PROCEEDINGS
OF THE
14th ANNUAL MEETING
FERTILIZER INDUSTRY ROUND TABLE
1964

Held at the
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Washington, D. C.
November 11-12-13, 1964

Executive Committee

Vincent Sauchelli, Chairman
Housden L. Marshall, Sec'ty-Treasurer
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Executive Committee

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Fertilizer Industry Round Table

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Wednesday Morning Session, Nov. 11, 1964

The Fourteenth Annual Meeting of the Fertilizer Industry Round Table.

Chairman Vincent Sauchelli presiding.

CHAIRMAN SAUCHELLI: Welcome to this our 14th annual meeting of the Round Table. It is always a keen pleasure to greet you on this occasion. We are particularly glad to see among our older associates a good number of new and younger men.

The aim and purpose of these Round Table sessions is to promote the best interests of our industry and in particular of those men in it who are directly engaged in the manufacturing function, the plant superintendents, the quality control staff, the foremen, the materials handling and the maintenance personnel.

These sessions provide a forum for this particular section of the industry. A frank, open discussion of common problems, by all in attendance, has been a feature of our meetings and this we encourage in every way we can.

A characteristic of top-ranking personnel is that they want to know why things happen as they do. These men want programs that tell them not only what to do, but also why.

Such persons, of course, belong to the group of better-than-average operators and they are not necessarily limited to the large complex type of manufacturing plant. The size of the plant has nothing to do with it. They are for the most part the younger men who are quicker to adopt new practices, who are less burdened with traditional procedures.

Your Executive Committee has been motivated to provide a program which emphasizes the why of operations. It used to be that the average superintendent in a typical old type fertilizer plant operated according to rule-of-thumb procedures which he acquired through some sort of apprenticeship. Very few of them had better than a high school education.

In modern complex manufacturing facilities, however, most of the responsible operating personnel have had an education in chemical engineering and many in economics and management. They are able to understand the chemistry and physics involved and want explanations based on these sciences.

They have to deal with a more sophisticated technology and their decisions require an understanding of the scientific principles. Today's managerial personnel must have not only a deeper knowledge of the newer technology, but in many organizations they must be oriented to the broader requirements of the market.

The ability to organize production by itself is not enough. Manufacturing now is required to fit the market requirements and plant managers are spending more time now with product development and marketing men.

What I refer to is for example, standardizing granule shape and size, or maintaining a range in size of materials to satisfy the needs of the local bulk blender, or concentrating on granular diammonium phosphate rather than on triple super.

In other words, the modern top ranking production man must work even closer with the new product development and sales organizations. As is true of other branches of the chemical industry so it is becoming increasingly apparent in our fertilizer industry that marketing orientation means first finding a market and then building a product to fit it, rather than making a product and then trying to find a market for it.

No one can doubt that we shall experience many technological innovations in the coming years. Ours is a dynamic industry. Change is its motto. The operating staff needs to keep abreast of these developments in technology and managerial skills and be able to interpret and adapt the new knowledge to its particular operations. To be able to do this becomes a personal satisfaction of knowing why.

We need to look back only a decade or so to manufacturing practices to realize how far we have come in our industry and to appreciate its dynamic quality. I am reminded of a conversation reported between a visitor to this capitol and the proverbial sage taxi driver.

On passing the National Archives Building, the visitor noted and was puzzled by an inscription which is meant to indicate, the past is but prelude. Asking the driver for an explanation, he got the quick answer. "It means you ain't seen nuthin yet." We, too, in this amazing industry of ours are just about in the prelude stage of what is bound to be an exciting, rewarding period of change and progress. So much for introduction.

Let me throw the spotlight on our agenda. With your splendid cooperation we of your executive committee have put together a program we feel will prove worth your time, effort and expense to be with us. There is something here for everyone.

As promised you. at last year's meeting, we have scheduled two popular sessions as questions and answer periods. They are the open forum on the program. one for this afternoon and one for tomorrow afternoon. A monitor was selected for each forum and he was asked to select a panel that would initiate the discussion on a set of questions you sent in for this purpose.

Frank Nielsson monitors today's forum. Elmer Perrine will monitor tomorrow's. You all know these leaders and you can be sure of a most interesting session. Don't miss them. We expect a lively give and take interchange of experience from the floor. The old-time Round Table tradition. No tape recording will be made of these open forum sessions. A summary only of the high points of each forum will be prepared by each monitor for the proceedings, but without names.

Friday's session will be a spendid symposium on orthophosphoric acid. Al Phillips of the staff of the TVA Research Center will monitor it. Al is particularly qualified in this field. He has organized an excellent program. The production and use of wet process acid is currently one of the most important developments in our industry. All of you will want to hear and participate in this symposium. I am as anxious as you to get the program started. Before we get to the meeting proper I have a couple of notices here. I'm going to ask Dr. Marshall, our Secretary-Treasurer, to make a statement.

Dr. Housden L. Marshall: Your Secretary has been rather active in sending out all your proceedings, notices, etc. We have been trying to keep up with supplying the back numbers, and we have a little bargain sale. We have 18 sets, 18 only, for 1958 through '63

The reason there are only 18 is because there are only 18 copies of the 1960 proceedings existing, therefore we can only give you 18 complete sets. First come, first serve, and when they are gone that is the end of the story as far as a complete set of proceedings is concerned.

We have talked to you folks about membership lists which is the list of people attending this meeting, which will be mailed to you in late December. Your proceedings, a part of your registration fee, will be mailed to you when we complete editing and printng around the first of April, 1965. Remember this is a love labor and the executive committee is working on it on weekends and nights, so we are not in a position to really expedite it as swiftly as we would like to, but we'll get it out. We don't waste any time.

I wish to thank most of you for giving us good addresses last year. I hope you repeat it this year. Our return mail was rather low for the number that we have. Thank you.

Moderator Sauchelli: Now to get back to our program.

Corrosion is one of the big problems facing every phase of our industry. The losses caused by corrosion run into hundreds of thousands of dollars annually.

Our first speaker, representing one of the major companies in our country, will discuss a new problem of combating corrosion. Today so much is concerned with plastics. It is interesting to see that plastics also has been applied to the problem of corrosion. It is a great pleasure to call on Harvey E. Atkinson, of E. I. du Pont de Nemours Company, to give his subject on Plastics to Combat Corrosion.

Mr. Harvey E. Atkinson: Thank you, Mr. Chairman. Ladies and gentlemen of the Fertilizer Industry Round Table, it is a pleasure to be here.

Plastics To Combat Corrosion

Harvey E. Atkinson

PLASTICS as engineering materials have accounted for several million dollars cost reduction for Du Pont in the past five years.

The key to savings, by Du Pont or any user of plastics, lies in recognition that plastics are a family of new engineering materials; that successful use of plastics involves learning a new technology in the field of materials engineering. There are:

New *criteria* of mechanical behavior. Mechanical properties of all plastics are time dependent, even at room temperature. Under loads that are small compared with indicated shorttime strength, plastics will deform, continue to creep, and eventually fail. The creep and stress-rupture behavior is temperature dependent, and changes in magnitude with relatively small temperature changes.

New *criteria* of chemical resistance. Plastics do not corrode as do metals according to the classical electrochemical process. A corrosion rate cannot be calculated on the basis of weight changes. Plastics degrade while maintaining their shape and appearance. Chemical resistance must be determined by measuring changes in strength, hardness, and dimensions over periods of six months to a year to establish trend and rate of change.

This behavior, both mechanical and chemical, introduces problems in equipment design that are unfamiliar to many design engineers. It is in this respect that the materials engineer must work closely with designers. Design also must accommodate methods of fabrication and assembly that are peculiar to plastics.

Purchasing also can present problems, particularly in fabricated equipment. There are as yet no industry standards nor national codes governing tanks, for example, and other custom fabricated items. The user must prepare detailed specifications if he is to obtain competitive bids. Without specifications, bids are not competitive, since they represent only the individual bidder's idea of what is needed.

Despite these different *criteria* of mechanical behavior and chemical resistance, new *problems* in design, new fabrication techniques, and more stringent requirements for purchasing, learning how to use plastics is a very profitable undertaking. One word of caution, however, as stated by the editors of "Modern Plastics," " . . . the plastics world is inhabited by a breed who become over-excited about every new material and, despite all previous lessons, seem to forget that it usually takes six or seven years for any important material to become well established." Major applications should be restricted to those plastics having the highest degree of engineering development.

These materials are:

Thermoplastics:

Polyethylene	PE
Poly (vinyl chloride)	PVC
Acrylonitrile-butadiene-styrene	ABS
Fluorocarbons	TFE and FEP

Reinforced Thermoset Plastics:

Polyester—Glass	PES
Epoxy—Glass	EP
Phenolic—Asbestos	PH
Furan—Glass	F

Sufficient engineering data are available on these materials to satisfy the needs of the materials engineer. The data are scattered, however, and require search of technical literature, review of ASTM and Commercial Standards, and consultation with manufactures.

These plastic materials find their major uses in the areas shown on the chart.

Major End Uses of Plastcs

Materials	Piping Systems Pressure	Piping Systems DWV	Sewer	Vent Systems	Tanks and Equipment
PE	X	X			
ABS	X	X			
PVC (Type I, Grade 1)	X	X	(D)	(S)	(S)
TFE and FEP (Linings)	X				(D)
Reinforced Plastics:					
PES	X		X	X	X
EP	X		X		
PH	X				(S)
F		X		X	X

(S) Specialty uses
(D) Major development in progress

With this outline in mind, let us illustrate, by typical example, how economic use of plastics has been accomplished.

Piping Systems

Piping systems are of major interest because they represent about 25 to 33 per cent of total investment in a process plant. An intricate system involving about 5000 feet of pipe with hundreds of fittings was installed with Type I PVC at a cost $40,000 less than conventional piping alloys (Figure 1).

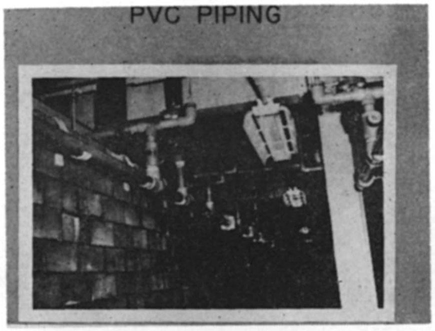


Figure 1.

The performance of PVC has been superior to that of type 316 S/S and higher alloys, a saving both in investment and in maintenance costs.

A 10 in. diameter PVC line, designed for specific wall thickness and purchased as a special extrusion, was installed to handle waste sulfuric acid. This line is over two miles long and cost $60,000 less than rubber-lined steel, the alternate material. In three years of operation, it has been trouble free compared with considerable leakage experienced with a wood stave line formerly used.

Today many plants use PVC routinely for mineral acid and salt services, particularly.

For exposure involving organic solvents or temperatures above about 150°F., the reinforced epoxy resin or phenolic-asbestos piping materials come into the picture. In one application epoxy-glass is used for vent lines to withstand chlorinated solvents, HCl, and high temperature. The alternates would be "Teflon"® lined or glass-lined at much higher cost.

Steam traced epoxy-glass is used to handle acid slurry at temperatures and pressures above those suitable for PVC. The reinforced epoxy pipe in one system saved 8 to 10 thousand dollars over rubber-lined steel. It is performing well after 4 years' service.

Pipe lined with "Teflon" TFE has saved as much as $700,000 annually in service involving HCl and chlorinated hydrocarbons at temperatures up to 300°F and pressure of 100 psi. No other piping material would provide economic service under such process conditions.

"Teflon" FEP also is moving into the lined piping picture. The fluorinated ethylene propylene lined piping is slightly lower in cost than TFE. A test installation of 2 in. pipe has handled about 36 MM lb. of 70% sulfuric acid at a temperature of 250°F in 14 months with no sign of attack.

An FEP lined and covered dip pipe has been in use over a year feeding a 50:30 sulfuric-nitric acid mixture at about 195°F. Externally the dip pipe is exposed to concentrated HNO_3 vapors at 230°F. There has been no evidence of attack in this service where previously glass lined steel was required and whose performance was erratic.

Valves also are an important part of piping systems. Ball valves are available completely lined with "Teflon"—either TFE or FEP. They have provided reliable service in extremes of chemical service.

"Teflon" TFE also upgrades performance of other valves such as alloy plug valves. A molded sleeve of "Teflon" around the plug eliminates need for lubrication and provides a tight seal even when wear or corrosion occurs on the plug. The cost premium of this increase in valve reliability is small.

No discussion of piping systems would be complete without reference to TFE unsintered tape as a thread sealant. This thread "dope" provides almost foolproof sealing of any threaded joint, and maintains its lubricity such that the joint can be unscrewed at any time. Contamination of pipe lines with conventional thread dopes is eliminated along with the cans, brushes, and rags that are a housekeeping and personal cleanliness problem.

A typical example of performance was an installation of steam and condensate piping involving 6000 screwed joints in the size range ½ in. to 2 in. The system was put on stream without a single leak, something never before accomplished with ordinary thread dopes.

Piping Auxiliaries

There are certain auxiliaries for conveying chemicals that are not classified as pressure piping, but which are important items in certain chemical operations.

First is flexible spiral tubing, used, for example, to isolate weigh tanks from pumps and from reaction vessels.

This unarmored tubing is for low pressure application. It is more economical in these applications than swing joints or other flexible connectors.

A different type of flexible conductor is chemical transfer hose. This, in essence, is an industrial rubber hose having a liner of "Teflon." It is suitable for 150 psi service at temperatures to approximately 300°F. Its utility lies in the fact that a single hose can handle any chemical. Tank truck, tank car and barge unloading are practical uses of this multi-purpose hose.

Metal overbraided hose is available in diameters to 1¼ in. With metal overbraid the full temperature potential of TFE becomes available in a flexible conduit. This is particularly useful in the case of need for flexible steam lines, hot oil lines, and the like. But one precaution! These hoses should not be used to handle liquid chlorine, which is under high pressure. Permeation may cause failure of the stainless steel overbraid with resultant pressure failure of the "Teflon" hose.

In all hose applications, particular attention must be paid to end connections. Overbraided hose usually has a metal connector that comes in contact with the fluid being handled. Chemical transfer hoses have flanged ends with the TFE liner flared over the flange face such that only TFE is in contact with the fluid. Where frequent connect and disconnect operations are encountered, it is advisable to install a replaceable adaptor on the hose end so that damage to the gasket face does not require replacement of the entire hose. It is such details as this that make the difference between economic success and failure in use of TFE lined flexible conduits.

Drain, Waste, Vent (DWV)

In gravity piping systems, polyethylene is becoming important for laboratory drainage. Polyethylene systems, including cup sinks and traps, can be installed at considerably lower cost than conventional silicon iron. Light weight and freedom from breakage contribute much to the low cost.

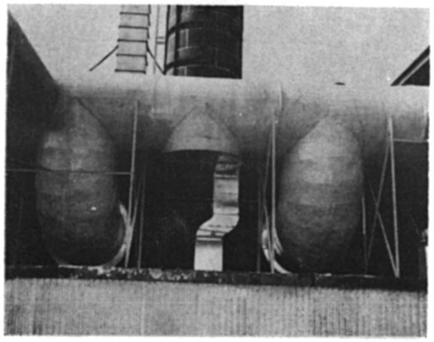
For floor drains, where corrosion is a problem, PVC or ABS are available with drainage type fittings. For severe acid conditions, heavy weight Schedule 80 systems are desirable. For mildly corrosive conditions, Schedule 40 systems are adequate. The latter are lower cost installed than bell and spigot cast iron systems.

The cemented socket joint with PVC and the light weight which gives ease of handling are major factors in the economy of such systems. These same techniques are now being applied to underground sewer systems.

Ventilating Systems

In the field of ventilating systems, plastics first entered the picture with PVC. Today, however, the reinforced polyesters, have relegated PVC to a minor role. This is because of broader range chemical resistance, higher temperature limits, better mechanical properties, and lower cost than PVC. Actually, reinforced plastics are the most important materials for custom fabricated equipment of any type.

Figure 2.



The polyester-glass fume system of Figure 2 measuring up to 9 ft. diameter in part has been maintenance free for more than 7 years handling vapors of hot dilute sulfuric acid. It cost less than Type 316 S/S which has a *total* life expectancy of five years.

A 20,000 cfm polyester-glass fan is in service exhausting steam and ammonia fumes. No signs of deterioration are evident after 4 years whereas steel fans lasted only 18 months. The purchase price was twice that of carbon steel, but the extended service life makes the installation economical.

The 160 ft. high glass-reinforced resin stack of Figure 3 has been in operation 4 years with every indication of a ten year minimum life. It handles chlorinated solvents, ammonia, and steam. The steel stack it replaced had two years life.

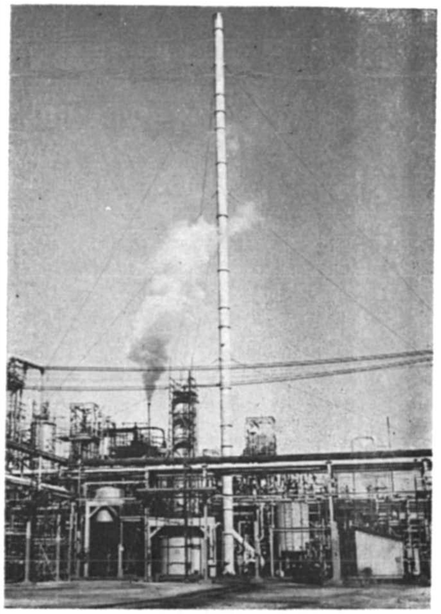


Figure 3.

An installation of polyester-glass skimmer hoods, fan, and 70 ft. stack exhausts hot acid fumes from pit tanks. Installed cost was $17,000 less than for Type 316 S/S primarily because light weight of the plastic minimized supporting steel and foundation requirements. Now about 2 years old, it shows no attack.

Lest we be lulled into a false sense of security, however, by these examples of savings through use of reinforced plastics; take a moment to look at factors affecting performance of this family of materials.

Resin-glass laminates are not homogeneous materials like the thermoplastics. The resin component provides chemical resistance. Glass fibers enhance mechanical properties, but detract from chemical resistance in varying degree depending on environment. To illustrate the point, consider the following results from laboratory tests of laminates exposed in 25% sulfuric acid at 212°F.

Plotting flexural strength versus exposure time as one measure of chemical resistance, Figure 4, an epoxy resin and a polyester resin lost strength only moderately when laminated with about 20% glass in the form of chopped strand mat (laminated 1 and 2). The polyester and epoxy have equal satisfactory performance.

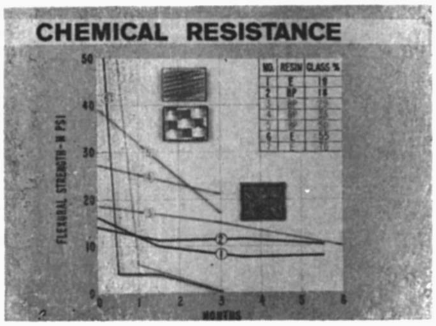


Figure 4.

As glass content in the form of chopped strand mat is increased progressively to 50% in laminates 3, 4, and 5, initial strength increases, but rate and magnitude of attack as measured by strength loss also increases.

If glass *form* is changed to large woven roving continuous strands, laminate 6, with glass content still in the 50% range, initial strength is doubled, but the laminate fails rapidly—complete loss of strength in 3 months.

The situation becomes progressively worse with filament wound structures, laminate 7, having 70% or more glass content.

With this knowledge, the highly chemical resistance mat laminates are combined with the high strength roving laminates to obtain a structure suitable from both standpoints — chemical resistance and strength, Figure 4.

The principles demonstrated by these tests have been borne out in actual practice. In relatively mild chemical service, tanks fabricated as laminate 6 have given erratic service life. Equipment fabricated in accordance with the laminating principle illustrated in Figure 5 has been reliable. These principles are incorporated in the draft industry standards now being considered by the Corrosion Resistant Structures group of the Reinforced Plastics Division of the Society of the Plastics Industry (S.P.I.).

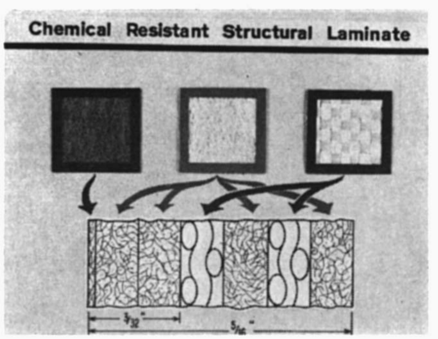


Figure 5.

It is common practice today, having developed this understanding of materials behavior, to use reinforced plastic equipment for agressive chemical service. The process tank of Figure 6, with complicated internals for coil supports, spargers, and dip legs—as well as baffles — has been used over five years in dilute acid slurry service. Its cost was approximately 50% that of rubber-lined steel. Similar process tanks and large storage tanks—up to 40,000 gallon capacity —are finding economic application in services formerly requiring stainless steel, rubber-lined steel, or brick-lined steel.

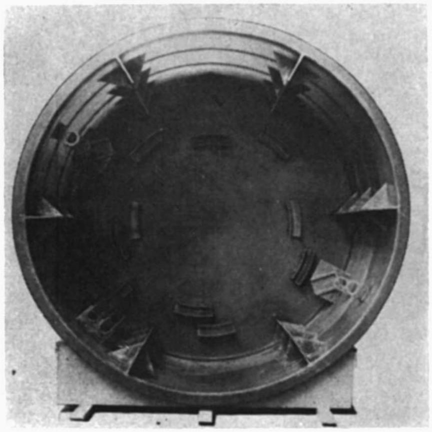


Figure 6.

Illustrations of plastics usage are almost endless, but the few given in this paper point up the general requirements to obtain satisfactory service. The areas where specific plastics find their greatest economic utility have been indicated. It is our conclusion that plastics properly engineered, can play a major role in reducing costs where corrosion control is a problem.

CHAIRMAN SAUCHELLI: Thank you Mr. Atkinson for a very informative talk. This discussion should stir up some questions. Who has the first question?

MEMBER: Mr. Atkinson, I think you should point out in the fertilizer industry the chief corrosion problem comes from fluorine and when we design plastic laminate structures we first have to protect the glass fiber from fluorine.

MR. ATKINSON: Yes, certainly you have fluorides in all parts of your industry, although I'm sure there must be plenty of areas where you are not combating fluorides. However, if you are combating fluorides, and want to use the reinforced plastic materials, we usually replace the inner layer of the lining, the glass reinforcement is replaced with the thermoplastic type, such as Dynel or something of this kind which is resistant in its own right to the fluoride component of the system.

MR. EVERETT MORTENSON: Is there any evidence or reason to think that the deterioration of the polyester plastics is disappearing over a period of time?

MR. ATKINSON: I think this can be answered categorically, no, with one exception. A properly fabricated polyester system has no plasticizer in it. Now if we are getting them with plasticizer someone is adulterating the system in such a manner that it will not do the intended job for severe corrosion.

CHAIRMAN SAUCHELLI: A good

beginning. Who else has a question? Mr. Atkinson you did a fine job. There are no more questions.

CHAIRMAN SAUCHELLI: The next subject is "Screens and Screening." Mention screens and at once you associate it with our old time friend Wayne King. Wayne has been an old hand at this and he is just anxious to get something off his chest. You all love to see Wayne come up on the platform here and tell us about his experiences, that is his business experiences, not his "Gaslight Experiences."

Screens and Screening

Wayne King

GOOD Morning — My assignment is to talk to you and it is your privilege to listen. I will make it as brief as possible and with a little luck we should wind up together.

Now—we all know, that you just can not get your foot in your mouth unless your mouth is open. But—also, a Turtle makes progress only when it sticks its neck out.

Perhaps, what I need is a band —i.e. a band-aid to put over my mouth. I say this—knowing that you may not agree with me on all points. In fact I will present some provacative opinions and perhaps meet some violent opposition; but you will be given equal time for rebuttal. My intention is to challenge your thinking and the conclusions you reach from my lecture may jolly well be your own. Also the jokes may be your own. I will eliminate the details and elementary remarks and get to the point of our discussion.

First let's talk about molecules.

A glass of water or a bar of steel looks tranquil; but the molecules are moving like mad. By somewhat similar thinking but with catastrophic results important to our thinking, would be three or more materials of same measurable size, trying to stay in the same cu. foot of space. I refer to materials having different bulk densities. Even with a close sizing, such as 8x10 mesh, .093″ x .065″ and regardless of how well we mix these constituents, they refuse to live with each other and non-segregation is impossible. The word homogeneous we use for a lack of a better definition. Certainly not heterogeneous. By definition homogeneous means alike in both nature and size. My point here is that a good job of granulation will approach a solution to this chemical problem.

In this day of specialization, it is no sin to be unfamiliar with subjects. As Will Rogers once said "We are all ignorant. We are just ignorant about different things."

We come here to the Round Table to become more knowledgeable. To share our experiences and findings. So—will you consider Granular with me. When you observe this, you can take it or leave it. We desire only unity of purpose.

Let us consider briefly, simplified practice as it may apply to *Granular* Standard sizing for granular has been requested by many interested parties, including the Spreaders. I intend to emphasize the chemical control, which may lead to a degree of size standardization. Obviously some variation of sizing may be with us, to maintain the desired chemical control and I am assuming this is important, altho; I appreciate the problem of the Blenders, as such. The Blender might gain something from my data, which must be studied before they step off in deep water. To be specific, I assume the chemical control is our major objective. Simplified Practice is in use on highway material for the Federal program; but with many "modifications" for valid reasons, sometimes.

Hence, we will talk about—Process — An Efficient Operation-Know How.

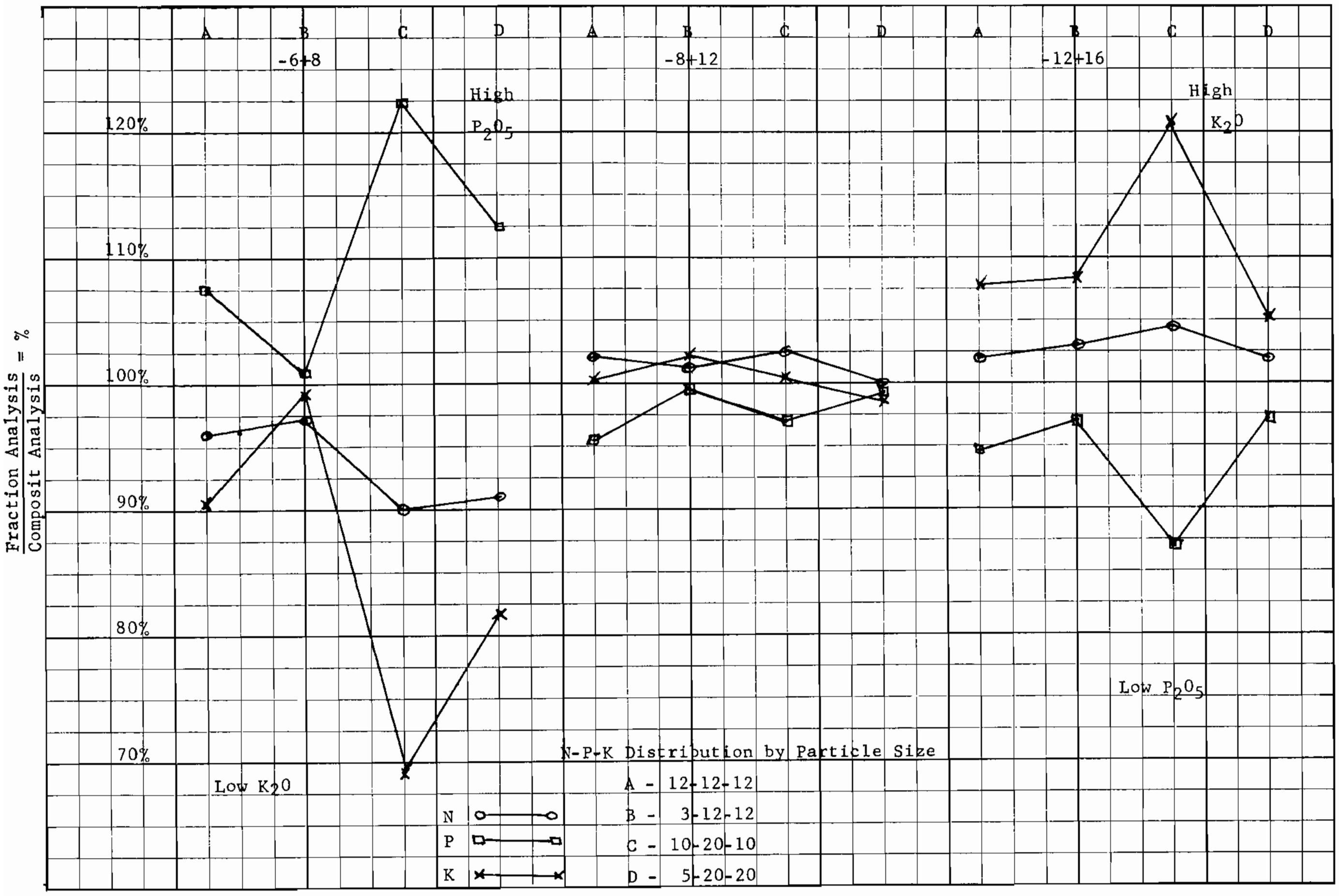
We, too frequently, have only consistency in change. We deal thruout our Flow with fluctuations of conditions and results. Many of our problems are continuing problems and with the advent of new techniques, we invite additional variables.

We must keep abreast or ahead of these situations and it is easier to stay out of trouble than to get out. I have been fussy about some few factors; because their control makes our production life more simple and free from strife.

To name a few—

1—Incoming voltage control.
2—Continuity of Feed and Feed Distribution to Screens.
3—Sufficient reduction capacity of distintegrators.
4—Profitable use of Testing Sieves to show us the way.
5—It is possible that we need an Expert on each shift for each Granular Mill.
6—And we need a definition of this Expert.
7—It is obvious that this Expert should be qualified as an Executive, Psychologist, Chemist, Engineer, Mechanic and or an understanding of these facets as they apply to our process.
This is an age of affluence — Everyone can either pay his bills or can charge it. To get and keep high class qualified men on this job at the "Do-It" level may be part of our problem. It is evident, that we do need someone on the job at some level, that knows the answers. Absence of these "brains" for even a short period of time can result in frustration.
8—Generally, knowledge doubles every ten years. We have to sprint to merely keep up in our Granular effort. The 10 year formula hardly applies to us. We, that are involved tend to take these changes in stride and I wish to emphasize that we keep our personnel equally in stride. For instance, modern math. is different as we understand it; but if our children understand it, then it may be alright.
9—So—to do our part, The W. S. Tyler Co. would propose a check off sheet, to cover production maintenance. This includes our call at your plant to cover production factors related to screening, i.e. size classification.

This may not be as good as the Happy Hour; but it is better than the Funny Farm.



So, as the British say, let's pull up our socks and get on with it. Slide. 6x8 – 8x12 – 12x16. This shows with certainty a high degree of chemical control with planned sizing.

Sieves showing cuts at 6-8-12-16 mesh and chemical analysis. This will be included in the FIRST release and further study is in order.

My associate Steve Janovac will give you different phases of this.

Screens and Screening

S. J. Janovac

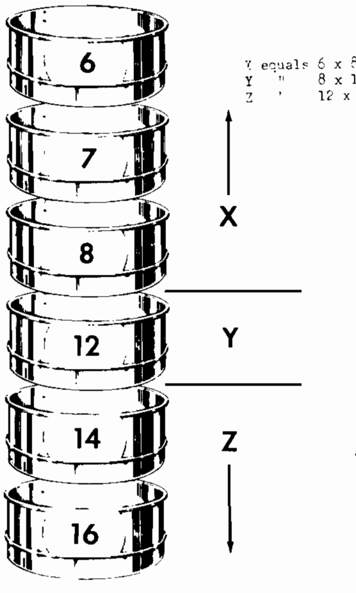
As all of us know, all too often there are misinterpretations of terms, which sooner or later must be defined. Now, it is not necessarily our intention to set the standard of definition but we can briefly outline what we consider standard terminology.

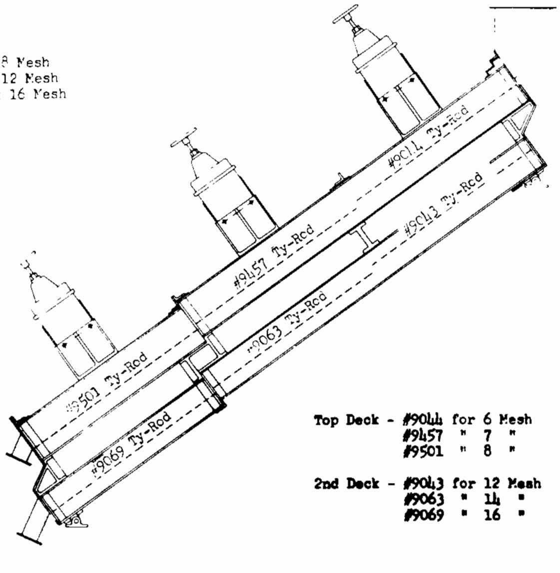
Screening can be simply defined as the separation of a mixture of various size grains of granules into two or more portions by means of a screening surface acting as a multiple go—no go gage with the final portions consisting of grains or granules of more uniform size than those of the original mixture.

Now "a screen" is a vibrating screen which in turn is simply a device set in motion by mechanical or electrical means imparting vibration to a screening surface usually of woven wire cloth called a screen section.

There are three basic types of wire cloth, with each having its own advantages and, of course, disadvantages.

The first is square mesh. The primary advantage of square mesh is that it provides for a closer control of the separation. However, it is more subject to blinding by hav-





ing four sides to hold the near mesh granule in the opening. Additionally, square mesh offers a relatively small percentage of open area in relationship to the wire diameter.

The next type of weave is what we call Ton-Cap. This type weave offers a larger percentage of open area with a heavier wire yet still maintaining the same opening. It also presents fewer possible blinding surfaces. However, it does not afford an accurate control of sizing and has less open areas than Ty-Rod.

Ty-Rod is freer screening than either of the other two, it has yet a heavier wire and a greater percentage of open area and is not as subject to blinding as the other types. Also the long slot wires have a tendency to develop a secondary vibration which helps keep blinding to a minimum. Again, the disadvantage is less control of sizing.

There are, of course, applications which dictate the need for a specific type weave and a wire size that will give adequate life. Obviously, on the raw material and product screens, we can use Ton-Cap and Ty-Rod sometimes more of necessity than choice, but even so, granular fertilizer is ideal for a slotted opening type weave, since essentially all the material is granular, whereas on a shipping screen, square mesh must be used to eliminate paper, wood slivers and other foreign material from the bag. The reason being that this trash is generally flat and thin and when turned end ways would pass through a slotted opening, but regardless of the presentation, it would not pass a square opening.

Therefore, in the selection of a type weave, attention must be given to the physical characteristics of the material, as just described.

The next criterion is the separation desired which should be premised upon a sieve number and the corresponding opening.

Once the opening or the separation has been established, then select the mesh count and—or wire size you feel will yield adequate life. As an example, we want a 6 mesh separation with a sieve opening of .131″. Now consider the mesh and wire from a catalogue.

(1) 4½ mesh .092″ ϕ .130″ opg. 34.2% O.A.
(2) 6½ mesh .023″ ϕ .131″ opg. 72.5% O.A.
(3) 6 mesh .035″ ϕ .132″ opg. 62.7% O.A.
(4) 5½ mesh .047″ ϕ .135″ opg. 55.1% O.A.

Now all four specifications have the same approximate opening. Now we can start eliminating. The first specification is much too heavy. Extended screen cloth life yes! But can we get anything through with only 34.2% open area? The second example leaves little question as to the ability to make the separation. It has a 72.5% open area, but with an exceptionally light wire, how long will that last at 40 to 50 tons per hour?

The remaining two selections are more logical. The same approximate opening, a respectable percentage of open area and an adequate wire diameter.

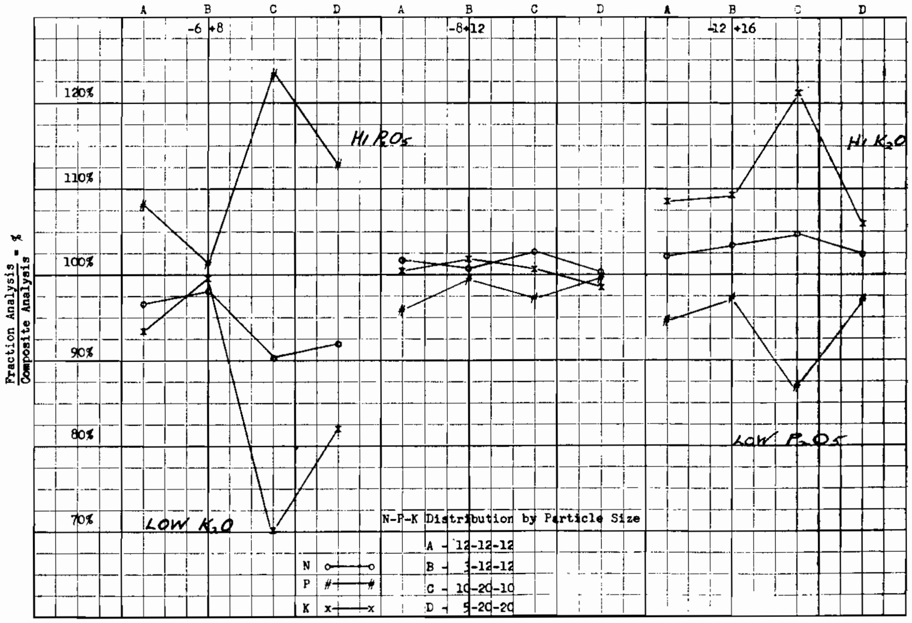
This same analogy would hold true for Ton-Cap and Ty-Rod.

The selection of a wire cloth specification is probably easier than keeping it on the screen. Naturally, we all would like to see every screen section wear out and frequently do, however, we all know that premature wire cloth failure does happen and can be caused by a multiude of reasons and in many cases a combination of conditions that exist in a particular plant. From our experience, we can cite a few of the main causes for wire cloth failure in order of their predominance.

Cloth not kept at proper tension—Approximately 90% of premature cloth failure is due to the screen section not being kept taut on the vibrating screen and allowed to flex or whip. The only positive way to assure adequate tensioning is with hook strips on the screen section. When installing a screen section, it is imperative the section be drawn to drum head tension and, also, it is very important to re-tension the section after a day or two of operation since wire cloth has a tendency to stretch with the section loosening up after initial tension. This is particularly true of Stainless Steel. If the wire cloth is allowed to whip, short life is inevitable.

Insufficient Wire Cloth Support — On mechanical screens we find many cases of breakage along a supporting strip indicating that the wire cloth is not sufficiently supported for the load being handled. The remedy for this would be the addition of more supporting rails built into the screen deck to afford greater support for the cloth.

Incorrect Feed—Poor feed distribution over the screening deck, overloading the screen cloth at one point will very often cause premature failure. Also in many cases, the feed comes onto the screen at too great a velocity or in surges



causing a heavy burden on the cloth at these instances.

Corrosive Action On The Fine Cloth—In many cases the material being fed over the screen promotes a corrosive action on the screen cloth. Sometimes this is in the form of minute pitting which can hardly be seen with the naked eye but is enough to start cracks in the wire cloth.

Oxidation of steel wire, once the galvanizing is worn off, is sometimes very hard to detect since the abrasive action of the material is continually removing the rust as quickly as it is formed, giving the appearance of a clean bright screen, where actually the high rate of oxidation gives the illusion that the screen is wearing out by abrasion only.

Improper Wire Cloth Specification — Frequently, the use of a heavier specification or the use of a Ton-Cap or Ty-Rod specification, to replace a square mesh, where applicable, will enable the use of a considerably heavier wire diameter and thus longer life.

We also feel that where Ty-Rod is applied, it should be applied so that tension is principally on the "warp" wires or the "many" wires.

Finally, there is also the possibility of a defect in the wire. However, this condition is very rare since we do exercise a great degree of quality control on all incoming wire making sure it meets our standards.

In summary then, we have a vibrating screen using wire cloth to separate material into two or more fractions using square mesh, Ton-Cap or Ty-Rod which must be kept at drum head tension using hook strips to avoid premature cloth failure generally caused by whipping or flexing.

CHAIRMAN SAUCHELLI: Thank you, Mr. Janovac.

We'll leave the questions until after the next speaker. "Screening Requirements can be Specified" is the title of the next speaker's talk. This whole business of screening, screen-size and so on, is very important in our industry, and since we hold no special axe to grind we thought it would be only right and proper to get as much information before this group as possible on "Screens and Screening."

With new organizations coming into the picture I am sure you will be interested in hearing what the next speaker has to say. He is a young engineer. He has had wide experience in industry. It is a pleasure to call on Mr. E. E. Ferry, Novo Industrial Corporation, to give his talk.

Screening Requirements Can Be Specified

E. E. Ferry

As the fertilizer industry continues its technological development and the processes become more complex, the need for more reliable performance by the process equipment also increases. An important means of improving the performance of this process equipment is the use of more definitive specifications for the product requirements and process performance.

While procedures for purchasing screening equipment vary between fertilizer producers, it has been our experience that the specifications for the performance of screening equipment generally have not been detailed nor specific. A typical inquiry gives the:

Fertilizer composition
The expected feed or product rate
The nominal size classification of the product and its purity limits
The particle size distribution of the feed material

There may be additional information regarding the product and process requirements, but the entire specification normally is contained on one side of an 8½ by 11 inch sheet of paper. Included with this specification, there are frequently four or five pages of specifications covering the type of electric motors that may be used with the screening equipment.

Possibly the reason that specifications covering screening equipment and its performance are so limited is that in the past we manufacturers have not given the fertilizer industry so much information as have the manufacturers of electric motors. This no longer need be the case. Screening is gradually changing from an art to a science. Improved laboratory methods now make it possible to study the vibration patterns developed in the screen cloth and to follow the action of the individual particles during screening. This information is now being related to capacities and efficiencies for various screening applications.

Most, if not all, screen manufacturers have determined the capacity and efficiency of their machines in separating fertilizer materials at the various separation sizes. This information can be correlated to your screening requirements in detail with reasonable accuracy. With this capacity-efficiency information available to you, it is only logical that your specifications regarding the process requirements and product particle size distribution should now become more definitive.

Your customer judges your product to a large degree on its particle size: Is there a large dust loss and does it spread evenly? This particle size depends primarily on the performance of your screening equipment.

But what factors should a complete screen specification include? Since information must flow in both directions between the fertilizer producer and the screen manufacturer, the complete specifications should basically include:

Process design specifications
Process information
Mechanical & electrical specifications

In return, with the manufacturers' recommendation and proposal should be included the information for evaluating screens for process performance.

Each of these items, with the exception of the mechanical and electrical detail, will be discussed this morning.

The process design specification that you write should cover the two most important factors regarding your requirements, capacity and purity. The minimum feed rate or product rate that is acceptable should be established, generally in tons per hour. The minimum product size requirements that will be accepted should be defined in terms of the maximum percentage of material larger than a given mesh size and of material smaller than a given mesh size that is allowable. These two factors constitute the entire process design specifications and cover the basic requirements for the screening equipment by establishing its minimum capacity and the product purity. Most other process conditions and performance characteristics then become factors which are useful in evaluating the screening equipment proposals.

As mentioned previously, various factors should be considered in the evaluation of a screen proposal. The following information regarding its predicted process performance should be required:

1. Conformance to the process design specifications. If the capacity, either in feed rate or product rate, and the product particle size distribution do not meet the minimum design specifications, then further evaluation has limited value. By insisting upon conformance, you assure yourself of a consistent base from which to evaluate all screening equipment proposals.
2. Efficiency of recovering the available product size material as product. Possibly the ability to recover the product size material is the most significant factor to be considered in the evaluation after conformance to the process design specification.
3. Rate of recovery of product within the design specification. While this may be more or less synonymous with the efficiency in recovering product size material, it has significance in relation to the types of screen cloth and aperture used with the screening equipment. If the capacity/efficiency ratio of the screening equipment is such that it can perform closer to the process limits and provide a higher "On Specifications" product rate, this is obviously to the producer's advantage.
4. Installed costs. The method of evaluating the cost of purchasing and installing capital equipment is established within each company and is a subject that will not be covered here in detail. Generally, the evaluation is on the basis of dollars to do a job, however, the efficiency in recovering product should also be considered.
5. Performance during upset process conditions. The producer should give consideration to the performance of the screening equipment under conditions other than design. While the purpose of this discussion this morning is to develop specifications for equipment so that the process will operate as it was designed, realistically, this will never be achieved. The fertilizer process will almost always be in some stage of upset. I use the term upset as being other than the designed feed rate, particle size distribution, etc. Since most of you probably consider these upset conditions as an every day way of life, the ability of the screening equipment to accommodate these variations in feed rate, particle size distribution, moisture, etc. is then an important factor in the evaluation. There are other factors to be considered in the evaluation. Some are finite and some will be intangible such as the number of machines required, space, tendency to blind, maintenance, etc.

So far we have established that the complete screen specifications should include:

Process design specification of rate and purity.

A listing of the screen performance information that allows you to evaluate each proposal.

One item of importance should be brought out at this point. The specification should avoid taking over the manufacturer's responsibility or function by specifying limits on feed rate per unit of screen area, screening angle, or screen apertures. While the producer may do this accurately from past experience with a given make of equipment, it does limit him to the capabilities of that equipment. You then cannot take advantage of new developments with which to meet increasing competition and the more demanding product requirements.

In order for the screen manufacturer to provide the performance data with a proposal, the following process information must be supplied with the inquiry:

I. General Information
  A. Chemical Classification — is the material a complex fertilizer, triple superphosphate, urea, ammonium phosphate, etc.
  B. Type — is it granular, spherical, prilled and so forth.
  C. Nominal Size Classification—is the product classified as a 6 by 16 or 8 by 20, etc. mesh product.

II Chemical and Physical Properties
  A. Chemical Composition — if a complex fertilizer is it a 10-10-10, a 16-8-8, or some other composition. Since the composition generally has an effect upon the other properties, this should be considered in the information furnished.
  B. Particle Size Distribution of the Feed to the Screen—as we know this will vary with the chemical composition and the process conditions. Because of this significant effect that distribution has on the screening efficiency, the coarsest and finest distributions expected to occur should be given in addition to the design distribution.
  C. Temperature
  D. Moisture
  E. Density
  F. Angle of Repose
  G. Hardness

All of the above characteristics are related to the material as it is fed to the screening equipment and provide information regarding its screenability.

H. Particle Shape—the shape of the particles also has an effect on the capacity and efficiency with which the material may be screened. Therefore, it is necessary to know if the particles are granular, spherical, prills, etc.

III Process Data

A. Feed Rate—the feed rate, along with the particle size distribution and the particle shape are the most significant factors in recommending screening equipment for an application. Variations in the feed rate will affect the efficiency of the separations. Consequently the process information should include, in addition to the design feed rate, the maximum and minimum rates that are expected to occur with any frequency.

B. Desired Product Rate—where the product rate is not used as a part of the process design specification, the rate desired is useful in establishing the recovery efficiency. In some instances, it is desirable to recover as much product as possible and in other cases it may not be desirable or practical.

C. Desired Recycle Rate — for certain applications it may be desired to maintain a special recycle rate rather than a product rate, in this instance this rate should be given and, if it is important, the recycle rate for either or both the oversize and fine materials.

D. Process System Equipment—the type of equipment conveying the material to the screen should be given. Each has it own delivery characteristics. The immediate preceding process should also be given, for as an example, screening characteristics of a drier discharge are different from those of a cooler discharge.

With the above process information, the screen manufacturer can analyze the process design separations required, the required capacity and efficiency under design conditions and make a recommendation that is related to the process requirements and material properties. The manufacturer can also develop the performance of the recommended equipment under the predetermined upset conditions.

Although each manufacturer may have different methods of compiling, cataloging and applying the capacity/efficiency capabilities of his equipment for various materials and processes, the procedure is more or less the same. One example is to present the capacity/efficiency data in a graphical form as shown in Fig. 1. The percent efficiency is plotted in relation to the feed rate to the screen. In this instance the material is a 16-8-8 granular fertilizer. The separation size is 6 mesh and the screen aperture used is 0.118 by 0.354 inches rectangular. The feed to the screen contains 20 percent plus 6 mesh material. Curve 1 depicts the efficiency in passing the minus 6 mesh material through the screen at the various feed rates. Curve 2 depicts the efficiency in retaining the plus 6 mesh material above the screen.

As you can see, in order to get an efficiency of 95% in passing the minus 6 mesh, a feed rate of X TPH/sq. ft. or less must be used in designing the screen. Under these conditions then, the efficiency of retaining the plus 6 mesh material above the screen will be Y %.

From these data, anaylses of the effect of specific properties or characteristics may be made. For example, the effect of particle size distribution on the efficiencies is illustrated here. The percent efficiency is plotted in relation to the amount of plus 6 mesh material in the feed. Again the material is the granular 16-8-8, the separation size is 6 mesh and the screen aperture is 0.118 by 0.354 inches rectangular. The feed rate for this curve is 2 tons per hour per square foot of screen area. As the amount of plus 6 mesh material in the feed decreases, the efficiency in passing the minus 6 mesh material increases and the efficiency in retaining the plus 6 mesh material decreases. This then allows the previous curve to be used at feed compositions of other than 20% plus 6 mesh.

To illustrate—the use of such data in developing a recommendation of screening equipment and a method of presenting this information to you for evaluation, the following example has been developed.

Assuming a screening requirement for a granular fertilizer having a composition of 16-8-8 and a size classification of 6 by 14 mesh. The process specifications are:

1. A minimum feed rate of 100 tons per hour, and
2. Product size requirement of a maximum of 5 percent plus 6 mesh material and a maximum of 5 percent minus 14 mesh material.

The required information for evaluations are:

1. Conformance to the process specifications
2. Efficiency in recovering the 6 x 14 mesh size material as product at design conditions
3. Recovery rate of product at design conditions
4. Performance under the upset condition limits

The particle size distribution of the feed under design conditions is;

Plus 6 mesh 20%
Minus 6 plus 14 mesh—50%
Minus 14 mesh—30%

The maximum feed rate under upset conditions is 200 tons per hour and the minimum is 50 tons per hour. The feed material particle size distribution during coarse conditions has

Plus 6 mesh—30%
Minus 6/plus 14 mesh—60%
Minus 14 mesh—10%

During fine conditions it has;

Plus 6 mesh—10%
Minus 6/plus 14 mesh—40%
Minus 14 mesh—50%

The analyses of the separations that would result are shown. Fig. 2. At the design feed rate of

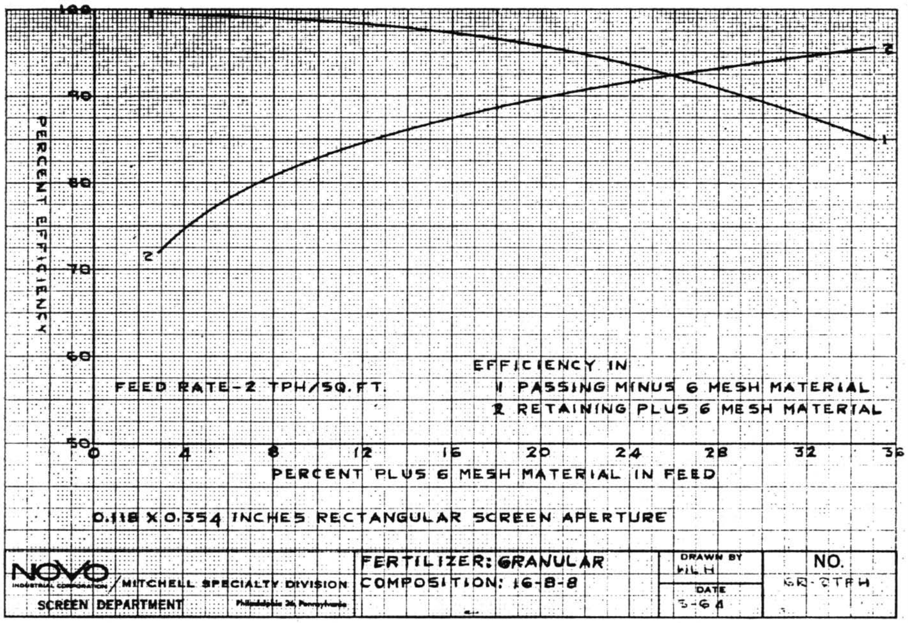


Figure 1.

100 tons per hour and the particle size distribution at design conditions, the product conforms to the specified range of 5% maximum plus 6 mesh and 5% maximum minus 14 mesh. The product recovery rate is 46 tons per hour and 91 percent of that product is minus 6 plus 14 mesh. This is a rate of 41.8 tons per hour of product size (6 x 14) material. Since 50 tons per hour of 6 by 14 mesh material is available in the feed the recovery efficiency of product size material is 83.6 percent. The performance during the maximum upset conditions is also given. An analysis of this type can be included as part of a screening equipment proposal, providing the design and maximum and minimum conditions will be established, and the process information mentioned previously is well defined.

In summary, the equipment manufacturer can supply detailed performance information regarding the separation of your materials with reasonable accuracy under design and upset conditions.

The establishment of the process design specification covering the minimum feed or product rate and the minimum product particle size requirements should be based on a design condition. Possibly the design condition may be difficult to define in specific terms, but it is desirable as a common base for your evaluation of all proposals and, certainly, you the producer are most qualified to do so. The process information is equally important to provide the manufacturer with sufficient data on which to base a reasonable equipment recommendation. By including in an inquiry, the process performance information required for evaluation, you assure yourself of receiving a proposal that has been carefully prepared with organized data on which to base a selection.

Although this may seem to be a very difficult task, you will see from a typical format that all of the information can be presented on three sheets of 8½ x 11 paper. (less than most motor specifications)

Through this flow of information from the producer to the manufacturer and back to the producer, you ultimately get the best equipment for your specific application . . . . I'm sure you'll agree it's worth the effort.

Process Specification For Screening Equipment Based On Design Conditions

1. Minimum feed or product rate
2. Minium product size requirements

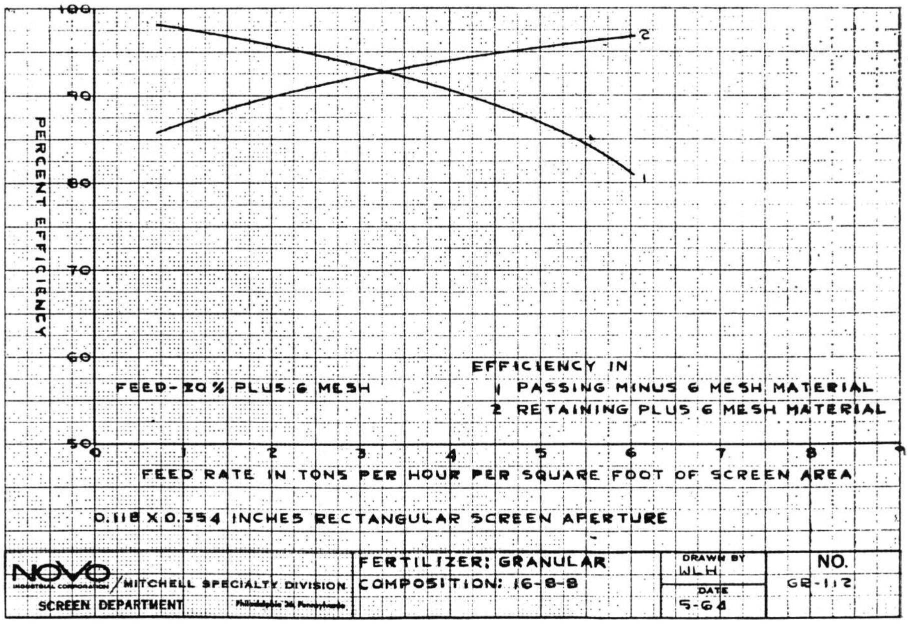
Information Required For Evaluating Screening Equipment For Process Performance

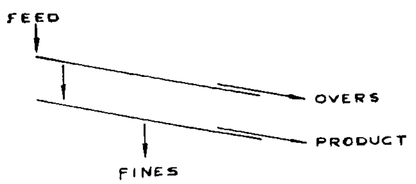
1. Conformance to the process design specifications
2. Efficiency of recovering the available product size material as product
3. Recovery rate of product within the specification
4. Installed costs
5. Performance during upset process conditions

Process Information Required For Recommendation of Screening Equipment

- I General information
  - A. Chemical classification
  - B. Type
  - C. Nominal size classification
- II Chemical and physical properties
  - A. Chemical composition
  - B. Particle size distribution of feed percent retained—cumulative mesh design coarse-fine
  - C. Temperature

Figure 2.





	Design	Maximum	Minimum
Feed Rate—TPH	100	200	50

Analysis	Design	Coarse	Fine
+ 6 M	20	30	10
6× 14 M	50	60	40
− 14 M	30	10	50

Feed Rate	Design — 100 TPH			Maximum — 200 TPH			Minimum — 50 TPH		
	Overs	Product	Fines	Overs	Product	Fines	Overs	Product	Fines
Design Feed									
Weight	21 TPH	46 TPH	33 TPH	53 TPH	91 TPH	56 TPH	9.5 TPH	21 TPH	19.5 TPH
% of feed	21%	46%	33%	26.5%	15.5%	28%	19%	42%	39%
Analysis									
+ 6 M	85%	4%	0%	71%	3%	0%	92%	6%	0%
6 × 14 M	15%	91%	16%	29%	87%	10%	8%	93%	26%
− 14 M	0%	5%	84%	0%	10%	90%	0%	1%	74%
Coarse Feed									
Weight	36 TPH	51 TPH	13 TPH	82 TPH	98 TPH	20 TPH	15 TPH	24 TPH	11 TPH
% of feed	36%	51%	13%	41%	49%	10%	30%	48%	22%
Analysis									
+ 6 M	79%	4%	0%	71%	2%	0%	88%	7%	0%
6 × 14 M	21%	91%	33%	29%	94%	24%	12%	92%	53%
− 14 M	0%	2%	67%	0%	4%	76%	0%	1%	47%
Fine Feed									
Weight	9.5 TPH	36 TPH	54.5 TPH	34 TPH	65 TPH	101 TPH	4 TPH	15 TPH	31 TPH
% of feed	9.5%	36%	54.5%	17%	32.5%	50.5%	8%	30%	62%
Analysis									
+ 6 M	87%	5%	0%	58%	1%	0%	95%	7%	0%
6 × 14 M	13%	90%	12%	42%	86%	10%	5%	92%	20%
− 14 M	0%	5%	88%	0%	13%	90%	0%	1%	80%

- D. Moisture
- E. Density
- F. Angle of repose
- G. Hardness
- H. Particle shape

III Process data
- A. Feed rate
  - 1. Design
  - 2. Maximum
  - 3. Minimum
- B. Desired product rate
- C. Desired recycle rate
  - 1. Oversize
  - 2. Fines
- D. Process system equipment
  - 1. Equipment conveying feed to screening equipment
  - 2. Upstream process equipment

SCREEN EQUIPMENT SPECIFICATIONS

1.0 *Materials To Be Screened*

1.1 Chemical Classification: ____________

1.2 Type: ( ) Granular ( ) Spherical ( ) Other ________

1.3 Nominal Size of Product: ________ Mesh X ________Mesh

1.4 Minimum Acceptable Product Rate Under Design Conditions: ____________ TPH

2.0 *Chemical & Physical Properties*

2.2 Chemical Composition (s) : ________; ________; ________; ________; ________; ________; ________; ________.

2.2 Particle Size Distribution of Feed·

	% Retained, Cumulative		
Mesh	Design	Coarse Feed	Fine Feed

(*Continued on Page* 16)

Chairman Sauchelli: Thank you Mr. Ferry. We have time for a few questions for Mr. Ferry, Mr. Javanac and Mr. King.

Mr. Quentin Lee: We are looking for a fast efficient way to clean our screens.

Mr. King: You are looking at the fastest screen brusher in the business. It was a year or two ago that we told you about this motorized wheel brush. That's the best way I have found to clean a screen. It beats this pushing a broom. It saves the wire. It is a little motor with a 12 foot flexible shaft on the end of which there is a 6 inch diameter wire wheel brush.

Chairman Sauchelli: Do you have anything to add to that, Mr. Ferry?

Mr. Ferry: No, I think that is just fine.

Chairman Sauchelli: Any other questions?

Mr. King: If you desire Tyler Bulletin 63 (1964 Edition) write us a postcard and we will send you one. It is not a catalog. It is a bulletin on testing sieves and the profitable use of it.

Mr. Steve Long: These data on efficiencies and screen capacities are right impressive. Were they obtained from the laboratory or production runs that go off week after week, and month after month?

Mr. Ferry: Most of these data has been done under laboratory control conditions. Many of them have been checked out through production equipment and check out very well. In fact, I would really say, they are probably on the conservative side.

Mr. Al Henderson: I want to direct this question to Wayne King. What happens when a 6 inch lump hits that apparatus you just described between the screens?

Mr. King: All hell breaks loose! I'm not selling these things you know. I just tell you to make one. Actually you can make your own, just buy a 12 foot flexible shaft and hook it on an 1800 RPM motor. It will do your job. If you want to play it safe, wait till the feed has cut off or is light

Chairman Sauchelli: Any other questions?

2.3 Feed Temperature at Time of Screening:
Normal ________ °F; Maximum ________ °F

2.4 Moisture Content: Normal: ________% Maximum: ________ %

2.5 Density: ________ #/Cu. Ft.

2.6 Angle of Repose: ________° from Horizontal

2.7 Hardness: ________

2.6 Particular Shape: ________

3.0 *Process Data*

3.1 Feed Rate:

  3.1.1 Design ________ TPH

  3.1.2 Maximum ________ TPH

  3.1.3 Minimum ________ TPH

3.2 Desired Product Rate: ________ TPH

3.3 Desired Maximum Recycle Rates:

  3.3.1 Oversize: ________ TPH

  3.3.2 Fines: ________ TPH

3.4 Other Process Data: ________

____

4.0 *Minimum Product Specification*

4.1 Nominal Size of Product: ________ X ________ mesh

4.2 Maximum Allowable Plus ________ mesh in product: ________%.

4.3 Maximum Allowable Minus ________ mesh in product: ________%.

____

____

5.0 *Mechanical Specifications*

5.1 Materials of Construction:

  5.1.1 Parts Contacting Process Material: ________

  5.1.2 Screen Cloth: ________

  5.1.3 Parts Not in Contact with Process Material: ________

  5.1.4 Finish or Paint Requirements: ________

  ____

5.2 Maximum Head Room Available: ________ feet

5.3 Maximum Floor Space Available:

  5.3.1 Length: ________ Feet

  5.3.2 Width: ________ Feet

5.4 Number of Screening Machines Desired:

5.5 Materials Handling Equipment Used in Conjunction with Screen:

  5.5.1 To Feed Screen: ________

  5.5.2 Remove Product: ________

  5.5.3 Remove Overs: ________

  5.5.4 Remove Fines: ________

5.6 Connection Sizes Desired (Approximate):

  5.6.1 Feed: ________

  5.6.2 Product: ________

  5.6.3 Overs: ________

  5.6.4 Fines: ________

5.7 Screen Enclosure:

  5.7.1 ( ) Open

  5.7.2 ( ) Dusttight

  5.7.3 ( ) Suction Connection

5.8 Processing Equipment Immediately Preceding the Screen:

____

6.0 *Electrical Specifications*

6.1 Power Characteristics: ________ volts/ ________ phase/ ________ cycle

6.2 Control Circuits: ________ volts/ ________ phase/ ________ cycle

6.3 Enclosures: ( ) Noma ________

  ( ) Dusttight

  ( ) Explosion-proof, Class ________ Group ________

  ( ) Other ________

6.4 Motors:

  6.4.1 Enclosure: ________

(*Continued on Page* 17)

6.4.2 Material of Construction: ____________________
6.4.3 Bearings: ____________________
6.4.4. Insulation Class: ____________________
6.4.5. Service Factor: ____________________
6.4.6 Other: ____________________
____
____

7.0 *Other Pertinent Facts and Requirements*:
____
____
____
____
____
____
____
____
____

Thank you again, Mr. Ferry, Mr. Javanac, Mr. King for your splendid presentations.

The business of sampling is a very serious one in our industry. It's difficult I know. I have had some experience in organizing research sampling bag fertilizers, but sampling bulk fertilizers really presents a problem. Our next speaker, Mr. L. J. Pircon, has given a great deal of time and thought to this problem. He is going to explain to us what his own organization has found in their intense study of the problem of sampling bulk fertilizer.

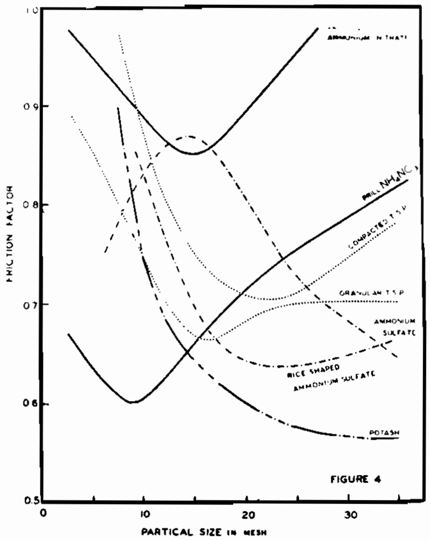
Problems Sampling Bulk Blended Fertilizers

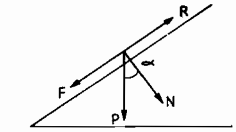
L. J. Pircon

SOME time ago Central Farmers and others observing physically blended fertilizer materials noted the relationship between the tendency of these materials to segregate and the matching of their particle sizes. However, continued work showed other factors to affect this relationship and the need for correction in the correlations developed. From this need was born our "K" factor concept. In plain, simple language this means that mathematically it can be shown that the angle of repose of the material has built into it size as well as all the other factors that can be considered and corrected for (Fig. 1).

The force vector analysis shows that the tangent of the angle of repose equals the retardent to motion constant K (Fig. 2). Furth-

Figure 1





$N = P\cos\alpha$, $F = P\sin\alpha$, $R = kN$, $P = Mg$

Where a = acceleration, M = mass & k = friction factor

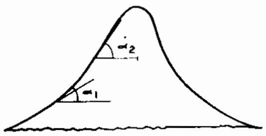
$$a = \frac{F-R}{M} = \frac{Mg\sin\alpha - kMg\cos\alpha}{M} = g(\sin\alpha - k\cos\alpha)$$

When $F = R$, $a = 0$ & $g(\sin\alpha - k\cos\alpha) = 0$

$\therefore k = \tan\alpha$

Figure 2

Figure 3



$a_1 = g(\sin\alpha_1 - k_1\cos\alpha_1)$ & $a_2 = g(\sin\alpha_2 - k_2\cos\alpha_2)$

$\Delta S = S_2 - S_1 = \frac{1}{2}t^2(a_2 - a_1) = \frac{1}{2}t^2 g(\sin\alpha_2 - \sin\alpha_1 - k_2\cos\alpha_2 + k_1\cos\alpha$

Where S = distance of particle travel and t = time

When $k_2 = k_1$, since $k = \tan\alpha$, then $\alpha_1 = \alpha_2$

and $\Delta S = 0$

Figure 4

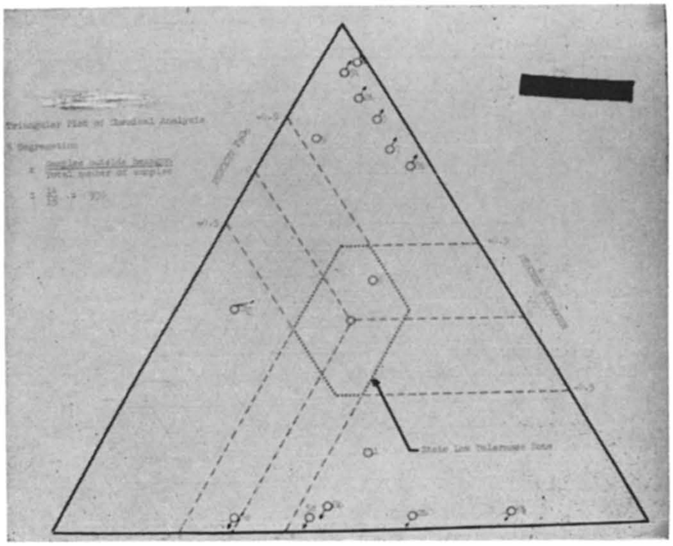

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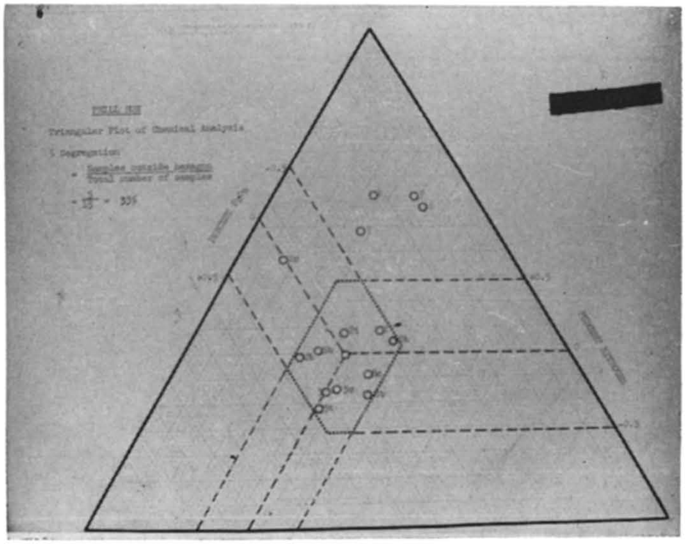
Figure 5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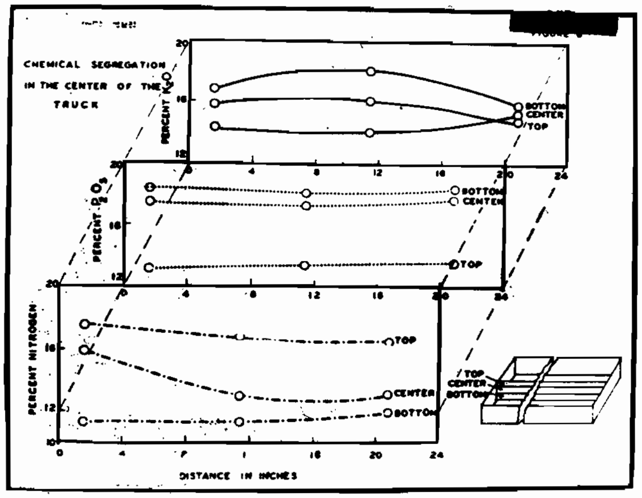


Figure 6

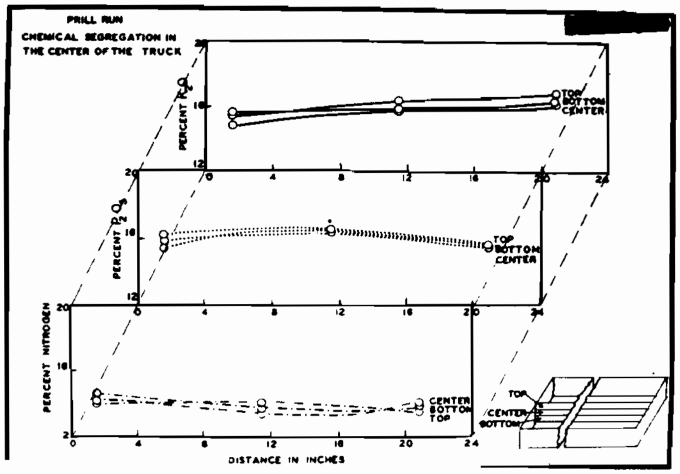


Figure 7

ermore, the equation describing the distance traveled shows that when K's of different materials are equal, $\Delta S = O$, and they will not move apart (Fig. 3).

How did this stand up in the laboratory and the field? Pilot sized equipment was made up and the truck was probed following a variety of haul conditions. When average weighted K's were not equated, 93 percent of the samples fell outside of the 0.5 tolerance hexagon (Fig. 4). More equal K's dropped this to 33 percent. (Fig. 5).

Variation in chemical and physical (size distribution) composition were noted in different parts of the truck at three levels immediately after loading the truck, then later after the haul. The chemical and physical difference was immediately apparent even for size matched particles whose shape factor differed considerably (Figs. 6,7,8,9). Where the K's were matched, the analyses matched and the two lines representing concentration versus truck dimension were parallel to the abscissa or horizontal axis and N, P_2O_5 and K_2O overlapped because it was a 1-1-1 analysis. But, as is seen, where the K's did not match the lines developed curvatures and spread apart showing segregation of variant pattern within the geometry of the truck. The size distribution at the three bed levels followed the same behavior pattern. No relative shifting with similar average K's, but appreciable changes in percentages for different K's. In the latter case, the larger particles walked away from the smaller ones.

Where K's were unequal, great care was taken to load the truck in a non-conical fashion and the curves almost overlap (1-1-1) grade and are parallel to horizontal axis, but curved showing wall effect. (Fig. 10) Following haul the material unmixed from top to bottom, but retained some relative

Figure 10

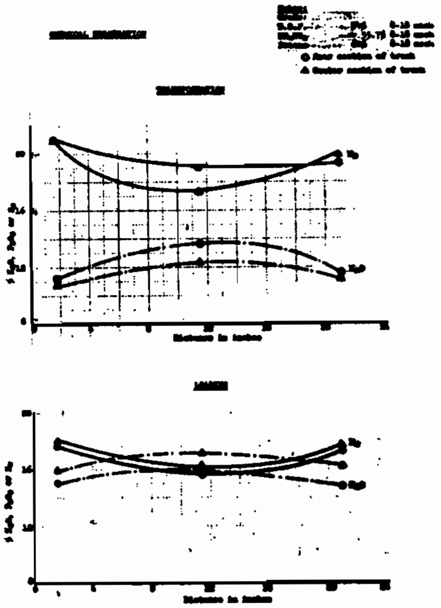


Figure 8

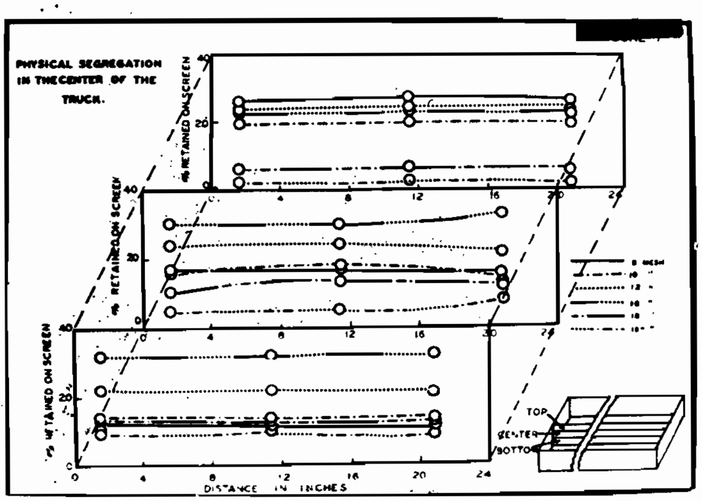
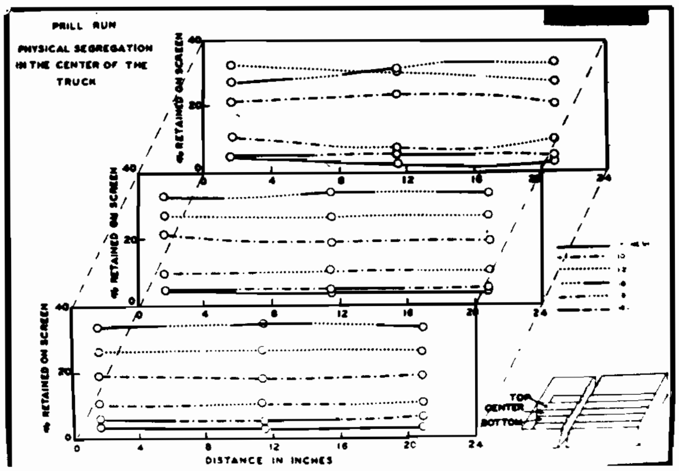
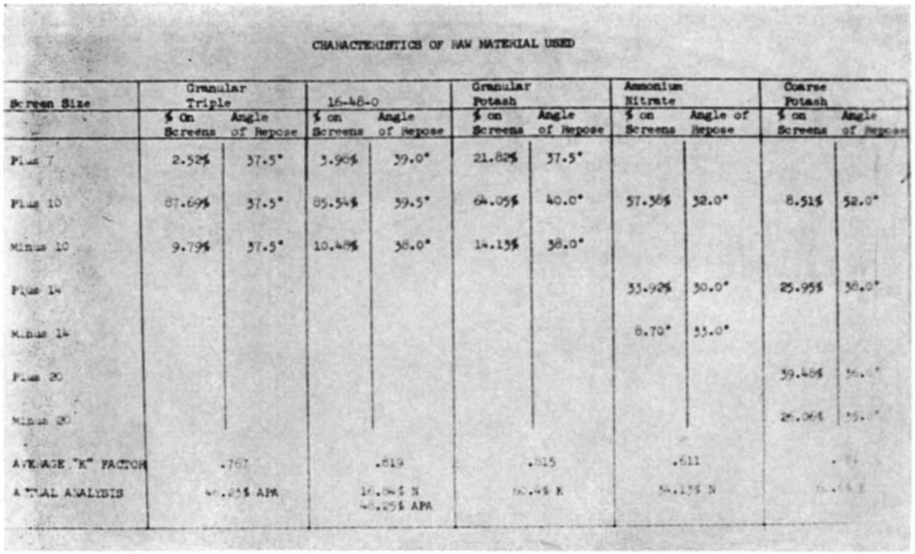


Figure 9





CHARACTERISTICS OF RAW MATERIAL USED

Screen Size	Granular Triple		16-48-0		Granular Potash		Ammonium Nitrate		Coarse Potash	
	% On Screens	Angle of Repose	% on Screens	Angle of Repose	% on Screens	Angle of Repose	% on Screens	Angle of Repose	% on Screens	Angle of Repose
Plus 7	2.52%	37.5°	3.96%	39.0°	21.82%	37.5°				
Plus 10	87.69%	37.5°	85.54%	39.5°	64.05%	40.0°	57.38%	32.0°	8.51%	52.0°
Minus 10	9.79%	37.5°	10.48%	38.0°	14.13%	38.0°				
Plus 14							33.92%	30.0°	25.95%	38.0°
Minus 14							8.70°	33.0°		
Plus 20									39.48%	36.[illegible]°
Minus 20									26.06%	35.[illegible]°
AVERAGE "K" FACTOR	.767		.819		.815		.611		[illegible]	
ACTUAL ANALYSIS	4[illegible].25% APA		16.84% N 48.25% APA		60.4% K		34.13% N		[illegible]	

Figure 11

patterns front to back and side wall to side wall.

To summarize, most of the segregation occured during conical loading of the truck, but could occur if K's were too far apart, even with careful loading followed by haul.

Additional work was conducted on bagged goods. The raw material was screened, piles of each fraction measured for angle of repose and weighted average K's computed. (Fig. 11) Care at this point must be exercised to measure free piles without drag force from side walls imposed. A short cut was attempted using a parallel plexiglass plate assembly and those pseudo K's with inhibited pile rest did not correlate. After the bags were filled, diagonal probes were taken and the results represented graphically (Fig. 12 and 13). These show that even with bagging the K correlation persists. Each set of points represents a bag and the only bags that would have met the state minimum tolerances were those where the K's were almost identical. A slight difference in K's produced bags not meeting the minimum, and bags containing matched sizing did not pass. This work showed how sensitive such measurements must be. Incidentally, these extended studies were conducted independently by another group of researchers outside of our company using our K concept and technique of measure .

This sensitivity of measure brings us to some practical considerations. Just how much control do we have over the material we are shipping, receiving and working with? Suppose the prime supplier decides to narrow the size range of his product. Especially with processes involving recycle, a plant of fixed size operating at near capacity would result in producing at a reduced rate. If additional or new facilities were incorporated to maintain production rate, increased capital requirements would still result in costlier product. Somewhere along the line this will have to be passed on to the customer. It is apparent from this that a narrowing of size will find a practical limit.

How about product and consequently production reproducibility? Certain of these materials are produced under precise controls and conditions and are remarkably similar from day to day. Others depend upon the state of the art, atmospheric humidity, number of sun spots and who knows what else—and the variance reflects this. Not only does the percentage on each screen vary from car to car and within the car, but even its granule structure and rugosity will vary.

How do these conditions reflect in the raw material bins? Since K varies with size it will vary with the pure material in a cone loaded pile in the bin (Fig. 14). When the shovel bites into the base of one pile and the top section of the next, even if they were originally matched, the shovel fills do not match each other. This can

Figure 12

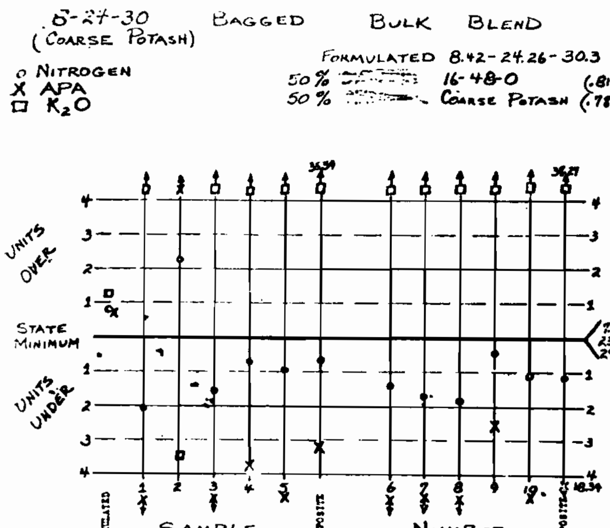
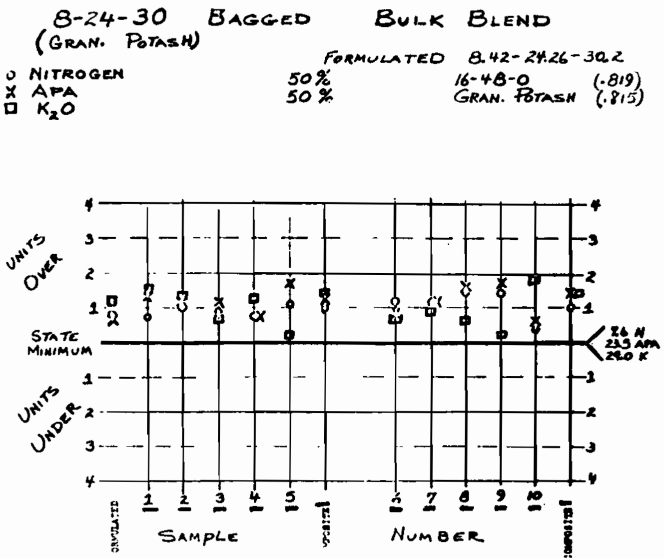


Figure 13



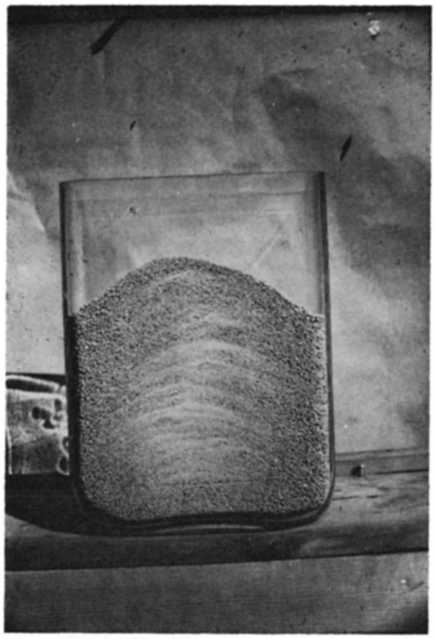


Figure 14

be minimized by not loading the pure materials in their respective bins in a conical fashion; but, instead, attempting to load with level surfaces. The same applies in loading the blends into trucks. This can be accomplished by use of a spout consisting of a rotating elephant nose or oscillating deflection board or other suitable mechanical devices. In the case of the truck this can be facilitated by pulling the truck forward during the loading, thereby piling the length of the hopper bed.

Do these conditions mean that we are forced to throw up our hands and attempt to solve these problems by forgetting about them? Let us look at what some of this knowledge has accomplished already. Ever since we at Central have known of the effect of pile coning we have instructed our operating personnel accordingly, thereby minimizing these ill effects as far back as four years ago. If typical specifications are established wherein average weighted K's are equated for the basic materials used, the odds will favor the majority mixes by minimizing the segregating tendency. True, some will always be separated enough to make it appear as though the proper amount of nutrients were not formulated initially, but equal K specifications will favor the majority.

This poses another question. Is it desirable to penalize some shipments even though the same honest effort was expended to produce the blend as the statistically favored? Chemical detective work is done by analyzing a sample of the shipment and calculation determines the acceptability of the product. All the nutrients are there, but the physics prohibits our finding them unless we sample in a manner satisfying this physical condition. Chemical "backtracking" is acceptable as an indirect means of regulation of a homogeneous product, but how heterogeneous must it be before the procedure is not applicable? Even if the K's are equated within practical limits there will always be some random innocent victims among the statistics.

What does all this suggest then?

1. Continue moving in the direction of matching materials. To improve the odds of a greater number of legally acceptable shipments, corrections for factors other than size should be made, and the use of the K factor will achieve this.
2. Alter some of the present practices in the blending operation in view of information developed by various groups studying segregation. Eliminate cone loading of bins, trucks, etc. Make certain that the mixer mixes to begin with. After maximum mixing is achieved, minimize handling in order to "leave well enough alone." Avoid excessive vibrations in containers, both stationary and moving. This means good suspension systems to isolate vibrations, and even proper choice of geometry and structural materials for the bins and trucks.
3. After everything feasible is done as per items 1 and 2, additional measurements regarding segregation and sample variance must be made and practically evaluated. Certainly product composition will vary, but to what extent and, more importantly, what extent is agronomically feasible? It has already been shown that certain crops of considerable economic stature permit surprisingly considerable variance. If it is found that the present regulatory philosophy and methodology is adequate to the needs of the blend program, which brings the manufacturing into the salesmens' and the farmers' lap, then not a stone need be turned. But on the other hand, if not, then both will have to be custom designed to suit the needs of this rapidly growing class of fertilizer business.

Chairman Sauchelli: Thank you, Mr. Pircon. That was certainly a flattering example of cerebration. We certainly enjoyed the presentation and I think that we can see how every problem can be made, can be analyzed thoroughly and in the analysis why a lot of it will get away from us, but it is possible to do a thorough job in chemistry and physics.

Those of us who have had the privilege of traveling abroad, recognize the pleasure and the privilege we receive when a foreign host shows his hospitality to us. We have among us today guests from overseas and I hope that all of you will make our foreign guests feel at home. I wish particularly to single out Dr. S. K. Mukherjee. Will you stand up, please, Dr. Mukherjee. Dr. Mukherjee comes from India. He is one of the outstanding technical authorities there in his country, and he showed me many hospitalities when I was over there. So it gives me much pleasure Dr. Mukherjee to acknowledge your visit with us today.

Dr. Mukherjee arose.

Chairman Sauchelli: We will stand adjourned until two o'clock.

Wednesday Afternoon Session, November 11, 1964

The Round Table reconvened at 2 o'clock P.M.
Joseph E. Reynolds, Moderator, presiding

MODERATOR REYNOLDS: Good afternoon and welcome. This afternoon and tomorrow afternoon we will have sufficient time to thoroughly discuss "Questions and Answers." I am confident these open forums will be informative, interesting, timely and useful. The questions to be discussed were picked by your Executive Committee from many submitted by our Round Table members, for importance and answers many of us are looking for to do a better job. Frank Nielsson will be Panel Leader. His Panelists are David W. Bixby, Allen S. Jackson, Robert E. Szold, C. R. Moebus, Harry L. Cook and Robert Heck. Frank needs no formal introduction. Most of you know "Our Slugger."

FRANK NIELSSON: Hi: Your Round Table Executive Committee are real "Eager Beavers." They are a good bunch of Guys. They contact you and convince you to accept the job, so here I am. Will my Panelists please come forward and take your seats around the table on the platform. Each Panelist has a question to answer which will be recorded in the Proceedings. However, as announced, "Questions and Answers" from the floor, will not be recorded. I suggest you take your pencil and paper to record some of the discussions from the floor of interest to you.

What Is The Effect Of Sulphuric Acid Strength On Ammoniator Performance?

David W. Bixby

IF we assume that concentrated sulphuric acid (93% = 66°Be) is introduced into an ammoniator only in an amount necessary to neutralize a given amount of ammonia, then it will contribute exactly as much heat to the total mass of material as does a chemically equivalent amount of sulphuric acid in a more dilute form (77.7% = 60° Bé). Dilute acid, however, dissapates some of this heat into its own water content so, per pound of 100% H_2SO_4, somewhat more heat will be available to the other materials when concentrated acid is used. Also, the liquid phase will be more effective because it is more concentrated, will crystallize more rapidly and the product will require less drying. On the other hand, the heat contribution of concentrated sulphuric acid may be further increased by heat of dilution if additional water is added in the ammoniator. Parenthetically we might note that per unit *weight,* concentrated acid contributes 15.3% more heat. Per unit *volume,* concentrated acid contributes 29% more heat.

Although there is a certain amount of extra heat contributed by using concentrated acid, (keeping in mind that we are comparing it with chemically equivalent dilute acid), this heat of itself is not the most important consideration when we are thinking about ammoniator performance (*as related to acid concentration*). The key word here, I feel, is *distribution.*

In the first place, one gallon of 60° Bé acid can be replaced by less than 0.8 gal. of 66° Bé acid, a 20% reduction in volume. With a sparger system already properly set up to deliver *dilute* acid, this means that there will be considerably less pressure at the orifices, more likelihood of blockage. With less water and therefore with less liquid volume, the increased heat effects have less opportunity to become distributed. Blockage of any of the outlets of course aggravates the problem. Excessive wetness in some areas at the expense of others then can lead to formation of plastic masses of material which further confine the heat, and then fume formation or fire may follow which of course are more serious when concentrated acid is being used.

I have no statistics on how many manufacturers use concentrated acid in ammoniators but I do know that many use 93% and some even use 98%. Some manufacturers have experienced flashing with 98% but not with 93%, others have had trouble with 93% and now prefer to stick to dilute acid exclusively.

Ideally, if a manufacturer intends to use, say 93% acid exclusively, he should set up his sparger so as to distribute it, and ammonia as well, through many smaller holes with great accuracy. However, there are practical limits to the size and number of holes which can be drilled in spargers. What should be kept in mind is that there is little margin for error in control when using concentrated acid and that trouble is more difficult to correct when it occurs.

Comparison of the effects of concentrated acid versus dilute acid on *granulability* is propably possible only when the formulation is taken into consideration also. Concentrated acid introduces less water, thus reducing plasticity, but, as previously mentioned, absence of this water can free more heat, creating more plasticity, and the two effects can conceivably neutralize one another. I know of no way to predict these effects other than by comparing dilute and concentrated acid experimentally.

I should like to repeat that accuracy of control and evenness of distribution are the key to effective use of concentrated acids but that the manufacturer must decide whether this extra effort is worthwhile in the light of his own particular needs.

What Kinds Of Spent Acid Are Suitable For Fertilizer Manufacture And Which Are Not?

David W. Bixby

SPENT acid is acid which has been used for another purpose and contains varying amounts of impurities. As far as I know spent *sulphuric* acid is the only one consumed to any extent by the fertilizer industry.

It is estimated that about 3.4 million tons of spent sulphuric acid was available in 1962, most of which was treated or reused as such by the operations which produced it. Approximately equal quantities were reused as such, decomposed and refortified. About 40% of the spent acid came from the petroleum industry, 30% from alcohols, 3-5% each from acrylic resins, petroleum catalysts, petroleum sulfonates, detergents, pigments and lesser amounts from dyestuffs and insecticides.

I. *Spent acid suitable for direct use in fertilizer manufacturing*

The spent acids suitable for use in fertilizer manufacture are considered to be those which contain only minor amounts of impurities and those which have not been diluted beyond the point of usefulness in at least some fertilizer process. The following fit into these categories, in addition to which they can be easily reclaimed by simple heat and vacuum or hot air blowing.

Alcohol spent acid

Spent acid from ethyl alcohol units averages 40-50% H_2SO_4 with only a small amount of organic material. Although too dilute to use directly in superphosphate manufacture it can be, and is, used directly in the manufacture of wet process phosphoric acid in at least two locations that I know of. Since alcohol spent acid is easily reclaimed, however, most of it is reused at the source.

Insecticides spent acid

Most of this acid comes from DDT manufacture and is also suitable for direct use, having been used merely as a dehydrating agent. It may contain some monochlorobenzene although this material will have been mostly removed by steam stripping. Any amount of contained chlorides can give rise to corrosion problems, however. The acid is still fairly concentrated, since 20% oleum was used to start with. There are about four basic producers of DDT in the U.S.A. although I do not know how many of them sell their spent acid for fertilizer manufacture.

Petroleum catalyst spent acid

The acid available from production types of petroleum catalyst is known to be suitable for direct use in ammonium sulphate and phosphate fertilizer manufacture.

Chemicals NEC spent acid

The most important source of acid in this category is that which is obtained from caprolactam manufacture. As far as I know it is recovered almost entirely as ammonium sulphate. Some is recovered as an ammonium nitrate-ammonium sulphate solution by virtue of using nitric acid to clean up the ammonical ammonium sulphate stream. The ammonium sulphate may be sold as such or mixed with an ammonium phosphate melt to produce 20-20-0 ammonium-sulphate-phosphate.

Acrylic resin manufacture also yields some spent acid, generally used to make offgrade ammonium sulphate.

Nitration spent acid

Some byproduct sulphuric acid is available from nitration operations (i.e. nitroglycerine), after it is separated from the nitric acid with which it was used. This source of spent acid is declining in importance. One reason is the partial replacement of nitroglycerine by ammonium nitrate. However, one man mentioned to me earlier this morning that his group is considering taking some spent acid from an analine plant yet to be built. This acid would be about 70% H_2SO_4 and presumably O.K. for direct use in wet process phosphoric acid manufacturing if no foaming problems are encountered because of the contained nitrogenous impurities, probably mostly nitrobenzene.

II. *Spent acids not suitable for direct use in fertilizers.*

Petroleum spent acid

Almost all sulphuric acid is recovered from petroleum refinery processes in the form of an acid sludge, not suitable for direct use in fertilizers. As is the case with the other spent acids, increasing amounts are being regenerated by the original users or the producers which in some cases are fertilizer companies having their own sulphuric acid plants.

Acid sludges may range all the way from 20% to 90% H_2SO_4, and from less than 1% to 60% hydrocarbons. Sulphonates and water are also present. Certain sludges, notably the paraffin based types, may be separated into two layers by hydrolysis, the inorganic layer being a dilute "black" sulphuric acid which, where the market situation is adequate, may be reacted with ammonia to produce fertilizer grade ammonium sulphate. The ammonium sulphate is heat treated to decompose organic impurities, and recrystallized to get rid of the remaining carbon.

There are several other acid sludge treatments which involve (a) the use of the organic hydrolysis product, or the sludge itself, as a fuel which destroys the sulphuric acid; (b) reconcentration of the hydrolysis acid mentioned previously, usually to 66° Bé before reuse in the refinery; (c) high temperature decomposition and reduction, producing SO_2 which may be reoxidized to form pure sulphuric acid, suitable for any use.

Alkylation acid, obtained from reactions where sulphuric acid serves as a catalyst, normally contain 85-90% free sulphuric acid with 5-8% carbon content. Usually, refineries make further use of it and it ends up as a sludge also. However, some finds its way to the fertilizer via burning in lead chamber sulphuric acid plants associated with normal superphosphate operations. Direct use of alkylation acid in superphosphate manufacture and wet process phosphoric acid manufacture has been tried experimentally but with unsatisfactory results, mainly because of odors generated.

Benzene spent acid

This acid is not available in large quantities, but is similar to an intermediate oil refinery sludge and is usually recovered by hydrolysis or used to make ammonium sulphate.

Spent acid from *Pigments and Pickling.*

These acids are too dilute for use in fertilizer process. Those from a titanium pigment operation may contain from 9-15% H_2SO_4 and 4-10% $FeSO_4$. Pickle liquor contains the same materials but in an even more dilute form. Reprocessing, if any, is accomplished by the manufacturers primarily to solve a waste disposal problem, in order to avoid air and water pollution.

Flight Designs For Dryers And Coolers

Allen S. Jackson

THE purpose of a flight is to lift material and permit it to fall through the heating gas in a dryer or the cooling gas of a cooler. Because the material dries or cools as it falls through the air stream, the flights must be designed to drop the material across the entire cross section of the cylinder being used as a dryer or cooler. The material should also be dropped in a uniform pattern across the cross section.

The rate of material fall is proportional to the length of the surface of material exposed. Figure 1 shows the amount of material that will fall as the flight moves through angle A as compared to the amount that will fall as the flight moves through angle B.

In fertilizer design we are always compromising between efficiency on one hand and cost and practicality on the other. Flights are a compromise also.

Figure 2 shows the theoretically perfect flight, a circular section with a top to it. The top of the figure shows the flight in various

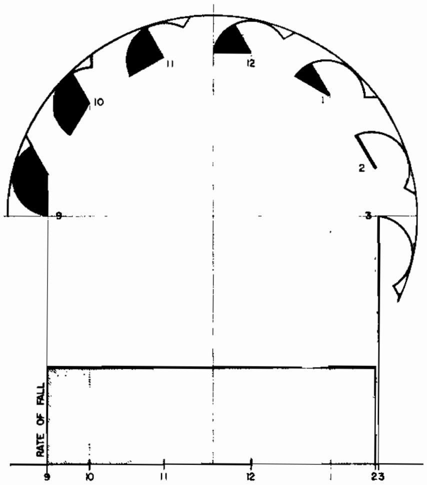
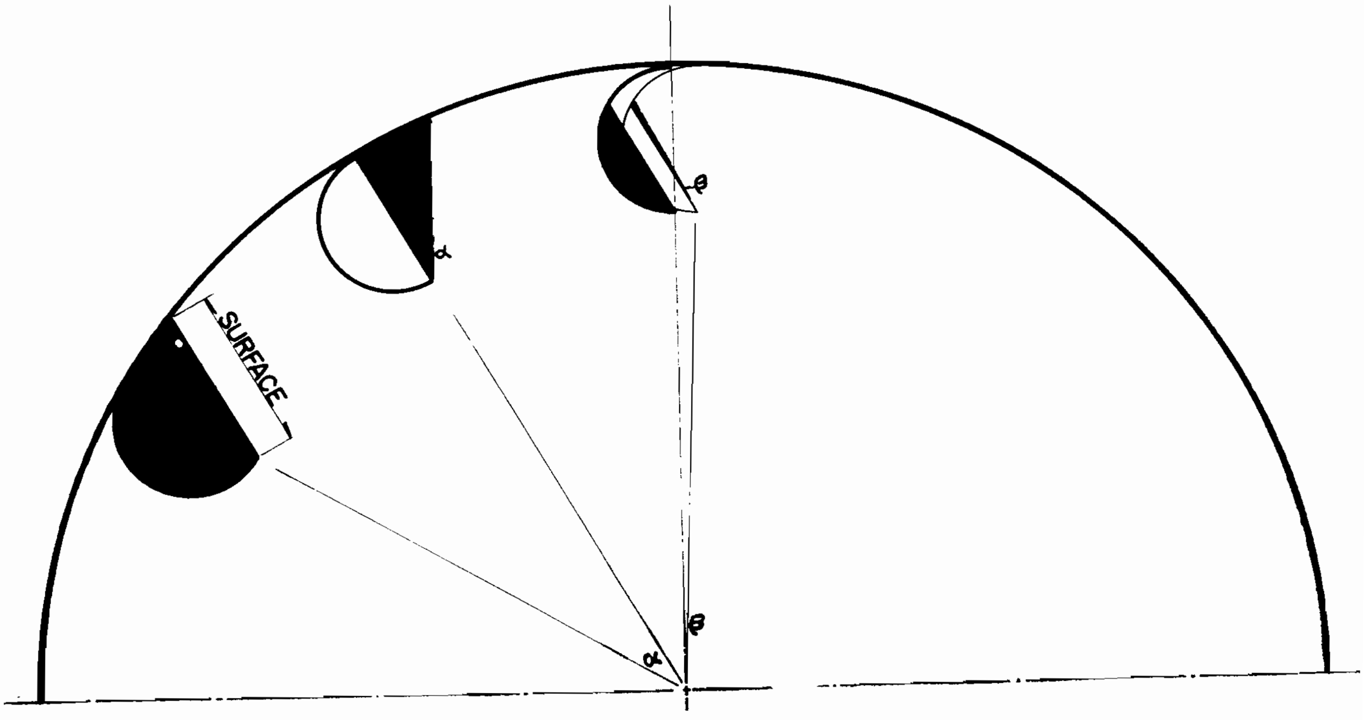


Figure 2

Figure 1

RATE OF MATERIAL FALL AT ANY INSTANCE IS RELATIVE TO LENGTH OF SURFACE



positions as the cylinder rotates expressed as hours hand position on a clock face, while the bottom of the figure shows the rate of fall of the material from the flights as the cylinder rotates. The rectangle indicates that a uniform curtain would extend across the cross section of the cylinder.

In Figure 3, we show a flatbar flight on a radius. The bottom of the figure shows that all the material has been dropped by the time the cylinder passed 11 o'clock (clockwise rotation).

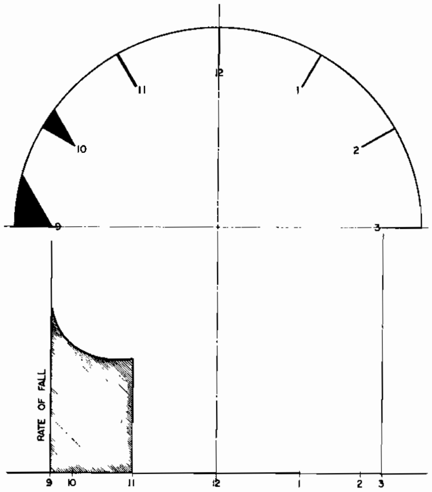


Figure 3

In Figure 4, we have taken a channel as a flight. The data indicate that most of the material falls in the first quarter of rotation with a decreasing ratio fall from 12:00 to 3:00 o'clock.

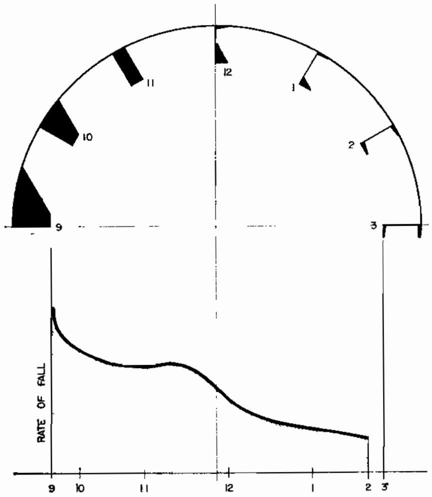


Figure 4

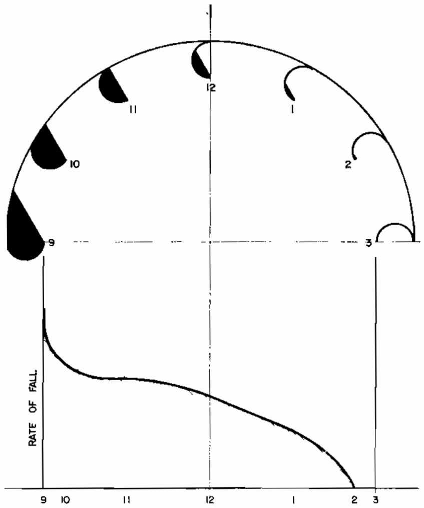


Figure 5

In Figure 5 we have taken a half circle flight, (split pipes) on a radius. The flight pattern is only slightly better than that found with the channel in Figure 4.

If the half circle were rotated slightly to extend the inward lip above the radius, better distribution would result.

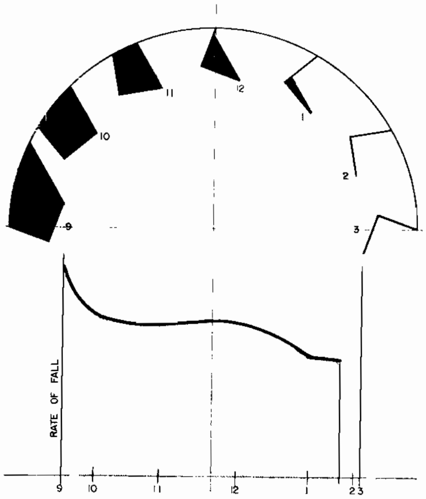


Figure 6

In Figure 6 we show an angle set below the radius. Although a substantial portion of material falls between 9:00 and 10:00 o'clock, the remaining rotation provides a rather uniform rate of fall across the cylinder cross section.

In Figure 7 we show an angle set on the radius. This shows the most uniform distribution pattern of all shapes examined except for the theoretical. When one considers ease of fabrication and availability, the angle design of Figure 7 appears to be the best compromise available for a practical dryer or cooler flight in a fertilizer plant.

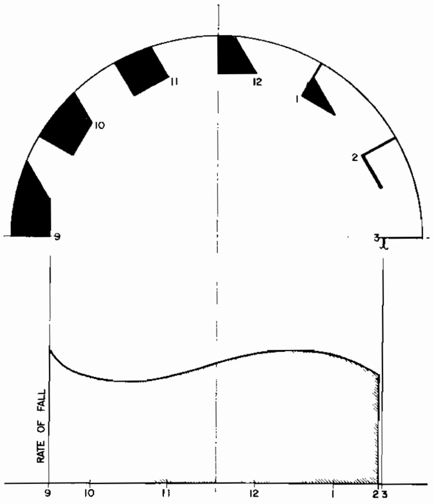


Figure 7

Figue 8 shows the plastic model used to demonstrate the various flight designs dynamically.

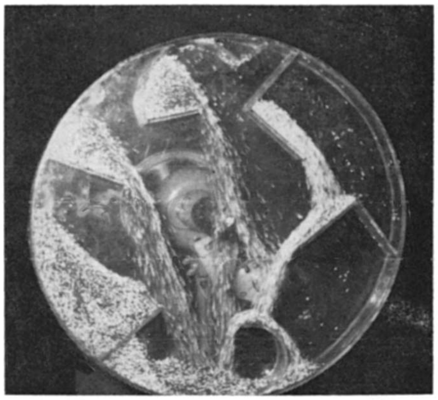


Figure 8

Spargers

Figure 9 shows an early sparger design in which a flexible rubber sheet was wrapped around a

Figure 9

drilled solution pipe, another one was wrapped around a drilled acid pipe, the entire assembly was bolted together and installed in the ammoniator. It was believed that the rubber lips would provide non-clogging spargers. This belief became a fact. Figure 10 shows the spray pattern for this sparger. At high rates of flow the pattern in the ammoniator was like that shown in Figure 10. However, at low rates of flow, the stream might come out at any place along the sparger.

Figure 10

In an effort to assure equal distribution some tests were made to determine how large and how many holes are needed for spargers.

Figure 11 shows the simple homemade setup where a pipe having a 1/16 inch hole and a 3/32 inch hole was made to discharge a water stream into separate buckets. The rate of discharge was measured through each hole as a function of the water pressure in the pipe.

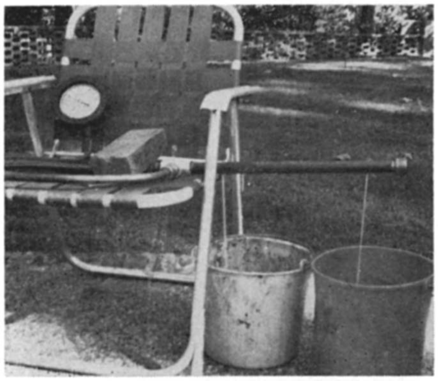


Figure 11

Figure 12 shows a plot of the data obtained. The data indicate why we prefer to use sparger pipe with a large number of 1/16 inch holes rather than a pipe with fewer large holes.

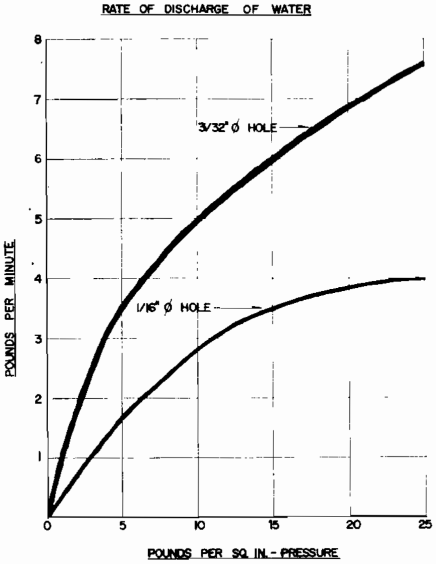


Figure 12

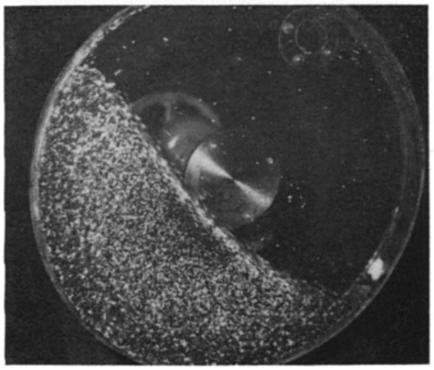
High velocity is needed to keep the holes from plugging. Using small holes permits higher pressures and higher velocities. Over the range of 5 to 20 pounds, the flow through the 1/16 hole varied from 1.5 to 3.9 lb. per minute. Over the same range of pressure, the flow through the 3/32 inch hole varied 3.5 to 7 lbs. per minute.

Where should sparger pipes be placed?

We have built a small plastic model to simulate a cross section of a continuous rotary ammoniator. It is powered by a variable speed sewing machine motor. It contains pellets in three size groups; red, about 6 mesh; white, about 10 mesh; and blue, about 16 mesh.

As the cylinder rotates you can see that classification occurs, with the larger pellets distributing themselves throughout the bottom and the top of the bed. The small pellets remain pretty well in an area near the top center of the bed, but below the stream that is flowing downhill. (Figure 13)

Figure 13



You can see that if you spray water on the top of the bed you will wet the large particles only, and tend to make them larger.

If you want to wet the fines for agglomeration, the water sparger should be placed at about 7:30, for clockwise rotation and about 6 to 8 inches under the surface of the bed.

If a cylinder were being used for granulation alone, say in 0-20-0, or 0-20-20, the preferred setup would be a series of short water spargers spaced at intervals under the bed as described above. This arrangement would cause the fines to become wetter, they would agglomerate and become classified, and in the next section the remaining fines would become wetter, etc.

By the same token, as granulation proceeds along the length of an ammoniator, the amount of fines decreases and their axis of location comes closer to the top. Therefore, it is logical to assume that the ammonia and acid spargers should be slanted relative to the bed surface to permit the fines the greatest exposure to the ammoniating solution.

Dry Nitrogen Solutions

Robert E. Szold

THE term "dry nitrogen solutions" as used in this presentation refers to nitrogen solutions containing 0.5% maximum water. These solutions were introduced commercially in 1960 by Commercial Solvents Corporation under the brand name DRI-SOL®.

Charts have been provided which show the properties of DRI-SOL nitrogen solutions and DIXSOL® nitrogen solutions, CSC's conventional nitrogen solutions which contain 6 to 18% water. Table I shows some of the properties of a dry nitrogen solution, DRI-SOL 466 (25-75-0), and a conventional solution DIXSOL 440 (24-70-0). These solutions were

Table 1. Properties of Solutions

	Dri-Sol Solution 466(25-75-0)	Dixsol Solution 440(24-70-0)
Total Nitrogen, % by Wt.	46.6	44.0
Composition, % by Wt.		
Ammonia, Free	25.0	23.8
Ammonium Nitrate	74.5	69.8
Water	0.5	6.4
Ratio Free Ammonia Nitrogen To Total Nitrogen	1:2.268	1:2.248
Percent Free Ammonia Nitrogen of Total Nitrogen	44.09	44.48
Properties		
Approx. Sp. Gr. at 60/60°F	1.150	1.147
Lb Per Gal at 60°F	9.58	9.55
Vapor Pressure at 104°F, PSIG	29	18
Vapor Pressure at 60°F, PSIG	1	26
Approx. Crystallization Pt. °F	5	26

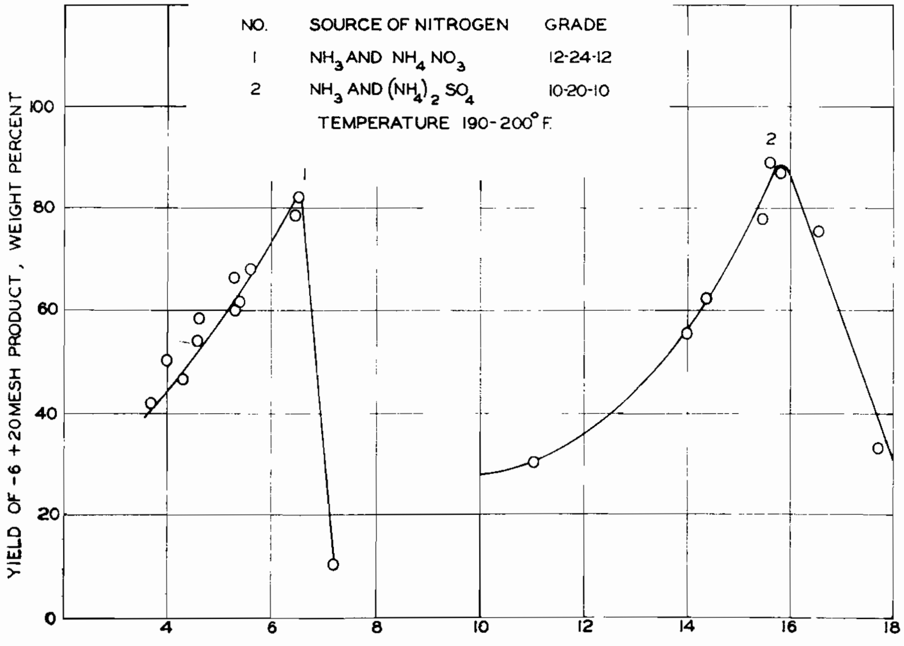
selected because they have similar ratios of ammonia nitrogen to total nitrogen. The lower water content of the dry nitrogen solution increases the total nitrogen from 44% to 46.6%. The crystallizing point of the dry nitrogen solutions is 21°F lower than that of the conventional solution. Dry nitrogen solutions are shipped at temperatures near 60°F. At 60°F the vapor pressure of this dry solution is 1 psig and the solution weighs 9.58 lb/gal compared to 0 psig and 9.55 lb/gal for the conventional solution. The viscosities of dry nitrogen solutions are similar to those of conventional solutions. When calibrating flow meters, only differences in specific gravity need be considered.

The dry nitrogen solutions offer many advantages for the production of both granulated and pulverized fertilizers. These advantages will be illustrated by a number of examples selected from the experiences of fertilizer manufacturers who have used these solutions.

Figure 1 taken from an article by Hardesty, et al.,[1] illustrates the importance of moisture control to obtain maximum yield of "on-size" particles. In the first example, 12-24-12 was formulated with ammonia and ammonium nitrate as the sources of nitrogen and was granulated at 190-200°F. The maximum yield of 82% product size (—6+20 mesh) particles was obtained when the moisture content of the mix was 6.5%. A slight increase in moisture to 6.9% caused over-agglomeration, and reduced the yield of product-size granules to 12%. The second example illustrates the behavior of 10-20-10 formulated with ammonia and ammonium sulfate, and granulated at 190-200°F. Ammonium sulfate is not as soluble as ammonium nitrate and more moisture is required to granulate mixes containing ammonium sulfate. In this case, a yield of 89% "on-size" particles was obtained at an appreciably higher moisture content of 15.8%. As moisture content was increased to 17.5% the yield of product-size particles dropped to 35%. However, the shape of the curve for 10-20-10 is similar to that for the 12-24-12. These graphs show that moisture must be controlled within narrow limits. The optimum moisture content varies, not only with different raw materials, but also with changes in temperatures, and with the production of different grades.

FIGURE I

VARIATION IN AGGLOMERATION WITH MOISTURE CONTENT OF 1:2:1 RATIO FERTILIZERS



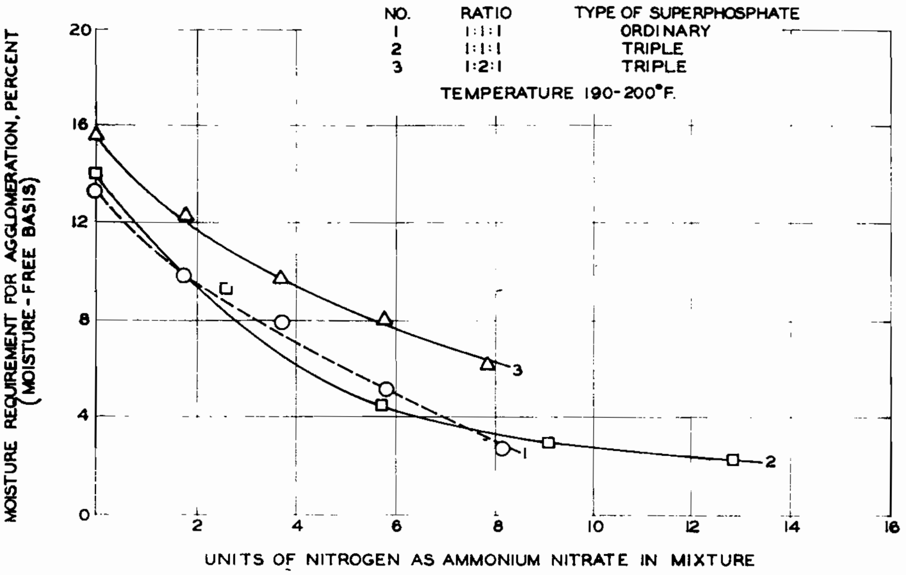
HARDESTY, J.O., ET. AL. J AGR. FOOD CHEM., 4, 60-63. (1956)

The specific formula used and the moisture contents of the raw materials determine the minimum moisture content of a mix. If more water is needed to obtain maximum yield of "on-size" granules, the operator can always add water. Adjustment of water addition is the simplest means of controlling granulation. However if excess water is present in the ingredients and over-granulation is occurring, control by water addition is obviously impossible and it is necessary, therefore, to increase recycle rate.

The use of dry nitrogen solutions affords a simple and positive method of controlling granulation conditions without using excessive recycle which inevitably reduces plant capacity. Table 2 shows typical formulas for the production of 12-12-12. Assuming the ordinary superphosphate contains 8% moisture and the triple 4%, there will be 96 lbs. of water/ton when a 440 (24-70-0) solution is used. Fertilizer produced by this formula

FIGURE 2

EFFECT OF AMMONIUM NITRATE CONTENT ON MOISTURE REQUIRED FOR AGGLOMERATION OF MIXED FERTILIZERS



HARDESTY, J.O., ET. AL., J AGR. FOOD CHEM., 4, 60-63 (1956)

normally granulates easily without the production of excess "off-size" granules. However, if the moisture contents of the raw materials are high, over-granulation results and it becomes necessary to increase recycle rates.

When using the equivalent dry nitrogen solution, 466 (25-75-0), under normal conditions the operator must add an additional 27 lbs. of water per ton to get the same amount of granulation which occurs without the addition of water when using solution 440. This gives greater latitude for adjustment in water flow rate before it becomes necessary to make a change in recycle rate.

The moisture content of raw materials usually varies from one section of a pile to another. This can lead to severe control problems. The use of dry nitrogen solutions generally makes it possible to minimize control problems by changing water flow instead of changing recycle rate.

At a plant in Iowa, production rates using a 440 (24-70-0) solution were 12 tons per hour. Using dry nitrogen solution 466 (25-75-0), production rates were increased to 15 tons per hour. Assuming that conversion costs were $12.00/hour, this increase in capacity reduced these costs by 20¢ per ton. This 25% increase in production capacity becomes even more important during the peak season when profit on the extra production can be realized. At a plant in Louisiana, using a pug mill mixer, the use of DRI-SOL solutions led to a 20-50% increase in production capacity depending upon the grade being manufactured.

In order to reduce formulation costs, it is desirable to use as much solution nitrogen and ordinary superphosphate as possible. However, the use of too much solution can result in excessive liquid phase in a mixture. Ammonium sulfate is used in place of solution nitrogen to reduce liquid phase and prevent over-granulation. Formulation costs increase as more ammonium sulfate is used as ammonium sulfate is usually more expensive per unit of nitrogen than ammoniating solution. Also ammonium sulfate's low analysis leaves less room in the formula for superphosphates and more of the higher-priced triple superphosphate must be used[2]. When a dry nitrogen solution is used, the amount of moisture in the mix is reduced allowing the use of more solution nitrogen and less of ammonium sulfate. In a grade such as 12-12-12, it is easy to get at least one more unit of nitrogen from dry nitrogen solution compared to using conventional solution.

Table 3 shows the advantage of a dry nitrogen in reducing raw material costs for a 12-12-12. Prices used in this example were:

Table 3. Formulas and Raw Material Costs for 12-12-12

Raw Materials	Formula Using 440 (24-70-0) Lbs./Ton	Cost $	Formula Using 466 (25-75-0) Lbs./Ton	Cost $
Nitrogen Solution	455	13.92	481	15.53
Ammonium Sulfate 21-0-0	209	4.08	95	1.85
Ordinary Superphosphate 0-20-0	546	4.64	606	5.15
Triple Superphosphate 0-46-0	300	8.62	274	7.87
Muriate of Potash 0-0-61	400	7.88	400	7.88
Sulfuric Acid 93%	156	1.56	189	1.89
Total	2,066	40.70	2,045	40.17

Table 2. Formulas for 12-12-12

Raw Materials	Material Usage Lbs./Ton		Contained Water Lbs./Ton	
Nitrogen Solution 440 (24-70-0)	455	—	29	—
Nitrogen Solution 466 (25-75-0	—	428	—	2
Ammonium Sulfate 21-0-0	209	209	0	0
Ordinary Superphosphate 0-20-0 (8% H_2O)	546	546	44	44
Triple Superphosphate 0-46-0 (4% H_2O)	300	300	12	12
Muriate of Potash 0-0-61	400	400	0	0
Sulfuric Acid 93%	156	156	11	11
Total	2,066	2,039	96	69

Raw Material	Dlvd. Price Per Ton, $
Solution 440 (24-70-0), $1.30 per unit plus $4.00/ton Frt.	61.20
Solution 446 (25-75-0), " " " " " " "	64.58
Ammonium Sulfate, 21-0-0	39.00
Ordinary Superphosphate, 0-20-0	17.00
Triple Superphosphate, 0-46-0	57.50
Muriate of Potash, 0-0-61	39.40
Sulfuric Acid, 93%	20.00

Delivered price of solution 440 $1.391/unit
Delivered price of solution 466 $1.386/unit

Net savings on nitrogen in the formula using the dry nitrogen solution would be 29¢/ton. There would be an additional savings of 24¢/ton as a result of substituting 0.6 unit of ordinary superphosphate for triple superphosphate. The total savings in cost of raw materials would be 53¢/ton. In the production of a 16-20-0 it is easy to get at least two more units of nitrogen from dry nitrogen solution and savings in the cost of raw materials, based on the above prices, would be $1.26/ton. Some users of dry nitrogen solutions have reported even further savings by supplying all of the nitrogen from solution in grades such as 12-12-12 and 16-20-0.

In the preceding examples, DRI-SOL solution 466 (25-75-0) was compared to the conventional 440 (24-70-0) solution because these solutions have essentially the same ratio of ammonia nitrogen to total nitrogen. Additional small savings are possible if DRI-SOL solution 462 (24-76-0) is used. This is due to a reduction in the amount of sulfuric acid required to neutralize free ammonia. Naturally, total savings possible in the cost of raw materials by using the dry nitrogen solutions will change from plant to plant since the delivered costs of raw materials are different at every plant.

Dry nitrogen solutions are being used to make grades such as 15-15-15 and 20-10-5 without the use of a preneutralizer. Typical formulas are shown in Table 4.

The use of dry nitrogen solutions can reduce drying costs. In Figure 2, data published by Hardesty, et al.,[1] are reproduced showing that the moisture required for granulation can be reduced by increasing the ammonium nitrate content of the fertilizer. In a 1:1:1 ratio made with ordinary super, and containing four units of nitrogen as ammonium nitrate, 7% moisture was required for agglomeration. Where there were 8 units of nitrogen as ammonium nitrate, only 3% moisture was required. In a 1:1:1 ratio made with triple superphosphate 6% moisture was required with 4 units of nitrogen from ammonium nitrate compared to 3.5% moisture with 8 units.

In the example of 12-12-12 formulas shown in Table 3 there are 5.56 units of nitrogen as ammonium nitrate in the mix made with solution 440 (24-70-0), and 6.27 in the mix made with dry nitrogen solution 466 (25-75-0). As there is also additional ammonia in the formulation with dry nitrogen solution, the heat of reaction and temperature of mixing are raised. Both the increased concentration of ammonium nitrate and the higher mixing temperatures reduce the moisture required for granulation. With less moisture in the feed to the dryer, less fuel is required to dry the fertilizer to a given moisture content, and drying costs are reduced. As an example, at one plant the moisture content of the ammoniator discharge averaged 4% when using conventional 440 (24-70-0) solution and 2.9% when using DRI-SOL solution 462 (24-76-0).

Table 4. Formulas for the 15-15-15 and 20-10-5

Raw Materials	Grade	
	15-15-15 Lbs./Ton	20-10-5 Lbs./Ton
Dri-Sol Solution 462 (24-76-0)	497	368
Ammonium Sulfate 21-0-0	354	0
Ammonium Nitrate 33. 5-0-0	0	701
Ordinary Superphosphate 0-20-0	216	352
Triple Superphosphate 0-46-0	0	297
Phosphoric Acid 0-54-0	489	0
Muriate of Potash 0-0-61	498	171
Sulfuric Acid 93%	36	130
Total	2,090	2,019

Drying capacity is the bottle neck at some plants. If heat input is limiting, the only way to dry a fertilizer with excess moisture is to decrease plant through-put which increases operating costs/ton. If heat input is not limiting, higher than normal drying temperatures are often tried. Excessive temperature can make the particles soft and sticky and cause over-agglomeration in the dryer. The particles will accumulate and fill up the flights, lowering dryer efficiency. When dry nitrogen solutions are used, the feed to the dryer contains less moisture and these difficulties are minimized.

At many plants where dry nitrogen solutions are used, the residual heat in the ammoniator discharge is high enough, and the products dry enough, so that little or no additional heat is required for drying. The dryer can then be used as a supplemental cooler. Fertilizer handled this way holds up well during storage. Obviously, drying costs are at a minimum.

The dry nitrogen solutions are also of value in the production of pulverized fertilizers. When producing pulverized fertilizers with dry nitrogen solutions, mixing temperatures of 189-190°F are common compared to 150-160°F with conventional solutions. The higher mixing temperatures aid in completing the reaction in the mixer and reduce the amount of reaction which must occur in the pile. Curing time is thus reduced. Manufacturers of bases such as 8-16-0 take advantage of the reduced curing requirement and use the base 2-3 days after it is laid down, compared to two or more weeks required for curing a base made with conventional solution.

In pulverized grades such as 6-12-12 the replacement of conventional solution with DRI-SOL solution reduces the moisture content of the mix by 16 pounds per ton. Lower moisture in the raw materials and higher reaction temperatures both contribute to a dryer product with excellent storage characteristics.

Many manufacturers of pulverized fertilizers have reported decreased citrate insoluble phosphates when dry nitrogen solutions are used. Perhaps this is due to the combination of a drier product and less reaction in the pile.

If extremely dry superphosphates are used with nitrogen solutions, the product may be dusty and ammonia efficiencies low. These problems can easily be overcome by spraying small amounts of water into the mixer or adding water directly to the nitrogen solution. No appreciable thermal effect is noted when water is added to the solution.

No special problems are encountered in handling DRI-SOL solutions. Since they are shipped cool, the vapor pressure is low. If tanks are accidently vented, ammonia loss would be no greater than for conventional solutions of similar free ammonia content. Any type of solution standing in steam-traced lines will become warm and the vapor pressure will increase. If there is a slight leak in the line, ammonia will evaporate and salting out may occur. For this reason, it is recommended that all lines and, particularly, steam-traced lines be emptied when the plant is shut down. Frankly, we at CSC recommend this precaution for handling all solutions.

The dry nitrogen solutions help the fertilizer manufacturer select the moisture level needed to do the best overall job in any particular situation instead of having to compromise optimum formulas and/or operations to accommodate the higher water level of conventional nitrogen solutions. It is a simple matter to add water as required but if too much moisture is present it can often be a critical problem to get rid of it.

To summarise, many fertilizer manufacturers are finding that dry nitrogen solutions provide one or more of the following benefits:

1. Better Process Control
2. Increased Production Rates
3. Decreased Drying Costs
4. Decreased Conversion Costs
5. Decreased Raw Material Costs
6. Quicker Curing
7. Improved Product Quality

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(2) Hignett, T. P., "General Questions of Operating Techniques, Equipment, and Practices in Manufacturing of Granular Mixed Fertilizers," *Chemistry and Technology of Fertilizers,* edited by Dr. Sauchelli, pages 269-298, (1960).

Anti-Caking Agents

C. R. Moebus

BEFORE embarking on details concerning the use of anti-caking agents in the mixed fertilizers industry, perhaps a little background on our entry into the field of producing anti-caking agents is in order. Back in 1958 and 1959 we were trying to interest a major AN producer in the use of a South Carolina kaolin to prevent caking of prilled AN. Tests indicated that kaolin alone was only partially effective in providing the required protection. At the same time this producer was studying the effect of a product known as Petro Ag Special with approximately the same results. One of the chemists involved asked us what the effect would be if the two materials were combined. Our response, was that we would be happy to prepare the mix if they would test it—and so our company started on the road to producing a very effective anti-caking agent for use in the AN industry, known as AC Clay—or, as our products have subsequently become known, the AC series.

At the suggestion of several AN producers we made contacts in the mixed fertilizer industry where the caking of high nitrogen granular mixes was reported to be a problem. It was reasoned that—if Clay-Petro Ag combinations could effectively condition AN, then they should logically be able to condition high nitrogen granulars.

Late in 1960 and early in '61 I was assigned the job of contacting the fertilizer industry to determine their needs and gather as much information as possible on the subject of making in the high nitrogen granular mixtures. Calls revealed that commonly used anti-caking agents were:

(1) Diatomaceous earth
(2) Kaolin
(3) Cocoa shells
(4) Peanut hulls
(5) Vermiculite
(6) Limestone
(7) Cotton seed hull flour
(8) Magnesium oxide
(9) Phosphate rock
(10) Miscellaneous inexpensive powders

No pattern seemed to exist regarding what was used on what product.

In the course of this survey I called on Mr. Spillman who advised me that if a plant is producing a high nitrogen granular under the optimum conditions of granulation, drying, cooling, and sizing, the probability of requiring a conditioner to prevent caking would be slight. He pointed out that usually production conditions were something less than optimum for a variety of reasons. He also advised that probably it would be difficult to develop a pattern from plant to plant. He was so right.

He also advised that most producers would be reluctant to condition unless it was absolutely necessary because of the added cost. Here again he was so right!

However, we did find a few

companies who had problems and who were willing to evaluate the possible benefits derived from surfactant treated kaolins. In some instances one or more of the conditioning agents mentioned previously were in use. Others were not conditioning at all. Tests conducted were the usual 80 pound bagged samples stacked 15 high and torn down every 30 days for the duration of the test. Samples of regular production material were simultaneously stacked and torn down for comparison. The quantity of caked goods was determined in many instances by screening through a ¼ inch mesh screen and weighing the quantity of fertilizer in lump form retained on the screen. Typical test time involved ranged from 30 to 180 days. In most cases the application rate was from 1 to 2 percent but in isolated instances up to 3 percent was used.

We prefer to have tests run in units where a conditioning drum is an integral part of the system. However, for test purposes, should no drum be available, a portable gasoline or electric powered cement mixer can usually be rented and the various test quantities handled on a batch basis. The cement mixer will insure good distribution of conditioner applied at the rate of 20 to 60 pounds per ton of granular fertilizer.

Successful tests results have resulted in a number of fertilizer producers using surfactant treated clays to condition:-

10-10-10
12-12-12
15-15-15
16-8-8
20-0-20
14-0-14
30-10-0
29-14-0

and I'm sure there are other combinations which are difficult to keep in shape.

As previously mentioned, we believe the optimum anti-caking protection occurs where the conditioning agent is introduced to the fertilizer in a conditioning drum. However, many plants do not have this facility and have instead introduced the material into screw conveyors moving the product from the cooler to the pile—or from the pile to the bagger bin. The more tumbling that can be affected the greater the opportunity for the conditioning agent to adhere to the granule—and theoretically the better the chances for good anti-caking protection.

Feeder mechanisms used to introduce the conditioning agent include

Vibra Screw Feeder
Jeffrey Weightrol
Syntron Vibratory Feeder
Miscellaneous screw feeders
and/or

any metering devices which will permit the conditioner to be introduced to a known continuous flow of fertilizer at a given rate. Devices which are adjustable are best because there is sometimes a need for conditioners of various densities to be employed in one plant. For example—Diatomaceous Earth with a density of 9 to 12 pounds per cubic foot may be used interchangeably with an AC clay having a density of 40 pounds per cubic foot.

There are presently seven different AC clays produced commercially. Each is the same base clay with various percentage additions of Petro Ag Special. For high nitrogen granular fertilizers we recommend AC-3 or AC-4, used at the 1 to 2 percent application rate. AC-3 clay contains approximately 98.75 percent clay and 1.25 percent Petro Ag Special. AC4 contains 99.25 percent clay and 0.75 percent Petro Ag Special.

At this point it is probably well to dwell for a moment on the effect of the surfactant addition to a kaolin with regard to its anti-caking capabilities. There are many theories concerning why materials high in ammonium nitrate want to cake. Perhaps the most logical one is that ammonium nitrate being a highly hydroscopic material, tends to draw water from the atmosphere to its surface, thus each granule of fertilizer probably is enveloped with a very thin film of water. It is reasoned that this water film dissolves the readily soluble AN and then temperature and/or humidity changes result in the dissolved salts coming out of solution, recrystallizing, with caking the result. The surfactant addition to kaolin makes this film of water wet the clay more readily. Everyone knows how tenaciously clay sticks to a dampened surface. Therefore, the combination of clay, plus surfactant, adheres more readily to the slightly wet surface of the granule, thus imparting more effective protection from caking. In effect, the mechanism is purely mechanical with the film of kaolin interrupting the crystal growth between granules, which normally is ascribed to the cause of caking.

You may be interested in cost of mineral conditioners delivered to various sections of the country:

	AC—Clay (#4)	Airfloated Kaolin	Diatomaceous Earth
Florida	$32.86	$20.67	$52.00-$53.00
Iowa	$38.67	$26.86	$48.00-$49.00
North Carolina	$33.53	$20.29	$52.00-$53.00
New Jersey	$37.28	$25.24	$52.00-$53.00

Therefore, you can see that in most areas of the country the delivered cost of a conditioning agent will run between .01¢ to .025¢ per pound, depending on the type chosen. When applied to granular fertilizer at the rate of 20 to 40 pounds to the ton, your cost will range from $.20 to $1.00 per ton for conditioning. In return you will gain in these areas:

(a) Freedom from pile set and attendant difficulties in moving the material from the pile as well as possible breakdown of granules from pile set.

(b) Freedom from bag set and complaints of your customers.

(c) Less returned merchandise with costly reprocessing and complete loss of the bag in which the material was originally packaged (estimated at approximately 10¢-11¢ per bag or $2.50 to $2.75 per ton), to say nothing of the irritation of your customer.

With the marketing patterns of your industry changing, many of you have had to store bagged goods in warehouses near the site on which the fertilizer will be used. This warehousing, in advance of spring demand, has been the source of much returned goods because of poor condition. Anti-caking protection in instances like these is a virtual must.

Finally we have recently had reports from bulk blenders who have found it advantageous to use 1 to 2 per cent conditioner to not only keep their product in shape, but to also provide a uniform color to their fertilizer, thus masking the different colors of the base materials they blend. Evidently the farmer, in some cases, prefers to have a fertilizer that appears uniform in color rather than a blend of several colors which may lead to questions concerning the blend uniformity.

Factors Involved In And Methods Of Measuring Shrink In A Fertilizer Plant

Wayne H. Shidaker and Harry L. Cook

IN any discussion of shrink, we are dealing with a subject that some people in our industry hate to discuss and regard as a necessary evil; and in all probability, some shrink is. The question being how much. Traditionally, our experience with the old pulverized plants was about two per cent, and this was more or less accepted and ignored. When we went to granulation, we expected losses to be considerably higher and finally budgeted 4.5% shrinkage the first year. Actually, our dollar shrinkage for our first year of operation was 5.43%. Unfortunately, we had no information as to where this shrinkage occurred.

In studying this whole problem of shrink, definitions, of course, are necessary. We had established that our shrink is the difference between material received and materials shipped including, of course, any change in internal plant inventory. In addition to this, we recently began to consider these on a unit of plant food basis. It does not matter too much how you measure as long as it is possible to keep proper records. These are very difficult and the unit basis eliminated most other sources of error.

There are, of course, many definitions of shrinkage in various industries, and some such as loss of value, loss of volume, that are applicable there, are not applicable in fertilizer manufacturing, since the value of plant food ingredients, if properly processed, is the same when sold as when received and the volume is purposely changed.

Table 1. Points of Shrinkage

1. Receiving
   Shipper—Plant
2. Storage (Materials)
3. Processing
   a. Ammoniation
   b. Granulation
   c. Drying
   d. Cooling
   e. Dust Collecting Systems
4. Storage
5. Shipping
   a. Bulk Scales
   b. Bag Weights

The first point of loss is in receiving of raw materials into the plant. The first thought is that the supplier did not ship the weight he billed. We spot check our materials; however, our first year of study on this when we routinely weighed incoming materials gave us a very definite indication that this shortage is relatively rare and did not make a real contribution to our shrink.

The remaining items in Table 1 are those points where losses can occur and will be discussed in the remainder of this paper.

I am sure you have all many times seen piles of fertilizer along the tracks, along your siding, and in the yards. I am sure you have also seen the piles of materials at the receiving docks.

Photo 1

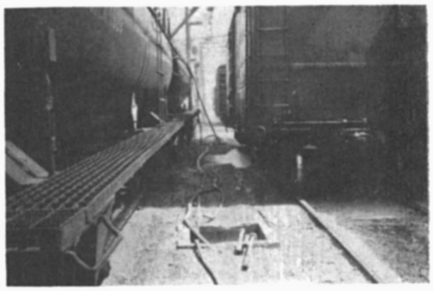


Photo 2

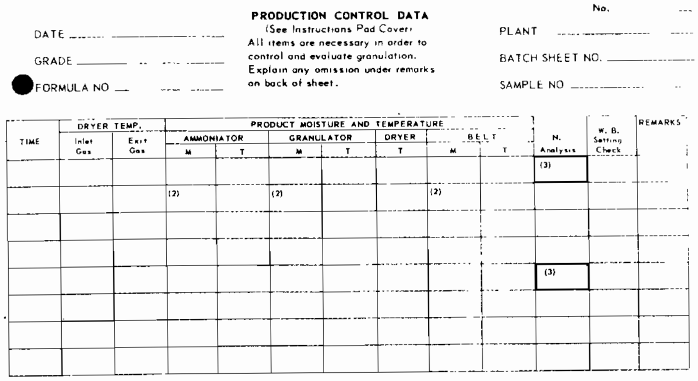
Even tank cars can sometimes leak, and I have even heard of cars that were only partially unloaded.

Photo 3

Plant people used to handling large volumes of bulk material sometimes do not realize the real value in a small pile of material dockside.

A second point of loss can be in the storage of raw materials. The dust from some materials from around the plant and materials inadvertently placed in the wrong storage can contribute in this area.

Now, let us consider manufacturing losses (processing). When we went to granulation, we were very much in the opinion that this would be an area of considerable loss, and as we look in the possibilities, I am sure that it is one area that needs constant attention to prevent a high loss. In studying this area, we set up a Production Control Data sheet.



No.

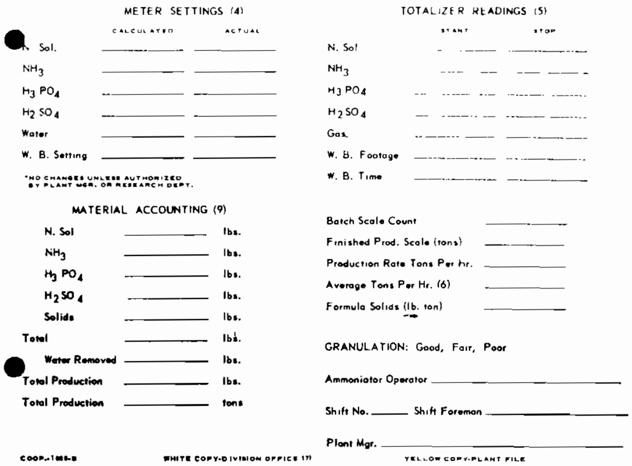
PRODUCTION CONTROL DATA
(See Instructions Pad Cover)
All items are necessary in order to control and evaluate granulation.
Explain any omission under remarks on back of sheet.

DATE
GRADE
FORMULA NO

PLANT
BATCH SHEET NO.
SAMPLE NO

TIME	DRYER TEMP.		PRODUCT MOISTURE AND TEMPERATURE							N. Analysis	W. B. Setting Check	REMARKS
			AMMONIATOR		GRANULATOR		DRYER	BELT				
	Inlet Gas	Exit Gas	M	T	M	T	T	M	T			
										(3)		
			(2)		(2)			(2)				
										(3)		

Photo 4



METER SETTINGS (4)	CALCULATED	ACTUAL
N. Sol.		
NH_3		
H_3PO_4		
H_2SO_4		
Water		
W. B. Setting		

*NO CHANGES UNLESS AUTHORIZED BY PLANT MGR. OR RESEARCH DEPT.

TOTALIZER READINGS (5)	START	STOP
N. Sol		
NH_3		
H_3PO_4		
H_2SO_4		
Gas.		
W. B. Footage		
W. B. Time		

MATERIAL ACCOUNTING (9)

N. Sol		lbs.
NH_3		lbs.
H_3PO_4		lbs.
H_2SO_4		lbs.
Solids		lbs.
Total		lbs.
Water Removed		lbs.
Total Production		lbs.
Total Production		tons

Batch Scale Count
Finished Prod. Scale (tons)
Production Rate Tons Per hr.
Average Tons Per Hr. (6)
Formula Solids (lb. ton)

GRANULATION: Good, Fair, Poor

Ammoniator Operator
Shift No. Shift Foreman
Plant Mgr.

COOP.-1855-B WHITE COPY-DIVISION OFFICE (7) YELLOW COPY-PLANT FILE

Photo 5

As you will notice, this half gives us processing information.

In this second half, we have meter settings, calculated and actual; the totalizer reading for both liquids and dry materials; both a footage and time indicator on our weigh belt.

In the material accounting section we use the instrument figures in all calculations, removing water as formulated, and this will many times furnish us with reasons for an analysis that does not jibe.

We were told that this equipment would not work in a fertilizer plant, but it will if you want it to. Then you will see that we have a Batch Scale Count, and in this case the scaleman keeps his own count, and we also have an electronic counter. We have on this sheet a Finished Product Scale tonnage to storage, and I will return to this later. From the standpoint of loss in processing, we have losses at the ammoniator. This can be a dust loss as well as the actual loss of gaseous nutrients. These losses can continue through granulation and drying if equipment is not properly constructed, installed, and operated.

Early in our operation if an error in analysis occurred, it was easy to blame the instruments, not scales so much, since our people understood these, but the rotometers were new. We installed a tank on a platform scale to check actual delivery. It is possible to check not only the accuracy of the instrument, but also the accuracy of your setting.

This is an example of curves prepared in order to obtain an actual setting.

In order to know what losses are occurring and where in the processing equipment it is necessary to measure stack losses, this is relatively simple equipment designed to measure both ammonia and dust losses.

While these may not be an absolute value, they are very valuable in spot checking your operation.

Early in our experience, we learned that sampling fertilizer was a nigh impossible job. So we installed automatic samplers.

Dust losses will continue, of course, through the cooling operation. I am sure all fertilizer manufacturers have dust collection systems for the drying and cooling operation, but this is still a source of loss which must be accounted for, and we are sure that if you have regular cyclones this loss is considerably more than you realize and more than you can afford. As indicated in Table 2, six tenths (.6%) per cent loss recovery gives a pretty short pay off time.

Photo 6

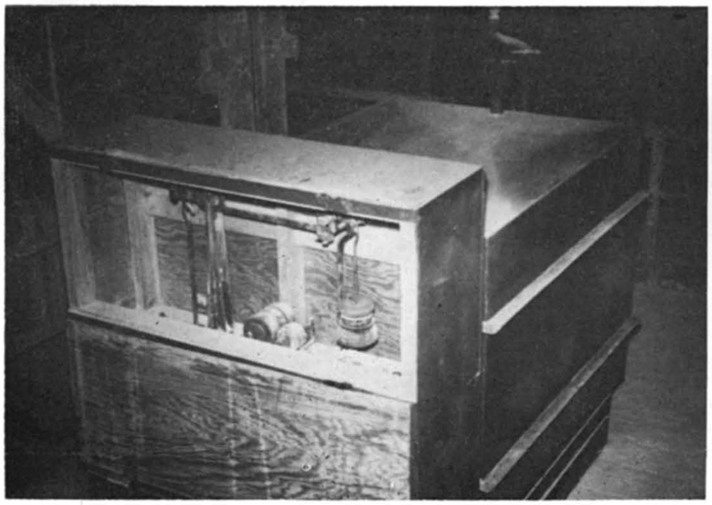
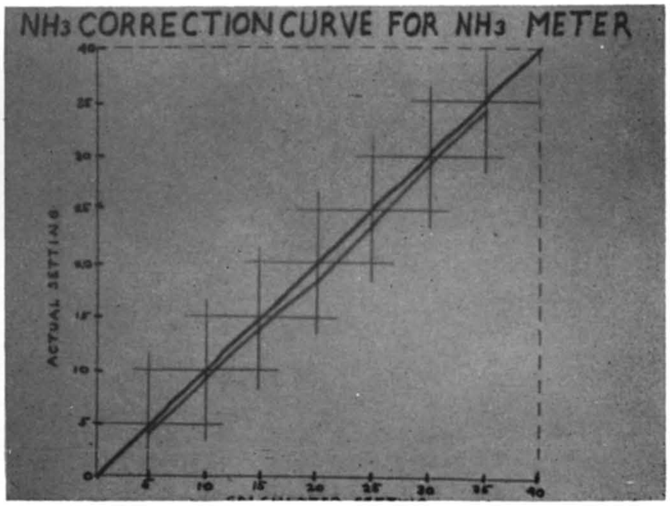


Photo 7



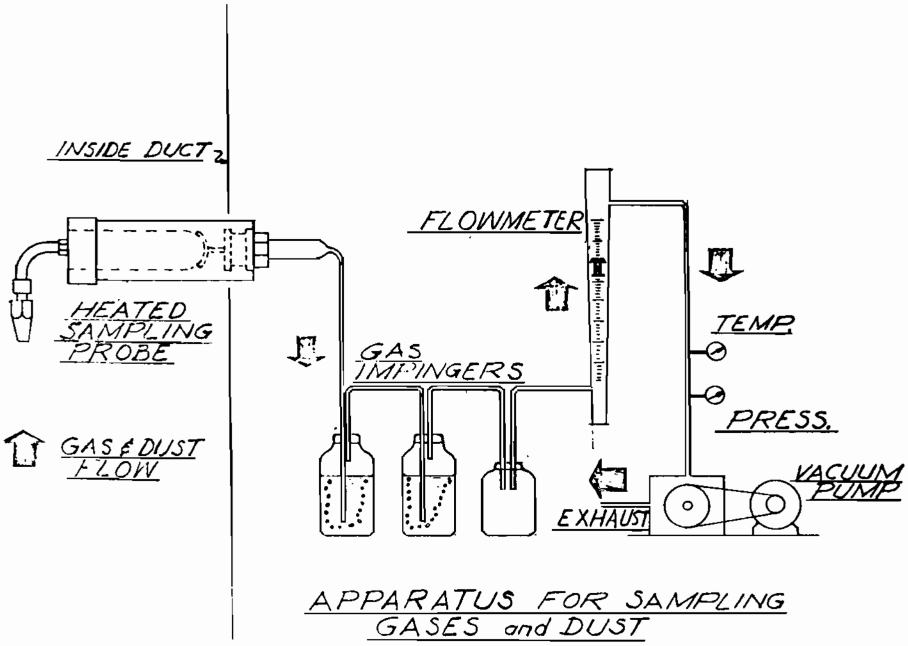


Photo 8

Table 2. Dust Collectors Evaluation

	Cyclones Loss Lbs./hr.	Per Cent	High Efficiency Collectors Lbs./hr.	Per Cent
Dryer	124		4.38	
Cooler	99		2.75	
Total	223	.625	7.13	.02

Photo 9

As I indicated in the discussion on the Production Control Data Sheet, we have a scale which automatically weighs and records the tonnage of material going from our classification screens to the bins. This enables us to separate the losses in the plant into three areas. The definite manufacturing losses. We weigh our materials into this process and weigh the final product out. The difference between the product scale weight and our shipping reports will give us losses in the shipping areas. The difference between these two and total losses would be losses in receiving and other handling not included in the process of shipping.

In Table 3, I want to illustrate that unless you measure you just do not know what is going on, and constant attention to all details of operation is essential.

Table 3. Shrinkage by Grade
Per Cent

Grade	Year	Tons	Mfg. Shrinkage	Shipping Shrinkage
3-12-12	62	6,111	.12	.80
	64	3,290	+ (1.12)	1.12
5-20-20	62	13,701	.41	.44
	64	17,902	.26	1.40
6-24-12	62	10,671	(+ .28)	1.62
	64	8,904	.43	1.01
12-12-12	62	13,344	.22	2.77
	64	14,652	.38	(+ .31)
6-30-15	62	2,807	2.13	1.09
	64	2,765	-o-	-o-
Average	62	59,782	.33	1.16
	64	58,060	.39	.78

As you put less interest on a grade as illustrated by the 3-12-12, errors can become greater. Undoubtedly tail ends of other grades were included, but shipping lost it. As in 5-20-20, you can place emphasis to improve and lose it in another area.

A new grade such as 6-30-15 will probably be high the first year. I doubt, however, that we improved this much, but these do give you information to work on.

In Table 4, a comparison of 1962 and 1964 shows that receiving and miscellaneous shrinkage have remained about the same. We lost on manufacturing, but gained on shipping. This shipping loss is too high, but we know what it is. Six ounces per bag if it is all shipped in fifty pound bags. In this area, how finished product storage is handled can be a factor. Placing material in the wrong bin, contamination with raw materials, shipping scales, both bulk and bagged, can be major contributors.

Table 4. Comparison 1962 and 1964 Sources of Shrinkage

	Budget	Actual	Receiving	Mfg.	Shipping
1962	3.00	2.69	1.20	.33	1.16
1964	2.25	2.36	1.19	.39	.78

Table 5. Comparisons 1958-1964

Date	Budgeted Shrinkage	Actual Shrinkage
1958	4.50	5.43
1959	4.50	5.34
1960	4.50	4.31
1961	3.50	3.20
1962	3.00	2.69
1963	2.75	2.13
1964	2.25	2.36

Table 5 illustrates that the progress made by knowledge of point of loss and attention to details.

I am sure you recognize in dealing with tonnages and our final product scale, in particular, an error can be included. If we formulate at one per cent moisture and dry to one-half per cent, for example. To eliminate this we must make all our calculations on a unit basis. This year, in cooperatin with our Central Farmers organization we are making an interplant study on a unit basis.

Table 6. Summary of Losses Nominal

Units x 1000	N	P_2O_5	K_2O
Received	429	1,044	868
Beginning Inventory	32	72	64
Total	461	1.116	932
Shipped	422	1,048	888
Ending Inventory	24	41	37
Total	446	1,089	925
Loss	15.4	26.7	7.2
Per Cent Loss	3.34	2.39	.77

Average Per Cent Loss — 1.93

In Table 6 we see these losses on a nominal basis. This means if we pay for a twenty unit Superphosphate, it is carried as twenty unit goods. If we make and sell a 5-20-20 analysis, this is the unit basis for this table.

On the other hand, if we use actual analysis, we see another picture. In Table 7, actual losses are shown.

Table 7. Summary of Losses Actual

Units x 1000	N	P_2O_5	K_2O
Received	429	1.044	868
Beginning Inventory	32	72	64
Total	461	1,116	932
Shipped	413	1,934	878
Ending Inventory	24	41	37
Total	437	1,075	915
Loss	24	40.6	18
Per Cent Loss	5.22	3.64	1.93

Average Per Cent Loss — 3.3

IMC's Experience With Defoamers In Phosphoric Acid Production

Robert Heck

IMC has been producing phosphoric acid in its plant at Bonnie, Florida since 1953. The digestion system in the original plant consisted of five stainless steel tanks with acid brick lining in the first and second tank. The slurry moved from one tank to another through an overflow system.

The slurry generated in these digestion tanks was filtered in Eimco disc filters which were originally designed for filtering a dicalcium phosphate slurry:

The filtered acid was concentrated in submerged combustion evaporators.

The first defoaming agent used in the original plant was a tall oil fatty acid. After 1960 an improved product consisting of 70% tall oil with a proprietary additive was used. This latter defoaming agent reduced the defoaming cost per ton of P_2O_5 by approximately one-third.

In November of 1962 a new phosphoric acid plant was built at Bonnie. This plant consists of a digestion unit containing 10 concrete tanks, carbon brick lined, with an underflow, overflow arrangement between tanks.

The slurry from the digestors is filtered on a Prayon filter. After filtration the resulting 30% P_2O_5 acid is concentrated to 54% P_2O_5 in three Swenson forced circulation evaporators in series.

The first defoamer used in the new plant was a tall oil with a proprietary additive. The combination of the new production system and tall oil derivative further reduced defoaming cost.

In 1963/64 the operating rate of the plant was increased from 300 TPD to 440 TPD and the defoamer cost rose to the late 1962 levels.

The various types of defoamer tried in the Bonnie plant are as follows:

1. Tall oil from animal fat.
2. Tall oil from vegetable fat.
3. Tall oil from pine oil.
4. Hodag PH55.
5. Swift, Type F67A

The defoamer is fed to both the number one digestion tank and the feed to the evaporators. The split is in the range of 70-80% to the digestion tank and 20-30% to the evaporator feed. At present we are using 4-7 pounds of defoamer per ton of P_2O_5 at a delivered cost of 11-12 cents per pound.

We have found that the amount of defoamer required is a function of at least five variables:

1. Operating rate
2. Digestion system design, including cooling and fume disposal
3. Defoamer selection
4. Rock source and grade
5. Amount of aeration taking place.

FRANK NIELSSON: Many thanks to Our Panelists for a thorough, interesting discussion on each respective paper presented and to this audience for contributing many questions and interesting, helpful answers. I was glad to have the opportunity to monitor this session. I now turn the meeting back to Moderator Reynolds.

MODERATOR REYNOLDS: Thank you Frank and your Panelists for a job so well done. We shall adjourn until tomorrow morning 9.30 A.M.

Thursday Morning Session, Nov. 12, 1964

The Round Table Reconvened at 9:15 o'clock A.M.

Vincent Sauchelli and Albert Spillman Moderators

CHAIRMAN SAUCHELLIS Ladies and Gentlemen please come to order. We have an interesting program today and with your cooperation it will be very successful and enjoyable. I shall now turn the program over to Albert Spillman who will moderate this morning's session.

MODERATOR SPILLMAN: Good morning Ladies and Gentlemen. I shall shorten my remarks to save a little time. It is always a real pleasure for me to have the opportunity to meet many of my friends here and also to make acquaintances with a good many of you attending our Round Table for the first time.

I wish to take this opportunity first, on behalf of you folks and your Executive Committee, to thank Tom Athey and his Associates of the Raymond Bag Company for the splendid entertainment given to us last evening.

Your Executive Committee has had the usual cooperation from the Membership on topic suggestions for this two and a half day program. I am confident that this program will meet with your approval. Finalization for topics to be discussed were carefully analyzed by your Executive Committee to be most important and timely. Each Speaker, assigned to lead the various discussions, is highly qualified by his experience and training.

The first discussion this morning "Steam, Acid or Water in Granulation," was prepared by William (Bill) E. Adams, Elmer C. Perrine and George R. Gilliam. They have contributed a great deal to a number of Round Table conferences. Bill Adams will present this paper.

The Application Of Steam, Acid Or Water In Granulation

William E. Adams, Elmer Perrine, George Gilliam

THE word "granulation" has been very common in the fertilizer industry for twenty-five years. Granular fertilizer is generally considered as a product containing very few granules smaller than twenty mesh. In this discussion we will consider also semi-granular fertilizer which has the fines in the product except for those picked up by the air system and returned as recycle. The coarse material is crushed since oversize from both granular and semi-granular will be sized to less than five mesh.

Considerable work has been published on granulation of 1-1-1 and higher ratios of P-K fertilizers; but, we believe, less work on low nitrogen grades. The Eastern Seaboard has a high percentage of 1-2-2 and 1-3-3 ratios which appear to change slowly. We might mention one reason for this is the split application of nitrogen for various crops on lighter sandy soils.

The purpose of this paper is to discuss the use of steam, acid or water in granulating. A comparison is shown of finished product with respect to screen sizes along with temperature and moisture data throughout the individual runs. The production rate versus drier load is given for the different ways in co-current and counter-current drying equipment. This data was taken from commercial plants producing from ten to twenty tons per hour, and essentially agrees with data previously run in our pilot plant.

It is estimated that a sulfuric acid installation with storage will cost $5,000. A steam installation will cost $10,000. A pound of acid is estimated to cost 1.25¢ where steam costs .23¢ per pound. The "rule of thumb" method is that a pound of steam will replace about one pound of sulphuric acid in granulation. This represents approximately one-dollar's saving per ton in the co-current system where data were taken.

In running these tests an effort was made to granulate about the same with either steam, acid or water. Figure 3, 4, 9 and 15 reveal this very well and are generally as close as can be expected by visual observation. The time for the individual tests was generally based on that required to give the same general appearance in granulation. The quantity of recycle from cyclones was observed to help establish this result. Temperature of the product throughout the entire system was taken for guidance in the granulation.

In view of vigorous discussions between the value of co-current

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Acknowledgements

Elmer, George and I are indebted to Central Chemical Corporation, Hagerstown, Richmond Guano Company, Richmond, and Tilghman Fertilizer Company, Salisbury, for supplying the service and equipment to obtain this data. We are delighted to present this production data and hope you find some benefit from it.

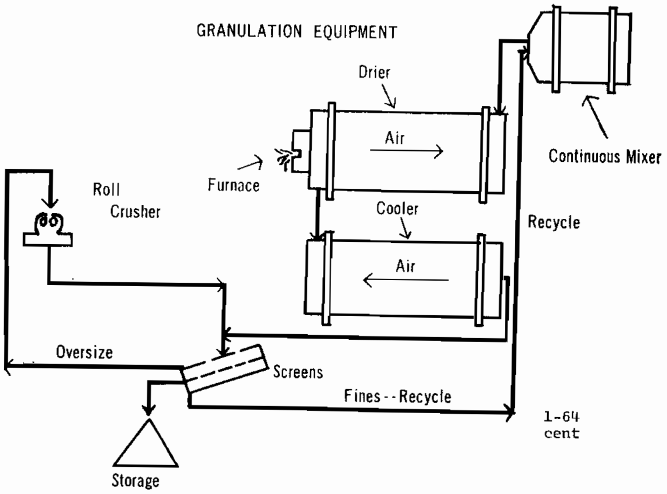


Figure 1.

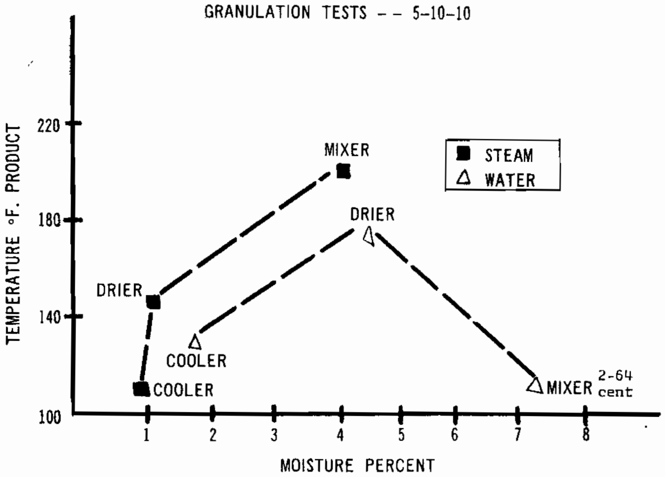


Figure 2.

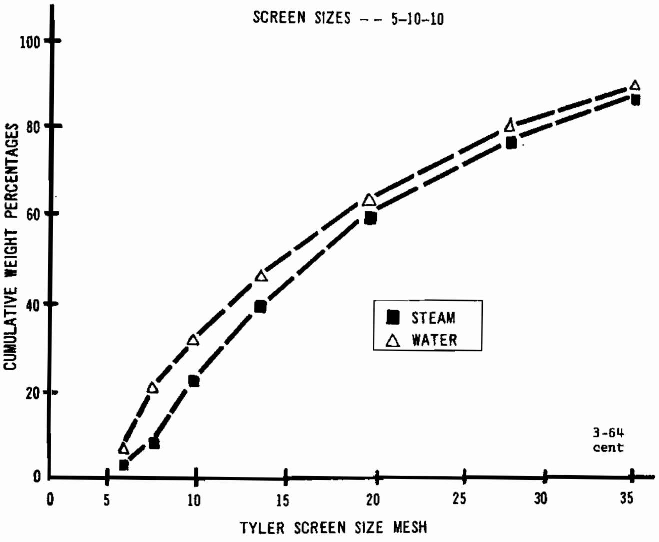


Figure 3.

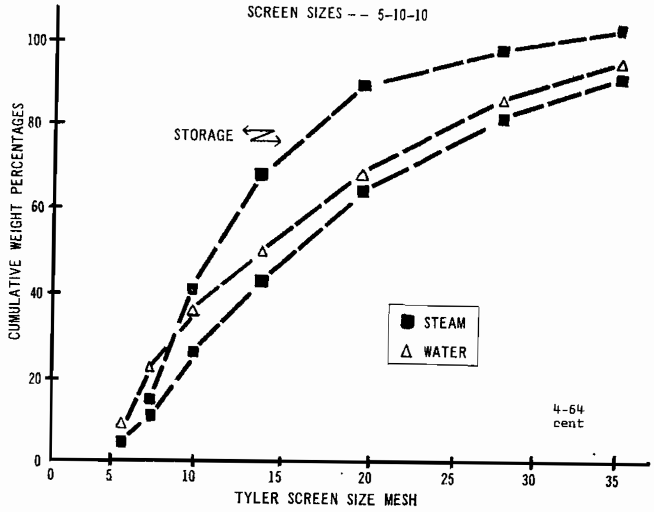


Figure 4.

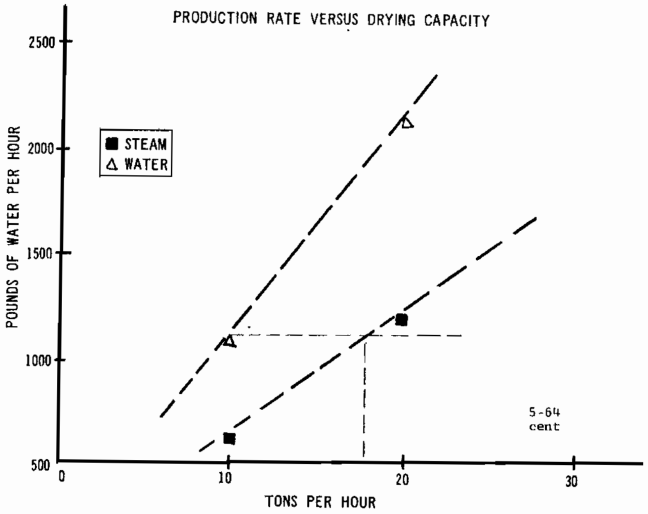


Figure 5.

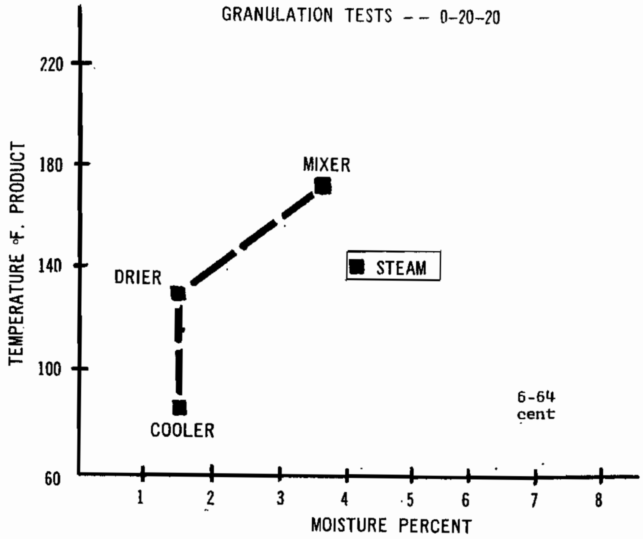
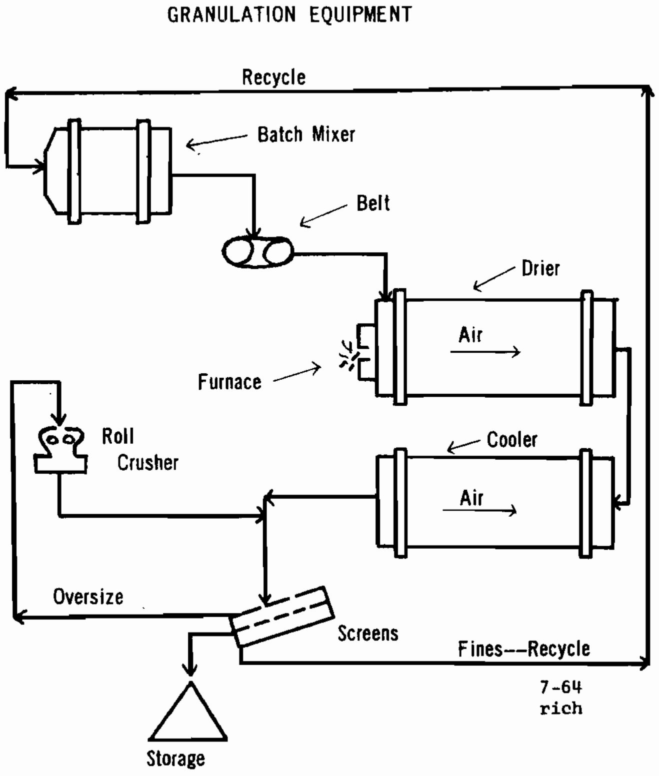


Figure 6.



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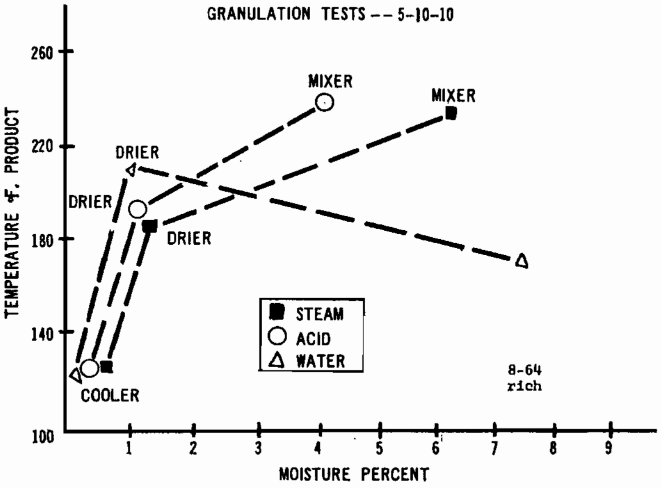

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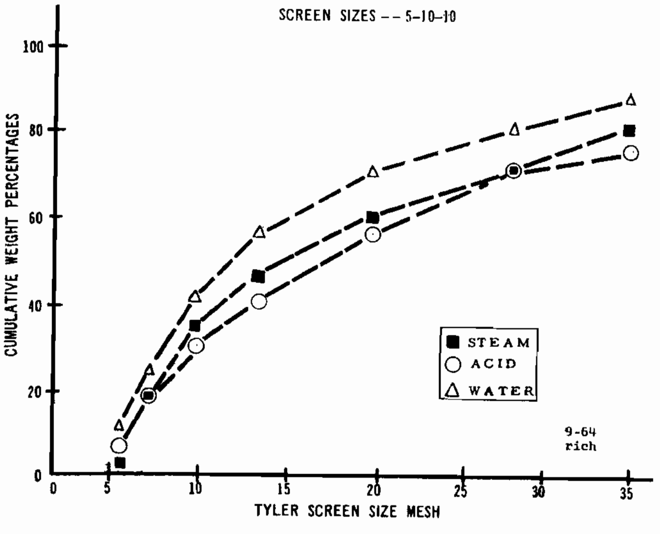


Figure 9.

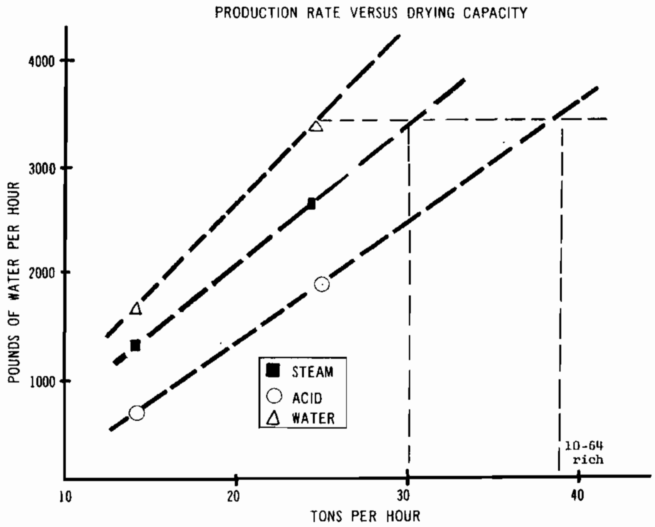


Figure 10.

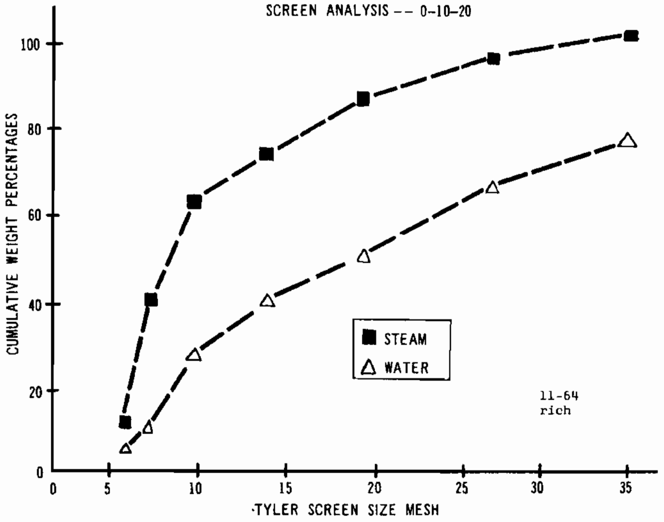


Figure 11.

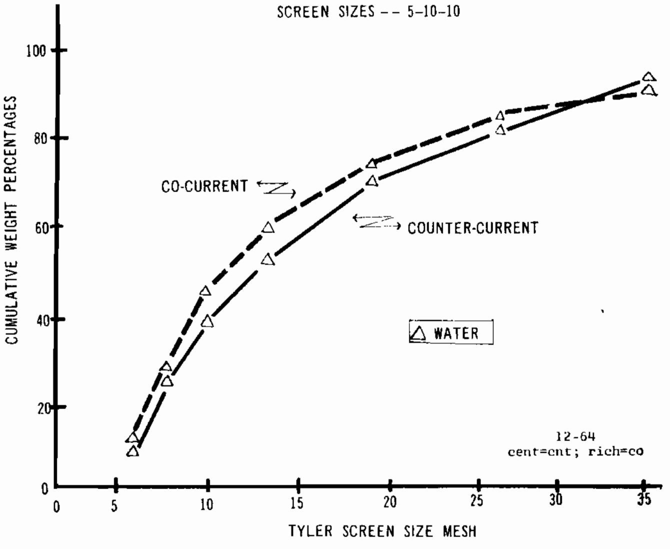


Figure 12.

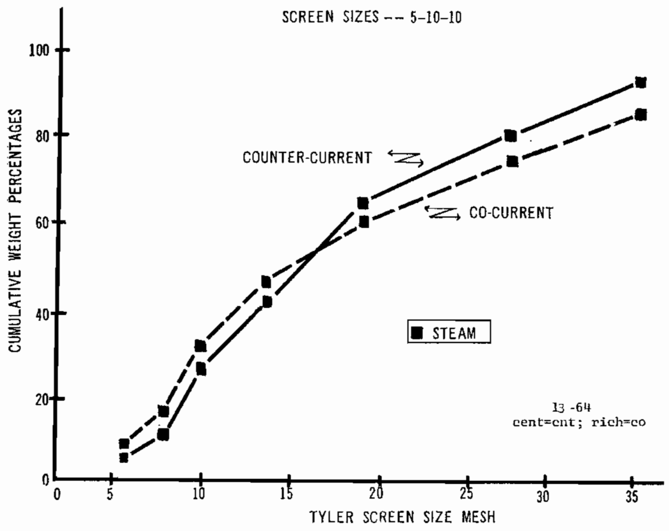


Figure 13.

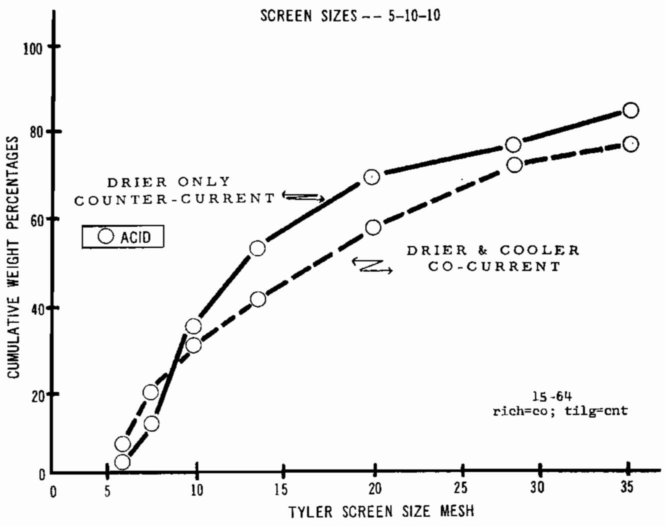


Figure 15.

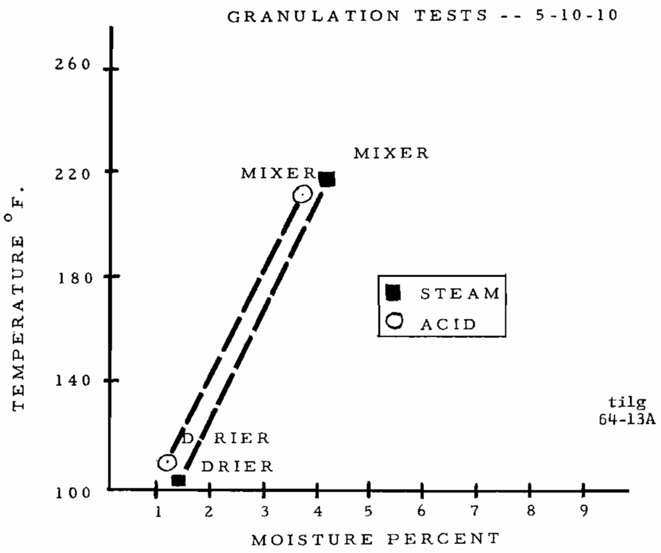


Figure 13A.

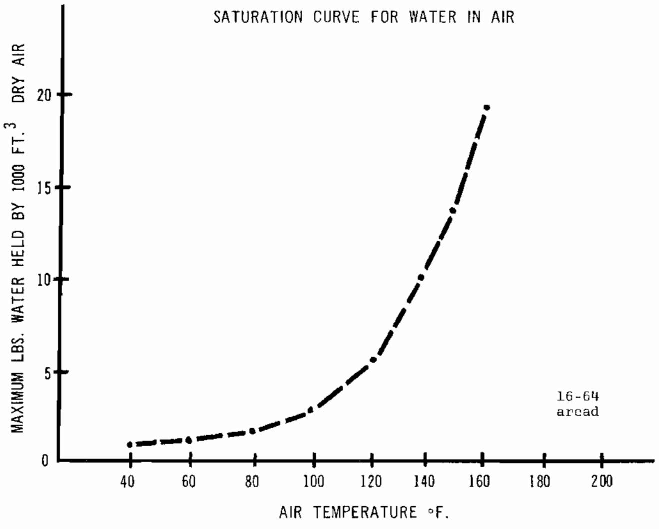
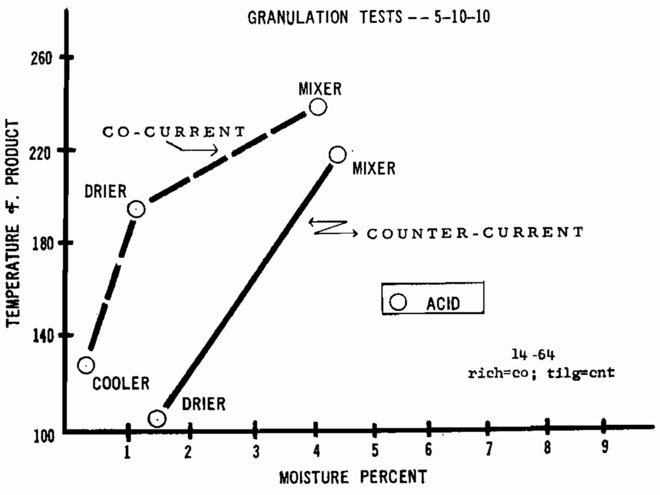


Figure 16.



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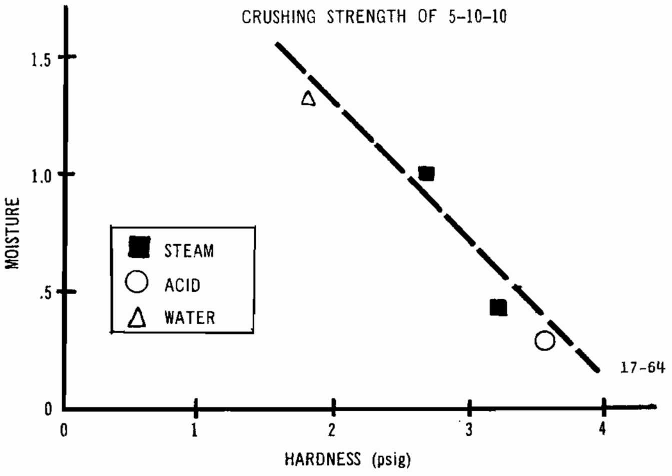

Figure 17.

Equipment Listing. (Counter-Current System)

Pulverizer #2
Make—Prosser
Size
RPM—850
h.p.—10 1170

Screen
Tyler Hummer
4 x 10—#9063—14 mesh
4 x 5 —#9465—5½ mesh

Steam Boiler
Make—Ames Iron Works
Oswego, New York
General Furnaces Corp.
Size—90 h.p.
Type—Cyclotherm
Year—1943
Serial—52219
3000 pounds @ 150° F.
Maximum water pressure—125

Drier
Make—Prosser
Size—5′ 6″ x 25′ 9″
RPM—7¼
h.p.—15 U. S. Motor Gear Head

Elevator #3
Make—Sackett
Size—6 x 12
RPM—40
h.p.—7½
Westinghouse

Burner—Cleveland, Ohio
Make—North American
Size—212-8A

Fan
Make—Westinghouse
Size—426 M
Wheel—45″ Diameter
11.81″ Circ.
Inlet 26″ Diameter
Outlet 24-5/16″ x 22-3/8″
RPM—1190
h.p.—50 (runs 95 ampers)
R.P.M.—1760.

Ammoniator
Make—Prosser
Size—7 x 12
RPM—16
h.p.—30 (runs 15 amperes)
Crocker Moto Wheeler

Cooler
Make—Blue Valley
Size—5′ x 30′
RPM—9¾
h.p.—15

1-64 Cent.

5-10-10	Formulations	0-20-20
228 pounds	Nitrana 6-M	
990 "	R.O.P. Superphosphate—20%	881 pounds
—	Triple Superphosphate—47%	483 "
328 "	Murate of Potash—62%	656 "
520 "	Dolomite	20 "
2066 "	TOTALS	2040 "

Cent.

Table I. Granulation Tests Using Steam 5-10-10

		Run #1			Run #2	
	°F.	Cenco Moisture	Karl Fischer % Moisture	°F.	Cenco Moisture	Karl Fischer % Moisture
Mixer	202	4.5	3.85	202	4.6	4.71
Drier	157	1.6	1.48	140	1.4	1.44
Cooler	112	2.0	1.01	112	1.4	1.35
Pile	100			100		
Stream lbs/ton		127			127	
Acid lbs/ton—66° Be.						
Oil gal/ton						
Boiler		2.60			2.60	
Drier		.52			.52	
Total		3.12			3.12	
Recycle lbs/tons (Cyclones)						
Recycle lbs/ton (Oversizet)						
Rate tons/hr.						
Air Flow (ft./min.)						
Horizontal		300			300	
Vertical		230			230	
Ambient Temperature		62° F.			62° F.	
Relative Humidity						

2-64 Cent.

operation versus counter-current operation, we have taken data in both systems. Figures 12 and 13 show the screen size comparison of semi-granular product using steam or water for both types.

We were also interested while running these tests to determine whether more BTU's would be required for drying to a finished product using steam for granulation or water for granulation. Information obtained suggests oil consumption per ton of fertilizer is about the same. See table No. I & II. Careful temperature measurements were taken at the discharge of the mixer along with moisture determinations when using steam or acid or water to establish the moisture necessary for proper liquid phase at that temperature. Figure 2, 8, and 13-A show the results.

In general, the data show the higher the temperature, the lower the moisture which results in a lower load on the drier. Since driers are the "bottle neck" in many granular operations, any assistance in lowering the drier load will increase the tons per hour produced. Figures 5 and 10 show various production rates using steam, acid or water where water removal by drier is the controlling factor.

The use of steam in no-nitrogen grades was run for observation and comparison with water. Figures 6 and 11 show the results of using steam for granulating no-nitrogen grades compared with water. The low-moisture from the mixture using steam as compared to water is quite encouraging for increasing production.

Small plants have improved their product some by adding sulfuric acid and allowing the material to roll in the mixer. We have added a drier to this type operation to further improve the product. Figures 13-A and 14 show the moisture of the finished product produced when using steam or acid for granulation with limited equipment.

Figure 16 shows the need for increasing the air temperature before it enters the drier. Dry air at approximately 162° F. will pick up twenty pounds of water per

1,000 cubic feet. Compared with less than one-half pound at 40° F. This reminds one that high humidity air at low temperature can be changed to low humidity air at higher temperatures. This also reminds us that high temperatures are not absolutely necessary to do a fair job of drying.

Samples of the various products were tested for hardness. Figure 17 shows the hardness of the pellets versus the moisture for products made with steam, acid or water in a 5-10-10.

Table IV. Granulation Tests Using Steam
0-20-20

	Run #1		Run #2	
	°F.	Cenco % Moisture	°F.	Cenco % Moisture
Mixer	171	3.5	174	3.7
Drier	127	1.6	126	1.7
Cooler	77	1.3	80	1.5
Pile				6-64 Cent.

Table II. Granulation Tests Using Water
5-10-10

		Run #1			Run #2	
	°F.	Cenco Moisture	Karl Fischer % Moisture	°F.	Cenco Moisture	Karl Fischer % Moisture
Mixer	120	6.6	7.41	113	6.6	7.48
Drier	180	3.1	4.25	210	4.0	4.93
Cooler	130	1.7	1.83	138	1.7	1.68
Pile	128			128		
Steam lbs/ton						
Water lbs/ton		217			217	
Acid lbs/tons—66° Bé						
Oil gal/ton						
Boiler		—			—	
Drier		3.1			3.1	
Total		3.1			3.1	
Recycle lbs/ton (Cyclones)						
Recycle lbs/ton (Oversize)						
Rate tons/hr.		10			10	
Air Flow (ft./min.)						
Horizontal		300			300	
Vertical		230			230	
Ambient Temperature						2-64
Relative Humidity						Cent.

Table III. Screen Sizes
5-10-10

Tyler Screen Size Mesh	Steam Exit Cooler Cumulative Weight Percent	Water Exit Cooler Cumulative Weigth Percent	Product to Storage Belt to Pile Cumulative Weigth Percent
6	5	10	None
8	12	22	15
10	24	35	38
14	41	48	65
20	61	65	89
28	79	81	97
35	92	93	99
Thru 35	8	7	1
			3-4-64 Cent.

Table VI. Granulation Tests Using Acid
5-10-10

	°F.	Karl Fischer % Moisture
Mixer	235	4.20
Drier	190	1.20
Cooler	126	.46
Pile		
Steam lbs/ton		
Water lbs/ton		
Acid lbs/ton—66°**	106	
Oil gal/ton		
Boiler	—	
Drier	1.0	
Total	1.0	
Recycle lbs/ton (Cyclones)	400	
Recycle lbs/ton (Oversize)	140	
Rate tons/hr.	24	
Air Flow (ft./min.)		
Horizontal		
Vertical		
Ambient Temperature		
Relative Humidity		8-64 Rich.

**Acid was used to absorb excess ammonia over that absorbed by the R.O.P. Superphosphate.

Table VII. Granulation Tests Using Water
5-10-10

	°F.	Karl Fischer % Moisture
Mixer	170	7.50
Drier	207	1.30
Cooler	128	.24
Pile	92	
Steam lbs/ton		
Water lbs/ton	112	
Acid lbs/ton 66°Bé	13	
Oil gal./ton		
Boiler	—	
Drier	2.5	
Total	2.5	
Recycle lbs/ton (Cyclones)	400	
Recycle lbs/ton (Oversize)	220	
Rate tons/hr.	24	
Air Flow (ft./min.)		
Horizontal		
Vertical		
Ambient Temperature		8.64
Relative Humidity		Rich.

**Acid was used to absorb excess ammonia over that absorbed by the R.O.P. Superphosphate.

Equipment Listing. (Co-Current System)

Mixer
Make—Atlanta Utility Works, Inc.
Size—Two-ton
h.p.—15

Screen
Make—Atlanta Utility Works, Inc.

Drier
Size—7′ x 50′ Air System—30 h.p.
Make—Prosser
h.p.—50

Cooler
Size—6 ′x 50′ Air System—25 h.p.
Make—home-made
h.p.—30

Screens
Sizes—6′ x 14′
6′ x 14′
Make—Carwell Machine Company

Chain Mill
Make—Atlanta Utility Works, Inc.
h.p.—10

Steam Boiler
Make—Schultz & James
Richmond, Va.
h.p.—125
BFA: dew

7-64
Rich.

0-10-20	Formulations	5-10-10
—	Nitrate 3-M	230 pounds
—	H_2SO_4—66° Bé	13 "
1000 pounds	R.O.P. Superphosphate—20%	1000 "
660 "	Murate of Potash	330 "
17 "	Dolomite (Calcined-CaOMgO)	—
323 "	Filler	427 "
2000 "	TOTALS	2000 "

Rich.

Table V. Granulation Tests Using Steam 5-10-10

	Run #1 Karl Fischer		Run #2 Cenco		Run #3 Cenco	
	°F.	% Moisture	°F.	% Moisture	°F.	% Moisture
Mixer	235	6.40	235	5.00	235	7.00
Drier	190	1.20	185	——	195	2.00
Cooler	126	.51	115	1.40	130	1.60
Pile	92		92		92	
Steam lbs/ton	52		60		72	
Water lbs/ton						
Acid lbs/ton—66° Be.**	13		None		None	
Oil gal/ton						
Boiler	.6		.5		.6	
Drier	.9		.8		1.0	
Total	1.5		1.3		1.6	
Recycle lbs/ton (Cyclones)	400		400		400	
Recycle lbs/ton (Oversize)	——		——		——	
Rate tons/hr.	24		——		28	
Air Flow (ft./min.)						
Horizontal	450		450		450	
Vertical	450		450		450	
Ambient Temperature	72° F.					
Relative Humidity						8-64 Rich.

**Acid was used to absorb excess ammonia over that absorbed by the R.O.P. Superphosphate

Table X. Granulation Tests Using Steam 0-10-20

	°F.	Karl Fischer % Moisture
Mixer	215	5.70
Drier		.44
Cooler		.23
Pile		
Steam lbs/ton	120	
Water lbs/ton		
Acid lbs/ton—66° Be.		
Oil gal/ton		
Boiler	—	
Drier	2.4	
Total	2.4	
Recycle lbs/ton (Cyclones)	40	
Recycle lbs/ton (Oversize)		
Rate tons/hr.	25	
Air Flow (ft./min.)		
Horizontal		
Vertical		
Ambient Temperature		
Relative Humidity	11-64 Rich.	

Table XI. Granulation Tests Using Water 0-10-20

	°F.	Karl Fischer % Moisture
Mixer	92	6.2
Drier		.2
Cooler		1.2
Pile	90	
Steam lbs/ton		
Water lbs/ton	173	
Acid lbs/ton—66° Be.		
Oil gal/ton		
Boiler		
Drier		
Total		
Recycle lbs/ton (Cyclones)	400	
Recycle lbs/ton (Oversize)		
Rate tons/hr.	25	
Air Flow (ft./min.)		
Horizontal		
Vertical		
Ambient Temperature		
Realtive Humidity	11-64 Rich.	

Equipment Listing (Semi-Granulation)

Mixer
Size—One-ton
Make—Sturtevant Mill Company
Drier
Size—6′ x 30′ Air System —25 h.p.
h.p.—15
Screen
Size—7′ x 14′

Table VIII. Screen Sizes
5-10-10

Tyler Screen Size Mesh	Acid Cumulative Weight Percent	Water Cumulative Weight Percent	Steam Cumulative Weight Percent
6	7	11	6
8	17	25	17
10	20	42	31
14	41	57	44
20	56	72	59
28	71	83	71
35	74	91	82
Thru 35	26	9	18

9-64 Rich.

Table IX. Screen Sizes
0-10-20

Tyler Screen Size Mesh	Steam Cumulative Weight Percent	Water Cumulative Weight Percent
6	14	5
8	41	14
10	63	27
14	76	39
20	86	53
28	93	65
35	98	76
Thru 35	2	24

11-64 Rich.

Table XII. Semi-Granulation Demonstration
5-10-10

Acid	Steam	Solution		Temperature	Moisture
80		3-M	Mixer	210° F.	3.48
			Drier	110° F.	1.63
80		2-M	Mixer	215° F.	3.75
			Drier	125° F.	1.25
15	60	3-M	Mixer	220° F.	3.85
			Drier	115° F.	1.69
	80	2-M	Mixer	220° F.	4.39
			Drier	110° F.	1.44
40	40	3-M	Mixer	230° F.	3.58
			Drier	115° F.	1.25
40	40	2-M	Mixer	220° F.	3.79
			Drier	110° F.	1.19

13A-64 tilgh.

Table XIV. Screen Sizes
5-10-10

Tyler Screen Size Mesh	Run #1 Weight Percent	Acid Run #2 Weight Percent	Weight Percent Averaged
6	5.5	4.5	5
8	11	11	11
10	29	32	30
14	46	54	50
20	59	69	65
28	67	78	72
35	76	86	81
Thru 35	24	14	19

tilgh.

Table XIII. Granulation Tests Using Acid
5-10-10

	Run #1		Run #2	
		Karl Fischer		Karl Fischer
	°F.	% Moisture	°F.	% Moisture
Mixer	215	4.85	220	4.04
Drier	105	1.51	110	1.46

tilgh.

Table XV. Water Saturation In Air

Maximum Pounds Water Per 1,000 Cubic Feet of Dry Air	Air Temperature °F.
.413	40
.843	60
1.64	80
3.05	100
5.56	120
10.12	140
13.79	150
19.10	160

arcad.

Table XVI. Crushing Strength of 5-10-10

	Steam	Acid	Water
Richmond			
*Hardness	3.05	3.67	
Moisture	.51	.46	
Central			
*Hardness	2.85		1.93
Moisture	1.01		1.35

*psig

In summarizing, the use of steam looks encouraging toward increasing the production rate as compared with water, reducing fumes as compared with acid, and reducing cost as compared with acid. Many superintendents of plants feel they have less down-time due to less build-up and plugging of equipment when using steam. This can all be accomplished without increasing the cost of drying, as related to total oil consumption.

The use of a drier looks very promising toward improving semi-granular product made with sulfuric acid. The use of steam will reduce the costs measureably on this type product.

The use of steam, acid or water will give a good granular product and the dryness of the product determines its shelf life as indicated by the hardness tests.

MODERATOR SPILLMAN: Bill,

Elmer and George, thanks for this excellent, thorough report which will be most useful to many of us considering installing equipment for steam. We had many inquiries to discuss the economics of "Steam for Granulation." We ought to have some questions. Bill, I shall ask the first question. What do you consider the minimum capacity for a steam boiler to give the most economic operation.

Mr. Adams: The boilers used by most of the fertilizer industry are in the neighborhood of 125 to 150 horse power. This is satisfactory for average tonnage of 20 to 30 tons per hour. Most of these boilers are operated at around 100 lbs. to 125 lbs. pressure.

Moderator Spillman: Bill, did I understand you to say installation cost, including the equipment, is around $10,000.00?

Bill Adams: I may have squeezed that cost a little bit. Several people have told me their costs are as much as $12,000.00 or $13,000.00 and this depends on whether it is 125 or 150 horsepower boiler. It also depends on how this equipment is set up.

A Conferee: How do you calculate the amount of steam that remains in your ton.

Mr. Adams: Actually we took the samples of the product and analyzed it for moisture. I might also mention that these steam generators have water meters in front of them. One pound of water used is one pound of steam and we measure steam consumption by that, but the way to tell the amount retained would be to analyze the mixture for moisture from the ammoniator.

Conferee: How did you measure your granule hardness.

Mr. Adams: I shall call on my co-partner, George Gilliam, to answer that question since he handled the "hardness tests."

Mr. Gilliam: These hardness tests are nothing more than an average figure. It depends on how much energy is applied. We used minus 8 plus 10 mesh pebbles and I think this data was determined on approximately fifty individual pebbles. We put the pebble on a little flat metal block placed on a scale. Above it we can bring a plunger down at a very slow rate, applying pressure, and observing the indicator on the scale as it will go up until you get a definite crush of the pebble. You will have a little jump there and where it jumps, that is the crushing pounds to crush that individual pebble.

Mr. Adams: George, before you leave, what would you say would be a good satisfactory average crushing strength figure? I think the man asked that question.

Mr. Gilliam: I don't think you have any worry above a half pound. Most of the granular fertilizers today will run somewhere from one to four pounds crushing. The interest is, will these pebbles break down in handling and bagging. I think if you have as much as one pound pressure strength you're reasonably safe. For example, a pebble of urea will have a crushing strength of approximately one pound average. Ammonium nitrate will go around two, two and a half pounds. It depends on the composition. Your NPK fertilizers, granular materials, will usually run somewhat higher than this if your moisture is below one and a half per cent. Of course, the crushing strength will drop off as the moisture content of your material increases.

Moderator Spillman: We have enough time for one or two more questions.

A Conferee: Are you finding any difference between the crushing strength in the granules when you use water, acid or steam?

Mr. Gilliam: The graph that we showed towards the end of our paper gave the crushing strength. This was primarily the basis of the dryness of the product. In other words, we can't tell any difference from the test work, that we did in this case, whether it's steam, acid, or water. All we can determine is that it is harder when it drier. Using steam we found that we could get granulation in the mixer at much lower water content, due to the higher temperature, than you could with using water for granualtion.

Moderator Spillman: Bill, did you make any studies on bag storage conditions with these experiments?

Mr. Adams: No actual bag tests in storage were run on these various tests.

Moderator Spillman: Thanks to all of you for an excellent discussion.

Three years ago we had a thorough discussion on materials handling, packaging, packer equipment, palletizing and storing. This year we were asked to discuss an up to date review on paper sacks versus plastic sacks. We are fortunate to have an excellent presentation on Paper Versus Plastic. Mr. C. G. Peterman, Secretary Paper Shipping Sack Manufacturers Association, is Panel Leader. His Panelists are John H. Dively, St. Regis Paper Co., G. W. McDuffie, International Paper Co., with whom I worked way back in 1933, when we first started packaging paper bags at our Baltimore plant and Shelby Brown, Jr. Will the panel come forward, please.

Paper Versus Plastic Bags

C. G. Peterman

Panel Leader

Once again, those of us representing the Paper Shipping Sack Manufacturers' Association are grateful for the opportunity of leading a concise and updated discussion on fertilizer packaging. Our name is quite a mouthful, but for those who prefer initials we are called P.S.S.M.A. or PISMA and sometimes we are called other names.

So that you will all have our Association in prespective, we have 21 members operating 69 manufacturing plants that produce 85-90% of all paper shipping sacks made in this country.

The title of our Association is

a slight misnomer since it seems to imply that our interest is only in "paper." Like the railroads who discovered they weren't in the railroad business but were in the "transportation" business, we have long ago discovered that our industry must be customer-oriented and not product-oriented. Our first job is to get your products to the final consumer with the highest degree of protection at the lowest possible cost and does not allow our interest to be confined to paper—but to plastics or combination paper-plastics or whatever material that might come along in the future that serves you best.

We are fortunately endowed with skilled and hard-working men such as those here on the panel with me today who give freely of their time to build a better industry. Many of these men who donate their time to work in our Association are experts not only in paper making and bag converting but in chemistry, packaging machinery, materials handling, traffic, package engineering, etc.

We have an active Technical Committee which is instrumental in establishing minimum requirements for materials used in paper shipping sacks and sack closures. Another Committee works on test procedures for components of shipping sacks. This helps both you and your paper shipping sack suppliers to test for uniform properties in sack materials and to maintain a high degree of quality in your packages.

In the year 1963, our industry produced over two billion eight hundred and thirty million multiwall sacks for the packaging and shipment of about 400 different commodities used in every major segment of our economy. Within this group, the largest single market for multiwalls is for packaging fertilizers. In 1963, 34% of all multiwalls were used for packaging and shipping agricultural & food products; 17% for building materials; 38% for chemicals; 8% for minerals and 3% miscellaneous products.

For fertilizer packaging the sewn open mouth type of sack continues to be predominant. In 1963, 71% of all fertilizer paper and combination plastic/paper shipping sacks were sewn open mouth; 17% were pasted valve type: 11% were sewn valve type and less than 1% were pasted open mouth sacks. New ideas and new equipment are right now being offered that will drastically change these percentages in the next few years.

With the advent of the all plastic bag, the trend toward 50 lb. capacity fertilizer sacks made of other materials continued to increase at a faster rate. A comparison of capacity of fertilizer sacks between 1960 and 1963 shows:

1960	1963	Capacity of Fertilizer Sacks
38.48%	49.70%	25 - 50 lbs.
40.83%	32.05%	51 - 80 lbs.
20.69%	18.25%	81 - 100 lbs.

Some of you may recall that our industry made a survey 5 years ago to determine the number of different sack widths used in the mixed fertilizer industry. We collected from our members two-hundred and eight different sack constructions, not, of course, including all plastic bag constructions which were then only a gleam in the chemical industry's eye. 97% of these sacks were made from 12 paper widths. I think it is important in the light of rapid changes and improvements in sack constructions, materials used, bag making equipment and materials handling methods to periodically have your suppliers make a study of your sack width requirements with the thought of getting your sacks into that 97% bracket, where only 12 paper widths are used. You might give your company the benefits of worthwhile economies, and I am sure you would obtain better service from your sack suppliers.

It is our intention here this morning to present to you an open, frank appraisal of the three major types of sacks used for packing and shipping Fertilizers. We have refrained from criticising bulk handling and segregation of ingredients during handling and shipping in bulk; nor are we advocating or promoting one bag vs. another. We hope to give you a run down on the variety of bags available; the good and bad features, and some of the advantages and disadvantages of each in filling, handling, shipping and storing.

With me are three gentlemen with whom I am proud to be associated. They are experts in their fields and join me in offering to help in any way we can to promote a better understanding between your industry and ours. Other representatives from our industry are here today and have also volunteered to be available to answer questions that might be raised following this presentation of papers, or any time during our stay at this convention:

1. Heavy Duty Plastic Sacks
2. Multiwall Paper Shipping Sacks
3. Plastic Film-Kraft Paper Combination Sacks

May I introduce Mr. Shelby Brown, Jr., Sales Manager of Bemis Bro. Bag Co.'s Plastic Package Plant, Terre Haute, Indiana — a man well qualified to speak to you on "Heavy Duty Plastic Sacks."

Our next speaker is Mr. O. W. McDuffie, Sales Promotion Manager, International Paper Co., Bagpak Division, New York, N. Y. Mr. McDuffie will cover the "Multiwall Paper Shipping Sack."

Mr. John H. Dively, Marketing Manager, Industrial Packaging, St. Regis Paper Co., New York, N. Y. has appeared before this meeting on a previous occasion and will cover the subject, "Kraft Paper/Plastic Film Combination Bags For Fertilizer."

Heavy Duty Plastic Sacks

Shelby Brown, Jr.

ETHYLENE gas is available in limitless quantities and from many sources. It was once a waste product of the oil industry. It is low in cost. It can be cheaply transported by pipeline. Ethylene is the basic raw material from which polyethylene resin is made.

Polyethylene resin is made from ethylene gas by polymerizing the gas under heat and pressure and through the use of certain catalysts. Variations in the processing results in different characteristics in the polyethylene resin produced. The processes readily lend themselves to complete automation with its great cost savings.

Polyethylene resin is thermoplastic. The resin is put into a machine called an extruder which melts and works it into a uniform molten viscous state. It then extrudes this material through a die into the film, which solidifies as it cools, and it is wound into rolls ready for printing and manufacture into bags.

We are taking only a bird's-eye view of how polyethylene film is made. I feel this is sufficient for our purpose here today. With your producton knowledge, and it, too, leans heavily to automation, you can readily foresee the low cost possibilities for polyethylene packaging. Its technology is still so very young as compared to other packaging materials such as paper.

Polyvinyl chloride, most frequently referred to as P.V.C. has been promoted in Europe for heavy duty plastic sacks. At least one of its principal European promoters manufactures the resin and film, and also the chemicals and fertilizers which are packaged in it. Its use does not appear to be on the increase in Europe. It has not been used in this Country primarily because of its poor strength in cold weather and because it is higher in price than polyethylene.

Polypropylene film is sometimes mentioned as a possibility for heavy duty packaging. Again, poor performance in cold weather and higher price thus far have eliminated it as against polyethylene.

The improvements that have been made in polyethylene in the last few years are so great that they are almost dramatic. They have earned its virtually uncontested place as the heavy duty plastic sack film of today. These improvements have not only been in the resin but also in the extrusion processes and in the sealing and fabricating techniques of bag printing and manufacture. These improvements are continuing. As in fertilizer, there can be great differences in bags of the same specification from one plastic bag manufacture to another.

Improvements, such as mentioned, generate high obsolescence in bag manufacturing equipment. None-the-less, research and development are being constantly pursued by the better bag manufacturers, and the quality of their heavy duty plastic sacks climbs accordingly.

To clarify, henceforth when I refer to the heavy duty plastic sack, I will be talking about a bag made from polyethylene. For the sake of of brevity, let's refer to it by its abbreviation "P.E."

Heavy duty plastic sacks have very high impact resistance and are extremely strong. Drop tests results are many times better than for the paper multiwalls they replace. By comparison with multiwall. P.E. does have lower puncture resistance. Punctures occurring in plastic sacks, however, will not spread as in the case with tears in multiwalls. Punctures in the plastic sack may be easily and quickly repaired with plastic patches available on the market. The original plastic sack is thus not only saved, but also the sack's contents plus labor cost of re-sacking. The puncturing problem is rapidly fading out of the picture due to gain of experience in proper handling. Continued improvements in the film also increase its resistance to puncturing.

Plants used to handling multiwalls have to change their bag handling practices when converting to P.E. Packing and handling equipment must be altered to eliminate sharp corners, protrusions, etc., which would snag and puncture plastic sacks. Many of you will remember the problems encountered when you switched from textile bags to multiwalls. It is one of the dues to progress. To stand still in business today can be very fatal.

The same equipment used to fill open-mouth multiwalls can be used to fill heavy duty plastic bags. Plastic sacks, however, are more flexible than multiwalls, and packer operators have to be re-trained on handling practices to equal production speeds. It is now being achieved in many places. New opening features, such as opening lips on bag tops and anti-static formulations in films, will materially aid the filling operation.

P.E. bags can be closed at the same speed as multiwalls. The heat seal closure of plastic sacks does require additional investment in plant. The amount of investment will vary in round figures from $900 to $5,000 depending upon closing speed desired. There have been vast improvements in the heat sealing equipment. Doughboy, for example, now have their SCH Model sealer, which has been designed for heavy duty plastic sacks and especially for the fertilizer industry. It will close in excess of 30 bags per minute. Current limitations are packer speeds.

Doughboy is also coming on to the market with another band sealer designed with particular emphasis to the fertilizer industry. It is their ED Model 2. The basic sealer sells for about $895. With tape-over attachment, trimmer, etc., the unit comes to about $1,700. It will close eight to 12 bags per minute. This unit should be of great interest to the smaller packer. It may also be of interest to other fertilizer packers who want to enter the lawn and garden products field. P.E. is ideal for this field because of its "impulse" appeal, plus product protection around the home, garage, etc.

The Bemis "Thermair" heavy duty plastic sack sealer has recently been turned over to Doughboy through a licensing arrangement. It is currently in Doughboy's engineering department for some modification.

The only other known current heavy duty plastic sack sealer is the duPont sealer. It was first placed in fertilizer packaging at the beginning of this year on a limited basis. Some modifications have been made on it as a result of these field studies. They claim their corrosion experience has been good so far. They plan to stay in the fertilizer field with the sealer on a limited basis. By the end of next year, they will complete their observations and determine where they stand.

The heavy duty plastic valve sack is, in my opinion, still in the embryonic stage. There are a couple of designs being shown, but they do not appear to be aggressively promoted. Price and production capability appear to be the current deterrents. I am sure our competitors have been and are working on a valve bag design just as aggressively as Bemis is.

Handling of P.E. bags is no longer a problem if the bag size is right and the filled bag is handled as a plastic shipping sack and not as a multiwall. Many users report the bag as being easier on the hands. Consult your bag supplier on the best bag size for your needs.

Some users of heavy duty plastic sacks have encountered stacking problems. Bag size and piling methods have much to do with this. The big manufacturers are working diligently on the problem. As of this date, it is not possible to offer the anti-slip qualities in P.E. sacks that are available in multiwalls through the use of crepe paper, etc. A solution will be found. Current surveys indicate the stacking problem to be on the decline as users are becoming more proficient in handling the heavy duty plastic bag. Experience in handling leads to reduced problems.

The moisture barrier characteristics of the heavy duty plastic bag stands out clearly as the single, most important advantage cited by the farmers, farmer dealers, and dealers and other authorities contacted in a very recent survey. They can be field stacked for longer periods of time than multiwalls of the same cost performance. Prolonged exposure to sunlight causes a breakdown in P.E. which makes the bag brittle. The solution to the problem lies in the finding of suitable ultra-violet ray inhibitors for the film. The plastic bag is already helping to relieve the seasonal aspect of the fertilizer business, and its future in this regard is bright.

Plastic shipping sacks must be perforated to allow the entrapped air in the filled bags to escape. "Set-up" in some fertilizer formulas under certain conditions occurs adjacent to these perforations. Under the auspices of the National Flexible Packaging Association, the major plastic shipping sack manufacturers are making an intensive study to find out the true cause of the "set-up," so that corrective steps may be taken. "Set-up" is, of course, not new in fertilizer and occurs in other types of bags as well.

Where plastic bags were available in 1964, the re-purchase rate was 84% among the farmers and 85% each for the farmer-dealers and dealers according to a current mail survey. This was significantily above the 1963 re-purchase rate of 72%.

All indications are that the use of heavy duty plastic sacks will continue to grow, and with great benefits to the fertilizer industry.

Multiwall Paper Shipping Sacks

O. W. McDuffie

THE concept of a "a bag" for storing and transporting products of value has existed for a long, long time—in fact, prehistoric man used a flexible animal skin. Archeologists also confirm that a glued package, made from the leaves of the Papyrus plant (which also comprised the Egyptian writing paper) was in use on the trade routes from the Eastern Mediterranean area to the Far East by, at least, as early as the year 2,000 B.C. The paper bag, as an industrial package, however, did not appear on the American scene until about 1850. A U. S. Patent, issued in 1859, covered a hand-crank powered machine for making pasted style paper bags. Although these first machines were crudely whittled from Rock Maple, some of their principles are employed in the modern paper bag making technique in use today.

The multiwall paper bag gained acceptance for packing Portland Cement during the 1920's and, by 1938, was in fairly wide use for bagging mixed fertilizers. Although most multiwall bag industry statisticians would probably say that the peak year for mixed fertilizer in paper bags was 1955, there was approximately 75% of the fertilizers produced during 1963 conveyed to the farmer in multiwall paper bags—and this excludes ammonium nitrate.

It has been said by "old timers" of our Industry that we, in reality, do not qualify to be classified as "Industry" because our output is so diversified that we are only a small part of all the consumers of industrial shipping sacks. To the extent that the multiwall bag is tailor-made to the user's specific packaging requirements, this is true, and this is favorable for bag users as well as producers because when your seasonal demands are at a low ebb, we are serving other consuming markets where cyclical demand patterns differ from your own. Our flexibility in this respect results in more economical utilization of converting facilities, which means lower packaging cost for all packers of industrial products.

To convince you that the multiwall paper bag of today differs from the first one you ever saw is somewhat analogous to a similar problem of a certain foreign car manufacturer, whose unattractive product has not changed appreciably since the first one rolled off the assembly line. I notice in the local newspaper that his advertisement is currently captioned, "It looks like the 1947 model but it is 5,001 ways improved." Now, a multiwall bag does not have nearly so many parts but virtually every detail of its construction has been improved since 1957. You have noticed, perhaps, that today's bag can be made of Extensible Kraft and, although it is lighter in basis weight, it is much stronger. Further, these improved papers are available in various asphalt laminated and polyethylene coated grades, and bags made from these improved extensible materials provide a broad range of product protection levels. Its outer surface can be more highly dressed to better present your company's name, trade-mark, and product brands to your customer. Its outer surface is now frictionized to insure stacking stability during transit of the filled sacks. Both Valve and Open Mouth style bags have recently been improved. For example, the Stepped-End style Pasted Valve multiwall presents possibilities for packaging economies and increased efficiency which may yet not be fully recognized by the Fertilizer Industry. A new style of Open Mouth bag, which can be closed without the necessity of sewing machines, is also making its debut. This latter style package provides most of the attributes of the Pasted Valve bag in appearance after it is filled. O course, all earlier styles are still available and in a wider assortment of constructions.

In summary, I would like to leave with you three thoughts which characterize the multiwall paper bag as an industrial package. These are they:

First—It is flexible and, by this I mean, it can be tailored to your specific needs. If moisture barrier properties are required, the proper level of protection can be provided by one or more special plies and, in the same construction, be insured of resistance to puncture, rupture and tearing of additional plies of the more economical Kraft paper. The combination of walls of the multiwall bag's construction will permit you to buy the performance requirements that you need and to obtain these several necessary characteristics from the most economical packaging materials capable of supplying them.

Secondly—It is available. The multiwall paper bag comes from a domestic source of fiber, the reserves of which are being constantly replenished. It is unique in this respect, that its raw materials can be re-created, and whether it be the Open Mouth style bag or Valve bag, there is a supplier who can meet your seasonal requirements on short notice.

Lastly—It is a dependable industrial package which performs well in almost any bagging and product distribution system. It is easy for workmen to fill, convey and load; it ships well and, in normal application, over its entire end use area, the failures rate averages about 0.5%.

I have but one recommendation to make, which is this—if you have not had time to review your packaging requirements in the last year or so, that you should make a mental note here to do this before the rush of another season is upon us. The cost factors of your existing packaging system are constantly changing, labor costs increase, your customer requirements change. These changes may be affecting your over-all bagging cost adversely. I recommend that you call in your current supplier of bags and review with him all possibilities which could lower your cost. If you can find time to do this, I am confident that many of you would uncover opportunities for improved profitability.

Kraft Paper/Plastic Film Conbination Bags for Fertilizer

John H. Dively

WHEN I first entered the field of packaging, nearly 20 years ago in Baltimore, I concluded within a few short months that your industry could save a lot of money if it would follow the path of the nation's cement producers. The cement industry had really standardized its packaging operations. Virtually every cement plant I called on during the first few months of my travels, used the same types of packing equipment and bags, similar bag constructions, and practically the same size bags. Also, all of them had one man packer lines operating at 20 to 24 bags per minute.

The fertilizer packing lines I saw, on the other hand, demonstrated almost no evidence of standardization. Even, while in the same plant, it was not too uncommon to find both valve type packaging systems and open mouth packers. One could also frequently see both multiwall paper bags and textile bags being used.

Since I was a young engineer with a large paper company, hopefully knowledgable in my duties, but as I later learned, woefully naive to the needs of your industry, I formulated a great idea—standardization of packaging for the fertilizer producers.

One day way back in 1946, I mentioned this great idea of mine—standardization—to Al Spillman. Al, being the perfect gentleman and wonderful person he is, did *not* throw me bodily out of his office for wasting his time. Instead, he very patiently explained that even though a somewhat higher degree of standardization could be achieved in fertilizer packaging, especially in certain parts of the country, there were simply too many variables between plants and between the many grades and types of fertilizer produced to accomplish anywhere near the standardization achieved in cement.

About all I can say is, "How right can an intelligent man like Mr. Spillman be?" Here we are, almost 20 years later, and after listening to Mr. McDuffie and Mr. Hoepner I am certain you will agree that there is even *less* standardization in fertilizer packaging today than there was in 1946 and I haven't even covered the paper/-plastic combination bags that are now *also* available to you.

You may rightfully ask, "Are all these different bags and constructions really necessary and *especially* a bag that *combines both plastic and paper?*" My answer to that is this: "No perhaps they are not necessary, or, more properly put, *would not* be necessary if the bag industry or someone had been successful in finding the perfect bag for fertilizer."

As I see it, and I hope I am much smarter about packaging now than I was twenty years ago, for today's highly competitive and diversified fertilizer industry, a bag must do all of the following:

- Provide moisture protection for virtually all grades.
- Be resistant to attack by acid and/or chemical vapors of certain grades.

- Withstand a wide range of temperature changes and other adverse climatic conditions.
- Be easily closed with either adhesives, thread, or heat sealing.
- Be tough and resistant to puncture and scuffing.
- Handle and stack easily for high-speed operations.
- Be slip resistant for stable stacks and loads.
- Provide an excellent printing surface for market appeal.
- And yet, be economical.

These are very demanding requirements and so far no one has come up with a single low-cost material that will do all of these things well on all types and grades of fertilizer.

Paper, for instance, is, among other things,

- strong
- scuff resistant
- handles and stacks well, and
- is low in cost

but paper by itself does not provide moisture protection and is not resistant to acid attack. On the other hand, plastic film is an excellent moisture barrier and is resistant to acid attack. But many of you who have had experience with plastic report that it does not handle and stack as well as paper; that it punctures more easily, and that it is less stable on trucks and pallets.

However, when these two materials kraft paper and plastic film are used in the right combination with each other you can in effect pack your fertilizer in a *light-weight plastic bag* which is *wrapped with* paper. This is truly a superior bag for many grades of fertilizer and fertilizer materials because,

- The plastic film provides resistance to attack by superphosphates and the P-K grades.
- The plastic film also provides relatively low-cost moisture protection for the more hygroscopic nitrogen goods and materials such as ammonium nitrate.
- The paper provides a tough, puncture resistant *exterior.*
- The paper also provides excellent *handling and stacking qualities* and stable loads on pallets and trucks.
- And, finally, these bags can be packed with your present equipment.

Although the paper/plastic combination is available in all types of bags being manufactured today, the four types most commonly used for fertilizer and fertilizer materials are flat tube sewn open mouth, gusseted sewn open mouth, gusseted sewn valve, and stepped end pasted valve.

The following are some examples of why and how members of your industry are using them.

The first is a large fertilizer company in Texas that had serious problems with bag failure on its P-K goods. The corrosive action to the paper by the gases given off by the superphosphate caused the bags to fail. This company replaced the 100# asphalt laminated extensible ply formerly used in their gusseted sewn open mouth bag with 2 mil blown tubing and switched to a flat tube sewn open mouth. Performance is completely satisfactory in both 50# and 80# bags and for both truck and rail shipments. Although many people do not like the pillow shape of filled flat tube open mouth bags, for the paper/plastic combination construction, flat tube bags are more economical than gusseted bags. This is due to the fact that in flat tube bags, blown tubing can be used that is ¼″ narrower than the paper tubing that surrounds it. As a result, when the pre-weighed charge is dropped from the scale into the bag, the film which is smaller in diameter than the paper plies contributes significantly to the total strength of the package. This makes possible worthwhile reductions in the total basis weight of the paper plies to help off-set the cost of the film.

The next example is that of a large fertilizer producer in Ohio that had the same problem on *its* P-K goods in gusseted sewn open mouth bags. When this company switched from a PE coated ply to the paper/plastic construction, it chose to stay with gusseted bags and, therefore, flat film was used rather than tubing. Tests showed that 1½ mil film was adequate for this company. When flat film is used, it has the same dimensions as the paper plies and, therefore, does not contribute as much to total bag strength as the blown tubing in flat tube bags. This company reduced basis weight by 20# in its 80# bag to help compensate for the cost of the film. Here again, bag failure due to corrosive attack to the paper has been eliminated.

The third example is a company in New England that uses sewn valve bags. This company replaced 90AL with light weight film as the second ply to get increased moisture protection and better performance during the winter months. Average GFMV transmission is approximately *4.0 grams* for 90AL. For light weight film GFMV transmission is *approximately 1.5 grams.* This means double the moisture protection and greatly improved bag performance in the winter months since plastic film is more flexible than asphalt in cold weather.

The fourth example is a company in Wisconsin that is using stepped end pasted valve bags. As the company in Texas this one, too, was plagued with bag failure on their P-K goods. Here the problem was solved by simply adding light weight film to the bag between the two inside kraft plies. They continued to retain the PE coated Kraft ply next to the product to assure that the Kraft sheet did not break up and contaminate the fertilizer in case it was weakened by the P-K goods. This new paper/plastic construction has been used with complete success by this company since last December.

My final example is an ammonium nitrate producer in Florida and demonstrates the versatility of the paper/plastic combination. As all of you know, ammonium nitrate is a very hygroscopic product that has caused problems for years because of its tendency to form lumps after it is bagged. Like my first example of the fertilizer company in Texas, this ammonium nitrate producer switched from gusseted sewn open mouth to flat tube sewn open mouth and a 2 mil blown tubing inside ply. For ammonium nitrate, however, this company went one step further. It had the bag supplier heat seal the film ½″ above the stitchline in the end of the bag that was closed at the bag factory. The packing line was also equipped with a heat sealer so that the film at the top of the bag could be sealed ½″ below the sewing line after it was filled. This provides a *filled plastic bag* or *pouch heat sealed at both ends* that is inside of a paper bag. Performance has been outstanding—lump free storage for at least three months through the hot, humid summer months in Florida.

I rushed through the range of bag constructions in my five examples pretty rapidly. Since they are important I'd like to take a moment and review them.

First there is flat tube sewn open mouth. For this type, blown tubing ¼″ narrower than the paper walls can be used as the inside ply and if desired it can be heat sealed. Also flat film can be used between the plies but this construction can not be heat sealed.

Next there is gusseted sewn open mouth. For this type, flat film is generally used for either the inside ply or for placement between plies. Some bag suppliers can heat seal the end when the film is positioned next to the product. Equipment to seal gusseted bags in your plant is now available.

The next is gusseted sewn valve. It can be manufactured in the same construction as gusseted sewn open mouth and some bag companies can heat seal both ends.

Last is pasted valve bags both conventional flush cut and stepped end. Conventional flush cut pasted valve bags can be manufactured with either blown tubing as the inside ply or with flat film between plies. However, more work needs to be done on this bag to improve strength at pasted ends especially for temperatures below freezing. Currently, stepped end pasted valve can only be furnished with flat film. If positioned as the inside ply, the film must be securely attached to, and supported by, the adjoining kraft ply. A recent break through has been announced on this.

For the most part in all of these bags 1½ mil PE film is the minimum gauge that can be furnished as the inside ply and still maintain acceptable quality. Although the bag industry has been working on manufacturing techniques that will make possible the use of 1 mil or even thinner film on the inside, I'm not at all sure that the light weight PE films available *now* will be satisfactory anyway for the high analysis granular and blended fertilizer you produce today — especially for extended storage periods. Blended fertilizer in particular that contains both superphosphate and ammonium nitrate is especially difficult to store in bags during humid weather. Frequently bags for this type of product must provide high resistance to both moisture and acid attack.

To summarize briefly your industry needs better bags for certain grades. The use of film as an integral part of multiwalls is relatively new but the technical and production people in the bag industry have quickly met the challenge of successfully combining these two materials in a wide variety of bag types. The result, the paper/plastic combination looks like the best answer available today. But like any new item, some further refinements are desirable and development work is continuing especially in seeking out techniques for handling the light weight films and in finding new and better adhesives.

In the meantime, if you are having problems with material lumping during storage or with bag failure on superphosphates or on your P-K grades, you should look into the paper/plastic combination bags. Chances are good that one of them will solve your problem at little or no additional cost to you.

Mr. Peterman: Thank you Panelists. We are a little short on time. We have time for perhaps one or two questions. However, since there may be more questions on packaging, and there are a number of bag industry representatives here, I wonder if it would be out or order if we could have the bag industry representatives stand up, just to be identified so that you can ask them questions while you are here. Would you stand up, please?

A Conferee: I believe in the discussion here there has been an indication or a feeling on my part that it's quite a bit of a trial and error method in determining the type of bag to be used. I'm curious as to what steps have been taken to come up with an optimum size and type of bag to suit a particular requirement or need? The other question I have. Are there any industry standards for testing the various types of bags or bag materials?

Mr. Peterman: I think on the second part of that question Mr. Dively touched upon the wide variety of materials that are used for packaging fertilizer and the wide variety of fertilizers that are being packaged, and as I see this, panel might disagree with me, however, this comes right down to an individual problem of an individual company and there is a tailor making of a package to your product. Can you gentlemen add anything to the first part of the question?

Mr. Dively: In my opinion there certainly is a lot of available information on the performance of various kinds of bags, however, there is so much difference between the requirements that one company may have versus what another company may have, or even between plants within the same company, that although we have broad guidelines, for instance, on fifty pound fertilizer bags, if there is any such thing as a standard construction, it's probably two forties and one fifty with a PE coating. There are many companies that are using one forty and two fifties or three fifties, or some other construction that they found better suits their needs, than a so called standard bag. Using the broad guidelines, to my knowledge, you then generally go through a trial and error stage to see whether the typical bag will do the job as a starting point and then make changes as you see fit from experience with performance.

Mr. Peterman: I don't see trial and error as being a deterrent to designing good packaging. I think this should be a constant process. Our industry does change. There are new developments in both materials and equipment and I think we should all be in the trial stage with packaging.

A Conferee: We are very interested, sir, in plastic sacks. We use for 50 pounds five mil, six mil for the other and then on the eighty pound sack what is recommended?

Mr. Brown: A lot will depend on how you are shipping, how far, whether it's by your own trucks, driver's trucks, shipping by rail or are you shipping within a one state area, a four-county area, or all over the United States. Mixed fertilizer plants, generally speaking, probably ninety five per cent of all the fifty pound mixed fertilizer bags that are being used today are in a five mil construction. This has been found generally satisfactory. However, if you are doing something unusual, say you are packing fertilizer in Richmond and want to ship it to the West Coast, then certainly, I would suggest that you start with something heavier, probably a six or seven mil, for fifty pounds. On the eighty pound bag, the big deterrent to plastic has been, when you get up to an eighty pound bag and you even get in to as light a mil gauge as a six or seven, it's not always competitive in price with the paper bag. The same way with the 100 lb. bag. A fifty pound package is very competitive. In many cases a lower cost than the paper bag. On eighty pounds we would recommend starting out, depending on what you are packing and how you are shipping, anywhere from a six to a ten mil. This would depend on the situation.

Mr. Peterman: Gentlemen, I think we will have to shut off the questions because of time. However, this afternoon most of the bag company representatives will be here including the panel representatives and I certainly thank you for your time this morning.

Moderator Spillman: Thank you, Mr. Peterman and your panelists, for this splendid discussion. I will now turn the meeting back to Doctor Sauchelli.

Chairman Sauchelli: At one time in the fertilizer industry we heard nothing about plant food elements but the big three, N. P. K, or as some will have it, N, P_2O_5, K_2O. Then in time as knowledge grew others questioned "Why limit it to the big three?" It should be the big six, as NPK plus calcium, magnesium, and sulfur. In recent years we have seen how sulfur has made tremendous advances regarding its importance in plant nutrition.

Well, that went on for some time. Then came the observation that, no, it isn't only the big six, but actually there are fifteen or sixteen essential nutrients. Among them are what were called the "trace minerals," or the trace elements or the oligochemicals, or minor elements.

We have seen how by classification, which the human mind likes to do for convenience, the nutrients classified as "major" were those elements, NPK which were used in major quantities: not because they had any major importance but simply because they were used in major quantities and similarly, "minor" elements, became used in minor quantities. The minor elements so far comprise iron, copper, boron, manganese, molybdenum, and zinc and we begin to see evidences in the literature that more are about to be added to the list perhaps, two or three more.

As our knowledge of plant nutrition increases so does the importance of these other elements also increase. I think most people still like to use the term "trace elements," in identifying those elements that are used in minute quantities.

But how mix these minor elements, or micronutrients, in a large quantity of other materials and have uniform distribution? It's a big problem that the industry has had to recognize the situation and try to find ways and means of mixing them so as to maintain uniformity of distribution in the bag and in the field.

It takes research to find the answers and, as in so many other problems of basic research, there is one organization in this country to do the job that is of outstanding quality. I refer to the Research Center of the Tennessee Valley Authority. When you mention it you always associate with it its great leader, internationally known, who is going to be the next speaker. Mr. Travis P. Hignett. He is going to talk on the problems met within the production of micro-nutrients-enriched fertilizers. It's a pleasure, Mr. Travis P. Hignett, to have you here.

Problems In Production of Micronutrient Enriched Fertilizers

Travis P. Hignett

SEVERAL months ago Vince wrote me suggesting that I speak on the topic of the chemistry of micro-nutrient materials when mixed with various fertilizer materials in mixed fertilizers. We have scattered information on this subject, hardly enough to be of much value, I'm afraid in our present stage of research, so I prefer to talk on problems because that permits me to talk about the things that I don't know.

When I first started to work as a young chemical engineer, my boss whom some of you know, P. H. Royster, I used to come and report to him on progress on projects that I was working on, and then tell him what we still didn't know. And he'd break in and say "Don't start trying to tell me what you don't know. It will take too long."

So this morning I will not try to tell you all that we don't know, but I do mean to tell you some of the things that we have found out, as well as some of the things that we are trying to find out.

Last year Mr. Holden spoke to you on micro-nutrients and emphasized their importance, so I shall not dwell on this phase of it except to underline what he said, that we feel that they are of growing importance to the fertilizer industry.

A speaker who talks about *problems* gives himself the broad latitude of the unsolved — a convenient area in discussion of micronutrient-enriched fertilizers. We believe that research in this field is one of the most important needs in agriculture. I shall discuss with you some of the things that we are trying to find out, particularly about the reactions of micronutrients with other fertilizer materials and the effects of these reactions on the quality of the products.

We at TVA have a substantial program on micronutrient fertilization — a program including both agronomic research and chemical research. We have recently prepared a total of 120 tons of experimental fertilizers for micronutrient tests and demonstrations in several states. Included are 80 materials; 67 contain one or more micronutrients, and 13 are comparison materials without micronutrients. We hope to learn from these tests how the agronomic effectiveness of the micronutrients is affected by the method of mixing (blending, coating, or incorporation in granular or liquid fertilizers) and by the water solubility and other characteristics of the mixtures. We hope to learn how to use effectively low-cost sources of micronutrients.

Additional objectives of these trials are to learn more about the frequency of economic responses to micronutrient-enriched fertilizers, to delineate areas of micronutrient need, and to compare the effectiveness of various combinations of micronutrient sources and fertilizers when combined by practical procedures and applied by common techniques.

I think we have all been impressed by the percentage of trials that have shown significant response to one or more micronutrients. Sometimes the response has been spectacular; more often it has been moderate but economically significant.

When micronutrient materials are added to fertilizers, chemical reactions often result. Through these reactions, water-soluble materials may become insoluble or insoluble materials may become soluble. So we need to consider how changes in solubility of the micronutrients affect the agronomic usefulness of the products.

There is very little information on the availability of micronutrient sources in terms of the combined effects of granule size, water solubility, and placement. But we do have some evidence that water solubility is important when the micronutrient is supplied in a granular fertilizer. For example, some water-insoluble zinc materials (such as zinc oxide) are good sources when finally ground and mixed with the soil but are much less effective in granular form. When zinc oxide is incorporated in granules of ammonium phosphate, it remains insoluble and relatively ineffective. When incorporated in granules of ammonium polyphosphate, it becomes soluble and much more effective. We have too little information for generalized conclusions, but we can conclude tentatively that at least partial solubility in water is desirable for micronutrients in granular fertilizers.

A preference for granular fertilizers is growing among farmers, and industry is responding. So we assume in our research that nearly all solid fertilizers soon will be granular. We see little future for water-insoluble materials whose effectiveness depends on fine grinding and intimate mixing with the soil. A possible exception is suspension fertilizers; broadcasting on the surface, with disking or harrowing into the soil, may provide favorable placement for these finely ground, insoluble micronutrient sources.

Chemical Characterization and Evaluation

The water solubility of the micronutrient content of materials for our agronomic test program is determined by an empirical extraction method. Selected products are characterized more completely. We are also doing leaching tests, intended to simulate dissolution in the soil, and diffusion tests in which a pellet of fertilizer is placed in the soil and the rate and extent of movement of its micronutrient content into the soil are measured. We hope that the results of these tests will shed some light on the cause of differences in agronomic effectiveness of the materials.

Prescription Mixes and Premium Fertilizers

One of the first problems that the manufacturer of micronutrient-enriched fertilizer must consider is what elements to add and in what percentages.

In the prescription or symptom-treating approach, only those micronutrients known to be deficient in a particular soil for a particular crop are supplied. The amount is intended to be sufficient for correction of a serious deficiency. In the shotgun approach, smaller amounts of several micronutrient elements are added to fertilizers as insurance against mild deficiencies, and the resulting mixtures are often called "premium" fertilizers. A popular combination of micronutrients is boron, zinc, iron, manganese, copper, and molybdenum.

Premium fertilizers have the advantage that they can be manufactured in volume at low cost, whereas prescription mixing would be expensive or even infeasible in a large granualtion plant producing mixed fertilizers. A premium fertilizer may waste the farmer's money by supplying elements already present in adequate amounts. But if the farmer waits until the need for each element is clearly evident, he may suffer much greater economic loss; methods for determining whether addition of one or more micronutrents will result in an economic increase in yield are cumbersome, uncertain, or entirely lacking. This is particularly true when the deficiency is not severe enough to cause unquestionable deficiency symptoms in crops. As one farmer put it, "Our state college tells us not to use trace elements unless we need them, but they don't tell us how to find out what we need."

Perhaps a compromise between the symptom-treating and shotgun approaches would be best; to supply fertilizers that contain those micronutrients that are most likely to be needed in specific areas and for specific crops. More research will be needed for formulations of such fertilizers in most areas.

Liquid Fertilizers

The main problem in addition of micronutrients to liquid mixed fertilizers is solubility. Boron as sodium borate and molybdenum as sodium molybdate are sufficiently soluble for practical purposes in both orthophosphate and polyphosphate solutions. Compounds of zinc, copper, iron, and manganese are almost insoluble in ammonium orthophosphate solution such as 8-24-0 and in mixed liquid fertilizer made from it. When a solution of zinc sulfate is added to 8-24-0 orthophosphate solution, the zinc is precipitated as zinc ammonium phosphate ($ZnNH_4PO_4$). Similar precipitates are formed when salts of manganese, iron, and copper are added.

Polyphosphate solutions such as 11-37-0 and 10-34-0 sequester most micronutrient elements, thus dissolving and holding in solution substantial concentrations of them. The amount depends on the polyphosphate content of the solution and on the micronutrient material added.

We found that at least 2 percent zinc was dissolved by 11-37-0 from zinc oxide, zinc sulfate, or zinc carbonate. Zinc sulfide was not soluble, but calcined zinc sulfide (impure zinc oxide) was soluble. Some of the solutions containing over 2 per cent zinc precipitated zinc ammonium pyrophosphate after about 1 month.

Practical methods of dissolving the zinc materials were studied in the laboratory. Zinc carbonate, a byproduct from the zinc industry, dissolved readily in 11-37-0 at 80° F. with moderate agitation. Calcined zinc sulfide ore concentrate (impure zinc oxide) also dissolved readily. Zinc sulfate dissolved very slowly, even with vigorous agitation. A more practical way of adding zinc sulfate was as its water solution.

When finely powdered zinc oxide was added to 11-37-0, it formed globules that dissolved only after several hours of vigorous agitation. When prewetted with an equal weight of water, it dissolved in 12 minutes with moderate agitation.

In tests of the solubility of manganese in 11-37-0, the best results were obtained with hausmannite (Mn_3O_4); it gave solutions containing 0.2 percent manganese that remained free of precipitate on standing for 1 month. Manganese oxide (MnO) and manganese sulfate monohydrate ($MnSO_4 \cdot H_2O$) also dissolved in 11-37-0 to yield solution containing 0.2 to 0.3 percent manganese, but precipitates were formed in a few days. Manganese dioxide (MnO_2) and manganese carbomate ($MnCO_3$) were much less soluble.

Copper oxide (CuO) was sufficiently soluble in 11-37-0 to yield a solution containing 0.7 percent copper. Copper sulfate ($CuSO_4 \cdot 5H_2O$) was more soluble (1.5% Cu). Ferric sulfate [$Fe_2(SO_4)_3 \cdot 9H_2O$] dissolved in 11-37-0 to the extent of 1 percent iron.

Tests were made to establish the feasibility of dissolving several micronutrients in 11-37-0 simultaneously. The micronutrients were added as a mixture of sodium borate, copper sulfate, zinc oxide, manganese oxide (Mn_3O_4), and sodium molybdate in the proportions required to yield a solution containing 1 percent each of boron, copper, and zinc—0.2 percent each of manganese and molybdenum. When a dry mixture was added to 11-37-0, the formation of globules of zinc oxide hindered dissolution. This trouble was avoided by prewetting with 1.6 parts of 11-37-0 per part of dry mix. Dissolution of the resulting slurry in hot 11-37-0 (180° F.) was complete in 20 minutes. This base solution was then used to prepare mixed liquid fertilizer grades of 8-8-8, 5-10-10, and 24-12-0. The micronutrients remained in solution for at least a month.

Although magnesium salts will idssolve in liquid mixed fertilizers, a crystalline precipitate of magnesium ammonium phosphate ($MgNH_4PO_4 \cdot 6H_2O$) forms in orthophosphate solution. In polyphosphate solutions the precipitate is a magnesium ammonium pyrophosphate. The time required to form the precipitate depends on the pH of the solution and the concentration of magnesium. When 1 percent magnesium (as magnesium sulfate solution) was added to ammonium polyphosphate solutions, precipitation occurred after 1 day at pH 6.0, after 7 days at pH 5.5, and after 21 days at pH 5.0. Results with magnesium oxide were similar. The grade of the ammonium polyphosphate solution before addition of the magnesium materials ranged from 11-37-0 (pH 6.0) to 10-41-0 (pH 4.5).

Micronutrient-Enriched Suspension Fertilizers

With suspension fertilizers the solubility of the micronutrient carrier is immaterial except as it affects agronomic response. If the micronutrient carrier is insoluble in the suspension, its fineness should be such that it will neither settle rapidly nor clog spray nozzles. Materials smaller than 60 mesh generally are satisfactory; somewhat larger particles sometimes are acceptable. Experimental suspensions have been made with micronutrient additions from fritted trace elements and from sulfates of copper, zinc, and iron. In general, these materials had no important effect on the properties of the suspensions when the amount added was about 5 percent.

Incorporation of Micronutrient Materials in Superphosphate

Since superphosphates are quite acidic, many insoluble micronutrient materials may become soluble when incorporated in them. The boron in colemanite reportedly becomes soluble when the mineral is mixed with unammoniated superphosphate. Micronutrient frits react with unammoniated superphosphate during storage and become at least partly water soluble.

Incorporation of sodium borate in concentrated superphosphate (54% P_2O_5) has been studied by TVA in both pilot-plant and large-scale tests. In the pilot-plant tests, run-of-pile superphosphate and fertilizer borate (14.3% B) were granulated with addition of steam and hot water. The product was dried, cooled, and sized at 6 to 16 mesh. The yield of onsize product was 83 percent of the throughput; the remainder was recycled. The product contained 44 percent available P_2O_5 and 3.6 percent boron.

Operation in the large-scale plant was similar, except that dehydrated borate (20% B) was the source of boron. The operation was satisfactory, but the cooled product was dusty.

Storage properties of the products were very good. Free acid content of the superphosphate was low (0.6%). Water solubility of the phosphorus in the products was 5 to 10 percent lower than that in superphosphate without boron. The boron content was essentially all water soluble.

The boronated superphosphates were blended with unborated superphosphate and other granular materials to yield products of various grades. The blends were considered satisfactory.

A zinc-enriched concentrated superphosphate was prepared in bench-scale tests by granulating high-analysis superphosphate with zinc oxide. The product contained 51% available P_2O_5 and 9% zinc; it contained no free acid, and 12 percent of the zinc was water soluble. Further tests are planned in which the solubility of the zinc will be increased by addition of phosphoric acid during granulation. Production of tonnage lots of this material is planned for use in blends. In similar tests the addition of zinc sulfate increased the free acid content of the superphosphate of 7.6 percent; addition of zinc sulfide resulted in no apparent reaction. These materials are being tested in the greenhouse.

Incorporation of Micronutrients in Ammonium Nitrate

Incorporation of zinc and boron in ammonium nitrate was studied in the pilot plant. Borax, zinc-oxides, or both were mixed with hot concentrated ammonium nitrate solution (95%), and the mixture was granulated in a pan granulator. The composition of the products before addition of the usual conditioning agent was:

Percent by weight		
N	Zn	B
33.8	0.0	0.5
33.6	2.3	0.0
32.4	1.9	0.7

The boron and zinc compounds did not seem to affect the granulation or further processing of the granular products. Storage properties of the products were at least as good as straight ammonium nitrate. Sensitivity of the ammonium nitrate to detonation was not increased by boron or zinc. The boron content of products was water soluble; 30 to 40 percent of the zinc content was water soluble. The compound $3Zn(OH)_2 \cdot NH_4NO_3$ was identified in the zinc-containing materials.

Incorporation of Magnesium in Diammonium Phosphate

The possibility of incorporating 2 percent magnesium in diammonium phosphate was studied in the laboratory. Olivine, serpentine, or calcined magnesite was added to wet-process phosphoric acid, which was then ammoniated to produce granular diammonium phosphate (about 17-48-0). The products contained magnesium ammonium phosphate ($MgNH_4PO_4 \cdot H_2O$). The phosphorus content of the products was all citrate soluble, but the water solubility was somewhat lower than when no magnesium was added.

Incorporation of Micronutrients in Ammoniated Mixed Fertilizers

The effect of ammoniation of mixed fertilizers on water solubility of the zinc content was studied by USDA workers. When zinc sulfate ($ZnSO_4 \cdot H_2O$) or basic zinc sulfate [$ZnSO_4 \cdot 4Zn(OH)_2$ was incorporated in mixed fertilizer containing unammoniated ordinary or triple superphosphate, 80 to 90 percent of the zinc was water soluble. Ammoniation of the superphosphates lowered the water solubility of the zinc, sometimes to zero. Inclusion of dolomite in the mixtures also lowered the solubility of the zinc. When zinc chelate ($Na_2ZnEDTA$) was added, the zinc remained water soluble. The amount of zinc added was 0.5 or 0.05 percent of the mixture.

The lack of solubility of zinc in fertilizers containing ammoniated superphosphate or ammonium phosphate is attributed to the formation of zinc ammonium phosphate ($ZnNH_4PO_4$).

The behavior of micronutrients other than zinc and boron when incorporated in mixed fertilizers has not been studied extensively. Soluble salts of manganese, iron, and copper probably would become insoluble when incorporated in mixed fertilizers that were ammoniated to near neutrality.

A little information on the behavior of managnese was obtained in TVA studies. When manganese sulfate ($MnSO_4$) was incorporated in ammoniated mixed fertilizer (6-24-24 grade) at levels that supplied 1.4 or 2.8 percent manganese in the product, about 20 percent of the manganese was water soluble. The extraction was made with a weight ratio of 10 parts of water to 1 part of sample. The pH of the water extracts was 4.6. When manganese oxide (MnO) was incorporated in similar fertilizers, very little water solubility resulted. Similar results were obtained when manganese sulfate or oxide was applied as a coating to the surface of the granular fertilizers. Addition to unammoniated mixed fertilizers was not tried.

Addition of Micronutrients to Ammonium Polyphosphate Fertilizer

Ammonium polyphosphate sequesters some metal ions, including zinc, iron, manganese, and copper. The effect of this sequestration on solubility of micronutrients in solid ammonium polyphosphate generally is similar to that of 11-37-0 liquid fertilizer, which I have discussed.

A rather large number of ammonium polyphosphates containing various combinations and amounts of micronutrients have been prepared in the TVA pilot plant in several-ton lots and are being field tested in several states. Solubility of the micronutrient content of these materials is governed by a complex set of conditions, but some tentative conclusions can be drawn.

When zinc oxide was incorporated in ammonium polyphosphate at levels of 1 to 3 percent zinc, 80 to 100 percent of the zinc was water soluble, the proportion depending on the polyphosphate content and perhaps other factors. When higher levels of zinc were added, a smaller proportion was water soluble. The method of incorporation was not critical; results were similar whether the zinc compound was dissolved in the acid prior to ammoniation, added to the melt after ammoniation, incorporated during granulation, or applied as a coating to the finished granule.

In one series of tests, dry mixtures of zinc oxide and ammonium polyphosphate were tableted under pressure. Microscopic examination showed an absence of chemical reaction between the two. when the pellet was placed in the soil, the ammonium polyphosphate and about 80 percent of the zinc dissolved and diffused into the soil. The solubility of zinc in combinations with ammonium polyphosphate thus does not depend on chemical reaction of the components prior to placement in the soil. In similar tests with pellets containing diammonium phosphate and zinc oxide, most of the diammonium phosphate dissolved and diffused into the soil, but the zinc remained at the pellet site as zinc ammonium phosphate ($ZnNH_4PO_4$).

Substantial amounts of iron, manganese, and copper were soluble when appropriate compounds of these elements were incorporated in ammonium polyphosphate. Several lots of ammonium polyphosphate contained combinations of micronutrients (up to five: Cu, B, Mo, Mn, and Zn). There was some evidence of interaction; manganese, for example, was more soluble when added with other micronutrients than when added alone. These systems are so complex, however, that few clear-cut conclusions are possible right now.

Field tests showed that the zinc oxide—ammonium pholyphosphate combination was quite effective in granular form, even when the application rate was as low as 0.5 pound of zinc per acre. Zinc oxide incorporated in ammonium orthophosphate, or straight granular zinc oxide, was much less effective.

Practical Problems

Manufacturers of compound fertilizers cannot economically manufacture small lots of special products. If micronutrients are to be incorporated in mixed fertilizers economically, the tonnages will have to be substantial. Perhaps further study will lead to a general recommendation for specific crops and areas that will permit volume production of micronutrient-enriched fertilizers.

An alternative to incorporation proper is blending of granular mixed fertilizers with micronutrient carriers. Blending can be done economically in small lots. Methods are being developed for preparing uniform mixtures by causing powdered micronutrient carriers to adhere to the surface of granular fertilizers. However, this method results in odd or fractional grades which are not permitted in some states. When 87 pounds of manganese oxide is added to 1 ton of 8-16-16, for example, the grade of the resulting mixture is 7.7-15.3-15.3. Some states permit blending of micronutrient carriers with approved grades — a concession that greatly encourages use of micronutrients on a prescription basis.

Bulk blenders are in a more fortunate position than compound fertilizer manufacturers for supplying mixtures containing micronutrients on prescription basis. In most states they are not required to stick to approved grades. In states where approved grades are required, blenders can make them by using granular fillers.

The main problem is to obtain a uniform mixture of the micronutrient material with the granular fertilizer materials. Perhaps the best solution is to add powdered micronutrients and cause them to adhere to the surface of granular blends by means of oil, water, or other binders. This method is being developed by TVA and has been used successfully by some blenders, but further work is needed to evaluate it. The method works best when the micronutrient material is a fine powder, preferably minus 100 mesh. The granular fertilizer materials, the fine micronutrient material, and the binder are stirred in a batch mixer until the fine material adheres to the surface of the granules. With oil as binder, the requirement is in the range of 1 to 3 percent. Diesel fuel oil is suitable, but it discolors paper fertilizer bags and adversely affects the strength of some types of plastic bags. Used motor oil is more effective as a binder than dieasel oil and is less likely to discolor or weaken bags. Both oils are cheap and widely available.

CHAIRMAN SAUCHELLI: Thank you, Travis.

I once heard William Beebe, the great naturalist, say, "our present trend is to find out more and more about less and less," in other words, to concentrate in the small field. We've seen, in Mr. Hignett's presentation, how much more is being done with these micronutrients and how much more will be done in the future, and so our industry should plan for more and more chemical studies because the days that Joe Whittington tells us about are very, very simple as compared with what the future fertilizer man is going to be faced with. It's getting to be a fascinating but complicated industry. Who has the first question to ask Mr. Hignett? This is a very interesting subject, a very fascinating one.

A CONFEREE: Would you hesitate to use this oil technique when the ammonium nitrate is high?

MR. HIGNETT: We would advise not using oil on any mix containing more than sixty per cent ammonium nitrate.

CHAIRMAN SAUCHELLI: Any more questions? You have a great opportunity here to ask questions of a man who is very, very familiar with this problem and it's perfectly probable that the industry will face more and more — any questions?

A CONFEREE: Sir, in your

group of micronutrients and liquids you said an 8-8-8 with a one per cent zinc copper and boron concentrate. Did you have much problem with the three elements and one liquid or did this go into solution very nicely?

MR. HIGNETT: The base solution contained one per cent zinc copper and boron and two tenths per cent of the other materials. The base solution was a 10-30-0, I believe, used to make 8-8-8, so the amount of the micronutrient in 8-8-8 is proportionately less, say about three tenths per cent instead of one percent.

CHAIRMAN SAUCHELLI: Any other questions? Here is a question.

A CONFEREE: Did you ever consider the possible effect of the zinc phosphorus in the used motor oil?

MR. HIGNETT: No, I don't think we ever did. I have no information on how much zinc phosphorus and other materials may be in used motor oil. We considered the possibility of lead in used motor oil and surprisingly there is quite a volume of information on the effect of lead on crop production. We concluded that the amount in motor oil would not have any adverse effect on crops. I don't know about phosphorus or zinc.

CHAIRMAN SAUCHELLI: Another question?

A CONFEREE: I think that there might be one per cent zinc in used motor oil, also there would be lead salts and other contaminates and I think it is a good question, what would be the eventual effect on crops with all these miscellaneous contaminates—

MR. HIGNETT: Does anyone have any suggestions where one can get a representative analysis of used motor oil?

CHAIRMAN SAUCHELLI: Thank you, Travis, once more, for a very splendid presentation.

We will have to move on. I think it was back in 1928 that I first got interested in the subject of potassium nitrate. At that time I tried to induce the Aluminum Company of America to get interested in its marketing by bringing to the United States some of the potassium nitrate that was being produced just outside of Rome, Italy, in an aluminum plant, with which they were associated. I did get one hundred pounds of that Italian potassium nitrate and used it in test work at an experiment station that I was interested in. I was impressed with its effectiveness as a source of N and K. From time to time I tried to induce American potash producers to produce potassium nitrate in this country. It is a good fertilizer.

Finally it has happened and the next speaker, who is a real good friend of the Round Table, he has been on our program a number of times and he's always welcome. John O. Hardesty, of the United Sttaes Department of Agriculture, is going to tell us something about the use of nitrate of potash in granular mixed fertilizers. John?

Use of Potassium Nitrate In Granulated Fertilizers

John O. Hardesty

POTASSIUM nitrate was first suggested for use as fertilizer more than 300 years ago, but owing to previously high costs of production it has only recently come within the category of competitive fertilizer materials. Its agronomic value as a source of both N and K has long since been demonstrated. The technical literature is replete with references to the necessity for nitrate-nitrogen on various crops. Recommendations of the Tobacco Workers' Conference this year call for—at least 35% of the total nitrogen in preplant fertilizers for flue-cured tobacco to be in the nitrate form—and 50% of the total nitrogen in sidedressers to be in the nitrate form. These are in addition to the usual requirements for low chloride ion and for nominal amounts of SO_3 ion, recommended at the rate of 75 to 100 lbs. SO_3 per acre.

In a previous paper, published in 1961, we discussed the physical and chemical properties of potassium nitrate with respect to its use in tobacco fertilizers, as well as its use in the formulation and granulation of high-analysis grades, such as 8-16-16, 10-10-10, and 15-10-10. The results of that study and of previous investigations show that:

(1) The low hygroscopicity of potassium nitrate favors satisfactory physical condition of mixtures containing it,
(2) Its low salt index facilitates formulation of mixtures for salt sensitive crops. In this respect it has been widely used for solution feeding in greenhouses,
(3) Its physiological basicity favors its use in non-acid forming fertilizers, and
(4) Its chemical stability favors retention of nitrogen during processing and the safety of the product with respect to fire hazard.

In regard to safety from fire hazard, recent experiments at Beltsville with 5-10-15 grades containing up to 308 lbs. of KNO_3 per ton showed no sign of oxidation reaction up to 300° F. The same results were obtained with 8-0-24 containing up to 492 lbs. KNO_3 per ton. Results on these mixtures were confirmed by the Bureau of Explosives, Association of American Railroads. Their explosion tests on the mixtures were sol negative. Their report indicated that the materials examined would not be subject to Interstate Commerce Regulations for shipping purposes.

The above mentioned attributes of potassium nitrate are widely recognized and would appear to present little reason for further study except as they may be examined for accomplishing a specific purpose in fertilizer manufacture and use. However, one characteristic of this material that is less widely recognized by fertilizer technologists, — and one that certainly deserves further study in fertilizer processing,—is its high solubility coefficient. Its rapid increase in solubility with increase in temperature has a unique influence on the liquid phase content of most mixtures containing it. I say, "unique" because of all the common nitrogen and potash materials, KNO_3 has the highest solubility coefficient and appears to have an influence on the liquid phase of the mixture peculiar to it alone.

In 1937-38, several of our published articles demonstrated that soluble salts were effective in promoting granulation of mixtures. Later work showed the phenomenal effects of highly-soluble urea and ammonium nitrate in promoting granulation. At that time, our results on the granulation of 8-16-16 grades containing 120 lbs. KNO_3 per ton showed the granules to have the highest crushing strength of some 16 other mixtures studied. During this period of granulation research and development, the percentage of soluble salts in the mixture was an important item on our formula sheets.

Soluble salt content is important to the liquid phase content of the mixture, both with respect to granulation processing and with respect to the physical condition of the granular product. The "liquid phase" or "solution phase" of mixed fertilizers are familiar terms in fertilizer technology. Rader, in 1947, measured the liquid phase of mixtures and found that, in general, the liquid phase consists of salts in solution, involving chiefly the ions, NH_4^+, NO_3^- and Cl^- followed by moderate amounts of K^+ and smaller amount of HPO_4^{--} and SO_4^{--} ions in that order. Calcium and magnesium were seldom present in the liquid phase. The method of measurement requires weeks of exacting work on a single sample and is impractical for routine determinations. However, the results of the earlier work provided a basis for estimating the solubility effect of fertilizer materials on the liquid phase and granulating properties of mixed fertilizers.

Table I. Comparative Solubility

Material	lbs. soluble per 100 lbs. water at 68° F.	194° F.	Ratio
KNO_3	32	202	6.31
KCl	34	54	1.59
K_2SO_4	11	23	2.09
$(NH_4)_2SO_4$	76	98	1.29
NH_4NO_3	195	740	3.79

Today, I would like to apply solubility data to mixtures that have been used in recently reported laboratory, pilot-plant, and commercial-scale tests, where potassium nitrate was found to aid granualtion and improve the physical quality of the granular product.

Table I shows the comparative solubility, in lbs. per 100 lbs. water, of common nitrogen and potash materials at 68° F. (an arbitrary average atmospheric temperature), and at 194° F. (an arbitrary average granulation temperature).

The last column shows the ratio of the solubilities of the materials at these two temperatures. This value will be referred to in this paper as the solubility coefficient. The increase in solubility between these two temperatures is greater than 6-fold for KNO_3 as compared with less than 4-fold for its nearest rival NH_4NO_3. Used at the rate of 200 lbs. per ton of mixture, KNO_3 and NH_4NO_3 have the same capacity for entering the liquid phase at a granulation temperature of 194° F. It is worthy of note that 200 lbs. of NH_4NO_3 is equivalent to the NH_4NO_3 in 356 lbs. of No. 440 (32-56-0) ammoniating solution. This is about the average proportion of ammoniating solution in the average mixed fertilizer. Used at rates higher than 200 lbs. per ton of mixture, NH_4NO_3 has far greater capacity for entering the liquid phase than does KNO_3; for example, at 194° F., 740 lbs. of it is soluble in 100 lbs. of water (col. 3) while only 202 lbs. of KNO_3 is soluble. The high liquid-phase content of such mixtures containing large proportions of ammonium nitrate from ammoniating solution necessitates their granulation by slurry methods rather than by the conventional nonslurry method.

The high solubility coefficient of KNO_3 (last col. Table I) also indicates that this fertilizer material crystallizes most rapidly from the liquid phase with a decrease in temperature of the mixture following ammoniation and granulation. This tends to decrease the proportion of liquid phase, inhibit further agglomeration and formation of oversize granules, cause crystal knitting, and increase the hardness of the granule, and thus improve processing and physical stability of the product.

Table II shows the solubility of KNO_3 in 100 lbs. water at the granulation temperature of 194° F. as compared with that of salt pairs containing the same amount of nutrients. Figures in parenthesis

Table II. Solubility of Salt Pairs Having Nutrient Content Equivalent to KNO_3. Temp. 194° F.

Salt		Amt. soluble per 100 lbs. water	
Kind	lbs.[1]	lbs.	Percent of total solids
KNO_3	202 (202)	202 (202)	100
NH_4NO_3	78	78	
	(226)	(132)	58
KCl	148	54	
$(NH_4) SO_4$	124	98	
	(272)	(152)	56
KCl	148	54	
$(NH_4)_2SO_4$	124	98	
	(302)	(121)	40
K_2SO_4	178	23	
NH_4NO_3	78	78	
	(256)	(101)	39
K_2SO_4	178	23	

[1] Figures in parentheses are totals.

indicate the total pounds of salts required (Col. 2) and the proportion soluble (Col. 3). The total quantities of salts required (Col. 2) show that only 202 lbs. KNO_3 supplies the same amount of nutrients as 226, 272, 302 and 256 lbs. of the salt pairs listed in descending order. This gives KNO_3 a distinct advantage in formulating high-analysis fertilizers, especially when space in the formula is limited. The last column shows that 100% of the KNO_3 is soluble whereas only 39 to 58% of the salt pairs (listed in ascending order) are soluble at the granulation temperature.

Low Nitrogen Grades

Let us observe how these known solubility data correlate with the results of recently reported laboratory studies, pilot-plant tests and commercial plant practice on the granulation of mixtures formulated with and without KNO_3. Recent laboratory granulation studies conducted at Iowa State University by Boylan and Kamat (Ag. & Food, Sept-Oct. 1964) on low nitrogen grades, formulated as shown in Table III, showed that the 5-10-15 grade (right hand column) containing 460 lbs. KNO_3, as compared with the 2-10-15 grade, middle col.) containing 425 lbs. KCl, was more cohesive and more sensitive to moisture change at the agglomeration stage, and required only 6.5% moisture for optimum agglomeration vs. 15.8% for the 2-10-15 mixture. The 5-10-15 mixture containing KNO_3 gave somewhat higher yields of granular product which was more uniform in appearance and nutrient distribution than that which the 2-10-15 grade without KNO_3.

Table III. Formulation with- and without KNO_3[1]

Ingredient	Pounds per ton of 2-10-15 Without KNO_3	5-10-5 With KNO_3
Norm. super	1020	1020
NH_3	52	52
Sul-po-mag	222	222
KNO_3	none	460*
KCI	425	75*
H_2SO_4	50	50
Sand	281	171*

[1] From Boylan and Kamat (2).
* Significant change in formulas.

Table IV. Liquid Phase in 2-10-15 and 5-10-15[1] Per 100 pounds Water, Temp. 194° F.

Ingredients involved	2-10-15 Amt. present	Amt. sol.	5-10-15 Amt. present	Amt. sol.
	pounds per ton			
KNO_3	none	none	460	202
KCl	425	54	75	54
Sand	281	none	171	none
Totals	706	54	706	256
Amt. Soluble	8%		36%	

[1] Data from Boylan and Kamat (2).

The three units of nitrogen derived from KNO_3 in the 5-10-15 grade are not present in the 2-10-15 grade. Therefore these experiments compare directly the effect of replacing KCl with KNO_3 in the formula. The difference in these two formulas is indicated by the asterisk in the last column of the table;—namely,

460 lbs. KNO_3 vs. none
75 lbs. KCl vs. 425
171 lbs. sand vs. 281

When this difference in formulation is projected in Table IV to show the solubilities of the involved materials at the granulation temperature, we find that 36% of total solids involved were soluble in the 5-10-15 mixture formulated with KNO_3 and only 8% in the 2-10-15 mixture without KNO_3. This could well explain why the mixture with KNO_3 required only 6.5% moisture for optimum granulation as compared with 15.8% required by the mixture without KNO_3.

In general, the granulation of low-nitrogen grades, such as 3-9-9, 4-16-16, and 5-20-20, requires high temperature and high moisture content. The heat and moisture are usually derived from various combinations of water, occasionally steam or applied heat and ammoniation. There is little room in the formula for the inclusion of high-nitrogen salts present in ammoniating solutions. The chief source of heat, therefore, is from anhydrous ammonia.

Table V shows the effect of salt solubility on a 5-20-20 mixture when 100 lbs. KNO_3 is substituted for some of the nitrogen and potash in KCl and $(NH_4)_2SO_4$. Of the materials involved in the substitution, the KCl and $(NH)_2SO_4$ combination (top of table) furnishes only 17% (right hand Col.) of soluble salt per 100 lbs. water at the granulation temperature, whereas the KNO_3, KCl, $(NH_4)_2SO_4$ combination (bottom of table) furnishes 31% of the involved salts in the soluble form. The effects of the KNO_3 on this 5-20-20 formula should be similar

Table V. KNO_3 Effect on Liquid Phase 5-20-20 Grade 5% Moisture, 194° F.

Ingredient		Nutrient (lbs.)		Amt. soluble per 100 lbs. water	
Kind	lbs.	N	K_2O	lbs.	Percent of total solids
KCl	667	—	400	54	6
$(NH_4)_2SO_4$	250	50	—	98	11
Total	917	50[1]	400	152	17
KNO_3	100	13	44	100	12
KCI	600	—	356	54	7
$(NH_4)_2SO_4$	113	24		98	12
Total	813	37[1]	400	252	31

[1] Remaining N from ammonia.

to those obtained by Boylan and Kamat on the 5-10-15 grade.

Indeed, various reports from industry on the granulation of low-nitrogen grades with nominal amounts of KNO_3 subsituted for KCl in the formula, indicate:

(1) Less moisture requirement for granulation
(2) More onsize granular product
(3) Less recycle, and
(4) Better appearance and stability of the product.

Results of laboratory granulation tests on 8-16-16 by Boylan and Johnson were reported at the recent ACS meeting in Chicago. These authors substituted 200 lbs. KNO_3 for equivalent nitrogen and potash in $(NH_4)_2SO_4$ and KCl respectively. Otherwise the mixtures were identical in formulation. The solubilities of the ingredients involved in the substitution are given in Table VI. Of the ingredients involved, only 15% are soluble in the mixture without KNO_3 (top of table, right hand Col.) whereas 39 percent (lower right of table) are soluble in the mixture containing KNO_3. The authors of this (unpublished) paper reported that the mixture without KNO_3 required 12.2% moisture (not shown) for optimum granulation while the one with KNO_3 required only 9.6%; also, that the KNO_3 increased product yield from 62 to 68% and improved the drying characteristics and physical quality of the product.

Table VI. Effect of KNO_3 on Liquid Phase of 8-16-16 Grade at 194° F. 5% Moisture

Ingredient involved Kind	lbs.	Nutrients N lbs.	Nutrients K_2O lbs.	Salt soluble per 100 lbs. water lbs.	Salt soluble per 100 lbs. water %
without potassium nitrate					
$(NH_4)_2SO_4$	438	90	—	98	10
KCl	543	—	320	54	5
Totals	981	90	320	152	15
with potassium nitrate					
$(NH_4)_2SO_4$	313	64	—	98	11
KNO_3	200	26	88	200	22
KCl	397	—	238	54	6
Totals	910	90	326	352	39

Boylan and Johnson (6).

Table VIII. 10-15-15 Potato Grades Comparison of Liquid Phase at 194° F. 5% Moisture

Ingredient	Mixt. No. 1 Solid lbs.	Mixt. No. 1 Liquid lbs.	Mixt. No. 2 Solid lbs.	Mixt. No. 2 Liquid lbs.
NH_4NO_3	271	271	271	271
Urea	25	25	25	25
$(NH_4)_2SO_4$	142	98	76	76
KNO_3	None	None	100	100
KCl	254	54	254	54
K_2SO_4	294	23	208	23
Totals	986	471	934	549
Amt. soluble	48%		58%	

High Nitrogen Grades

Mixtures containing 10%, or more, of nitrogen are usually formulated with sufficient ammoniating solution to supply heat and soluble salts (NH_3NO_3 or Urea) for effective granulation. There would appear to be little object, therefore, in formulating such mixtures with KNO_3, merely to increase the liquid phase. However, in commercial granulation operations involving 136 tons of 10-15-15 mixture formulated as shown in Table VII, the insertion of 100 lbs. KNO_3 in the formula was claimed to:

Improve agglomeration.
Reduce fines recycle by 50% which would permit increase in production rate from 20 to 23 tons per hour without changing the load on equipment.
Improve appearance of the granule, and
Improve the hardness of the granule.

Table VII. Formulation 10-15-15 Potato Grades (commercial operation, 136 tons)

Ingredient	Pounds per ton without KNO_3	Pounds per ton with KNO_3
	(1)	(2)
N Soln. 440	410	410
$(NH_4)_2SO_4$	142	76[1]
H_3PO_4	200	200
Concd. super	214	172
Norm. super	512	616
KNO_3	none	100[1]
KCl	254	254
K_2SO_4	294	208[1]
MgO	62	62
Borate	2	2

[1] Significant changes in formulation.

These mixtures were reported to require about the same amount of moisture for optimum agglomeration.

When we project these two formulas on the basis of ingredient solubility (Table VIII) we find that there is not a great deal of difference in their soluble-salt contents; it being 48% vs. 58% of the ingredients that are chiefly responsible for the liquid phase. The slightly greater amount of soluble-salt in the mixture containing KNO_3 might make the mixture a little more sticky and thus be partially responsible for improved agglomeration. However, the high solubility coefficient of KNO_3 likely is most responsible for the good behavior of this 10-15-15 mixture in processing and in storage. There is rapid crystallization of KNO_3 from the liquid phase when the mixture begins to cool following the maximum temperature of agglomeration. This has the same ef-

Table X. 8-0-24 Grades Comparison of Liquid Phase at 194° F. 5% Moisture

Ingredient	Without KNO_3 Solid	Liquid	With KNO_3 Solid	Liquid
NH_4NO_3	260	260	184	184
$(NH_4)_2SO_4$	356	98	253	98
KNO_3	None	—	410	202
K_2SO_4	978	23	649	23
Mg limestone	361	—	471	—
Clay coating	25	—	25	—
Totals	1980	381	1992	507
Amt. Soluble	19%		25%	

fect as the addition of dry recycle material to the wet granular material. It decreases the quantity of liquid phase at the surface of the granule, inhibits further agglomeration, and improves the shape and hardness of the granule.

These conclusions, substantiated by commercial-plant tests, are also verified by the chemistry of the soluble salt system in the 10-15-15 mixture shown in Table VIII. Of the salts present (Col. 1) including those that are likely to be formed by chemical reaction in this mixture, the KNO_3, $(NH_4)_2SO_4$, NH_4Cl, and K_2SO_4 are the stable salts, while KCl and NH_4NO_3 are the unstable salts in the saturated liquid phase of the mixture. Of the four stable salts that will crystallize from the liquid phase on cooling, KNO_3 is present in largest amount (Col. 4) and crystallizes faster per unit drop in temperature because of its higher solubility coefficient.

N-K Grades

More striking examples of the effect of KNO_3 as an aid to granulation are the pilot-plant tests by Huillet and coworkers reported on at the recent ACS meeting in Chicago. These tests were with N-K grades, such as 8-0-24, 10-0-32, 15-0-15, and 16-0-16 with and without KNO_3. These authors suggest that the use of KNO_3 in these grades increases the liquid phase for efficient granulation in conventional equipment, and, at the same time, obviates the use of a preneutralizer which wastes heat and lowers production rate. At first thought, these two statements seem contradictory.

However, let us compare the formulas of 8-0-24 mixtures shown in Table IX, one without KNO_3 and the other containing 410 lbs. KNO_3 per ton. Note what the KNO_3 did to the formula; it reduced the Nitrogen Solution requirement from 371 to 263 lbs. per ton of mixture; the sulfuric acid requirement from 335- to 242 lbs.; and the K_2SO_4 requirement from 978 to 649 lbs. It increased the magnesium limestone from 361 to 741 lbs. per ton of mixture. In consequence the heat and moisture contents were greatly reduced, and this could well provide the difference in operating conditions; that is, the requirement of a preneutralizer for the mixture without KNO_3 and conventional equipment for the mixture containing 410 lbs. KNO_3.

Table IX. Formulation 8-0-24

Ingredient	Pounds per ton Without KNO_3	With KNO_3
N Soln. 440	371	263
H_2SO_4	335	242
KNO_3	None	410
K_2SO_4	978	649
Mg limestone	361	471
Clay coating agent	25	25
Total	2070	2060

Table X shows the proportions of salts soluble in the 8-0-24 mixtures with—and without KNO_3. Indeed, the proportion of soluble salt is higher in the mixture containing KNO_3; 25% versus 19%. But here again, the high solubility coefficient of KNO_3, or in other words, the rapid crystallization of this stable salt on cooling below the agglomeration temperature likely is most responsible for the processing behavior of the mixture containing this material. At least one commercial plant claims to have increased production from about 13 to 20 tons per hour on 8-0-24 grade, by formulation with KNO_3 and processing in conventional equipment, as compared to formulation without KNO_3 and then using a preneutralizer to dissipate heat.

Storage Quality

Most high-analysis mixtures are dried to a moisture content of 1%, or less, before storage. The hardness or physical stability of the

Table XI. Solubility of Salt Pairs Having Nutrient Content Equivalent to KNO_3. Temp. 50° F.

Salt Kind	lbs.[1]	Amt. soluble per 20 lbs. water lbs.	Percent of total solids
KNO_3	202 (202)	4	2
NH_4NO_3	78	32	
	(226)	(38)	17
KCl	148	6	
NH_4NO_3	78	32	
	(256)	(34)	13
K_2SO_4	178	2	
$(NH_4)_2SO_4$	124	15	
	(372)	(21)	8
KCl	148	6	
$(NH_4)_2SO_4$	124	2	
	(302)	(17)	6
K_2SO_4	178	15	

[1] Figures in parentheses are totals.

granule during storage and handling of the product is dependent on the moisture content and the proportion of soluble salt dissolved in this moisture. Table XI shows the solubility of KNO_3 and salt pairs having nutrient contents equivalent to that of KNO_3 in 20 lbs. water (i.e. 1% in the mixture) at a storage temperature of 50° F. Only 2% of the KNO_3 (right hand Col.) is soluble under these conditions, whereas 6 to 17% of the salt pair, selected in ascending order, is soluble. This accounts for the favorable effect of KNO_3 on the storage quality of mixtures.

In conclusion, these solubility data corroborate the results of granulation tests by previous investigators showing that, in addition to the value of KNO_3 as a source of nitrate nitrogen and potash, it possesses properties which make it useful as a granulation aid in the processing of mixed fertilizers, including low- and high-nitrogen grades of N-P-K fertilizers and concentrated N-K fertilizers. The most important property of KNO_3, in this respect, is its high solubility coefficient, which increases the soluble-salt content of the liquid phase at the granulation temperature, provides rapid decrease in liquid phase in the latter stages of processing, and allows minimum solubility in the liquid phase during storage and handling of the resulting products.

KNO_3 has been reported to aid in the commercial granulation of 12-24-24 fertilizers. Further laboratory investigations are in progress to determine the solubility relationships in high-analysis mitxures such as 12-24-24 and 20-20-20. It appears likely that slurry methods of granulation offer the best means of processing such grades. It will be interesting to see what contribution potassium nitrate can make here.

CHAIRMAN SAUCHELLI: Thank you, John, for such a thorough study and excellent presentation.

CHAIRMAN SAUCHELLI: We stand adjourned until 2 p.m.

Thursday Afternoon Session, November 12, 1964

The Round Table reconvened at 2. P.M.
Joseph E. Reynolds and Albert Spillman, moderators

CHAIRMAN REYNOLDS: Fertilizer processing technology continues to change and has many advances to meet the many changes in the requirements of our fertilizer industry. Innovations, and processing techniques have kept pace, or actually in many cases, have preceded the trend.

A new process to produce an extremely high analysis mixed fertilizer was recently announced by the Scottish Agricultural Industries.

The fertilizer technical knowhow and the high stature of the work of the Scottish Agricultural Industries are well recognized on a worldwide basis. We are indeed very fortunate today to have two representatives largely responsible for this process from SAI with us. Dr. George Bremner and Mr. John Harris have travelled a considerable distance to be with us and to discuss the process right down to the very beginning knowhow of this SAI-R process.

Our next speaker was actively engaged in the development of the process from the very beginning, and is highly qualified to discuss the subject. It is my provilege and pleasure to introduce my good friend, Mr. John Harris.

MR. JOHN HARRIS (Scottish Agricultural Industries, Limited.): Thank you very much. Good Afternoon, Gentlemen:

First, I would like to say that Doctor Bremner and I count it a real privilege to have been invited to give this paper and to be with you today. We always enjoy coming over to the United States, of course, because of the wonderful treatment we receive, and this occasion has certainly been no exception. Now to tell you a little about this new process that we have developed.

The SAI-R Process For High Concentrated Granular Fertilizers

Dr. George Bremner and John Harris

THE continuing increase in concentration of compound fertilizers has been achieved by the greater use of materials such as ammonium phosphate in place of superphosphate and either ammonium nitrate or urea in place of ammonium sulphate. These new materials are more soluble and more temperature sensitive than the previous constituents, and thus have resulted the need for modification of existing methods or to the development of new methods for manufacture.

The desirable features of a process specifically designed for granulating materials of this type into compound fertilizers can be indicated by a consideration of the physical properties of the materials themselves.

Diammonium phosphate is thermally unstable. It has a measurable partial pressure of ammonia at 80°C and above 100°C, this pressure rises very rapidly. Therefore to minimize ammonia loss

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(1) U. S. Patent No. 3,049,419 and No. 3,907,833

when processing diammonium phosphate, it is necessary to restrict the temperature of operation, and also to minimize these air volumes used in the system. It can only be in this way that high ammonia losses from the system are avoided. However, a reduction in the temperature of operation has, in practice, the disadvantage of reducing the rate at which moisture is removed from the system, i.e. the drying rate of the material may be lowered significantly, and larger driers operating at lower temperatures are required.

The principal differences in the properties of compound fertilizers containing ammonium nitrate from formulations which contain ammonium sulphate as the principal source of nitrogen are—

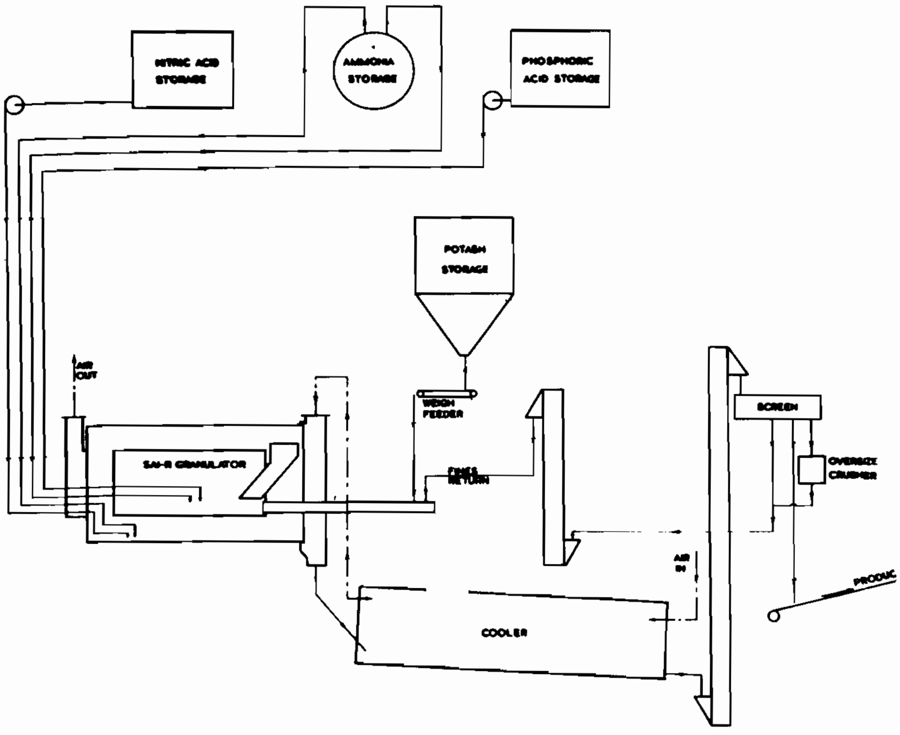
(1) The materials soften or become plastic at lower temperatures (when dry 95° to 120°C as against 140° to 160°C.

(2) They are considerably more hygroscopic (about 2½ to 4 times) and tend to form a surface "skin" which inhibits moisture release.

(3) They are less thermally stable.

It is worth considering the differences in a little more detail. The sensitivity to moisture and temperature of fertilizers containing ammonium nitrate necessitates granulation at low temperatures and low moisture levels if serious overgranulation is to be avoided. With the usual granualtion processes, this means that a low moisture is required in order to avoid very large external recycling of dry solids. (For example, ammonium phosphate slurries at 12-14% water are used.) In practice, some sort of balance is required between these two, but limitations in the handling of low moisture slurries in general mean that relatively high recycle systems cannot be avoided. The other approach to the slurry handling problem is to do a partial preneutralization followed by the final neutralization during the granulation stage.

The observation, that formulations containing ammonium nitrate, are considerably more hygroscopic, is simply a reflection of the very low water vapour pressures above these solids so that it is difficult to dry them. It is well known that granulation by the normal techniques generally involve a high proportion of agglomeration. That is, solid particles are brought together and held in that form by a "glue," usually ammonium/ammonium phosphate slurry or ammonium nitrate slurry. To dry granules so formed, it is necesfor that moisture inside the granule to diffuse to the surface before it can be evaporated. During this movement the moisture carries with it the more soluble salts present — among them ammonium nitrate — and this material is brought to the surface. The water evaporates and if the temperature is too high leaves a "skin" which reduces the rate at which further moisture can reach the surface and be evaporated. Recent techniques suggested by T.V.A. (e.g. preliminary cooling before drying) ten dto minimize this difficulty and have been applied with success by them when handling pure ammonium nitrate.



SAI-R GRANULATION PLANT FLOW DIAGRAM

Figure 1. SAI-R Granulation Plant Flow Diagram.

To turn to thermal instability, it is well known that ammonium nitrate when heated beyond a certain point, say about 300°F, can begin to decompose even in the absence of air. Decomposition can be catalyzed by the presence of a chloride. During the decomposition acids are formed which further catalyze and accelerate the rate of decomposition. This particular aspect of decomposition can be minimized by having a reservoir of ammonia present so that it neutralizes any acid as soon as it is formed. A suitable "reservoir" is diammonium phosphate, and we have found that to have this material in intimate contact with ammonium nitrate does minimize the tendency to decompose.

The features then which seem to us to be desirable in a process for making a high analysis fertilizer containing a high proportion of ammonium nitrate such as the 23:11½:11½ which we now produce are—

(1) One which operates at low temperature and so overcomes the problem of "skin" formation and thermal instability.

(2) One which avoids the possibility of hot spots (e.g. at drier inlets).

(3) One which can produce diammonium phosphate readily and produce it in intimate contact with ammonium nitrate

(4) One which operates at a low moisture level and so avoids the production of large quantities of oversize, and minimizes external recycling.

As many of you well know, we in Scottish Agricultural Industries following the trend to high concentration, developed successfully to the pilot plant scale, a process for the manufacture of potassium metaphosphate,[1] a 0:57:37 fertilizer. We decided reluctantly, however, that in our particular circumstances in Britain and at that particular time we should not go ahead with full scale application. We therefore turned our thoughts to the nitrogen component where up to that time (1959/60) ammonium sulphate had been our chief nitrogen carrier. Our largest plant was of Dorr design for the production of ammonium phosphate compounds of the following formulations — 12:12:18, 13½:13½:13½, 12:24:12 and 15:10:10.

Our choice for the new nitrogen source fell on ammonium nitrate and our agronomists told us that any plant we put in should certainly be capable of producing a high analysis 2:1:1 grade.

Our first efforts were naturally directed towards the production of these new compounds on the existing type of plant. Trials were consequently run on a pilot plant of this form, and also on another well known method. The difficulties revealed by these trials suggested that an entirely new approach was well worth considering. By that time we had already conducted small scale trials on what eventually developed into the SAI-R process, and it was decided to build a large pilot plant of output 1-2 t.p.h. On this unit some 2,000-3,000 tons of a variety of grades were produced and used for testing in the field and under large scale bag and bulk storage conditions. As a result of this we were convinced that this process had considerable advantages and also it produced a product of excellent physical properties.

SAI-R PROCESS (11)

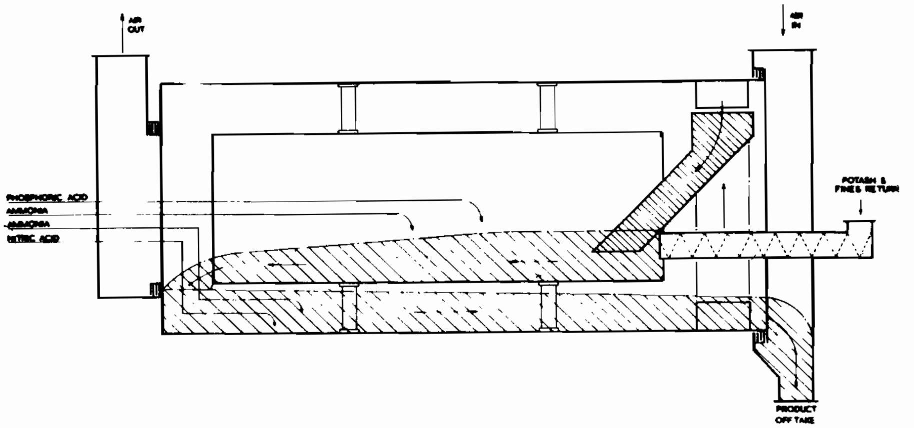


Figure 2. SAI-R Granulator Diagramatic.

The general solids flow diagram of a granulation plant based on the SAI-R process has one significant difference from more conventional plants in that the reaction, granulation and drying take place in the one unit.

The salient features of the process are—

(1) The complete drying of the product is by chemical reaction only. This means that the acid concentrations have to be chosen so that on reaction with ammonia the heat liberated will drive off all the water present and give a dry product. Thus hot spots are avoided.

(2) Normal external recycling is minimized, the solids being recirculated within the unit. This allows the addition of the acids direct with the complete elimination of reaction vessels for the preneutralisation of phosphoric and nitric acid and the avoidance of slurries. Further, ammonium nitrate is never handled as such, and by a suitable choice of addition point the possibility of reaction between nitric acid and KCl is avoided.

(3) The high recirculation ratios allows granulation to be achieved by a layering mechanism. Moreover, granulation is carried out at low moisture levels and all drying takes place on the surface by the heat release from the chemical reaction.

(4) A distinct zone with a restricted air flow to increase the ammoniation efficiency in the formation of D.A.P.

(5) External recycle can be limited to that required solely for screening out product.

(6) Ammonium nitrate and D.A.P. are produced in intimate contact so that thermal instability is minimized.

To turn to details of the SAI-R granulator itself. The granulator consists of two concentric drums with the solids progressing along the outer drum, elevated via buckets, hopper and chute into the inner drum and being displaced along the inner drum until they spill over and get into the outer drum again. In a large scale production unit the rate of solids movement is about 600 tons per hour. Excess solids are removed from the system at the elevator end of the outer drum and returning fines and cracked oversize along with any necessary solids feed such as potash are returned through an augur into the inner drum.

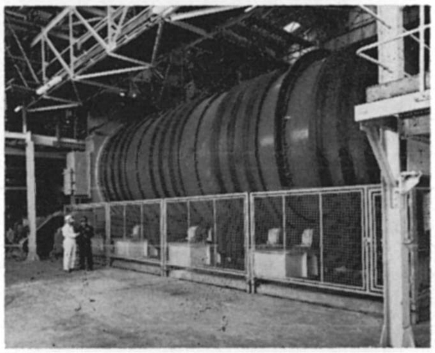


Figure 3. SAI-R Granulator Leith Fert. Co.

Air is drawn through the outer drum in a direction countercurrent to the movement of solids in that part of the granulator. The air movement in the inner drum is restricted since the only inlet is through the chute carrying the solids. This inner drum is used for the formation of D.A.P. by distrib-

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(11) U. S. Application No. 188,766, No. 377,368 and No. 206,622

uting phosphoric acid on the rapidly moving granular bed and injecting gaseous or liquid ammonia under the bed of acidified granules.

The other acids, nitric acid and/or sulphuric acid are distributed and neutralized on the bed in a similar manner at one end of the outer drum. The material then traverses along the outer drum where it is dried before the cycle commences again into the inner drum.

It is at the elevator end of the outer drum that the material is at its lowest moisture level and so it is at this point that the material is removed for size grading and extraction of product.

A large scale product unit based on this SAI-R process is now in operation and some practical data obtained from this point exemplify the various points mentioned earlier.

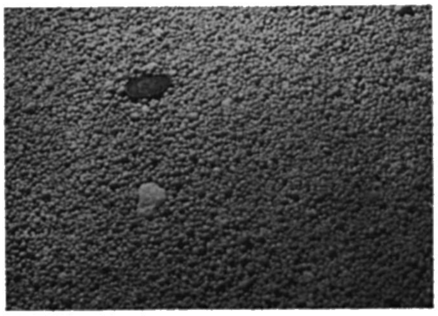

Figure 4. Material Wisch from SAI-R Granulator.

There are two granulator units, each 15 ft. diameter and about 36 ft. long, rotating at 14 r.p.m. The relatively high speed of rotation (about 70% of critical speed) is simplified by mounting the unit on pneumatic tires.

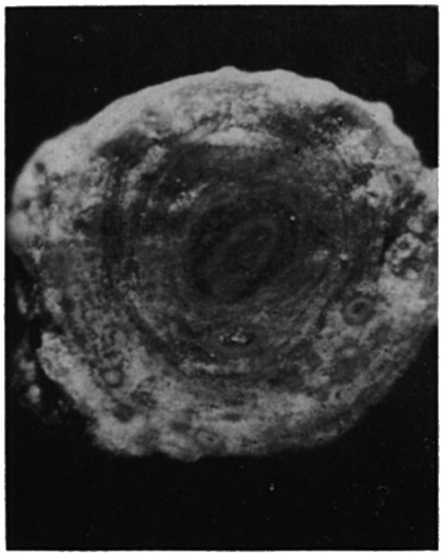

Figure 5. Section of Granule.

There is no slurry handling in the process, and with the exception of potash, all raw materials are metered into the plant as gases and liquids.

The internal solids recirculation ratio varies with the formulation being processed and is generally in the range of 30:1 to 60:1.

The granulation efficiency within the unit is so high that this does not determine the size of the recycle. This is determined by the need to crack a proportion of product for reintroduction of fresh nuclei, and in practice design allows for a recycle ratio of about 1½:1.

The acid concentrations which are required depend on the extent of the ammoniation and formulations. For example, phosphoric acid of 40 to 42% P_2O_5 is used with high potash formulations such as 16:16:21 and in the range 44 to 46% P_2O_5 for high nitrogen formulations such as 23:11½:11½. The nitric acid supplied by our Kuhlmann plant is 69% HNO_3 and it is our practice to use it at this concentration, any water required for balancing heat evolution being supplied by incorporating a proportion of unconcentrated phosphoric acid.

The ammonia slip from a granulator has been so far about 5-7% of the ammonia feeds and as such is readily recovered in conventional gas scrubbing equipment.

The operating temperatures have been in the range of 80°C to 95°C depending again on the formulation being processed and the moisture level of the product is generally about 0.2/0.3%.

The plant is designed to produce a number of compounds to give an overall annual production of about 150,000 long tons per annum.

Since granulation is achieved by layering in preference to agglomeration, the granules are extremely dense and round being built up of successive layers. This process leads to more uniform granulation and the discharge from the granulator normally operates with greater than 70% product size material. Another advantage is the lack of oversize material in the system since this reduces the serious problem of cracking materials of a plastic nature.

In conclusion then, we believe that the merits of the process may be summarized as follows—

(1) The elimination of external reaction vessel and slurry handling.
(2) No handling of ammonia nitrate as such.
(3) No fuel and no hot spots.
(4) A product of superior quality of well recognizable characteristics.
(5) Safer working.
(6) Simpler feed control.
(7) A process which should lend itself well to being made automatic in operation. Perhaps this is a large step in changing granulation from an art to a science.

CHAIRMAN REYNOLDS: We have a few minutes for questions. Actually, John has also consented to talk to us here about another process that they have developed so we're going to keep him up here for another fifteen minutes, but first of all are there any questions particularly concerning this process that we can direct to John?

A CONFEREE: I don't believe you gave production tons per hour. You gave us the annual tonnage. What is your per hour production?

MR. HARRIS: This figure varies as the grades change. The output is at its lowest in the case of the 2-1-1, very much higher with compounds such as 1-1-1, 1-1-1½, etc.

We didn't start up our large units until August so our operating experience has not been great enough to give you really accurate figures at the present time.

A CONFEREE: Would you care to tell us the size of your cooler?

MR. HARRIS: Yes, I can tell you the size of this unit and I hope that Mr. Weber will be able to confirm it. Our previous plant used the Dorr process and we are now using the drier of the Dorr process as the cooler on the SAI-R plant. You see, it was just convenient for us to do so, and if I remember correctly it is about ninety feet long by eleven feet in diameter, certainly larger than is necessary.

Can you confirm these dimensions, Mr. Weber?

Mr. Weber: That's right.

A Conferee: Do you have any problems of buildup scaling, in the granular—

Mr. Harris: Our troubles with this difficulty have been fairly small up 'til the present time. You see, before we built the large plant we built a pilot plant which would produce between one and two tons an hour and operated this for 2-3 years. On it we overcame most of the difficulties that were likely to give trouble on the full scale plant.

We used the two to three thousand tons of product which we made in this pilot plant unit for agronomic trials in the field and for large scale bag and bulk storage tests. The results of these tests were large factors in the choice of this process. That is, the excellent properties of the product made by this process.

You will see that we gained a lot of experience on the pilot plant scale. Indeed, I have another film with me about the pilot plant if anybody would like to see it. Certainly this unit allowed us to avoid a large amount of possible trouble on the big plant.

A Conferee: Mr. Harris, of the ratios 2-1-1, 1-4-4, how many different grades do you produce at those ratios, by this process?

Mr. Harris: In our first year of operation we are restricting the main plant to two grades. They are the 16-16-21 and the 23-11½-11½, but we put out about five or six grades and the plant will certainly make a 1-1-1 which will be about a 17½-17½-17½, and 1-2-1 which if I remember right is 14½-29-14½.

A Conferee: But your market would only require one grade of each of these different ratios, is that correct?

Mr. Harris: This is what we hope.

A Conferee: You wouldn't have two grades of a 1-1-1, for example.

Mr. Harris: We try to persuade the market to accept just one concentration, that is one grade, per ratio as soon as ever we can. The reason is, of course, that it is pretty expensive having storage and so forth for several different grades of the same ratio, and after all there is very little to be gained by it.

A Conferee: Mr. Harris, could you tell us the speed of rotation, the rpm, on your granulator and cooler?

Mr. Harris: I don't remember the figure for the cooler but I believe that it has not been changed from when it was working as a drier. For the granulator the speed of rotation is 14 rpm. This means that it has a peripheral speed of around 700 feet a minute and is turning at about 70 per cent of critical speed. The shape of the bed which we can obtain and the shape of the roll is very important for granulation.

A Conferee: I seem to have missed how you get the product out of the ammoniator. In other words, you're feeding it and you come on around back and then you lift and bring it back in.

Mr. Harris: You will remember from the film the chute that material passes down on its way from the outer drum into the inner. The way product is at present being removed is to have a second leg in that chute. In other words, we have a bifurcated chute, one leg of which feeds the inner drum, and the second feeds the offtake, so that we can take out, at a controlled rate, as much material as we want for screening out product and perhaps cracking a bit for further nuclei production. This is an alternative method to that we show in the film.

Chairman Reynolds: Perhaps there will be questions after the next comment here but, as perhaps you've read in the trade journals and also seen and heard from people who have travelled from Europe and from England and Scotland, the SAI people have developed another process which produces a product called PhoSAI and Mr. Harris has kindly consented to mention a few things along this line and give us a little insight to that product, so John?

A New Form of Ammonium Phosphate With Granulation Properties Similar To Superphosphate

***Dr. I. A. Brownlie**
Paper given by John Harris

Summary

Scottish Agricultural Industries Limited have developed a new form of ammonium phosphate which is sold in large quantities in the United Kingdom under the registered Trade Mark 'PhoSAI'. This product has properties very similar to superphosphate and can be used directly as a replacement for superphosphate in T.V.A. and conventional types of NPK granulation plants. The product is in the form of a powder similar in properties to superphosphate and it can be transported easily in bulk by sea, rail or road. It is cheaply produced by a novel process, stores without caking and has excellent granulating properties thus allowing conventional granulation plants to produce concentrated NPK products at an increased rate in terms of plant food units. No alterations are required to be made on these granulation plants and the product can, if desired be transported to them over long distances from a central production factory. Savings can thus be made compared to the expensive handling, storage and transport of phosphoric acid. The product can, if necessary be further ammoniated either at source or destination to various levels of diammonium phosphate content.

I Introduction

Superphosphate which was first manufactured in Great Britain about the middle of the nineteenth century has formed the basis

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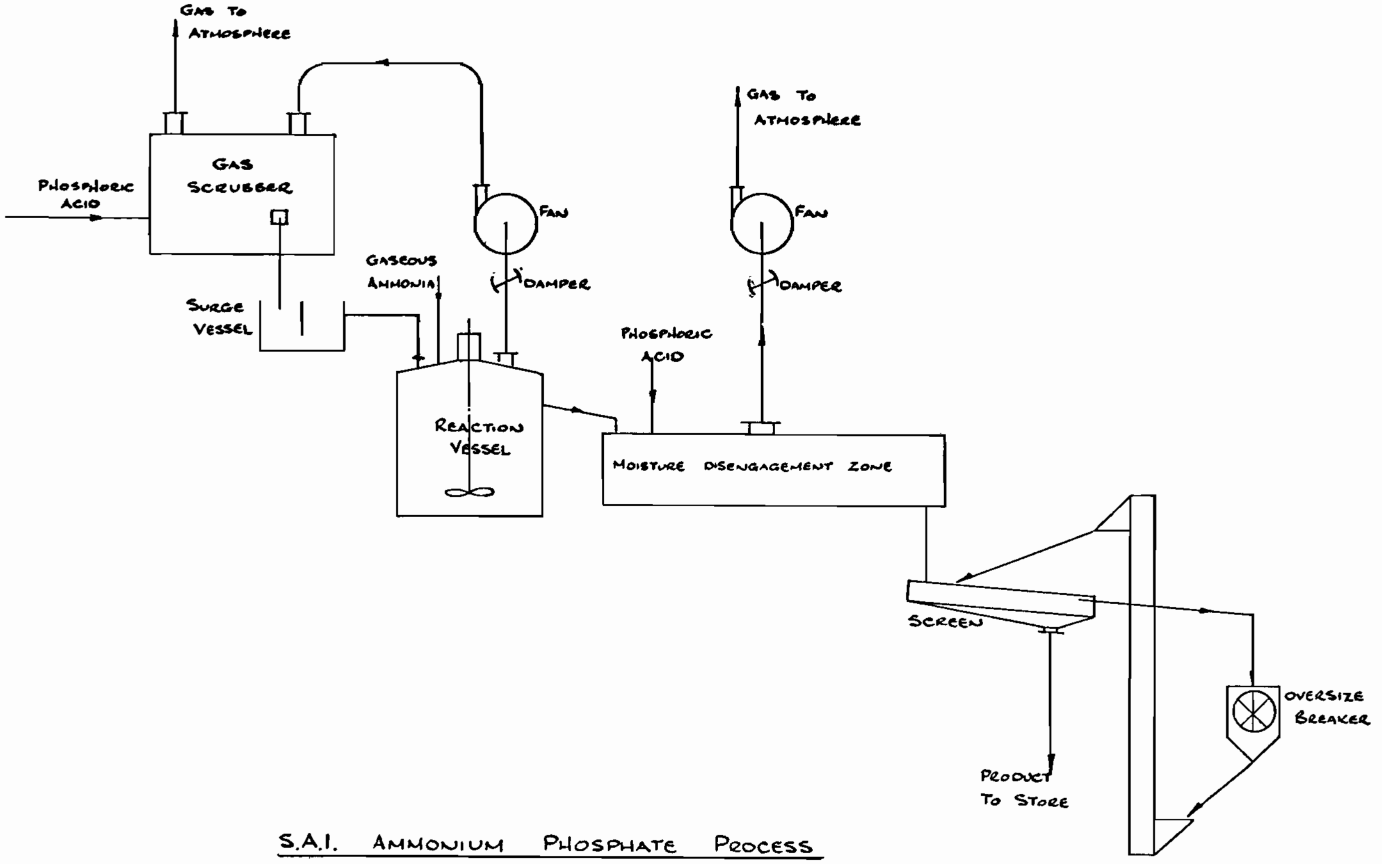
*Scottish Agricultural Industries Limited.



S.A.I. Ammonium Phosphate Process

of the fertilizer industry for over a hundred years and its production has grown steadily to a world wide figure of about 4½ million tons of P_2O_5 per annum. There is, however, a growing tendency for superphosphate to be replaced by products based on phosphoric acid.

The prime reason for replacing superphosphate is the need for increased concentration of plant foods. Many types of plants, both batch and continuous, were designed for superphosphate manufacture over the years but basically superphosphate remained a low cost form of soluble P_2O_5. The need for greater concentration of plant foods has inevitably led to greater manufacturing cost, particularly as the first stage normally involves phosphoric acid manufacture. It is necessary therefore that (a) the succeeding stage of conversion from phosphoric acid to the solid form should be a cheap one; (b) the material should be as concentrated as possible; (c) it should be easily incorporated in granular NPK fertilizers; (d) it should store well; and (e) it should be capable of being transported easily in bulk and handled in conventional equipment. The production of such a material is a worthwhile challenge not only because of the tremendous global target of P_2O_5 tonnage as superphosphate which could be converted but also because of the increasing tendency to convert phosphate at the source to a soluble concentrated form of P_2O_5 with the object of saving shipping and transport costs.

II Development of the Product

Scottish Agricultural Industries Limited worked for several years on an ammonium phosphate replacement for superphosphate and it was shown that the use of a form of crystalline monammonium phosphate, from which had been removed certain impurities such as iron and aluminum phosphates, originally present in wet process phosphoric acid, would not granulate with ammonium sulphate and potassium chloride. About three years ago S.A.I. found that solid monammonium phosphate had the property that if the moisture content was above, say, 15% the product could be worked into a fluid mass although it appeared to be solid and easily handled if not so worked. Moreover, if the moisture content was lower, no amount of physical handling, vibration or pressure could soften or liquefy it. The thixotropic property was thus found to be critically dependent on moisture content. From these experiments it was deduced that if solid monammonium phosphate could be produced at a moisture content between 6% and 12% it would have properties beneficial to granulation in a conventional granulation plant. If such a solid monammonium phosphate were fed into a plant using steam for granulation, its incorporation in an NPK fertilizer would be similar to that achieved with superphosphate. Additional moisture in the form of steam would quickly bring the monammonium phosphate to the thixotropic stage and would aid granualtion. This is the basis of the S.A.I. process for granualtion of NPK fertilizers[1] based on the S.A.I. form of ammonium phosphate, which is described below. This is different from granular monammonium phosphate where the initial moisture content is similar to that of the ammonium sulphate and potassium chloride, i.e. below 1% and

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[1] British Patent Specification No. 951,475.

addition of moisture, increasing the moisture content of all the components by approximately the same amount, overwets the ammonium sulphate and potassium chloride constituents. It was, therefore, believed that solid monammonium phoshpate containing 6%-12% moisture would give a more homogeneous NPK product than that obtained with granular monammonium phosphate.

III Description of the Process

Development of a process for producing a powder form of ammonium phosphate containing between 6% and 12% moisture according to requirements was successfully completed. The S.A.I. process[2] shown in Figure 1 is basicly simple with a capital cost about one-quarter of the cheapest published process for ammonium phosphate using ammonia and wet process phosphoric acid. The process is carried out in two stages:—

(1) Gaseous ammonia and wet process phosphoric acid are reacted in an agitated stainless steel reaction vessel to produce an ammonium phosphate slurry of N/P atomic ratio about 1.3.

(2) Phosphoric acid is added to the slurrry from stage 1 in such proportions that a magma of N/P about 1.0 is produced. The magma is then treated in a novel machine developed for the purpose of encouraging moisture disengagement. The moisture released is vented to atmosphere and the solid monammonium phosphate discharged from the mixer is screened before being transferred to storage. Any oversize material is milled before being recycled to the screen.

Process capital for a 100,000 t/a year plant under U.K. conditions is about £100,000 for battery limits, i.e. import or raw materials, gaseous ammonia and 40% P_2O_5 average strength phosphoric acid to export of screened product from the plant. Raw material and utility consumptions are given in the table below.

If desired the process can be adapted to produce directly ammonium phosphate containing a substantial proportion of diammonium phosphate. 'PhoSAI' can also be ammoniated easily to the diammonium phosphate form which in turn possesses excellent granulation properties.

IV Specification of 'PhoSAI'

A typical chemical analysis of the product using Nauru phosphate as the P_2O_5 source is 11% nitrogen, 47% water soluble P_2O_5 and 48% total P_2O_5 with a moisture content of about 6%. The corresponding figures for Morocco phosphate are 11% nitrogen, 50% water soluble P_2O_5, 51.5% total P_2O_5 and 6% moisture. A typical screen analysis of this material is approximately as follows:—

	%
+ 5 mesh B.S.S. =	6
− 5 + 7 " " =	10
− 7 + 12 " " =	15
−12 + 16 " " =	12
−16 + 30 " " =	19
−30 " " =	38
	100

The product which is sold in the United Kingdom under the registered Trade Mark 'PhoSAI' has excellent storage properties and can be stored satisfactorily in bulk. Storage of ammonium phosphate in this way reduces the requirements of storage for P_2O_5 in the form of phosphoric acid.

Table I. To produce 100,000 tons/annum of 'PhoSAI'

Raw Materials	
Phosphoric acid (40% P_2O_5) as tons P_2O_5/annum	48,000
Gaseous ammonia (tons/annum)	14,000
Sulphuric acid (94% H_2SO_4) as ton/annum 100% H_2SO_4	5,000
Utilities	
Power K.W./ton	10
Steam ton/ton	0.1
Labor 1 man/shift	

V Use of 'PhoSAI' for Concentrated Granular NPK Production

'PhoSAI' has been used to manufacture a wide range of concentrated fertilizers. It can be used to produce concentrated NPK fertilizers containing ammonium phosphate as a sole source of P_2O_5 or it can be used in conjunction with superphosphate. The form of granulation is similar to that obtained with superphosphate based products giving a homogeneout-product as distinct from granular mono- and diammonium phosphates which give a coating of nitrogen and potash salts on the phosphate nucleus.

The granulation moisture requirements on full scale plants for a 12:12:18 fertilizer were examined using different forms of monoammonium phosphate, the other ingredients being ammonium sulphate, potassium chloride and single superphosphate.

The results showed clearly that the granulation moisture content is considerably lower with 'PhoSAI' (0.8-1.4 tons H_2O/ton P_2O_5) than for other forms of monammonium phosphate (1.9-2.5 tons H_2O/tons P_2O_5.) This means that drying costs per ton of product are lower if 'PhoSAI' is used as the source of P_2O_5 in fertilizers. Furthermore the average moisture content of the raw materials is highest when 'PhoSAI' is used and consequently the amount of added moisture necessary to achieve granulation is smaller. This moisture is normally added as steam so that the significant reduction in steam requirements means a reduction in the cost of steam per ton of product.

The source of P_2O_5 in concentrated fertilisers manufactured on conventional granulation plants is usually either triple superphosphate, monammonium phosphate or diammonium phosphate. Results for the manufacture of 12:12:18 fertilizer shows a much lower granulation moisture content for 'PhoSAI' when compared with diammonium phosphate and as a consequence fertilizer drying costs and steam raising costs are greatly reduced.

Similarly results for two 16:9:9 formulations made from 'PhoSAI'

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[2] British Patent Specification No. 951,476.

and triple superphosphate respectively demonstrated the lower steam requirements necessary with 'PhoSAI 'as well as the lower granulation moisture requirements which would result in reduced drying costs.

A large number of different grades of fertilizers have been manufactured in conventional plants using 'PhoSAI'.

In general the products made are:—

(i) high P_2O_5 fertilizers ranging from 14:7:7 to 20:10:10
(ii) high P_2O_5 fertilizers ranging from 10:15:10 to 12:24:12
(iii) high K_2O fertilisers ranging from 9:9:25 to 13:13:20

The 1:1:1: fertilizers such as 13½:13½:13½ have also been produced.

An important aspect of the use of 'PhoSAI' is its suitability for use as a replacement for superphosphate in T.V.A. type granulation plants. Experiments have shown that it incorporates easily in the production of NPK products by the T.V.A. granulation methods.

When 'PhoSAI' is used to supply all or part of the P_2O_5 in granular compound fertilisers, the temperature of the solids leaving the drier can be maintained at 120/125°C with no ammonia loss and no reversion of soluble P_2O_5 to a water insoluble form. When other materials which are more heat sensitive than 'PhoSAI', e.g. urea, ammonium nitrate, etc. are used in the mix then the temperature of the solids leaving the drier must, of course, be reduced.

VI Production of Non-Caking NPK Fertilizers

Tests were carried out by one manufacturer on the keeping qualities of fertilizer made with 'PhoSAI'. Plant runs were made on the same grade of fertlizer using (a) 'PhoSAI' and (b) triple superphosphate as the main source of P_2O_5. The results showed that for the same plant load and exit drier temperatures the moisture content of the product made with 'PhoSAI' was lower than that for the product made with triple superphosphate. Furthermore on concentrated fertilizers this manufacturer finds that with 'PhoSAI' he is able to dry the product to a moisture level low enough to prevent caking. On the same fertilizer made with triple superphosphate he is unable to do this.

'PhoSAI' can be used in the production of powder NPK fertilizers for horticultural applications and in these products a reduction in caking has been obtained when compared with the equivalent superphosphate based products.

VII Other Applications for 'PhoSAI'

'PhoSAI' can be granulated by itself with a high granulation efficiency in conventional plants. Moisture requirements using a horizontal pan as the granulating unit vary from 0.4 to 0.5 tons water per ton P_2O_5. With a product moisture content of under 1% a typical analysis of the granular material is 12.5% nitrogen, 50% water soluble P_2O_5 and 51% total P_2O_5. The granules produced are hard and well formed and can be used for bulk blending or direct distribution as required. Similarily the production of the diammonium phosphate form of 'PhoSAI' mentioned earlier and its granulation to produce 18-46 D.A.P. is most important for application in bulk blending. 'PhoSAI' has proved itself to be a suitable replacement for superphosphate in the move towards more concentrated fertilisers, and it is believed that it will be suitable for phosphate rock producers who wish to solubilise P_2O_5 as the source for supply to world markets. It is particularly suited for wet process phosphoric acid manufacturers who wish to transfer P_2O_5 over long distances to granulation plants since it handles more easily than phosphoric acid and is cheaper to transport.

VIII Patents

S.A.I. have filed patent applications in all the major manufacturing countries of the world in respect of (i) the process of manufacture of S.A.I. ammonium phosphate and (ii) the process of manufacturing NPK fertilisers based on S.A.I. ammonium phosphate.

CHAIRMAN REYNOLDS: Thank you very much, John.

Earlier I mentioned that we were also fortunate in having another gentleman with us from Scotland. I would like to ask Dr. George Bremner to stand up so that he might be recognized.

Dr. Bremner is Director of Research for Scottish Agricultural Industries. Would you care to add anything to the comments, Dr. Bremner?

DR. GEORGE BREMNER: Good afternoon. There is one point I should like to mention. Both to-day and yesterday we have heard about fertilizer products containing in the region of 1% of moisture, and you may wonder why we in S.A.I. lay stress on the fact that the moisture content of our products from our new processes are so small—down in the 0.2-0.3% moisture region.

Obviously we have good reason for working in this low moisture range as the lower the moisture content, the more work has to be done to get it into the desired range. The reason why we operate at such low moisture contents is to ensure good physical properties of the product. There is, of course, much evidence to show that moisture content is critical in this respect. Work we have carried out in our own laboratories has brought us to conclusions similar to those given at your Conference last year by Mr. Wm. Lewis of Du Pont who pointed out that, as the content of ammonium nitrate in a fertilizer increases, good physical properties are more difficult to come by.

We concluded therefore that low moistures were necessary to ensure good physical properties of our ammonium nitrate fertilisers, and even so—and I think this is the point that Mr. Harris did not mention—we also carried out a surface treatment for the two grades we are producing at the moment, namely 23:11½:11½ which contains about 60% ammonium nitrate, and the 16:16:21 grade.

Although we produce these products at such low moisture contents, we still believe that surface treatment for both is necessary to confer excellent physical characteristics.

I think that is all, Mr. Reynolds.

CHAIRMAN REYNOLDS: We're

going to ask these gentlemen to stay close by. The panel which comes on following my comments I'm sure will have some questions that, although they are pertinent here they will also apply to some of the processes that would be in Scotland. So we are going to call on them as the other questions come along later, for any comments that they might have, so they will be around with us the rest of the afternoon.

But as for the official talks, again, we really appreciate your efforts in coming over many, many miles to be with us. We really feel that you brought us a wonderful process. From the standpoint of description, the product is most impressive, so again, thanks very much.

Question And Answer Period

MODERATOR ALBERT SPILLMAN: Yesterday afternoon our "Questions and Answers Period" was most interesting, timely and with much activity from our audience. This afternoon's session, I am confident, will also be interesting and helpful to analyze some of our technical manufacturing problems. The questions assigned to our panelists this afternoon, similar to yesterday's session, were picked by your Executive Committee from your inquiries, to be most urgent for thorough discussion.

Panel leader for this afternoon is our good friend Elmer Perrine. Elmer has contributed many important papers to this Round Table. Elmer will you please come to the platform and introduce your panelists.

ELMER PERRINE: Thank you Albert. About 30-60 days before these Round Tables convene each year I can generally count on getting several telephone calls from your hard working Executive Committee. These messages mostly get me hooked to appear as a speaker to discuss a subject or two. I am always glad to help.

My panelists are John Surber, John C. Frederick, E. D. Appling and Gus Mautner. Questions and answers by each panelist on the subjects assigned, will be included in the proceedings. Discussions from the floor will be off the record and will not be included in the proceedings.

What Modifications are Necessary to Convert A Typical TVA Type Granulator Plant to an Ammonium Phosphate Plant

John C. Frederick

IN this discussion, an ammonium phosphate plant is a granulation unit capable of producing ammonium phosphate mixed grades; and in this approach, part or all of the P_2O_5 is supplied by phosphoric acid (wet process) and part or all of the nitrogen is supplied by ammonia, either from solutions or as anhydrous ammonia, depending on the grade being produced. As the average nutrient content of mixed fertilizers is increasing yearly, the use of phosphoric acid in the production of granulated fertilizers is expected to increase.

Since the early 1950's the TVA continuous granulator has been widely used by the mixed fertilizer industry and is now found in every part of the United States. These units, when originally placed in the field, were for the most part using nitrogen solutions as the primary source of nitrogen. In addition, super and triple were principally being used as the sources of phosphate, with sulfuric acid being added in order to react with ammonia not tied up by the super and triple. This ammonia-sulfuric acid reaction forms ammonium sulfate; and from this heat of reaction additional heat is supplied which is usually necessary for granulation.

Slide No. 1

6-24-12

Sol. 490 (34-60-0)	—	6 units
46% Triple	—	18¾ units
20% Super	—	5¼ units
66°-Be Sulfuric Acid	—	100 pounds
60% Potash	—	12 units

This slide is an example of a grade where all of the nitrogen is obtained from solution, and super and triple both are being used as the sources of phosphate. Probably many of you recognize a formula similar to this which you have used or are presently using. In actual plant practice, slight over-formulations to compensate for plant food losses are customary; but since this example and the following examples are for comparison purposes, no plant food over-formulations are shown. However,, moisture losses due to heat of reaction and drying have been allowed based on a finished product's having 1% free moisture.

Slide No. 2

6-24-12

Sol. 490 (34-60-0)	—	4 units
Anh. Ammonia	—	2 units
75% Phos. Acid	—	6 units
46% Triple	—	9½ units
20% Super	—	8½ units
60% Potash	—	12 units

Slide No. 2 is an example of a formulation for this same 6-24-12 with four units of nitrogen from solution and two units from anhydrous, the phosphate sources being divided among phosphoric acid, triple, and super, as shown. The phosphoric acid replaces the sulfuric acid used in Slide No. 1 and in addition supplies part of the P_2O_5 required. The triple has been reduced from 18¾ units as shown in the previous slide to 9½ units. The super usage has increased from 5¼ units to 8½ units. The additional heat necessary for granulation is provided by the reaction of the anhydrous ammonia and also the ammonia in the 490 solution with phosphoric acid, resulting in the formation of ammonium phosphate salts. The formula shows two units of anhydrous ammonia and four units of nitrogen solution; however, phosphoric acid may also be used if all six units of nitrogen are from solution.

A TVA type granulator plant would likely require only minor modifications to produce ammonium phosphate grades using formulations such as Slide No. 2.

Slide No. 3

6-24-12

Anh. Ammonia	—	6 units
75% Phos. Acid	—	12¾ units
20% Super	—	11¼ units
60% Potash	—	12 units

Slide No. 3 shows an example in which all of the nitrogen is supplied by anhydrous ammonia, and the triple has been completely eliminated by the introduction of additional phosphoric acid. Also, the super has been increased over the two preceding slides. This No. 3 formula will probably require the addition of a pre-neutralizer or reactor tank for product moisture control and minimum nitrogen losses because of the addition liquid phase introduced by the 12¾ units of phosphoric acid along with the 6 units of anhydrous ammonia. To the pre-neutralizer, usually all of the phosphoric acid and a part of the anhydrous are introduced, with the remaining ammonia being added to the granulator for ammoniation of the super. An 8-32-16 formula, which is also a 1-4-2 ratio as is 6-24-12, would be possible using this approach in a TVA granulator.

A 16-8-8 is next shown to illustrate a grade having a different ratio.

Slide No. 4

16-8-8

Sol. 440 (24-70-0)	—	8 units
21% Amm. Sulf.	—	8 units
46% Triple	—	5¼ units
20% Super	—	2¾ units
66° Be Sulfuric Acid	—	150 pounds
60% Potash	—	8 units

Slide No. 4 shows a non-phosphoric acid formulation where 8 units of the nitrogen are being supplied by solution and the additional 8 units by ammonium sulfate. Sulfuric acid is added in order to supply heat and also to tie up the ammonia as ammonium sulfate above and beyond the ammonia absorbed by triple and super. If phosphoric acid were added to this example, the sulfuric acid and triple usage would be reduced and the super usage increased.

Slide No. 5

16-8-8

Sol. 440 (24-70-0)	—	16 units
75% Phos. Acid	—	1 unit
20% Super	—	7 units
66° Be Sulfuric Acid	—	390 pounds
60% Potash	—	8 units

This 16-8-8 formulation shows phosphoric acid being added, and in this example all nitrogen is from solution. In this approach, a pre-neutralizer would undoubtedly be necessary due to the 16 units of solution and the 390 pounds of sulfuric acid, plus 1 unit of P_2O_5 from phosphoric acid.

There are many other grades where phosphoric acid can be added to produce ammonium phosphate mixed goods. The two grades, 6-24-12 and 16-8-8, are merely examples of this approach. Formulation costs have been purposely omitted due to the variation in material cost in different sections of the county.

Phosphoric acid is corrosive to most of the common materials of construction such as steel and cast iron; and for this reason piping for handling this acid is most often 316ss, rubber-lined steel, or plastic piping such as polyvinylchloride. The phosphoric acid sparger pipe in the TVA granulator is usually 316ss or Hasteloy C. Wet process phosphoric contains small quantities of suspended solids, usually gypsum, and for this reason is extensively metered by magnetic type flow meters, whose accuracy is unaffected by these solids.

The use of phosphoric then for most plants would require suitable piping from the tank car or storage tank to the granulator, a meter, and an additional sparger; or the phosphoric acid could be added through the existing sulfuric acid sparger. In most cases the air compressor used for solution unloading could be utilized for unloading phosphoric acid tank cars.

With high nitrogen grades such as 12-12-12 and 20-10-10 if all of the nitrogen is to be obtained from solution, or if all of the nitrogen is to be obtained from anhydrous ammonia on grades such as 6-24-12, the use of a pre-neutralizer may be necessary. These pre-neutralizers are most often stainless steel tanks of varying size depending on the desired production rate. All of the formulated sulfuric acid and/or phosphoric acid is usually metered to this tank with sufficient nitrogen solution and/or anhydrous ammonia to maintain an exit slurry slightly on the acid side. This slurry overflows to the granulator, where it is added to the potash and solid phosphates along with the additional nitrogen solution or anhydrous ammonia required.

It is hoped that many of you participating in the Round Table who have used phosphoric acid in manufacturing mixed fertilizers will present your thoughts as to the modifications you have found necessary in order to produce these ammonium phosphate type grades and what the economics are when phosphoric acid is used. These modifications would include phosphoric acid handling system, phosphoric acid meters, sparger arrangements in the granulator, production experience such as operating temperatures in the dryer, moisture control of the final product, and any other modifications you have found necessary.

Nitrogen Losses

John Surber

DURING the past two decades we have enjoyed several remarkable changes in the pattern of manufacturing, storage, and shipment of fertilizer. Granulation is perhaps the most significant change that has occurred in the past two decades. We readily recognize the extreme importance of the introduction of the T.V.A. Ammoniator and the friendly exchange of information that is generated by these Round Table conferences. But, with all the manufacturing progress and development, we still experience nitrogen losses.

After many years of real progress in curtailing nitrogen losses, it is still most important and timely that we examine the topic "What Improvements Have Been Made, or can be Made, to T.V.A. Ammoniation equipment to cut down nitrogen losses?"

The study of nitrogen losses has generally been divided into three categories namely, (1) from the ammoniator, (2) from the dryer, and (3) from storage. In that the decomposition in storage occurs under adverse conditions and the remedy is generally known to all, we will confine our remarks to losses from ammoniation and drying, ammoniation being by far the greater of the two evils.

Some of the factors that have been and will continue to be upper most in our analysis of this problem are presented for your consideration.

1. Metering of Liquids.

The replacement of variable area meters with magnetic meters, positive displacement meters and turbine meters for anhydrous ammonia has greatly reduced the losses resulting from inaccuracies in metering. Further improvements on the magnetic meters such as variable range units and more efficient integrators are a step in the right direction to reach our ultimate goal of *A Flow Within Plus or Minus .5% at Any Setting for NH_3 and 2% at Any Chart Setting for Other Liquids.*

It is extremely important that all liquids including water be metered to the process.

2. Dry Material Feeders.

In our opinion a dry feeder should be capable of feeding dry material within a tolerance of one quarter of one percent at all times. This does not mean ½% for each and every ton produced. The dry feeder should weigh a present amount, not act as a proportioning belt that needs continual changing throughout the shift.

3. Size of Ammoniator.

The physical size of an ammoniator is not nearly as important as the effective length over which liquids are evenly distributed. The industry's original 7′ x 7′ ammoniator for 20 tons per hour was wrong and even the present 7′ x 16′ ammoniator for 20 tons per hour requires full length sparger distribution and in some cases double solution spargers are paying dividends.

4. Depth of Bed in Ammoniator.

Many years ago our recommendation was that the depth of bed be ¼ the diameter of the drum, thus 20″ retaining ring for a 7′ diameter unit. This recommendation is still a gool rule however, the retaining ring should not contain doors or divits that effectively lower the bed depth. Ample horsepower should be provided to obtain at least 20″ depth on 7′ unit.

5. Sparger Design and Arrangement

This is probably the most important element in the control of nitrogen losses and there have been and still exist within the industry as many designs as there are people in this room. In general, a sparger should be designed and arranged to distribute the liquids evenly throughout the length of the ammoniator. It is imperative to obtain uniform reaction thereby eliminating "hot spots" or "flash fires." It is equally important to observe care in selecting hole sizes, number of holes, and location of holes. Periodically check and replace any sparging pipe that has plugged or deteriorated in any way.

6. Ammoniation Rates.

With carefully selected materials and excellent operators, we should obtain good results with these rates:

Superphosphate (ROP)
  6 lbs. NH_3 per unit P_2O_5
Triple Super (ROP)
  3.5 to 4 lbs. NH_4 per unit P_2O_5
Triple Super (Coarse)
  0 to 3 lbs. NH_3 per unit P_2O_5
Phosphoric Acid
  7.5 lbs. NH_3 per unit P_2O_5

7. Operator.

The operator must know the capabilities of the equipment in the process. We obtain our best results where the operator understands meters, feeders, spargers and formulation. Take time to train each operator to the best of your ability and continue this training as new developments occur.

8. Pre-Neutralization

The two common methods of pre-neutralization reasonably achieve a controlled reaction of ammonia and acids and mix the resulting slurry with the dry materials. The pre-reactor is a very valuable tool in the fertilizer plant and for our purpose today it materially curtails the loss of nitrogen because of the controlled reaction.

9. Data About Typical Ammoniator Sparger Arrangement

(a) Length of ammoniating section .... 14′
(b) Diameter of ammoniating section .... 7′
(c) Rotating speed of ammoniator .... 11 RPM
(d) Height of retaining ring .... 20″
(e) Product rate .... 30 Tons Per Hour of Product
(f) Screen sizes of product .... −6 mesh (9044) +14 mesh (9069)
(g) H_3PO_4 Sparger
    (1) 1½″ 316 SS pipe
    (2) 10′ 0″ long
    (3) 54—¼″ holes 2″ center to center within 6″ blank at each support and 3″ blank at each end.
(h) NH_3—Solution Sparger
    (1) 1½″ 316 SS pipe
    (2) 11′ 0″ long
    (3) 58—¼″ holes 1¾ center to center with blanks of 5¼″, 11½″, 9¾″ and 4″ at entrance end, 1st support, 2nd support and exit end respectively.
(i) H_2SO_4 Sparger
    (1) 1″ Hastaloy "C" Schd. 40.

(2) 11′ 0″ long
(3) 104—⅛″ holes 1″ center to center with blanks of 6″, 10″ 12″ and 3″ at entrance end, 1st support, 2nd support and exit end respectively.

(j) Sparger in bed 10″ from shell at approximately 4:00 o'clock with 6″ spacing between NH_3 solution sparger and H_2SO_4 sparger. H_3PO_4 sparger on top of bed 6″ from shell at 1:30 o'clock.

10 Drying

The drying temperature may vary slightly with the formulation but in general the product temperature should not exceed 180°F. This is considered to be the maximum safe temperature for drying DAP. The burner, combustion chamber, and/or plenum on single shell driers should be so designed to keep flame from coming in contact with material.

I would like to present some thought to equipment manufacturers for their consideration:

1. Ammoniator should be driven with adequate horsepower to permit retaining ring to be at least 25% of diameter of drum.
2. Scraper bats to be eliminated. Oscillating scraper is the best idea to date but is not good enough. Spargers should always be at a constant distance from the shell, therefore we don't want build-up on shell. Rubber lining rather makeshift.
3. Retaining ring on ammoniator should be designed to vary bed depth very easily.
4. Discharge doors need vast improvement.
5. Keep exit end of ammoniator frame reasonably open in order for operator to observe bed action.
6. Meters should be provided for .5% accuracy for NH_3 and 1% accuracy for other liquids at any chart setting. Cheap and inaccurate metering is false economy.
7. Need for ammoniators 7′ x 20′ or 7′ x 24′
8. Need for exploration into reactors superior to the present T.V.A. reactors.

Safe Methods of Reclaiming Fertilizer Piles and Operating Payloaders

E. D. Appling

This is a most common problem and I'm sure all here are very familiar and concerned about both phases of the general topic. Actually, reclaiming piles or operating payloaders safely is each a very major problem in any fertilizer plant. These are both troublesome problems that have been associated with the fertilizer industry for a great number of years. Usually, when an accident occurs in either of these hazardous conditions, it is quite severe. This accentuates the need to implement the safe methods of performing these operations that are presently known, and also to continue to study and develop ideas and procedures which will improve on what is considered the safest way now.

I am going to assume for this discussion that we consider the safe operation of payloaders specifically as it concerns removing fertilizer from a storage pile, rather than the broad complete range of activities these units actually perform.

The most generalization I think we should do when considering this subject is to say that to some degree it is a common problem in all fertilizer plants.

Generalization on solutions to these problems is quite dangerous. It probably will suffice to say that to some degree any logical approach will work in any plant. The two phases of the topic separately or combined should be considered on an individual plant basis. The variations in personnel, physical facilities, types of material handled and many other conditions are sufficiently different as to cause effective safety procedures to be different. By this I do not mean the basic idea or principle, but that any good concept of safe operation can be modified and adapted to a particular situation.

The major problem encountered in reclaiming a pile of fertilizer is in the formation of an overhang. This is real serious because overhangs are potentially deadly. Therefore, a method of reducing piles without forming overhangs, vertical faces or slide areas would be of great value in correcting this hazardous condition. Where applicable, overhang cranes or clamshell or power shovels with extended booms and now a reclaiming machine are effective ways of reducing piles. These are effective because they are able to perform the desired function without personnel having to work in potentially dangerous areas.

Possibly of more general interest, is the larger front-end loader, this being more adaptable to more fertilizer operations than the larger, more-expensive, less-mobile units mentioned earlier. This type front-end loader has a long reach bucket and rather high vertical position of the operator, plus heavy total weight of unit means the operator can work rather large piles without being in extreme danger. I realize that even a machine of this size is too large to operate in most of the older fertilizer plants.

This, then, brings us back to the problem as it now exists, which is having large fertilizer piles to reclaim and rather small front-end loaders with which to work. The small front-end loader does not have the capability of completely reclaiming a large fertilizer pile safely, regardless of how efficient the operator may be. The primary method of reducing a fertilizer pile to the safe operating potential of a payloader presently is by blasting down with dynamite. The use of dynamite is a two-prong safety hazard. First, the actual physical handling of dynamite itself, especially without trained personnel, is dangerous. Secondly, the operation of drilling, placing, tamping and exploding the dynamite necessarily means that personnel work in areas of extreme danger of fertilizer falling on them.

In connection with using dynamite quite a few novel innovations all directed toward increased safety for the blasting personnel have appeared in recent years. These consist of such things as (1) using an observer, (2) safety harness, and (3) cages on fork lifts to raise a person above the level of accumulation should a slide occur.

As a supplement to blasting, various types of probes attached to front-end loaders have been used experimentally. Also, hand poles used to remove overhanging materials with cracks behind them. Lumps caught behind columns, braces, etc. can be dislodged with hand poles.

Various types of haul down machines of the ditch digging type or back hoe design using a telescoping boom have been and are being used to bring high piles down so the payloader can handle the material.

A safety conscious, well-trained operator is an absolute necessity in obtaining safe operation of payloaders. The operator who meets the above qualifications should make the final determination as to the safety of payloader operation within a particular area as it relates to removing material from a fertilizer pile.

Various types of cages, protective bars and tops have been installed on payloaders for the operators protection. I'm sure we can get arguments pro and con as to the advisability of using these types of devices.

Personally, I'm one of the cons even though the picture circulating among you is strong evidence that protective bars are indeed a safety factor.

As to the operation of payloaders in general, a device I would like to see is a master control switch of some type built into the seat. This would function in such a way that the machine would be completely inoperative unless the driver was sitting in the seat in the proper operating position.

Ammoniation Fires

Gus Mautner

A. Philadelphia 16-8-8

1. Conditions at time of blast. Magician 3rd class bit Flame Bit
2. Condition allow combustible NH_3 and air mixture (16% NH_3).
3. Ignition of organic substance, paper, wood, etc. from ammonium nitrate temperatures of 270°F.
4. Formulation 8 units Nitrana 7, 8 units ammonium sulfate Formula

288	Super
221	Triple
200	66° Sul. Acid
355	450 (25.3-69.2-0) Solution
763	21 Sul. Ammo.
263	Potash KCL
2,090	

5. Free Ammonia 89.8#, Phosphate (NH_3) + Sulfuric Acid 101.0#, % Ammoniation 88.9%.
6. Effect of phos. acid 100# PA reduces S/A reg. to 150#

435	Super
44	Triple
100	Phos. Acid.
150	Sul. Acid
355	450 (25.3-69.2-0) Solution
763	Ammo. Sulfate
263	KCL
2,110	

7. Free Ammonia 89.8#, phosphates plus sulfuric acid 102.0# ammonia % ammoniation 88.0%.

B. Causes of Fires

1. Interrupted dry material feed
2. No recycle
3. Damaged spargers (hole enlargement
4. Improper formulation
5. % ammoniation over 100
   a. Insufficient 66° sulfuric acid to react with excess free ammonia.
6. Dry bed in ammoniator
7. Overgranulation
8. Ammoniator shell buildups prevent free passage of material under spargers.
9. Lumps on spargers impedes flow through ammoniator
10. High temperatures during granulation melts ammonium nitrate and fertilizer appears wet.

C. Cure

1. Formulate with 100 - 125# phosphoric acid.
   a. Reduces sulfuric acid needed for NH_3 neutralization
   b. Ammonium phosphates fire retardants and may prevent ignition of organic matter
2. Use 60° Be sulfuric acid.

PANEL LEADER PERRINE: Thank you Panelists for an excellent job of discussing your respective subjects. My compliments to each of you and to our audience for joining from the floor with pertinent questions and many helpful answers. Time is passing fast, now 4:45 P. M. Again, may I tell you, it was my pleasure to be part of this most interesting session. I shall now turn the meeting back to Moderator Spillman.

MODERATOR SPILLMAN: Thanks Elmer and your Panelists for a real good performance.

Friday Morning Session, Nov. 13, 1964

Round Table reconvened at nine o'clock.

Dr. Vincent Sauchelli, presiding

CHAIRMAN SAUCHELLI: Good morning, ladies and gentlemen, it is good to see this number of folks at this early hour on the third day.

We are going to start our program this morning in accordance with the printed program, with a brief business meeting. Since our organization is such an informal one, we would like to keep it that way. We have no minutes or anything of that sort to read and have approved, but we do have a statement from our Secretary-Treasurer regarding our financial status.

Secretary-Treasurer Report

HOUSDEN L. MARSHALL

Members of The Round Table. The Order existed fairly close to the equilibrium point all this last year although it did go short a little bit.

Financial Status:

Carryover, Oct. 31, 1963		$ 576.42
Income Nov. 1, 1963 to Oct. 31, 1964		3,375.93
Membership list—Attendance 1963		187,50
Sale back proceedings		939.26
Total Cash Available		$5,079.11
Disbursements.		
1963 Meeting and Incidental Costs	$ 403.28	
Membership Lists Costs	226.57	
Printing Proceedings, Transcripts, etc.	3,202.66	
Office Expenses, Stationery, Postage, Secretarial	984.26	
Total Cash Expense	$4,816.77	
Balance on hand Nov. 1, 1964		$ 262.34

I have eight kits of proceedings from 1958 Thru 1963. First come first served at $20 per kit. Thank you.

CHAIRMAN SAUCHELLI: Thank you, Dr. Marshall. I want to take this opportunity to express my sincere appreciation for the hearty cooperation given me by the members of the Executive Committee. It is a hard-working group and particularly Al Spillman and Dr. Marshall who have taken most of the burden.

Dr. Marshall, of course, you just can't keep him down. He is constantly wanting to be doing things and he keeps pushing the rest of us. Sometimes we just have to hold him down. He is full of pep and particularly when it comes to money matters, why, there is no holding him at all. He has done and excellent job.

These proceedings are a valuable by-product of our sessions here. They are becoming better known and the demand for them is increasing. Whoever receives a copy of the proceedings knows that he has some very worthwhile information concerning our fertilizer technology and so on.

The dates for next year's meeting are November 10, 11, 12, 1965, and we have scheduled the meeting for this hotel, The Mayflower.

It is a very difficult thing to try to get reservations at different hotels for a meeting of this kind, and Washington particularly during November is a very busy place as you can see the number of conventions here now at the Mayflower. Also daily, there are two or three groups meeting here. We are committed here for next year.. For how many more years Mr. Secretary?

DR. MARSHELL: All of these are provisional reservations. We are not absolutely bound to them and we did vote on the 1965 meeting last year. We have our name in the book at this hotel until 1973.

I happened to be in here one day when they had some new sheets floating around and I thought we would fix it up and we took it until 1973, but we do have to go ahead, I can tell you now after seeing what they have. It is really filled up.

I have made every effort and I am keeping my eye on that book to get the week before or after election. That is the way we have worked it so far. It seems to be the most popular week.

CHAIRMAN SAUCHELLI: Let me have a show of hands of all those who would like to continue to come to Washington for this meeting? (A showing of numerous hands.)

Thank you. The question comes up from time to time as to the desirability of holding the meeting elsewhere in the country. I know every year we have taken a census here of the desires of the membership and invariably the vote is for holding the meeting here in Washington. People like to come to Washington. It is convenient. I see Frank Nielsson has a different viewpoint, but let's hear what Frank has to say.

DR. NIELSSON: I think we ought to give some consideration to holding the meeting at some place other than Washington. Every year we come to Washington but I feel that if we would hold our meeting in some other city, perhaps out West, we would get more of those fertilizer people interested from that area than we now have. We have to come to Washington for 1965 but we don't have to come to Washington every year. I think we ought to think about going to Denver, St. Paul, Miami Beach and other cities. I think if we do this and move about from city to city, we will get more and more people to come into our membership.

CHAIRMAN SAUCHELLI: We have given thought to this question.

MR. NIELSSON Yes.

DR. MARSHALL: As I said, we are not totally committed yet, but if we are going to some other city and to get a hotel to accommodate this group, you really have to get in way ahead of time. If you want this, we can do it. I think you would be surprised but if you checked you would find that the date we would start with would be 1968 or '69.

CHAIRMAN SAUCHELLI: I have participated in two regional meeting, one in the Southeast, held in Savannah and one in Dallas for the Southwest. They were huge successes. I know that the regional meeting can be a success if you get local support and local participation in organizing it.

We take for granted how the organization goes on here but your Executive Committee does the work. There are just four of us that live and meet in Baltimore. It is convenient to get together and we are willing to give the time. We have not been able to get this same kind of cooperation from local groups in other parts of the country.

We haven't always met in Washington. We've had a meeting in Chicago, we had a meeting in New York, we had a meeting in Atlantic City. When we started, we used to meet at the same time the American Chemical Society met and the competition for space and time was such that we decided it was better to hold our own independent meeting. So that's the status. We will give your suggestion some thought, Frank.

MR. WILLIAM HARWOOD: I would like to ask why is it necessary to meet here?

DR. MARSHALL: The distance story, Bill. You check the people who have registered.

MR. HARWOOD: I think if we met in some other place, you might find out that we would attract bigger crowds. As far as reservations, I feel confident two years from now we could get wonderful reservations in Miami Beach at $13 a day.

DR. MARSHALL: I've got one better than that. We can go to Puerto Rico.

MR. HARWOOD: I move we go to Puerto Rico.

(Laughter.)

CHAIRMAN SAUCHELLI: I think that the majority wants to come to Washington and we will consider the others. We have been active on it. We haven't just forgotten about other locations but so far, the best proposition is here in Washington.

We have a splendid schedule for this morning's session. We are through with the business meeting unless you have something very special to add. This meeting is your meeting. If it is successful, it is because of your participation in it and if it is going to be successful in the future, it can only be successful by your wholehearted support by giving us questions and problems from which we can select our agenda. Please, there is a cigar box on the registration desk waiting for suggestions. We haven't had many so far. We hope before the meeting is over that we will get suggestions from you or you may write to any one of us on the Committee your suggestions for our next 1965 meeting.

Phosphoric Acid; it is one of the most significant developments in recent years in our fertilizer industry. I am sure all of you appreciate the impact of phosphoric acid and its derivatives in our technology. It has been an amazing development in the last five years and the literature on phosphoric acid and its derivatives is growing rapidly.

Some of us had the privilege of attending the demonstration at Wilson Dam last month, organized by the research staff at the Tennessee Valley Authority Research Center. We saw there something of the development in phosphoric acid and its derivatives that gave us an insight into some of the potentials for future developments. We decided, because of its importance, to hold a symposium on phosphoric acid. When I thought of the subject, I immediately associated the idea in my mind with Alvin Phillips of T.V.A. staff, who has done so much in research with Phosphoric Acid.

It is really a privilege to have the cooperation of Alvin Phillips. He has always been generous everytime we have called on him, as is true of the other members of the T.V.A. staff. Always a hearty response from them. It is a pleasure for me to acknowledge that cooperation with our Round Table and to appreciate what they are doing.

Alvin Phillips has organized this symposium Panel and I am going to turn the meeting over to him. He will introduce his Panel. I know they have something very much worthwhile to give us.

Remarks On Phosphoric Acid

Alvin B. Phillips

Panel Leader

Thank you very much Dr. Sauchelli. Ladies and Gentlemen. This Panel was organized for the purpose of discussing the production and some of the uses of phosphoric acid.

Of course this is a very broad subject, however, we are going to try to cover it this morning despite the fact this is the last day of the meeting and it is also Friday, the 13th.

I could start out by boring you with a lot of statistics on the phenomenal growth of phosphoric acid production in the last decade, however, all of you know about that. In fact, a lot of you here in this room are responsible for it. Besides, any figures that I might have gotten together last week probably wouldn't be very accurate this week. Instead I am going to introduce the first speaker.

We are very fortunate to have leading off our panel a man who has been associated with the production of phosphoric acid for many years. He has authored many articles and he has even written chapters of books on the subject of phosphate fertilizers.

He is Director of Fertilizer Projects of Dorr Oliver Incorporated, Stamford, Conn. Most of you know him. It is a pleasure to introduce to you Mr. William (Bill) C. Weber.

Phosphoric Acid Production Technology

William C. Weber

Introduction

THIS introduction to our panel discussion will deal almost exclusively with the production of wet process ortho phosphoric acid by the sulfuric acid acidulation of phosphate rock.

Phosphoric acid can be produced by two principal methods:

a) Thermal decomposition of phosphate rock with silica and coke in an electric furnace or in a modified blast furnace.

b) Decomposition of phosphate rock with mineral acids.

The thermal process will not be discussed because, while this is a very important industry and the production of thermal phosphoric acid is very large, most of such phosphoric acid is used for the production of phosphate chemicals rather than fertilizers.

In addition to sulfuric acid, phosphoric acid can also be produced with hydrochloric acid or nitric acid. The former is not economical unless a large quantity of waste or byproduct acid is available for which there is no better use. Furthermore, it does not usually produce phosphoric acid as such since the reaction products are soluble and the P_2O_5 is usually precipitated in the form of feed or fertilizer grade dicalcium phosphate. There are, however, processes (IMI and Dow) for producing phosphoric acid by hydrochloric acid acidulation using solvent extraction, but these are not yet widely used, nor do they seem to have broad application.

The nitric acid acidulation of phosphate rock is theoretically very attractive economically because the nitrate radical can end up as a constituent of the ultimate fertilizer. The various so-called "nitrophos processes" do not produce phosphoric acid as such but, except in one or two instances, convert the P_2O_5 to dicalcium phosphate. This is a complicated subject but one which I believe has considerable economic potential and should be given more consideration in this country. Nitric acid acidulation is widely used in Europe. I would suggest that it be the subject of a panel discussion at a future Round Table.

Phosphoric acid production is becoming a large and important segment of our fertilizer industry. The capacity of wet process phosphoric acid plants in North America has expanded from approximately 3800 tons of P_2O_5 per day in 1960 to 6500 tons in 1962 and to over 10,000 tons in 1964, based on plants now under construction. There are about forty phosphoric acid plants in the U. S. A. and Canada.

Processes

Methods for producing wet process phosphoric acid divide themselves into two groups depending on whether the acid produced before evaporation has a concentration of 30-32% or upwards of 40% P_2O_5.

1. *Strong Acid Processes*

a) *The TVA Foam Process*

This has been under development by TVA for some time and was demonstrated on a pilot scale at the recent TVA Demonstration Meeting. It is not yet in commercial production.

The novelty lies in the distribution of the sulfuric acid on a layer of foam maintained in the reactor. A stable semihydrate is produced with good filtering properties which can be separated and washed with conventional filtration equipment without further hydration of the calcium sulfate. A 40% P_2O_5 acid is obtained from the filter. Recoveries are still not sufficiently good to be competitive with conventional processes. The washing losses in particular are rather high, but TVA engineers feel that this can be improved with larger and better filter equipment.

TVA will undoubtedly have to demonstrate this process on a larger scale before it will be ready for commercial exploitation. In particular there is some question as to the scale-up of the reactor and its practicability in very large sizes.

b) *The TVA Fuming Sulfuric Acid Process*

This is a process originally conceived, patented and partially developed by the Davison Chemical Company and known at that time as the "clinker" process. TVA undertook its further development and has considerably improved the operation. It was demonstrated on a small pilot scale at the recent TVA Demonstration Meeting. It will have to be demonstrated by TVA on a larger scale before it will be ready for commercial exploitation as there are several unsolved problems and questions as to the feasibility of extrapolating the present equipment to commercial size.

This process produces a 50-54% acid without using any filter or evaporator. Unground phosphate flotation concentrates, together with fuming sulfuric acid (105% are introduced into a pug mill and mixed for about half a minute, after which the mix is "denned" in a rotating drum for about forty minutes to granulate the mixture of anhydrous phosphoric acid and anhydrous calcium sulfate. 85% of the fluorine is evolved during the pugging and denning operation as well as a significant amount of SO_3. By addition of silica, the fluorine evolution can be increased sufficiently to produce a low fluorine feed grade acid.

The granules, which are between about 10 mesh and ½″, are then leached in a rotary drum extractor. This drum is set on a slope and provided with a continuous helical flight. The granules are fed in at the lower end and the leach water at the upper end. The strong acid overflows the feed end, and the extracted granules are discharged at the opposite end.

This process has considerable promise, but there are still several difficult problems to be solved. To be economical, the SO_3 and fluorine evolved during the early stages must be separately recovered in concentrated form. The acid carries considerable calcium sulfate in suspension, which would render the acid unattracive for many uses, and the acid, because of its high concentration and viscosity, is probably difficult to clarify by conventional means. The water soluble losses with the calcium sulfate are still too high but can probably be improved by further study or, alternatively, it may be necessary to reduce the produced acid strength.

The really big problem is whether a rotary extractor is the best arrangement for leaching the granules and whether it can be expanded to handle the large capacities which are now commonplace in the phosphoric acid industry.

c) *The Landskrona Process*

A company by the name of Kemiska Patenter, associated with the Swedish Superphosphate Company, developed a process some years ago for producing 40% P_2O_5 based on producing a stable anhydrite. This was subsequently changed over to a stable hemi-hydrate process and has been used by the Swedish company in their plant in Landskrona, Sweden. A small plant installed in Italy some years ago has, we understand, been abandoned.

d) It is possible to produce stronger acid, 38-40% P_2O_5, in a dihydrate system; but work done by my Company indicated that it would be necessary to go to two-stage filtration and we concluded that this would be uneconomical. St. Gobain has announced and patented a dihydrate process for producing 35% P_2O_5 acid and possibly up to 45%, but we do not know of any commercial installation.

2. *Normal Acid Strength Processes*

The principal variation in processes for producing 30-32% P_2O_5 phosphoric acid lies in the type of calcium sulfate produced. A great deal of work has been done over the years by many investigators on methods of producing the calcium sulfate in the form of stable anhydrite or hemi-hydrate or, alternatively, of producing hemi-hydrate initially and then recrystallizing this to dihydrate (gypsum).

a) *Nissan Process*

A process has been developed by the Nissan Chemical Company and several small plants are operating in Japan. Three large plants are being constructed in Australia. This process is based on initially producing, in a strong acid at high temperature, a relatively unstable hemi-hydrate and subsequently diluting and cooling to effect hydration to the dihydrate which is then filtered and washed. The process produces a 30% P_2O_5 acid. The advantages claimed are higher extraction (lower insoluble mainly citrate soluble losses) and an excellent gypsum well suited for use in making gypsum plasters and wallboard. The plants are somewhat more complicated and more expensive in first cost than the conventional dihydrate processes. The rock must be ground finer.

b) *Nippon Kokan Process*

This is similar to the Nissan process but uses a synthetically produced seed additive for promoting the hydration.

c) *Conventional Process*

Most of the phosphoric acid plants in the world and most of those being constructed at the present time produce a dihydrate of calcium sulfate directly in the reaction. This is then filtered off and washed, and if a stronger acid is required, the acid is concentrated by vacuum evaporation.

My Company has for about thirty-five years studied various variations on the conventional process, primarily with the objective of producing stronger acid directly from the filter and involving the production of stable hemi-hydrate or anhydrite, or, alternatively, unstable hemi-hydrate and subsequently recrystallizing this to dihydrate. Up to the present time we have not been able to advocate these approaches as being more economical. There is an additional incentive for a so-called "recrystallization process' in that it might be applicable to some phosphate rocks which are difficult to treat in conventional plants. We have found that certain chemical additives often make such rocks amenable to normal treatment.

Equipment

Over the years the conventional dihydrate process has by no means stood still. The biggest advances have been in the development of better and bigger equipment and in the construction of larger and larger plants and the refinement of designs, resulting in considerably reduced first and production costs. The flowsheets have been simplified and stream-lined and the plants are now well instrumented.

1. *Grinding*

In this country there has been a steady drift to air-swept ball mills rather than the older roller mills. This trend has been largely due to the increased size of plants. When the capacity exceeds the largest size of roller mill, it is more economical to use one ball mill than two of the smaller roller mills. Power is about the same and the maintenance is slightly lower on ball mills; but the determining factor is usually first cost. The breakpoint is somewhere between 20 and 30 tons of rock per hour (150 to 250 tons of P_2O_5 per day) The ball mill can be counted on for 24-hour, 7-day per week operation; whereas roller mill installations should be based on 20-22 hours per day.

2. *Ground Rock Handling*

The preferred arrangement is for the grinding installation to be considered an intergral part of the phosphoric acid plant since, if they are put together, labor can be saved and the ground rock can be handled with airslides and elevators. If the grinding plant is at some distance, pneumatic conveying must be used which is more expensive and has some other disadvantages.

3. *Raw Material Feeding*

Gravimetric feeders for the ground phosphate rock are essential. The trend has been to use automatic batch scales, but in the very large plants now being built these are being replaced by gravimetric belt feeders.

The sulfuric acid is controlled by either magnetic flowmeters or orifice-type electronically-controlled instruments.

The wash water is metered carefully and the weak filter wash solutions, which are returned to the reaction, should preferably be accurately controlled. This is usually done with magnetic flowmeters.

4. *Reaction or Digestion*

The reaction system is the heart of a phosphoric acid plant as it is here that the P_2O_5 is extracted and the gypsum is produced, which determines the performance of the filter.

The older system, where a series of agitated tanks in cascade was used, has been pretty well abandoned. The modern practice is to do the entire operation in one large tank.

The first so-called "single tank reactor" was that offered by the St. Gobain Company of France (an invention of Union Chimique Belge). This was, in effect, one large agitated tank with multiple agitator mechanisms. The slurry was pumped directly to the filter.

The Dorr-Oliver Companies are offering a novel and patented "Single Tank Reactor" with two compartments, an outer annulus equipped with several agitator mechanisms designed to provide a controllable swirl or recirculation of the slurry and an inner agitated core for stabilization of the reacted slurry before it goes to the filter via a surge tank or so-called "filter feed tank."

Prayon, who initially offered a reaction system consisting of two tanks, each divided into four compartments, are now offering in most cases and in all large plants a single rectangular compartmented tank. This is still, in effect, a multitank system as the slurry progresses from one compartment to another over and under baffles.

Most other systems use variations of these or the older multitank arrangement.

In all cases, slurry recirculation is provided whereby reacted slurry is recycled or pumped back to the first reaction tank or compartment to control supersaturation which is necessary for good gypsum control. In the Prayon system, pumps are used for this purpose; whereas in the Dorr-Oliver Single Tank Reactor the recycling or swirl is obtained by the placement of the agitators in the annular reaction compartment, and much greater recirculation ratios can be used. In the St. Gobain reactor, no outside recycling is necessary as the complete reaction is effected in one large agitated tank.

These reactors are now being built in very large unit sizes. In one Prayon plant in Florida each unit or compartmented tank is handling as much as 400 to 500 tons of P_2O_5 per day. The largest Dorr-Oliver Reactor in operation is 200 long tons per day, but units are being built with capacities of 450 and 600 tons per day. Capacities of to 700-800 tons per day do not seem unrealistic.

5. *Cooling and Acid Dilution*

Most phosphoric acid plants use a concentrated contact process sulfuric acid (93-98%). It is possible to use weaker acids but this reduces the strength of acid obtainable and/or the washing efficiency obtainable on the filter. The heat of dilution of the sulfuric acid and the exothermic heat of reaction make it necessary to provide cooling of the reaction slurry.

This cooling is effected by evaporation, either by blowing air across the surface of the slurry or introducing air into the slurry, or by subjecting the slurry to flash or vacuum cooling. In either case, the amount of water evaporated is considerable and this adds to the amount of water than can be introduced on the filter for any given strength of produced acid.

In the St. Gobain system, air is drawn in around the periphery of the tank and sweeps across the surface and is exhausted from the center. This method uses large quantities of air and is probably not efficient enough for large installations. It is simple.

With the Dorr-Oliver Single Tank Reactor, cooling can be effected either by injecting air in a novel impingement arrangement, (patented) or by circulating the slurry through a vacuum cooler. Prayon combine their slurry recirculation with the cooling and pump the slurry from a later stage of the reaction through a vacuum cooler, returning part of it to the first stage reaction and part of it to the latter or stabilization stages.

It is possible to reduce the cooling load by diluting the sulfuric acid and cooling the diluted acid. Prayon use this system. Dorr-Oliver does not generally use it because they feel that it is inadvisable to waste the heat of dilution and thereby reduce the amount of water than can be used on the filter. They prefer to dilute the sulfuric acid with returned filter wash solutions and reverse the possibility of reducing the cooling load by acid dilution (at a slight loss in recovery) for use when it becomes desirable to increase the throughput of an existing plant.

Regardless of what cooling system is provided, a fume exhaust must be provided from the reactor and these fumes must be scrubbed to avoid atmospheric pollution from evolved fluorine. Where air cooling is used, the volume of gases to be exhausted and scrubbed is greater, and this may be the determining factor in the choice of air cooling or vacuum cooling, since the regulations on fluorine emission, especially in Florida, are becoming very stringent. Various types of scrubbers have been used, including impingement scrubbers of the Doyle type, cylonic scrubbers and spray scrubbers with packed sections, depending mainly on the efficiency required.

6. *Foam Control*

Most phosphate rocks contain varying percentages of organic material and CO_2 in the form of calcium carbonate. These combine to create a foaming problem which varies considerably with the type and grade of rock. In Florida, where low grade rocks are almost universally used for phosphoric acid production, the use of an antifoam reagent is almost obligatory. Some of the reagents which have been found effective are:

a. Hodag, a mixture of Tall oil, fatty acids and wetting agents
b. Tall oil and fatty acids
c. Oleic acid
d. Silicones
e. Turkey red oil
f. Sulfonated oleic acid

Normal average usage is about 5.0 lbs. per ton of P_2O_5. With poor rocks, up to 10.0 lbs. may be necessary.

7. *Filtration*

Next to the evolution of efficient reaction systems, the most important development in the large-scale production of phosphoric acid has been in the design of the filtration equipment.

Over the years a variety of filters has been used. These have been band filters (Landskrona, Lurgi or Mercier), Oliver Horizontal Filters, in-line pan filters such as the Dorr-Oliver TP or Giorgini, and the rotary tilting pan filters of the Prayon and Eimco type. The TP and Horizontal Filters are still economically attractive for small plants, particularly where high acid strengths are not essential; but the tilting pan filters are practically standard equipment now for all large plants.

The Eimco and Prayon filters are very expensive, but they are ideally suited for phosphoric acid work; they are well-developed and, of most importance, they can be built in very large sizes. The largest single unit now offered has 950 sq. ft. of active area which, with a good reaction system, can handle 450 to 500 tons of P_2O_5 per day on one unit.

I would not attempt to pass on the relative merits of the two makes as they are both very good, well-developed and offered by reliable companies. There are slight differences but they are not significant, and we generally evaluate them on the relative installed cost.

8. *Pumps*

Phosphoric acid plants involve a number of pumping operations, and there has been a considerable development in this area and the choice of the proper pump for the particular service is important.

For filtrate service, the choice has been between horizontal Wilfley glandless pumps and vertical submerged pumps. It is very difficult to maintain packing in phosphoric acid service and the use of water seals is objectionable because of the introduction of additional water. For slurry service, vertical pumps are also used, but it is difficult to improve on the Type K Wilfley slurry pump. Axial flow or elbow pumps are used for evaporator circulation. Pumps are usually of all alloy construction, but rubber-lined casings for large slurry pumps have been found entirely satisfactory.

9. *Evaporators*

At one time direct heat and submerged combustion evaporators were used for phosphoric acid, but with the increasing emphasis on avoidance of atmospheric pollution and because of other problems, they have been generally abandoned. The vacuum evaporator is now almost standard equipment.

These consist of a large flash chamber with an outside steam-heated tubular exchanger. Originally circulation through the exchanger was by natural or thermosyphon means. It was found that, by increasing the recirculation and reducing the temperature rise, scaling—one of the most serious problems—could be reduced and, therefore, forced circulation evaporators using axial flow pumps are now universal.

One installation is now being constructed which will use a novel method of discharging the supersaturation in a maintained crystal bed which, it is hoped, may reduce or even eliminate tube scaling.

These forced circulation units have been built with up to 12 ft. 6 in. diameter, rubber-lined flash chambers and up to approximately 2600 sq. ft. (inside) Karbate tube exchangers. The only limitation seems to be the maximum diameter flash chamber that can be shipped conveniently. One unit can handle up to 150 tons of water evaporation or 100 tons of P_2O_5 per 24-hour day. The units must be boiled out on a regular schedule, and they are usually figured for 85% on-stream time.

Where the capacity is in excess of that obtainable from a single unit, it is customary to use 2, 3 or even 4 units in series. This is preferable to operating them in parallel as it reduces the amount of heat exchange surface and condenser cooling water requirement because the early stages are operating at low acid concentration.

Swift & Company have developed a process for recovering the fluorine evolved during the evaporation as a 15-23% hydrofluosilicic acid with a recovery of 90-95% of the evolved fluorine. This was described in a paper presented at the ISMA Helsinki Meeting in September 1963 by the writer and Mr. Dwight Sanders of Swift & Company.

10. *Instrumentation*

Phosphoric acid plants are now being fairly heavily automated. This permits better control and more efficient operation. Phosphoric acid plants require about two skilled operators per shift, although this number may be increased in large plants. We know of one plant being operated by one man per shift.

Magnetic flowmeters are generally used for controlling phosphoric acid and phosphoric acid slurry flows and also for sulfuric acid.

The evaporators are completely instrumented and automatically operated except when they have to be taken out of service for boil-out. With vacuum control, the temperature of the evaporated acid is a good measure of the strength of evaporated acid and it is customary to use this temperature to control the weak phosphoric acid input to the evaporation circuit.

Several companies are working on the development of an automatic sulfate analyzer for the reaction slurry, hoping to use this to automatically control the proportioning of the phosphate rock and sulfuric acid. This is the most important control point in a phosphoric acid system and such a controller, if thoroughly practical, could make a valuable contribution. At least two companies allegedly are using such controllers, but they are not generally available or been adopted.

Other possibilities being looked at or reasonably well developed are automatic control of reactor solids concentration and automatic control of defoamer addition.

11. *Acid Clarification*

The phosphoric acid coming from the filters contains a significant amount of fine and suspended gypsum. It is usual to provide an in-process storage for this acid, arranged so that the acid is clarified and the gypsum is returned to the process.

During evaporation some of the impurities contained in the phosphoric acid are precipitated and these must be removed if the acid is to be used for the production of high analysis fertilizers or if it is to be shipped. The present practice is to clarify it as much as possible, using a flocculant such as Separan, to then subject it to sheer, cooling and aging and to finally clarify the acid in centrifuges before shipment.

12. *Materials of Construction*

Wet process phosphoric acid is very aggressively corrosive, not only because of its phosphoric acid content but because it also contains an excess of sulfuric acid and hydrofluosilicic and hydrofluoric acids. Therefore, the choice of proper materials and the development of better materials has been one of the significant improvements in phosphoric acid technology.

Rubber linings are used extensively, but when handling slurries, they are preferably protected with brick and, under most conditions, this has to be a carbon brick as ordinary silicious acid-proof bricks are attacked by the fluorine. Brick-lined concrete tanks can be used. The evaporator heat exchangers are now almost universally made of Karbate tubes cemented into Karbate tube sheets. Nionel tubes can be used for the second or third stages, or single stage when concentrating to 54% P_2O_5. The flash chamber and acid and vapor piping are rubber lined. The filters, pumps and agitators are made of stainless steel. 316 or 317 ELC are quite commonly used, or alloy 20. With some phosphate rocks deficient in silica, higher alloys must be used or can be used profitably to minimize corrosion and maintenance.

Piping is of rubber-lined steel or plastic. Epoxy-protected fiberglass, Saran lined, and PVC have been used. Rubber hoses are also used generally.

13. *Dust and Fume Control*

In a well-designed plant, all tanks are covered and vented to the reactor scrubbing system. The discharge and feed sections of the filter are hooded and similarly vented.

All dust is eliminated by proper venting and dust collection and the rock dust feeding system is best placed in a separate dust-tight room.

MODERATOR PHILLIPS: Thank you very much, Mr. Weber, for that very splendid review of phosphoric acid production technology. I know that many of you have questions and perhaps remarks you would like to make. We would appreciate it if you would remember them. We will have a question and answer period after the other speakers have presented their papers.

Our next subject is the use of orthophosphoric acid in granulation. I had asked Bill Jones, of Northwest Cooperative Mills to, take it because I knew that his company had pioneered in this field. When he found that he couldn't come he sent a substitute. I am glad he did because this gives us a chance to meet this gentleman for the first time on the Round Table. He is Manager of Northwest Cooperative Mills, Winona, Minnesota plant. I am very glad to introduce Mr. Rocco Russo

The Use of Phosphoric Acid In Granular Mixed Fertilizers

Rocco L. Russo

DURING the past few years the fertilizer industry has been forced to make many changes. Our customers want a better product. They want a product that will spread more evenly. They want a product that will not cake in their spreaders. They need a product, more uniform in shape and size, that will eliminate variations in fertilizer distribution. By using phosphoric acid we can give them the quality to meet these new demands.

Phosphoric acid in the manufacturing process yields many benefits to the finished product. One of the most important is more selective screening—because of the great amount of liquid phase and the heat of reaction that we have with the phosphoric acid as opposed to a dry form of P_2O_5, we can screen out particles which are minus 20 mesh. The fines can then be recycled and made into larger granules. With phosphoric acid we produce harder granules that will not break up into fines or dust. This assures us that the quality of the product in the bag will be as good when it is opened as it was when the fertilizer was originally produced. The fertilizer is less hygroscopic, with less tendency to cake because we have cut down on the nitrogen solutions eliminating conditioning problems. Most of the time we can eliminate the solutions completely. This alleviates the problem of obnoxious gases. Inside the plant these gases are a health hazard to the workmen, outside the plant our public relations suffer when the wind carries these gases to nearby neighborhoods. With phosphoric acid we can produce higher analyses without diammonium phosphate and also less triple superphosphate.

There are also many benefits to the manufacturing process itself. Perhaps the best way to illustrate this is to tell you what we do in our plant. Because we can obtain the correct liquid phase and heat required for good granulation there is no need for steam. Sulfuric acid is not needed for additional heat. It is needed only to neutralize any excess ammonia when it is impractical to increase the phosphoric acid. For example, by using too much phosphoric acid our recycle ratio becomes too great which then cuts our tons per hour. Because we can react 9 pounds of ammonia per unit of P_2O_5 from phosphoric acid as opposed to 6 pounds per unit from superphosphate and only 3.5 pounds for triple we can formulate more ammonia thereby using less nitrogen solutions. The solutions are not needed to arrive at a proper liquid phase. By using phosphoric acid we can increase the normal super phosphate in our formula. We do not need granular super to aid in sizing; therefore, all of our normal super is R.O.P. For the same reason our triple super is R.O.P. rather than granular. This is a big boost to the economics of our plant operation. We do use coarse potash in most of our grades but we are beginning to substitute some fine potash. 6-24-24 offers a very good possibility for eliminating coarse potash. Another benefit of phos. acid in granulation is the reduction of water in our formulas and in many cases water is not used at all. Besides improving the quality with phosphoric acid, the costs are much lower by cutting the solutions, sulfate of ammonia, triple super, and diammonium phosphate. We do not put any ammonium nitrate as such into our formulas.

The phosphoric acid is pumped from storage by a Worthington centrifugal pump. Our meter is a one half inch magnetic flowrator which has given excellent results and is the most trouble free piece of equipment in the plant. The acid is used in a scrubber before entering the pre-neutralizer. We scrub ammonia fumes from the ammoniator and pre-neutralizer. We pre-neutralize some of our grades and control the ammonia at a mole ratio, according to individual grades, between 1.2 and 1.5.

The temperature in the pre-neutralizer ranges between 215°F and 225°F. The slurry from the pre-neutralizer is discharged on top of the bed and then is ammoniated further in the ammoniator. We have a "saw-toothed" open trough for feeding the slurry when pre-neutralizing and a closed sparge bar with holes spaced as needed when not pre-neutralizing. Our fines are screened out and introduced into the ammoniator.

	Formula Without Phosphoric Acid		Formula With Phosphoric Acid	
	Lbs./Ton	Cost/Ton	Lbs./Ton	Cost/Ton
Anhydrous	124	5.69	124	5.69
Normal Super (20.5%)	382	4.59	942	11.34
Triple Super (46.5%)	697	21.38		
Phosphoric Acid (54%)			388	15.06
Potash (62%)	650	12.25	650	12.25
Sulfuric Acid	166	1.22		
Total	2019	$45.13	2104	$44.34

We operate at production rates between 16 and 20 tons per hour. The rate is influenced by our final screen analyses and moistures. 6-24-24 granulates so well that there is no problem keeping minus 20 mesh fines out of the finished product. On this grade we can run the maximum number of tons. The 6-24-24 moistures are at the 1% level; therefore, the increased rate is no problem for drying. The moistures on the rest of the grades vary between 1 and 2.5%, most averaging 1.5%. It is necessary to add more water when granulating 5-20-20 and the finished product moistures would be between 2 and 2.5%. A typical screen analysis for 5-20-20 is:

minus 6 mesh	99.3%
minus 8 mesh	73.3%
minus 12 mesh	18.6%
minus 16 mesh	4.0%
minus 20 mesh	0. %

The screen analysis for 6-24-24 run much larger in size and are as follows:

minus 6 mesh	99.3%
minus 8 mesh	54.6%
minus 12 mesh	5.9%
minus 16 mesh	0 %

Here is a comparison from the economic standpoint of a 5-20-20.

The 5-20-20 with phosphoric acid is less expensive by $0.79. In addition to this there is no need for steam.

Our formulas which include phosphoric acid are 4-12-36; 5-20-20; 6-24-24; 6-24-12; 6-24-24; 8-24-12; 8-32-16; and 12-36-12.

I hope these facts and figures will be helpful to you in your own plant operations. Thank you for your attention.

Moderator Phillips: Thank you very much Mr. Russo for that fine review.

Again, if you will just keep in mind and jot down your questions, we will try to answer them a little later.

At this point in our discussion of phosphoric acid technology, I would like to try to answer a few questions about superphosphoric acid and the production of it.

Production of Superphosphoric Acid

Alvin B. Phillips

The questions I shall try to answer are:

What is superphosphoric acid?
How is it made?
What are its properties?
What does it cost?
What is its future?

What Is Superphosphoric Acid?

Superphosphoric acid is a term used rather loosely to define a range of mixtures of ortho and polyphosphoric acids. It is the product you get when you remove enough water from ordinary strength phosphoric acid, such as merchant-grade wet acid—or when P_2O_5 is absorbed in a limited amount of water, as can be done when the acid is made by burning phosphorus.

Perhaps this can be understood more clearly by referring to Figure One.

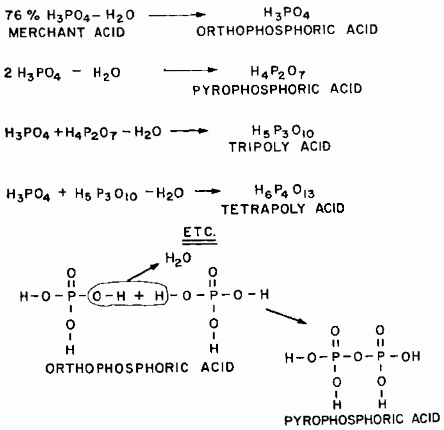

FIGURE I
DEHYDRATION REACTIONS OF PHOSPHORIC ACIDS

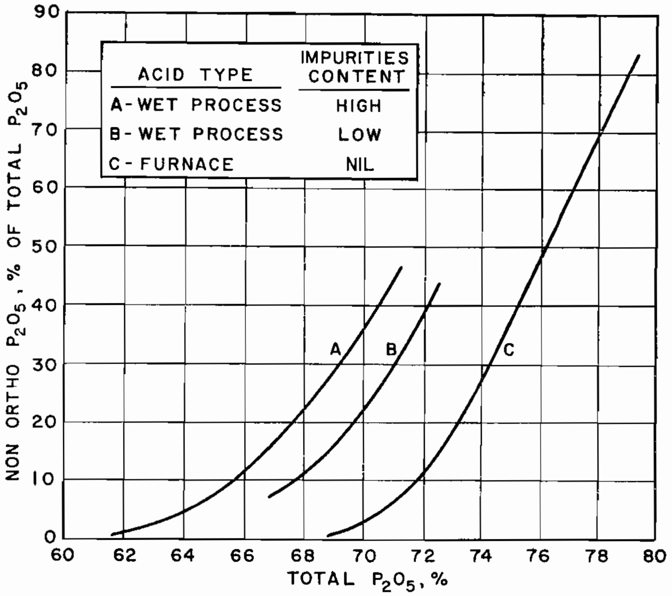

FIGURE 3
EFFECT OF TOTAL P_2O_5 CONTENT ON
NON ORTHO P_2O_5 CONTENT OF SUPER ACIDS

When water is removed from weak acid, the only change that occurs is an increase in the concentration of the acid—until 100 percent orthophosphoric acid (H_3PO_4) is reached. Then, as more water is removed, different types of phosphoric acid are formed—first the pyro form ($H_4P_2O_7$) when one mole of water is removed from two moles of ortho acid—then the tripoly is obtained as another mole of water is removed from a mole of ortho and pyro acid. At the bottom of the figure is a graphic representation of how this works. This process can continue through a whole series of acid—tetra, penta, hexa, and so on.

Superphosphoric acid is a mixture of these acids. The composition of the mixture—the proportions of the various acids—depends on the concentration—or perhaps more accurately—the degree of hydration. It can be shifted either by removing water from the system or by adding water.

Figure Two shows how the distribution of these various species in electric-furnace phosphoric acid varies as the P_2O_5 content is varied. The pyro builds up rapidly above 72 percent P_2O_5 and the ortho drops. Then as the concentration increases, the tripoly starts to rise and the pyro drops. The more condensed phosphates build up as the P_2O_5 content reaches the 80's, and the tripoly decreases.

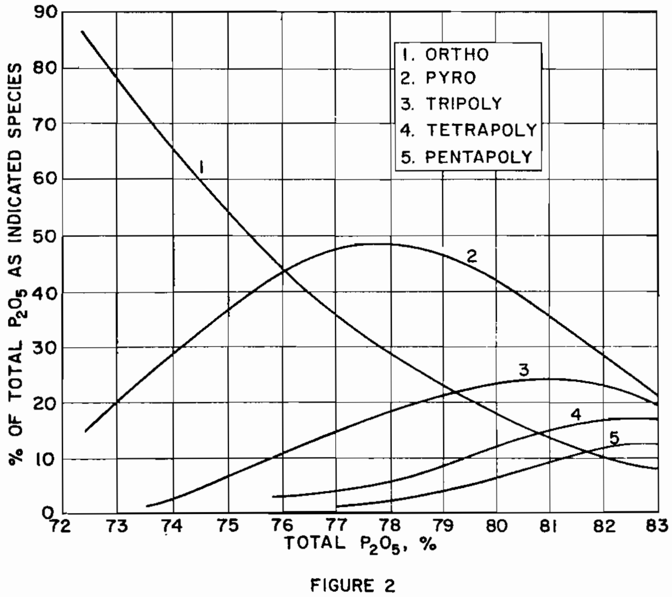

FIGURE 2
APPROXIMATE DISTRIBUTION OF SPECIES IN
ELECTRIC FURNACE SUPERPHOSPHORIC ACID

The same type of distribution is found in wet-process superphosphoric acid. However, as we can see by Figure Three, the nonorthophosphate content of wet-process acid starts to develop at lower P_2O_5 levels. This is simply because of the impurities in wet-process acid. At a given P_2O_5 content, as the impurities increase, there is less room for water so the phosphorus is present in less hydrated (or more condensed) species. Thus, a 40 percent nonorthophosphate level is reached at 70 percent P_2O_5 in acid which contains the most impurities, and at 75 percent in the relatively pure electric-furnace acid.

So we see that superphosphoric acid is a mixture of orthophosphoric acid and various species of nonortho acids such as pyro, tripoly, tetrapoly, and even more highly condensed phosphates. The proportions of these species present depend on the total P_2O_5 content of the acid and are influenced also by the purity of the acid.

How Is Superphosphoric Acid Made?

There are two general methods for making superphosphoric

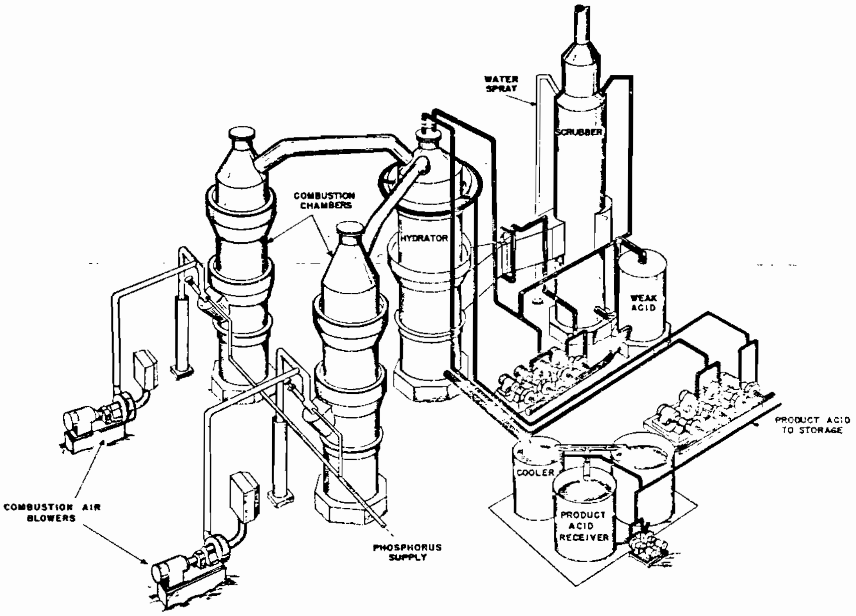

FIGURE 4
SUPERPHOSPHORIC ACID BY COMBUSTION OF PHOSPHORUS
(TENNESSEE VALLEY AUTHORITY)

acid in commercial use. Figure Four is a flowsheet of one of them.

This is the arrangement of our stainless steel acid plant at TVA. We burn elemental phosphorus in the two combustion chambers. The oxidized phosphorus then goes to a hydrator tower where it is absorbed by weak acid sprayed in the top and distributed on the walls of the tower. The combustion chambers and hydrator are water-jacketed open towers of stainless steel. From the hydrator the gases go through a venturi scrubber, a spray tower, and finally a demisting pad to remove acid mist. Water is sprayed in this tower. The resulting weak acid is pumped to the venturi and to the hydrator. The product acid flows from the bottom of he hydrator. A stream of product acid is cooled and recirculated to the hydrator to control the temperature and to prevent corrosion.

This plant has operated very well, producing either orthophosphoric or superphosphoric acid containing as much as 83 percent P_2O_5 with almost complete recovery of P_2O_5. The main difference between the production of orthophosphoric and superphosphoric acids is in the amount of water added.

But most of you do not have phosphorus to burn, so you'll probably be more interested in how superphosphoric acid can be made by concentrating wet-process acid. There are two methods of concentration in use; direct contact with combustion gasses and vacuum concentration.

Figure Five shows the arrangement of a pilot plant we built at TVA to study the direct-contact method (*3*). Natural gas is burned in a combustion chamber. After the addition of some tempering air, the combustion gas is passed through a pool of acid in the carbon-lined evaporator body. Weak acid is fed continuously to the bottom of the evaporator and overflows at the side to a sump that contains cooling coils. The exhaust gases pass through an entrainment separator after which they would require scrubbing to remove fluorine and residual P_2O_5. Products from most acids contained 70 to 72 percent P_2O_5, but higher concentrations were practical when starting with exceptionally clean wet-process acid.

There is at least one commercial plant that uses a system similar to this.

A somewhat similar approach to superphosphoric acid production involves the well-known system of submerged combustion (*4*). In this case a part of the burner—at least the discharge nozzle — is submerged in the acid pool. It has the advantage of somewhat better thermal efficiency but the design tends to be more complex.

There have been two types of

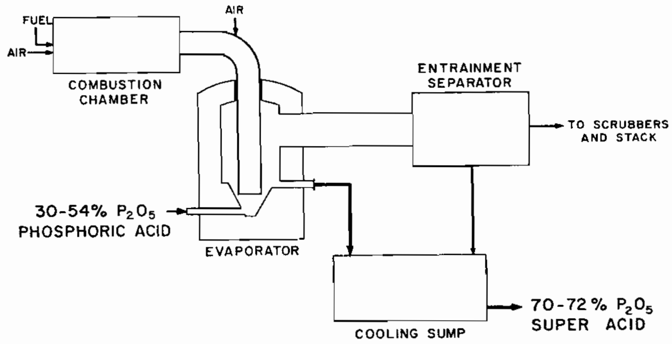

FIGURE 5
SUPER ACID BY DIRECT CONTACT WITH COMBUSTION GAS
(TVA PILOT-PLANT)

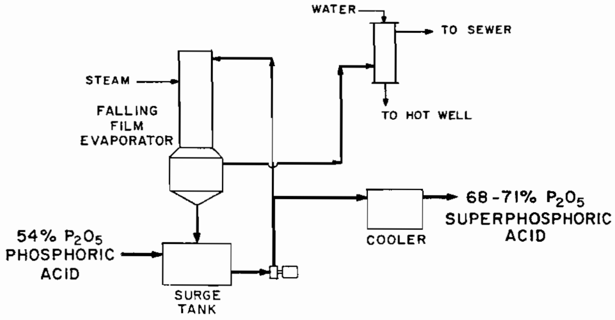

FIGURE 6
SUPERPHOSPHORIC ACID BY VACUUM CONCENTRATION
(WESTERN PHOSPHATES, INC.)

vacuum evaporators developed for superphosphoric acid production and placed in commercial operation. The flow diagram of a plant described by Petersen (*1*) that uses a steam-heated, falling-film vacuum evaporator is shown in Figure Six. In this plant, the feed acid—54 percent P_2O_5 — enters a surge tank from which it is pumped to the top of the evaporator. It then flows downward as a film on the inside vertical tubes and into the same surge tank to which the feed is introduced. Vacuum is provided by a barometric condenser and an ejector system. Acid is recirculated from the surge tank to maintain the required concentration in the product which is removed through a cooler to storage. The product concentration is around 68 to 70 percent. The tube chest of the evaporator is heated by steam. The metal used in the tubes was not specified by Petersen, but was said to be corroded at a rate less than 15 mils per year.

Another vacuum concentration system, shown in Figure Seven, was described in a recent article by Rushton and Smith (*2*). It uses a Swenson forced feed evaporator. Clarified feed acid is fed into the bottom of the evaporator and is forced upward along with recycle acid through the tube bundle of the evaporator. Heat is provided by a Dowtherm system in this case. Vacuum is provided by the usual condenser and ejector. Product acid is withdrawn through a cooler to storage.

In pilot-plant studies, forced circulation was found to be preferable to natural circulation because it decreased the tendency toward scale formation. Several alloys, including Nionel and Type 317 stainless steel, showed satisfactory corrosion resistance for tube construction. As was the case with the direct-contact evaporators, the retention time in the unit had to be limited to prevent the formation of insoluble iron-aluminum tripolyphosphate, although the limitations presumably were not so stringent because of the lower operating temperature. Products containing 68 to 72.5 percent P_2O_5 were produced with 40 to 55 percent in the nonortho form.

A large-scale plant of this type is in operation at the J. R. Simplot Company in Pocatello, Idaho.

Each system of concentration —direct contact with hot gas or vacuum—has its advantages and disadvantages. The chief disadvantage of the direct-contact method is that the exhaust gases contain fluorine and acid mist that must be recovered to avoid loss and atmospheric pollution, while the vacuum system does not require scrubbing equipment. The fluorine and what little acid is entrained in the vapor are recovered in the condensate.

On the other hand, the direct-contact method permits higher operating temperature and higher product concentrations. It does not have heat transfer surfaces to scale which require frequent down time for cleaning. At present the vacuum system is most favored by industry — I believe there are four commercial-scale units in operation compared to one or two of the direct-contact type.

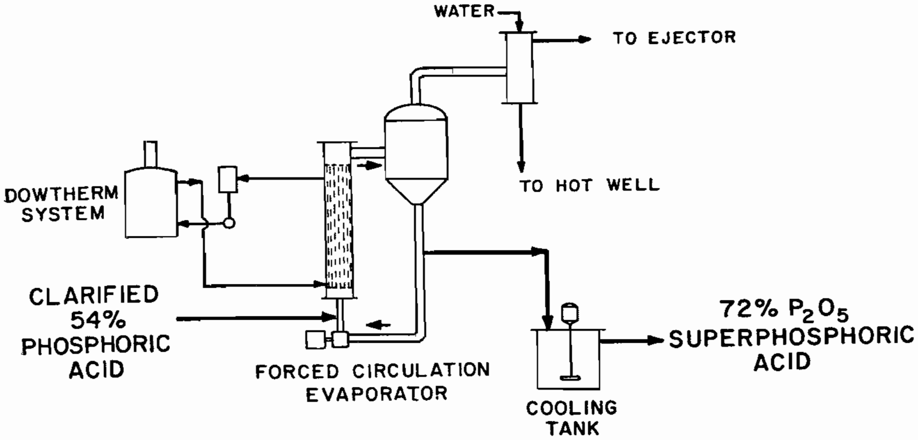

FIGURE 7
SUPERPHOSPHORIC ACID BY VACUUM CONCENTRATION
(SWENSON EVAPORATOR COMPANY)

What Are the Properties of Superphosphoric Acid?

The most interesting property of superphosphoric acid is its ability to sequester much of the impurities that ordinarily would appear as solids in ortho acid or its liquid derivatives. This is important because it greatly reduces or eliminates sludging of the product —a major problem with wet-process acid—and permits the production of clear liquid fertilizers from the acid. The extent of the sequestering depends on the level of polyphosphate and the amount and type of impurities present. Trivalent ions, such as iron and aluminum, are readily sequestered but divalent ions, such as magnesium, are difficult. Petersen reports that phosphate rock destined for superphosphoric acid production should contain less than 0.3 percent MgO.

The viscosity of the acid is another property of great interest. Superphosphoric acid — particularly the wet-process variety—is quite viscous, and this probably is its greatest disadvantage. The viscosity depends on the concentration of the acid and the impurities content.

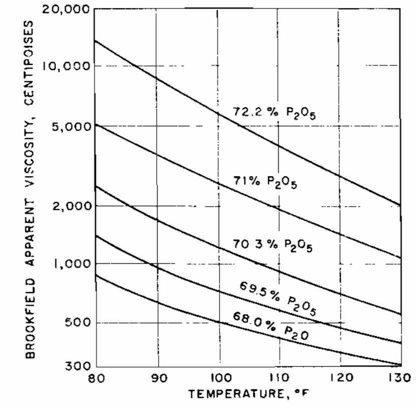

FIGURE 8
VISCOSITY OF WET PROCESS SUPERPHOSPHORIC ACID

Figure Eight gives temperature-viscosity curves for superphosphoric acid of different concentrations made from 54 percent acid that contained about 8 percent impurities (i.e., Fe_2O_3 + Al_2O_3 +

SO_3 + F). This acid was quite viscous at high concentrations and 80° F., but the viscosity was not unreasonably high at intermediate concentrations and slightly elevated temperature. In our pilot-plant work at TVA, we were able to pump all of the acids we worked with at reasonable rates with a positive displacement pump when they were heated to about 120° F.

Wet-process superphosphoric acid is much less corrosive than orthophosphoric acid. The corrosion rate of mild steel in superphosphoric acid made from a wide variety of orthophosphoric acids was in the range of 10 to 20 mils per year at temperatures of 120° to 150° F. This is much lower than electric-furnace superphosphoric acid, indicating an inhibiting effect of the impurities in the wet acid. A sample of wet-process superphosphoric acid was stored in a steel drum at room temperature for a period of 16 months without appreciable corrosion. These results indicate shipping of wet-process superphosphoric acid in mild steel should be possible. However, it would be wise to test the individual acid under consideration since the inhibiting effect of the impurities is not well understood. Also, dilution of the acid to the corrosive ortho range would have to be carefully avoided — including any that remains in the carrier on the return trip.

One more property worthy of mention is the low fluorine content. A considerable amount of fluorine is removed by either method of concentration. In our work with the direct-contact process the concentrated products usually contained 0.2 to 0.3 percent F compared with about 0.8 to more than 1 percent in the 54 percent acid used as feed. Fluorine contents of about 0.4 to 0.5 percent in superphosphoric acid concentrated by the vacuum method are reported.

What Does Superphosphoric Acid Cost?

The production of electric-furnace superphosphoric acid should not cost any more than the production of electric-furnace orthophosphoric acid in a properly designed plant.

The production cost of wet-process superphosphoric acid is, of course, more than the cost of wet-process orthophosphoric acid because additional concentration is required.

An estimate made at TVA on the direct-contact method indicated that a plant for concentrating 100 tons of P_2O_5 per day from 54 per cent to about 70 percent P_2O_5 would cost about $600,000. The operating cost would add about 6.5 cents per unit of P_2O_5 to the acid. A figure of 5 cents per unit was mentioned in an estimate from another source.

The only estimates I have seen for vacuum concentration give a wide range of 3 to 5.2 cents per unit. Presumably the lower figure would apply in a situation where steam was available at no cost.

I am not sure that the estimates are very comparable, but they may be useful to give some idea of the cost of superphosphoric acid. Certainly costs will vary from one location and situation to another, and estimates are not very reliable unless calculated for the individual case.

What Is Its Future?

The future of wet-process superphosphoric acid will, of course, depend on what advantages have been or can be developed to offset the added costs involved.

One obvious advantage is the high concentration that allows the shipment of about one-third more P_2O_5 per ton. If mild steel equipment could be used for transport, this certainly would add to the advantage—so would the freedom from sludge formation. On the other hand, the high viscosity reduces this advantage. Favorable economics undoubtedly would depend on rather long hauls.

Another possibility for offsetting the added production cost is to take advantage of the high concentration or polyphosphate content of the acid in its end use. For example, at TVA we have developed a high-analysis superphosphate (54%) using superphosphoric acid and are working on the production of ammonium polyphosphate which we believe may have particular merit as a carrier of micronutrients.

The use for superphosphoric acid that has received most attention to date—and the one that accounts for most of the present production of superphosphoric acid—is the production of liquid fertilizers. Another use that is not so fully developed, but is now under study by one of the manufacturers of wet-process superphosphoric acid, is its replacement of ortho acid in the production of granular fertilizers.

Both of these applications do take advantage of the high concentration of the acid to improve the product or the process. They will be described in detail by the next two speakers.

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2. Rushtton, W. E., and Smith, J. L. Superphosphoric Acid from Wet Process Phosphoric Acid. Chem. Eng. Prog. *60,* No. 7, 97-99 (July 1964).
3. Scott, W. C., Patterson, G. G., and Elder, H. W. Wet Process Acid: Pilot Plant Production. Ind. and Eng. Chem. *53,* No. 9, 713-716 (September 1961).
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Mr. Phillips: We are fortunate in having a representative here who has graciously agreed to substitute for Jim De Long who is listed on your program but was called away only yesterday. This gentleman absolutely needs no introduction to you. He is Dr. Frank Nielsson.

Dr. Nielsson: Mr. De Long has a prepared paper "Use of Superphosphoric Acid In Granular Fertilizers." I had a choice of either reading it or thought I would talk off the cuff and just talk about super-acid because super-acid is something new. It is something that we ourselves don't know too much about. Dr. Nielsson continued his remarks off the record.

Use of Superphosphoric Acid In Granular Fertilizers

J. M. De Long*

*Mr. De Long could not attend meeting. Filed for proceedings.

As shown by the topic emphasis of today's program, there is a great deal of interest and activity in the production and use of superphosphoric acid, both domestically and abroad. Most of the recent activity deals with the conversion of wet process superphosphoric acid into intermediates for the manufacture of high analysis liquid fertilizers and cold liquid blends. The most popular of these is 10-34-0 because of its manufacturing simplicity and its relatively trouble-free shipping characteristics. To a somewhat lesser degree, super acid has become an ingredient of high analysis NP solid materials. In this same vein, TVA has recently published their investigations on the use of super acid in the manufacture of nitric phosphate fertilizers (like 20-20-0, and 15-15-15). Certainly, all of these independent activities properly summed up, indicate the major role superphosphoric acid will play in the future of the fertilizer industry.

IMC, as one of the world's largest producers of wet process superphosphoric acid, has accepted the challenge of defining and projecting this role.

Our market development group in collaboration with research and technical personnel conducted laboratory and full-scale plant tests to establish and evaluate several end-uses for superphosphoric acid. In the course of this testing program, several tank cars of super acid were converted to various liquid and solid products. The manufacture of granular fertilizers took place in our plants at Plymouth, Indiana and Buffalo, New York. (TVA granulator and batch ammoniator).

Before describing this phase of our test work, I would like to give some background which leads us to believe superphosphoric acid would be beneficial in the process of fertilizer granulation. Super acid contains from 30 to 50% of its P_2O_5 in a form quite different from that of regular 54% phosphoric acid. Referred to as non-ortho phosphates, or generically, as polyphosphates, this portion of the acid acts quite differently than the orthophosphoric acid. Although relatively stable at moderate temperatures in moisture-free systems, the polyphosphate acids exhibit pronounced hygroscopic activity when exposed to moisture at elevated temperatures, extracting water from its immediate surrounding. And, this water is chemically absorbed without altering the free moisture content of the acid. This is because the water is instrumental in the conversion of polyphosphoric acid back to orthophosphoric acid. Taking our cue from this property of super acid, we felt that it could be turned to advantage in operations where it is desirable to reduce the free water content of the product. The most obvious consideration was the batch ammoniator operator who has to rely completely on chemical heat of reaction for drying as well as granulation. Since there are a large number of batch plants which have, as most a cooler and screens, and are thereby restricted to the manufacture of semi-granular products, we concentrated our efforts in this direction. Our thought at this time was that superphosphoric acid would substitute for a dryer while, at the same time, provide heat and liquid phase plasticity in the form of a highly concentrated plant food ingredient.

One of the first problems encountered was the method of proper handling and accurate measuring of the super acid. Previous experience taught us that this material must be moved into the plant sufficiently hot to overcome the rather high viscosities of the acid. In other words, we had to have a source of heat to raise and maintain the acid temperature in the tank car. Our cars are rubber-lined with stainless steel steam coils and bottom-discharge ball valves for unloading.

A study of the car design and the heating characteristics of the acid helped us select a hot water heater, moderate in cost with sufficient BTU heat output to bring the acid temperature up in 12 hours. Our first thought was to use a pump to empty the car, however, this was found unnecessary if large diameter, insulated pipe was used with sufficient air pressure on the car. All of our test work was carried out in this manner using mild steel pipe, however, permanent installation would require 316 stainless pipe to minimize corrosion.

We found that the acid boot measuring system was inadequate for accurate metering of the acid because of the intense black color and the retarded flow characteristics of the acid. Discrepancies in the levels of the boot and sight glass forced us to use a stainless steel positive displacement meter with indicator calibrated in volume units. For batch manufacturing purposes, we found an inexpensive 1½" water meter was suitably accurate. Previous testwork in continuous systems required a magnetic flowmeter which seemed to compensate for acid volume and temperature variations.

The acid was introduced into the ammoniator via an over-the-bed sparger pipe drilled at frequent intervals to insure distribution over the rolling bed. Dry materials, including screen undersize or fines, were fed to the ammoniator followed by nitrogen solution and acid. Of the numerous grades tested, only one called for additional sulfuric acid. This was added late in the cycle via the conventional acid boot.

Before describing the product characteristics and observed advantages to process and product alike, I would like to go back and describe the considerations made in formulation with superphosphoric acid. Because of its tendency to absorb water, we had to predict the amount of polyphosphoric acid which would hydrolyze back to orthophosphate. Taking the many variables into account, we estimated approximately 80% of the polys would revert to ortho form, thereby establishing the quantity of water which would be absorbed, in this case approximately 5 pounds for every 100 pounds of super acid.

Ammoniation rates based on previous experiments with 10-34-0 were another thing. Only through experiment did we find that the anticipated 7.2 lbs. NH_3/unit of P_2O_5 was too high. Although there appeared to be no detectable ammonia loss at the higher rate, there was an observable loss in the product analysis. We consequently backed off to 6.5 lbs. ammoniation rate and found this was ideal.

In most of the formulas calculated, the addition of super acid made some noteworthy changes over the conventional formulas. First, the addition of an extremely high phosphate source eliminated the triple requirement with a corresponding increase in the allowable quantity of normal superphosphate. Second, because of the excellent agglomerating characteristics of the acid, we found that coarse dry materials were no longer necessary. This benefitted in several ways:

(1) because of the increase in surface of the input solids, we found we could add as much as two units more N as nitrogen solution.

(2) this added liquid phase, together with the finer dry materials contributed to more uniform ammoniation and product homogeneity, and

(3) as you may have guessed by this time, there were slight formula cost savings as a result of using R.O.P. normal and standard potash as well as a lower-cost nitrogen source.

The batch ammoniator discharge was distinctly different from any we had seen before. Instead of the usual mass of partially formed granules which generally appear wet, hot, and steaming, the discharge looked much like hot carmel corn, that is, there were definite granules formed but they appeared to be sticky and efflorescent, massing together like a popcorn ball. Had these same balls been formed with sulfuric acid, there would have been all hell to pay in the crushing circuit following the screens. As this hot mass cooled, however, most of these balls broke down, into on-size granules, hard, dry, and essentially dust-free. There was a small quantity of oversize which made up the recycle after passing through a closed circuit crushing system. Generally speaking, the quantity of fines recycle was quite low and in no way affected the overall throughput of the system.

As a matter of fact, production was increased slightly by the elimination of in-between-batch delays such as filling up the acid boot and making that extra trip to the triple bin for solid P_2O_5.

It is important to point out here that we worked with the regular operating crews in the plant who, for understandable reason, are always leary about new process gimmicks. Before a morning had passed, they were completely sold on the simplicity of operation and the elimination of ammonia fumes from the operator's working area. Because of the limited affinity of super acid for KCl, there was no evidence of the formation of ammonium chloride in the stack gases.

Products to storage were approximately 92% 6 x 16 mesh with essentially no minus 35 and, even though the pile temperature exceeded 120° F., the product remained free-flowing, dust-free, and well conditioned indefinitely.

Several comparison tests were run including hardness, attrition, condition, and storage tests using conventionally prepared fertilizers of the same analysis as the standard.

Generally speaking, there was a 30% increase in hardness, 30% less attrition when subjected to rather severe tumbling, and total elimination of caking. This was determined by bagging several tons for palletizing and then checking bottom, middle and top bags at regular intervals for set.

A soft chemical set was observed and attributed to the residual polyphosphate content as it reached equilibrium with respect to temperature. We considered this desirable for bagged products as it precluded any possibility of segregation. A single drop from waist height, simulating normal handling of bagged or bulk product in shipping, resulted in a free-flowing product which contained no lumps or dust.

Chemical analysis of the several grades showed that 98-99% of the P_2O_5 was in available form and from 50 to 75%, depending on the formula and the quantity of acid used, was in the water soluble form of P_2O_5. Prior to analysis, all samples were ground thru 80 mesh and hydrolized by conventional methods.

Since each of the grades manufactured were premium products, containing SPM, and minor elements, we were acutely interested in what effect the poly content would have with respect to chelation. Interestingly enough, from 35 to 75% of the magnesium in the product wound up as chelated Mg.

Our test program has been extended on the basis of these unusual findings and we foresee the day when the batch ammoniators alike, will find it practical, economic, and agronomically desirable to use superphosphoric acid as an essential part of their formulations.

MODERATOR PHILLIPS: Thank you very much, Slug, for substituting for Jim De Long and for those pertinent remarks. They were very helpful, I am sure.

Last Monday Jack Smith of Simplot called me and said that he could not make this meeting because of a plant start up. He did send me a copy of his talk on the use of superphosphoric acid and liquids.

A very good friend of mine kindly volunteered to deliver this paper. This good friend works with me at TVA. Most of you know him. I am very proud to introduce Mr. Frank Achorn.

MR. FRANK ACHORN (Tennessee Valley Authority): Well, I am glad I've got one real good friend.

I feel like I am trying to substitute for *Johnny Unitas* in the last 30 seconds of the ball game, however, I have read the paper and I do agree with everything that is in it. I had the pleasure of working with Simplot a little in this program, therefore, I am a little familiar with it.

The Use of Wet-Process Superphosphoric Acid In Liquid Fertilizers

Jack Smith

Paper presented by Frank Achorn

A RECENT issue of Chemical Week magazine lists eleven producers of wet-process superphosphoric acid who concentrate about 1,000 tpd of P_2O_5 to the superacid level. Most of this acid is used in the production of high--analysis liquid fertilizers.

So far as we know, all of these liquid fertilizers are true solutions although slurries and suspensions based on wet-process superacid may become popular in time if higher analysis products may be produced in this manner. Presently, the most popular grade is 10-34-0, which is made by reacting superacid with ammonia and diluting, simultaneously, with water. Some of the other products made from the same materials are 9-34-0, and 7-21-0. Of course, 8-24-0 and 7-21-0 may be made from ordinary wet-process acid, but this usually results in the formation of large quantities of sludge as the iron and aluminum present will precipitate, as phosphate salts.

Liquids Produced From Super Acids

These products are quite stable for reasonable periods of time, and other materials may be added without producing precipitates. By the addition of urea ammonium nitrate solution and muriate of potash, it is possible to make a wide variety of solutions with good handling characteristics. The grades of some of these solutions are: 13-26-0, 16-24-0, 21-7-0, 12-16-0, 7-21-7, 8-8-8, 6-6-12, 21-14-0 and 16-16-0.

Because of the peculiar properties of superacid, it must be handled differently than ordinary wet-process acid. The viscosity, for example, may be as high as 50,000 centipoises at 80°F, however, by heating the acid to about 150°F it becomes fluid enough to be pumped with a centrifugal pump. At lower temperatures a positive displacement pump must be used. One approach to this problem has been to load the superacid at 160° to 180° Fahrenheit into insulated tank trucks which carry the acid as far as three hundred miles with a temperature drop of only about ten degrees. Another approach is to equip tank cars and trucks with steam coils or jacketed outlets to heat the acid at its destination. Where a superacid concentrator and reactor exist at the same site, as at Pocatello, the problem is reversed. The hot acid leaving the concentrator is cooled to a desired handling temperature before it is fed to the reactor.

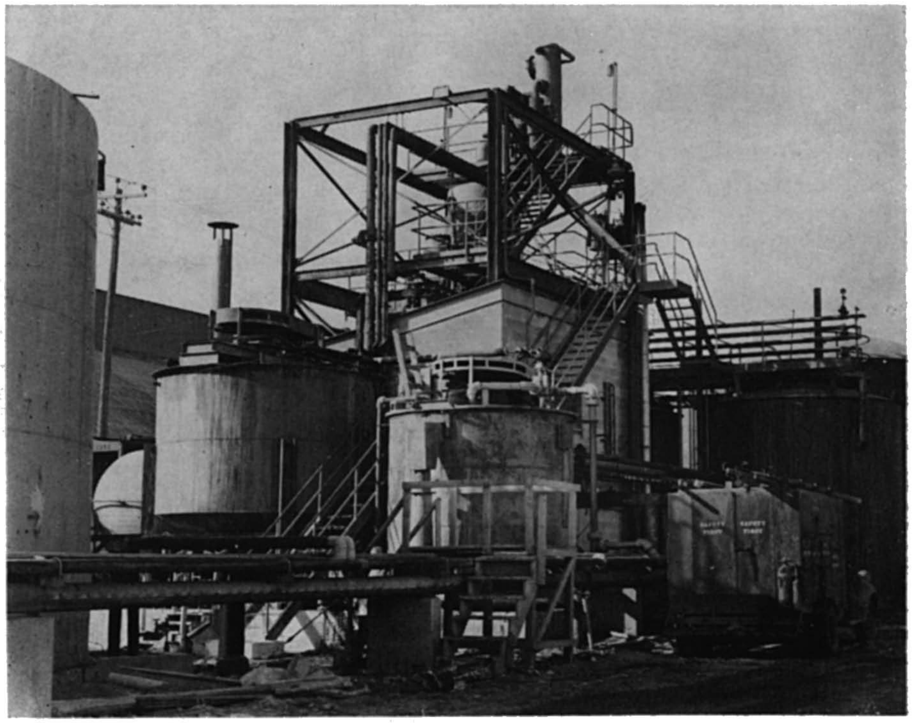
Another of superphosphoric acid's unusual properties is its great density, about 16.7 pounds per gallon, which must be taken into account when designing storage tanks.

The corrosive nature of superphosphoric acid is quite different from that of its cousin, merchant-grade acid. Although superacid is quite corrosive at high temperatures, it attacks mild steel very slowly at ambient temperatures. For this reason, Simplot is storing superacid on a test basis in a mild steel tank. We do not presently have enough experience in this area to recommend that others do the same.

A variety of reactor designs have been successfully employed to make solution fertilizers from superphosphoric acid or to convert one of these solutions into another product such as 8-24-0 by the addition of ordinary wet-process phosphoric acid, ammonia, and water. Such a product has superior clarity and handling characteristics because of the polyphosphates present in the solution. Sequester or essentially dissolve the impurities introduced by the wet-process acid.

The Pocatello reactor is patterned after a unit in use at the TVA, Wilson Dam, Alabama. It operates water flowing through continuously with the heat of reaction being taken away by heat exchange which is installed in the reactor. A 3-hp mechanical agitator keeps the materials well mixed. Superacid is fed at a controlled rate by a speed-controlled gear pump; this fixes the rate of reactor operation. Anhydrous liquid ammonia is sparged into the reactor and controlled automatically by a pH controller which measures the pH of the product at the reactor outlet. Water is added through a rotameter and manually controlled to give the desired product density. This reactor, which is

Photo #1. Overall View of Liquid Fertilizer Plant, Pocatello, Idaho.



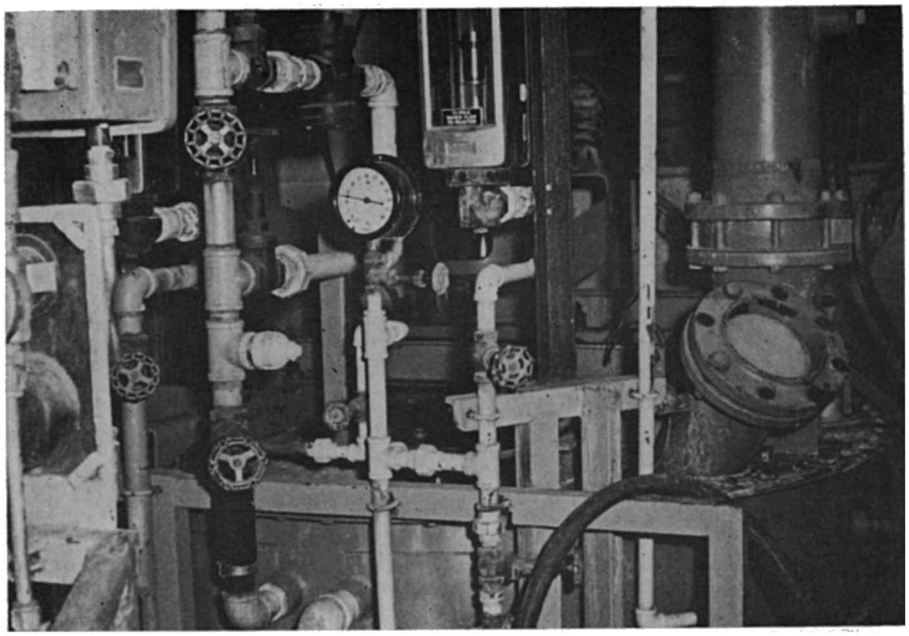


Photo #2. Top of Reactor Pocatello Liquid Fertilizer Plant.

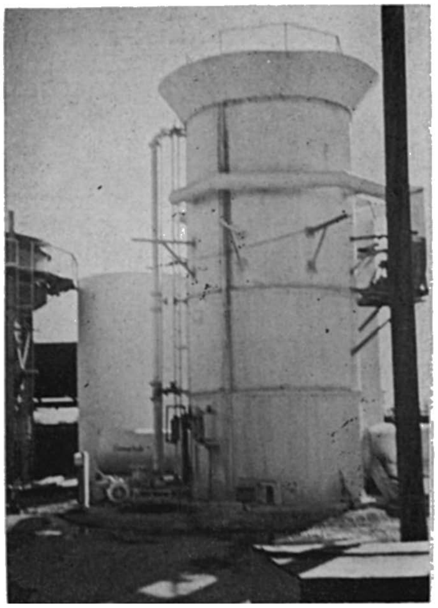


Photo #4. Liquid Fertilizer Reactor, Twin Falls, Idaho

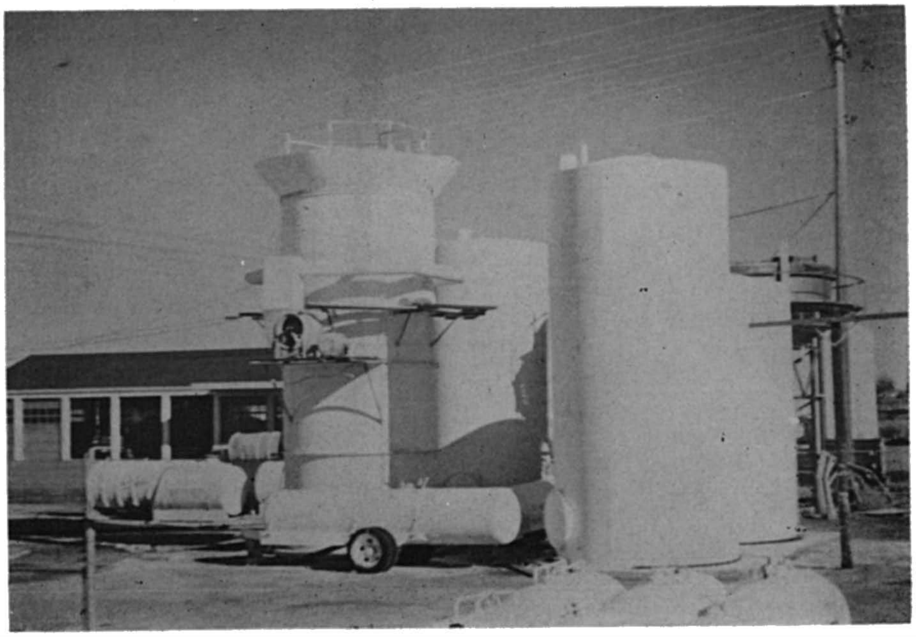
only about eight feet high x five feet in diameter can produce 200 tons per day of 10-34-0 solution. The acid and water are fed through open end pipes and enter the surface of the liquid in the reactor. There are two perforated ammonia ring spargers, one just below each of the two turbines. The flow of cooling water through the heat exchanger is automatically controlled so that the reactor temperature is held constant at about 160°F. The product leaving the reactor is pumped to a two-pipe heat exchanger where it is cooled by water to about 110°F before entering the day tanks. This cooling helps to keep the rate of polyphosphate reversion low. Material in a full day tank is circulated for about two hours, sampled, and analyzed. If the material is off-grade, additional acid, ammonia, or water may be added. When it has been determined that a day tank is on grade, its contents are transferred to the large storage tank, which has a capacity of about 70 0tons.

A second type of reactor is one commonly used in the Intermountain West near the points of use. It consists of a mild steel storage tank which is equipped for recirculation to ammonia and acid spargers so that the entire tank is a reactor. The reactors at the Simplot Soilbuilder Units in Blackfoot and Twin Falls, Idaho, are of this general type. One of the serious problems in operating a reactor of this type is dissipating the heat generated by the reaction. If the heat were not removed, the contents of the reactor would soon reach their boiling point, and ammonia losses by vaporization would become substantial. A cool reactor also runs more smoothly as it is free of the bumping associated with collapsing vapor bubbles. The Blackfoot reactor uses a thin film of water running down the outside of the reaction tank to remove heat from the product. This water film is produced by an ordinary lawn soaker wrapped around the circumference of the tank near the top.

The Twin Falls reactor uses air to cool its product. A cooling zone in the top of the tank contains a number of spray nozzles which spray hot solution down toward the surface of the product in the tank. A blower on the outside of the tank directs a stream of cool air upward against the spray and the solution is cooled by evaporation.

The reactors at both Twin Falls and Blackfoot were designed for the production of 8-24-0 solution from wet-process phosphoric acid but could be easily modified to convert superphosphoric acid to 10-34-0.

Photo #3. Liquid Fertilizer Plant, Twin Falls, Idaho.



Another means of contending with the problem of reaction heat is to make a less concentrated products than 10-34-0. One liquid fertilizer producer, makes a 7-21-0 product from superphosphoric acid and cools the freshly-made product by mixing it with a large quantity of previously-made product held in storage. In this way, the heat is dissipated without the use of any cooling equipment.

One of the advantages of making a neutral solution is that corrosion rates are very low, only a few mils per year on mild steel. As a result, it is possible to use piping, storage tanks, pumps, and valves made of steel in handling the products.

The neutral solutions made from superphosphoric acid normally store well, especially in cool weather. Usually there are enough polyphosphates in solution to prevent the settling out of iron and aluminum compounds such as occurs in solutions made from regular wet-process acid. A persistent problem with Western superacid has been the settling out of magnesium compounds in the finished solutions, but this difficulty is gradually being overcome.

The duration of allowable storage time is dependent on outdoor temperature, the acidity and polyphosphate content of the solution, and the concentrations of metallic impurities in the product. High storage temperatures, low pH's and high concentrations of the metallic impurities all limit the storage period. This is because a certain minimum ratio of polyphosphates to dissolved metals introduced by the acid must be maintained, and high temperature and low pH hasten the reversion of the polyphosphates to ordinary orthophosphate, which has no sequestering value.

Superphosphoric acid enjoys several economic advantages over merchant grade wet-process acid:

1. Because of the high P_2O_5 concentration of superacid (70% vs 52% for merchant acid in Simplot's case) the superacid may be shipped for about 25% less, per unit of P_2O_5. This, coupled with relatively low cost of converting the superacid to neutral solutions, makes the installation of reactors attractive in areas remote from the acid source but near the points of use.
2. Because more concentrated solutions may be made from superacid than from merchant acid, they may be transported and applied more cheaply. For example, if a 1,100 gallon application truck is to apply 10-34-0 made from superacid or 8-24-0 made from merchant acid, the advantage of the 10-34-0 may be seen as follows:

Products	8-24-0	10-34-0
Density	10.85 lb/gal	11.85 lb/gal
Weight of 1,100 gallons	11,935 lb	13,035 lb
Units of Plant Food	191.	287.

   The load of 10-34-0 contains about 50% more units of plant food and, as a result, may be handled for about 33% less cost per unit of plant food applied.
3. The high-analysis, high-density products, because of their lower application and transportation costs and the extra mileage gotten out of application equipment, permit liquid-mix plants to broaden their distribution areas and handle increased volume without purchasing additional application equipment. According to one operator, his radius of operation is doubled when he uses 10-34-0 as compared to 8-24-0.

At present the liquid fertilizer producer still has some problems to contend with in the manufacture of solutions from wet-process superacid.

For example, if a liquid-mix unit is not properly equipped to handle superacid, difficulty may be experienced because of the high viscosity of the acid. Some difficulties may also arise if the superacid is stored at high temperatures for extended periods of time or stored at too low a polyphosphate content. In the case of high-temperature storage, tripolyphosphates may form which will cloud both the superacid and subsequently the solutions made from it. There are at least two recorded instances of too-low analysis superacid turning solid in storage; this apparently is because there were not enough polyphosphates in solution to sequester the metallic impurities, and the impurities precipitated out in large quantities as sludge. Also the solubility of the acid itself is lower at the lower P_2O_5 contents.

The operation of well-designed liquid-mix reactors is almost completely trouble-free. An exception to this rule is the off-control operation of a reactor, which if severe, can result in the production of a sludgy product.

Most of these difficulties can be avoided completely through the use of proper equipment and procedures, and it is the author's opinion that they are more than offset by the advantages of using superphosphoric acid.

Commentary on Slides

1. This is the liquid fertilizer plant at the J. R. Simplot Company's Pocatello complex. The large, light-colored tank in the right foreground is for superphosphoric acid storage, and the large, dark-colored tank in the left background is for 10-34-0 storage.
2. In this view, the superacid surge tank is shown at left of center, and the concentrator feed tank at the center.
3. Here, the top of the 10-34-0 reactor is shown through a maze of piping. The mixer drive is at center background and a vent line appears at the right. This line goes to a blower outside the building and provides protection for the operator in case a large quantity of ammonia is accidentally released.
4. This shows the reactor outlet where the product passes through a weir box in which stands a pH measuring assembly. This is part of the pH control loop which regulates the ammonia flowing into the reactor.
5. This is the control panel from which both the superacid con-

centrator and the 10-34-0 reactor are operated.

6. At the center foreground is the alloy gear pump which feeds superacid to the 10-34-0 reactor. The variable speed drive is remotely controlled from the panel shown in the previous slide.
7. This is the two-piece cooler which cools the 10-34-0 being pumped to storage. The 10-34-0 is in the outside pipe allowing some heat loss by convection.
8. This is an overall plant view with a typical tank truck being loaded with 10-34-0 solution.
9. In this view, the superacid storage tank is seen at the left. Part of the superacid concentrator may be seen at extreme upper right.
10. This slide shows the shell and tube heat exchanger which heats superacid being taken out of storage.
11. This is the Blackfoot, Idaho, Simplot Soilbuilder liquid fertilizer plant.
12. The tank in the center is the Blackfoot solution reactor. It contains a Maas reactor unit and is equipped with circulating equipment. The cooling water distributor is at the top of the tank wall.
13. This is the Twin Falls, Idaho, Simplot Soilbuilder liquid fertilizer plant. The solution reactor is in the center.
14. This view shows the cooling air blower and air ducting.
15. The warm air leaves the reactor above the angular ring at the top. The product is made below the level where the air ducts enter. This reactor utilizes swirl rings and spargers and the reaction occurs throughout the liquid mass.
16. This view shows the reactor controls and circulating pump.
17. Another load of Simplot liquid fertilizers.

MODERATOR PHILLIPS: Frank, thank you very much for doing a splendid job of reading this paper, but don't go away.

I'd like to ask the panel to come up here to the table now so we can try to answer any questions that you might have on this series of talks.

A CONFEREE: I would like to ask a question about the corrosion of the Nionel tubes. They have considerable trouble, I understand, in the Florida phosphate concentrations with Nionel tubes but from Mr. Weber's talk, I understand that the first stage seems to be the only problem that you have in this series. My question is this: Are there no problems in the use of Nionel tubes when you go all the way from 30 to 54 per cent P_2O_5?

MR. WEBER: While Karbate is now generally used, we are successfully using Nionel on the second or third stages of series-operated units and so long as only strong acid is in contact with the tubes, the Nionel stands up quite well. Therefore, you could use Nionel on a single-stage unit if you were concentrating to at least 47 per cent P_5O_2.

Is there another question? Yes, Doctor?

DR. MUKHERJEE: I would like to pose a few questions to the panel, if I may.

One is whether the type of rock affects the transition temperatures from gypsum to hemi-hydrate and from hemi-hydrate to anhydrite in the wet-process?

Mr. Weber mentioned a sufficiently designed reactor system and I was wondering whether the design of the reaction system had to be varied to handle different types of rock.

The next question is, are there legal restrictions anywhere in the United States as to fluorine emissions in phosphoric acid plants?

Next, is the byproduct gypsum from phosphoric acid plants utilized in this country?

On the super acid, I have two questions. One is, most of the discussions this morning has been on 70 to 72 per cent super acid. I have seen references to 81 to 83 per cent acid. What is the experience on this?

From Mr. Phillips' paper, we gather that there are two factors involved in corrosion, not merely the water content but also the effect of the various other materials present in the acid. Now does it mean, therefore, that for every batch of acid one has to run a corrosion test before one could depend on whether it could be shipped in carbon steel containers or not? The next point is, what is the present status of phosphoric acid as a commodity for sale? Is it available for sale and if so, what tonnages are being handled?

MODERATOR PHILLIPS: The panel here has been taking some notes and we will take these questions up one at a time in the order you asked them.

MR. WEBER: First, Dr. Mukherjee asked whether the type of rock affected the transition temperatures from gypsum to hemi-hydrate and from hemi-hydrate to anhydrite?

The amount of impurities in the acid would affect the temperature and concentration transition point but not to a very great degree except in the case of sulfuric acid. In this case, the transition concentration is essentially a function of the total amount of sulfuric acid and phosphoric acid.

The second question on whether the reaction system has to be varied to handle different types of rock. With most normally used commercial rocks there is not much difference. If the rock is highly reactive, such as Morocco, we might use lower detentions than with, for instance, Florida rock. On the other hand, there are some phosphate rocks which are much less reactive, such as Kola rock where we normally use greater detentions.

As to legal restrictions on fluorine emissions in phosphoric acid plants, this is becoming more and more restrictive, particularly in Florida where they have experienced damage to vegetation and they have made fairly exhaustive studies.

They are now putting limitations on the total fluorine emission from any plant and, whereas at one time this was 100 pounds of fluorine per day, it has been reduced to 50 pounds and they are now talking 25 pounds.

They are also putting limitations on the total fluorine emission in any particular area and refuse to issue licenses for the construction of new plants if there is already a concentration of plants in any particular area.

As to whether the byproduct gypsum from phosphoric acid plants is utilized in this country, to my knowledge, at the present time there is no such usage. Natural gypsum of high quality is very generally available at quite low prices. The byproduct gypsum is used in other countries, particularly in Japan, for gypsum plasters and wallboard and in some cases as a cement retarder. Gypsum is also used in some parts of the world as a material from which to produce ammonium sulfate.

Moderator Phillips: In answer to Dr. Mukherjee's questions on super acid, I do not have any information on the production of 81 to 83 per cent P_5O_2 wet-process super acid. As you mention, there have been references to the projected production of acid of this strength in recent news articles. Perhaps there is someone in the audience who can furnish some information?

I am sure it would not be necessary to measure the corrosion rate of each batch of acid to determine whether it can be shipped in carbon steel. However, I believe corrosion tests should be made on acid from a given plant and they should be repeated when there is a drastic change in conditions, such as the use of a new type of rock, at least until more experience is gained in handling the acid in carbon steel. I think there has been little wet-process super acid offered for sale; most of it as produced for internal use by the producer. Perhaps there are some in the audience who could verify this?

Edwin Cox, III: I would like to say something on the corrosion problem, sir.

The only reason I would like to talk about the corrosion problem is not from our own experience with superphosphate but we did do quite a bit of work on corrosion. There are many factors that must be taken into account in the use of laboratory corrosion data. These are the effect of welds, stresses from mechanical working, electrolytic corrosion because of dissimilar metal and so forth.

Moderator Phillips: Thank you. We try to consider these in our corrosion tests but I am sure there is no substitute for practical experience.

Yes, Dr. Waggaman?

Dr. Waggaman: I would like to get your opinion as to whether 0.8 per cent fluorine would be objectionable in a phosphate rock used for making phosphoric acid. In the case of this new deposit in Peru which is being developed, fresh water is not available.

Mr. Weber: It would be extremely dangerous. We have had experience with rocks with much lower fluorine than this or where the water supply contained significant amounts of fluorides and it had caused very severe corrosion in the phosphoric acid plant. The stainless steels normally used do not stand up to a mixture of fluorine, phosphoric and sulfuric acids.

For moderate amounts of fluorine, it is possible to use higher and much more expensive alloys and other materials of construction, but this adds considerably to the cost of the plant and, if the rock were being generally merchandised, plants not specifically designed to handle such high fluorine rocks would not be able to use the rock.

Dr. Waggaman: That would not apply, I don't suppose, to superphosphate?

Mr. Weber: I don't think so, no.

Dr. Waggaman: Thank you.

Moderator Phillips: Thank you.

A Conferee: I believe one of the gentlemen on the panel stated that magnesium was chelated by the polyphosphate in his work on granulation, but the paper submitted on the Simplot Company operation stated that magnesium was difficult to sequester in the production of liquid fertilizers.

Moderator Phillips: I believe Dr. Nielsson went off to catch a plane. I know this was one of the problems mentioned by Petersen in his paper on super acid production. He said that phosphate rock with magnesium could not be used because the magnesium was not readily sequestered.

Mr. Achorn: I, too, questioned this. This is one of the reasons why water softeners have to be used in liquid fertilizer plants. The magnesium reacts with phosphoric acid and ammonia to form a relatively insoluble compound. The water softeners are used to remove the magnesium prior to the introduction of the water into the liquid fertilizer mixture.

Moderator Phillips: Is there another question for the panel?

I certainly want to thank this panel for the fine papers they have contributed and their companies for releasing the information that made these papers possible.

Chairman Sauchelli: That expresses the opinion of all of us. I think we have had a splendid session this morning, worthwhile in every possible way, very practical and useful information.

I want to take this opportunity to just mention a few things. We have had 13 visitors or guests here from overseas, one from Morocco, three from Italy, two from Scotland, one from England, one from India, four from Puerto Rico, one from Aruba of the Antilles and a delegation of 48 friends from across our northern border. We don't consider the latter foreigners. We are certainly pleased always to have visitors here from overseas and from Canada.

I would like to recognize our foreign visitors. Please stand. We always like to welcome our visitors from overseas. I know several have already left.

One of our great aids is The Trade Press. Will you please stand so that we may recognize you. It is always helpful for the fine publicity from their respective magazines.

They are really fine workers. They keep busy here.

Mr. Secretary, do you have anything else you want to add.

Dr. Marshall: Nothing more.

I thank my associates on the Executive Committee for their fine cooperation. Thanks to the management of the Mayflower Hotel for their helpful cooperation in making our stay here pleasant. They have tried to help in every way to make our meeting pleasant.

We stand adjourned until next year. Now remember let us have your suggestions for our 1965 meeting, November 10-11-12.

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Note: Many more participants who did not announce their names when asking or answering questions.

Notes

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