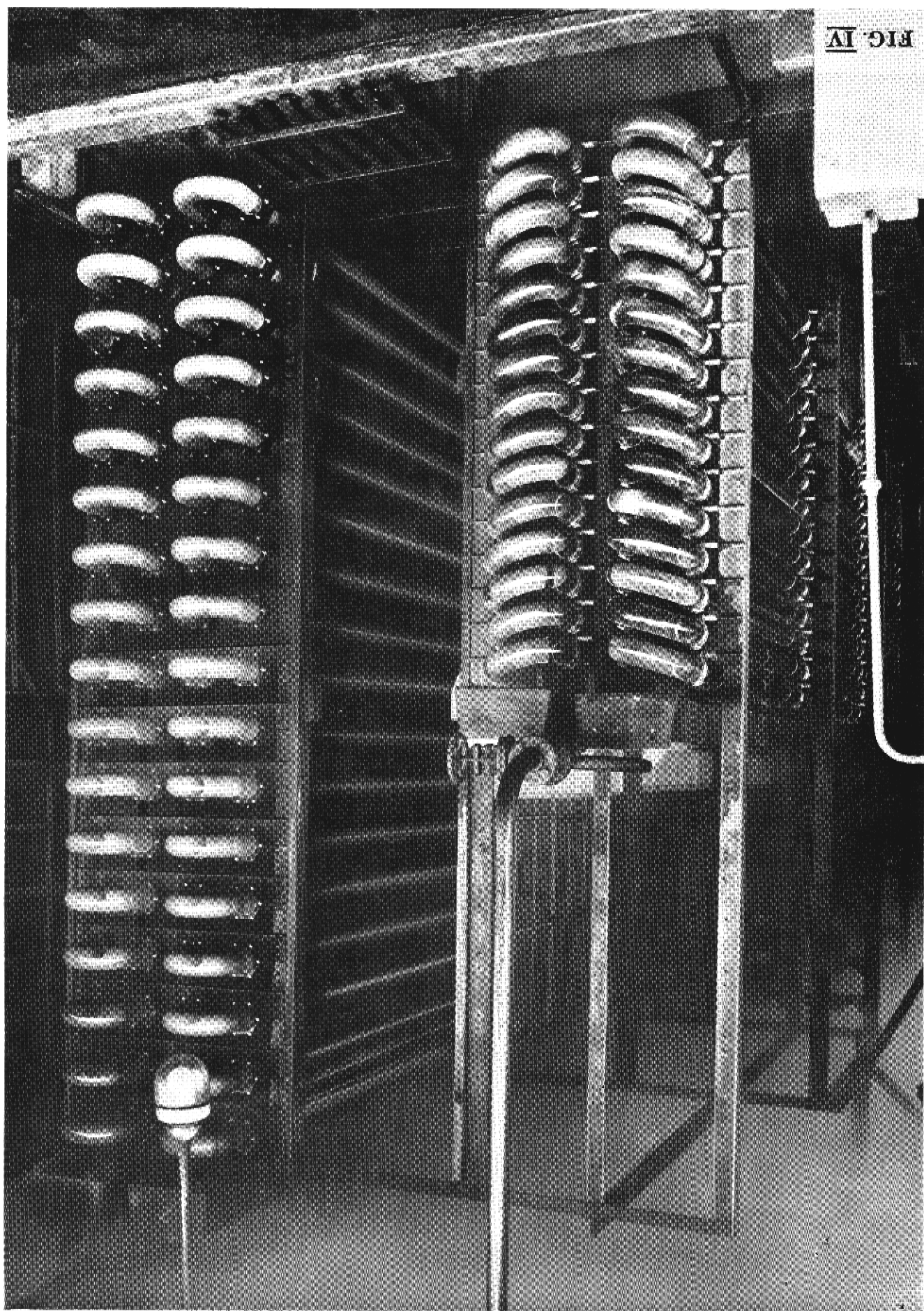
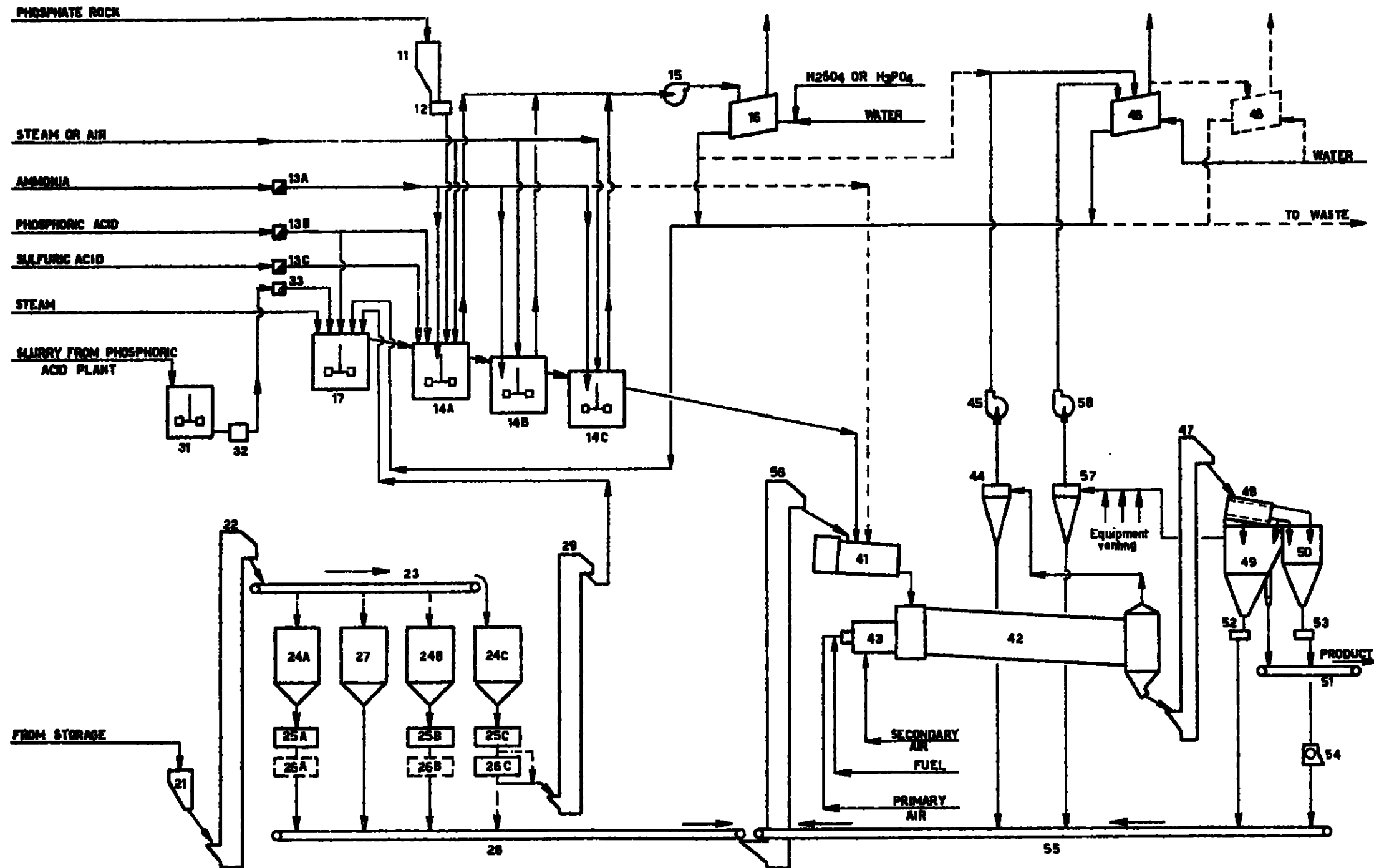


Fig. 6

THE DORR-OLIVER  
GRANULAR FERTILIZER PROCESS  
TYPICAL FLOWSHEET



- 11 PHOSPHATE ROCK BIN
- 12 GRAVIMETRIC FEEDER
- 13ABC FLOW CONTROLLERS
- 14ABC REACTION AGITATORS
- 15 FUME EXHAUST FAN
- 16 SCRUBBER
- 17 DISSOLVING AGITATOR
- 21 FEED HOPPER
- 22 RAW MATERIAL ELEVATOR
- 23 RAW MATERIAL CONVEYOR N°1
- 24A DRY FILLER BIN
- 24B POTASH BIN
- 24C AMMONIUM SULFATE BIN
- 25ABC RAW MATERIAL FEEDERS
- 26AB RAW MATERIAL MILLS
- 27 SALTS BIN
- 28 RAW MATERIAL CONVEYOR N°2
- 31 FILLER SLURRY STORAGE
- 32 FILLER PUMP
- 33 FILLER FLOW CONTROLLER
- 41 BLUNGER
- 42 DRIER
- 43 COMBUSTION CHAMBER
- 44 DRIER CYCLONE
- 45 DRIER FAN
- 46 SCRUBBER
- 47 PRIMARY ELEVATOR
- 48 VIBRATING SCREENS
- 49 FINES BIN
- 50 OVERSIZE BIN
- 51 PRODUCT CONVEYOR
- 52 FINES FEEDER
- 53 OVERSIZE FEEDER
- 54 PULVERIZER
- 55 FINES CONVEYOR
- 56 SECONDARY ELEVATOR
- 57 DUST CYCLONE
- 58 DUST FAN

Fig. 4

SYSTEM  $\text{NH}_3 - \text{H}_3\text{PO}_4 - \text{H}_2\text{O}$

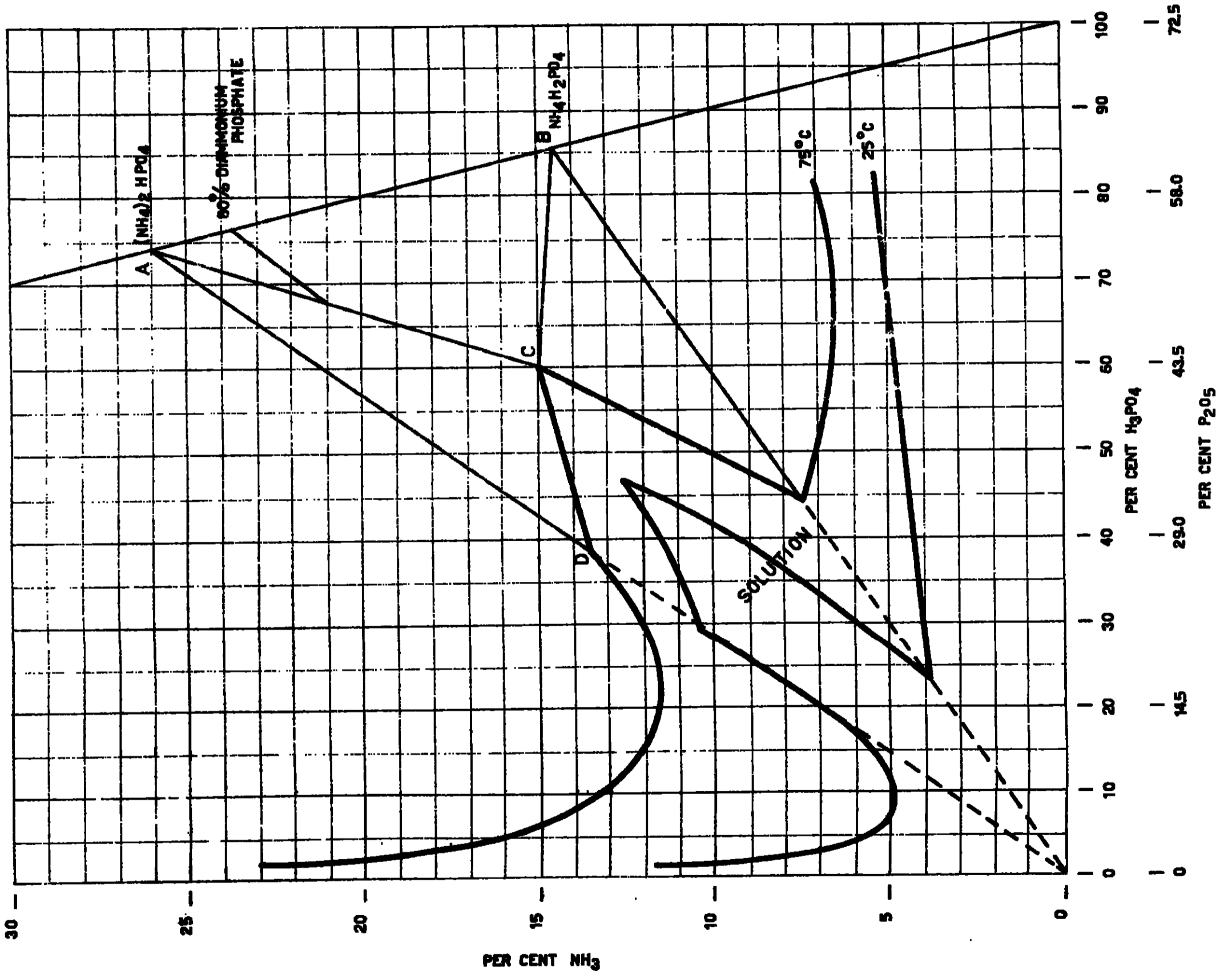


Fig. 5

APPROXIMATE CHARACTERISTICS  
OF SYSTEM NH<sub>3</sub> H<sub>3</sub>PO<sub>4</sub> H<sub>2</sub>O

When NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>, (NH<sub>4</sub>)<sub>2</sub> HPO<sub>4</sub> &  
solution are all present

