PROCEEDINGS
OF THE
FERTILIZER INDUSTRY ROUND TABLE
1957

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OPENING REMARKS

Vincent Sauchelli, Chairman

WELCOME, friends, to the 6th annual Round Table.

This Round Table is somewhat different in organization, from previous meetings. Because of the large attendances at the last two get togethers, it was necessary to do something different if we were to maintain the essential character of the Round Table—namely, free and open discussions of the problems put before the meeting. Many suggestions were received from you on how to meet the situation. Mr. Hignett, TVA, one of our loyal, most cooperative associates, sent me the germ of the idea which your Executive Committee worked out. We hope it meets with your approval. The plan is this. We asked you all to send us the problem you would like to have discussed. About 100 questions and problem statements were received and classified as to main phases of operation. Next we forwarded to a randomized selection of our members a copy of these same questions with the request that they suggest those among our members who they thought best qualified to answer the questions in a practical, satisfying manner. It was gratifying to receive your unstinted help. From these suggestions we made up this year's agenda.

The men chosen to answer and discuss the qusestions will be grouped and constitute the active Round Table. Each of them will have not more than 5 minutes in which to do his work. The rest of the members will be given an opportunity to comment on the question if it is felt necessary to complete the discussion initiated by the Round Table leader.

We hope our procedure works. It can if each of us cooperates in observing the time limits.

We have a tape recording service which will record your comments. Please note this: We plan to print all the questions and answers as a "Proceedings" of our meeting. Most of you have requested that we do this. The cost of printing the proceedings will come out of our own funds. Since the discussions are always helpful to an outstanding of the problem, it is our purpose to publish them, also. But before final publication each person whose comments are recorded will be given the opportunity of reviewing the comments and sending in to us final copy for publication. This procedure should have your approval.

This Round Table is your Round Table the more so as you contribute to the discussions which in our opinion form the most important contribution of this meeting to future developments in fertilizer technology. We are looking forward to you for a critical and constructive discussion of the several questions before the Round Table.

We can all greatly assist one another by the fullest possible exchange of information on the results of research and your practical experience in the application of those results.

We have come a long way in five years in the matter of exchanging ideas in our industry. These Round Tables have been most helpful in bringing this about. The entire industry has been the beneficiary. No one can go it alone. Except for certain patentable information on which a firm may have spent considerable money, the fund of information in an industry—the largest portion of pertinent information—can be freely exchanged and discussed with benefit to all.

The meeting is now open.

SECTION 1 — GENERAL

Optimal Particle Sizes of Various Fertilizer Salts for Various Soil Conditions and Crops

G. F. Terman

BECAUSE of the high solubility of most nitrogen salts and their high mobility in soils, there is little or no problem in regard to granule size of nitrogen fertilizers. Granule size of nitrogen materials such as ammonium nitrate, nitrate of soda, and sulfate of ammonia should be the size range which is best for storage, handling and application.

The bulk of the potassium fertilizers used, muriate and sulfate of potash, are also highly soluble. Potassium is less mobile in the soil since it is sorbed more strongly than nitrogen and much of the potassium applied in fertilizers may be fixed by the clay minerals in slowly soluble forms. There is no evidence that differences in availability exist over the usual range of size of muriate or sulfate particles. There is some evidence that larger particle sizes of some experimental fused potassium phosphates produced by TVA are more slowly soluble and available to plants than are finer particles. Additional experiments are under way at present on this problem.

The problem of granule size with various phosphate salts is vastly greater and more complicated than with nitrogen and potassium. The problem of granule size of NP and NPK fertilizers in relation to its effect on crop yields is largely related to the phosphorus component. Phosphate salts vary from high water solubility (monoammonium, diammonium, and monocalcium phosphates) to very ow water solubility (dicalcium and ricalcium phosphates) and even to ow citrate solubility (fluorapatite in ock phosphates). Consequently, one must specify the phosphate salts presnt in the granule in discussing availbility to crops. Highly ammoniated uperphosphate based fertilizers and itric phosphates usually contain comlex mixtures of ammonium, monoalcium, and dicalcium phosphates nd apatites.

The relationships between grane size and availability are much o complex to cover in a few mines, but a few generalizations can made:

1. With the low water soluble phosphates, such as dicalcium phosphate, rate of solution of phosphorus from the granule is usually the dominant factor in availability of the phosphorus to plants. Thus, granules of low water soluble phosphates should be relatively fine. For example, in a greenhouse experiment conducted by TVA at Wilson Dam, 6-14 mesh dicalcium phosphate was only half as available to wheat as —35 mesh material.

2. With water soluble phosphates, dissolution from the granule and fixation of phosphorus by the soil are usually the dominant factors in availability. Granules of these phosphates should be relatively large, and the optimum size will vary with the capacity of the soil to fix phosphorus and with other factors. From the availability standpoint, banding of finely divided phosphates near the seed is in part a satisfactory substitute for granulation under many soil conditions.

3. Differences between early growth response and final yield response to phosphorus have been a source of confusion in the interpretation of results from granule size, water solubility, and other types of experiments. Yield responses in greenhouse experiments and early growth responses in field experiments on phosphorus deficient soils are usually found to soluble phosphates properly applied. Whether these early growth responses follow through to final yields of grain or forage in the field depend on several factors. These include amount of available phosphorus in the soil, adequate supplies of other nutrients, length of season, and many other factors. Final yield responses are found more frequently on potatoes and vegetable crops than on corn and small grains.

4. In general, for use on acid soils, fertilizers containing about 50 percent or more of the phosphorus in water soluble forms should be granulated coarsely (about 6-20 mesh) and phosphates of about 25 percent or less water solubility should be rather finely divided (about —35 mesh). The optimum granule size

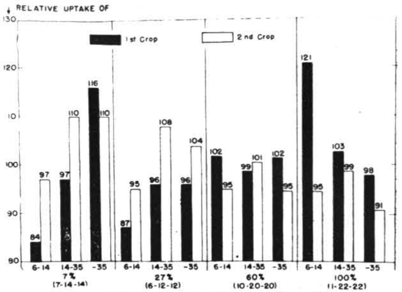

Figure 1. Effect of Granule Size and Percent Water-Soluble P on Mean Relative Uptake of P by Two Greenhouse Crops of Sudangrass and Oats.
(Mean uptake of each crop from all phosphate fertilizers on 2 Tennessee and 3 Virginia soils = 100.)

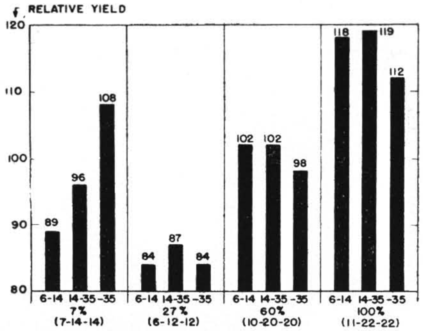

Figure 2. Effect of Granule Size and Percent Water-Soluble P on Mean Relative Yields of Wheat Forage in Mississippi.
(Mean yield from all phosphate fertilizers in 4 field experiments = 100.)

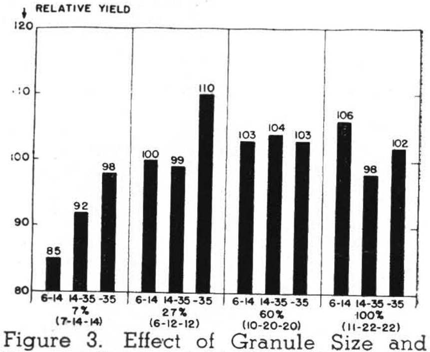

Figure 3. Effect of Granule Size and Percent Water-Soluble P on Mean Relative Yields of Vegetable Crops in Washington.
(Mean yield from all phosphate fertilizers in 5 experiments = 100).

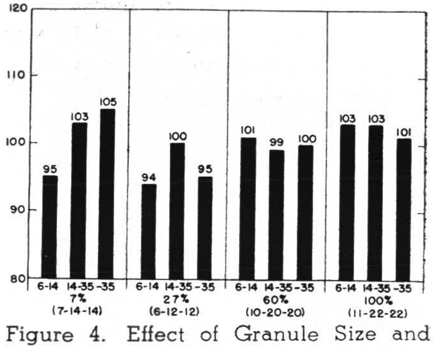

Figure 4. Effect of Granule Size and Percent Water-Soluble P on Mean Relative Yields of Corn Grain and Seed Cotton in Georgia, Kentucky, and Tennessee.
(Mean yield from all phosphate fertilizers in 6 corn and 1 cotton experiments = 100).

and water solubility will vary among soils, and data are not available to define this relationship exactly. The strong interaction found in many experiments between granule size and water solubility indicates that at some point in the range of water solubility between about 25 to 50 percent, granule size may not be important.

5. Broadcast application of finely divided phosphates is frequently as good or better on calcareous soils than banding. Similarly, there appears to be less advantage in regard to availability of the phosphorus of granulating phosphates for use on calcareous soils than for use on acid soils.

Why Do We Have So Many Nitrogen Solutions?

H. H. Tucker

THE question given for me to help discuss is "Why do we have so many ammoniating solutions? What is a practical basis for selection? Would it be possible to standardize the nomenclature for these various solutions?"

This in reality is not one but three separate and distinct questions on the one general subject of solutions.

Why do we have so many ammoniating solutions? Probably for the same reason that fertilizer manufacturers have so many grades. And it is the fertilizer manufacturer's need for different grades that largely determines the need for a variety of solutions.

Fertilizers can be divided into two general classes, dry and liquid. The requirements of these two classes are almost completely opposite. Dry fertilizers require solutions which supply nitrogen materials that will salt out and stay dry after manufacture. With liquids exactly the opposite is preferred.

Fertilizer can be further divided into high, medium, and low nitrogen grades. Since there are limits as to the amount of ammonia that will react with phosphates, each of these categories might logically use a different ratio of nitrogen salts to free ammonia.

Fertilizer manufacturing equipment varies as to type and efficiency to the point where the same solution is not universally best for any one given grade.

Handling problems due to temperature preclude the use of some highly desirable solutions at certain times of the year.

In general, there are three types of ammoniating solutions. Those containing urea, ammonium nitrate, and a combination of both urea and ammonium nitrate. This further multiplies the number of solutions available for use. Fertilizer manufacturers have their own convictions based upon their experience as to which type of solution best meets their needs. Ability to use particular nitrogen solutions may give some fertilizer manufacturers definite competitive advantages in manufacture and in sales.

This is part of the answer as to why we have so many nitrogen solutions.

What is a practical basis for selection of a nitrogen solution? As previously stated, fertilizers may be classified into three groups — high, medium, and low nitrogen. Each group needs a matching solution as far as the ratio of salts to ammonia or the fixed-to-free ratio is concerned. The need for water in manufacturing and facilities for removing excesses will determine whether the normal or the concentrated solutions are preferred. Salting out temperatures may make some solutions desirable in summer that are not desirable in winter.

These points must all be considered in selecting solutions and show the need for a variety of solutions. Preferences for ammonium nitrate, urea, or combinations of salts in solutions will be based on many factors. In general, urea in solutions costs more than ammonium nitrate. Many manufacturers feel its use is justified because of the qualities it gives to their complete fertilizers. Since it is impossible to handle a different solution for each manufacturing process and condition and each grade of fertilizer, the selection then becomes a compromise. The solution selected will likely be the one that most nearly fits the majority of requirements for the majority of the tonnage to be manufactured.

We at Sohio have tried to cut down on the number of solutions to those which we felt might serve the needs of the fertilizer industry. The number of ammoniating solutions as shown in the following table from Solution 1 to Solution 22 has been reduced to 12 in number and are classified as to type of solution, that is, ammonium nitrate or urea or ammonium nitrate and urea solutions. They are further classified as to the ratio of nitrogen from salts to nitrogen from ammonia, that is to say, fixed to free nitrogen ratio.

It would seem that further simplification might be brought about by having either Solution IX or 7, since perhaps both should not be required.

Solutions 3 and 3X are no longer in our product line because they have very high salting out temperatures and have been largely replaced by Solutions 11 and 16. To fill in this table it would seem that perhaps Solution 13 might be a logical and worthwhile addition, giving a solution with a low fixed-to-free ratio and one containing a combination of both salts.

The low, medium, and high fixed-to-free ratios would logically go along with low, medium, and high nitrogen grades. The very high nitrogen ratio solutions fill a need for high nitrogen granulated materials with adequate drying where it is desired to obtain particularly high quantities of nitrogen from solutions.

The newer concentrated ammonium nitrate solutions fall only in the medium, high, and very high fixed-to-free ratios. The low fixed-to free ratio Solution No. 4 already wa a very concentrated material wit only 6% of water.

Urea solutions for dry fertilize manufacture fall only in the low an medium fixed-to-free ratio group With higher fixed-to-free ratios i this group salting out temperatur become excessive.

The ammoniating solutions co taining both urea and ammoniu nitrate are limited to those contai ing large amounts of ammoniu nitrate to urea. Solutions containi large amounts of urea to ammoniu nitrate in general have high salti out temperatures or must conta large amounts of water which give low nitrogen contents.

Urea is added to ammonium nitrate-ammonia solutions to reduce salting out temperatures, to increase the volume of liquid phase at granulation temperatures, and to condition the products made from this solution. Twenty to forty pounds of urea per ton of end product will change the crystal structure of ammonium chloride without excessively increasing its solubility or hygroscopic properties. Increasing solution phase at granulation temperatures permits granulation with a minimum of water.

Solution 11 containing 11% of urea is formulated so as to use up to 350 pounds of Solution per ton of end product while Solution 16 with 6% of urea may be used up to 650 pounds of solution per ton of fertilizer without exceeding the limit of urea that can be used with ammonium nitrate.

Would it be possible to standardize the nomenclature of these various solutions? In general I believe we all agree that there is need for standardization. This no doubt would be possible and will probably have to be the result of a very careful study of the entire problem by a standardization committee.

There is a lot of confusion on this subject. This confusion has increased in the past two or three years. Three years ago, there were only a half dozen or so solutions producers with only a dozen or so solutions. A paper recently presented by Dr. J. Richard Adams of the USDA to the American Fertilizer Control Officials on October 18, 1957, shows 22 producers supplying 38 ammoniating solutions. Also listed are some 15 additional nitrogen solutions containing no free ammonia. These are used primarily for direct application to the soil or to increase the nitrogen content of liquid fertilizers. In addition to these nitrogen solutions anhydrous ammonia and varying strengths of aqua ammonia are available.

Insofar as nomenclature is concerned, companies of course use their trade names. This is good in that trade names establish the source of supply. Beyond this point confusion is rampant. The solutions are designated by numbers or letters or both. Some identical or near identical solutions may be referred to by as many as 10 different numbers or letters or combinations of numbers and letters.

Greatest confusion in nomenclature seems to exist in the ammonium nitrate-ammonia-water solutions. Perhaps this is because more companies are making these solutions and perhaps because these solutions are more universally used in the production of both conventional and granular fertilizers.

Two years ago the majority of solution manufacturers were using the numbers 1, 2, 3, and 4 for the four ammoniating solutions containing ammonium nitrate. These numbers were readily translated to or from letters such as A, B, C, and D. The more recent rash of concentrated and new solutions has largely obscured the uniformity that then seemed to exist.

The area of greatest uniformity in numbering two years ago and yet today is in the four component, ammonia-ammonium nitrate-urea-water solutions. These are all numbered in

Table 1. Classification of Sohiogen Ammoniating Solutions

Solutions Classification		Ammonium Nitrate			Urea	Ammonium Nitrate & Urea	
Fixed to Free Nitrogen ratios*	Characteristics	Regular	Concentrated				
	Solution No.	4	——		21	13	
Low	%N	49.0			45.4	49.0	
	Fixed to Free Ratio	.75 to 1			.5 to 1	.75 to 1	
	Salting-out temp. °F	—52			+16	—13	
	H_2O%	6.0			30.7	8.8	
	Solution No.	2	2X		22	15	
Medium	%N	41.0	47.0		45.2	44.0	
	Fixed to Free Ratio	.90 to 1	.92 to 1		.8 to 1	.91 to 1	
	Salting-out Temp. °F.	—25	—30		+46	—1	
	H_2O%	18.2	5.8		26.3	17.0	
	Solution No.	1	1X	7		10	
High	%N	41.0	44.0	45.0		44.4	
	Fixed to Free Ratio	1.25 to 1	1.25 to 1	1.18 to 1	——	1.16 to 1	
	Salting-out Temp. °F	+21	+26	+5		—20	
	H_2O%	12.8	6.4	5.9		10	
	Solution No.	3**	3X**			11	16
Very High	%N	37.0	41.4		——	41.0	41.4
	Fixed to Free Ratio	1.65 to 1	1.65 to 1			1.63 to 1	1.65 to 1
	Salting-out Temp. °F.	48	64			+7	35
	H_2O%	16.6	7.0			12.0	9.4

* Ratio of Nitrogen from Salts to nitrogen from neutralizing ammonia.
** Solutions recently replaced by Sohiogen 11 and Sohiogen 16.

the teens—that is between 10 and 19.

Because of this and the fact that most of the ammonia-ammonium nitrate solutions were numbered under 10, we at Sohio assigned the twenties to the ammonia-urea-water solutions. The solutions of two salts and water were numbered in the thirties since Solution 32 was the lone example of this type of solution.

Sohio certainly does not claim to have a perfect numbering system by any means and we have already had occasion to seriously question certain aspects of it. I have outlined some of our thinking. I am sure other companies have had equally adequate reasons for the systems they have used.

I certainly hope that something is done to standardize nomenclature of nitrogen solutions. Perhaps this is a task which should be assigned to an industry committee working through this Round Table or some other organization. Where there is a need there is usually an answer to this need.

Standardization of Superphosphates Is Desirable as to: (a) Fineness; (b) Moisture Content for Granulating

C. E. Floyd

WE believe that the superphosphate particle size should be somewhat less than the maximum size of the finished product of the complete grade which we are attempting to granulate. Taking for granted that we are using a coarse potash in the formula and that these small granules of potash are serving as seed for the soluble salts and ammoniated phosphates to build upon so that in theory as well as fact, each granule of finished product would to some degree be a homogeneous mixture, then it is obvious that the larger particle sizes of superphosphate would turn out to be partially ammoniated phosphates and the resulting product would be a mechanical mixture rather than an agglomerate. While we can do a fair job of granulating such grades as 12-12-12 using fine potash, we do not believe that the potash is soluble enough to be absorbed to any great extent by the larger particles of superphosphate and therefore, our product would not be as homogenous as desired. In general, we would say that the optimum size particles of superphosphate should be no more than half the optimum size of the finished product granules.

We believe that the moisture content of superphosphate should range from no less than 5% to more than 9%. Since water is essential to chemical reaction, we do not feel that too dry a superphosphate will take up ammonia efficiently. As you all know, each grade of fertilizer is an individual problem insofar as satisfactory granulation is concerned; however, in general, the less added water we have to use, the better; which is all the more reason that we should have enough water in our materials to serve as an aid to the reactions which take place.

In summary, we would say that it is very desirable to standardize our superphosphate as to fineness, as well as moisture content, when used in the process of granulation.

W. L. Hill

The best answer to this question that I can think of runs about like this: Superphosphate should be neither too fine nor too coarse and it should also have a moisture content less than some necessary minimum, for the efficient operation of some particular granulation process (laughter) on a given feed in the particular facility that happens to be at hand (laughter).

Now this statement merely means that the specifications for superphosphate in technical use lie in three-pronged dependence on the modus operandi—the mode, the operand and the apparatus. Thus, general specifications ought not be attempted. Now, truly, you're all going to put ammonia into the fertilizer mixture, which introduces another consideration. You have ammonia-absorption rate and granulative water demand all mixed up together and going hand in hand. Now, I think that fineness governs the other two; hence, we can talk with profit on the fineness of fertilizers. In particular, we can talk about fineness of superphosphate. I put down on paper some things that are basic to appreciation of this intrinsic property. Copies are in the rear of the hall. You may want to take one to read at home. At the moment I shall console myself by making a few comments on the nature of pile-cured superphosphate.

Now if you take a crumb of superphosphate (or triple superphosphate) and look at it rather closely, you will perhaps notice an aggregate of smaller crumbs. Look at the smaller crumbs, using a microscope, and you will see that there are holes in them and that they are often made up of individual grains. The grain is the thing I wish to talk about. Now, you have the crumb size and the grain size. Some of my friends tell me that those people down there will not appreciate what you're saying when you talk about grain size but they will understand porosity. But, since porosity doesn't have very much to do with grain size, I'm going to stick to my guns and talk about grain size as distinguished from crumb size. Why are these things important? Well, the crumb size is determined over there on the shipping end by the mesh of the sizing screen, whereas the grain size is determined and controlled by conditions that occur in the acidulator and the den. The terms describe different kinds of fineness. There are different types of crumb. If you survey various and sundry superphosphates by examining crumbs, you will find one type of crumb that is a loosely bound aggregate of smaller crumbs and grains. The outside is very irregular, made so by grains sticking out and pores reaching inward. In supers that have been stirred considerably during the plastic stage you will find crumbs that have been compacted at the surface often to the point of being practically impervious. You can find stil other crumbs that have been compacted throughout until they've become a giant nonporous grain. These are the three extreme types. If you grind the intermediate type you get a very nice response in ammoniation rat and perhaps also in granulation. I you grind the other two you won't find too much response.

Now, this compacting occurs in manufacture as a result of stirring the acidulate when it's in the plastic stage and as a result of the curing process where you don't get sensibly complete conversion in the den, say only 80%. You lay it out on the pile where the continued reactions deposit salt that fill up the pore spaces. Some of these compacting processes you do deliberately in the granulation step, out they also occur incidentally in the manufacturing step to a greater or less- extent and thus modify the character of the product.

Now to finish, I should like to comment that triple superphosphate is likely to have a coarser grain size than ordinary superphosphate. Why? Well, ordinary superphosphate is mainly calcium sulphate with monocalcium phosphate as cement holding the calcium sulphate grains together. The phosphate lies all around on the surface of the sulfate, so that the affective grain size is smaller than in triple super, where monocalcium phosphate grains are cemented together with some more monocalcium phosphate. Despite all this you can make triple superphosphate that is too one for best work and how would you do that? Well, I can only theorize. After all, we're dreamers, you know. And one way to do it is to use a rock that is inherently very reactive, grind it very fine, whip a lot of air into it, mix it with strong phosphoric acid, preferably warm, and mix as quickly as possible without prolonged stirring. The latter condition can be met by using a cone mixer, from which the rapidly blended acidulate drops to a belt, hooves up, sets, and presently breaks off the belt under its own weight. Thus, you could have a triple superphosphate with very fine grains and highly porous. I'm not saying that that would give it, but that's the direction you would have to move in order to get it. Suppose you had such an extremely fine-grained triple superphosphate. What trouble might you have with it? You would have something that would grab up ammonia at a great rate, but then you would find when you put it into your granulation process that the granulative water demand is so high that you couldn't apply enough water with the equipment at hand. You would have to back up toward coarser material. The moral of the whole thing is that when you get ready to write specifications, you're going to need to compromise by having a material that is fine enough to give you a reasonable good rate of ammonia absorption and at the same time not so fine as to require more water than can be supplied by the delivery system or disposed of by the drying equipment.

Dr. Sauchelli: Thank you, Mr. Hill. I don't know personally of any greater student of phosphates and all phases of phosphates than Mr. Hill, and I think he has given us a very fine exposition in answer to this question. We have time for one or two brief comments; who is willing to make them?

Question: I was wondering about this binding of phosphates. Our people here use granular potash and hope to coat it with phosphate. How would the finer phosphate adhere? Would it make any difference, finer adhere better?

Mr. Hill: I think I follow what you're driving at. If you want to wrap it around the potash you would have to put enough water in it to make it plastic and then it will wrap around, but if you've got a little grain of sand, you'll just have to stick it on because you can never make it plastic. So I think after all that the crumb size is less important than the grain size, but if you have a material that is fine grained, you perhaps can compact it so that you get the crumb size the way you would like to have it. However, you have to do this job in the acidulator and the den, you cannot do it with the sizing screen.

Dr. Sauchelli: Well, we've had two good comments on that—Mr. Floyd and Mr. Hill, an industry man and a research man.

Discussion on Relative Humidity and Moisture

Benjamin Makower

The remarks of the preceding speaker might be amplified by referring to a graph of relative humidity vs. moisture content. We can take as an example a graph for commercial sodium nitrate as given in the book (Fertilizer Technology and Resources, Acadamic Press Inc., N.Y., 1953). (See Fig. 1.)

The curve plotted in this graph shows the variation of moisture content of this fertilizer with relative humidity of the surrounding air. The higher the relative humidity, the more moisture will be taken up from the air. At higher than approximately 50% relative humidity, which is called the "critical humidity," the curve becomes very steep. This steepness signifies that a liquid phase is formed in the product. Enough moisture is present to convert some of the solid sodium nitrate into a water solution. From the curve we can see that the steep portion starts out at approximately 0.5% moisture.

We can say, therefore, that if we wish to keep the amount of the liquid phase in sodium nitrate at a desired low level, the moisture content should not exceed about 0.5% and the material should not be stored at relative humidities higher than about 50%. It is generally recognized that the less liquid phase the fertilizer contains, the better it stores and handles.

For other fertilizers, the "critical relative humidity" will be different and must be determined experimentally with each material. Considerable useful information of this type has already been accumulated and is presented in the book by K. D. Jacob cited above.

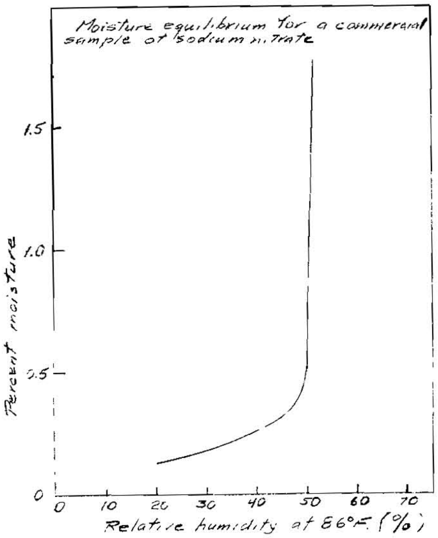

FIGURE 1

What is the Optimal Mesh Size of Muriate of Potash for (a) Granulation, (b) Blending with Other Coarse Materials, (c) Direct Application as a Fertilizer

J. O. Hardesty

(a) The reply to the first portion of this question was covered under Question 5.

(b) In producing a mixture of individual granular materials the granular potassium chloride preferably should be of the same particle size as the other materials. However, this does not eliminate the possibility of segregation since other factors, such as particle shape and density, influence the degree of segregation. Neither does it eliminate the possibility of chemical reaction with other ingredients of the mixture during storage.

(c) Considering the ease of handling in distribution equipment, the optimal size of potassium chloride for direct application is probably in the range of 6 to 20-mesh (3.327-0.833 mm. screen opening). The selection of optimal-size fractions within this range will likely depend on rate and method of application.

A count of the granules per unit weight of 6-mesh material shows that there are approximately 9626 granules per pound. On the basis of the relative volumes of different sized granules, the following tabulation shows the approximate number of variously sized granules of potassium chloride per square foot, when applied at the rate of 167 pounds per acre (43,560 sq. ft.).

Screen		Granules/sq. ft. Rate, 167 lbs./acre
Mesh	Opening	
	m.m.	
6	3.327	37
8	2.362	103
10	1.651	301
14	1.168	897
20	0.833	2342

R. D. Tayloe

Potash is currently available in three sizes, granular, coarse and standard. These fall more or less in the following ranges, 6 to 14 mesh, 10 - 28 mesh and minus 28 mesh. These are approximate as each manufacturer works in a slightly different range. The standard grade exhibits the widest variation, some products being almost all plus 50 mesh and others being less than 50% plus 50 mesh.

There are four general uses for potash; side-dressing, plow-down, blending and mixing. By side-dressing I mean to a growing crop. Here the granular material is required, so the potash will fall to the ground and not damage the growing plant.

Material for plow-down is put on broadcast prior to plowing; for this purpose the finer grades do better as better distribution can be obtained.

Blending refers to the mixing of dry materials to make such grades as 14-0-14 or 0-20-20. The materials used are generally granular and a large part of this tonnage is bulk-spread. Care should be taken to prevent segregation, and I would suggest a potash the same size or just a hair finer than the other materials.

Mixing to me implies ammoniation, with or without granulation and for the most part sold in bags. The tonnage here outweighs the other uses combined by many times.

For mixing without granulation the standard potash should be used. Perhaps one of you may care to comment on this when I am through.

When granulation is involved the current trend is toward the use of coarse potash as a seeding agent in grades high in potash. Because of several factors coarse potash is not much used in 1-1-1 ratios, but is in 1-4-4's. Since these are two leading ratios the manufacturer should have two potash bins, using standard as much as possible with coarse available as needed to granulate high potash grades.

This is not the official sales policy of my company nor, as far as I know, any other potash company. It is my suggestion, based on the facts that (1) there is more standard available, (2) it is cheaper and (3) it gives a more homogenous product.

There is not a great deal of data available on the effect of potash particle size. Mr. Phillips presented the results of some work by the TVA at the recent ACS meeting, and I have seen some data from a few manufacturers. They all find that increasing the potash particle sizes increases the yield of on-size product. The homogeneity of the product is bound to be affected, however, as the lower size limit is raised. Mr. Hardesty at the Round Table last year gave 10 mesh as about the upper limit for good granulating practice and as I recall suggested about 30 mesh as the lower limit, which permits the inclusion of potash coated with ammoniated super in the entire product size range.

I would say that more data are needed on the subject, with particular emphasis on the homogeneity of the product and the potentiality of segregation. In closing, let me leave this thought: blend coarse and standard for use as needed, using the highest proportion of standard consistent with good operation rather than going for the maximum yield of on-size product at the first pass.

Results of Tests Using Potash of Different Particle Size In Granulating 5-20-20

T. J. Bosman

The data in this paper were obtained from test runs at the Federal Chemical Company, Danville, Illino Plant. The work was under the direc tion of Dr. Ed. C. Kapusta and myself. It was a follow-up to previous work done by Dr. Kapusta at Kingsbury & Company and the Buhner Fertilizer Works. My results are very much in line with the work done in these previous tests.

Four runs were made. On runs #1, #2, and #3, three samples were taken at the granulator, dryer-cooler, recycle belt and finished product belt respectively. The first was taken one half hour after getting on stream, the other two at one half hour intervals from the first. On run four only one set of samples was taken after being on stream one half hour as all storage space was full and we had to stop batching. This gave us 16 samples on which screen tests and the analyses of each cut were made, in addition to the screen test on raw materials. In some of the tables we have condensed these results by taking weighted averages of all samples from each source in the same run.

From the data obtained I sum up the results as follows: With the coarse potash less recycle is formed thereby cutting down on the quanity of acid and water needed for granulation. However, the coarse potash tends to greater variations in analyses. You can pick separate particles of this coarse potash adhering to the other granules.

The fine potash makes more recycle, thereby causing the use of increased quantities of water and acid. The analyses are slightly more uniform but still leave a lot to be desired. You are not able to distinguish the potash from the other granules in the finished product as readily as with the coarse.

On both days these tests ran about as ideal as a 5-20-20 granulation could. The quantity of water was the lowest we have ever been able to use and get granulation. Most of the time we have had to bypass our instrument to get enough water for granulation. On a 30-ton per hour basis this would be over 166# water per ton. How much over, it is hard to estimate.

Once, before we had a pump put on our water line, Dr. Kapusta and I had to postpone our test because we couldn't get enough water for granulation using the coarse potash, even though we were using considerably more than we used in any of the runs given in this paper.

This follows much in line with other work done by Dr. Kapusta. At one place he had to use 180# H_2SO_4 and from 258# to 363# of water per ton to get granulation.

Albert Spillman

I wish to state that I am not an advocate for using coarse potash. I hope some day through more research we will find the answers, possibly through some additive material or some changes in our equipment to granulate low granular mixtures without the use of coarse potash. But until that information is found, I don't believe we have any other recourse to keep operating except to use coarse potash in some of our low nitrogen, high potash mixtures.

In most formulas of our high nitrogen granular mixtures, we had sufficient soluble salts to enable good production of on-size granule recovery enabling our holding down to a minimum the recycling of fines. Using the standard minus 35 mesh fine potash in these mixtures is no problem. Our product recovery before screen classification minus 5 mesh plus 20 mesh will average around 65 to 80 per cent. In our particle installation at Baltimore, this is a good recovery and keeps us operating on a full-plant scale.

Our low nitrogen granular formulas are difficult to produce because of insufficient soluble salts. Using the standard minus 35 mesh fine potash in these mixtures we get a low recovery average of 45 to 50 per cent on-size product before screen classification. Coarse potash used in mixtures difficult to granulate with fine potash is very helpful to produce sufficient on-size product recovery to permit good plant operating conditions. We use coarse potash in our granular low nitrogen mixtures 6-12-12, 6-12-18, and 6-18-18. When using the fine potash in these mixtures, we obtain a product recovery minus 5 mesh plus 20 mesh of approximately 45 to 50 per cent compared to recovery on-size 65 to 80 per cent when we use the coarse potash.

Using coarse potash is very tempting from an operator's standpoint and a real utopia to keep production of granulation up to a maximum capacity of our grading equipment. However, we must be careful not to use coarse potash in formulating mixtures if there is the slightest chance regular fine potash can do the job. For the present, until other means of equipment of formulation is found to granulate difficult low nitrogen formulas, I can see no alternative to using coarse potash.

To enable more uniform distribution of potash into all the range of particle sizes of the complete mixture, best results can be obtained when using the minus 35 mesh potash. For difficult mixtures to granulate where coarse potash is used, I recommend using potash particle size with a minimum plus ten mesh or if it can be obtained, all through a ten mesh if obtainable and a minimum of finer than 35 mesh 85 to 95 per cent. In other words, I feel that if we could get a coarse potash one hundred per cent minus ten mesh and about 85 to 95 on a 35 mesh screen, I think we can do a fair job and still hold down the possible least segregation.

Careful experiments made by us on plant production scale conform with the findings of experimental work reported by John Hardesty, USDA. Analyzing mixtures containing coarse potash by fractions show five and ten mesh size carry a high potash content and 20 mesh size carry a low potash content. However, the complete mixtures show the potash to grade is guaranteed. In order to do some testing at our plant we ran two separate tests on two types of coarse potash. Grade No. 1 of coarse potash used had 7 per cent on a ten mesh, 74 per cent on a 20 mesh, 11 per cent on a 24 mesh, six per cent on a 35 mesh, or 98 per cent on a 35 mesh. The second grade of coarse potash used had 4 per cent on ten mesh, 52 per cent on 20 mesh, 18 per cent on 24 mesh, 22 per cent on 35 mesh, or a total of 96 per cent on a 35 mesh. The two mixtures made were 6-18-18 and 6-12-12 granular. We ran two different tests on screen fraction size five mesh, ten mesh, 20 mesh, and also a composite sample to see which of the two grades of potash did the best job. In our 6-18-18 five mesh particle size on one sample we had 21.40 per cent for 18 per cent. On the second sample we had 20.9

per cent. On the ten mesh particle size, the first test showed 19.6 per cent. The second test 19.42. On the 20 mesh size, the first test showed 16.55 per cent; second test, 16.4 per cent. On a composite, the first test showed 18.24 per cent and the second test 18.06 per cent.

This information is on the first coarse potash used with a larger percentage of the ten mesh. Using the coarse potash with four per cent on a ten mesh on a 6-18-18 on two results, on the five mesh we showed 19.32 per cent potash on the first result. On the second result 19.15 per cent. On the ten mesh fraction we had 18.70 and 18.58.

On the twenty mesh we had 17.5 and 17.32. And on a composite we showed 17.98 and 18.05. On the 6-12-12 using the coarser potash, on the five mesh fraction we had 13.95 and 13.88. On the ten mesh fraction 13.20 and 13.42. On the twenty mesh fraction 11.4 and 10.95. And on a composite sample 12.22 and 12.15.

Using the coarse potash practically all through the ten mesh screen, on the five mesh fraction, we had 13.65 and 13.15. On the ten mesh, 12.95 and 13.04. On the twenty mesh 11.34 and 11.20. And on a composite 11.98 and 12.05.

We had about ten per cent more recovery of on-size product using the coarser potash than we did with the coarse potash practically all through a ten mesh. However, we had better potash analysis with the No. 2 grade potash carrying a bigger spread of particle size between ten and thirty-five mesh.

Recovery of product before recycle. On the very coarse potash we had about ten per cent more recovery than we did with the finer coarse potash. On a 6-18-18 we had 81 per cent and 78 per cent recovery, that's through a five mesh on a twenty, using the No. 1 grade. And using the No. 2 grade potash we had a 70 per cent and a 72 per cent recovery on a 6-18-18. On a 6-12-12 using the very coarse potash, we had a 76 and a 73 per cent recovery against the finer coarse potash of 69 per cent and 67 per cent.

I can't emphasize too strongly the importance of an operator doing a little fussing in his plant. There are times, I know we have a lot of difficulty with our operators. After all, they're told to produce tonnage and if you don't get the tonnage you just can't do a job. And sometimes I'm afraid we use coarse potash where we could get by with a little fine potash. So my recommendation is to do a little more fighting in the plant and maybe we can get this coarse potash down a little less than what we think we need.

If there are any questions, I'll be glad to answer them. Vince, what I'm saying is that there are a number of different types of potash on the market today—here's an illustration. When we first decided to use coarse potash, I made a survey for our company. I had about fifteen different samples of potash, and it just scares you to death. I'll just read a few of them off here to you. On-size on a ten mesh, the range was anywhere from two per cent on a ten mesh, some of the samples were as much as 52 per cent on a ten mesh. I'm just scared. I think a lot of us fellows may know what we're doing in a big plant, but there are a lot of small mixers who are ordering coarse potash and I'm really afraid if they don't know, if somebody doesn't just caution them on the type of coarse potash to use, they're going to get themselves in a lot of difficulty from segregation. So my recommendations are from what I've been able to find out, if we're going to use coarse potash, let's get it as much as possible through a ten mesh, a hundred per cent would be my way of thinking.

Mr. Sauchelli: Thank you, Albert. It just shows you this question is very important. That's why we've been willing to give the time this afternoon to this discussion, and I think we need more of it . . . more plant men should be interested and checking for themselves. I know the potash companies all are investigating it and the trend, as I said previously, is toward producing potash to specification as to mesh sizes, and that's all to the good.

J. G. MacArthur

The question as originally stated was, "Do you have any experimental data on the relationship of potash to particle size, on-size product before screening, recycle load or homogeneity of the granular fertilizer?". Although in no way complete, some information was obtained during startup operation of a commercial fertilizer plant which demonstrated, to a degree, the effect of varying the quantity of coarse potash and filler in the production of various fertilizer grades.

Figure 1 is a picture of four different fertilizers, and shows visually the degradation in particle size as the amount of coarse potash used in a formulation is decreased. Reading from left to right materials are:

Beaker 1; an 8-8-8 manufactured with fine potash and is included for comparative purposes.
Beaker 2; contains 3-9-27.
Beaker 3; 3-9-18.
Beaker 4; 4-12-4.

All materials are finished product and were taken as a 6/20 mesh cut from the process stream. The degradation in particle size, as the amount of coarse potash used is decreased, is apparent.

Figure 2 shows formulation and some operating data for the various fertilizer grades. Commenting on this briefly, the 8-8-8, even though it contained 500 pounds of filler, had sufficient heat-furnishing reactions and soluble binding agent present to adequately agglomerate; in fact, the throughput percentage of 162 indicates that it was necessary to bring back some recycle as a granulation control. This condition did not exist in the other three grades.

In case of 3-9-27; 3-9-18, and 4-12-4, it was found necessary to add extraneous heat to maintain the ammoniator temperature in the neighborhood of 200°F. This was accomplished by feeding live steam beneath the bed of fertilizer. It is worthy of note that as the amount of coarse potash added decreased, the demand for steam and total moisture increased. It is also worthy of note that although 3-9-27 and 3-9-18 contained over 1,000 pounds of material normally considered difficult in granulate, namely potassium chloride and dolomite, and the 4-12-4 contained only slightly over 700 pounds of these materials, the first two grades appear to have a larger particle size. This condition has been attributed to two factors; namely, the overall particle size of the raw materials initially fed to the system were larger, and the undersize particles did lend themselves as nuclei for fertilizer granules.

Figure 3 shows the difference in sieve analyses for the various raw materials and mixed fertilizers with the following amount of each raw material being held on a 20 mesh sieve:

Fine Potash	18.6%
Dolomite	44.6%
Coarse Potash	82.6%

Analysis of the materials exit the cooler, that is before separation and grinding of the oversize and separation of the recycle, shows little indication for particle size degradation in the first three grades. However, the 4-12-4 shows a 20% decrease in the amount of material held on the 20 mesh when compared to a 3-9-18 or 3-9-27. Finished product analysis shows a general degradation of the last three grades.

Figure 4 is a photomicrograph showing a nucleation of fertilizer particles by coarse potash and other materials. The granule in the lower part of the picture shows quite plainly a crystal of pure potash in the center. Close examination shows other smaller particles of potash scattered throughout the matrix, around the outside. Apparently, concentration of a large quantity of the potash carrying materials in the center of the particle left enough matrix and soluble materials to effectively bind the remaining insoluble material into the granule. The particle directly above that containing the potassium chloride crystal had a nucleus of a dolomitic limestone, whereas the third particle, which by the way is not very visible in this picture, has a nucleus of superphosphate. Figure 5 is another photomicrograph of a fertilizer grade, this time 4-12-24 manufactured with fine potash. This material was manufactured in a small bench-scale fertilizer unit and although very few fines were obtained exit the ammoniated granulator, a small amount of handling caused appreciable breakdown of the particles. This grade was not demonstrated on a commercial basis; however, attempts to granulate a 3-9-18 using fine potash met with failure due to extreme dusting and inability to consume the large amount of recycle sent back to the ammoniator. It should be pointed out that the filler can also be critical in manufacture and in the case of one grade, namely 3-9-18, when a silica type filler was substituted for the dolomitic limestone, satisfactory granulation was not achieved.

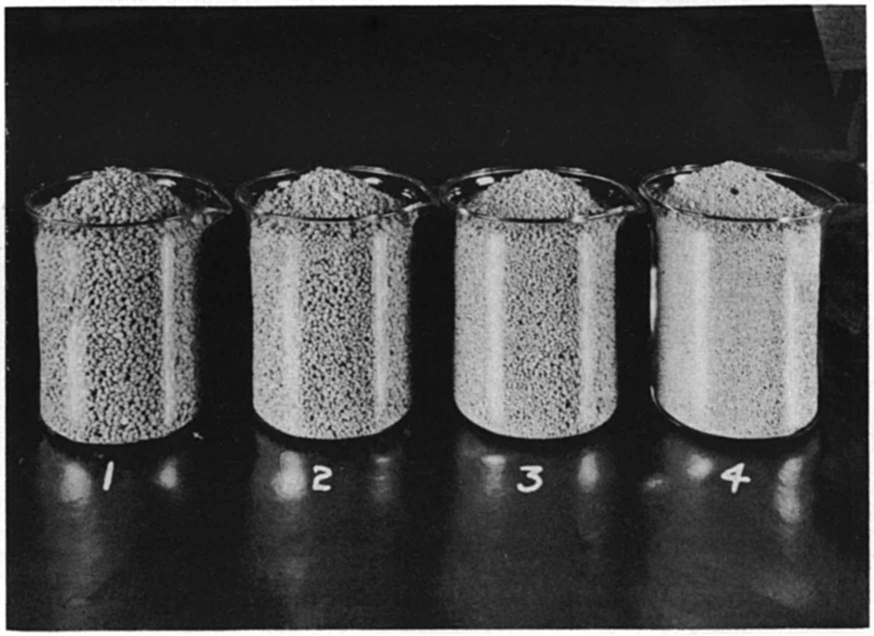

FIGURE I

FIGURE II

GRADE	8-8-8	3-9-27	3-9-18	4-12-4
Formulation #/ton				
Ammoniating Solution 37%	433	108	109	162
Anhydrous Ammonia 82%	—	25	25	25
Sulfuric Acid	98	—	—	—
Superphosphate	800	915	900	1200
Potash (Fine)	267	—	—	—
Potash (Coarse)	—	900	600	133
Filler	500	110	412	575
Total	2098	2058	2046	2095
Production T/hr.	12	15	12	10
Recycle T/hr.	7½	2¼	3	¾
Throughput % of Production	162	115	125	108
Ammoniator Temp. °F.	195	205	211	209
Steam Feed #/ton	—	107	133	160
Water Feed #/ ton	—	83	73	88
Total Added #/ton	—	190	206	248

FIGURE III

	Dolomite	Potash (Coarse)	Potash (Fine)	8-8-8	3-9-27	3-9-18	4-12-4
Held on					*Cooler Exit*		
6	0.8	0.0	0.0	21.9	19.5	16.0	2.3
14	27.2	31.5	0.5	82.9	79.0	83.5	63.5
20	44.6	82.6	18.6	94.1	95.5	97.5	90.4
40	62.2	96.1	82.9	99.5	99.5		
65	89.0	98.8	97.2				
Held on					*Finished Product*		
6				5.4	5.5	0.0	0.0
14				70.8	76.0	73.2	29.6
18				98.3	98.0	88.7	42.1
20				100.0	99.5	99.9	97.0
40							99.9

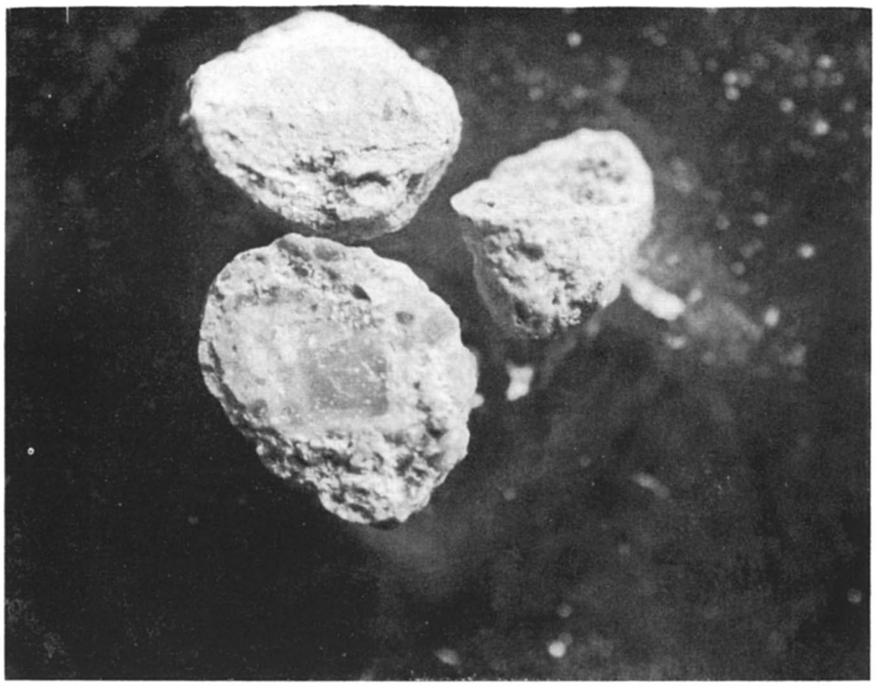

FIGURE IV

FIGURE V

J. O. Hardesty

Answer:

Experimental data, as well as data from commercial- and pilot-plant practice, indicate that the optimal particle size of potassium chloride for obtaining high product yields of granular mixed fertilizers is not the same as that for obtaining uniform nutrient distribution in the product. These data are available in the literature cited at the end of this paper, and indicate that:

(a) Solid potassium chloride tends to resist agglomeration with itself (*11*) and with other ingredients of the fertilizer mixture (3), *12*, *18*).

(b) The resistance to agglomeration is most apparent in the low-nitrogen, high-potash grades (3), *12*).

(c) As would be expected, an increase in the proportion of granular potassium chloride in the initial mixture, especially if it is the same particle size as that of the granulated product, increases the yield of granulated product and reduces the yield of recycle fines (*12*, 18). A particle size of 8- to 14-mesh* potassium chloride may seem optimal for producing high product yields of low-nitrogen, high-potash grades but it does not contribute to homogeneity in the finished product.

(d) Distribution of plant nutrients among granules of the final granulated product becomes less uniform as the particle size of the initial materials approaches that of the final product (6, 10, 17). The result, — if the procedure is carried to its ultimate limit,—is a mixture of (a) granules of individual materials, in which no agglomeration has occurred, or (b) granules of crushed oversize, in which the low degree of agglomeration has resulted in poor distribution of plant nutrients. Either type of product is inferior to a granulated mixture in which a high degree of agglomeration has caused a wide distribution of initial ingredients among the granules of the product. Plant nutrient distribution is far more uniform in a granulated product made from materials passing a 40-mesh screen than in one made from materials of which an appreciable proportion is retained on a 20-mesh screen (10).

(e) A comparison of the relative volumes of spherical granules of equivalent density shows that, even though an 8-mesh particle of potassium chloride is uniformly coated with the other ingredients of a 5-20-20 mixture, it is not possible for the final granule to be up-to-grade in the 6- to 20-mesh product fraction It can only be up-to-grade in the oversize fraction.

The data in the references cited indicate that (a) all initial materials for use in granulation processes should be finely-divided, and (b) initial granular materials, when used as a production aid, preferably should include no material larger than 10 mesh in particle size.

General Comment:

The successful operation of a modern fertilizer mixing plant calls for professional skills and knowledge that previously were not necessary in making mixed fertilizers. The physical sciences are now being applied to the production of fertilizers. The questions presented here for discussion in the 1957 Round Table Conference are evidence of this transformation. Some of the questions refer to problems that have not been fully explored. They point to the need for further applied research in the field of fertilizer technology. As rapidly as the answers to these questions are obtained they will ordinarily appear in scientific, agricultural, and trade publications.

Plant personnel who are responsible for the production of mixed fertilizers usually find that a systematic inspection of the technical literature yields valuable assistance in solving many production problems.

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* Mesh size in all cases refers to the opening in Tyler Standard Screen Scale Sieves.

Edwin C. Kapusta

Plant experience has indicated the important role of potash particle size in the granulation of low nitrogen-high potash grades in this process. To obtain maximum production rates and reduce problems in plant operation, manufacturers have resorted to the use of granular potash in the production of these grades. Since little plant scale data was available as to the optimum potash particle size for this application, arrangements were made to conduct plant scale tests which might provide more information on the effect of potash particle size on granulation.

Plant scale tests on the effect of potash particle size on granulation of 5-20-20 fertilizer were carried out at the Buhner Fertilizer Company plant at Danville, Illinois. This plant employs the TVA continuous ammoniator process.

Three sizes of granular muriate of potash were studied in these tests. The coarsest was essentially a minus 6 plus 20 mesh product, the intermediate muriate was a minus 8 plus 20 mesh; and the smallest size muriate was essentially a -10/48 mesh product.

The 5-20-20 mixture was formulated from anhydrous ammonia as the sole source of nitrogen. Normal and triple superphosphates were used as the sources of phosphate. Sulfuric acid and water were added to promote agglomeration. The amount of water used was the sole variable. The water rate was adjusted to yield the best results for each type of potash used. Normal plant operating procedures were followed in these studies. The plant was operated at a capacity of 20 tons of product per hour.

All three sizes of granular muriates resulted in equally satisfactory plant operation. The amount of water required for granulation was about the same for each of the granular potash products tested. The recycle load was the same for all of the granular potash products studied.

The 5-20-20 products made with granular potash were angular and irregular in appearance and contained appreciable amounts of uncoated potash crystals and exposed potash surfaces. The use of the smallest size granular potash resulted in a product containing considerably less exposed potash than the use of the two larger sizes of granular muriate. On the basis of appearance, plant operators stated that the smallest size granular muriate was most suitable. All of the products made with granular potash were very similar in particle size distribution; approximately 70 per cent of the product was in the minus-6-plus-10 mesh range in each case. Each of the products made with granular potash contained approximately the same amount of moisture.

In regard to plant food distribution, the potash accumulated in the product particle size fractions approximating the size of the granular potash raw materials. In general, the potash content of the larger particle size fractions of the product was higher than the desired amount; whereas the smaller particle size fractions were deficient in potash. The phosphate content varied inversely with the potash content in the various screen fractions. The nitrogen was fairly evenly distributed throughout all of the product and recycle particle size fractions.

From the standpoint of plant food distribution throughout the product and recycle, the intermediate size granular potash proved to be most satisfactory. The largest particle size potash gave a product with an overabundance of potash in the larger particle size fractions and a deficiency of potash in the smaller screen fractions. The use of the smallest particle size granular muriate resulted in a product containing too little potash in the coarser particle size fractions and too much potash in the smaller screen size fractions.

What is an Ideal Size for Granules or Pellets: a) Agronomically, b) for Processing, c) for Handling in Fertilizer Distributors on the Farm?

Arthur M. Smith

Consumer preference is the most important thing about particle size. It is often influenced, and may even be created by advertising. But it won't last unless it is supported by sales appeal. Sales appeal results from appearance, feel and peformance.

Appearance is an important factor, and it is either positive or negative. It can vary with the composition and physical properties of the product as well as with particle size.

Feel is a rather intangible property. It is influenced, but usually not dominated by, particle size. Seldom of major importance as a favorable factor, feel can be of great importance as an adverse factor; and this can be entirely independent of any caustic, roughening, or burning effect on the skin of those who handle it.

To a farmer the most important factors in performance are:

1. That the fertilizer be free flowing;
2. That it be free of hard lumps and not caked;
3. That it be packaged so as to effectively minimize any hygroscopic properties of the product;
4. That top and side dressing mixtures and materials contain no dust to stick on the foliage.

Within reasonable limits uniformity in size means more to a farmer than what size the particles are. That is because with most fertilizer compositions uniformity of particle size gives a freer, more uniform flow. However, since no process, either spraying, rotating, or crystal growth, gives a product so uniform that it need not be screened, some acceptable criteria as to particle sizes and percentages thereof seem desirable. These should allow manufacturers reasonable flexibility so that recycling may be avoided as much as possible.

Based on my experience I suggest the following:

0-6 mesh — none
6-10 mesh — 30% maximum
10-20 mesh — 50% minimum
20-40 mesh — 20% maximum
finer than 40 mesh — none to 2% maximum.

It should be recognized that buyers' preferences as to particle size of fertilizers varies in different parts of the country according to crop, climate, type of application equipment used, etc. It also varies according to products. For example, granular cyanamide is fully accepted by farmers at an approximate uniformity of particle size much smaller than they will accept for either ammonium nitrate or nitrate of soda. Sulfate of ammonia for direct application sales has suffered more from its fine crystalline form than from tackiness, hygroscopicity or caking due to a slight free acid content. When made by the gypsum process, or when offered in granular or blocky crystal form, the particle size was usually satisfactory to the farmers.

Terms used to describe coarser particles include: granular, pelleted, pelletized, prilled, shotted, pebbled, and crystallized. Old style mixed fertilizers, even when "pilly" are usually described as pulverized or powdered. Size of particles *per se* is not always of first importance. The nature of the surface of the particles may be of equal or even greater importance; and so may be the shape of the particles whether round, oval, irregular, cubical, or angular and coarsely crystalline. The composition and density of the product usually determines whether the surface of the particles is hard or soft, smooth, rough, or pitted.

The importance of the surface characteristics of the particles increases as the particle size decreases.

In the past much importance has been attached to the angle of repose. It is a true index of the drillability of a fertilizer, and is the best single indicator of whether the particle size distribution of the product will be popular with farmers.

However, composition is always important and often is the cause of what was free-flowing fertilizer when it left the factory, showing up on some farms as caked, lumpy, or damp fertilizer having poor drillability.

Products in which the ingredients have approached a reaction equilibrium or by other means have been homogenized prior to aggregating into larger particles, usually give the least trouble after shipping. Mixtures of granular, or other coarse particle materials, which may react with each other, have to be selected carefully in formulating in order to avoid segregation on the one hand and hygroscopic, caking, and lumping effects on the other.

Free flowing are the two most important words in describing the physical characteristics of fertilizers. Whether producing pulverized fertilizers or products consisting of coarser particles, those two words are both the challenge and the measure of success or failure of the chemists, chemical engineers, plant superintendents, and managers who are responsible for production.

What are the Merits of any of Ammoniated Superphosphate and Ammonium-Phosphate Based Fertilizers.

U. S. Jones

In answer to this question of the merits, if any, of ammoniated superphosphate and ammonium phosphate based fertilizers, my remarks will be confined to the agronomic considerations. In answer, the following I, think, in a few words will cover it. The crop growth and yield response from ammonium phosphates and ammoniated superphosphates are closely related to the ease with which the fertilizer is dissolved in water. For many years plant nutrition experiments have been carried on to determine utilization of nitrogen, phosphorus, and potassium fertilizers by crops. Of the total amount of nitrogen fertilizer added to the soil, it is possible for 60 to 80 per cent of it to be taken up by the current crop. Of the total amount of potassium fertilizers added to the soil, it is possible for something on the order of 70 to 80 per cent of the potassium to be taken up by the crop. Most of the inorganic nitrogen and potassium salts used in fertilizers are completely soluble in water. Most of the phosphorus fertilizers are not completely water soluble and are not so efficiently used. It has been determined that not more than ten to fifteen per cent of the phosphorus added is taken up by the current crop. Drs. Olson and Dryer of the Kansas station recently concluded that a much greater recovery of fertilizer phosphorus by the current crop is possible than the ten to fifteen per cent or lower recovery commonly observed in the past. That data indicate that phosphorus fertilizer efficiency can be improved by proper placement, by nitrogen association with the phosphorus as well as the initial solubility. As ammonium phosphates and ammoniated superphosphates both have nitrogen chemically compounded into goods and they are both placed in the soil by the same equipment and in the same manner, the primary agronomic consideration then in the discussion of the merits of the two materials narrows down to the question of the initial water solubility of the two materials. Both are citrate soluble according to the chemical procedure approved by the AOAC. Ammonium phosphates of monocalcium phosphates are water soluble according to this method, Dicalcium phosphates are only slightly soluble in water, and tricalcium phosphates are not water soluble.

Variation in Density of Fertilizers Creates a Problem in Weight Control at Bagging Units. Is There a Practical Way to Counteract the Density Problem?

W. F. Jacobi

This problem has confronted manufacturers and shippers of a variety of products for some considerable time. Some manufacturers, especially those concerned with packaging small consumer units have found the answer to the problem. The method of approach used in the solution of the problem as it applied to small packages was basically the same in all instances and there is no reason why the same method should not be used in the case of larger unit packing operations. In fact the same system has been and is being employed today in 25, 50, 80 and 100# unit packing operations in some Chemical, Feed and Fertilizer plants.

It is accomplished for instance by Unions' Model UB-101A I & C Bagger, which will pack free and semi free flowing materials having variable densities, at accuracies with ± 2 ounces, and at speeds of 20 B.P.M.

The I & C UB-101A utilizes the same basic method of weighing so successfully used by the small unit packers—it divides the weighing cycle into two parts—Bulk and Dribble.

By means of a belt type constant speed feeder equipped with a cut-off gate the bulk portion of the cycle delivers 90-95% of the total weight into a weight hopper. During this function the cut-off gate is fully open to permit maximum volume of flow.

The remaining 5-10% of the total weight is fed into the weight hopper through a restricted opening of the cut-off gate. This is the Dribble portion of the cycle which ultimately controls the weight tolerance.

Material in suspension, that is, material which has run over the feeder but which has not reached the accumulation in the weight hopper thereby registering weight, controls the weight accuracy.

It can be recognized that if a uniform volumetric flow of material having a consistent density is fed into a weigh hopper, and, at a preset interval is cut-off completely, the material in suspension no matter what the volume, would be identical for each separate weighing, and the weights would be constant and accurate.

Using the same example but with material being inconsistent in density it can be seen that the material in suspension would vary, not in volume, but in weight thereby resulting in erratic weighings. It naturally follows that a large volume of material in suspension, will result in bigger weight variations but by means of a Dribble cycle the volume of material is cut down to a point where any change in density will have no appreciable effect in the ultimate weight tolerance.

It would be entirely possible to cut down the Dribble flow to a volume small enough to guarantee a weight accuracy which would have no readable variation on a conventional check weight dial scale. However, this method of obtaining absolute weighing accuracy though possible and desirable, is not practical, nor is it necessary in weighing fertilizer, because to limit the Dribble flow as described above the other important factor in bag filling—SPEED—would have to be sacrificed.

The ideal situation then is to be able to bag and weigh units at an accuracy that will insure against shortweights and reduce overweight or giveaway to minimum without sacrificing speed.

Union's I & C Bagger Model UB-101A has effectively demonstrated in Fertilizer, Chemical and Feed Plants that a weight tolerance of 2 oz. on unit sizes of 25, 50, 80 and 100 lb. can be maintained at speeds up to and above 20 BPM.

Robert J. McDonald

The problem of getting accurate weight control at bagging units due to variation in density of fertilizers can be divided into two general categories. The first category includes the manufacturing of fertilizer to minimize the variation in density insofar as is practical. The other category has to do with weighing equipment designed to be affected as little as possible by density variations.

All too often these people who work out the manufacturing process, conveying equipment, and storage methods give too little thought to producing and supplying a product to the bagging unit having characteristics that would promote efficient and accurate packaging. Packaging of the product should be treated as an important and final step in the manufacturing process. It is important to make a product not only acceptable to the consumer, but also suitable for packaging.

There is not time to go into the various means to produce a product having uniform density, except to mention the general areas in which consideration should be given. It is important to have uniformity of ingredients, accurate batching, thorough mixing, uniform particle size and shape, prevention of separation, moisture control, etc. The food and chemical industries have done a great deal along this line to devise means of better control of the product.

Much can be done in the way of designing weighing equipment that will minimize the effect of density variation of the product. The most important single item is to keep the amount of product in suspension at the time of cut-off to a minimum. The amount of product in suspension at time of cut-off refers to the product which has passed the cut-off gate but hasn't yet reached the bucket. This product cannot be weighed because the scale has already been satisfied in order to close the cut-off gate. The unweighed portion, then, falls into the bucket.

The amount of product in suspension varies directly as the length of stream in suspension. It is then apparent that the length of stream in suspension should be kept as small as possible. If there is only one product and one unit size involved, the problem of getting a short stream in suspension is simple. On the other hand, if several sizes must be run on the same equipment, such as 50's, 100's and 200's, the problem gets more involved. The scale bucket will fill at different levels for each unit size. If the bucket is designed for 200-lb. weights it will be only half full for 100-lb. sizes and one-fourth full for 50-lb. sizes. In a scale designed to have a short stream in suspension for a 200-lb size, the stream in suspension gets progressively longer for each smaller size. Various means have been used to help the situation. The bucket can be proportioned to hold one-half the maximum capacity and dumped twice when the maximum capacity is required. Frequently this works out very well because often the maximum capacity is only a small percentage of the total production.

Another method to compensate for variation of product level in the bucket is to make the scale feeder and cut off gate adjustable vertically. Some scales have had this feature, but where size changes are frequent, it is not practical to make the change.

The amount of product in suspension varies directly as the thickness of the stream; therefore, the thickness of the stream should be thin as practical at the time of finishing the weight. This can be accomplished by two-speed filling in which a heavy stream is used to get the bulk of the product into the bucket and a finer stream to finish bringing the bucket to weight. Another method to accomplish a thinner stream at the time of cut-off is a compromise bulk and dribble feed. To do this the feeder is allowed to run long enough after the cut-off gate has closed to collect a bulk charge for the next cycle. When the cut-off gate opens the bulk charge drops into the bucket, making it possible to use a thinner stream for finishing the weight.

A change in product density usually results in a change in flow characteristics which adversely affects the weights. The feed rate of a belt feeder is affected less by variation in flow characteristics than other types of feeders. This is a point in favor of belt feeders.

In conclusion, we can say that the manufacturing processes should be worked out to produce a product having a reasonably uniform density. Also, bagging equipment should be selected that has been designed to minimize the effects of product density variation. Keeping the minimum of product in suspension at the time the weights are finished is important in combatting density variations.

Allen S. Jackson.

First, I would like to state the problem as I see it. If there is a fall of one foot between the gate of a bagger and the bed of material in the weight hoppers the time of fall of the material is ¼ of a second. If the shut off gate of the bagger requires ¼ of a second to operate the total time lapse between the sensing of the scale and the complete operation is then ½ second. At twenty 100 pound bags per minute operation this represents a quantity of 16 pounds of material "en route" to the scale hopper that has in effect been timed and not weighed. If the density of material, that is rate of feed, changes 10% this would cause an error of 1.6 pounds per bag.

Two approaches can be made to the problem:

First, by the design of the bagger and second by the operation and maintenance of the bagging equipment. The design of a bagger is a compromise of several things; lowering inertia of moving parts by reducing weight vs durability of said parts, simplicity of operation and maintenance vs compensating gadgets, quickness of electric power vs powerful operation and repeatability of air; basic design to minimize time effect vs possible head room requirements of the machine.

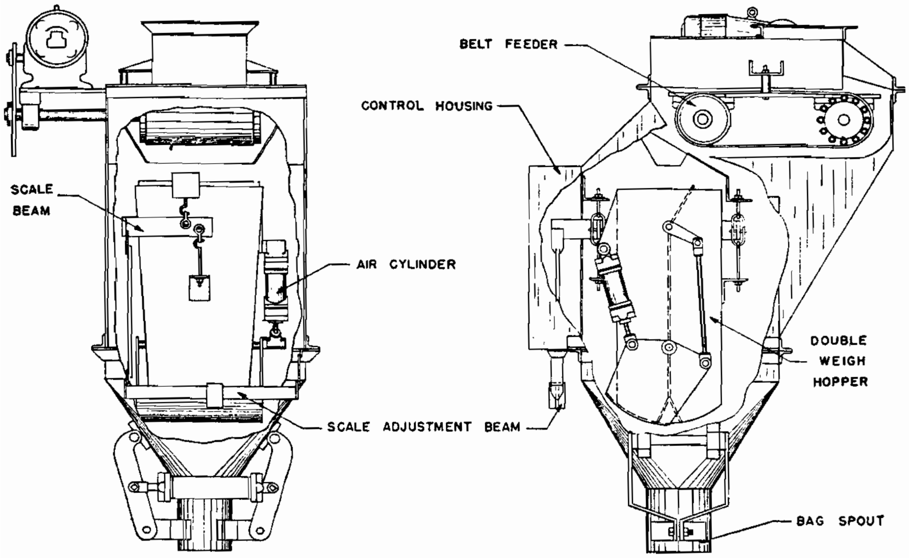
A well maintained gravimetric rather than a volumetric feeder feeding the scale would obviously eliminate the varying density problem, but operationally it would probably be impractical and the initial cost of a bagger would probably be increased by several thousand dollars.

We have approached the problem by placing the shut-off gate on the weight hopper itself, practically eliminating the fall of material past the gate, and used a continuous running belt to make rate of flow a minimum. We use a dual hopper on one scale. We have, however, used air power to move our gates which are fairly heavy. The air power plus the weight of the gates causes somewhat slower operation than could be obtained with electric power but it was our choice in the compromise of simplicity and durability.

In the operation of existing bagger already in mills, I believe there could be some improvement in operation of many of them. Visiting many mills as I do several errors in operation of automatic baggers are noted.

1—It is surprising to note how many operators have not taken the time to learn exactly "how" and "why" their bagger works and what its limitations are.
2—Some mills have their bagger operating at speeds greater than they can supply the material to it, maintaining high possible error yet not getting any increase in throughput.
3—Normal scale maintenance is so often ignored. Possibly because the pressure of the short shipping season or possibly because the bag weight is set by trial and error rather than by scale measure the operators do not realize that pivots and other parts are depreciating.

I feel that if mills in general would examine their bagging program and make certain that they are operating their bagger no faster than necessary and then give the unit the same care that they would give any other sensitive scale their trouble would be at a minimum.



R. E. Harrison

We as a company have been aware of the problem of varying density for the last 20 to 25 years and have devoted a great deal of our engineering time to finding a practical solution. We have not only persued the ideas of our own engineering staff but have on many occasions worked with the engineers and operating people of your industry.

Items like telemeters and automatic compensators have been analyzed very carefully both in our laboratories and in your plants. Although many of them have some merit, to date they do not appear to be the practical solution you are looking for.

The real problem, of course, is controlling material in suspension, impact, roll-off force, inertia in friction. Most packing equipment today in weighing an 80# charge at high speed cuts off at 72# to 73# leaving 7# to 8# in suspension to give desired weight. Variation of bulk density between grades and also within grades causes this amount of material in suspension to vary as much as 2# to 3# and in some cases more.

Many improvements have been made by packing equipment people in scale design, scale control circuits, scale greeding methods etc. Your industry has contributed through better fertilizer conditioning and better maintenance programs. However, economic conditions have demanded that these improvements be used for increased production rather than improved weight control in much the same manner as engineering improvements in automobile engines were used, by popular demand, to tremendously increase horsepower rather than gasoline mileage.

Now that packing rates of 20 to 30 bags a minute are obtainable, we feel that packing rates have reached a plateau and future improvements will be used to improve weight results and control variations in densities. We presently have a staff of specialtists, making a comprehensive study on the behavior of materials and the flow of solids. Controlling the behavior of materials will take us a long way toward solving this problem since we have established that at high speeds constant motion control is basic.

At present we do not believe that mechanical devices can be incorporated in the scales to solve this density change problem and we are looking to the electronic and pneumatic fields. The final solution will probably come in an electro-pneumatic device which will automatically compensate for variation or reduce the amount of material in suspension to a point where it is no longer a major factor.

Until these developments are perfected you can alleviate this problem in your own plants with your present equipment in part. Most packing equipment of the modern type today has a reasonable amount of repeatability. If you are packing a fertilizer of 70# cu. ft. density, let's say it is 5-10-10, your operator should observe exactly where the adjustments are. When he switches to another grade and comes gack to the 5-10-10 if he will replace the adjustments exactly as he had them when last running 5-10-10 of 70# bulk density, he will have the desired weight or very close to it. This is known as repeatability of a scale.

A great deal can also be done to solve this weight variation problem by maintaining constant moisture control of your material. Also, stockpiling methods to eliminate or greatly reduce stratification and segregation of particles would greatly help.

What Are the Ammonium Characteristics of Supers (Normal and Triple) of Different Granularities?

C. E. Waters

WE presented some data on the ammoniation of superphosphate of different particle sizes in 1954, and the information was published in 1955. The work was done in a laboratory ammoniator, using fractions of different size ranges obtained by screening normal superphosphate. A solution containing 65% ammonium nitrate and 21.7% ammonia was used. The grades of fertilizer made were 5-13-10, 5-10-10, and 5-7.5-10, corresponding to ammoniation rates of 4.13, 5.33, and 7.17 pounds of ammonia per unit of P_2O_5. Analyses of products made with 3-minute absorption time (including the time required for spraying the solution) indicated nearly complete absorption by 30- to 40-mesh fraction at the 4.13-pound rate, by the 40- to 50-mesh fraction at the 5.33-pound rate, and by the 50-to 60-mesh fraction at the 7.17-pound rate. Somewhat different limits were estimated on the basis of analyses of the air in the ammoniator after a three-minute reaction period.

We did not make similar tests with triple superphosphate. A reasonable working assumption is that similar results would have been obtained at ammoniation rates about two-thirds as great as those used with normal superphosphate, i.e., 2.8, 3.6, and 4.8 pounds of ammonia per unit of P_20_5.

Data on ammoniation of normal and triple superphosphates with anhydrous ammonia were published by Kumagai, Rapp, and Hardesty in 1954. They showed that low ammoniation rates, long reaction time, and fineness, softness, and high moisture content of the superphosphate were conducive to efficient absorption of ammonia.

In the booklet describing the pilot plant demonstrations of fertilizer technology by the T. V. A. in June, 1957, it was mentioned that granulated, concentrated superphosphates produced in the continuous ammoniator have a granular structure that allows them to be ammoniated to about the same degree as nongranular superphosphates.

SECTION 2A

Discuss Methods for Proper Drying and Formulation so as to Take Care of Shrinkage. Most Manufacturers End up with Shortages at the End of the Year Where Large Tonnages of High Analysis Goods are Made.

F. T. Nielsson

TWO sets of calculations are necessary. The first shows the amount of moisture and CO_2 added with the raw materials. Subtracting the estimated moisture content of the product from the total moisture and CO_2 input shows the evaporation that should take place during the process. The evaporation is placed into the formula as a minus quantity.

The second calculation shows the dust and handling losses that can be expected in the process. These latter losses are what I call shrinkage. Usually shrinkage is of the order of two (2) per cent. Two ways of handling shrinkage can be used. In one, all of the items in the formula are multiplied by 1.02 to show the actual quantities required to yield one ton of dried product. In the other, a 2% shrinkage factor can be taken to show that 2,000 lb. of input will yield 1960 lb. of product. In plants where the dust catcher exhaust is scrubbed and the scrubbing water is added back to the system, the theoretical input of product into a storage bin must be checked against the actual shipping weights to arrive at the shrinkage.

In calculating evaporation, the following points should be taken into account.

1. Triple and super dry out in storage. The loss of moisture is especially marked in phosphates that have been left in inventory during the summer when the relatively small piles have had a good opportunity to dry.
2. Granular triple usually contains less moisture than does run of pile triple.
3. DuPont urea solutions contain CO_2 in addition to the water reported by the manufacturer. The CO_2 loss should be calculated as part of the evaporation loss.
4. Limestone reacts with sulfuric acid at the temperatures found during ammoniation. About 12 per cent of the weight of fine limestone is lost as CO_2 while for coarse limestone the corresponding figure is about 3 per cent.
5. Dry limestone and sand contain about one per cent moisture while in wet limestone and sand the moisture content is about four per cent.
6. Electric furnace phosphoric acid of 54 per cent P_2O_5 concentration contains 25 per cent water, while wet process acid of the same P_2O_5 concentration contains about 15 per cent water. Spent acids contain water in varying amounts depending upon the type of spent acid. The water content of acid as found by the Karl Fischer method yields good results when used in calculating evaporation loss in granular formulae.

Joe C. Sharp

We should first define what we mean by shrinkage. Are we talking about loss of plant food or merely loss of weight? If it is only loss of weight we are talking about and all the plant food can be accounted for, then it is caused by water evaporation and can be easily compensated for by treating the raw materials as though they contained the same amount of moisture as the finished product. That means if the finished product is to be dried to 1% moisture, the raw materials should be treated as though they contained 1% moisture. In some raw materials like normal superphosphate, treating it in this manner will increase the analysis of the raw material to be used in calculating the formula. For example, if the superphosphate contains 20% P_2O_5 and 7% moisture and the finished product is to be dried to 1%, the analysis of the superphosphate should be 20 divided by 94, which is equal to 21.3% P_2O_5.

When the raw material contains less than 1% moisture, the analysis to be used in calculating the formula will be lowered on that particular material. For example, if 60% muriate of potash contains 1/10 of 1% moisture, the analysis of this product will be 60 divided by 100.9 which is equal to 59.5% K_2O.

For bookkeeping purposes, this means that all materials must be converted to tons of plant food to avoid bookkeeping losses.

Plant food losses may occur in many different ways. Over-ammoniation will cause nitrogen loss. Even with efficient reactor conditions, ammonia losses become appreciable at a rate above 3.5% for triple superphosphate and 5% for normal superphosphate. If either triple or normal superphosphate contained large percentages of hard particles, losses will occur even below the above ammoniation rates. If the reactor becomes too wet so that over-agglomeration takes place, ammonia is lost to the atmosphere. Poor distribution of solution and acid also causes ammonia losses. The use of too much sulphuric acid will cause local overheating and result in ammonia loss. Too shallow a bed depth in the reactor will let the ammonia escape before it can react with the superphosphate or acid.

Overheating in the dryer will cause plant food losses. The critical temperature for different mixtures has not been established, but there is some evidence that decomposition in the dryer occurs when any part of the material exceeds 230°F. A portion of the material may reach or exceed that temperature, even though the stream of the material leaving the dryer may be much below that temperature. This may occur by the flame coming into direct contact with the material or material may adhere to the flights or shell of the dryer and become exposed to high temperatures for a long period of time.

Another source of plant food loss is through inefficient dust collectors. Keeping discharge doors operating properly on cyclones and keeping the inside wall clean help to minimiz such losses.

C. E. Floyd

We believe that the construction of the formula has more to do with the loss by shrinkage than any other one factor. Each grade is individual within itself and after we have worked out a formula which will give us good agglomeration, then we have to adjust to take care of free water losses and in some cases other gaseous losses which no doubt occur. In addition we have to allow for dust losses through the dryer and cooler collectors. Also, we have to allow for normal losses in handling and bagging. Gas losses and dust losses through the collectors are dead losses and naturally increase costs. In order to minimize these losses, we need a certain amount of heat of reaction in the ammoniator in order to attain a liquid phase without the addition of water. This condition will promote drying at relatively low temperatures, thereby decreasing the liability of gas losses. In addition, proper agglomeration on the first pass decreases the amount of free dust to be handled through the collectors. So from these facts, we can conclude that the formula construction is of paramount importance. During the discussion period we will be glad to give you our idea of overages to be allowed on any one grade. Generally speaking, after allowing for free water losses which will give us a residual moisture content of 2% or less, we add about 1% for dust and gaseous losses in low nitrogen goods; then add about 2% for handling and bagging. In our experience we have found that we have to go as high as 3% on gaseous and dust losses in high nitrogen grades such as 12-12-12.

Summarizing: in order to get good agglomeration, we try to construct a formula which will give us a mass temperature of approximately 200°F. in the ammoniator. If we can attain a liquid phase of the soluble salts, then we get good agglomeration without the addition of water; with this condition existing we dry at low temperatures and produce very little dust in the process, thereby effectively controlling our inventories.

Question: Discuss the Economics of Using Phosphoric Acid in the Manufacture of Complete Fertilizers.

A. V. Slack

PHOSPHORIC acid is used in production of complete fertilizers in two ways. One is in slurry-type processes where it is used as the sole source of phosphate in making products of the ammonium phosphate or ammonium phosphate-sulfate types. The other is in mixed fertilizer production — usually granular — where it is used to supplement normal superphosphate in mixes which are so high in analysis that grade cannot be made with normal superphosphate alone. The latter type of usage is the one covered in this discussion.

At first glance phosphoric acid does not appear to offer any economy. In most areas it is more costly than triple superphosphate, which is the material normally used in upgrading to make high analyses. The acid has the following advantages, however, which tend to offset the cost differential.

1. It can tie up more ammonia than triple superphosphate.
2. It supplies hydrogen ion needed in granulation for producing heat of reaction in making low-nitrogen grades. Thus it replaces part or all of the sulfuric acid requirement.
3. The ammonium phosphate produced on ammoniation is more concentrated than ammoniated triple superphosphate. Therefore there is more room for relatively inexpensive normal superphosphate in the formulation.

The greater ammonia holding power of the acid is an important advantage. Data from TVA and industry tests indicate that the acid can be ammoniated without excessive loss — under average operating conditions—to about halfway between mono-and diammonium phosphate, which corresponds to 7.2 pounds ammonia per unit of P_2O_5. Triple superphosphate, on the other hand, is

TABLE 1

Formulation Costs for 12-12-12 and 5-20-20 With and Without Phosphoric Acid

			12-12-12				5-20-20			
			Conventional formulation		H_3PO_4 formulation		Conventional formulation		H_3PO_4 formulation	
Raw material	Grade	Price, $/ton	Lb./ton	$/ton	Lb./ton	$/ton	Lb./ton	$/ton	Lb./ton	$/ton
Nitrogen solution[a]	41% N	53	494	13.10	446	11.80	—		—	
Anhydrous ammonia		90	—		—		125	5.60	125	5.60
Ammonium sulfate	20.5% N	38	195	3.70	293	5.57	—		—	
Superphosphate										
Normal	20% P_2O_5	20	537	5.37	761	7.61	293	2.93	902	9.02
Triple	46% P_2O_5	60	300	9.00	—		761	22.83	—	
Phosphoric acid	54% P_2O_5	79	—		168	6.64	—		412	
Potassium chloride	60% K_2O	36	400	7.20	400	7.20	667	12.00	667	12.00
Sulfuric acid	60° Bé.	23	162	1.86	62	0.71	140	1.61	—	
				40.23		39.53		44.97		42.88

[a] 22.2 per cent ammonia; 65.0 per cent ammonium nitrate.

limited to about 4.0 pounds ammonia. If sufficient gypsum is available from the normal superphosphate in the mix, both the phosphoric acid and triple superphosphate can take up more ammonia by reaction between gypsum and the ammonia phosphate formed in the initial ammoniation step. However the reaction of triple superphosphate is slower because the gypsum and ammonium phosphate are in separate particles. Therefore, phosphoric acid gains more ammonia capacity from the presence of gypsum than does triple superphosphate. Good data on the relative increases are not available.

Some idea of the savings involved can be obtained by considering a formulation in which 10 units of P_2O_5 is supplied by either phosphoric acid or triple superphosphate. The acid would take up 32 pounds ammonia more than would the triple superphosphate (neglecting any effect of gypsum present). Sulfuric acid is normally added to neutralize excess ammonia in formulations in which more ammonia is used than the superphosphate will take up. About 100 pounds of sulfuric acid (66° Be.) would be required to neutralize the 32 pounds of ammonia, at a cost of $1.00 or more per ton, assuming that the acid costs $20 or more per ton.

Phosphoric acid not only supplies phosphate for low-nitrogen grades but also replaces sulfuric acid in producing heat of reaction. However, the heat produced is lower per unit of ammonia reacted and more water is introduced with the acid. As a result, the ammoniator temperature is lower, the moisture content of the material leaving the ammoniator is higher, and a greater load is thrown on the dryer. The higher water content also increases drying and recycle requirements for high-nitrogen grades. A recent survey indicated that most producers who use phosphoric acid in high-nitrogen grades use some sulfuric acid along with it and may also use more ammonium sulfate in the formulation. The use of concentrated electric-furnace acid (superphosphoric acid) developed at TVA may be helpful in avoiding this difficulty since it contains no free water.

The degree of advantage realized from being able to use more normal superphosphate varies widely, depending on the relative cost of phosphoric acid, triple superphosphate, and normal superphosphate. The greatest advantage accrues to producers who have their own normal superphosphate plants and therefore have already incurred some of the costs of production. With the advent of higher analyses, some of these have had to reduce production of normal superphosphate because of the requirement for a more concentrated phosphate in order to make grade.

The estimates given in Table I indicate the overall economic situation in regard to use of phosphoric acid. The formulations listed are believed to be fairly typical of those used in industry, although widely different formulations are used by some producers. More saving is indicated for the 5-20-20 than for the 12-12-12, because more ammonium sulfate had to be included in the phosphoric acid formulation for the latter to counteract the effect of the greater amount of water introduced with the acid. The estimates show that for both grades the principal benefit results from the reduction or elimination of sulfuric acid.

Phosphoric acid has a further advantage which is difficult to evaluate from the economic standpoint. Producers who have used it report that fume from the ammoniator is substantially reduced or eliminated.

What are the Economics of Using "Wet" Phosphoric Acid in Place of Furnace Acid; Other Advantages? Can Sludges from "Green" Phosphoric Acid be Used in Granulating Fertilizers?

Rodger C. Smith

THE primary advantage of wet process phosphoric acid in comparison with furnace grade phosphoric acid is cost. Any other advantages are very much secondary and there are some disadvantages to the wet process acid.

Production of wet process acid involves the reaction of sulfuric acid with rock phosphate and the filtration or other separation of the insoluble residue. There results a dilute acid, about 30% H_3PO_4, and containing varying amounts of calcium sulfate residue. The acid is normally concentrated to about 65% acid strength for use in mixed fertilizer manufacture.

The production of furnace acid results from the reduction of rock phosphate in furnaces at high temperatures, the oxidation of volatilized phosphorus to phosphorus pentoxide, and then the reaction with water to form phosphoric acid. With this process, 85% H_3PO_4 can be produced readily, but the 75% grade is normally available commercially because the 80% or 85% grade crystallizes under temperatures which can be experienced in the United States.

The use of phosphoric acid in mixed fertilizer granulation accomplishes two things, (1) reaction with ammonia with its attendant addition of heat to the mixture and (2) provides the nutrient P_2O_5.

Assuming 93% sulfuric acid costs $25 per ton, the neutralizing value of 65% acid is about $17 and that of 75% acid about $20 per ton.

65% phosphoric acid has virtually the same P_2O_5 content as triple superphosphate. Therefore, it can be said that, roughly speaking, 65% phosphoric acid has a value of the cost of triple superphosphate plus $17 per ton. Conversely, where 65% grade is available at the price of triple superphosphate less the cost of equivalent acid, it is good economics to use it.

It appears likely that wet process acid will continue to be lower cost than equivalent furnace grade considering the two processes of production, the general availability, the amount of electricity required for making elemental phosphorus and the generally lower freight charges applicable to wet process acid delivered to the mixed fertilizer plant.

There have been some reports, both experimental and production, that wet process acid contributes more to granulation than furnace acid. Our experience is that there is very little difference in their effect on granulation. Wet process acid has been used successfully at our Wilmington, Delaware, plant in producing 10-10-10, 8-16-16, 6-18-18 and 6-12-18 as has been furnace acid.

Any significant difference in effect on granulation is related to the concentration. The necessity of using an increased amount of lower concentration acid has the effect of lowering the temperature of the ex-mixer material which necessitates a slightly higher moisture level to accomplish agglomeration. Drying requirements are slightly higher. If the dryer has limited capacity in relating to the rate of production, percentage of acceptable size granules may be reduced slightly and the moisture level of the product endangered.

Perhaps the most important difference between the two grades of phosphoric acid has been in the matter of metering to continuous ammoniators. The viscosity of phosphoric acid varies substantially with temperature. The presence of calcium sulfate precipitate adds to the problem of satisfactory meter operation. The meter manufactured by the Foxboro Company which measures the flow magnetically is being installed in many plants for use with phosphoric acid. Reports on its performance appear to be very satisfactory.

T. E. Martin

There has been little competition between wet phosphoric acid and furnace acid in the solid fertilizer industry. This is primarily since furnace acid is too expensive for such use. It is true that, during the period of high-analysis phosphate scarcity just after the war, furnace acid was used by a few fertilizer manufacturers for making triple superphosphate. Also in a few cases manufacturers have used furnace acid in mixed goods formulation. Generally speaking, however, wet process acid has entered the market as a new product, not as a replacement for furnace acid.

During the past summer the price of furnace acid increased $5.00 a ton or about $0.09 a unit of APA. This was taken in some quarters to indicate that furnace acid was being withdrawn from the dry mixed goods and triple superphosphate markets. Tending to reinforce this conclusion is the fact that one of the furnace acid producers has recently offered for sale wet process acid produced by a major fertilizer manufacturer.

Furnace acid for fertilizer use is now priced, f.o.b. producing point, at $85.00 a ton of 75 per cent acid containing 54.5 units of APA. This is equivalent to $1.56 per unit of APA. Wet process acid containing the same 54.5 units of APA sells for $1.32 a unit or $71.94 a ton f.o.b. middlewestern producing point. The difference is $13.06 a ton or $0.24 a unit. One large wet process acid producer absorbs up to $0.08 a unit freight if necessary in order to hold the delivered price down to $1.45 a unit. This delivered price at distant points is still $0.11 a unit under furnace acid price at point of origin.

It now appears that wet process acid has driven furnace acid from the rather minor use position it had a few years ago in the manufacture of mixed fertilizers and triple superphosphate. At present furnace acid still enjoys a dominant position in liquid mixed goods manufacture.

Advantages other than price offered by wet process acid in comparison to furnace acid for dry fertilizer manufacture are:

1. Wet process acid contains several per cent of sulfuric acid which is free to the fertilizer manufacturer. This is not present in furnace acid. It provides ammonia pickup and additional acidulation acid.

2. A second advantage of wet process acid is that it contains desirable trace elements — iron, aluminum, magnesium, calcium, potassium, zinc, and manganese. These are absent from furnace acid.

3. A third advantage of wet process acid is that it contains about one quarter less water than furnace acid, for the same 54.5 units of APA content.

4. A fourth advantage of wet process acid—it contains metallic impurities, particularly iron, which are desirable granulation aids.

5. A fifth advantage of wet process acid lies in the peculiar and apparently permanent depression of the crystallization temperature far below that for furnace acid. This is characteristic of the product of at least one middle western producer.

Several advantages have been claimed for furnace acid as compared to wet process acid for dry fertilizer manufacture. These are more imaginary than real. They are:

1. The claim has been made that the solids content of wet process acid will plug piping, erode pumps, jam instruments, and so on. However the major producer of wet process acid for sale to the fertilizer industry has been increasingly able to control the solids content at a low level satisfactory to customers. It should be realized that the major portion of the solids present is iron and aluminum phosphates, which are desirable aids to granulation. Iron has even been added to furnace acid for this purpose.

2. The green or black color of wet process acid hinders visibility in the "see-through" type of variable-orifice flowmeter known by the trade name of Rotameter, Flowrator, and so on, particularly when the float is near the top of a large size Rotameter. Little trouble of this sort has been experienced with the small size Rotameters commonly used in fertilizer plants. This problem does not exist with the magnetic follower indicators of armored Rotameters, where the operator never sees the material being metered. There are other much more serious problems in the use of Rotameters for metering either wet process or furnace acid.

3. For the same APA content, wet process acid contains less water than furnace acid since it contains in solution desirable quantities of sulfuric acid and iron and aluminum phosphates. This results in a higher viscosity for wet process acid. The high viscosity of furnace acid at winter acid temperature introduces a problem of accuracy of a Rotameter for metering. The higher viscosity of wet process acid gives the same problem. This problem is a major objection to the use of Rotameters to meter either type of acid accurately. With care the application of temperature correction factors to flow settings can be made with fair

accuracy. At best this is a considerable nuisance.

4. Wet process acid contains very small quantities of fluorine, which is not present in furnace acid. The fluorine content is very much lower than that of normal superphosphate, however, and has not been objectionable in the fertilizer plant. There seems to be no extraordinary corrosion problem associated with this fluorine.

5. Wet process acid with 54.5 units APA content is not quite as high analysis a material for formulation purposes as furnace acid of the same strength. This apparent contradiction is due to the fact that on the dry basis wet process acid runs about 66.5 per cent APA while furnace acid is about 72.7 per cent APA. This difference is due to the presence of sulfuric acid and iron and aluminum phosphates in the wet process acid, which are desirable in the fertilizer formula, and may even be added if furnace acid is used.

6. As a small part of the APA in wet process acid is made up of the granulation acids iron and aluminum phosphates, wet process acid does not contain quite as much phosphoric acid as furnace acid of the same APA content. This results in a little less ammonia pickup, which, however, is compensated for by the sulfuric acid content of the wet process acid. In general it is correct to assume the same ammoniation rate for wet process acid and furnace acid.

R. R. Hull

OUR experience on this problem of sludges has been very limited and I do not think it can be used as a worthwhile guide in answering the question. The major problem seemed to us to be whether the sludge is used wet or is dried prior to use. When the wet sludge was used we had great difficulty in weighing or measuring the material into the system. Its use resulted in plugged elevators and chutes. It was difficult to obtain representative moisture samples which in turn upset the water balance in the granulation system. The P_2O_5 content varied considerably and caused wide variations in analytical results. Since the plant design was not readily adaptable to changes to permit the use of wet material its use was abandoned. We considered the possibility of drying the sludge prior to use but found drying costs were excessive and therefore did not attempt to evaluate it.

A limited amount can be utilized by adding it to the pan mixer when making ordinary superphosphate. In this way the P_2O_5 value can be recovered in the finished product but of course it dilutes the finished analysis.

What Methods can be Used for Manufacturing High Nitrogen Ratios Such as 2-1-1 and 1-0-1, 16-8-8 and 14-0-14, for Example.

C. E. Floyd

WE have successfully made 14-0-14 by reacting a low ammonia solution with enough sulphuric acid to take care of the free ammonia in the solution. We then overflow this reacted solution onto a rolling bed of potash and dolomitic limestone, as well as some sulphate of ammonia. So far we have managed to derive about 10 units of the nitrogen from the reacted solution. We will be glad to give you the details of our process; however, will in no way be responsible for results obtained or hazards involved, even though we think these hazards to be of minor concern.

This 14-0-14 can be bagged shortly after manufacturing and will stay in very good condition for a considerable length of time. Coating with a suitable diatomaceous earth will no doubt improve the carrying qualities.

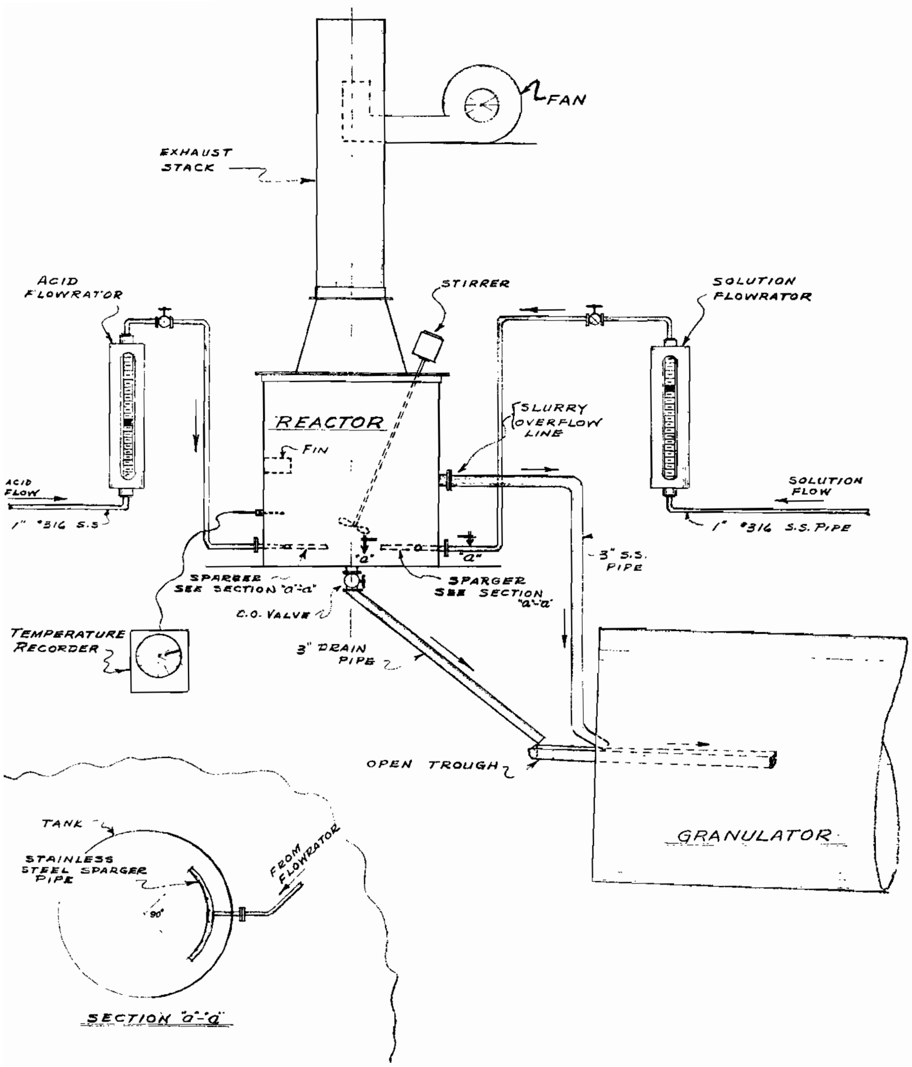
16-8-8 of course can be made by conventional methods; however, we are limited in the amount of Solution we can use due to an excess liquid phase. We believe that we could use a modified 14-0-14 method for this grade and perhaps cut the formula cost to some extent.

We might mention the fact that we have also made 8-0-24 Tobacco Top Dresser by the same method as that employed for 14-0-14. Of course in the 8-0-24 T.D. most of the potash was derived from sulphate of potash. Our product was unusually good and stable for a mixture of this nature and we have been highly pleased with results obtained. In the 8-0-24 grade we have obtained all the nitrogen from low ammonia solutions.

We feel that there will be numerous questions with regard to the above mentioned processes, and will be glad to answer them to the best of our ability.

We believe that a blackboard sketch will help us explain the details of the process. (Insert sketch attached.) We set out to make 10 tons of 14-0-14 per hour and arbitrarily designed a stainless steel reaction tank 36″ high and 30″ in diameter. We introduce the solution and acid through sparger pipes as shown on the sketch. We also keep a stirrer in operation to minimize the direct contact of the Acid and Solution. Fins were placed slightly beneath the overflow to prevent the formation of a vortex. With the type Solution we use, reacted with just enough or perhaps a slight excess of 60° Bé. acid needed to neutralize the free ammonia in the solution, we maintain a constant temperature about 280° F. in the reactor. The over-flow pipe is approximately 18″ above the bottom of the reactor tank. The reacted solution flows into an open trough which extends into the granulator. The solution overflows from the trough onto the rolling bed of dry materials. The dry materials consist of muriate of potash, limestone, and sulphate of ammonia. Deriving 10 units of nitrogen from the reacted solution, we have to add a very small amount of water to get good agglomeration. The product is easily dried to less than 1% free moisture. Very little recycle occurs in the process. Analyses indicate a very homogenous product.

Question from the floor: What type stainless steel was used in con-



struction of the tank? Answer: Number 316.

Question from the floor: What type solution was used? Answer: Allied #4 - 37% Total nitrogen - 16.6% water - 16.6% free ammonia. 60° Bé. acid was used to supply enough water to complete the reaction and to maintain a liquid phase until it was flowed onto the bed of dry materials. The muriate of potash used was of fine texture.

Are there Any Data from Tests or Experience Regarding use of Diammonium Phosphate in the Production of High-Analysis Goods Such as 16-20-20, 6-24-24, 13-13-13, 15-15-15? Compare with Phosphoric Acid and Ammonia, i.e., Mechanical Condition, Carrying Qualities and so on.

T. P. Hignett

Very good results were obtained when diammonium phosphate was used in the production of granular high-analysis fertilizers. The following advantages were apparent.[1]

1. Higher analysis products can be made with diammonium phosphate than are possible with conventional raw materials.
2. Diammonium phosphate contributes water-soluble P_2O_5, thereby increasing the water solubility of the products.
3. Inclusion of diammonium phosphate decreases the amount of ammoniator reactions that would otherwise be required. This may be an advantage in making some high-analysis grades in which conventional formulations may involve more heat of reaction than is desirable for good granulation.
4. Substituting diammonium phosphate for equivalent amounts of nitrogen solution and superphosphate or phosphoric acid lowers the moisture content of the formulation. This is an advantage when the moisture content is higher than is needed for granulation. The advantage of lower moisture content may result in economies in drying the granular product, and it may facilitate higher production rates in plants of limited drying capacity. In fact, such high-analysis grades as 13-39-0 and 12-24-12 have been made in commercial plants that have no dryer.
5. In making standard grades such as 12-12-12, use of diammonium phosphate rather than triple superphosphate leaves more room in the formulation for ordinary superphosphate. Therefore, formulations using diammonium phosphate are often cheaper than conventional formulations.

Grades of granular fertilizer that have been produced in our pilot plant with diammonium phosphate in the formulation include 16-20-0, 20-20-0, 15-30-0, 13-39-0, 8-24-24, 10-30-20, 12-24-12, 12-12-12, and 15-15-15. Incorporation of the diammonium phosphate has been accomplished without difficulty. The proportions used varied from 150 to 600 pounds per ton of product.

Good granulation was obtained in these grades. The recovery of onsize product (—6 +28 mesh) ranged from 83 to 97 per cent. Granulation was readily controlled by the addition of small amounts of water, steam, or cooling air. The granules were hard and strong. The storage properties of the products were about the same as high-nitrogen grades made without diammonium phosphate.

Many commercial plants are using diammonium phosphate in

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[1] "Use of Diammonium Phosphate in Production of Granular, High-Analysis Fertilizers." T. P. Hignett, G. C. Hicks, nad J. E. Jordan. *Com. Fert. and Plant Food Ind.* 93, No. 4, 24-6 (October 1956).

formulations for granular fertilizers. In addition to the grades listed above, 15-30-15, 6-24-24, 5-20-20, 8-24-8, and 10-30-10 have been made. Most of the plants have reported that the diammonium phosphate formulations work very well and in many cases give better operation, particularly in grades in which too much plasticity was a problem. One producer who has had considerable experience with both diammonium phosphate and phosphoric acid reported that grades made with diammonium phosphate had much better physical properties than those made with phosphoric acid.

In our pilot-plant work we have made 15-15-15 with either diammonium phosphate or phosphoric acid. This is the only grade for which we have a direct comparison. When both were dried to 0.5 per cent moisture, the unconditioned product made with diammonium phosphate did not cake during 3 months of storage whereas the one made with acid caked. Conditioning the latter product with 2.5 per cent of kaolin or kieselguhr prevented caking. The phosphoric acid formulation contained more water; hence, more drying was necessary.

From the operational viewpoint, diammonium phosphate formulations seem preferable in grades in which it is otherwise difficult to reduce the moisture level, plasticity, or temperature such as 16-20-0. Phosphoric acid formulations seem preferable for grades that need more plasticity and in which the water content of the acid is not objectionablee such as 6-18-36.

Some precautions must be taken in formulating with diammonium phosphate. If superphosphate and diammonium phosphate are premixed and held for even a short time before ammoniation, the ability of the superphosphate to absorb ammonia will be impaired seriously. It is important to bring the diammonium phosphate, ammonia, and superphosphate together simultaneously in order to retain the normal degree of ammoniation of the superphosphate. Also, it is advisable to ammoniate the superphosphate fully in formulations which contain diammonium phosphate. Any unammoniated superphosphate remaining in the fertilizer may react with diammonium phosphate in the storage pile. This reaction might weaken the particles. Some producers have reported disintegration of granules in the storage pile presumably due to this cause.

Ray Sorenson

For the past two years we have used diammonium phosphate in manufacturing the following analyses fertilizer: 13-39-0, 12-24-12, 10-30-10, 10-20-20, and 6-24-24. In these fertilizers we have used from a minimum of about 50 pounds to a maximum of about 400 pounds diammonium phosphate. However, the carrying qualities are not quite as good as some of our lower analyses grades where less nitrogen is used. We attribute this fact to the high percentage of nitrogen rather than the use of diammonium phosphate. Granulation of these grades has been excellent. For instance, we believe our 13-39-0 to be as good as the same analysis made in the form of ammonium phosphate in both granulation qualities and carrying quality. However, our formulation costs are somewhat higher because of the amount of sulphuric acid required in some of the higher nitrogen grades.

J. Howard McNeill

Many manufacturers are beginning to use DAP in high analysis goods in order to compete with high assay grades in their areas. There has been some confusion regarding the incorporation of DAP in these grades. As high analysis granular goods continue to gain popularity, the use of DAP is likely to continue to increase in certain sections of the U.S.A.

Monsanto has been successful in the use of an electronic computer to help some manufacturers work out formulation programs. DAP is one of the optional materials which may be studied to determine if it is profitable. Projects for customers have involved both the broad economic studies and operational formulas as well as actual plant demonstrations. Once the economics of DAP as a raw material for formulation are justified by the computer method, the most profitable formulas are selected for plant-scale use.

Monsanto did research on numerous combinations of raw materials with DAP in order to obtain some of the basic data needed in the field.

Results which are of particular interest to the manufacturer of granular fertilizer are briefly mentioned below. Results of our pilot plant and plant tests show that grades, such as 16-20-0 (300 lbs. DAP/ton), 11-48-0 (650 lbs. DAP/ton), 13-39-0 (295 lbs. DAP/ton), and 15-15-15 (250 lbs. DAP/ton), can be made satisfactorily in standard granulating units. Formulations typical of those produced by the computer when DAP is allowed, and verified by actual plant runs are attached to this paper.

DAP has been found to release as much as .3 mole of its 2 moles of ammonia when heated to temperature suitable for granulation. This free ammonia must be tied up with some other material or it will be lost. Therefore, Monsanto has adopted a system of formulation which allows for the reation of the .3 of ammonia with the acid materials.

Several people have raised an objection to the use of DAP in granulation because of this so-called unaccountable loss of nitrogen in the finished product. It appears that in all cases there has been no allowance made in the formulation for absorption capacity in companion materials for the NH_3 which will be released from the DAP.

In working with DAP in high analysis grades, Monsanto has found these advantages worthy of consideration:

1. DAP contributes "space" to a formulation because of its concentration, 74% plant food.
2. In many areas the use of DAP reduces the cost per ton of an analysis either directly on the cost of N and P_2O_5 or, more commonly, by increasing the relative use of low cost materials such as normal superphosphate.
3. DPA tends to increase the total water solubility of some mixtures because DAP is completely soluble.
4. Often, higher production rates

can be obtained with DAP. As DAP is substituted for other materials in formulations, "cooler" formulations are usually the result since most materials being displaced have higher heats of reaction.

5. Drier formulations can be obtained by substituting DAP for nitrogen solutions and super phosphate or acid.
6. As a raw material, DPA requires less storage space for equivalent plant food than does sulphate of ammonia plus triple superphosphate. Also, only one storage area is needed.

The addition of potash to most DAP mixtures results in about the same behavior as in non-DAP formulations.

Some manufacturers have preferred to use large amounts of DAP at slightly higher cost per ton in order to get higher production and better quality products. Some have found that use of DAP enabled them to get that "additional production" which is very vital during peak seasons in the fertilizer business. Properly executed, this action can add a nice profit to any operation.

Formulation 16-20-0

Raw Material	Wet Weight
Lion Solution 5	547
Ammonium Sulphate	53
Diammonium Phosphate	303
Normal Superphosphate	
20.0 7.0	545
Triple Superphosphate	
46.0 6.0	279
Sulphuric Acid 66	341
Theoretical Water	155
Total Wet Weight	2,222
Total Dry Weight	1,997
Cost Per Ton	$53.08

Formulation 16-20-0

Raw Material	Wet Weight
Lion Solution 5	385
Ammonium Sulphate	399
Diammonium Phosphate	303
Normal Superphosphate	
20.0 7.0	391
Triple Superphosphate	
46.0 6.0	345
Sulphuric Acid 66	223
Theoretical Water	104
Total Wet Weight	2,151
Total Dry Weight	2,000
Cost Per Ton	$54.95

Formulation 11-48-0

Raw Material	Wet Weight
Diammonium Phosphate	650
Triple Superphosphate	
Phosphoric Acid	338
Lion Solution 6	198
Theoretical Water	50
Total Wet Weight	2,150
Total Dry Weight	1,997
Cost Per Ton	$89.77

Formulation 13-39-0

Raw Material	Wet Weight
Anhydrous Ammonia	103
Lion Solution 1	312
Diammonia Phosphate	225
Phosphoric Acid	477
Normal Superphosphate	
20.0 8.0	347
Triple Superphosphate	
46.0 5.0	719
Theoretical Water	83
Total Wet Weight	2,266
Total Dry Weight	2,000
Cost Per Ton	$67.78

Formulation 15-15-15

Raw Material	Wet Weight
Anhydrous Ammonia	68
Lion Solution 3	588
Diammonium Phosphate	128
Phosphoric Acid	174
Normal Superphosphate	
20.0 8.0	97
Triple Superphosphate	
46.0 5.0	256
Potash 600	500
Sulphuric Acid 66	335
Theoretical Water	128
Total Wet Weight	2,273
Total Dry Weight	2,001
Cost Per Ton	$52.98

Formulation 15-15-15

Raw Material	Wet Weight
Anhydrous Ammonia	29
Lion Solution 3	603
Diammonium Phosphate	252
Normal Superphosphate	
20.0 8.0	36
Triple Superphosphate	
46.0 5.0	342
Potash 600	500
Sulphuric Acid 66	342
Theoretical Water	109
Total Wet Weight	2,213
Total Dry Weight	2,001
Lost Per Ton	$53.57

What, if any, work has been done on use of diammonium phosphate as a base goods for the small mixer? (Physical, chemical considerations.)

T. P. Hignett

TVA has, and is, carrying out extensive tests on the use of diammonium phosphate as a base goods in dry-mixed (blended) fertilizers and has studied its use in the production of high-analysis granular fertilizers. Two papers have been published[1],[2], and additional information is being developed because continued interest by industry in using diammonium phosphate in a wider range of mixtures brings up new problems. Our work has shown that diammonium phosphat is compatible with commonly used fertilizers, and that it can be used as a base to make a variety of grades of mixed fertilizer of good physical properties. Some of the mixtures prepared and tested in our laboratory are discussed below.

Mixtures of diammonium phosphate with ammonium sulfate and potassium chloride (10-20-30, 10-20-20, and 15-15-15) were satisfactory when conditioned with 3 per cent of kaolin clay, kieselguhr, or calcined dolomite. The mixtures conditioned with 3 per cent of kaolin clay or kieselguhr had medium bag sets after 1 month but no lumps after the bags were dropped. Only very light bag set was observed when calcined dolomite was used as the conditioning agent.

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[1] "Storage Properties of Diammonium Phosphate Alone and in Admixture with Other Fertilizer Materials." Julius Silverberg and F. G. Heil. *J. Agr. Food Chem.* 5, 436-41 (June 1957).

[2] "Use of Diammonium Phosphate in Production of Granular, High-Analysis Fertilizers." T. P. Hignett, G. C. Hicks, and J. E. Jordan. *Com. Fert. and Plant Food Ind.* 93, No. 4, 24-6 (October 1956).

Mixtures of diammonium phosphate with ammonium nitrate and potassium chloride (10-20-30, 12-24-24, and 20-20-5) tended to cake more than did mixtures containing ammonium sulfate and potassium chloride. Calcined dolomite cannot be used as a conditioner for these mixtures because of its reaction with ammonium nitrate to release ammonia and to form calcium nitrate and water.

With these mixtures, about 3 per cent of kieselguhr gave satisfactory storage. Kieselguhr was somewhat more effective than kaolin clay since, generally, about 10 per cent of the latter material was required. Best results were obtained and less conditioner was required in most tests in which coarse instead of fine potash was used. The 20-20-5 product is produced by several mixers in fairly large quantities for use as a fish pond fertilizer.

Mixtures of diammonium phosphate, calcium metaphosphate, and potassium chloride (10-30-20, 8-24-24, 6-18-36, and 4-24-24) had practically no bag set and no caking when conditioned with calcined dolomite. These mixtures had medium bag sets when conditioned with 3 per cent by weight of kaolin clay.

When diammonium phosphate was mixed with concentrated or ordinary superphosphate, there was a chemical reaction which resulted in some ammoniation and generation of heat. There was no loss of plant nutrient. However, the mixtures tended to set up while "fresh." Conditioning the mixtures or permitting them to cure for about a week before bagging or both prevented caking in storage. Satisfactory results have been obtained with this combination of raw materials for the following grades: 6-24-24, 5-20-20, 8-24-24, 6-18-36, 10-30-20, 5-15-30, and 9-27-18. Calcined dolomite was found to be an excellent conditioner for mixtures that did not contain ammonium nitrate.

Diammonium phosphate, as produced by TVA, is a fairly coarse crystalline material containing 21 per cent N, 53.7 per cent P_2O_5, and 0.1 per cent moisture. Its hygroscopicity is about the same as that of ammonium sulfate and potassium chloride since it has a critical humidity of about 85 per cent at 86° F. Diammonium phosphate will cake in bags or in bulk over a period of months unless treated with a conditioner. Bulk material formed a firm cake in a 20-foot pile but was easily reclaimed with a power shovel. Conditioning diammonium phosphate with 1 to 3 per cent of calcined dolomite prevented bag set and caking and was somewhat more effective than kaolin clay or kieselguhr. Calcined dolomite also was very effective as a conditioner for mixtures that did not contain ammonium nitrate. For mixtures containing ammonium nitrate kieselguhr was somewhat more effective than kaolin clay.

Experimental work is being continued to identify other grades that can be advantageously made with diammonium phosphate.

Discuss the Merits of Ammoniated Superphosphate vs. Ammonium Phosphate Based Fertilizer.

G. L. Terman

Agronomically speaking, the merits of ammoniated superphosphates versus ammonium phosphate based fertilizers are largely in relation to solubility, especially water solubility, of the phosphorus component. Ammonium phosphates, either as straight materials or in mixtures, are largely water soluble. High water solubility of the phosphorus is frequently advantageous on acid soils to push early growth(to help avoid a weed problem, etc.) and, under some conditions and crops, to increase yields. On calcareous soils, there usually is a yield advantage of high water soluble over low water soluble phosphates.

There is also some evidence that ammonium ion stimulates early growth of some crops such as small grains. This may result in greater utilization of phosphorus from ammonium phosphates or mixtures containing ammonium salts.

Ammoniation of concentrated superphosphate, a high water soluble phosphate, and subsequent formulation into high analysis grades seldom cause the water solubility to be reduced below 40 to 50 percent. The components of low water solubility formed in the process are largely dicalcium phosphate and apatites. Little or no difficulties in the availabilities of CSP based and ammonium phosphate based grades can usually be measured in experiments in which the crops are grown to maturity.

High ammoniation of ordinary superphosphate has been found to reduce water solubility of the phosphorus to as low as 10 to 15 percent. The phosphates of low water solubility formed during ammoniation have also been identified petrographically as dicalcium phosphate and apatites. Because of the presence of gypsum, much larger amounts of these phosphates of low solubility are formed than in the case of CSP, which contains little or no gypsum.

Marked decreases in availability to crops with increase in degree of immoniation of superphosphates have been found in greenhouse experiments. This is true for both fine and granulated fertilizers. In one experiment conducted by TVA at Wilson Dam, ammoniated ordinary superphosphates were compared in which 70, 50, 30, and 15 percent of the phosphorus was water soluble. Uptake of phosphorus and yields by wheat were highly correlated with amount of water soluble phosphorus applied. Marked decreases in early growth and appreciable decreases in final yields have also been found in field experiments in the Southeast.

One interesting finding in our experiments is that phosphorus in the material identified as calcium phosphate in the ammoniated ordinary superphosphates is apparently much less available than the phosphorus in commercial dicalcium phosphate. The reason for this is not apparent, and further studies are being made.

Have Ureaforms been Found Suitable for Use in Formulations of Complete Goods—Powdered? Granulated? What Specific Conditions Should Be Met to Assure Stability of Ureaforms in Fertilizer Mixtures?

C. E. Waters

IT is assumed that this question refers primarily to solid ureaforms, manufactured and sold as separate products. The use of materials to make ureaform right in the mixture is taken to be outside of the scope of the present question. Yes, ureaforms have been used in formulations of complete goods, with considerable success. As far as we know, all such formulations have been for specialty fertilizers, a 10-6-4 lawn fertilizer being typical.

The mixtures known to us are mostly powdered, though sometimes a ureaform, which is somewhat granular, is mixed with a base that has been granulated separately. We know of no case in which a solid ureaform is added to the mixture of ingredients before granulation.

I assume the term "stability" as applied to ureaform means that the product does not react so far that the nitrogen becomes unavailable. To assure such stability, it is best to avoid hot, moist storage, as well as high acidity. Just to illustrate the effect of temperature, a fertilizer, approximately 10-10-11, was made in the laboratory by adding a good solid ureaform to a previously made base, so that three units of nitrogen came from the ureaform. A high A.I. was found for the water-insoluble nitrogen in the mixture, namely 64%. After one month at room temperature, the A.I. was 54, and the same value was found seven months later. After a month at 50°C. (122° F.), the A.I. had fallen to 24 and after 7 months more at this temperature it was only 12. Rather surprisingly, during storage for eight months the cold-water-insoluble nitrogen increased by only 0.2 unit.

K. G. Clark

THE generic term urea-form is used to denote urea-formaldehyde reaction products of low solubility suitable for fertilizer use as a nitrogen source. Other urea-formaldehyde reaction products also exhibit low solubility but are not properly classed as urea-forms owing to their high resistance to bacterial decomposition in soil media.

Both urea-form and nonurea-form type urea-formaldehyde reaction products result from acid catalyzed reactions between urea and formaldehyde and are chemically related in that both types are polymethylene polyureas. Urea-forms, however, exhibit urea to formladehyde mole ratios >1 which limits their structure to straight or branched-chain compounds, whereas nonurea-form materials have U/F mole ratios <1 and consequently are cross-linked polymethylene compounds.

Unlike ammonium sulfate, ammonium nitrate and other chemical nitrogen materials, urea-forms as produced and marketed do not represent single chemical components. Rather they are mixtures of several components capable under the proper conditions of temperature, moisture and acidity of condensing to higher polymers with an increase in the uncombined urea content and decreases both in the water-insoluble nitrogen content and in the activity index of the product. Fortunately these condensation reactions are inhibited by neutralization of the acid catalyst and cooling and drying the product. Such a product remains stable throughout extended storage periods. The undesirable condensation reactions will proceed slowly as the acidity and moisture content are increased at normal temperature and somewhat more repidly if the temperature also is increased.

Urea-form has been found suitable for incorporation in powdered mixtures when about 100 pounds of dolomitic limestone per ton of product are added with the urea-form. The quality of the urea-form in mixtures of this type with moisture contents of about two percent and pH values as low as 5.5 has not decreased significantly in more than 200 days storage at room temperature. Likewise a commercial mixture, reportedly formulated so that the reaction between urea and formaldehyde occurred during ammoniation, showed little if any reduction in the quality of its water-insoluble nitrogen content after storage for more than a year under similar conditions of moisture content, pH and temperature. Successful use of a concentrated urea-formaldehyde solution together with solid urea to produce urea-form during ammonation of N-P-K- mixtures also has been reported.

In view of the above it would appear to be feasible to produce either granular or powdered products containing urea-form. When the urea-form is produced in place during the ammoniation and granulation stages it may be necessary to use a somewhat higher urea to formaldehyde mole ratio of reactants than otherwise would be required to obtain a fixed activity index. When urea-form is introduced as a solid component entering the ammoniation-granulation process some reduction in the activity index may be expected owing to the unfavorable conditions temporarily encountered. However, the undesirable condensation reactions are relatively slow compared to ammoniation and granulation so that an excessive decrease in activity index would not be anticipated providing the product is rapidly cooled and dried to 100°-120° F. and about 2 percent moisture. The degree of ammoniation and the inclusion of dolomitic limestone in the formula should be sufficient to insure that the pH of the product will not be below 5.5.

It should be emphasized that owing to the lack of direct experimental evidence on the ammoniation-granulation of urea-form containing mixed fertilizers the conditions suggested above should be considered as tentative.

Has the Accepted Availability Index (A. I.) Method for Predicting the Response of Ureaforms Been Found Satisfactory When Applied to Ureaforms in Mixed Fertilizer?

C. E. Waters

STRICT interpretation of the note, "Applicable to urea-formaldehyde compounds only," in the book of "Official Methods of Analysis of the A.O.A.C." would not allow the application of the A.I. test to mixed fertilizers. However, the phrase might be construed to permit the testing of mixed fertilizers in which ureaform is the only source of water-insoluble nitrogen. Although the collaborative test run by the A.O.A.C. included no such product, the original presentation by Morgan and Kralovec included data on mixed fertilizers, one of which contained only 6% water-insoluble nitrogen.

The size of sample required for the determination of hot-water-insoluble nitrogen can cause serious difficulty. This sample must contain 0.3 gram of water-insoluble nitrogen (W.I.N.). If the fertilizer analyzes 3% W.I.N., a 10-gram sample is required, and this is quite manageable. If only 1% W.I.N. is present, a 30-gram sample must be taken, and this is hard to handle. Besides, it may seriously change the pH of the buffer solution.

We have only scant information as to the actual use of the A.I. test by industrial and governmental laboratories in analyzing mixed fertilizers. There is also little or no information on the correlation between A.I. tests on mixed fertilizers and actual response of plants to the nitrogen contained in them.

At present there is no accepted method for testing mixed fertilizers containing both ureaform and natural organics. The A.O.A.C. neutral permanganate method is used to some extent for want of a better one, but some chemists hold it to be unreliable. In some cases there is also substantial interest in distinguishing between the part of the water-soluble organic nitrogen that represents free urea, and that which represents water-soluble reaction products of urea and formaldehyde. A separate determination of free urea nitrogen by the urease procedure is helpful in such cases.

W. A. Morgan

THE "accepted method" refers to article 2.38, p. 16 of the A.O.A.C. Methods of Analysis, 8th Ed., applicable to urea-formaldehyde compounds only, for determining the activity of the water-insoluble nitrogen.

The principle of the method is the difference in solubility of the U-F material in cold water (25° C.), and in hot water (98°-100° C.). A fixed quantity of sample is specified for the two determinations. The amount of sample used for the hot water digestion depends upon the amount of cold-water-insoluble nitrogen found.

From a limited number of experiments with laboratory samples of mixed fertilizers in which all of the cold-water-insoluble nitrogen was derived from U-F material, approximately the same A.I. was found as in the U-F material used. When the mixture also contained U-F material with a low A.I., the A.I. was lowered considerably. This provides a means of detecting the addition of insoluble U-F resin scrap in such mixtures. For example, a 10-6-4 mixture containing 23% of a U-F material, A.I. 52.8 (35 mesh), gave A.I. = 50.6. When the mixture was changed to 17.6% of the U-F material and 10.3% of the 3 A.I. material, found A.I. = 40.4, a reduction of 10 A.I. units.

So far as I know, a satisfactory application of this method has not been made to fertilizers that contain cold-water-insoluble nitrogen not derived from U-F material. The weight of sample used for the cold-water-insoluble nitrogen should contain at least 0.5 g. of the U-F material. Additional work on mixed fertilizers is certainly desirable.

Problems Connected With the Use of Sulfate of Potash for Granulating

R. A. MacDonald

THERE is no particular problem using sulfate of potash in fertilizer mixtures. Most of these products are essentially neutral and water solutions tend to be neutral.

Sulfate of potash tends to undergo base exchange when incorporated, in the presence of water, with other material quite the same as potassium chloride. The products of this exchange are generally less hygroscopic than products of KCl. The physical size of potassium sulfate can be somewhat of a problem since surface area is often a limiting factor in exchange reactions in fertilizer.

There are no special reasons why potassium sulfate cannot be incorporated into a granular fertilizer.

The second question: Is it possible to use sulfate of potash like muriate "to seed" granules in granulation processes.

When granular particles are needed to assist in the granulation of fertilizers, it may be convient to supply them with granular sulfate of potash. There is no indication that the kind of raw material supplied in granular form has any unusual effect on granulation.

In using granular sulfate of potash it would be well to select a material in the size range needed to build the proper sized finished granule taking into consideration the formulation for the grade.

Do Certain Formulations Have a Better Influence on Effecting Heat of Reaction and Assisting Granulation in the Ammoniator?

Rodger C. Smith

THERE are factors which influence the heat of reaction and assist granulation in the ammoniator. I will use "formulation" in its broader sense to include grades and ratios.

The manufacture of high-analysis grades is basically necessary for granulation to be practical. This is readily appreciated, I am sure.

Likewise, granulation is more readily accomplished with ratios high in nitrogen—those of 1-2 N-P_2O_5 ratio or higher. In the narrow N-P_2O_5 ratios in high-analysis grades, there is adequate amount of nitrogen to enable inclusion of suitable levels of ammonia and nitrogen salts. Four forms of nitrogen comprise the nitrogen ingredients used in most granular mixtures—ammonia, ammonium nitrate, urea and in lesser quantity ammonium sulfate.

In formulation, it is important to use sufficient ammonia which reacted with superphosphates and the necessary amount of acid will cause a mixer temperature of at least 180° F. and 220° F. To attempt granulation at lower temperature requires unnecessarily high level of moisture which adds to the size of drying equipment necessary and the cost of its operation. Attainment of this temperature can be controlled largely through the choice of nitrogen solution or use of anhydrous ammonia to utilize fully the ammoniation capacity of the superphosphates and a reasonable amount of acid. Additional heat can be obtained through excess use of sulfuric acid. This is impractical, however, for continued operation because heat can be provided in a dryer more economically than by excess use of sulfuric acid.

Attainment of 180° F. or higher in the mixer dissolves salts present adding greatly to the liquid phase. Agglomeration with the aid of dissolved salts is much more satisfactory than with high levels of water. During drying and cooling, agglomeration and hardening of granules is more rapidly accomplished with resultant efficiency of operation.

This is not too say that the importance of moisture can be minimized. With high liquid phase, the moisture level is still important, no doubt more so. Adequate moisture must be provided to insure a state suitable to agglomeration sufficiently long to attain a desirable range of granule size.

The nitrogen salt or salts formulated affect the granulation. We have, and the industry in general has, experienced largely with ammonia-ammonium nitrate solutions. The high solubility and steep solubility curve appears to give this compound an advantage in contributing to granulation.

Ammonia-urea solutions can be and are used in granulation. Equipment of larger size and greater range of drying and cooling capacity appears to me to be required. The "Urana" solutions containing very limited amounts of urea for the purpose of depressing the crystallizing point appear to be satisfactory for granulation. Solutions containing substantial amounts of urea along with ammonium nitrate are not satisfactory.

The source of potash affects the liquid phase, sulfate of potash being less soluble than muriate. Addition of certain minor element carriers in appreciable quantity such as borate increases the liquid phase.

Formulation is of first importance to granulation efficiency, yet the miracle material or combination of materials has not been introduced.

Discuss Pros and Cons of Phosphoric Acid Versus Sulfuric Acid in Granulation.

Rodger C. Smith

IT can be said at the outset that both sulfuric and phosphoric acid (furnace and wet process production) can be successfully employed in mixed fertilizer granulation. The use of sulfuric acid in granulation has been much more general than other acids because of general availability. Only recently has very widespread use been made of phosphoric acid.

In choosing between the two acids, perhaps the first consideration is the grades manufactured or contemplated. Phosphoric acid enables the production of more concentrated grades for the simple reason that phosphoric acid accomplishes two purposes, that of phosphorus carrier and an agent for neutralizing ammonia. 100 pounds 65% phosphoric acid provides the same amount of P_2O_5 contained in 100 pounds triple superphosphate plus the neutralizing capacity of 70 pounds 93% sulfuric acid. The effect on space in the formula is obvious.

The adaptability of phosphoric acid to various ratios varies with the proportion of nitrogen to phosphorus. The higher the phosphorus in relation to nitrogen, the greater the adaptability of phosphoric acid from the viewpoint of ammoniation capacity. Specifically, the use of phosphoric acid in a 1-1-1 ratio or higher in nitrogen is difficult with the nitrogen solutions normally used. The ammoniation capacity of phosphoric acid is greater than that of superphosphates but its use reduces the poundage of the superphosphate, the net effect is less ammoniation capacity. With sulfuric acid use, its neutralizing capacity is additives with that of the superphosphate.

At this point, it is possible to evaluate cost of ingredients for the grades manufactured or contemplated. As stated earlier, it is my opinion that the principle advantage of phosphoric acid is that of materials cost. With current prices of the two acids, it is likely that phosphoric has some cost advantage in most locations. Recent new production facilities further improve the cost advantage.

An important difference between the two acids is their heat of neutralization and heat of dilution since there is a substantial difference in concentration of the two acids as normally used. The temperature of the fertilizer as discharged from the mixer is somewhat less with the phosphoric acid. To offset this lower temperature and resultant lower amount of salt in solution, granulation must be accomplished at slightly higher moisture. These differences are not great, as much as 1% higher moisture being required, but they are a factor. Comparable increased dryer capacity is required to dry to the product moisture found necessary, in our case 1½%.

This discussion assumes the use of ortho-phosphoric acid. If the superphosphoric acid, which is a combination of ortho-phosphoric acid and pyro-phosphoric acid should become commercially available, then heat-moisture relationships would be considerably different. Those of you attending the T.V.A. demonstration observed the rapid agglomeration and crystallization when using the 76% P_2O_5 superphosphoric acid.

Another practical difference observed in operation of granulation plants is the amount and nature of fumes. The yellowish-brown nitrous oxide fumes which accompany uneven distribution of ammonium nitrate solutions and sulfuric acid in either batch or continuous mixers are noticeably absent when using phosphoric acid. Working conditions in the area of the mixers are substantially improved. No mixer fires have occurred, to my knowledge, when using phosphoric acid only.

In summary, both acids have a place in our granulation work. If there is any appreciable cost advantage with phosphoric acid at your location, its consideration appears to be in order.

Is it Possible to Replace X Percent of Triple Superphosphate with Fortified Superprosphate in Granulation Processes? Granulation With Triple Superphosphate Doesn't Seem to Be the Answer.

James E. Seymour

THEORETICALLY and practically it is possible to replace triple superphosphate with enriched or fortified superphosphate.

Chemically, the enriched or fortified superphosphate is identical to the mixture of triple superphosphate and normal superphosphate required to produce a phosphatic mixture equivalent in P_2O_5 content.

Physically existing differences are that the enriched or fortified superphosphate particles are homogeneous, and for practical purposes identical with one another with respect to chemical composition whereas the triple superphosphate—normal superphosphate mixture is merely a physical blend of two chemically varying components.

Theoretically the excess gypsum of normal superphosphate can be utilized by monocalcium ortho phosphate or essentially triple superphosphate to enhance its ammonia absorption or holding capacity. I assume that a homogeneous granule or particle containing the chemical equivalent of a mixture of triple superphosphate and normal superphosphate would do a better job of utilizing the excess gypsum than the physical blend of triple superphosphate and normal superphosphate.

Generally, the surface area of the solids of reaction mixture is one of the primary factors determining the amount of liquid phase required for granulation. Dense solids of low permeability require less liquid phase for granulation. Dense phosphatic reaction mixtures of a low order of permeability are also difficult to ammoniate. It is therefore mandatory to effectively manipulate the process for the production of the enriched or fortified superphosphate to give the density and permeability essential for optimum economics with respect to both granulation and ammonia absorption.

The economic value of triple super-normal super replacement is dependent upon the price of the phosphatic ingredients at a location and the degree of replacement.

SECTION 2B

How Does One Determine That a Given Plant is Operating at an Optimum Granulation Level?

Robert B. Smith

1. Using maximum amount of solution possible consistent with good formulation and a product of good physical appearance. Solution at present is most economical source of nitrogen.
2. Keeping acid usage within bounds, bearing in mind that acid added above that required adds to the over-all cost without contributing to value of the product.
3. Keeping recycle at the very minimum consistent with good formulation and with keeping the final product in good physical condition. There appears to be at least two schools of thought on this subject, those who believe in plenty of recycle to soak up the excess liquids even going to the extreme of grinding acceptable product to create fines. We subscribe to the second school, which believes that we should try to produce as much acceptable product on the first pass through the equipment as possible. We know it costs money to granulate and if a product makes 2 or 3 passes through the process before it lands in the storage bins that does not appear to be economical production. We also feel that it is extremely important that fines should be returned to the process as quickly and as consistently as possible. Otherwise, the analysis will be affected.
4. Controlling oversize; it would appear that reducing oversize is one of the bottlenecks in producing granular fertilizer. If it is allowed to get out of hand the mills plug, making it necessary to remove material from the system, which immediately affects the analysis or the operations come to a halt.
5. Watching the moisture in the finished product; we like to have the moisture below 1½% when the acceptable product goes into storage. We have experienced bin set and difficulty with excessive fines in the final bagged product when moistures are 2% or above. During ammoniation we like to have the temperature of the mixture at 200° to 225° F. which in turn helps to keep the moisture at the very minimum, making it easier on the drying equipment and the pocketbook to reach the desired moisture level of 1½% or below.
6. Keeping track of the analyses; each grade should meet the guarantee with a little to spare, but it should be borne in mind that excessive over-runs in analysis cost money. We penalize our plants for over-run on analysis just as severely as we do for under-run.
7. Classifying the product; the product should have the fines removed and should not have granules of sufficient size to plug fertilizer distributing equipment. Farmers are interested in obtaining a uniform product that will stay in condition, work well in their equipment and will meet the guaranteed analysis, no more no less. He is not interested in paying extra money for fancy bags, extra special uniform granules, etc. He does not intend to admire the fertilizer or display it. He intends to use it for only one purpose, namely, to produce more profitable yields of farm commodities.

If a plant will meet all of these specifications, then in our opinion, that plant is granulating at optimum efficiency.

The Pros and Cons of Using Steam In Granulation

T. J. Bosman

STEAM at the present time is a necessary nuisance in our 1-4-4 ratios and sometimes in our 1-2-2 ratios. Its main use of course is to offset the cooling effect of the water that has to be used in formulas of these ratios.

It will cause a build up on your sparger, although not to the degree that an unbalanced ammoniation rate with phosphoric acid would do. Then it costs money to generate steam. We have a Clayton Boiler rated at 100 H.P. at each of our three granulation plants.

Generally when we start granulating a 1-4-4 ratio, the recycle gets out of hand before we get on stream. When this happens we have to increase the recycle input. To keep this from making still more recycle we have to add more water. The more water added the cooler the material becomes, so before long we would have a wet mass, like wet sand, instead of the plastic granular particles that we desire. To stop this condition we turn on steam which brings the temperature up and lets us get some granulation. In a way more recycle and more water creates a vicious circle that is hard to overcome, even by the addition of steam. This could go on to the point where some recycle might have to be by-passed to finished product.

At times we get on stream soon after starting. On such occasions we do not have to use so much water. When we can keep the water low enough to maintain a temperature 220° F. or above in the ammoniator then the necessity for steam is overcome.

L. Hein

GRANULATION always requires the proper proportion of liquid and solid phase. Liquid phase is usually increased in high analysis grades by heat released from chemical reactions. The higher temperatures resulting from this heat, increases the solubility of salts and therefore increases the amount of liquid phase present. The result is the promotion of granulation.

In low nitrogen grades the heat released by chemical reactions is so small that insufficient liquid phase is present in the mixture. Therefore, it is necessary to increase the amount of liquid phase or to utilize solid granules as raw material so as to decrease liquid phase requirements.

An increase in liquid phase can be obtainable by either increasing the amount of liquid used in the formulation, adding heat, or both. For example, increasing the amount of water in the formulation is effective. The water may be added as free water or by the use of raw materials containing higher moisture. More effective would be the use of hot water sprayed directly into the ammoniator. This increases the liquid present and adds heat. The heat introduced by water is limited to that amount which can be carried as sensible heat. Much more heat can be introduced per unit of moisture by the use of steam. For example, with the temperature in an ammoniator at 150° F. one pound of steam will introduce about 20 times as much heat as one pound of water at 200° F. The over-all effect for the use of steam is granulation at lower moisture than with hot or cold water with the resulting decrease in drier load. The disadvantages to steam are the investment and costs involved in producing it. Sulfuric or phosphoric acids are also used to increase the liquid phase. The effects are an increase in liquid and heat, and possibly an alteration of the physical character of the liquid.

Another method of promoting granulation of low nitrogen grades is to use a granular raw material.

Publications show that granular potash is effective for this purpose, apparently the granules of potash act as seeds or nuclei for the build up of the other ingredients. The liquid phase required is greatly decreased because the potash is already granular and therefore, needs no liquid phase for cementing into granules.

Discuss the Advantages of Using a Granulating Drum as Against a Pugmill for Mixing and Introducing Solutions

Walter J. Sackett, Sr.

THE opinions I am about to express on this subject are based on the following assumptions:

A. That the scope of the question is limited to batchless processing.

B. That the granulating drum referred to in the question is a T.V.A. type Ammoniator.

C. That the word "mixing" relates to the mixing of preblended solids with liquids.

In our work of designing and fabricating contiuous processes for the Fertilizer Industry, we have employed the pugmill as well as the T.V.A. type ammoniator. Our selection as to which machine we will use in any specific case is largely influenced by the nature of the processing to be accomplished. For example, in building what may now be considered the conventional process of producing granular mixed fertilizers, in which the mass remains in the solid state, we use the T.V.A. ammoniator because of its operating simplicity and its inherent low power and maintenance expense. The T.V.A. ammoniator gives us a uniform introduction of liquids into the cascading mass of powdered blend so long as care is exercised in formulation and operation to avoid excessive plasticity of the mass.

On the other hand, however, there are other processes in which the T.V.A. ammoniator cannot too well be adapted. Slurry-to-plastic and plastic-to-solid phase processing, such as we use in the production of nitraphosphatic fertilizers, falls into this category. Here, we need the vigorous agitation as well as the positive discharge the pugmill gives us, even though very substantially higher power and maintenance expense must be expected in its operation.

In conclusion, we believe each machine has its advantages as well as its limitations. In short, each, we feel, has its own job to do.

J. E. Reynolds, Jr.

THIS discussion will present the advantages and disadvantages of using a double shafted pug mixer for mixing and granulating mixed fertilizers as compared to the ammoniator-granulator drum. It is recognized that other types of shafted mixers are often grouped synonomously in the classification of a pug mixer. However, for this discussion, the pug mixer described refers only to the double shafted pug mixer. Several desirable features of the double shafted pug mixer are given in the following paragraphs.

The Davison-Trenton Process is a Davison double shafted pug mixer. The liquid distribution manifold for submerged introduction of all liquids is shown along the front side of the mixer, and the pre-mixed dry raw materials poido-meter is shown on the right. The fertilizer materials move from right to left with the recycle portions introducted at the feed end.

One of the foremost features of the pug mixer is its ability to achieve positive action agglomeration while mixing dry and liquid raw materials. The raw materials are continuously agitated and mixed during the ammoniation operation. The paddles "chop" and "shear" the semi-slurry mass. This type of action further reduces the tendency for large lumps to form, or for localized over or under wetting conditions to exist. It is believed that a more homogeneous mixed fertilizer is also possible with a pug mixer. The pug mixer is also versatile enough to handle formulas containing a large liquid phase, thus permitting extreme flexibility in the choice of raw materials. The mixing and granulation function of the pug mixer is accomplished through controlled recycle conditions which maintains the total liquid phase at its optimum level.

The premixed dry raw materials are first mixed with the returned portions, and the resulting mixture is then combined with the formulated liquids. The liquids are introduced at three or four submerged locations conveniently spaced down the middle of the mixer. The distribution pipes are brought from an outside manifold which permits isolation of any piping system while operating. The feature of optional distribution pipes is desirable due to any potential mechanical failure, or the desire to maintain a predetermined rate of liquid flow into the mass of material, even though the total volume of liquid has been reduced.

The advantages outlined above can be summarized as contributing to a reduction in operating costs through fewer interruptions which permits a high daily production output. The labor, maintenance, power, fuel, and certain other expense items represent a relatively fixed number of dollars per operating day. These dollars directly affect the cost of sales as an increase or a decrease in the cost per ton, depending on the daily production output of the plant. The use of a pug mixer also permits wider flexibility in the choice of the most economical formulations.

The most controversial considerations confronting a person who is choosing between a granulating drum and a pug mixer are: (1) the horsepower requirements, (2) the initial cost of the two mixers, and (3) the maintenance and lost production costs associated with the operation of the two mixers. As a basis of comparison, let us review these costs for a 30-ton per hour unit.

	Pug Mixer (3' x 4' x 16')	Ammoniator-Granulator-Drum (7' x 15')
Connected Horsepower	100	40
Cost Not Installed (Motor Included)	$18,000	$13,500
Electrical	1,500	500

The increased capital investment for a pug mixer as compared to an ammoniator-granulator is estimated at $4,000-$5,000. This represents approximately 2% of the total cost of a granulation unit. When this sum of money is proportioned over ten (10) years, the indirect cost is $.01 per ton on the basis of 40,000 tons of yearly production.

The simultaneous consideration is to determine the power cost for operating the ammoniator-granulator versus the pug mixer. This extra power cost for the pug mixer is in the range of $.08-$.10 per ton of product.

During the past five (5) years, much information has been gained concerning the techniques of operating a pug mixer. The problems of maintenance and "downtime" were the first to be recognized. Improvements in the design of the paddles and the choice and location of distribution pipes has materially aided in reducing the operating costs. The pug mixer has more potential maintenance spots, due to the use of paddles which are subjected to corrosion and erosion conditions. The cost for maintaining the mixer and the paddles is approximately $.03-$.04 per ton of product. The other maintenance costs such as distribution pipes are considered comparable for the two mixers.

The above extra costs of purchasing and operating a pug mixer must be justified, or otherwise the pug mixer would not be selected. The evaluation of the relative merits of any mixer must include the investment costs, the operating costs, the formula costs, and the other tangible and intangible advantages of the mixing unit. The conclusion should be based on the return on investment, rather than separate items which add to or subtract from the total number of advantages.

Rodger C. Smith

Our direct experience with a pug mill type of mixer is with a one ton capacity batch mixer having fixed shell, center shaft and sigma blades. Observation has been made of several different types of paddle mixers used continuously for ammoniation in both granulation and pulverized fertilizer production.

As to ammonia absorption, much depends in each unit upon the solution or ammonia inlet pipes and operation of the mixers. Adequate distribution is necessary. Furthermore, the U.S.D.A. and others have repeatedly demonstrated the importance of time in relation to reaction of ammonia with superphosphates. About 4 minutes is required before the rate of uptake appreciably decreases. With either of these mixers that time is seldom accomplished. On ammoniation, it is our experience, after several years' operation, that ammonia take-up can be very similar between the rotary drum mixer and an enclosed pug mixer having fixed shell and sigma blades. Paddle mixers used in continuous mixing in a few plants are inferior, according to the information and observations which I have made. Time for ammoniation is inadequate and thoroughness of contact between ammonia and solid or liquid materials is lacking.

As to mixing, the drum such as a Stedman rotary mixer is a batch unit where the pug mill can be either batch or continuous. In batch operation, they both do satisfactory mixing assuming reasonably uniform particle size of ingredients and other conditions favorable to good blending. In continuous mixing, uniformity of blend depends upon the uniformity of ingredient feed to the mixer.

The power requirement of pug mixers is greater than rotary batch mixers. In our enclosed Besser mixer, which I include in the classification of pug mixers, cleaning is negligible and maintenance is normal. With paddle mixers, my observation has been that both cleanup and maintenance are excessive.

For pulverized fertilizer production, I consider the rotary drum preferable to a pug mixer. For use in granulation, both are inferior to the T. V. A. continuous ammoniator.

W. W. Whitlock

One of the most important arguments for the pug mill is the homogeneity of the product. With more and more higher analyses and closer governmental scrutiny, particle homogeneity points toward better on-grade analysis. By the same token, a greater liquid phase and higher permissible temperatures in the pug mixer allow for more completion of both primary and secondary chemical reactions.

Bed control on the pug mixer is easier inasmuch as there is not the critical liquid-to-solid phase constantly cycling as in the granulating drum mixer.

The pug mixer allows greater flexibility as to location of recycle.

Construction and maintenance costs generally will be somewhat higher in the pug mixer than in the granulating drum.

Control of liquid ingredients is more difficult in the granulating drum because of the other pressing problems of sparger sizing, sparger build-up, and proper distribution of liquids. The criticalness of sparger location, size of holes, etc., is minimized in the pug mill.

Power requiremennts can be expected to be greater in the pug mill.

Ingredient cost generally should be lower in the pug mixer because of the ability to utilize more nitrogen from nitrogen solutions.

Particle size is generally easier to control in the pug mixer; however, more recycle is usually needed. The granulating drum mixer will require much closer sizing of ingredients for good operations and product analyses. Proponents of the pug mill mixer regard the angular particle shape to be better, while those favoring the drum mixer argue for the round particle. More information is needed on the flowability and drillability of these different shapes of particles. The pug mill mixer is likely to impart a more dense particle.

Space requirements in an existing plant would indicate an easier adaptability for the pug mill mixer.

Operating is a solution or similar slurry state, the pug mixer probably will require more particle drying and cooling. Generally speaking, in the granulating drum, more water is lost in process than in the pug mixer.

The above mentioned points are somewhat controversial and intended to reflect the pros and cons of these mixers, as reflected in discussions with many fertilizer men throughout the country. They are to create a basis for discussion, rather than a firm argument for either one of the other types.

Comparative Advantages—Pug Mill Vs. Granulation Drum

	Pugmill		Granulating Drum Mixer	
	Pro	Con	Pro	Con
Homogeneity	best			
Chemical Reaction	most complete			
Bed Control	easier			
Recycle Flexibility	location easier			
Construction Costs		higher	lower/ton	
Maintenance Costs			lower/ton	
Liquid Control				sparger a problem
Power Requirements		greater		
Ingredient Cost	more solution			
Particle Size	easier to control	more recycle needed		ingredient size critical
Particle Shape	more angular		rounder	
Particle Density	more dense			
Space Requirements	easier adapted			
Drying		more drying needed	more water lost in process	
Cooling			less cooling required	

The Analysis of Granulating Fertilizers Made in Systems Using Continuous Feeding of Solid and Liquid Materials Often Shows Wide Discrepancies; Can These be Reduced by Improvements in Processing and How?

F. T. Nielsson

The discrepancies are caused usually by (1) feeders and meters improperly calibrated or out of calibration; (2) the use of improper materials in the formula; (3) improper operating technique and (4) the production of small tonnages of one grade at a time. The following paragraphs expand each of the preceding statements.

1. Although solids feeders are of the gravimetric, or weighing, type, those of the belt type apparently have a slightly different rate of delivery for each type of material. Not only does this show up when changing from, say sulfate, of ammonia to muriate of potash, or when changing from a 5-20-20 base to a 12-12-12 base, but also when changing from cured super to green super. Feeders must be calibrated initially over enough points to be able to plot a graph of delivery against feeder setting for each type of material to be handled by the feeder. Periodically, one or more points must be checked to determine if the curve has shifted. Checking can be done simply by timing the flow of a large quantity of weighed material from a surge hopper. The more feeders being used, the greater the probability of one of the feeders being out of calibration.

Most plants use rotameters to meter the liquids entering the process. These meters probably were purchased to handle a liquid at a definite specific gravity and viscosity. In the case of nitrogen solutions, as various solutions are used, a meter factor must be established for each solution that has a specific gravity different from the one for which the meter was purchased. In addition, the effect of viscosity becomes important when metering acids. Viscosity, or the degree of "syrupiness" increases as the temperature decreases, and is expressed in centipoises. A given rotameter float will operate satisfactorily over a fairly wide range of viscosity. However, when the reference viscosity is exceeded, large meter errors develop, especially at the lower range of the scale where the float is close to the walls of the tapered tube used in rotameters. It is necessary to obtain a curve from the supplier of acid showing the effect of temperature on viscosity and prepare a calibration curve for the acid meter if you are to operate in ranges outside the reference viscosity. For example, when using furnace phosphoric acid, the actual flow at 25°F is about 10 per cent less than that found at 80°F at the same float reading.

2. Urea solutions tend to yield small granules, ammonium nitrate solutions tend to yield large granules while urea-ammonium nitrate solutions yield granules in between the other two types. All anhydrous formulae tend to yield large, irregular granules. The desired particle size of your product may determine the type solution you should use to get the most onsize material. The use of a feed of large particle size results in excessive oversize. Excessive oversize results in excessive production of fines with large recycle.
3. Some operators do not follow the given formula, especially in regard to the use of sulfuric acid. If a formula is not granulating properly, they will keep adding acid until the desired degree of granulation is reached. Because the excess acid acts as a solid diluent, the final product is offgrade if the extra acid is not a part of the formula.

   It is well known that recycle and product are of a different analysis. Some operators tend to accumulate recycle and then stop the raw material feed while they granulate the recycle and remove it from the system. Such a procedure results in a pile having layers of different analysis.
4. Because recycle and product are of different analysis, the first part of a run is deficient in the components that are high in the recycle, and the last part of a run when the recycle is being run out is high in those same components. The longer the run, the greater the portion of ongrade material, and in large tonnages the offgrade beginning and end portions are not significant. In small tonnages the offgrade portion may be a large part of the product pile. Cleaning the recycle out of the system at the end of an operating period has the same effect as running a small tonnage of that grade.

J. E. Reynolds, Jr.

The control of the flow of all fertizier materials is vital to the successful operation of any granulation process. The successful proportioning of the correct ratio of solids to liquids is reflected not only in the analyses of the finished product, but in the degree of granule formation. This discussion will be divided into: (1) preparing the dry mixed raw materials, (2) storage and feeding from the surge hopper, and (3) checking the accuracy of the flowmeter equipment.

1. *Preparing the Dry Mixed Raw Materials.*

The solid raw materials can be brought into the continuous process either by continuous weighing or by batch weighing. Due to the simplicity of scales and the ease of maintaining accurate weights, a batch operation is standard for the Davison-Trenton process. This batching system may either be controlled by an automatic repeating time cycle or by manual operation. In any event, it is desirable to mix the dry raw materials systematically on a regular time sequence.

The pre-weighed dry raw materials are mixed in a rotary drum mixer. The length of time required for mixing the dry ingredients has been determined, and to maintain uniformity, a time cycle system has been adopted for this operation.

The thoroughly mixed dry raw materials are then elevated to the pug mixer surge hopper.

2. *Storage and Feeding from the Surge Hopper.*

The flow of the pure-mix dry batch is regulated by a system of level indicating lights and a poidometer which discharges material at a constant rate. The surge hopper will usually hold 5-8 tons of dry material. The size of the hopper is designed to provide sufficient reserve capacity to forestall any interruption in the continuous operation. The hopper is, however, small enough to discourage "un-mixing" or a holdup of the dry batch due to packing, bridging, or caking.

The surge hopper is a transition section which converts a batch system to a continuous system. It is most unsatisfactory to operate the surge hopper on a mixing cycle which permits the level of material to fluctuate between the top light indicator and the bottom light indicator. A fairly constant level is desirable. The poidometer operation provides an indicating reference of the cubic feet of material which is discharged from the hopper. A correlation between (1) the number of batches recorded at the batching scale, (2) the level of material in the surge hopper at the start and end of a certain check period, (3) the cubic feet of dry materials calculated to pounds, and (4) the number of equivalent batches of liquid materials for the check period are used to verify the accuracy of the liquid and solid mixing system.

3. *Checking the Accuracy of the Flowmeter Equipment.*

The amount of liquids which has combined with the solid ingredients should be periodically checked. Experience has shown that most flowmeter inaccuracies are repetitous. If a deviation from the indicated scale of flow is determined, then the defined per cent of drift can be interpreted as a correction factor for the meter scale. The usage can be checked by isolating a known quantity of liquid, and determining the equivalent number of tons produced before the quantity is depleted. This operation should be repeated for calibration accuracy.

In the case when liquid storage is available, the rate of flow from the tank can be determined by gallon or pound relationships for the volume between the two depths for a certain mixing time. Sight glass accessories are recommended.

The above systems are based on inventory checks. Periodically, the flowmeters should be calibrated with a known quantity of water, which is weighed after passage through the meter. The flowmeter can be removed and taken to a test rack in the shop, or provided with necessary water piping to and from the meter. A curve of the liquid flows at different rates should be determined.

All liquid facilities are equipped with pumps for positive movement.

Adequate flow control equipment is also provided to compensate for any possible restriction or interruption of liquid flow. Pressure gauges to indicate distribution pressure are also provided.

Pros and Cons of Phosphoric Acid Versus Sulphuric Acid In Granulation

T. J. Bosman

SULPHURIC acid on the whole will make agglomeration somewhat easier than the use of phosphoric acid as it gives higher reaction temperatures.

The granules are larger but not as hard.

In 1-4-4 formulas water still has to be added but not to the extent that it does with phosphoric acid.

Sulphuric acid is easier to handle.

Phosphoric acid does not give the heat of reaction that sulphuric acid does. This makes it harder to agglomerate low nitrogen grades, but is an aid in high nitrogen formulas where the tendency is toward over agglomeration.

In 1-4-4 ratios it makes a smaller granule than with the use of sulphuric acid, but the granule is much harder. There is less tendency for the granules to bind together, therein making an easier blowing material.

Where only small quantities of phosphoric acid are used, it may become necessary to use some sulphuric acid as an aid to granulation. This could become necessary even with larger quantities of phosphoric where you vary from the 7.4# ammoniation rate.

Where you can maintain the 7.4# rate and do not have to use sulphuric acid it is more economical to use. You get double value, available P_2O_5 and neutralizing value.

Phosphoric acid is much harder to handle than sulphuric. A change in temperature causes such a change in viscosity that it throws your instrument off. Victor Chemical Company furnished us with a correction curve for temperature changes that works fairly well with furnace acid, but not with wet process acid. Even then, we measure our storage tank from time to time to check against our instruments.

With wet process acid you have the further complications of solid impurities. Although these solids help in agglomeration, they do not help in instrumentation. We have had times when our lines have choked off completely. Then they had to be torn down, washed out and reassembled.

A better type instrument no doubt would help. It would let us get more accurate data.

Potash Granules are said to Act as Seed in Production of Granules or Pellets. Can Other Granular Materials such as Granulated Superphosphates, or Nitrogenous Compounds Act Similarly? Any Data? Relate These to the Several Existing Processes and to High and Low Nitrogen Grades

J. O. Hardesty

ANY solid particle may act as "seed" when its surface is wetted with a solution or slurry. Some fertilizer granulation processes (*1*, *14*) depend on repeated wetting of undersize—or seed-granules with a solution or slurry, drying the resulting coating, and repeating the operation until the granules have grown to the desired size. The desired size of granule passes over the screen to product and the undersize granules return to the process. Particle growth is well-controlled in such processes and each granule has, virtually, the same composition. However, with processes in which the initial ingredients are predominantly solids, agglomeration or growth of the granule depends on the aggregation of small, solid, particles through contact of their wetted surfaces to form stable granules. Control of such processes on the assumption that one kind of ingredient particle acts as a seed for collecting the other solid ingredients of the mixture into stable aggregates does not seem feasible.

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F. T. Nielsson

"Seeding" is a term used in crystallization process when the crystals salt out of solution on solid nuclei already present in the solution. In a granulation process, granulation is a combination of salting out and agglomeration. This combination is affected by the liquid phase present. To be effective the liquid phase must have some thickness. With many fine particles present, the liquid phase is spread so thin over the surface area of the particles that it is not effective in promoting agglomeration. Large sulfate of potash particles therefore will have the same effect as large muriate of potash particles in providing more liquid phase for effective granulation. Granular triple also increases the effective liquid phase. Sulfate of potash has a secondary effect in that the sulfate reacts with the calcium of the phosphate to yield calcium sulfate. The formation of calcium sulfate during the granulation process appears to increase the degree of caking in the ammoniator and drier.

Richard G. Powell

UNFORTUNATELY, at this time, we have not fully collected data for a quantitative answer to this question. All we can say right now is yes—other granular material under proper conditions, can be used for promoting granular products. We have used granular superphosphate and coarse to granular triple superphosphates in both batch and continuous operation in conjunction with acid and solutions for producing a wide variety of grades. In low analysis grades, such as 3-9-12, 3-9-18, etc., we have also used limestone filler of about 14 mesh in conjunction with solution and acid and were able to aid the granulation. However, we have never attempted to appraise this on a quantitative or economic basis to see how much the use of such granular raw materials aided granulation of the final product.

What Are the Factors Involved in the Segregation of Granulated Fertilizers?

J. O. Hardesty

TRULY granulated fertilizers do not segregate. Segregation becomes apparent only as the mixture approaches a state in which it is composed of individual granular materials rather than a granulated mixture having uniform distribution of plant nutrients.

The discussion of the above questions has been based on normal conditions, procedures, and results observed in applied research and in commercial practice. It is recognized that the rules of practice and the results obtained are very likely to vary in accordance with local variation in such factors as temperature, humidity, storage conditions and trade requirements. All such factors must be carefully considered in formulating and processing present-day fertilizers.

Can Granulation be Carried out in the Usual Ammoniating Equipment Using Nitric Acid as the Acidulant?

T. I. Horzella

WE do not have plant experience using nitric acid in the TVA ammoniator or the paddle mixer, however, we would anticipate a positive answer with the following comments.

The reasons why acids are used in the granulation of fertilizers are mainly the following:

(1) The heat of reaction evolved during neutralization of the acids with ammonia enhances granulation. For some grades, addition of steam to the ammoniator has the same effect as the formulation with sulfuric or phosphoric acids. If this heat of reaction between the acid and ammonia is the main factor, nitric acid would be suitable.

(2) Sometimes in order to obtain low nitrogen grades, it becomes necessary to use either ammonium sulfate or phosphate. Since it is not practical to transport and handle these salts in solution like ammonia nitrate or urea solution it is self explanatory why sulfuric and phosphoric acids and ammonia are used to make the salts directly in the ammoniating equipment. These raw materials are almost as concentrated as the solid salts and the cost of manufacturing the solid ammonium sulfate and phosphate are eliminated.

In the case of nitric acids the outlook is different:

(1) The heat of reaction would be available as in the case of sulfuric and phosphoric acids, however it is anticipated that some corrosion and air pollution problems would arise while handling nitric acid in the ammoniator. Nitric acid reacts easily with potassium chloride, forming highly corrosive mixtures and undesirable nitrosil chloride gas.

(2) Transportation and handling of nitric acid is not as simple as that of sulfuric or phosphoric acids, especially because of the lower concentration at which it is commercially available.

In a more general case we would answer the proposed question, stating that if nitric acid should become available to fertilizer manufacturers the TVA nitrophosphate processes would be practical and highly desirable. Most or all of the P_2O_5 would be used in the form of phosphate rock and only small amounts of sulfuric or phosproric acids would be required to prevent the formation of the hygroscopic calcium nitrate that is likely to appear in these processes.

Discuss Process of Producing a Granulated Fertilizer Without Subsequently Drying?

Ray Sorenson

WE feel that we have had excellent results in producing a granulated fertilizer without subsequent drying. However, this does not mean that we would not install a dryer if our fertilizer plant and tonnage were larger or our product ran more to the higher nitrogen grades. Because of our careful control of formulas, our larger cooler and our double screening process, we feel that our fertilizer is as good as any being produced by plants with special dryers in the process lines. However, it is likely that our formulation costs are higher on some grades because of the additional sulphuric acid required to produce sufficient heat for drying in our cooler. I doubt that a gas or distillate fired dryer would reduce the overall cost in our size of operation. If our annual production were twice as high a separate dryer might be a profitable investment.

Is it Possible to Granulate Satisfactorily P-K Grades? How Can it Be Done?

T. P. Hignett

IN the granulation of fertilizer grades that contain nitrogen the reaction of ammonia with superphosphates or acid plays an important part in providing conditions that are favorable to granulation. This reaction is not available as an aid to granulation of P-K grades. Granular P-K grades are usually made by granulating potash salts with superphosphate or mixtures of superphosphates. In our first experiments we sought to granulate these materials by using the TVA continuous ammoniator as a rotary granulator. Water or water and steam were added in sufficient quantity to cause granules to form. The granulation efficiency was fairly good, but the granules were weak and dusty. A high percentage of moisture was required for granulation which made the drying step difficult and expensive.

In seeking to overcome these difficulties, adding phosphate rock and sulfuric or phosphoric acid to mixtures of superphosphate and potassium chloride was tried. This method proved to be quite beneficial; the granulation efficiency was improved, the granules were stronger, and the moisture content was so low that little or no drying was necessary. These improvements were attributed to the heat of reaction of phosphate rock and acid and to the plasticity of the fresh superphosphate formed in the granules. The reaction of the acid and rock was rapid and substantially complete in a short time.

A good example of the granulation of no-nitrogen grades in the pilot plant is a series of runs on 0-14-14. The first run was made at a rate of 1.4 tons per hour. The formulation per ton of product was 720 pounds of cured superphosphate, 438 pounds of phosphate rock, 326 pounds of sulfuric acid (66° Be.), 150 pounds of water, and 466 pounds of potassium chloride. The acid and water were injected under the bed through parallel spargers similar to those used for ammoniating solution and acid. The proportions of acid and water were equivalent to 64 per cent H_2SO_4.

No recycle was used in this run. Granulation was good; 70 per cent was onsize as granulated, and 86 per cent was onsize after crushing the oversize. The product was cooled but not dryed. The granules after curing overnight were firm, hard, and free flowing; the P_2O_5 availability was 95 per cent.

In a second run, 0-14-14 was made without using cured superphosphate. The formulation was 888 pounds of phosphate rock 613 pounds of 66° Be. sulfuric acid, 140 pounds of water, and 425 pounds of potassium chloride. The granules were cooled, but not dried, and screened; the minus 15-mesh fines were recycled. About 46 per cent of the throughput was recycled. Granulation was controlled to produce this amount of fines for recycling by varying the water input. The combination of recycle and control of water input was used to control granulation. The product contained about 4 per cent moisture and appeared to be quite satisfactory.

In another run, 0-14-14 from one of the previously described runs was ammoniated the next day to produce granular 6-12-12. The formulation was 1607 pounds of 0-14-14, 324 pounds of ammoniating solution, and 62 pounds of sulphuric acid. A well-granulated, ongrade product was produced, containing only 3.4 per cent water without drying. The degree of ammoniation was held at 3.0 pounds per unit because attempts to increase it resulted in high ammonia loss.

Other no-nitrogen grades that were produced successfully in the pilot plant included 0-20-20 and 0-26-26.

In bag-storage tests of the P-K products that had been cured 1 week, bag rot was experienced when no neutralizing agent was added prior to bagging, but addition of pulverized limestone or calcined dolomite prior to bagging was effective in preventing bag rot.

This method of making no-nitrogen grades is the most satisfactory and economical method that we know of. We also believe that the production of such materials for later use as a base material in ammoniation would be attractive, particularly for low-nitrogen grades such as 4-16-16 or 5-20-20 in which a granular ingredient is needed to assist in granulation.

Some problems might be encountered in some large-scale units. Raw material feeders in some granulation plants may not be well adapted to feeding ground phosphate rock at an accurately controlled rate. The gases evolved from the granulator and cooler are corrosive to mild steel ducts. The gases contain fluorine and hydrochloric acid. Some sort of protective coating might be suitable for preventing corrosion of the ducts. No corrosion of the process equipment was noted. To prevent atmospheric pollution, the gases should be scrubbed before discharge to the atmosphere. There was no evidence of corrosion of the ammoniator, granulator, or other process equipment in the pilot-plant runs. Presumably a coating of fertilizer material protected the equipment from corrosion.

If a dryer is used, the moisture content should not be reduced below about 4 per cent because lower moisture contents will prevent development of maximum citrate solubility.

We do not know of any case in which this method is being used by industry to make granular P-K grades, but we suspect that it is being used by some plants which prefer not to disclose their methods.

C. E. Floyd

I simply incorporated 100# of fine ground phosphate rock dust in the mixture and fed this mixture into the Ammoniator Granulator, adding enough water to cause agglomeration. Very good granulation was achieved; also, the free acids in the R.O.P. and Concentrate react with and make available about ⅓ to ½ of the P_2O_5

in the rock dust. The conversion takes place in the dryer. No acid was used as this will cause over agglomeration and a very sticky mass, which will gum dryer, screens, belts, etc.

The product should be dried to a residual moisture content of about 2%. Dried to this moisture content, the product is of sufficient hardness to insure good carrying qualities.

Discuss Method of Successfully Granulating Grades of Low Analysis, Such as Low Nitrogen Grades Prevalent in the Southeast

A. E. Henderson

A granulating low analysis goods and particularly low nitrogen grades is difficult because of the low percentage of soluble salts present, insufficient reacting vessel temperature and presence of inert materials, such as filler, which tend to retard agglomeration.

Generally speaking, considerably more H_2O must be added to produce the plasticity necessary for granulation and more drying is necessary because of the higher initial moisture requirements. Granulation can be achieved by use of excess acids, steam, and H_2O.

Probably the most promising method presently suggested is production of non-nitrogen grades by partial acidulation in the reactionary vessel. An example is TVA's pilot plant production of O-14-14 in the Continuous Ammoniator.

The most serious objection to this method will obviously be corrosion; however, I am sure it is less severe than the manufacture of superphosphate as a separate step.

Almost any grade can be granulated by use of H_2O, steam, excess H_2SO_4, and by partial or even complete acidulation of phosphate rock simultaneously with basing. In most cases, the cost will increase probably in the range of $.25 to $2.00 per ton. However, under certain circumstances it is probable that certain grades may even be produced at a lower cost. Let's assume that a new plant was built in an area where the predominant grades were 0-14-14, 4-12-12, 4-10-7, 8-8-8, and 2-1212. All of these grades could be produced by ammoniating O-14-14 and granular single superphosphate which would have been previously manufactured in the Continuous Ammoniator. Equipment generally used for superphosphate would be eliminated. Of course, double handling is necessary, however, the second step (ammoniation) would be simple and relatively inexpensive.

F. T. Nielsson

The difficulty in granulating successfully low nitrogen, low analysis grades is in generating enough heat during reaction to effect a high level of liquid phase. Several approaches are possible.

1. Steam permits granulation using nitrogen solution with a low acid requirements. Not many plants have steam and not enough work has been done using steam to evaluate the process adequately.
2. A fairly general method is to increase the anhydrous content of the ammoniating solution. This in turn requires the use of sulfuric acid to neutralize the excess ammonia that does not react with the phosphate. Some plants use varying proportions of anhydrous ammonia and of a basic solution like Spensol B to arrive at the right combination .for a given grade Other plants use the high free ammonia solutions like Spensol D and R to get the desired degree of heat with sulfuric acid.
3. Most low analysis grades require the use of some filler. Better results are obtained if fine limestone instead of sand is used.

General Discussion

A. E. Henderson: I'll try to summarize it. You need three things — heat, moisture, and soluble salts. Of course, in a low nitrogen grade, low analysis mixture, those three factors are at a minimum. As Mr. Nielsson has already said several times, you can granulate anything with enough sulphuric acid, so if we use an excess of sulfuric acid, we, of course, can granulate that type of mixture. I think probably one of the best approaches to this sort of thing is to use all anhydrous, if you're granulating a 2-12-12 or a 3-12-12, or a 4-12-12, something in that neighborhood, use all anhydrous and enough hot water or steam to granulate the mix, and I think that essentially answers the question. Of course, there is something that has recently been added and that is the TVA idea of manufacturing an O-14-14 from sulphuric acid, phosphate rock, and potash, and then later ammoniating that mixture. I asked the question late yesterday evening about what type of filler to use with this type of mixture. I feel a little bit confused about that and I think some more work would have to be done before we would really nw. But I do think that's a good idea. We will have some trouble in this industry and I presume with new materials of construction on the market constantly that we might be able to lick that one.

Mr. Spillman: Dr. Nielsson, you commented on this question, do you have any further comments on granulating low analysis formulas?

Mr. Nielsson: The only thing I would add is that if you use limestone instead of sand you usually get better results because acid and limestone react. If you'll add about 25 pounds of 60° Be acid for 100 pounds of limestone, you'll get remarkably better results than if you use sand as a filler on this low grade. And the other thing is that there has been a little bit of work on the fact that clay does help a little bit and if you start off with about 50 pounds of clay per ton, you start getting results, but there are very few places where you can afford clay because that stuff is going to cost you two to three cents delivered. But there have been tests made under controlled conditions starting off clay about 50 pounds per ton does do you a little bit of good.

I don't know, Al, did you talk about steam?

Mr. Henderson: I mentioned that. You can mention it again, though.

Mr. Nielsson: No, no, its just one of those things that there hasn't been enough done with steam to have any good data on it. From what I can gather, more and more people are beginning to use steam, and I think some people have tried this, and if any of you want to know what steam will do for your place, I think most of you have these steam jennies that are used to clean off tractors. They'll give you 700 pounds of a mixture of steam and hot water and if you connect one of these steam jennies up to your ammoniator, why, you can run a test at about five to ten tons an hour, you'll get enough hot water from your steam jenny where you can begin to tell the difference. And if you promise the maintenance man that you're not going to tear it all to pieces, he'll probably let you use it.

Mr. Spillman: At our Baltimore plant where we have a small tonnage of low analysis goods we use a little clay and it seems to help our granulation quite a lot.

Can Cost Data be Given Relative to: Formulation vs. Capital Investment; Total Cost vs. Recycle Rate; Rate of Production vs. Bulk and Bag Storage?

Joe C. Sharp & Paul C. Parker

THIS is a complex question. There are so many variables involved that it is necessary to make many assumptions and to arbitrarily specify conditions for the data to be of any use at all. Even then, plant management and planning can alter the data in either direction.

To answer the question a hypothetical plant producing 40,000 ton per year is arbitrarily chosen. To establish trends, two cases are examined: Case I where 25% of the production is recycle grade 12-12-12. It is assumed that all other grades can be produced at minimum recycle rates. That is, only fines that fail to reach on size on the first pass through is recycled. Case II where 50% of the tonnage is 12-12-12. Formula No. 1 represents a condition where the liquid phase in the reactor is controlled by recycle. Formula No. 2 represents a condition where the liquid phase in the reactor is controlled by formulation.

The prices shown in the formulas are current raw material prices with the following freight rates and delivered prices:

Material	Freight Rate	Delivered Price
Ammoniating Solution	$8.00	$57.50
Sulphate of Ammonia	$8.00	$41.00
Sulphuric Acid	$4.00	$27.00
Triple Superphosphate	$12.00	$58.00
Normal Superphosphate (Purchased)	$6.00	$24.20
Normal Superphosphate (Produced using spent sulfuric acid)		$15.00

Formula No. 1 12-12-12

Raw Materials	Pounds Dry Basis	Pounds Wet Basis	*Cost Per Pound	**Cost Per Pound	Cost Per Ton	Cost Per Ton
SPENCOL C-7	555	580	0.0287	0.0287	$16.65	$16.65
Sulphuric Acid	146	155	0.0134	0.0135	2.10	2.10
Muriate of Potash	400	400	....	....	....	....
Conditioner	30	30	....	....	....	....
Triple Superphosphate	203	216	0.029	0.029	6.26	6.26
Normal Superphosphate	666	710	0.0075	0.0121	5.32	8.60
Total	2000	2091			$30.33	$33.61

Formula No. 2 12-12-12

Raw Materials	Pounds Dry Basis	Pounds Wet Basis	*Cost Per Pound	**Cost Per Pound	Cost Per Ton	Cost Per Ton
SPENSOL C-7	364	387	0.0287	0.0287	$11.08	$11.08
Sulphuric Acid	58	62	0.0135	0.0135	.84	.84
Ammonium Sulphate	386	386	0.0205	0.0205	7.90	7.90
Muriate of Potash	400	400	....	....	....	....
Conditioner	30	30	....	....	....	....
Triple Superphosphate	290	310	0.029	0.029	9.00	9.00
Normal Superphosphate	472	503	0.0075	0.0121	3.77	6.08
Total	2000	2078			$32.59	$34.90

*Normal Superphosphate made with spent sulphuric acid.

**Normal Superphosphate purchased.

Formula No. 1 will require a 175% recycle rate to control the conditions in the ammoniator. In Formula No. 2, 30% of the material will fail to reach on size on the first trip through the reactor.

In this hypothetical plant it is assumed that the peak shipping season lasts 28 days.

FORMULATION VS. CAPITOL INVESTMENT

For this study a plant is sized to produce the entire 40,000 tons in 100 working days with an average of 20 production hours per day, when granulation by formulation is used. The size of the plant is then increased so that the same tonnage may be produced in the same period controlling granulation in 12-12-12 with recycle.

Case I — 25% of Production is 12-12-12

Basis:

Production: 40,000 tons Total—10,000 tons 12-12-12
Time: 100 working days @20 hours per day = 2,000 hours)
Recycle: Formula No. 1 — 30% on 12-12-12
Formula No. 1 — 175% on 12-12-12

Size of Plant:

$$\frac{40{,}000 \text{ tons}}{2{,}000 \text{ hours}} = 20 \text{ tons/hr. production}$$

With 30% recycle, equipment must handle 130% of production.
20 tons/hr. × 130% = 26 tons/hr.
With 175% recycle, effected equipment must be enlarged.
Size adjusted to permit 40,000 tons in 2,000 hrs. = 34 tons/hr.

Capital Costs:

Table I — Cost of Additional Equipment

Equipment	26 Ton Rate	34 Ton Rate	Difference
Rector—Size	6′ x 8′	6.5′ x 9′	$ 400
Cost	$15,000	$5,400	$3,000
Dryer—Size	7′ x 35′	8′ x 40′	
Cost	$12,000	$15,000	$3,000
Cooler—Size	7′ x 35′	8′ x 40′	
Cost	$12,000	$15,000	$3,000
Screen	$5,000	$7,000	$2,000
Hopper		$1,000	$1,000
Gravimetric Feeder		$3,000	$3,000
			$12,400
		Contingencies	2,000
			$14,400

Pay-Out Calculations:

	Normal Superphosphate Purchased	Normal Superphosphate Produced with spent acid
Formula Savings 10,000 Tons @ $1.29	$12,900	@ $2.26 $22,600
Increased Capital Investment (from Table 1)	$14,400	$14,400
Pay-out (Before Taxes)	1.2 years	.64 year

Case II — 50% of production is 12-12-12

Basis:

Production: 40,000 tons Total—20,000 tons 12-12-12
Time: 100 working days @20 hours per day = 2,000 hours)
Recycle: Formula No. 1 — 30% on 12-12-12
Formula No. 1 — 175% on 12-12-12

Size of Plant:

$$\frac{40{,}000 \text{ tons}}{2{,}000 \text{ hours}} = 20 \text{ tons/hr. production}$$

With 30% recycle, equipment must handle 130% of production
20 tons/hr. × 130% = 26 tons/hr.
With 175% recycle, effected equipment must be enlarged.
Size—adjusted to permit 40,000 tons in 2,000 hrs. = 40 tons/hr.
12-12-12—15 tons/hr.; other grades—31 tons/hr.)

Table 2 — Cost of Additional Equipment

Equipment	26 Ton Rate	40 Ton Rate	Difference
Reactor—Size	6′ x 8′	7′ x 10′	
Cost	$5,000	$5,800	$ 800
Dryer—Size	7′ x 35′	9′ x 45′	
Cost	$12,000	$19,000	$7,000
Cooler—Size	7′ x 35′	9′ x 45′	
Cost	$12,000	$19,000	$7,000
Screen	$5,000	$9,000	$4,000
Hopper		1,200	$1,200
Gravimetric Feeder		$3,000	$3,000
			$23,000
		Contingencies	3,000
			$26,000

Pay-Out Calculations:

	Normal Superphosphate Purchased	Normal Superphosphate Produced with Spent Acid
Fromula Savings 20,000 Tons @ $1.29	$25,800	@ $2.26 45,200
Increased Capital Investment	$26,000	$26,000
Pay-Out (Before Taxes) (from Table 2)	1.01 years	.58 year

TOTAL COST VS. RECYCLE RATES

An approach that will direct some light to this problem is to assume that the 26 tons per hour throughout is already fixed and that storage will not be increased. In such an event, control with recycle would be employed until the peak season (assumed to be 28 days) at which time formulation control would be used.

Case I — 25% of Production is 12-12-12

Basis:

Production: 40,000 tons Total—10,000 tons 12-12-12
Time: Assume 28 day peak season @ 20 hrs./day = 560 hours)
Production Rate: 20 tons/hour @ 30 % Recycle Rate
9.5 tons/hour @ 175% Recycle Rate

Production:

Total Production =40,000 Tons

Produced during peak season, 560 hours × 20 tons/hr. = 11,200 Tons

Produced prior to peak season = 28,800 Tons

28, Tons X 25% = 7,200 Tons 12-12-12

7,200 Tons = 758 hours required for 12-12-12 @ low cost formula rate 9.5 tons/hr.

7,200 Tons = 360 hours required for 12-12-12 @ high cost formula rate 20 tons/hr.

398 extra hours needed = 20 days extra

Production Costs:

Labor—6 men @ $1.50/hr. × 24 hours = $216.00 per day

Utilities = $5.30 per day

Production Costs = $221.31 per day

Pay-Out Calculations:

	Normal Superphosphate Purchased	Normal Superphosphate Produced
Formula Savings, 7,200 Tons @1.29	$9,288	@$2.26 $16,272
Added Production Costs, 20 days at @$221.30	$4,426	$ 4,426
Net Operating Savings	$4,862	$11,846
Increased Capital Investment (from Table 1)	$4,000	$ 4,000
Pay-Out (Before Taxes)	.82 year	.34 year

Case II — 50% of Production is 12-12-12

Basis:

Same as Case I except that production is 20,000 tons of 12-12-12 instead of 10,000 tons.

Production:

20 extra working days required (Case I) × 2 = 40 extra days

Pay-Out Calculations:

	Normal Superphosphate Purchased	Normal Superphosphate Produced with Spent Acid
Formula Savings, 14,000 Tons @ 1.29	$18,600	@ $2.26 $32,600
Added Production Costs, 40 days @ $221.30	$ 8,852	$ 8,852
Net Operating Savings	$ 9,748	$23,748
Increased Capital Investment (from Table I)	$ 4,000	$ 4,000
Pay-Out (Before Taxes)	.41 year	.17 year

PRODUCTION RATE VS. STORAGE

In studying production rate vs. storage costs, production during peak season seems to be the question. It is assumed that by using formulation control, production schedules can be met. That is, enough storage is already provided to take care of the difference in what can be produced during the 28 day period, and annual sales. Calculating pay-out on additional storage from the savings using recycle should be indication of whether additional storage is justified.

In figuring storage costs, a building 80 feet wide, with a cone shaped pile 30 feet deep with a 60 foot base is used. This allows a 20 foot aisle-way. These dimensions provide a 30 ton per linear foot storage, or 0.375 ton per sq. ft. including the aisle-way.

Case I—25% of Production is 12-12-12

Basis:

Production: 40,000 tons Total — 10,000 Tons 12-12-12

Time: Assume 28 days peak season @ 20 hrs./day = 560 hours)

Production Rate: 20 ton/hour @ 30% recycle rate
9.5 tons/hour @ 175% recycle rate

Storage Facilities Required:

560 hours × 20 tons/hr. = 11,200 tons produced during peak season

11,200 tons × 25% = 2,800 tons 12-12-12 produced during season

2,800 Tons = 140 hours required to produce 12-12-12 at high cost formula rate

140 hrs. × 9.5 tons/hr = 1,330 tons produced during peak season @ low cost formula rate

2,800—1,330 = 1,470 tons produced prior to peak season. Therefore, storage space is needed for 1,470 tons.

1,470 tons needed = 3,920 sq. ft. needed

.375 tons/sq. ft.

3,920 sq. ft. @ $4.00 = $15,680 storage cost

Production:

10,000 tons = 1,050 hours required to produce 12-12-12

9.5 tons/hr. @ low cost formula rate

10,000 tons = 500 hours required to produce 12-12-12

20 tons/hr. @ high cost formula rate

1,050—500 = 550 hours extra = 27.5 days extra

Production Costs:

Labor—6 men @ $1.50/hr. × 24 hours = $216.00 per day

Utilities = $5.30 per day

Production Costs = $221.30 per day

Pay-Out Calculations:

	Normal Superphosphate Purchased		Normal Superphosphate Produced with Spent Acid
Formula Savings, 10,000 Tons @ $1.29	$12,900	@ $2.26	$22,600
Added Production Cost, 27.5 days @ $221.30	$ 6,096		$ 6,096
Net Operating Savings	$ 6,804		$16,504
Added Storage Facilities	$15,608		$15,608
Increased Capital Investment (Table I)	$ 4,000		$ 4,000
Total Capital Expeditures	$19,680		$19,680
Pay-Out (Before Taxes)	2.9 years		1.2 years

Case II — 50% of Production is 12-12-12

Basis:

Same as Case I except that production is 20,000 tons of 12-12-12 instead of 10,000 tons.

Storage Facilities Required:

$15,680 storage cost (Case I X 2 = $31,360

Production:

27.5 extra days (Case I) X 2 = 55 extra days

Pay-Out Calculations:

	Normal Superphosphat Purchased		Normal Superphosphat Produced with Spent Acid
Formula Savings, 20,000 tons @ $1.29	$25,800	@ $2.26	$45,200
Added Production Costs, 55 days @ $221.30	$12,192		$12,192
Net Operating Savings	$13,608		$33,008
Added Storage Facilities	$31,360		$31,360
Increased Capital Expenditure (Table I)	$ 4,000		$ 4,000
Total Capital Expenditure	$35,360		$35,360
Pay-out (Before Taxes)	2.9 years		1.2 years

SUMMARY AND CONCLUSIONS

Pay-outs have been calculated based on an annual capacity of 40,000 tons of which (Case I) 25% is 12-12-12 and (Case II) is 12-12-12, with a 28 day peak season and the following delivered prices of raw materials:

Material	Analysis	Delivered Price
Ammoniating Solution	41.4% N	$57.50 per ton
Sulphuric Acid 66° Be'		$27.00 per ton
Ammonium Sulphate	21.1% N	$41.00 per ton
Triple Superphosphate	46.0% P_2O_5	$58.00 per ton
Normal Superphosphate (Purchased)	20.0% P_2O_5	$24.20 per ton
Normal Superphosphate (made with spent sulphuric acid)	20.0% P_2O_5	$15.00 per ton

(1) Formulation vs. Capital Investment
Assuming that prior to converting to granulation, a decision is to be reached whether to install equipment that will permit the use of recycle to control liquid phase in the reactor, the pay-outs on the cost of additional equipment are:

	Normal Super Purchased	Normal Super Produced
Case I	1.12 years	.64 year
Case II	1.01 years	.58 year

(2) Total Cost vs. Recycle Rates
It is assumed that the capacity of the plant has already been fixed. Since the equipment sizes cannot be increased, the use of recycle to control liquid phase in the reactor must be done at the lower production rate. It is further assumed that storage capacity will not be increased. Calculations are made showing the pay-outs achieved by using recycle control until the peak season, and formulation control thereafter.

	Normal Super Purchased	Normal Super Produced
Case I	.82 year	.34 year
Case II	.41 year	.34 year

(3) Production Rate vs. Storage
It is again assumed that the capacity of the plant has already been fixed, the same as (2) above, but in this case additional storage will be added to accomodate the slower rates of production necessary with recycle control. The pay-outs for the added equipment and storage facilities are:

	Normal Super Purchased	Normal Super Produced
Case I	2.9 years	1.2 years
Case II	2.9 years	1.2 years

Based on this hypothetical plant, which should be fairly representative, economics are in favor of liquid phase control with recycle in all cases.

J. E. Seymour

Unfortunately data of a universal nature relative to this question are not available. Ethical considerations preclude our disclosure of data from specific customer operations.

It is probable that individual problems of this nature (for a specific plant and location) can be solved by correlating and evaluating the correlation of the following factors.

1. Available market—total tonnage and grade percentage of the total tonnage.
2. General process for fertilizer manufacture.
3. Process requisites and general process technique employed.
4. Recycle capacity.
5. Sizing capacity.
6. Drying capacity.
7. Labor cost.
8. Availability of anhydrous ammonia.
9. Availability and cost of phosphoric acid.
10. Availability and cost of other fertilizer ingredients.
11. Availability, capacity and cost of steam and/or hot water.
12. Bagging capacity, and the relationship to manufacturing capacity.
13. Physical and chemical properties that give best results for:
    a. bagged storage
    b. bulk storage
14. Cost and location of bagged storage facilities.
15. Cost and location of bulk storage facilities.
16. Ambient conditions (humidity, temperature, etc.).
17. The degree of "gold plating" of various installations of the plant.

The following constitutes an illustrative example of total cost—recycle rate relationships existing based on two levels of production cost per ton of product and I.B.M.-702 formulae of various recycle values:

Case 1—25% of Production is 12-12-12

Assumption:
Production cost/ton of product at 27 ton/hr. rate = $3.00
Production cost/ton of product at 20 ton/hr. rate = $4.00
Grade: 12-12-12
Formula #1 50% recycle
Formula cost per ton = $40.36
Formula #2 100% recycle
Formula cost per ton = $39.56
Total Cost per ton of product for formula #1 = 40.36 + 3.00 = $43.36.
Total Cost per ton of product for formula #2 = 39.56 + 4.00 = $43.56.

Thus it is obvious that production cost per ton of product must be determined at each rate of production to be compared. The production cost per ton of product plus the formula cost per ton of product constitutes the total cost per ton of product. The foregoing illustrative examples indicate that the operation with the lower cost formula will be more expensive than operation with the higher cost formula.

What Minimum Ammoniation Temperature is Recommended for Manufacturing Granulated Fertilizer to Produce the Hard Strong Granule That Will Not Bag Cake?

W. G. Mautner

First of all, it is believed that the minimum material temperature required for efficient granulation is approximately 200° F. This temperature applies to the wet granular material as discharged from a TVA ammoniator. Although the granulation temperature is not the complete answer to the production of hard granules, the material must be partially agglomerated before drying. The product moisture and conditioning prior to drying by means of a granulator also play important roles in producing hard and firm granules.

Bag caking is caused primarily by high moistures or excessive sulfuric acid in the fertilizer. It has been found by experience that any grade containing moisture in excess of 1.5% to 1.75% moisture might bag cake in long storage.

J. Howard McNeill

Our observations are that the *minimum* temperature for best granulation is 185° F. under most conditions. Achieving good granulation under 185° F. presents a number of problems:

1. Control is much more critical because soluble salts do not contribute appreciably to the plasticity of the mixture.
2. More water is needed; this additional moisture must be removed and results in excessive dryer load. Many dryers are already overloaded under optimum operating conditions.
3. Granules are softer and tend to break down in storage and during handling because full advantage is not taken of the action of the soluble salts in forming strong bending forces in the granules.
4. Excessive fines are produced and low hourly rates of production of product are the result because of the poor bonding characteristics mentioned above.

Properly balanced fertilizer mixes with sufficient soluble salts for agglomeration and with the temperature raised to approximately 200° F. by heat of reaction will generally produce a hard, strong granule that will hold up well in storage.

Steam has been used very successfully to raise temperatures above 185° F. in many low nitrogen grades where the heat of the reaction is insufficient to produce suitable plasticity.

We have found that regardless of temperature in production, the granules with a high soluble salt content are often hygroscopic in humid weather. Holding the ammonium nitrate nitrogen content below 50% of the total nitrogen will contribute to less hygroscopicity.

The important factor is the control of the volume of the liquid phase. Three factors control the volume of liquid phase in the reaction mixture: (1) temperature, (2) moisture, (3) soluble salt content.

Successful granulation has been brought about at lower temperatures but our observations indicate that a good "rule of thumb" is to keep the temperature in the ammoniator above 185° F.

Although the subject matter for this presentation was restricted to minimum temperature for granulation, there are practical upper limits on temperature. The factors which contribute to this upper limit are:

1. Rapid, excessive drying reduces the time interval during which good granulation can be accomplished.
2. Increased danger of spontaneous decomposition of ammonium nitrate; especially in 1-1-1 grades in which ammonium nitrate, potash, and acid are combined.
3. Perhaps a prohibitive nitrogen due to urea break-down where mixed urea - nitrate solution are used is a similar danger.

Are Coating Agents in Granulation Beneficial, and When and How Should they be Introduced into the Process?

G. L. Bridger

COATING agents or conditioners are definitely beneficial in prevention of caking but they are needed for only certain formulations and grades which give more trouble than others in this respect.

Since caking is caused primarily by high moisture content and small particle size, the best way to overcome it is by granulation by which the moisture content of the product can be reduced to 1 or 2% and the particle size can be controlled so that most of the product is in the range of 6 to 20 mesh or whatever other specification may be set up. Even with well granulated and well dried fertilizers however, some grades (especially those high in nitrogen) still tend to cake under severe conditions, and conditioners can be used to advantage.

A laboratory study of factors influencing the efficiency of conditioners was published by Hardesty and Kumagai (Agricultural Chemicals, February and March 1952). A large number of conditioners were tested and it was found that in general the lower the bulk density of the conditioner and the greater its moisture absorption capacity, the better was its effectiveness.

Some of the more common conditioners are diatomaceous earth, clay, Fuller's earth, vermiculite, and various organic byproducts or residues.

With well dried granulated fertilizers, 20 to 40 lbs. of conditioner per ton is usually sufficient to achieve optimum benefit. Larger amounts of Conditioner simply tend to make the fertilizer dusty on handling.

With non-granulated fertilizers the moisture content is greater and the particle size smaller so that considerably more conditioner must be used to achieve a beneficial effect. From 100 to 400 lbs. per ton is required under these circumstances. Such high rates of course are expensive and also result in substantial lowering of the concentration of plant nutrients in fertilizers.

Conditioners can be introduced into the fertilizer either before pile storage or before bagging. If introduced before pile storage they will reduce the tendency of the fertilizer to set up in the pile and facilitate digging out the pile. However, usually a very dusty condition results in the plant from dumping the conditioned fertilizer into the pile and to a lesser extent on digging out the pile for bagging. For this reason many manufacturers prefer to introduce the conditioner just prior to bagging.

For maximum beneficial effect it is necessary to mix the conditioner with the fertilizer thoroughly so that the particles are evenly coated. It is not sufficient to depend on normal conveying and handling of the fertilizer to obtain good mixing. A rotary mixer providing good agitation should be used.

How Can the Effects of Hygroscopicity be Minimized, Especially Those of High Analysis Fertilizers During Storage and Bagging, i.e., Wet Equipment, Wet Slippery Floors, etc.?

David O. Conrad

ALL of us who make, store, and ship high-analysis fertilizers have this problem of wet floors and wet goods in the bins. Since I have been asked to answer this one, I want to express my gratitude to the writer of the question for including that word "Minimized."

We have been handling high-analysis fertilizers since 1931 and to date we have found no way to completely do away with the problem. Our experience would point to two ways to hold the ill effects to a minimum.

The first and most obvious is a matter of formulation. We avoid using combination of ammonium nitrate and urea. Such combinations frequently are worse offenders than either material alone. There is, however, so much information available on this subject and there are so many people here more able to explain it that I will pass on to a matter of Plant Management that we have found quite helpful in reducing the problem.

That matter is *Plant Housekeeping*. Clean floors, clean aisleways, do not get as wet as dirty floors and aisles. By keeping the mixed goods cleaned back to the pile faces we give the air-borne moisture a smaller target. Where mixed goods are in a high pile the relatively thin layer of moist goods can be lost in the much greater mass of dirty goods in the bagging operation. We feel that it is a paying proposition to keep our aisles cleaned back to the storage bins. To that end we have trained our Hough tractor operators to scrape back on the return trip from the shipping unit to the bin. They have been trained, too, to clean it at the elevator between grade changes. Plant laborers are not, as a rule, the best of housekeepers, but by keeping continually after them they can be trained.

Our men on the shipping end of the packers have been trained to keep their working area cleaned up. All broken bags are promptly disposed of by returning them either to the elevator, if we are still bagging the grade, or to the bins.

This practice of good housekeeping, if rigorously carried out, will do much to keep this problem of wet floors under control.

Outline the Causes of Hard and of Sticky Pulverulent Fertilizers

A. Henderson

The answers are given in the literature as follows:

DIFFERENT materials become caked from different causes, but the presence of moisture is usually the governing factor. Moisture may induce a caked condition (a) by causing the material to set much in the same way as plaster of Paris, (b) by increasing the attraction between particles through surface tension, cohesion, and other forces and (c) by causing the crystals to knit together by solution and recrystallization as the humidity of the air alternately rises and falls, or as the material undergoes changes in temperature. The rate and extent to which caking takes place in a fertilizer material or in a mixture of materials vary with the moisture content, the size of the particles, the pressure under which the fertilizer is stored, the temperature variations during storage, and the length of storage.

The effect of these factors differs with different materials, and the caking of a fertilizer mixture will therefore vary with (a) the materials used in the mixture, (b) the mechanical condition of the materials at the time of mixing, (c) the proportion of each material in the mixture and (d) the method of mixing. Thus, it has long been known that a mixture containing a large proportion of readily soluble salts cakes more readily as a rule than one which is relatively insoluble; that a finely divided mixture tends to cake more than a granular or coarsely crystalline one; and that the tendency of a mixture to cake decreases with increase in the proportion of any condition material that may be present. Chemical reactions between the components of a mixture may induce caking. (1)

The literature indicated that most previous investigators of the problem of fertilizer setting, had concluded that the solution phase was the prime offender. Researchers have listed many secondary factors to which they apply more or less responsibility for the damage caused by solution phase. These factors include: loss of moisture, change in temperature, pressure, metathesis, chemical reaction, hydration, crystal transformation, partial pressure of water above fertilizer, density and particle size.

Our interpretation of the literature was that temperature change and moisture loss were prime contributors and that pressure and metathesis were secondary contributors to the caking of well cured mixed goods." (2)

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(1) Relative Caking Tendency of Fertilizers by J. Richard Adams and William H. Ross, Ind. & Eng. Chem., Vol. 33, January, 1941.

(2) Chemical & Physical Properties of Ammoniated Superphosphate by F. L. Turbett and J. G. MacArthur, Spencer Chemical Company.

Is the Addition of Sulfuric Acid to a Mixture Helpful or Does it Make the Finished Product Hygroscopic? How does one Prevent Making a Gummy Product?

W. G. Mautner

THE addition of sulfuric acid definitely helps the granulation of mixed fertilizers especially in those grades containing 5% nitrogen or less. The sulfuric acid acts both as a plasticizer and a producer of heat. Both of these properties are essential to granulation efficiency.

The product will not be hygroscopic if the quantity of sulfuric acid added during granulation does not reduce the ammoniation rate of the superphosphates below 2.0 pounds of ammonia per unit of P_2O_5.

If the ammoniation rate is less than 2.0%, the mixture is on the acid side and will become very sticky and plastic in the drying operation. Fuming will be excessive due to the formation of hydrochloric acid and ammonium chloride. If this condition occurs and the dried product is not adequately cooled, these reactions will continue. Thermal decomposition will take place in the pile with resultant high temperatures, fuming and loss of nitrogen.

Sulfuric acid should be used in quanties which will allow a minimum of 2.0% ammoniation rate of superphosphates in grades containing 5% nitrogen or less.

In those grades which require sulfuric acid to react with the excess ammonia above the quantity required for ammoniation of the superphosphate, the maximum amount that can be used is approximately 150 pounds per ton. If the sulfuric acid required for the neutralization of the excess ammonia is much higher than 150 #/T, high nitrogen losses, excessive heat, and over granulation will occur.

DISCUSSION

W. G. Mautner: After some four years of wrestling a TVA type of a granulating unit, these are what we think are wise. In the first place, sulfuric acid is essential in this process for three reasons: First, you want the heat and plasticity which you get from the acid; secondly, you want some chemical dry-out from this heat; and the third reason for using acid, of course, is in those grades in which you have to neutralize whatever does not react with the superphosphate.

The addition of sulphuric acid definitely helps the granulation of mixed fertilizers, especially those containing five per cent of nitrogen or less. The sulphuric acid acts as both a plasticizer and a producer of heat. Both of these properties are essential to granulation efficiency. The product will not be hygroscopic if the quantity of sulphuric acid added during the granulation does not reduce the ammoniation rate of the superphosphate below two. In other words, you cannot make a 3-12-12 with three units of solution, and 175 to 200 pounds of sulphuric acid and expect to have any kind of a product. If the ammoniation rate is less than two for the mixtures on the acid side, it will become very sticky and plastic during the drying operation. Fuming will be excessive due to the formation of hydrochloric acid and ammonium chloride. If this condition occurs and the dry product is not adequately cooled, these reactions will continue, A thermal decomposition will take place in the pile with resulting high temperatures, fuming, and loss of nitrogen.

In those grades which require sulphuric acid to react with excess ammonia above the quantity required for the ammoniation of the superphosphate, the maximum amount—and this is our opinion—which one can use is 150 pounds per ton. We have set that maximum because in a 12-12-12, for instance, we formulate so that we do not have to use over 125 pounds of 66 degree Baume acid because we find that our nitrogen losses are excessive. Now if I may just have a minute on another subject. We heard quite a bit of talk yesterday about nitrogen losses and you make piles of fertilizer with low nitrogen. We have a small plant, and I say small, and we have in that plant a control laboratory in which we run every hour, we run moistures, nitrogen, and screen tests. The lab is operated by ordinary laborers in the plant. We get our moistures in eight minutes; we get our nitrogens in half an hour; and we get our screen tests in fifteen minutes. Now if any of you people — the whole installation will cost you for equipment, around $750 to $800. Now we find that especially in making the 12-12-12, if our flow raters are off, if our feeders aren't functioning properly, we at least know within an hour as to whether they are on or off, and that is very important. There isn't any use in making a thousand tons of stuff if you don't know the analysis for two weeks. And I think that that is one of the things that has helped us to stay on—I mean to have the proper analysis. Your screen test would show whether your unit is functioning properly. Your nitrogen is a check on your flow raters, its a check on your operators, it's a check on your feeders, your dry feeders. And the analysis routine is quite simple and it can be run by anyone that can read and write his own name.

Mr. Spillman: The very thing that Mr. Mautner talked about, we do at our Baltimore plant. We don't make our analysis tests at Baltimore, but we very carefully do analyze our moistures and screen tests. And if you don't do anything in your plant, it will keep you on the right track. We have no difficulty from nitrogen losses because we don't operate with what we call a high ammoniation rate. We use a pretty good ammoniation rate . We try to hold about 5.8 pounds of ammonia per unit of P_2O_5, we run a pile and we hold it to about three pounds of ammonia per unit of P_2O_5 in triple superphosphate, and we find through that method we can do a pretty good job on holding our analysis. On our control work it takes time there again and you have to pay a man to do the job. We run moisture tests every hour and we run screen tests every hour, both to find out how our cooler is operating, how our drier is operating, and how our screens are performing. And if it were not for that type of work, we'd be getting into a lot of difficulty because we find at certain times through running these tests that the screens are off, they plug, and we find that the moistures are off and through this control we can keep on a pretty straight track. It's very helpful, but it takes time and it costs a little money to do the job.

Another Question: This refers to using ammonium nitrate solutions. We run the nitrogen by the Devarda method which is used essentially for ammonium nitrate, but it works very nicely in this. We use, as the liquid for receiving the ammonia from the distillation, we use a saturated solution of boric acid. We back titrate that with half normal sulphuric which we purchase, and we use 1.4 gram sample and every cc is a half a per cent of nitrogen. Now what we're interested in is not whether it runs 501 or 499; we're interested in whether it's running 450 or 550, And we can check our laboratory within .15 of a per cent which, after all, is just a control.

It's surprising that you can hit your main control lab within .1 per cent and you'll be getting 99, you'll get ten or you'll get 10.1 and that is—the only trouble is when you get over to using ammoniating solutions you can't say that the Devarda method will give you the ammonia and amminium nitrate and the difference with urea, it won't come out that way. If you got two units from urea, the difference may be about 1.6, if you're on straight solution, this method is really good.

What Influences are Responsible for Caking of Granulated Fertilizers? Particle Size of Raw Materials, Chemical Composition (e.g., Ammonium Nitrate and Urea), Hygroscopic Fines, in the Finished Product? Would Standardizing Particle Size Reduce such Undesirable Caking?

T. P. Hignett

MANY factors influence the caking of granular fertilizers, including moisture content, chemical composition, curing, particle size and shape, amount and type of coating agent (if any), climatic conditions, type of bag, temperature when bagged, and duration and condition of storage.

Of these factors, probably moisture content and chemical composition are the most important. These factors are interrelated; the composition influences the moisture content at which caking may occur.

Most manufacturers consider that high-nitrogen grades should be dried to 1 per cent moisture and that low-nitrogen grades should be dried to 3 per cent moisture. This is a good general rule, but like most general rules, it is subject to exceptions. The more common granular high-nitrogen grades 12-12-12 and 10-10-10 are usually free from caking if dried to 1 per cent moisture. The popular granular low-nitrogen grades 4-16-16 and 5-20-20 are usually free from caking if dried to 3 per cent moisture.

Somewhat higher moisture contents may be satisfactory when other factors are favorable. For instance, in TVA tests, 12-12-12 granular fertilizer appeared to be satisfactory when dried to 1.4 per cent moisture and cured 7 days before bagging. Still higher moisture contents may not cause caking if an effective coating agent is used. For instance, in USDA tests, one sample of 12-12-12 (10 to 20 mesh, cured 7 days) did not cake at 3.7 per cent moisture when coated with 2 per cent diatomaceous earth.

European and New Zealand manufacturers have reported that undried granular ordinary superphosphate has satisfactory storage properties even at 7 per cent moisture. In TVA tests, no-nitrogen grades such as 0-14-14 and 0-20-20 did not cake appreciably at about 4.5 per cent moisture.

At the other end of the scale, pure crystalline soluble salts will usually cake unless conditioned even at very low moisture contents such as 0.2 per cent or less. Ammonium phosphate-nitrates such as 15-15-15 and 16-22-16 required drying to less than 1 per cent moisture, curing, and coating with an effective conditioner to prevent caking (2.5% kaolin clay was effective). Nitric phosphates 14-14-14 and 12-12-12 were satisfactory if dried to about 0.5 per cent moisture and cured or if dried to 1.0 per cent moisture, cured, and conditioned. Nitric phosphates without potash such as 17-22-0 were satisfactory when dried to 1.0 per cent moisture without curing or conditioning.

There is little evidence to support the popular opinion that caking is related to hygroscopicity when the caking properties are evaluated on the basis of bag-storage tests using bags containing one or more moisture-resistant plies. For instance, Raistrick's data reported at the 1956 Round Table indicated that NPK fertilizers in which the nitrogen was derived from ammonium sulfate required drying to less than 1 per cent moisture to prevent caking. The same is true of similar grades containing ammonium nitrate or urea.

In TVA tests the caking tendency of granular fertilizers was not appreciably affected by substitution of urea for part or all of the ammonium nitrate content of typical grades, as measured by the amount of lumps present after dropping the bags. However, the urea-containing products had harder bag sets.

Caking is much less severe in granular fertilizers than in nongranular when other factors are equal. However, within the range of particle sizes usually covered by granular fertilizers, the effect of particle size is not a major one. We believe that standardization of particle size of granular fertilizers is not the answer to the caking problem.

There is no evidence to indicate that the particle size of the raw materials has any direct effect on caking. Granular potassium chloride is often used as an aid to granulation of low-nitrogen grades. To the extent that its use is effective in producing more uniform granulation, it may indirectly improve storage properties. The storage properties of several grades of granular fertilizer made with and without granular potassium chloride were compared in TVA tests. No difference was found when granules of the same particle size were compared.

The use of fine potash may improve the roundness and uniformity of the granules in some grades. This may be true of high-nitrogen grades or of low-nitrogen grades when made with granular superphosphate. These effects may indirectly improve storage properties.

The presence of fines in the final product is likely to increase the caking tendency. This effect is not necessarily dependent on the hygroscopicity of the material. In one case in which the fines were principally small crystals of potassium chloride, caking was severe. Microscopic ammoniation showed that caking was caused by reaction of the small loose crystals of potassium chloride with the ammonium nitrate content of adjacent granules.

Casehardening of granules is claimed to occur in some granulation processes, and it is claimed that this casehardening results in improved storage properties. We do not know of any actual data on this subject. Since no method of quantitatively measuring the extent of casehardening has been developed, it is difficult to evaluate its effect.

To summarize, of the many factors that affect caking of granular fertilizer, probably moisture content is the most important. Common high-nitrogen grades (such as 12-12-12) are usually satisfactory at 1 per cent moisture, and most low-nitrogen grades are satisfactory at 3 per cent moisture when packaged in bags containing suitable moisture barriers. The use of coating agents deserves more attention since effective ones are available at low cost. Coating agents are necessary for prevention of caking for some very high analysis grades and are good insurance against caking in medium high analysis grades.

J. O. Hardesty

The data in references 4 and 8 indicate that:

(a) Caking is caused by crystal knitting.

(b) Crystal knitting, and consequently, caking, is potentially enhanced by high initial moisture content of the product, high content of very soluble hygroscopic salts, a drop in temperature during storage, pressure (for example, on the lower bags in a storage pile), and the presence of fine particles.

(c) Standardization of particle size if interpreted to mean "the requirement of a narrow particle-size range," would reduce caking but not eliminate it until all the factors that cause crystal knitting are controlled.

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What Experience Has Anyone With a Dust Collector for Hygroscopic Materials? With Bags Plugging up With Dampish Dust?

Louis F. Cizek

ONE of the by-products of the entrance of granulation processes into the mixed fertilizer industry is the dust control problem. Any of the methods used involves the drying and cooling of the fertilizer. The use of rotary type dryers and coolers are universally agreed to be the most satisfactory, considering the amount of heat and moisture required to be transferred in relation to value of the product mixed fertilizer.

During the past 8 years of experience with granulation, our organization, and I am sure this experience is similar to other, has found it necessary to adjust its thinking as to the moisture and temperature levels most suitable to granulation under given conditions and the temperature and moisture of the product necessary. The effect is increasing volumes of air and moisture to be handled. With the use of existing equipment in a plant, the effect is to exceed the capacity of dust collectors and to reduce the efficiency of certain collectors because of the presence of moisture.

In an effort to cope with this problem, Eastern States has recently completed installation at the Cambridge, Massachusetts, plant of two dust filters in conjunction with two classifiers, for the air exhausted from the drier. Manufactured by the Kelco Cooperative of Baltimore, similar equipment has been used on our shipping units for several years.

The equipment for filtering the ex-drier air consists of two identical units which operate alternately. Each unit consists of 84 orlon cloth tubes 6¼″ in diameter and 80″ long. The air enters the bags at the top and is exhausted through the cloth with use of fan of 12,000 c.m.f. capacity. With the use of a gate mechanism located at the fan inlet, the flow of air is alternated between the two units. Shaking mechanisms are installed to clean the tubes during the period that air is not being exhausted through the given unit, this accomplished with a timing device.

To avoid the condensation of moisture on tubes and metal, the entire filtering unit for the drier air is housed in a masonry constructed room equipped with a hot air furnace. The purpose of the room and furnace is to insure that the exhaust air is maintained at a temperature above its dew point. Thermocouples located in the masonry room control the furnace operation.

Our experience with this particular equipment during the few weeks of operation to date are as follows:

What Are Some of the Proven Scrubber Systems to Control Effluents from Getting into the Atmosphere? Can Fumes Be Controlled Without Expensive Scrubbers?

J. E. Rissman

Scrubbers are employed by the fertilizer industry for two main purposes:

1. The prevention of atmospheric pollution by effluent dusts and noxious vapors.
2. Recovery of valuable materials from effluent gas streams.

Both the pollutants and the materials to be recovered can be in either gaseous or solid forms.

In actual practice, these purposes are frequently combined. Thus a scrubber may be used for preventing atmospheric pollution by the recovery of dust which is returned to the process. Another example is the recovery of fluorine for sale as water treating or glazing compounds.

The most important constituents recovered from fertilizer plant effluent streams include:

1. Fluorine compounds evolved from the acidulation of phosphate rock.
2. Phosphate rock dust from grinding or mixing operations.

The most common types of scrubbers used in the fertilizer industry are discussed briefly as follows:

1. *Cyclones*
   Cyclones may be of either the wet or dry type. Dry cyclones are used to collect larger solid particles and wet cyclones are used to increase the efficiency of collection of small solid particles or to collect soluble vapors. In either the principle is that of spinning out the dense particles by centrifugal force. The usual device consists of a clyndrical vessel with a tangential gas inlet. The gas spins around in the cylinder and passes out the top. The solid or liquid particles are thrown to the vessel wall and fall to the bottom of the separator where they are removed.
2. *Spray Scrubbers*
   Spray scrubbers consist of many forms of chambers were a gas is brought into contact with a sprayed liquid. A spray scrubber may be as simple as an arrangement of shower nozzles in the top of a stack. While spray scrubbers are relatively simple and inexpensive their application is limited by low efficiency.
3. *Venturi Scrubbers*
   The venturi scrubber consists of a venturi section and a separator. The scrubbing liquid is sprayed into the throat of the venturi where the gas reaches its highest velocity. This high velocity gas stream striking the liquid causes

the droplets to break up and impinges the dust and fume particles onto the liquid. The gas is further cleaned as it expands and moisture condenses on the fine particles of dust. The gas then passes into a separator, such as a cyclone or chamber, where the liquid droplets and wetted dust particles are removed. Venturis are often applied as multiple units either in series or parallel or both.

4. *Inertial Scrubbers*
   Inertial scrubbers work on the principle of directing the dust laden gas stream toward a liquid film in such a manner that the inertia of the dust will carry it into the liquid whereas the gas stream is diverted in another direction. This principle is employed by placing baffles inside a chamber and applying a continuous liquid wash over the baffles.
5. *Packed Towers*
   Packed towers are designed to spread the collecting liquid over a large surface area and to impede the flow of gas as little as possible. Their principle application is in the removal of soluble vapors rather than the removal of solid particles which tend to plug the device.
6. *Mechanical Filters*
   Mechanical filters, such as panel and bag filters, are sometimes used in special cases but are not widely used by the fertilizer industry because of space, cost and maintenance requirements.

Many commercially available scrubbers combine two or more of the principles described in the foregoing.

The criteria for selecting a scrubber include:

1. The amount and nature of the impurities contained in the gas to be scrubbed.
2. Permissible limits of these impurities in the effluent stream.
3. The quantity of gas to be treated.
4. The temperature and pressure of the gas stream.
5. The corrosive nature of the materials involved.

Limitations of Sampling and Analytical Procedures; are Present Methods of Analysis Satisfactory for Granulated Fertilizers?

J. R. Archer

There is a definite need in today's modern fertilizer granulation plant for improved sampling techniques and instruments, and for rapid Methods of Analysis which will provide innerprocess control and assure product quality.

In 1954 the Chemical Control Committee of N. F. A., sent out a questionnaire to members of the fertilizer Industry in which the following question was asked:

> *"Do you consider the use of high analysis of granular fertilizers as increasing control problems?"*

Ninety-five (95) per cent of the members replying to this question stated that high analysis and granular fertilizers increased problems of Sampling and Analysis.

This question was asked:

> *"What percentage of erratic analysis do you attribute to improper sampling and sample preparation?"*

Eighty-seven (87) per cent of the members replying to this question stated that improper sampling and sample preparation attributed to erratic analyses.

It appears that a great majority of the Fertilizer Industry are concerned about the problem of sampling, and that there is a need for development work on sampling techniques and instruments.

No doubt, the laboratories reporting on the monthly Magruder check sample use the official methods of the A. O. A. C., yet, it is not uncommon to find the analysis of one laboratory varying from the average by as much as 1%. Perhaps, the Methods of Analysis should be in "cook book" form where the various techniques are described in more detail.

SECTION 3

Discuss Ammoniation in Regular Formulations, the Chemical Effects of other Compounds, and the Heats of Reaction

J. O. Hardesty

Ammonia neutralizes the acid constituents of superphosphate including any additional acid that may be present. The only compounds that interfere with this neutralization reaction are basic materials such as lime and quick-acting liming agents which serve to reduce the amount of neutralizing ammonia that can be absorbed. Considerable detailed information on this subject appears in the literature (*5, 9, 13, 16, 20*) related to ammoniation technique, the reactions involved, and the heat developed.

5. Hardesty, J. O. Agglomeration—A Chemical Engineering Tool for Granulating Mixed Fertilizers. *Chem. Engr. Progress* 51, 291-5 (1955).
9. Hardesty, J. O. and Ross, W. H. Heat Developed in the Ammoniation of Superphosphates with Anhydrous Ammonia. *Ind. Eng. Chem.* 29, 1283-90 (1937).
13. Keenen, F. G. Reactions Occuring During Ammoniation of Superphosphates. *Ind. Eng. Chem.* 22, 1378 (1930).
16. Kumagai, Rikio, Rapp, H. F., and Hardesty, J. O. Physical Factors Influencing Ammonia Absorption by Superphosphates. *Agr. Food Chem.* 2, 25-30 (1954).
20. White, L. M., Hardesty, J. O., and Ross, W. H. Ammoniation of Double Superphosphate. *Ind. Eng. Chem.* 27, 562-7 (1935).

L. Hein

Ammoniation of ingredients in mixed fertilizers results in the neutralization of acid constituents. These constituents may be calcium phosphate from ordinary or triple superphosphate, sulfuric, or phosphoric acids. The role of calcium sulfate has been discussed in the literature. In the reaction of ammonia, calcium sulfate and calcium or ammonium phosphate, the calcium from calcium sulfate enters the phosphate molecule to increase its calcium content. For example, monocalcium phosphate in these reactions is changed to di-or tricalcium phosphate with the accompanying formation of monoammonium phosphate or ammonium sulfate. The over-all effect is an increase in ammonia holding capacity of the mixture.

The calcium sulfate content of ordinary superphosphate is approximately twice that utilized in reactions of ammonia with monocalcium phosphate in the superphosphate. This excess is therefor available for reactions of additional ammonia with other phosphates such as triple super phosphate or phosphoric acid. For example, without phosphate: 2 $H_3PO_4 + 3\ NH_3 \rightarrow NH_4H_2PO_4 + (NH_4)_2HPO_4$. With calcium phosphate: 2 $H_3PO_4 + 2\ CaSO_4 + 4\ NH_3 \rightarrow 2\ CaHPO_4 + 2\ (NH_4)_2SO_4$.

Or should tricalcium phosphate be formed, 6 moles of ammonia would be held-twice as much as without calcium sulphate present. Similar effects are obtained with the monocalcium phosphate in triple superphosphate.

Let us assume that 196 lbs. of 100% H_3PO_4 are used in a formula per ton of fertilizer product without $CaSO_4$ present, this 196 lbs. would hold 51 lbs. of ammonia or about 7.2 lbs. ammonia per unit P_2O_5 (51 lbs. ammonia per 142 lbs. P_2O_5). If sufficient calcium sulfate were present, the ammonia holding capacity of the acid would be increased to 9.6 or 14.4 lbs. of ammonia per unit of P_2O_5 depending upon whether ammoniation was continued to di- or tricalcium phosphates. Assuming ammoniation is carried only to dicalcium phosphate in the product which is 9.6 lbs. NH_3/unit P_2O_5, the increased ammonia holding capacity is 9.6 minus 7.2 = 2.4 lbs. per unit of P_2O_5 or 17 lbs. ammonia per ton. If this 17 lbs. of ammonia were absorbed in sulfuric acid, about 50 lbs. of the acid would be needed.

The second part of this question concerns heats of reaction during ammoniation. Time will not permit more than a superfluous discussion. You are referred to published information in the Article "Heat Developed In the Ammoniation of Superphosphates with Anhydrous Ammonia" by Hardesty and Ross. The article can be found in Industrial Engineering Chemistry, 29, page 1283, November, 1937. In general, the heats evolved per pound of liquid ammonia in the ammoniation of the usual fertilizer ingredients are:

Material	Degree ammoniation	Heat released per lb. NH_3 liquid
Ordinary Superphosphate	6# NH_3/unit P_2O_5	1425
Triple Superphosphate	4# NH_3/unit P_2O_5	1635
H_3PO_4	MAP*	1900
H_3PO_4	DAP**	1750
H_2SO_4	Ammonium Sulfate	2850

* MAP = monoammonium phosphate
**DAP = diammonium phosphate

Since the data in the above table applies to the ingredients alone, it is not strictly applicable for detailed calculations for mixtures. However, it does show that absorption of a pound of ammonia in sulfuric acid releases almost twice as much heat as its absorption in superphosphate or phosphoric acid. The low moisture in 66 Be´ sulfuric acid also results in increased temperatures of the fertilizer mix.

The Successful Use of Five (5) Units of Nitrogen From Anhydrous Ammonia in a 5-20-20 Grade

J. E. Reynolds, Jr.

The successful use of five (5) units of nitrogen from anhydrous ammonia is accomplished through the features of submerged superphosphate ammoniation at many distribution points in close contact with the required amount of sulphuric and/or phosphoric acid. The basic needs to economically use large amounts of anhydrous ammonia are: (1) a steady flow of liquid anhydrous ammonia, (2) preparation of an aqua ammonia solution in the distribution manifold, and (3) submerged introduction of the aqua ammonia to achieve the greatest ammonia absorption and heat of reaction possible.

To assure a uniform flow of formulated liquid anhydrous ammonia, a pump is most generally provided. It is most desirable to experience a condition of ammonia vaporization ahead of flow control operated flowmeter. This indicates a mixture of liquid and vapor of an unknown amount. The ability to use large amounts of anhydrous ammonia is dependent on a minimum amount of fluctuations and maintaining a constant rate of reaction. Sufficient pressure is also maintained at the mixer manifold to retain a large percentage of the ammonia as a liquid.

The measured anhydrous ammonia is combined with the water in the distribution manifold. This practice is accomplished to achieve a certain amount of heat to accelerate the chemical reation between the ammonia and the superphosphate materials, and to discourage the vaporization of ammonia after it leaves the distribution pipe. The carrying and combining qualities of the ammonia are enhanced by this practice.

The actual practice of submerged introduction of the ammonia at many different points is probably the most important requirement. The distribution pipes enter the side or the bottom of the mixer, but in all cases these pipes are below the mixer shafts. The depth of material in the mixer covers the majority of the paddles to eliminate ammonia vapors from following the paddles through the surface. An instantaneous reaction is desirable, but a violent blowing reaction is undesirable. Previous work has shown that certain temperatures are desirable for granulation, and likewise, certain temperatures are desirable for ammonia absorption. The positive mixing of the warm water-anhydrous ammonia mixture with sulphuric acid and superphosphates at many locations utilizes the potential heat to the greatest advantage.

Although this discussion is primarily related to the use of five units of anhydrous ammonia in 5-20-20, this practice can also be duplicated in other grades. The operating and control techniques must be carefully supervised, however, when five (5) units of anhydrous ammonia are formulated. The formulas using five (5) units of anhydrous ammonia are limited to certain proven plants, whereas the use of four (4) units of anhydrous ammonia is wide spread throughout our organization.

Ray Sorensen

My answer to this question is yes. We have successfully used five units of nitrogen from anhydrous ammonia in most analyses containing five units of nitrogen. With our present equipment we feel that we lose approximately $\frac{1}{8}$ unit of nitrogen, consequently we over formulate by this amount. We have been using about 200 pounds of 66° Baumé acid per ton of fertilizer. However, we have changed our method of introducing acid and ammonia slightly and feel that we might be able to cut the acid down to as low as 160 pounds per ton. The products containing five units nitrogen from ammonia, made in our process, comes out in excellent condition and can be shipped as soon as it is reasonably cool. We have had practically no trouble with this type of material in bagged storage. It rarely sets up in the pile.

Heavy Nitrogen Losses Are Reported From Processed 1-1-1 Ratio Mixed Fertilizers, Particularly Granulated. Reactions May Continue in the Pile in the Case of High Nitrate High Chloride Ratios. Has Anyone Tried Adding an Inhibitor to Reduce or Stop Such Reactions?

T. P. Hignett

Nitrogen losses during processing are discussed on pages 67-72. The present discussion will be related to losses that may occur in the storage pile in the case of high-nitrate - high-chloride mixed fertilizers. Incidentally, we do not believe that losses occurring in the storage pile are at all common.

Fertilizers of high nitrate and chloride content made by usual formulations will decompose, losing nitrogen at high temperatures. The decomposition starts at 380° F. The heat of reaction then causes the temperature to rise further to perhaps 700° F.

For instance, in one test made by TVA, one end of a bag of 14-14-14 was heated with a blowtorch until fumes appeared. The flame was then removed. The material continued to smolder, giving off brown fumes as the reaction slowly ate its way through the bag of material. After about 4 hours the reaction had eaten its way through the entire bag. The residue remaining had lost 99 per cent of its nitrogen and 20 per cent of its chlorine.

The reaction could not be stopped by drenching the bag with water. It could only be stopped by raking the material out of the bag and drenching all of it with water.

Similar materials of high nitrate content that did not contain chlorine were tested. When heated with a blowtorch, fumes appeared, but when the blowtorch was removed, the reaction ceased.

Dry nongranular mixtures of ammonium nitrate and potassium chloride with or without superphosphate were subject to self-propagating decomposition when heated. As little as 3 per cent chlorine was sufficient to sensitize the reaction. Potassium sulfate did not sensitize the reaction.

Either monoammonium or diammonium phosphate inhibited the reaction. Mixtures that had high nitrate and chlorine contents were not sensitive when they contained enough ammonium phosphate to equal 60 per cent of the weight of their ammonium nitrate content. There is some evidence that ammonium sulfate inhibits the reaction, but no quantitative data are available. No other inhibitors have been identified insofar as we know, although not much work has been done along this line.

Acidic mixtures containing organic matter and nitrates are subject to spontaneous heating and consequent nitrogen loss, according to USDA studies *(Ind. Eng. Chem.* 38, 1298-1303, 1946).

To summarize, fertilizers of high nitrate and chlorine contents are sensitive to self-propagating decomposition when heated to about 380° F. Even a small lump of material reaching the pile at this temperature may cause the reaction to spread throughout the pile. Heating by external means of a small portion of the pile will start the reaction; this heating may be caused by contact with steam pipes, welding operations, dynamiting, or other accidental causes. Mixtures that do not contain chlorine are not sensitive. Ammonium phosphates inhibit the reaction if present in sufficient quantity. Thorough cooling before pile storage and guarding against accidental heating may be the best means of preventing the reaction.

Is There Danger of a Breakdown of Ammonium Nitrate in High-Nitrogen Fertilizers? Is Nitrous Oxide Formed at 200° C?

T. P. Hignett

Decomposition of ammonium nitrate is quite rapid at 380° F. (193° C.) and may be appreciable at lower temperatures. When chlorides are present, the decomposition of ammonium nitrate yields brown fumes which may be one of the oxides of nitrogen or nitrosyl chloride or both. It is not known whether nitrous oxide is formed (nitrous oxide is a colorless gas). Brown fumes are not usually observed when fertilizer containing ammonium nitrate but no chlorine is overheated. White fumes are often seen when fertilizers are heated to about 300° F. These fumes are believed to be vaporized ammonium nitrate.

(For further discussion, see pages 74-76.)

Dale Kieffer

Sometimes we experience what is interpreted to be a high nitrogen loss on 1-1-1 ratios when actually the loss may be no more in proportion than on 1-2-2, and other ratios. The nitrogen content in 1-1-1 ratios is usually double—or substantially higher—than in other mixed fertilizers, thus the loss may be more apparent than real.

The real losses that occur probably are due to efforts on the part of the formulators to get too much of the total nitrogen from anhydrous ammonia and solutions, which requires more than the average amount of sulphuric acid in order to neutralize the ammonia. A mixture of sulphuric acid, muriate of potash and ammonia solutions can, and invariably will, produce smoke and fumes containing nitric oxide, ammonium chloride and ammonia in sufficient quantity to represent a substantial percentage of the total nitrogen added.

If the mixture is overheated in the ammoniator and the dryer and carried immediately to the pile without cooling, decomposition might continue in the pile, contributing further to nitrogen losses. At times, this could reach dangerous proportions.

I do not believe nitrogen losses can be attributed to the pile reactions which may take place between chlorides and nitrate. It is true that ammonium nitrate and potassium chloride when mixed together tend to produce ammonium chloride and potassium nitrate. When there is an excess of ammonium nitrate the reaction seems to be more unstable, but when there is a slight excess of potassium chloride the mixture is likely to be quite stable, less hygroscopic, and not as subject to bag-set. None of these reactions will produce loss of nitrogen but, almost certainly, will cause a loss of good will.

E. C. Perrine

The loss of nitrogen that is most frequently encountered and which accounts for much loss in the industry is failure in combining the free ammonia with the superphosphates and to some extent with the added acids in the formulae.

The unintended actions of some chemicals in the formulae result in some nitrogen loss.

High heats of drying in some granulation plants are possibly causing losses.

Screening of fines from granulated grades can influence analysis if they are not returned to process.

Improper handling of ammoniating solutions can reduce their nitrogen content. One of these actions is the excessive venting of tanks during transfer of volatile solutions. Another would be the use of a solution that had partially salted out in storage or in tank cars.

It is true that many plants are retaining essentially 100% of the ammonia input at rates of 6.5 pounds per unit of P_2O_5 in 20% superphosphate and 3.5 pounds, for triple superphosphate. This requires equipment that is designed for the job and also requires considerable skill in operation. Some plants are operated as though the desire for the benefit of high rates of ammoniation is all that is required and they merely pour the nitrogen solution into the mixer without any recognition of the needs of equipment or techniques.

Beyond 3 pounds of ammonia per unit of P_2O_5 superphosphate considerable ammonia is usually lost to the atmosphere unless equipment and techniques are suitable for the requirements that are quite specific for high rates of ammoniation. Even with the best of equipment it seems impractical to hold more than 8 pounds of ammonia per unit of P_2O_5 superphosphate and this requires an input of 9.0 pounds, involving a calculated loss. Inputs as high as 12 pounds have generally failed to increase the retention much beyond 8.0 pounds. Much nitrogen is lost through this technique and not all operators are fully aware of the reasons.

Deterioration of equipment and the creeping tendencies of plant operation account for much loss of nitrogen. Where most of the ammonia is to be reacted with superphosphates it is the general practice to add 3.0 pounds of 66° Bé sulfuric acid for each pound of ammonia that is in excess of that ammonia which is expected to be reached with the superphosphate.

Where formulation demands the maximum ammonia take-up from the superphosphates, unless both the acid and ammoniating solutions are added in such a manner that each pound of ammonia has its chance to react with its alloted 3 pounds of sulfuric acid the conditions are present for loss of nitrogen from two sources. One loss is from unreacted ammonia that escapes to the atmosphere. The second loss could well be through ammonium chloride escaping to the atmosphere. This condition partially answers question on page 57.

Agglomeration before all of the ammoniating solutions have been added reduces or completely stops further ammonia take-up about as though the particle size of the superphosphate had been suddenly increased. The influence of particle size has been dealt with under questions on pages 6, 8, 17, 34. Changing to another solution has corrected this condition, which may help to explain the reason for the number of nitrogen solutions being used for granulation. There have also been cases of unwanted granulation in pulverized fertilizer manufacturing which caused problems in ammonia take-up.

Venting of storage or measuring tanks in handling volatile ammoniating media has reduced the nitrogen content in some cases.

Good operations in most granulating plants hold the products in the dryer to no more than 250 to 275° F. and these are reached for a very short time in the dryer. As indicated in other discussions in this meeting such temperatures are acceptable operations. Some of the nitrogen salts are possibly broken down in the much higher heats of some dryers that may be operating at higher temperatures. This is usually done to offset some other factor which might better be corrected directly. In one case a sample of about 25 pounds taken from the dryer exit had an average temperature of 350° F. Some particles had to change. Such temperatures are far in excess of normal operations and usually bring penalties of poor granulation and reduced tonnage per hour until the conditions that invite excessive temperature are corrected. Air pollution is often a result of excessive heats and other attempts to force a granulation plant beyond some of its limitations.

Why is so Much Trouble Experienced with the Nitrogen Content of high Analysis 1-1-1 ratio Fertilizers such as 12-12-12, 14-14-14?

Dale Kieffer

It is a fairly well established fact that a combination of sulphuric acid, potassium chloride, ammonia and ammonium nitrate in a fertilizer ammoniator can, and does, under most fertilizer plant conditions, produce rather heavy smoke and fumes that are very difficult to scrub—and are usually lost through the stack. Apparently, the most important losses are in the form of NO_2, ammonium chloride, and ammonia. Most of us know that under the right conditions the high analysis 1-1-1 ratios containing large amounts of sulphuric acid, ammonium nitrate, and potassium chloride generate enough heat in the ammoniator to cause a rather serious decomposition of the ammonium nitrate. This is more likely to happen when the formula contains phosphoric acid, but can—and has happened—when the phosphate was all obtained from triple superphosphate.

An acid environment in mixtures containing large amounts of ammonium nitrate is the most important factor causing decomposition. Perhaps, some of the trouble that has been experienced in this connection is due to the fact that the acid and ammonia feeds get out of balance—with an insufficient amount of ammonia for temporary periods being fed to neutralize the acid. It is very important that the operation be controlled so that the acid feed never runs ahead of the ammonia feed. Some help may be obtained from the addition of a small amount of burned Dolomite, or an equivalent neutralizing agent, which will aid in keeping the pH of the mixture up. The rate at which the decomposition of ammonium nitrates will take place will be slowed up if there is some neutralizing salt in the mixture.

In the formulas containing larger amounts of sulphate such as calcium sulphate and sulphate of ammonia, nitrogen losses are not as great as in the higher analysis grades where the phosphates must all be obtained from acid or triple superphosphate—and the nitrogen from ammonium nitrate.

G. F. Sachsel

Any dogmatic answers to this question might bring a raucous "Vass you dere Charlie" from the audience. Since I have never lived inside an ammoniator or granulator, I propose to merely suggest what seems to be some likely causes of trouble.

The research of Rader and others have shown that ammonium nitrate and potash will react to form potassium nitrate and ammonium chloride. The handbooks tell us that ammonium chloride decomposes at 350 C (662° F.). How does any part of the fertilizer run get that hot? Sulfuric acid may do this in two ways: (1) reaction with anhydrous ammonia, and (2) dilution with water. The second effect by itself would not heat the mix very far but the ammonia reaction could produce high temperatures locally.

It almost seems as if we were in a bind here: if we don't hit excess anhydrous with sulfuric, it will flash off and we lose nitrogen; if we do hit it with sulfuric, we lose ammonium chloride; if we don't use that much anhydrous, the goods will cake like mud because of the high ammonium nitrate content.

A couple of months ago, a Uramon solution might have looked like part of the answer. The data of Lehr and others presented at the last ACS meeting suggested that urea can cause as much caking as ammonium nitrate. Throwing the potash in last would be a possibility but it requires revamping the process or equipment. It may also create new problems, such as lack of uniformity in the goods, particularly if granulation is practiced. Good liquid distribution and solids mixng should prevent much of ths local overheating. A longer ammoniator may help in this way.

DISCUSSION

G. F. Sachsel: I believe a lot of this has probably been discussed before because this is really tied in with the problems of the reaction of nitrogen materials with other components of fertilizer mix. A mixture with a little higher than one per cent moisture has been satisfactory, and even as high as two per cent on 12-12-12 may be satisfactory if a good coating agent is used on the granule. Generally speaking, though, those grades, one per cent allows some margin of safety and is quite satisfactory. Now as we go a little higher in analysis, it may be necessary to reduce the moisture content. For instance in 14-14-14 we think the moisture content should be down around one-half of one per cent. In most formulations that we have tried for 15-15-15, we've found that it's almost impossible to dry it far enough so it will not cake without a coating agent. So as we go to the higher analysis, it becomes more and more necessary, more and more advisable to use a coating agent. On some of the low nitrogen but high analysis mixtures such as 7-28-28 it may be satisfactory at one per cent moisture without a coating agent. Our evidence indicates that there is very little difference between the caking properties of high analysis grades depending on whether urea or ammonium nitrate is present or whether there is a small amount of the urea in a mixture with ammonium nitrate. A part of this question concerns the particle size of the raw materials. We have answers to some specific questions in that respect. We find that the use of granular potash as compared with the use of fine potash in making granular fertilizers has not affected the caking properties of the fertilizer when both are granulated to the same extent. When fine potash is used, granulation is sometimes poor. We have found cases where due to poor granulation and the presence of unincorporated fine potash in the product, caking has been increased. The mechanism of caking in that case has a crystal growth between the potassium chloride fine which has not been incorporated and adjacent particle containing ammonium nitrate bridging across with ammonium chloride or potassium nitrate crystals. Potassium nitrate, incidentally, seems to be a stronger bonding crystal than the ammonium chloride.

As to standardization of the final granule size, we know, of course, that caking is less severe in granular fertilizer than it is in nongranular when both are of the same composition and same moisture content. I don't think that standardization of particle size of granular fertilizer is the answer. It's not within the range usually encompassed by granular fertilizer; that is, whether it is five to twenty mesh or whether it's six to sixteen mesh or whether it's eight to twenty-eight mesh, I don't think that makes very much difference. It may make a little difference, but it's rather a minor effect as compared with the major effects. Now, of course, the presence of fines if screening is not complete or not as perfect as it should be, the presence of fines in the product will certainly increase caking, and not always the hygroscopicity of the fines. either. There has been some evidence that case hardening of granules is helpful, but case hardening is a property that I don't know how to measure and so we haven't any specific data on it. I think the use of a coating may deserve more attention than it has been given, especially since there are some that are very effective in extremely small amounts, even one per cent or less, and there are some that are quite cheap. I think it may be good insurance and in some cases may cost only a few cents per ton to apply a coating agent to granular fertilizers, and the effects are certainly rather pronounced.

So to summarize the effect of very many factors that affect caking of granular fertilizers, probably moisture content is the most important. The common high nitrogen grades, such as 12-12-12, should be dried to about one per cent moisture or less. Low nitrogen grades to about three per cent moisture or less. Of course, it never does it any harm to dry it further than that if you can. The use of coating agents I think should receive a little more attention. Thank you.

Mr. Spillman: Would Mr. Hignett please comment a little further on that one item he skipped over, the temperature at bagging time. Where we get varying degrees of temperature between bagging temperature during the day and possibly a drop in temperature over the night in freezing weather. Many times with granular fertilizer it becomes very packed in the bag, but with a rising temperature it will come back to perfect condition. And some of the salesmen, not very highly technically trained, have said, oh, well, that's just a case of freezing; it will be all right when it warms up. Actually, I think the initial phase of some of the chemicals in formula are largely to blame for that. In other words, the solubility of those soluble salts is very extensive and I think they have a great effect on that. Would you comment on that further, please?

Mr. Hignett: I can't give you any authoritative data. Of course, you're quite right, as the temperature drops the soluble salts crystallize out and cause caking and that is the cause of unusual caking when the bagging temperature is high. Now how high you can get away with all depends on what product you're dealing with and other factors. I haven't seen much effect on high bagging temperature when the temperature is 140° F. or less, but on the other hand my experiences may not be as broad as yours.

Mr. Spillman: Mr. Nielsson.

Mr. Nielsson: I just want to ask a question on these moistures. Are you talking air flow method, desiccator for twenty-four hours or 100° C. oven moisture? It's quite important because most of the industry or a lot of them are using 100° C. moisture. The air flow method, in our experience with it, shows that two per cent by total moisture with the oven method would be about one and a half by air flow method. One thing that is rough then is if you tell the boys to use one moisture content and if the labs are using government literature they say another moisture content is supposed to be all right. If the different moisture methods don't give the same answer, we're not talking about the same moisture conditions.

Mr. Hignett: I am under the impression that most of the samples run by our laboratories are run by the desiccator method. I know that some years we ran air flow method on some materials, but I don't think we did on the general run of conventional fertilizers.

What Are the Factors Which Influence the Ammoniation Characteristics of Superphosphate?

J. O. Hardesty

Experimental data, confirmed by plant practice, indicate the following conditions for best ammoniation:

(a) Both ordinary and triple superphosphates should consist of fine, soft, porous particles rather than coarse, hard, dense particles.

(b) The ammoniation rate for ordinary superphosphates should not exceed 6 pounds of neutralizing ammonia per unit of P_2O_5; triple superphosphate, 4 pounds per unit of P_2O_5.

(c) The initial moisture content of the superphosphates, either ordinary or triple, should not be less than 3%.

(d) The superphosphates should contain as small a proportion of coarse particles (+20 mesh) as practicable.

(e) The reaction temperature for ordinary superphosphate is not important but for maximum absorption of ammonia by triple superphophate it should be near 212° F.

(f) The reaction time should be not less than 3 minutes.

(g) Cooling of the product at the end of the processing stage is essential.

Further details are given in the following reference: Kumagai, Rikio, Rapp, H. F. and Hardesty, J. O. Physical Factors Influencing Ammonia Absorption by Superphosphates. *Agr. Food Chem. 2,* 25-30 (1954).

The data in this reference show that granulated superphosphates are not suitable for ammoniation.

T. P. Hignett

The factors that influence the ammoniation characteristics of superphosphates have been studied by Kumagai, Rapp, and Hardesty *(J. Agr. Food Chem. 2,* 25-30, 1954). In the absence of granulation and when using anhydrous ammonia, the ammonia absorption increased as the particle size of the superphosphate was decreased from 5 to 80 mesh, increased as the moisture content of the superphosphate was increased from 1 to 9 per cent, and was more rapid with porous than dense superphosphate.

When using ammoniating solutions, the moisture content of the superphosphate may be less important. If the total moisture content of the mix reaches the point at which the mix becomes muddy, ammonia absorption my be poor.

When ammoniation and granulation are carried out simultaneously, the initial particle size of the superphosphate is relatively unimportant since the ammonia is absorbed in granules that form during ammoniation. It is important to prevent overgranulation since ammonia absorption in oversize granules is poor,

Also, when ammoniation and granulation occur simultaneously, the initial moisture content of the superphosphate is not particularly important since water is usually added if necessary to obtain good granulation. In making high-nitrogen grades, dry superphosphate may yield better results. If the superphosphate is too wet, it may be difficult to prevent overgranulation and consequent ammonia loss.

Porosity of the superphosphate strongly affects ammoniation absorption either in the presence or absence of granulation. It is probably the most important physical property that affects ammoniation.

Overacidulation in the production of superphosphates increases its ammonia holding capacity. This is not likely to be an important variable in the range of acidulation usually encountered in practice.

Elmer C. Perrine

Four properties of superphosphate that influence its ammoniation to some degree are particle size which is discussed in detail on pages 63, 66, free acid content, water, and porosity affecting the area exposed.

The equipment and the use of equipment have more influence on ammoniation than do the characteristics of the superphosphates themselves. Of the characteristics of superphosphate which do materially influence ammoniation, that of particle size is probably the most important.

Under plant conditions particle size alone can cause as much as 40% of the weight of a superphosphate to absorb only half as much ammonia per pound as that portion that is above 50 mesh.

Under plant conditions ammonia readily penetrates to the center of superphosphate particles that are no larger than around 40 mesh and that at larger than 30 mesh there is a rapid falling off in ammonia take-up.

When agglomeration of all ingredients has begun before all of the ammonia has been added, the result is similar to increasing the particle size of the superphosphate alone. It may be even more severe as some superphosphate is doubtless imbedded so deeply in the pellet as to be quite beyond the reach of ammonia. Economics and the great advantage of high ammoniation rates in granulated as well as in most pulverized fertilizer have stimulated usages in plants that have shown many of the early supposed factors to have little influence on ammoniation.

The influence of free acid on ammoniation in most commercial superphosphates can be calculated to a nicety. One grade of 20% superphosphate will absorb only about 0.6 pound more of ammonia per unit of superphosphate because of its higher acid content compared with one of a low acid content. (This is a factor of about 10%)

The amount of water in superphosphate itself or total water in the mixture has little direct influence, but indirectly the influence of water is sometimes quite great. At high rates of ammoniation water at 170° F. will hold very little ammonia. Where the water may permit premature granulation or agglomeration, as mentioned earlier, it does influence ammoniation but this is not necessarily a property of the superphosphate itself.

Triple superphosphates have shown an ability to absorb from 50 to 60% as much ammonia per unit of P_2O_5 as does 20% super, or from .5 to possibly 4.0 pounds of ammonia.

The long-held rate of 3 pounds of ammonia in 20% superphosphate was low because of equipment and techniques and not because of properties of superphosphates. Rates of ammoniation were more than doubled when simple changes were made to permit uniform mixing of the ammoniating medium as it entered the mass and also provided for reacting the ammonia gas that is generated as the temperature of the mass increases from the heat of the reaction. By a willingness to lose some ammonia through excess application without the use of acid and with equipment and techniques that hold essentially 100% of an input of 6.5 pounds of ammonia per unit of P_2O_5 in 20% superphosphate some operators are holding as much as 8.0 ponds of ammonia when 9.0 pounds are applied. Beyond these rates of application the loss of ammonia is nearly 100% for any additional applied. Comparable data for this excess ammoniation for triple super is not available.

What Are Advantages and Disadvantages of Concentrated Low Water Content Ammoniating Solutions in Manufacturing Non-Granular Fertilizers, Ditto, Granular?

Joe C. Sharp

In non-granular fertilizers, moisture is one of the principal causes of bag cake. If the moisture content is low enough in the finished product, large amounts of salts like ammonium nitrate or urea may be tolerated. Using concentrated solutions is one means of keeping the moisture content low in the finished product. Low moisture content also helps to control reversion. If the moisture content in the finished product is below 5%, reversion is usually not a problem.

In granulating high nitrogen grades, concentrated ammoniating solutions reduces the amount of recycle required to control liquid phase in the reactor. It also reduces the drying problem in such grades. The only advantage of concentrated solutions on low nitrogen grades is freight saving.

On the disadvantage side, because of higher vapor pressure more care must be exercised in transferring from tank car to storage to avoid nitrogen loss. The same applies to filling measuring tanks when the batch process is used.

B. E. Adams

Advantages—Permits more units of nitrogen from solution without increasing the water content per ton of fertilizer. More nitrogen can be shipped per tank car.

Disadvantages — Higher vapor pressure which requires better handling to prevent additional loss.

The above answers apply both to granular and non-granular fertilizer.

Effect of Triple Superphosphate Granular or Pellet Size on Ammoniation

D. Chapman

Yes, triple superphosphate of different particle sizes does have individual characteristics during ammoniation. We have laboratory tests under study on the subject; however, the completion unfortunately is still to be reached, and we have no new and conclusive information to release at this date.

I turn to work done by Messrs. Kumagai, Rapp and Hardesty for an answer to this question. They point out that moisture, density, hardness and length of ammoniation, as well as particle size, have a direct effect upon the absorption rate of triple superphosphate. For this work, a laboratory rotary ammoniator was used with a period of four minutes allowed for ammoniation and mixing. This is considered equivalent to the 3 minutes ammoniation time prevalent in the industry.

Two types of triple superphosphate were tested for absorption variation in relation to particle size, "Type A being composed chiefly of soft porous particles, while Type B triple superphosphate was a mere granular product composed chiefly of hard, dense particles." Comparative results of the two types were as follows:

Screen Fraction	*Percent Absorption*	
	Type A	*Type B*
— 80 mesh	100%	100%
30 mesh	92%	81%
6-10 mesh	85%	49%

The curve formed by the results on Type A parallel the absorption of ordinary superphosphate. Type B shows complete absorption at —80 mesh while the 6-10 mesh of Type B only shows 49% absorption as compared to 85% of Type A of the same size. "Type B has higher free acid and available and water-soluble phosphorus pentoxide contents than Type A, and on this consideration alone, should have greater ammonia absorbing capacity." However, "Type B has greater apparent density and granular hardness and a more encrusted particle than Type A. Apparently, many ammoniation difficulties encountered in commercial practice are attributable to a high portion of dense, hard encrusted granules in the superphosphates."

Reference: Physical Factors Influencing Ammonia Absorption by Superphosphates, *J. of Agricultural & Food Chemistry*, Vol. II, January 6, 1954.

Comparisons of Urea vs. Ammonium Nitrate Solutions in Granulation

Theodore R. Schmalz

At the outset, some qualifications regarding the following discussion should be made. The observations presented are based on operating experience with a couple of hundred thousand tons of granulated goods made with ammonium nitrate solutions and a couple of hundred made with urea solutions. In addition, some of the comparisons are based as much on empirical evaluations as on scientific type data.

A natural starting point would be a comparison of the solutions themselves. Urea solutions generally have a slightly higher ratio of free to total nitrogen. In most grades, this fact would probably not be significant. However, in 1-1-1 and in some cases 1-2-2 grades, ammoniation rates assume increased importance. Another point of consideration is the salting out temperature. Urea solutions generally have higher freezing points than nitrate solutions. These two considerations may not be of great importance, but an advantage in flexibility of formulation lies with the nitrate solutions.

On the other hand, urea solutions are not as corrosive as nitrate solutions. Another factor in favor of urea solutions is the safety factor. One urea solution producer is advertising a fire proofing quality, in that carbon dioxide liberated from ammonium carbamate during ammoniation blankets the ammoniator. In any case ammonium nitrate is more hazardous generally than urea.

Solutions containing urea were used to make several grades of granular goods at two of our factories. One of the units is a batch operation; the other a continuous operation. The same general tendencies were evident in both operations.

Goods made with urea solutions tended to be finer than those made with nitrate solutions. Initial granulation in the ammoniator appeared to be as good if not better than when nitrate solutions are used, but breakdown of particles was occurring in subsequent handling, Another indication of particle softness was reported in an informal manner when our chemist noted that, in preparation of samples, grinding was much easier on urea made goods than on nitrate made goods.

Higher temperatures and increased fuel consumption to obtain comparable product moistures was noted when urea solutions were used. This effect was probably due to the increased water consumption and the additional fines recycle load in the system.

Bag storage tests indicated that less bag set was experienced in the materials that had been made with urea solutions where product moisture was 2 to 3%. Goods made with nitrate solutions showed as good condition at 1% moisture as the urea made goods.

Based on the experience which we have had, it appears that any advantages which can be derived by using urea solutions for production of granular goods are appreciably offset by the results obtained using nitrate solutions. This statement takes no account of agronomic considerations and is based only on plant operating conditions and physical characteristics of the products.

What Data Are Available to Show How and Why Different Types of Ammoniating Solutions Work Better in Granulating Different Ratios?

A. V. Slack

There are now 35 or more different types of ammoniating solutions available to fertilizer producers. They differ mainly in the following ways:

1. Free ammonia content, ranging from 16.6 to 36.8 per cent. This can also be expressed as ratio of free ammonia nitrogen to fixed nitrogen, which ranges from 0.58 to 2.0. These values are for what might be called standard solutions. A few recently introduced solutions are outside the range given.

2. Type and content of supplemental nitrogen source, either ammonium nitrate, urea, or ammonium carbonate.

3. Water content, ranging from 5.8 to 38.6 per cent.

The variations of these factors among the various solutions have an effect on granulating efficiency of various grades. The free ammonia content has an especially important effect in granulation of high-analysis grades of the 1:1 $N:P_2O_5$ type. These require use of an acid to neutralize ammonia beyond that which the superphosphate can take up. If a solution with a high ratio of free ammonia to fixed nitrogen is used, more acid will be required and the higher resulting plasticity may give difficulty with overagglomeration. For example, in TVA tests on 15-15-0, 129 pounds of sulfuric acid was adequate to prevent excessive ammonia loss when an 0.83 ratio solution (21.7% NH_3; 65.0% NH_4NO_3) was used. When this was replaced with a solution of 1.1 ratio (26.0% NH_3; 55.5% NH_4NO_3), the acid requirement was increased to 191 pounds and more cooling air was required to prevent overagglomeration.

Presumably the standard commercial solution of lowest free ammonia:fixed nitrogen ratio (0.58; 16.6% NH_3 and 66.8% NH_4NO_3) would have given even better results than the 0.83 type used in these tests. This would have required only about 100 pounds acid to neutralize excess ammonia. Some of the granulation plants are using the 0.58 ratio solution and thereby reducing the amount of acid required; however, the majority appear to be using solutions with an intermediate ratio. One reason for this is the relatively high salting-out temperature of the low ammonia solutions, which requires insulation of tanks to prevent crystallization during cold weather.

Reduction of the free ammonia: fixed nitrogen ratio can be extended to use of a solution of such low ammonia content that no acid is required at all. Such a solution (0.26 ratio; 8.0% NH_3 and 72% NH_4NO_3) has been made available recently. A very few producers have used this in granulating high-nitrogen grades without acid; results were reported to be satisfactory. The solution has a low nitrogen content (32%) and very high salting-out point (85° F.), drawbacks which are unacceptable to many producers.

For grades of lower $N:P_2O_5$ ratio, such as the 1:2 and 1:4 type, the situation is reversed and a higher proportion of ammonia to fixed nitrogen is appropriate. The 1:2 $N:P_2O_5$ type can be granulated without acid but it is desirable in most cases to use as much ammonia as the superphosphate will take up so as to get maximum reaction heat. Solutions of intermediate ammonia: fixed nitrogen ratio (0.83 and 1.1) are usually required for this.

For the 1:4 $N:P_2O_5$ grades, most producers supply nitrogen as ammonia and use acid with it to furnish reaction heat. Some get along without acid by using the maximum allowable degree of superphosphate ammoniation in order to develop reaction heat and—in some cases—by using steam. In TVA tests with 5-20-20, good granulation was obtained without acid by using a combination of ammonia and ammoniating solution to give a high overall ammonia: fixed nitrogen ratio. (Supplying all the nitrogen as ammonia would have given overammoniation.) For the optimum degree of ammoniation—considered to be 3.8 and 5.8 pounds per unit of P_2O_5 in concentrated and ordinary superphosphates, respectively, in TVA work—the ammonia: fixed nitrogen ratio for 5-20-20 is about 2.2. None of the standard ammonia-ammonium nitrate solutions has this high a ratio; however, a 2.2 (42.6% NH_3 and 45% NH_4NO_3) and a 3.1 (49% NH_3 and 36% NH_4NO_3) have been introduced recently.

Rather than stock different solutions for different grades, some producers stock only one—preferably of low ammonia content—and use anhydrous ammonia with it when necessary. Although storage and handling facilities for anhydrous ammonia are somewhat expensive, the solution-ammonia combination is the most economical in respect to raw material cost. For those who prefer to minimize investment by using only one liquid source of nitrogen a solution of intermediate ammonia-fixed nitrogen ratio appears to be the best selection.

Substitution of urea for ammonium nitrate affects granulation characteristics. One difference is that the urea type has a higher average water content, which promotes overgranulation in high-nitrogen grades and makes more drying necessary in all grades. However, TVA tests showed slightly better granulations with urea solutions.

Substitution of only part of the ammonium nitrate with urea has some advantages. The water content is reduced and the salting-out temperature is considerably lowered. As noted previously, one difficulty in using a low-ammonia solution for high-nitrogen grades is the high salting-out temperature of the usual ammonia-ammonium nitrate solution. Partial substitution with urea reduces this to a safe level.

The presence of carbamate produced a lower temperature in the ammoniator in TVA work, presumably because of heat absorbed by decomposition of the carbamate. This made granulation of low-nitrogen grades more difficult but was advantageous in making high-nitrogen product. However, the high free ammonia content of these solutions requires a relatively large amount of acid.

The water content of solutions is especially important with formulations which tend to overgranulate. Standard solutions of reduced water content have been introduced in the past year or so, and these are helpful in such formulations by reducing the amount of liquid phase.

E. C. Perrine

With all phases of granulation operating at greatest efficiency different ratios in finished product will often call for different ammoniating solutions.

Heat in the earliest stages of granulation is a very important factor and the heat from the reactions in ammoniation is derived at the earliest stage that is desirable. This heat is a free by-product from the use of a low-cost ingredient. When the maximum potential of ammonia take-up is reached it is then advisable to obtain as much of the additional nitrogen from another low cost source. This usually involves the nitrogen salts that are contained for convenience and cost in the nitrogen solutions along with the ammonia. Judicious choice of the nitrogen solution may influence the ratio of triple superphosphate to normal superphosphate and any added acid. This influence on costs and on granulation itself may be even greater than the savings in the cost of nitrogen which savings are often appreciable.

Whether the operator of a granulating plant ever heard of the words he is almost a slave to "liquid phase." The plasticity or condition that disposes the mass to granulation is achieved through the relation of heat, moisture and character and amount of ingredients. The nitrogen bearing salts of the ammoniating solution are very important factors.

If the desired plasticity, liquid phase, or predisposition to granulate can be achieved by greater heat in the mixer and less water it is usual to produce more tons of granulated fertilizer per day through a system. Less fuel is used also.

To obtain granulation at an economical rate it becomes necessary at some point to decide whether the heat of reaction from any increase in ammonia is to be more desired than the influence of more or different ammonia salts. Additional heat early in the process results from using more sulfuric acid than the most economical reactions would call for. This is true because a pound of ammonia reacted with sulfuric acid develops about double the amount of B.T.U.'s of heat that are obtained from the pound of ammonia reacted with normal superphosphate. When the mass exceeds 190°F. so much heat is dissipated in the evaporation of moisture that efforts to exceed 200°F. become costly in time or in ingredients.

As valuable as heat is in the mass in the mixes it may become so costly at high levels that the function of more heat as a factor in the liquid phase may be served more economically by other factors.

One nitrogen solution with given other ingredients and formulae may result in a plasticity that promotes granulation in the mixer before that action is desirable. Another solution may correct this.

J. O. Hardesty

The type of an ammoniating solution ordinarily denotes the kind of soluble salts used in making the solution. For example, a urea-ammonia solution contains urea and ammonia in water. Other types are ammonium nitrate-ammonia, and urea-ammonium nitrate-ammonia solutions.

The term "*type*" as used in this question is interpreted to mean "ammoniating solutions of varying composition with respect to contents of soluble salts, neutralizing ammonia, and water."

Very little data are available in the permanent literatuure on the subject.

The first consideration in the selection of an ammoniating solution is the ability of the solution to supply neutralizing ammonia in sufficient quantity to give adequate ammoniation of the superphosphate present, without introducing an overage of nitrogen or water in the mixture. Thus, mixed fertilizer with a low N/P_2O_5 ratio requires a solution high in ammonia content and low in soluble salt content.

Another consideration is the additional ammonia required to neutralize any sulfuric acid that may be added to produce the heat required in granulation. Solutions of varying composition are provided by the solution producers to suit the individual requirements of the mixed fertilizer manufacturer. Varying the content of soluble salt or moisture, with respect to the unit amount of ammonia present in the solution, often provides an economical way of adjusting the volume of solution phase to that required for agglomeration of a given grade of fertilizer. The temperature and the volume of solution phase, however, can be adjusted within certain limits by the addition of dry recycle fines, steam or both.

What Are the Ammoniation Characteristics of Phosphates of Different Particle Sizes?

C. E. Franklin

PARTICLE size is a major factor in the ammoniating qualities of phosphate materials.

While much information and data are available on the physical factors influencing the ammoniation properties of single superphosphate, little information is available on the effect of the physical factors on the ammoniation of triple superphosphate. To fill this gap, Phillips Fertilizer Research Section undertook a study of the ammoniation of triple superphosphate. Random samples of various manufacturers' triple superphosphate were obtained and ammoniated under various test conditions to determine the effect of various factors, one of which was the effect of particle size.

To briefly review the procedure used, the ammoniation of various size fractions with a constant moisture content was conducted in a small steel ammoniating drum 14 inches in diameter which was rotated at a speed of approximately 30 RPM. Anhydrous ammonia was introduced through a ¼-inch steel tube. The ammonia was added over a two-minute period and the ammoniated product was tumbled further for two minutes. It was then aired and cooled for 24 hours and then analyzed. In the study on the effect of particle size on ammoniation a slight excess of ammonia was always added to insure a maximum amount of ammonia present.

Fig. 1 shows the results of the study on effect of particle size on ammoniation of triple superphosphate. The ammoniation rate, pounds of ammonia per unit of P_2O_5, obtained at different screen sizes, is plotted for three different types of triple superphosphate. While the different types of triple superphosphate were ammoniated to different degrees, the effect of particle size on each type is nearly identical. The material smaller than 80 mesh shows approximately 40 percent increase in ammoniation absorption over the 6-10 mesh particles in each case.

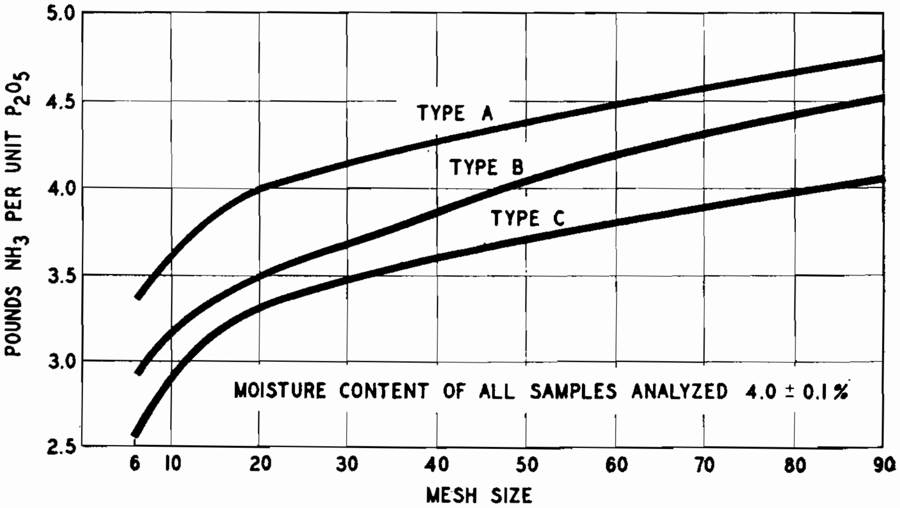

FIG. 1

In regard to the effect of particle size on the ammoniation of single superphosphate, I would like to review the work published by Mr. John Hardesty and co-workers of the U.S. Department of Agriculture. The effect of the particle size of superphosphate was determined by fractionating the superphosphate into different particle sizes and ammoniating each fraction separately. In this case each fraction was ammoniated with 6 pounds of ammonia per unit of available P_2O_5. The results shown on Curve 1, Fig. 2, ammonia absorbed per unit of P_2O_5 plotted against particle size fractions, indicates that only the 80 mesh fraction of the ordinary superphosphate absorbed all the ammonia reacted. The other fractions showed a decrease in ammonia absorption per unit of P_2O_5 with increasing mesh size.

Curve 2, Fig. 2, a composite sample, ammoniated with all particle sizes present, shows that ammonia is preferentially absorbed by the smaller particles when all size fractions are present during ammoniation. After ammoniation with 6 pounds of ammonia per unit of P_2O_5, the actual degree of ammoniation was 6.5 pounds per unit for the minus 80 mesh particles and 3.4 pounds per unit for the 4 to 6 mesh particles.

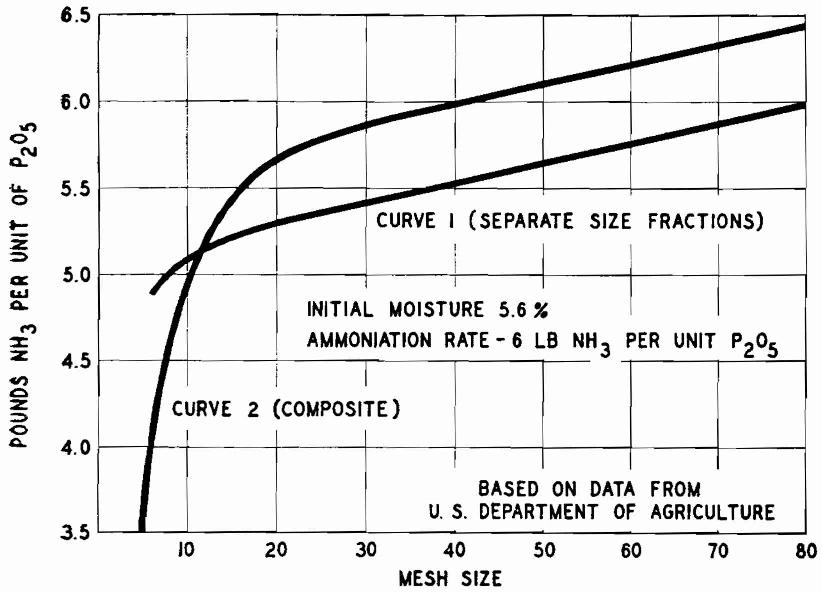

FIG. 2

Discuss the Loss of Nitrogen Occurring in Different Processes, That Is, How it May Vary and Whether or Not the Loss Occurs During the Processing Stages or as a Fume off the Dryer

C. E. Franklin

THE normal product temperatures upon leaving an ammoniator usually range from 170° to 220°F. However, these temperatures often do not reflect the maximum temperatures which are occurring in the area of the reactions between acid and ammonia. In this local area where exothermic reactions take place, localized temperatures often exist which are much higher than the average fertilizer temperature at the ammoniator discharge. It is in this area of localized high temperatures that thermal decomposition and sublimation of the fertilizer material take place.

In an effort to try to obtain a better understanding of nitrogen losses due to thermal decomposition and sublimation in the ammoniator, our Fertilizer Research Section has conducted laboratory tests to determine the effect of temperatures in the reaction zone on these types of losses.

In carrying out these tests on a 12-12-12 fertilizer in which the temperature was taken to be representative of the reaction zone and analyzing the off gases for ammonia, chlorides, and nitrogen oxides, it was found that with temperatures in the range of 320°F. and below the losses were less than two pounds per ton of nitrogen mostly in the form of free ammnia. At temperatures in the range of 350°F. the ammonium chloride losses were approximately five pounds per ton plus an additional three pounds of free ammonia per ton. When the temperature in the reaction zone was 370°F., dense white fumes were evolved from the fertilizer and analysis indicated a loss of 48 pounds of ammonium chloride per ton while free ammonia losses remained at approximately three pounds per ton.

At a reaction temperature between 370°F. and 390°F., there was a rapid, almost spontaneous decomposition of the fertilizer. In addition to ammonium chloride losses which amounted to 60 pounds per ton, mass spectrometric analysis of the gases evolved during the decomposition indicated that the losses also contained nitrogen oxides which brown fumes usually characterize.

Similar tests conducted with a 10-20-10 fertilizer gave very similar results.

There are, of course, several methods by which you can minimize these losses. One of the most important is to distribute the liquid material evenly throughout the length of the ammoniator through sparging equipment so as not to have large amounts of liquids which may react at any one place in the fertilizer mixer. Periodically check and replace any sparging pipes where corrosion may have enlarged any liquid outlets which may let large slugs of liquids out at any one point. Keep a good deep moving bed of dry materials moving by the sparger pipes which can rapidly dilute and disperse the area of localized high temperatures. The dry materials moving through the reaction zone serve as a cooling medium. A large percentage of the sublimed ammonium chloride may recrystallize as it moves up through the fertilizer bed. In one laboratory test we found that by increasing the bed depth only ½ inch, the ammonium chloride losses were eliminated.

If nitrogen losses are a serious factor after taking the above items into consideration, you may be using too high a ratae of liquid reactants for your equipment.

An example of how different quantities of nitrogen solution can effect the nitrogen losses due to thermal decomposition and sublimation: A 10-10-10 fertilizer produced in our laboratory batch ammoniator from 6 units of solutions had an average ammoniator discharge temperature of 190° F.and the nitrogen losses were equivalent to 4 pounds per ton. Changing the formulation from 6 to 7 units of solution raised the discharge temperature to 222°F., and the nitrogen losses were 19 pounds per ton. Going to 8 units of solution increased the average bed temperature to 240°F. and the nitrogen losses to 24 pounds per ton.

Most nitrogen losses occur as fumes from the ammoniator; however, at excessive high fertilizer temperatures in the dryer, it is possible to experience nitrogen loss in the dryer. At fertilizer temperatures of 210°F. and below in the dryer, no nitrogen losses are detectable. At fertilizer temperatures in the range of 220 to 250°F., the losses may range from one to five pounds of nitrogen per ton of fertilizer. At temperatures above 250°F., the losses increase rapidly with temperature.

T. P. Hignett

Nitrogen losses are often a serious problem in the production of high-nitrogen grades. To obtain information on the prevalence of nitrogen losses, published reports from a few states showing analyses made by the state chemists were examined. No attempt was made to make a complete survey. The reports showed that, in Wisconsin, Indiana, and Kentucky, more than half of the samples of 10-10-10 and 12-12-12 were deficient in nitrogen to an extent that exceeded the tolerance allowed by the state laws. In Missouri, 40 per cent of these grades were deficient in nitrogen. Probably similar situations exist in other states.

The reports show that some plants had a much higher batting average than others, but there was no clear indication that the type of process used affected nitrogen recovery.

Possible points of nitrogen loss are (1) from the ammoniator, (2) from the dryer, and (3) from the storage pile. Nitrogen losses from the storage pile are discussed on pages 58, 59. The present discussion will deal with losses from the ammoniator and dryer. These are believed to be the most important sources of loss.

Nitrogen losses from the ammoniator may result from one or more of the following causes: (1) unabsorbed ammonia, (2) fume consisting principally of ammonium chloride, and (3) decomposition of ammonia nitrate or urea.

Unabsorbed ammonia is believed to be the principal source of nitrogen loss. Poor ammonia absorption may be caused by overgranulation, poor distribution of ammonia or acid, overloading or underdesign of the ammoniator, or excessive degree of ammoniation.

Overgranulation is probably a frequent cause of ammonia loss when granulation and ammoniation occur in the same piece of equipment. Ammonia cannot penetrate rapidly into the center of a plus 6-mesh lump. Data from pilot-plant runs on 12-12-12 show that the ammonia loss was usually high when the product leaving the ammoniator contained more than 50 per cent plus 6-mesh particles. Under some conditions as much as 10 per cent of the free ammonia or possibly 5 per cent of the total nitrogen may be lost even at moderate degrees of ammoniation.

The cure for ammonia loss due to overgranulation obviously is better control of granulation by whatever means is available.

Distribution of ammonia and acid is very important. Mr. Perrine has given some excellent suggestions on ammonia distributors in various types of ammoniators in previous Round Table meetings.

In pilot-plant experiments, shortening the ammonia distributor from 30 to 12 inches increased ammonia and fume loss. On the other hand, installing two sets of the longer ammonia and acid distributors in parallel gave very low losses even at unusually high degrees of ammoniation. The longer distributors also helped prevent overgranulation, and this factor may be a cause of the improved ammonia recovery. The position, arrangement, design, and orientation of the ammonia and acid distributors may affect fume and ammonia loss. Enlargement of holes in the acid distributor due to corrosion is a frequent cause of high losses.

Retention time in the ammoniator in the range of 1.6 to 8 minutes did not appear to be an important factor. Presumably the effect of retention time was obscured by more important factors.

High ammonia losses may occur due to excessive degrees of ammoniation. It is not uncommon practice to use a high degree of ammoniation with the expectation of losing a certain percentage. This practice may be economical and satisfactory when the loss can be predicted accurately and compensated by increased supply of ammoniating solution. Standard degrees of ammoniation used in TVA's pilot-plant work were 3.8, 5.8, and 7.2 pounds of free ammonia per unit of P_2O_5 from triple superphosphate, ordinary superphosphate, and phosphoric acid, respectively. These degrees of ammoniation were such that ammonia losses were expected to be less than 3 per cent of the free ammonia input if granulation was reasonably well controlled. It was noted that considerably higher degrees of ammoniation could be used without excessive loss under some conditions. However, excessive losses sometimes occurred even when the degree of ammoniation was below the standard values if overgranulation or other difficulties interfered with a smooth rolling action of the bed.

There is a natural tendency to assume that, if the ammonia loss is high, it can be lowered by adding more acid. However, increasing the acid rate may increase the amount of overgranulation and thereby increase the ammonia loss.

There is a natural tendency to assume that, if the ammonia loss is high, it can be lowered by adding more acid. However, increasing the acid rate may increase the amount of overgranulation and thereby increase the ammonia loss.

Many continuous ammoniators produce a white fume which is composed mainly of ammonium chloride. This constitutes a loss of nitrogen. Because of its striking appearance, the fume is sometimes suspected of being the main source of nitrogen loss. However, the available data indicate that the percentage of nitrogen lost as fume is considerably smaller than that lost as free ammonia. Even heavy fuming has been found to account for less than 1 per cent of the total nitrogen.

The causes of fuming are similar to the causes of poor ammonia absorption, namely, overgranulation and too concentrated distribution of acid and ammonia. The cures are better control of granulation and more uniform distribution or greater length of distributors.

Decomposition of ammonium nitrate undoubtedly occurs at times in the ammoniator as evidenced by appearance of brown fumes. However, it is believed that decomposition of ammonium nitrate is seldom an important cause of nitrogen loss. The conditions that cause decomposition are so extreme that they are unlikely to be tolerated very long.

Urea decomposition is unlikely to be appreciable under conditions normally encountered in ammoniators.

Nitrogen losses from the dryer may be large under some conditions. The losses may be caused by vaporization of ammonia salts such as ammonium chloride, decomposition of diammonium phosphate which may be formed at high degrees of ammoniation, or decomposition of urea.

Nitrogen losses during drying of mixed fertilizers were studied by Bridger and Burzlaff (*J. Agr. Chem.* 2 1170-3, 1954). Appreciable losses were found to occur with some mixtures when the temperature of the material in the dryer was as low as 200° F. Any nitrogen losses occurring from ordinary formulations at 200° F. are probably due to diammonium phosphate decomposition. About 180° F. is considered to be a maximum safe temperature for drying diammonium phosphate. Overheating of the product is much more likely to occur in countercurrent dryers than in cocurrent ones.

Losses due to fume caused by vaporization of ammonia salts may become appreciable when the product temperature reaches about 280° F. Urea decomposition may be rapid at this temperature also. Some dryers may be operated at temperatures in this range.

In TVA pilot-plant operation in which the temperature of the material leaving the dryer ranged from 180° to 220° F., there was no appreciable loss of nitrogen from the dryer. In some plants in which the dryer is fired with an open flame in direct contact with the fertilizer granules, there may be appreciable nitrogen loss.

There are so many variations in equipment, process, and formulation that any general statement regarding nitrogen losses is likely to have many exceptions. With this qualification, we would express an opinion that the largest nitrogen loss is unabsorbed ammonia and that fume from the ammoniator or dryer is secondary.

We wish to take this opportunity to suggest that experiments be carried out in large-scale plants to decrease nitrogen loss by improved control of granulation and by improved ammonia and acid distribution. The latter might be accomplished by providing longer acid and ammonia distributors where this is feasible or by providing parallel distributors when the longer ones are not feasible. In many plants the acid distributor is not as long as the ammonia distributor so it would be relatively easy to lengthen it. This might be beneficial in improving nitrogen recovery and decreasing fuming.

Elmer C. Perrine

There are probably cases where nitrogen has been lost from granulated fertilizer while it was stored. But the relative simplicity of the formulation which is desired for obtaning granulation combined with the economy and other reasons for bringing all actions to completion ahead of storage would seem to preclude much loss of nitrogen during storage, particularly since good practice assumes that the finished product be dry and cool for storage.

Where fines are screened from the process and used elsewhere it is understandable why the remaining product could be off-grade if, as is sometimes the case, the particle size is related to the analysis.

G. L. Bridger

Discussion

The above two questions will be discussed together since they are both concerned with the same problem, namely, the introduction of nitrogen compounds into mixed fertilizers without loss.

Under certain conditions nitrogen may be lost in mixed fertilizer production in the ammoniation step, the drying step, and possibly the storage period. These will be discussed separately.

Ammoniation Step

In the ammoniation step the problem is to get as much free ammonia as possible combined with the superphosphates used in the formulation without excessive loss of nitrogen or excessive P_2O_5 reversion, since any free ammonia not absorbed by superphosphates requires addition of sulfuric or phosphoric acid for its neutralization. The free ammonia may be added as anhydrous ammonia or as a constituent of a nitrogen solution.

The factors controlling absorption of ammonia are the chemical and physical properties of the superphosphates used, the ammoniation rate, the time and temperature provided for the chemical reactions, and the distribution and mixing system for providing intimate contact between anhydrous ammonia, nitrogen solution, acid and solid ingredients.

Properties of Superphosphates. Physical properties of both normal and triple superphosphates are generally more important than chemical properties with respect to their ammoniation characteristics. Superphosphates consist principally of monocalcium phosphate monohydrate with a small proportion of free phosphoric acid and a considerable proportion of calcium sulfate in the case of normal superphosphate. The properties of superphosphates that are most important are particle size, moisture content and porosity.

In studies by Kumagai, Rapp and Hardesty (*Ag. & Food Chem.* 2, 25 (1954) at United States Department of Agriculture and by Waters, Arnold, and Payne and co-workers at Allied Chemical & Dye Corporation (*Ag. & Food Chem.* 3, 218 (1955), it was shown that only the minus 80 mesh fraction of both normal and triple superphosphate absorbed ammonia completely when the usual ammoniation rates were used (4 lbs. of NH_3 per unit of available P_2O_5 for triple superphosphates and 6 lbs. of NH_3 per unit of available P_2O_5 for normal superphosphate). As the particle size increased from 80 mesh to 4 mesh, the percentage absorption decreased to about 60% for normal superphosphate and 80% for triple superphosphate. In plant operation it is of course impractical to screen superphosphates to minus 80 mesh before use, but these results point to the desirability of keeping the particle size as small as practical with the equipment at hand.

The moisture content of normal or triple superphosphate should be at least 6% for good ammonia absorption. The above workers showed that superphosphates that will absorb 95% of the ammonia at 6% moisture will absorb only 90% at 4% moisture and 80% at 2% moisture.

The porosity of the superphosphate is a most important property but there is no quantitative way in which its effect can be measured except an actual ammoniation test. I am referring primarily to the microcrystalline porosity rather than the space between individual particles of superphosphate. Superphosphates may differ considerably in porosity depending on their method of manufacture. Some artificially dried superphosphates are less porous than storage cured products.

Ammoniation Rate. Ammoniation rate, or the ratio of ammonia to the available P_2O_5 content of the superphosphate, is one of the most important factors in ammonia absorption. If too much ammonia is used, much of it is simply not absorbed and goes up the stack, and furthermore reversion of the available P_2O_5 to an insoluble form may take place. Widely accepted ammoniation rates in the industry are 4 lbs. of NH_3 per unit of available P_2O_5 for triple superphosphate and 6 lbs. of NH_3 per unit of available P_2O_5 for normal superphosphate. With these rates at least 95% recovery of the ammonia can be expected if all conditions are favorable.

Time of Reaction. Sufficient time must be provided for the free ammonia to contact the superphosphate and be absorbed throughout the mass. More time must be provided for high ammoniation rates than for low ammoniation rates. At the commonly used rates mentioned above, a reaction time of about three minutes is necessary. This will depend somewhat on the type of equipment in which the reaction takes place and whether ammoniation is done batchwise or continuously.

Temperature of Reaction. Reaction temperature is not as important as some of the other factors and has relatively little influence on absorption of ammonia by normal superphosphate over a temperature range of 122-190° F. Higher temperatures favor ammonia absorption by triple superphosphate over a range of 149-216° F. Usually temperature cannot be controlled as well as other factors since it is influenced by climatic conditions and varies from one formulation to another.

Distribution and Mixing System. It is very important that the anhydrous ammonia, nitrogen solution and acid be added as uniformly as possible throughout the bed of materials in the mixer and that the mixing be as good as possible. The means to accomplish this are varied depending upon the equipment used and whether the system is batch or continuous. It is obvious that without good distribution there will be a tendency for over-ammoniation and consequent loss of ammonia in localized areas, and under-ammoniation in other areas. Furthermore poor distribution contributes to over-granulation and lump formation because of excessive moisture and temperature in localized areas. A more serious consequence of poor distribution is formation of ammonium chloride fume due to localized excess of acid; this attacks potassium chloride forming hydrogen chloride vapor, which forms ammonium chloride fume upon reaction with ammonia vapor.

Drying Step

After ammonia has been absorbed more or less efficiently in the mixing step further nitrogen loss is possible in the drying step if conditions are not properly controlled. The most important factors contributing to nitrogen loss here are temperature, time, and ammoniation rate. These factors were studied by Bridger and Burzlaff at Iowa State College (*Ag. & Food Chem.* 2, (1954)).

Temperature. The most important factor governing nitrogen loss in the dryer is temperature to which the product is heated. Nitrogen compounds are relatively unstable at moderate temperatures; for example, diammonium phosphate begins to lose appreciable amounts of nitrogen at about 200° F. Other nitrogen compounds are more stable but all have an appreciable vapor pressure of ammonia at temperatures which may be encountered in fertilizer processes.

In the studies at Iowa State College it was found that appreciable nitrogen losses occurred in some cases at product temperatures as low as 150° F. Nitrogen losses as high as 20% were found to occur at product temperatures in the range of 250-300° F. and with inlet air temperatures in the range of 400-600° F. These high losses were for fertilizers made from ammoniation of normal superphosphate at a rate of 5.4 lbs. of ammonia per unit of available P_2O_5 and with a dryer retention time of about twenty minutes.

The loss of nitrogen caused by overheating can be easily demonstrated in the laboratory by heating samples of fertilizer to various temperatures. Depending on their composition, fume evolution will generally be observed in the temperature range 300 to 500° F.

Time. The nitrogen loss increases gradually with increasing dryer retention time so that conditions should be such as to achieve the desired degree of drying in the minimum time possible.

Rate of Ammoniation. Nitrogen loss on drying increases as the rate of ammoniation is increased, since diammonium phosphate is more easily decomposed than monoammonium phosphate. If therefore ammoniation is high enough to form diammonium phosphate, nitrogen losses on drying are more likely.

Storage Step

Nitrogen loss in storage of mixed fertilizers does not occur except under very unusual conditions. In high-nitrogen grades, it is possible that decomposition of nitrogen compounds may occur if localized overheating is permitted, in which case the decomposition may spread and a serious fire result. If alkaline materials such as lime are mixed with the fertilizer, liberation of ammonia will take place.

B. E. Adams

Losses of nitrogen during processing, the stages of processing, will be considered in the mixer, granulator, drier and cooler.

Mixer. The presence of ammonium nitrate and acid together may cause the ammonium nitrate to break down, but large losses are expected to be ammonia.

Granulator. No loss is expected.

Drier. Losses are a possiblity when flame is applied direct to the particle causing overheating. This can be corrected by proper control of the flame and air flow to avoid over localized heating. The product temperature should be checked where losses are expected in the drier.

Cooler. No loss is expected.

J. O. Hardesty

Nitrogen loss during the production of mixed fertilizers is caused by (a) adding neutralizing ammonia in greater quantity or at a faster rate than it can be absorbed (b) adding sulfuric in such manner that it reacts with potassium chloride before it can be neutralized by ammonia, the result being the formation of hydrochloric acid and its neutralization with ammonia to form ammonium chloride which is partially liberated from the mixture at high temperatures.

To the writer's knowledge, it has not been definitely established that nitrogen is lost under normal storage conditions. Considerable research work is in progress regarding this question. Excessive heat in the storage pile may cause nitrogen loss.[2, 7]

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[2] Davis, R. O. E., and Hardesty, J. O. Organic Material and Ammonium Nitrate in Fertilizer Mixtures. Ind. Eng. Chem. 37, 59-63 (1945).

[7] Hardesty, J. O., and Davis, R. O. E. Spontaneous Development of Heat in Mixed Fertilizers. *Ind. Eng. Chem.* 38, 1298-1303 (1946).

DISCUSSION ON PREVIOUS QUESTION AND ANSWERS

Frank Nielsson: I personally believe that your analyses are off. You haven't put the nitrogen in Or if you put it in, it went some place. (laughter) It isn't as funny as it sounds. By that, I mean, we're having a lot of work going on by our trade association, testing for nitrogen loss. Is it leaking out in a pile, is it leaking out in a bag, is it leaking out by the time it gets to the farmer? My personal opinion is that if it's in when it leaves your cooler, it's going to be in from there on out. And if somebody out in the field isn't finding it, it was never there when it reached the storage pile. This is due to a variety of reasons: Some are formulating techniques; some are the fact that people don't realize that continuous feeders are wonderful except that they require calibration; and some are that your salesmen come in and say, "if you don't give me fifty tons of this grade I'm going to lose a thousand-ton customer." So against your better judgment you may give him fifty tons. With the system most of us have, even if we take a reasonable recycle of twenty-five per cent, when you make only fifty tons of material, you're not going to make analysis unless you really over-formulate to play safe. The preceding are largely cliches to all the old timers in the field; that is, anyone that's been at it more than six months. But let's take these cliches, repeat them, and go over some of the points; maybe you'll agree with them, maybe you won't.

Now, the word gravimetric means that it's a weighing-type feeder. And yet for some unknown reason these weighing-type feeders don't strictly weigh per se; that is, depending upon what you're putting through them, your calibration point will vary.

Now, if we take an Omega feeder, about which I am most familiar, you have a little arm, connected to the weight beam that swings between two mercury switches located either side of the zero point. If the weight beam goes up or down too much your signal light lights and the bell rings, showing an unbalance. So the operator feels that as long as the bells don't ring, and the lights don't light, he's doing fine. But actually every once in a while, as the Omega people warn you, the weight beam may ride continually above or below zero without hitting the alarrm switch.

And although the bells don't ring, and the lights don't light, you're still off grade because you're running continuously one or two ounces over. Then, let's say you've been using a pre-based material in making 5-20-20 and you switch over to 4-16-16 base, the zero point of the scale changes, but not enough to actuate the switches. Again, you won't notice that you're off grade but you are off grade because to stay on grade the weigh beam has to move above and below zero, not just ride on one side of zero. The feeder people have provided what they call a little tuning device. This is regulated until the weigh beam begins to swing around dead center. When the weigh beam swings around dead center, then the feeder is doing pretty much what it's supposed to do. But, as long as it's swinging either below center or above center, you're off two or three ounces per foot of belt, and that adds up over a day's run. A lot of people just try to calculate feeder factors. In my own company, as I go out and talk to the field, I find some hellacious factors for feeders. To me the simplest way to find out what a feeder will do is to have it feed a weighed amount of material and time it. If you're supposed to be feeding a thousand pounds an hour, you put in a thousand pounds and see if it takes an hour to feed it out. If it doesn't, I don't care what the factors are, something is wrong. That is something that would seem very, very, primitive, and yet it's surprising how many engineers who design plants don't make provision for you to come off an elevator, or off the end of your belt or wherever you're feed drops before you get into the ammoniator so you can put a buggy or a cart of some kind, and actually time how much is coming out in a given period of time. If a plant doesn't have a means of weighing feeder input or discharge, they're just fooling themselves. They can use all the factors they want, and they can put on dead weights and what not, but they won't know what they're doing until they can actually say that "one pound per minute is what I'm getting." Now I think that's the biggest reason why continuous feeders are off, because they aren't calibrated. And after calibrating once you get this odd behaviour I mentioned before: If you switch from a formula that calls for 500 lb. per hour of triple and you change to ordinary super, and don't recalibrate, the feeder probably isn't going to put out the five hundred pounds per hour that it was set for. The weigh beam will balance slightly off zero and you're going to be off. So you have to calibrate for every material that you use. You have to calibrate for every base that you use. You have to recalibrate every two or three days. You have to check chemical analyses as a check on the feeders.

Now, for the matter of rotameters; again I'm assuming that most people are using Fischer Porter's. Those people have written a great big book on how to compensate for everything. So if you really get in trouble, go and read their book. They have a section on fluorators. Once you begin to understand it slowly you can make your own calculations. In most cases with solution as the specific gravity, changes for one solution, it doesn't make too much difference, but if you go to anhydrous ammonia, that happens to be a compressible fluid; as the pressure varies, the density varies, and it varies quite a lot. It can go from 0.52 to 0.62, which is a sixteen per cent variation over normal, between the time you begin unloading a car and the time you empty. If you don't account for that change in density, your analyses are'nt going to come out right. The anhydrous manufacturer can supply you with density data.

Now, more and more of us are beginning to use phosphoric acid. Again you have to write to the manufacturer for a viscosity curve; that is, how does the viscosity of his acid vary with temperature. Now, by viscosity, we mean the thickening condition that occurs with decreasing temperatures. As the temperature drops, acid gets thicker. For instance, using phosphoric furnace acid, the actual flow at 25° F., with the same float setting at the low end of the scale: That is, on the rotameter, the tube is tapered and the float is closer to the walls at the low end of the scale. If you're running in this range, as the acid gets thicker, the float is dragged instead of pushed. That is, it may be showing 100 lb. per hour when actually the flow is ninety (90) pounds per hour. The Fischer-Porter book tells you how to make those calculations roughly. In our case we give the operators a chart showing the actual flow compared with the flow shown by the meter for various temperatures. I'm sure that the acid people, or the rotameter people will be happy to make those calculations for you, if you can't figure out what the devil they're talking about.

Now, another thing that I believe sometimes affects what you do, or what the results are, is that you may not be using the right solution for the kind of product you want to make. We've had a lot of discussion today about solutions. My personal observations are that urea solutions generally make small granules, ammonium nitrate solutions make large granules; and the urana type, (urea ammonium nitrate solutions) make granules in between. If you use all anhydrous and sulfuric acid you get large-looking granules. Now, if you're sizing very closely down in the small particle size range, you're just cutting your own throat if you're using solutions that make big granules, because when you make big granules, you make most of your product by grinding; and your product is going to show it because it's going to be a rough-looking product that has been formed by crushing. You can go out in the pile and tell whether a man has been making round granules in rotating equipment or whether he's generating granules in the cage-mill or hammer-mill. If you're going to make small pellets, get the solution that will give you small pellets. If you're going to make big pellets; if your salesman thinks that you ought to have minus five plus six, go to a solution that will give you those. Certain solutions in certain formulae want to granulate in certain ranges.

Then, we get over into operating technique, and this depends on individual plants. If you're your own boss, like Mr. Sorenson, you can go out and do anything you want within limits. But at most places, you have to sell your ideas to people in the plants. You're doing this by letter or by telephone; you don't have as close control as you'd like to. So the troubles of the multi-plant company may not exist for the one plant company. Many of the old timers, realize that sulphuric acid is almost like glue. Put enough in and you can make almost anything granulate. But, you may get some funny results. You pick up a bag and the bottom drops out (too much acid). (laughter) Let's say the boy in the office has made a mistake on a formula. It won't quite granulate. But the operator will say, "well, heck, it won't granulate. I still have to make my tonnage." And he will increase his sulphuric acid above that shown in the formula. If he doesn't have a recorder, you in the office think he's doing what you told him to do so you can't understand the big shortage in sulphuric acid. The operators don't seem to realize that adding sulphuric acid is just like adding sand from the analytical standpoint. If you add a hundred pounds of sulphuric acid too much, you've added, on a dry basis, nine percent diluent and your analysis is going to come down. So when you start asking questions, go out in the plant and make sure that the operator is doing what you told him to do. It is surprising when you go out in work clothes and just talk to the operator on the side. He doesn't know who you are the first couple of times, and you'll be surprised at what you find. The important thing to tell them that in an emergency we don't care what they do as long as they tell us what was done. If a formula requires a hundred pounds more of acid, tell us and we will reformulate. I believe the addition of recorders will clear up many discrepancies.

Then the last item is this matter of recycle, and that again depends somewhat upon the kind of plant you have. I think everyone will agree, that the recycle and the product have different analysis. Using fine potash, the recycle will be high in P_2O_5. So until you come on stream, what you're making is either low in potash or P_2O_5, depending on raw materials. When you get through with your run, you have recycle left in the system and during the last part of the run you granulate that recycle, and that will be either high or low in P_2O_5 or potash, depending upon your formula. Now, if you're going to make only fifty tons or so of a grade, the first part is off-grade, the last part is off-grade; the middle is O.K. So as you go through a fifty ton pile, you may start out with a 5-21-18 and end up with a 5-18-21. And nobody ever finds a 5-20-20. And its' just due to this business of recycle. Another thing happens when recycle gets out of hand. A pug mixer will granulate recycle easily while a TVA ammoniator will not. Once the system gets out of step with recycle, the operators will stop feeding base material, and just add water to granulate that recycle. Then there may be fifteen, twenty or thirty tons of good-looking material, but it's completely off grade, that is a 5-18-21. They start up again and go though this process where they're making a 5-21-18 while the recycle is building up. It gets out of hand and they stop feeding base, and they granulate the recycle. The pile then is 5-21-18, 5-18-21, and back and forth. You can't tell the production manager why the state found the wrong analysis. As far as you could tell the average samples were O.K. But the fertilizer actually went into the pile in layers. And if it is made in layers of varying analyses, it usually comes out that way.

Question 1. Are Coating Agents in Granulation Beneficial? When and How Should They Be Introduced in the Process?

J. O. Hardesty

Data are presented [15, 19] showing the beneficial effects of coating agents on fertilizers having a tendency to cake. Coating agents are added from a continuous volumetric feeder, immediately after cooling or immediately before bagging, at the rate of 20 to 60 pounds per ton of fertilizer in a rotating cylinder in the absence of an air current. Unattached coating agent is removed by a screen at the outlet end of the cylinder and recycled in the process.

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15. Kumagai, Rikio, and Hardesty, J. O. Relative Effectiveness of Granule Coating Agents. *Agr. Food Chem.* 4, 132-5 (1956).
19. Rapp, H. F., and Hardesty, J. O. Granulation of Mixed Fertilizers in Experimental Equipment and Determination of Storage and Drilling Characteristics of the Products. *Agr. Food Chem.* 5, 426-33 (1957).

What Is the Best Weight of Ammonia Per Unit of P_2O_5 in Ammoniating Phosphoric Acid Derived From Either the Wet Process or Furnace?

T. P. Hignett

When phosphoric acid is the only source of P_2O_5, the maximum degree of ammoniation that was attained in the TVA pilot plant without excessive ammonia loss was about 7.2 pounds per unit. This degree was attained with either electric furnace or wet-process acid. Loss of more than 3 per cent of the free ammonia was considered to be excessive. A degree of ammoniation of 7.2 pounds per unit corresponds to conversion of about half of the phosphoric acid to diammonium phosphate and the remainder to monoammonium phosphate.

In information containing both ordinary superphosphate and phosphoric acid, somewhat higher degrees of ammoniation can be attained under favorable conditions than the sum of the standard rates for the two materials. This is because the calcium sulfate that is present in the superphosphate enters into the reaction. Pilot-plant tests were made by TVA in which 8-16-16 was made from a formulation containing 10 units of P_2O_5 from ordinary superphosphate and 6 units from phosphoric acid. Good ammonia recovery was obtained at over-all degrees of ammoniation up to and including 7.5 pounds of free ammonia per unit of P_2O_5. If the usual degree of ammoniation of 6 pounds per unit is attributed to the superphosphate, then 10 pounds per unit must be attributed to the phosphoric acid to reach the over-all value of 7.5.

The over-all reaction whereby phosphoric acid in the presence of calcium sulfate may absorb 10 or more pounds of free ammonia per unit of P_2O_5 may be written as follows:

$$3CaSO_4 + 4H_3PO_4 + 9NH_3 = 3(NH_4)_2SO_4 + NH_4H_2PO_4 + (NH_4)_2HPO_4 + Ca_3(PO_4)_2$$

The reaction involving formation of ammonium sulfate is not as rapid as the direct reaction of phosphoric acid and ammonia; some plants may have difficulty in attaining the higher degree of ammoniation.

When formulations containing large amounts of phosphoric acid are used in continuous ammoniators, the acid distributor should be designed to match or slightly precede the ammonia distribution . If the acid distribution lags behind the ammonia at any point in the ammoniator, increased ammonia losses may occur.

T. J. Bosman

When we first started using phosphoric acid at our Danville plant, we were fortunate in having a representative of the Girdler Corporation present. He told us that in their Votator studies, they had found an ammoniation rate that gave the equivalent of ½ mono-ammonium phosphate and ½ di-ammonium phosphate would maintain its liquid phase better at our ammoniator temperatures 190°F to 240°F, thus letting the ammoniator discharge more freely.

The stoichiometric quantity of free ammonia (NH_3) needed to form these compounds in a 1:1 ratio is 7.2 lbs. per unit (20#) P_2O_5. Starting on this basis we figured our ammoniation rate slightly higher at 7.4 lbs. free ammonia (NH_3) per unit P_2O_5. This was done to compensate for some slight ammonia losses from the ammoniator. We have found this ratio to work very satisfactory.

At times when we have varied from this ratio we have run into trouble. This applies to variations in either direction. You first notice that the mix is not moving through the ammoniator as freely as it should. On closer observation you will notice a build-up on your spargers and their supporting brackets. Should you catch it in time the chances are you can rod this build-up off without stopping your ammoniation. Should you be very far off on your 1:1 ratio the build-up becomes so hard and gains so fast you cannot catch up with it. Then you will have to stop all materials flow until this build-up can be broken loose. If you do not do this, it will choke down the ammoniator. This material has the appearance of a hard, dry, mass with a very rough surface. The pieces are very hard to break and the fracture looks like a very dense structure of compressed granules.

This condition is much more noticable with furnace acid than it is with wet process acid. The solid impurities in the wet process acid tend to a little more plasticity. With furnace acid you will have to use water or steam quicker than you would with the wet process acid. We have found variations from the 7.4 lbs. rate will cause you to use more water, or more water and steam regardless of type acid, therefore to us it is the rate to use.

Why is so Much Trouble Experienced With the Nitrogen Content of High Analysis 1-1-1 Ratio Fertilizers (12-12-12, 14, 14, 14)?

Elmer C. Perrine

The loss of nitrogen that is most frequently encountered and which accounts for much loss in the industry is failure to combine the free ammonia with the superphosphates and to some extent with the added acids in the formulae.

Largely through faulty equipment or operating techniques the unintended actions of some chemicals in the formulae result in some nitrogen loss.

High heats of drying in some granulation plants are possibly causing losses.

Screening of fines from granulated grades can influence analysis if they are not returned to process.

Improper handling of ammoniating solutions can reduce their nitrogen content. One of these actions is the excessive venting of storage and measuring tanks during transfer of volatile solutions. Another would be the use of a solution that had partially salted out in storage or in tank cars. The influence of these two factors is not very great in the average plant but in isolated cases have been severe.

It is true that many plants are retaining essentially 100% of the ammonia input at rates of 6.5 pounds per unit of P_2O_5 in 20% superphosphate and 3.5 pounds, for triple superphosphate. This requires equipment that is designed for the job and also requires considerable skill in operation. Some plants are operated as though the desire for the benefits of high rates of ammoniation is all that is required and they merely pour the nitrogen solution into the mixer without any recognition of the needs of equipment or techniques.

Beyond 3 pounds of ammonia per unit of 20% superphosphate considerable ammonia is usually lost to the atmosphere unless equipment and techniques are suitable for the requirements that are quite specific for high rates of ammoniation. Even with the best of equipment it seems impractical to hold more than 8 pounds of ammonia per unit of 20% superphosphate and this requires an input of 9.0 pounds, involving a calculated loss. Inputs as high as 12 pounds have generally failed to increase the retention much beyond 8.0 pounds. Much nitrogen is lost through this technique and not all operators are fully aware of the reasons.

Deterioration of equipment and the creeping tendencies of plant operation account for much loss of nitrogen. Where most of the ammonia is to be reacted with superphosphates it is the general practice to add 3.0 pounds of 66° sulfuric acid for each pound of ammonia that is in excess of that ammonia which is expected to be reacted with the superphosphate.

Where formulation demands the maximum ammonia take-up from the superphosphates, unless both the acid and ammoniating solutions are added in such a manner that each pound of ammonia has its chance to react with its alloted 3 pounds of sulfuric acid the conditions are present for loss of nitrogen from two sources. One loss caused by improper use of acids is from the unreacted ammonia that escapes to the atmosphere. The second loss may be through ammonium chloride escaping to the atmosphere.

Agglomeration before all of the ammoniating solutions have been added reduces or completely stops further ammonia take-up about as though the particle size of the superphosphate had been suddenly increased. Changing to another solution has corrected this condition, which may help to explain the reason for the number of nitrogen solutions being used for granulation. There have also been cases of unwanted granulation in pulverized fertilizer manufacturing which caused a problem in ammonia take-up.

Good operations in most granulating plants hold the products in the dryer to no more than 250° to 275°F and these are reached for a very short time in the dryer. As indicated in other discussions in this meeting such temperatures are acceptable to many operators. Some of the nitrogen salts are possibly broken down in the dryers that may be operating at higher temperatures. Excessive heats are often resorted to in order to offset some other factor which might better be corrected directly. Air pollution is often a result of excessive heats and other attempts to force a granulation plant beyond some of its limitations.

Does Heat Produce a Mechanical Breakdown and the Occasional Sublimation During Drying of Concentrated Ammonium Nitrate Solutions?

C. E. Franklin

The question speaks of drying concentrated ammonium nitrate solutions. If it has reference to the drying of ammonium nitrate, the answer is "No," providing the drying is carried out within reasonable temperature limits.

If the question refers to the drying of mixed fertilizers using increased quantities of nitrogen solutions containing ammonium nitrate, we notice no tendency for a greater mechanical breakdown in the dryer, that is, the pellet or fertilizer granule crumbling. On the contrary, with increased quantities of solutions, the pellets appear more firm and have a greater resistance to crumbling than do granules which have a lower soluble salt content and are agglomerated by the addition of water.

The chemical breakdown or sublimation during drying operations is influenced primarily by the fertilizer temperature in the dryer and, too, the fertilizer formulation. Laboratory tests on the drying of high nitrogen mixed fertilizers indicate that there are no nitrogen losses if the fertilizer temperatures in the dryer do not exceed 210°F. At fertilizer temperatures in the range of 210° to 250°F the losses are small—one to five pounds of nitrogen per ton. Above 250°F the losses increase rapidly with temperature.

We have only a limited amount of data on nitrogen losses from drying operations and have not as yet been able to prepare a correlation between nitrogen losses and composition of the fertilizer.

B. E. Adams

Yes, this is possible when we have a major drop in temperature between the mixer and drier resulting in a lowering of the liquid phase below that required for granulation. If material has hot points you may have sublimation.

Richard G. Powell

Let us begin by examining a few pertinent facts and some history concerning ammonium nitrate. Ammonium nitrate, as indicated in the literature, in pure form melts at about 226.3°F (169.6°C)[3]. While there is some disagreement in the literature as to the temperature at which decomposition may begin, the general range may be considered to be around 347° - 392°F (175° - 200°C)[4]. At temperatures of about 500° - 602°F (260° - 350°C), the decomposition may be self-sustaining[5]. Since the decomposition of ammonium nitrate is a series of chemical reactions, it is difficult to isolate any given reaction. Generally, there are considered to be about seven reactions though some sources for this information have consolidated them to three major reactions. All begin with the decomposition to ammonia and nitric acid followed very closely by the production of nitrous oxide (N_2O) as well as nitrogen (N_2). M. Berthelot and H. L. Saunders[4] in separate investigations conducted about 50 years apart, came in close agreement when they observed that "Towards 392°F (200°C) it (ammonium nitrate) separates in a sufficiently definite manner into nitrogen monoxide and water without, however, there being a fixed temperature at which this destruction takes place."[1] Saunders further noted in his work on the Decomposition of Ammonium Nitrate by Heat that, "Pure dry ammonium nitrate decomposes only slowly at 392°F (200°C) and just melting at 336°F (169°C) and then dissociating into nitric acid and ammonia and begins to evolve a gas containing 98 percent of nitrous oxide."[4] The other two percent was found to be practically all nitrogen (N_2). Thus, it would appear that with pure dry ammonium nitrate, the temperatures needed for decomposition are above 350°F (176.6°C) and at about 392°F (200°C) we may evolve nitrous oxide (N_2O). We should point out, however, that as with all chemical reactions, there is an extended range of temperature and pressure (the only two intensive factors to be considered in this analysis) over which the reaction takes place. The reaction may occur only to an infinitesimal extent at low temperatures, but it is analytically thermodynamically demonstrable. With such a low temperature reaction with respect to time, the kinetics may dictate an extremely long time (approaching infinity) for the reactants to be consumed or converted to products, provided such products are swept away from the presence of the reactants. At the opposite extreme is a temperature so high that the reaction at a given pressure proceeds with violent rapidity. Such extremes are important, but in the present study we are considering the conditions which prevail in commercial apparatus. The temperature, pressures and kinetics catalysts are of a significant order, creating conditions that may cause the rate of reaction and the magnitude of conversion to be of importance.

Thus, important to us also, is the effect of temperature when we do not have pure ammonium nitrate alone, but in the presence of various salts and acids that exist in our fertilizer mixtures. H. Tramm and H. Velde in their work on the Spontaneous Decompositions of Ammonium Nitrate, found that, "When free of chlorides, ammonium nitrate may be held at 347°F (175°C) for a long time without decomposition, but that relatively small quantities of chlorides and free acid may cause decomposition at temperatures as low as 284°F (140°C)."[6] This decomposition as indicated, would rapidly proceed to the evolution of nitrous oxide (N_2O).

Now that we have the temperature ranges in mind at which this decomposition and possible subsequent formation of nitrous oxide may occur, let us examine the possible temperature conditions existing in the ammoniator. First of all, as you can appreciate, the reactions that are taking place at any one moment in the ammoniator are hard to predict. It is equally difficult to predict accurately, the temperatures existing in the ammoniator at the time of our maximum heat of reaction. Generally speaking, unless acid is being used, the temperature conditions which would promote decomposition of the ammonium nitrate are not readily attainable. Once we begin to neutralize a portion of the ammonia present with acid, however, considerable heat is evolved in addition to that which is normally encountered from the reaction of the ammonia and the normal or triple superphosphate. Assuming an average specific heat, based on our normal operating temperatures, of approximately .26 for fertilizer materials, .65 for solutions, and .4 for ammonium nitrate and also assuming a conservative heat of reaction of 2200 BTU per pound of ammonia from solution when reacted with 66° Baume' sulfuric acid and 1000 BTU per pound of ammonia from solution when reacted with normal or triple superphosphate, certain adiabatic equilibrium temperatures may be predicted. Considering an isolated point in the mixer at a given moment when the solution, acid and phosphatic material are being mixed, an estimation of the heat of reaction may be calculated. When this heat is applied to the various constituents present, individually or as a group, and over a range of initial temperatures for the constituents starting at 70°F, adiabatic temperature conditions of from 310° - 450°F (155° - 233°C) may be predicted. The more usual condition, that of dissipating the heat into the majority of the moving mass of fertilizer as opposed to consideration of isolated pockets where only solution and acid would be present, would indicate abiabatic temperatures in the immediate range of 374° - 392°F (190° - 200°). Therefore, assuming that pure ammonium nitrate begins to decompose at temperatures approaching 392°F (200°C), and ammonium nitrate in the presence of chlorides or free acid may begin to decompose significantly at temperatures above 284°F (140°C), it would indicate that under adiabatic conditions ammonium nitrate could decompose in the ammoniator and further that the decomposition products could be primarily nitrous oxide.

Fortunately, the mass of solids present, the evaporation of water, the agitation of the material and the movement of air in the case of the continuous ammoniator, all help to dissipate these heats of reaction very rapidly. Also, the dilution of the ammonium nitrate and reaction of the ammonium nitrate with potash, aids the prevention of its decomposition. That these abiabatic conditions do not exist long enough generally, to cause decomposition of the ammonium nitrate with potash, aids the prevention of its decomposition. That these adiabatic conditions do not exist long enough generally, to cause decomposition of the ammonium nitrate, is partially borne out by pdoduction records which seldom indicate temperatures in the ammoniator much above 200° - 250°F (93° - 139°C). Therefore, while it is possible to obtain the temperatures in the ammoniator that would cause ammonium nitrate to breakdown, under normal operating conditions, these temperatures are not reached or held long enough to cause significant decomposition. Primarily, the latter is due to the fact that true adiabatic conditions do not prevail and the heat is dissipated to the surroundings.

I believe many of you have seen the decomposition of ammonium nitrate taking place, usually accompanied by the issuance of gray-white vapors, which are partially finely divided ammonium nitrate resulting from the recombination of cooled, dissociated ammonia and nitric acid, and partially ammonium chloride, and then the formation of reddish brown vapors of nitrous oxide. In my experience, this has occurred when the fertilizer became too plastic in the ammoniator and the bed of material was not tumbling or being agitated in the normal manner. To point out an example of this, I would like to refer briefly to an experience of Swift and Company, which was quoted very widely recently in industry magazines.

Swift and Company experienced, while producing 12-12-12 using nitrogen solution, anhydrous ammonia, and 66° Baume' sulfuric acid, an unscheduled shutdown requiring their dumping the material from the mixer into a separate pile. The pile began to evolve dense, white fumes and gave evidence of spontaneous reacting, and a check indicated the pile had generated a temperature of over 500°F. In subsequent laboratory tests, they found that fertilizer salts promote the rapid breakdown of ammonium nitrate when heated to 420°F. Further, when the mixture contained about 6% or more of ammonium nitrate and was heated to 450°F, the breakdown became extremely rapid and self-sustaining. This would occur as indicated by the temperature condition we mentioned previously. Their recommendation for eliminating same was, of course, avoidance of large, hot lumps, a careful balance of the acids, solution, and anhydrous ammonia entering the mixer, and we add to this, the avoidance, as much as possible, of a static condition in the bed of material and an over-plastic condition.

We now come to consideration of the effect of heat during drying. The dryer inlet air is usually in the range of 300° - 500°F (147° - 260°C). The product temperature of the material in the dryer is generally in the range of 150° - 175°F (65.5° - 79.4°C). As we pointed out earlier, the presence of chloride salts depresses the melting and decomposition points of ammonium nitrate. If we consider the decomposition temperature lowered to 284°F (140°C), it does not appear likely that any breakdown would occur unless localized heating existed due to a poor pattern of the falling material or prolonged contact with inlet dryer air or with direct flame. Breakdown can be obtained, as we all know, if material entering the dryer is above normal temperature for material leaving the ammoniator, this being about 200°F (93.3°C) and inlet air temperature or direct flame is too high correspondingly. If a breakdown did occur, subsequent decomposition and loss of nitrogen would be expected. That though, does not generally happen if dryer conditions are normal. This is partially substantiated by the work done at Iowa State College under the direction of Dr. G. L. Bridger.[2] In their study of nitrogen loss in drying ammoniated superphosphate and mixed fertilizer, they conducted various tests at a constant inlet air temperature of 500°F (260°C). Their work showed that when the free acid was neutralized, the loss from ammonium nitrate was negligible. Using ammonium nitrate alone with superphosphate, the ammonium nitrate broke down readily in the dryer due to its decomposition by the free acid of the superphosphate. However, when ammoniating solution was used, losses were negligible under proper dryer conditions and whatever losses were experienced were "due primarily to compounds formed from free ammonia." The losses were also negligible when ammonium nitrate was the sole source of nitrogen when potash was included. The potash also helped prevent the decomposition of the ammonium nitrate by the free acid of the superphosphate even though chloride salts depress the ammonium nitrate decomposition temperature. Since most of the work done at Iowa State College included product temperatures of 200°F (93.3°C) and higher, with the nitrogen losses remaining negligible for the most part unless the product temperature reached 250° - 290°F (121.1° - 143.3°), we may reasonably conclude that the ammonium nitrate does not under normal operating conditions, break down in the dryer.

Insofar as sublimation is concerned, it is rather doubtful that any sublimation would occur. While pure ammonium nitrate may partially sublime above 212°F., the low decomposition temperature of ammonium nitrate in the presence of chloride salts or free acid and the very low partial pressure of the decomposition product would seem to indicate that sublimation would be superseded by decomposition.

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SECTION 4

What is Considered Adequate Instrumentation for Satisfactory Plant Control?

Walter J. Sackett, Sr.

MY opinions relative to this question are based on the continuous granulation of mixed fertilizers. Basic instrumentation, in my opinion, consists of:

A. Instruments required to measure and/or record the flows of liquids and solids to the ammoniator.

B. The instruments required to control the dryer.

A.—While an elaborate system of instrumentation can be used in a continuous process for the production of granular mixed goods, such an arrangement can become so complex that its cost cannot be justified. Rather than take the time necessary to dwell on a detailed description as to what the ultimate in instrumentation would consist of, I shall limit my thoughts to the practical aspects of instrumentation based on our own experience.

So far as the instruments required to measure and/or record the flows of liquids and solids to the ammoniator are concerned, it is imperative that adequate means be provided for accurately controlling and indicating the flows. For this purpose, rotameters are the most widely accepted means of accomplishing such control of the liquids. Our experience has shown that these meters should preferably, although not necessarily, be supplied with recording and totalizing devices. It strikes us that the extra money spent for such recording and totalizing devices is a wise investment in that it permits a day-to-day record to be kept of each operating period. In this way, should some problem of analysis or operating condition develop, the records made by said recording devices on previous runs of the same grade can be used to good advantage.

The continuous scale used to weigh the pre-mixed solids to the process should, likewise, be equipped with some method of determining the rate of flow being weighed by said unit. It is our opinion that tie-in devices, which attempt to correlate the flows of liquids with permitted fluctuations in the flow of the solid materials, are necessary and, as a matter of fact, can cause serious operating and maintenance problems. We have found it best to set all materials, both liquid and dry, to some pre-determined rate of flow and maintain them at that point.

B.—So far as the instrumentation of dryer control is concerned, we feel that it is necessary to employ accurate control instruments in the form of a Potentiometer to automatically regulate the temperature of the inlet gases and that this automatic equipment obtain its signal from a control unit set up to measure either the temperature of the exit gas or of the material in process as it leaves the dryer. This instrument is, perhaps, the most important control in the process. Its cost will be returned within a relatively short period of time by fuel savings alone.

Other dryer instrumentation includes such items as high temperature safety devices which prevent overheating of the combustion chamber and approved flame safety units which should be installed to afford safe operation of the burner equipment to satisfy insurance requirements. It is sometimes advisable, depending upon the size of the operation and the complexity of the formulation, to install a multi pen type recording instrument to record temperature at, perhaps, four to five places throughout the system. However, we feel that this type equipment can normally only be justified on the larger granulating units.

It must be understood that the opinions expressed in my discussion relative to dryer instrumentation actually revolve around the attempt to approximate material-in-process moistures through dryer temperatures.

I should like to say that we feel there is a definite need for one additional type of control instrument which, to our knowledge, is not available in any performance-proved unit. The unit we refer to would have the ability of continuously recording moistures of the material as it leaves the dryer.

R. Webber

The question should be broken down into two parts; namely, (1) batch ammoniation for production of either nongranular or granular fertilizers, and (2) continuous operations for the production again of either granular or nongranular materials. The initial consideration in batch systems for the production of nongranular fertilizer production, by far the biggest single consideration is the accurate weighing and liquid metering systems for the proportioning of raw materials. Weighing systems may consist of a scale mounted weight hopper or a floor mounted scale over which the raw material passes into the process. Liquid metering can be accomplished by a system of scale mounted measuring tanks either operated manually or automatically while your metric measuring tanks are a third and less frequently found system. Positive displacement type meters with provisions for automatic shut-off after delivery of a pre-set amount of liquid. Any combination of the above have been used successfully in the industry. The volumetric method is used for metering solutions, a calibration chart using water as a calibering fluid can be used in conjunction with published physical constants of the various nitrogen solutions to maintain desired accuracy. If an automatic shut-off meter is employed or an automatic scale tank, the error inherent in human judgment required for any type of manual operation is minimized. The positive displacement type of meter is also an excellent device for inventory control purposes because of its built-in totalizing feature. There are plants, the B part of the batch processing methods in which granular fertilizers are produced batch-wise normally by batch ammoniation followed by continuous granulation, drying and cooling. Generally in this type of operation the weighing, liquid metering, and batch ammoniation apparatus associated with nongranular fertilizer production is utilized in conjunction with some means of smoothing out surges to feed continuous drying, cooling, and classification equipment. Two and sometimes three additional liquid raw materials are used; namely, sulfuric acid, phosphoric acid, and water. The same remarks listed earlier in liquid metering apply to these three materials. The even larger number of liquid materials makes the choice of positive displacement instruments even more attractive from the control viewpoint provided that these materials are reasonably free of sludge. If spent or by-product sulfuric acid or wet process phosphoric acid is used in formulations, periodic cleaning of a volumetric type measuring boot or positive displacement type meter is recommended to insure sustained metering accuracy.

Next we get into completely continuous systems both for production of nongranular and granular fertilizers as carried out in various sections of this country. Continuous nongranular plants, again, the significant instrumentation problems are associated with solids weighing and metering, liquids weighing and metering, and in temperature control These cover the three general areas. Solids weighing devices may include a time-sequence batch weigh and mix system followed by some means of ironing out fluctuations in continuous feed system, or may be of the gravimetric weigh belt feeder type. Liquid metering devices include the variable area flow meter with its lower temperature limitations which were discussed previously, and just to recount the various methods available to meter viscous liquids, these are: Metering pumps, positive displacement meters which can be equipped with tachometers to give a measure of instantaneous rate as well as totalizing feature; round edge orifice meters which do exhibit some degree of immunity to viscosity changes; and the magnetic flow meters which have recently been introduced both by the Foxboro Company and the Fischer-Porter Company. In time sequence batch systems a search hopper equipped with variable speed belt is commonly used. An approved but more expensive method of continuously metering solids uses one or more gravimetric weigh belt feeders. Under such a system pre-mixed rock solids are fed continuously by single weigh belt feeder or solid raw materials are fed individually using a series of feeders feeding material onto a single belt. Either system is considered adequate provided that routine calibration checks are made. In the time-sequence system as well as the weigh-belt method, some independent means of check-weighing the total stream being delivered to the ammoniator is considered essential to good control of grade analysis. No straight volumetric feed system is adequate for a continuous ammoniation system where various grades are made at varying production rates.

The most common method employed for metering liquid materials in the industry is the variable type flow meter with either manual or automatic control. Nitrogen solutions, anhydrous ammonia, sulphuric, phosphoric, and water can be metered and/or controlled accurately with this device in the majority of cases, and in most cases this is the most economical approach as has been discussed previously. The question of manual vs. automatic control must be resolved on an individual basis, and I think the intent of the second part of this question as to how much instrumentation can be justified, the answer is well-known. Strictly, what is required to produce a quality product at adequate production rates and minimum costs. I think that no generalization can be made here without consideration of the individual plant size and peculiar problems. I believe the subject of metering viscous liquids has been covered adequately, so I will skip that and get on to the production of granular fertilizers on a continuous basis. Essentially the same system is used except that agglomeration is promoted and a drying and cooling problem is presented. The minimum instrumentation for carrying out the drying operation would consist of a flame protective device and temperature limit cut-off to eliminate the possibility of explosion from accumulation of fuel and air and to protect the equipment from excessively high temperatures. In addition, a device to indicate temperature of the input gases and temperature of the exit gases from the drying operation is considered essential.

At the other end of the scale almost completely automatic control oi the drying and cooling operations may be realized if production capacity and production costs will justify it. And rather than cover this subject, I would refer you to the several excellent discussions that were given in the 1956 Fertilizer Round Table. Mr. Foltz of General American Transportation and Mr. Leister on Fundamentals of the Renneburg Company in Baltimore, plus the other speakers who have been on the program today. Certainly an industry can justify much more additional instrumentation than is currently applied if considerations of post-grade control are considered.

Dr. Sauchelli: Thank you, Mr. Webber. Any questions for Mr. Webber? I'm sure it's a subject that's of very direct concern to the industry at this time and it is going to be increasingly so.

DISCUSSION

Mr. Perrine: In an interlock system whereby the acid and nitrogen solution is automatically cut off in a continuous system when the dry materials fail, I find too few of those going in and when they do go in and get out of order it is a simple matter to just turn them off. Do you run into that?

Mr. Webber: Very definitely, Mr. Perrine. We have recently installed pilot plant equipment and this was one of the features. We've interlocked the solids weigh-belt feeder with the automatic control valves for feeding aqueous solution and acid to preclude emptying these two trails together and initiating nitrate decomposition.

Question: Another situation parallel to that where all the operations may be good as far as the input is concerned, but in the continuous granulator, ammoniator, you may have the material just slide up and back without mixing and ammonium nitrate solution and the acid we pour in there underneath the bed in essence would be poured directly into each other. Is there any interlock system that might prevent that?

Mr. Webber: Off hand I can think of none at this time. I believe that a cheap five dollar ammeter put in the ammoniator drive circuit is an excellent investment. I think a device of this type will clearly indicate malfunction in the ammoniator as reflected by fluctuating power load on the drive.

Dr. Sauchelli: A question here. Mr. Slack.

Mr. Slack: I believe you said that you thought the time-sequence method was satisfactory. Is there enough advantage to other methods to install them?

Mr. Webber: I think, Mr. Slack, that economics dictate the choice in many plants that are converting over initially can do so at a minimum cost by adapting equipment that they have. They're already set up with scales; they're already set up usually with a batch mixer.

Mr. Slack: That's what I mean. If they already have that equipment, is there any point to using any other than the time-sequence setup?

Mr. Webber: I think most manufacturers have answered that by adopting the time-sequence system. New plant installations, I think, would have to—the economics would rest on a detailed approximation of the two systems side by side. As you say, most people who are making the conversion from nongranulation to granulation have quite readily adapted this time-sequence method. Again, there is no substitute for a scale mounted bucket or a grome and a stopwatch to check the accuracy of solid weigh-belt feeders or variable belt feeders, either the time-sequence method system or the gravimetric weigh-belt system. Certainly both techniques pay off.

Practical Methods for Checking Solution and Acid Flowmeters to Determine Actual Versus Indicated Rates of Flow.

A. Behler

At one time or another, a good many of you attending this Round Table have experienced the situation which caused you to question the calibration or accuracy of metering equipment handling the liquid fertilizer ingredients in your mixed goods plant. Perhaps it was low nitrogen analysis on a certain grade or discrepancy in tank car weights over integrator readings after a particular car was empty; in any event, you immediately pointed a finger of accusation at the flowmeter, because, perhaps, it was the one item in your system with which you were the least familiar.

It is my privilege to discuss with you some of the means of checking the calibration and alignment of liquid flowmeter equipment after it has been placed in operation and to answer any questions you may entertain in this regard.

In checking any device, it is mandatory that one understand the basic principle under which it operates, so for a brief moment, I would like to review some of the basic features of flowmeter equipment presently used in most continuous mix goods plants. The Variable Area or Rotometer consists of a tapered corrosion resistant metering tube into which a free float is introduced, the assembly being mounted vertically. Liquid is introduced into the bottom of the tube at the velocity of a moving stream causing the float to assume position in the tube proportional to instantaneous flow rate. The capacity of any meter being a function of tube taper and weight of the float. Meter tubes or armored meters are preferred for nitrogen solution anhydrous ammonia and acid because of their safe-proof construction. The black box above the meter contains a pneumatic transmitter which is actuated through a permanent magnetic coupling and provides a 3 to 15 lbs. air output to a remote controller—I say a remote controller deliberately, for in most of our plants the flow transmitter must necessarily be mounted in an atmosphere of corrosive fumes and in a location subjected to vibration. The controller, on the other hand, does not have to be mounted in the pipe line, can be located in a separate control room where the atmosphere may be kept clean and vibration entirely absent. A diaphragm actuated control valve is throttled continuously by the controller to maintain the rate of flow at a preselected value.

In checking the calibration of a flow control system, it is mandatory that the steps follow a sequence starting with the primary metering device then progressing through the rest of the loop. By the use of proper valving and a suitable container you can conveniently introduce plant water ahead of the flow transmitters and collect same into a 55 gallon or larger drum fed by a flexible connection downstream from the meter. Through a hand valve set the rate of flow at approximately 20% of maximum and hold there while accurately timing the run to fill the container. Repeat for 50% and 80% of full scale. The volume over time will give gallons per minute as well as pounds per hour of water equivalent to each of these settings. These should agree with the equivalent water capacity to the flowmeter within plus or minus 2%. If they do not, stop right here and check to see if there is any mechanical damage or corrosion to the float or metering tube. The meter manufacturer will gladly supply you with the proper float weight and key dimensions. With the assurance that the basic calibration is correct, the output air pressure of the pneumatic transmitter should be checked for various flow rates by observing the output pressure gage and flow indicating scale on the transmitter. A U-tube manometer is recommended for measuring this air pressure if highly accurate readings are desired. The indication of flow on the remote recorder should agree with the primary flow transmitter, and if this is not the case proper adjustments for O shift and linearity should be made in accordance with the meter instruction book. Other details concerning the air system and controller adjustments are contained in these same instruction books. A simple check should be made on the integrator at 20, 50 and 80% of flow rate by checking integrator readings over flow rates and timed for a period long enough to assure that any erring time would not affect the test by more than one percent.

It is suggested that one man in each plant be placed in charge of the instruments whether he be from maintenance or from production department. The metering equipment manufacturers provide free schooling for these men in the proper operation of servicing of specific instruments installed in your plants. A service contract with the metering company will insure annual or semi-annual checkup with proper maintenance made by an expert on these instruments at a reasonable cost.

In the northern part of this

country and other regions where acid temperatures may fall 30° F. and below increased viscosities of both sulphuric and strong phosphoric acid will necessitate the application of temperature correction to flowmeter settings in order to obtain accurate dosage from predictability data. Our company will supply a family of curves at nominal cost by plotting scale readings vs actual flow rates in PPH for the range of temperature over which the meter is to be operated. It is absolutely necessary that this be done since for example the 20°F. on typical 5,000 PPH sulfuric acid meter would result in errors of approximately 15% without this correction.

Obstructionless flowmeters of the magnetic type are currently coming into production and will be used particularly for wet process phosphoric acid. They are considerably more expensive than the variable area meters for this service, but we feel that the featuure of viscosity immunity justifies this extra cost. A reprint of a recent ISA Journal, "How to Select Flowmeters," is available for those interested in this subject.

How Can Acids Be Metered When the Temperature Is Below Freezing, or When the Liquids Are Quite Viscous?

Elmer C. Perrine

SOME rotameters or flowrators used in continuous operations are capable of accurate performance through a considerable range of viscosity. Also a meter can be chosen to handle quite viscous materials but would be unsuitable for metering like quantities at greatly reduced viscosities. Perhaps two meters would cover the range of viscosity and would be the better answer in the final analysis.

Since viscosity can be controlled by heating and the volume of acid by heating and if the volume of acid should be a fairly simple matter to warm the acid ahead of the meter in the piping to ranges of viscosity within the ability of the meter.

This may be a more simple and satisfactory answer than the choice of changing to a metering pump which may be designed for immunity from the influences of varying viscosity.

J. E. Seymour

The successful metering of phosphoric acid or sulfuric acid during freezing weather is dependent upon the selection of the proper metering equipment if the temperature of the acid in question is less than 40°F. or at acid viscosities above approximately 40 centipoise.

Relatively clean phosphoric acid or sulfuric acid solutions have been successfully metered during freezing weather with positive displacement meters, positive displacement pumps and magnetic type flowmeters.

Although variable area flowmeters do an excellent job of metering clean solutions of phosphoric acid or sulfuric acid above a temperature of approximately 40°F., temperatures below 40°F. (with the accompanying rapid rise in viscosity of these solutions) result in inaccurate metering unless the meter is recalibrated at each viscosity of acid solution (for each temperature) and at each flow rate desired. This is particularly unfortunate in that the majority of metering devices in use by the fertilizer industry are of this type. The most logical solution to using variable area flowmeters to meter phosphoric acid and sulfuric acid during freezing weather is to maintain the acid temperature above approximately 40°F. The foregoing statements applicable to the variable area flowmeter are also true for the round edge orfice meter.

Inasmuch as positive displacement meters, positive displacement pumps and magnetic type flowmeters effectively meter relatively clean phosphoric acid or sulfuric acid solutions independently of viscosity, other factors such as total equipment cost, maintenance time and cost, flexibility, accuracy, service and ease of calibration should dictate the specific choice of equipment.

Mr. James Malleck: We have a little information on measuring of these acids in cold weather. Our operation is located about twenty miles west of Salt Lake City, Utah. Our winter temperatures have gone as low as 15° below zero, and summer temperatures are as high as 110°F. This is a dry area so it isn't the humidity we notice, it is the temperature. I won't be as enthusiastic, I think, as the gentlemen were about the Foxboro. We have been using a Mega Rotodip liquid feeder for both sulphuric and phosphoric acids. The temperature of sulphuric acid runs from about 20°F. to about 90° or 95° over a year's weather changes. I have a few figures on the apparent accuracy of one feeder used on sulphuric acid during a six-month period compared to storage tank tonnage measurements. These figures are based on an average per month's delivery. In July, the actual delivery was one per cent higher than the tank measurement. August, it was one-tenth of a per cent higher. September, one-tenth of a per cent high. October, the feeder totals were the same as the tank outage. November was a little cold this particular year and we went to .9 of a per cent lower than the tank. December, it was .3 of a per cent lower. We assume these differences are due to viscosity changes because this being a dipper wheel, instrument viscosity would tend to reduce the actual delivery per rotation of the wheel. Apparently this more than offsets the density changes. Unfortunately we have no such seasonal comparisons on feeders used on green phosphoric acid for a variety of reasons. The tankage is not controlled so closely. However, the dipper wheels will develop some scale, particularly on the lower strength green phosphoric. These will introduce errors greater than the ones I have discussed on sulphuric. Viscosity changes no doubt do affect the delivery. This can be compensated for in setting the control points.

What Is the Extent of Fumes and Fires in the Industry? What Dangers Are Involved and How to Avoid Them.

Elmer C. Perrine

GENERALIZATION about fires could be as dangerous as fires themselves, but many of them that occur during the mixing process do so while operating conditions are quite inefficient, particularly in granulating operations.

The greatest hazards from fires and fumes as well as the costs from lost nitrogen and inefficient use of acids stem from allowing acids to contact some other ingredients in the mixer in concentration very greatly in excess of the over-all ratio of these products in the formula.

The subject is so important and the consequences can be so serious that short simple answers seem out of place. Fortunately a good measure of control has resulted when all operations were conducted as though all ingredients were very high priced and any serious deviation of established good practices would yield a product that could not be sold. That sounds like a simple answer and not all of the reasons may be known.

It is true that among the several products that are being used in high analysis granulated fertilizer some of them if used exclusively with each other would require equipment and techniques that differ considerably from the equipment now used in fertilizer manufacture. The best protection seems to come from the diluting effect of the other ingredients which far outweigh the ones in question.

The uniform distribution of all ingredients particularly as they are being added is a means of avoiding local heavy concentration of any of the materials. This includes even any added water and certainly includes any acids and ammoniating solution.

Means whereby the acid is neutralized promptly with the first part of the ammoniating medium are to be advised. Correctly performed there can be little free acid to act on any material after only a small portion of any large amount of nitrogen solution is applied. Creeping practices and corrosion or abrasion that slowly change good operations can create hazards in a plant that may have been quite safe when originally started. Frequent detailed inspection is very necessary and, of course, a knowledge of what is required of equipment and techniques.

Although this statement must not be accepted as a safety suggestion there are few fires and fumes when all of the ingredients are being used at their high efficiency. It would be interesting to determine what the cost in lost nitrogen and unnecessarily high acid consumption would be in the month preceding a fire in the mixer or the meeting of the town board to discuss the air polution from the local fertilizer plant. It could easily be a dollar a-ton of fertilizer and could also be two dollars.

How Does One Handle Nitrogen Solutions Having High Freezing Points?

Elmer C. Perrine

SOME part of the millions of dollars which the nitrogen producers have devoted to rolling stock has been in the interest of delivering those products to the customer which best suit his needs. The insulations of tank cars make it possible to deliver solutions at temperatures that are satisfactory in many cases of normal operations. In extreme cases, a change to another solution may be indicated.

One recent study of the formulating practice in a granulating plant revealed that the total savings in all plant foods by using solution X amounted to about two dollars per ton of fertilizer over the current formula using solution Y.

The economy of using Solution X was faced with the need for insulating the nitrogen solution pipe line due to the high salting out point or with using the solution only in suitable weather. At 100 tons of fertilizer granulated per 8 hour shift this involves a possible economy of $200 per shift. However, insulation alone is not enough in some cases.

It so happens that those managements that are interested in the nitrogen solution that happens to have a high salting out point are often using it at rates that will empty a tank car in two and often in one day thereby minimizing the influence of high salting out points.

When operations can be scheduled to use the solution directly into process shortly after arrival through insulated piping the operator greatly increases his choice of nitrogen solutions and can operate all year round with that solution which has proven most suitable from its merits independent of its salting out temperature—or in most cases its vapor pressure.

Solutions that are saturated at relatively high temperatures should not be permitted to remain static for very long in the pipe lines. To avoid the very disturbing feature of a solution slowly filling the pipe line during shutdown periods it has been a very satisfactory practice to empty the pipe lines and disconnect the tank car.

Where volume, irregularity of delivery or availability of track space may be serious problems then insulating and even heating and insulating the storage tank may be in order. Not only will insulation open the way to the use of nitrogen solutions of high salting out temperature but will also contribute to peace of mind in the use of higher pressure solution.

The storage and metering of these latter solutions also create some problems of heat transfer that are mitigated by insulation.

Has Anyone Used a Foxboro Magnetic Meter for Metering Viscous Fluids?

W. I. Strauss

THE Foxboro Magnetic Flow Meter is successfully applied to the fertilizer field. Over 50 such meters are already in service in this field alone. Most common is the measurement of phosphoric acid.

However, applications with sulfuric acid and phosphate slurries are also numerous.

In the manufacture of wet process phosphoric acid, for example, the measurement of the phosphoric acid is handled routinely by the magnetic flow meter, resulting in increased yield through better process balancing. Concentrated sulfuric acid and phosphate slurry are measured and controlled using the Foxboro Magnetic Flow Meter as the primary device. Crystal size can be better controlled through a combination of proper flow measurement and laboratory analysis. This leads to a minimum of filtration problems. Previously, measurement of these viscous materials (concentrated sulfuric acid and phosphoric acid), corrosive fluids (phosphoric acid and sulfuric acid of various concentrations) and slurries (phosphate slurries) were both a maintenance and measurement problem. Orifice plates of special design and metal were required, or the expensive venturi tubes were used. High maintenance seal systems and complex purge systems were employed.

As in the flow measurements for the wet process system, phosphoric acid by the furnace process has a common measurement problem. The phosphoric acid is approximately the same concentration as the wet process acid; therefore, the viscosity problem is the same. As the ambient temperature approaches 32°F., the viscosity changes rapidly. This would cause a significant error of flow measurement using conventional type flow meters.

The application of the magnetic flow meter for phosphoric acid flows affords accuracies to ±1% full scale with maintenance problems kept at a minimum. There are no seals or purges required. The meter measures volume rate of flow directly, such as gallons per minute, therefore turbulence or changes in viscosity have no effect on the instrument from an application or measurement viewpoint.

The principle of operation of this meter is not new. It is based on Faraday's well-known law of electromagnetic induction; the voltage induced in a conductor of a given length moving through a magnetic field is proportional to the velocity of the conductor. The voltage is generated in a plane which is mutually perpendicular to both the velocity of the conductor and the magnetic field.

A uniform magnetic field is induced through a portion of the flow tube (which is essentially a piece of pipe) by a coil outside the tube. The flowing liquid itself is the conductor moving through the magnetic field. Consider a cross-section through a cylindrical column of liquid which is a flat disc. As the flat disc is moved through the magnetic field, a voltage is developed. If a pair of electrodes is inserted in the tube wall in the proper relation to the magnetic field, this voltage can be measured. Length of the conductor becomes the distance from one electrode to the other; that is, the flow tube diameter.

The meter actually adds incremental velocities across the tube from one electrode to the other. It can be seen that turbulence or variation in flow profile due to flow rate or viscosity does not affect accuracy since the generated voltage is proportional to velocity. This permits installation of the flow meter adjacent to control values or pipe elbows. This can not be done with conventional flow measurement devices if reasonable accuracies are required.

If air bubbles or other material are entrained in the liquid, the meter will read the volume rate of flow of the total mixture. It is only necessary to have the tube completely filled with a homogeneous mixture of all times.

The basic requirement for the flowing fluid is that the fluid have a minimum conductance of approximately 200 micromhos in line sizes to 6″ and 50 mho in larger line sizes.

Various lining materials can be furnished to meet corrosive conditions. Neoprene, Kel-F and Teflon materials and glass are standard liners. Many types of electrode materials are also available for the same reason. Type 316 stainless steel, platinum, Hastelloy C Alloy, nickel, Monel Alloy and tantalum have been used. The maximum operating temperature is presently 365°F., but experiments are under way to increase this—possibly as high as 600°F.

The flow transmitter signal is recorded by a Foxboro Magnetic Flow Dynalog Instrument, calibrated in units of flow. operating temperature is presently 365° Fahrenheit, but experiments are under way to increase this possibly as high as 600° Fahrenheit. The flow transmitter signal is recorded by a Foxboro magnetic flow dynalog instrument calibrated in units of flow.

DISCUSSION

Mr. A. Spillman: Thank you, Mr. Strauss. Mr. Strauss, what are the costs of the Foxboro meter? Do they range in cost as to size?

Mr. Strauss: Yes, for a straight recording instrument, the dynalog, the recorder itself, is the same regardless of the size of the metering tube and that's $800. And we have the metering tube available in sizes from one-half inch which costs $750 approximately with the standard lining materials up to—we furnish them up to six feet. This is no reason why we can't go any higher than six feet.

Mr. Spillman: In other words, are you saying if we bought a Foxboro meter to measure, say, 4,000 pounds an hour we could buy one and install it for $750.

Mr. Strauss: No. The recorder costs $800 and on top of the $800 you have to add the cost of the transmitter which ranges from $750 in a half-inch size on up. The average installation in the fertilizer industry, I think the common size is one-inch and two-inch size, is approximately $1800 to $2000. The size of the transmitter incidentally, it doesn't make any difference how large the flow is. There is a minimum requirement in accordance with the sizes. For example, in a half-inch size, three gallons per minute is the minimum full-scale value, but if you could push a ridiculous amount of fluid through there up to 1,000 gallons, this would have no effect on a meter, a meter can read anything on upwards. There is a minimum requirement per size for maximum value.

Question: Can this meter be used as an indicating meter only?

Mr. Strauss: Yes, it can be used as an indicating meter. However, the price of the indicator is the same as the recorder.

Mr. Spillman: Is Tom Martin in the room? I was wondering, Tom, if you would give us some of your experience with the Foxboro meter. I understand you have several installed. We would appreciate any comments on it.

Mr. T. E. Martin: I sound like a Foxboro salesman which I am not, on this particular subject. Six or eight months ago I thought of this Foxboro meter as almost a toy, or something which was at the developmental stage. I am convinced now that it isn't. I hate to sound so enthusiastic about anything as I am about this machine. Phosphoric acid is something that's hard to meter accurately, at least continuously. That's where you can meter it very accurately very easily. Continuous metering of any type of phosphoric acid is very hard because of the fact that when the temperature changes the viscosity changes, and the stuff is viscous enough, not that it doesn't flow, but it's viscous enough so that almost any of the common types of instruments that are used for continuously metering acid have to be set at a different setting to get the flow you want depending on the temperature and depending on the viscosity, and the corrections you have to make for different temperatures are huge. For example, if it's 100° Fahrenheit summer temperatures, you require a setting of, say, 25 on a rotometer, which has been used for phosphoric acid, for a certain flow. At a winter temperature of 20° Fahrenheit you probably have to approximately double the setting. You have to set the rotometer at 50 to get exactly the same flow for furnace acid, that's 75 per cent furnace acid. For wet process acid you may have to multiply by something approaching four instead of doubling. And this would be fine if these factors by which you want to multiply the setting were accurately known but they're not and they vary from rotometer to rotometer. There is a variation in the accuracy of an orifice meter, similarly, and every meter we've looked at has some trouble other than a positive displacement meter. Some of these are being used rather satisfactorily. The trouble with positive displacement meters is that they will wear out if there are any solids in your acid and as is well known, there are tiny amounts, at the very best, of solids in any wet process phosphoric acid.

These meters have been in use, these Foxboro meters, in some of the triple plants in Florida for several years, and I understand from my friends down there that these things are very, very accurate, they're very rugged, they don't seem to require a great deal of maintenance. Now over the last six or eight months increasing number up to five or six that I know of personally of these instruments are in use in mixed fertilizer plants for metering phosphoric acid into continuous ammoniators. With the exception of one instance which I belive is minor, the reception of these—which is a minor defect which I think will be corrected fairly promptly—these instruments have been received with enthusiasm by the people operating them. Now this meter measures volume flow as Mr. Strauss told you, and, of course, we're interested in measuring a certain number of pounds into every ton of fertilizer, a weight flow. So if there is a great deal of variation in the density of the acid, then there will be a variation in accuracy of this instrument. Luckily the density of phosphoric acid varies only very slightly with temperature. The correction is .307 per degree Fahrenheit to a density which is 1.70 at 60° F. for wet process acid. It's a little less for furance acid.

The only reservation I have had in recent months about this Foxboro magnetic flow meter is the possibility that one might go out of order in the height of the fertilizer season and this thing is an electronic instrument full of vacuum tubes and everything. I certainly can't fix one. Mr. Strauss can, I guess, but I imagine not very many other people in this room can. I suppose you have to call a Foxboro man in. Mr. Strauss tells me, under some prodding, that he can pretty well guarantee very fast repairs on these things, and he also tells me that he hasn't had much occasion to demonstrate how fast he can repair these machines because, he says, they don't give much trouble. In installing these things it's important, I think, to keep them in as clean an atmosphere as possible, keep them out of dusty atmospheres if possible. There are two parts to this instrument: one is the transmitter which goes in the pipeline, the other is an instrument that hangs on a panel board or a well, or something. The instruments should certainly be kept out of a dusty atmosphere if at all possible, although it's in a dust-tight case. But you're going to open that case to change charts and for other reasons. The instrument itself, the dynalog instrument should be kept away from excessive vibration which is present in most fertilizer plants. It should be put in a location where there is as little vibration as possible.

As I say, I am very enthusiastic about this instrument. I think it's the way to measure phosphoric acid accurately continuously.

Mr. Spillman: Thank you, Tom, we certainly appreciate those very fine comments. I have one question for Mr. Strauss. Do you carry these meters in stock? How much time between the order date and delivery date?

Mr. Strauss: We would like to carry them in stock, but up to now we haven't been able to get the backlog up to the point where we could. The delivery varies according to the size, but as far as the size for the fertilizer field is concerned, it runs approximately ten to twelve weeks.

I would like to make one further comment on what Tom said about the density changing. We have sold installations, not in the fertilizer field but in other fields, the chemical industry to be specific, using a combination of an Omart cell which is a gamma measuring device for density and the magnetic flow meter, and we multiply the density figure from the Omart cell times the flow and we can actually give a flow rate in pounds per hour, a mass flow in other words.

Mr. Spillman: It looks like, Mr. Strauss, your company better get busy, put some more people on or more machinery or . . . you're going to get some orders.

How Does One Handle Anhydrous Ammonia and Pressure Solutions in Flow-Rate?

Theodore R. Schmalz

A DISCUSSION of the metering of high vapor pressure liquids with rotometers must include a discussion of the methods of pumping and piping used in handling them, since satisfactory metering is dependent upon these liquids reaching the rotometer in the proper condition.

The important aspect to keep in mind is pressure. The working pressure of the liquid must be kept well above the vapor pressure to insure against vapor bubbles forming in the handling system. This working pressure should also be constant since pressure fluctuation will cause flow fluctuation.

There are, of course, several methods of imparting a differential pressure to these liquids. An air compressor can be used in some cases to apply pressure to a storage tank or a tank car. Where air cannot be used because of hazardous air-ammonia mixtures, an ammonia compressor can be used. A small heated ammonia tank for rapid vaporization of ammonia to apply pressure to a larger storage tank or a tank car can be used, and of course a mechanical pump is a standard method of moving liquids.

In any case, the pressure attained must meet the requirements as previously stated, i.e., to be high enough and constant.

In order to stay within the five minute time limit, the balance of this discussion will be devoted to a description of the methods used at our Indianapolis factory for handling ammonia solutions and anhydrous ammonia, where we have had a modicum of success with this phase of our operation.

We have two 20,000 gallon aluminum storage tanks for solution storage. Tank cars are unloaded into these tanks by air pressure. Solution is pumped to the operation from these storage tanks in 2 inch stainless steel pipe by means of a Wilfley Model "AF" chemical pump, of stainless steel construction. In my estimation, this pump is unequaled in maintenance free, dependable operation. Impellor design is such that no packing is necessary and the mechanical seal is designed to seal only when the pump is not operating. We have had this pump operate for nearly a year on a 3 shift a day, 5 day week, with lubrication being the only maintenance.

A recycle line, from the pump discharge to the storage tank from which the liquid is being pumped, is equipped with a smooth acting, spring loaded differential pressure relief valve to insure steady pressure at the rotometer. Vapor locking and possible line or pump fracture is also eliminated by this means since flow of solution is maintained through the pump even though use of the solution is interrupted.

At this point it is worth mentioning that the pump suction piping should be as short as possible and sized to maintain a minimum pressure drop from the storage tank to pump. Vaporization of solution in the suction lines would render ineffective anything else that might be done to obtain good flow.

A Fisher-Porter, metal tube, flowrator is used to meter the solution, and a Fisher Governor, diaphragm operated, control valve controls flow.

A control valve, whether it be manual or automatic, should be placed down-stream from the rotometer, since the pressure drop across this valve is usually sufficient to cause vaporization at this point. After metering, however, this is not a serious detriment to the operation.

In the case of anhydrous ammonia, we have a system supplied by the Applied Engineering Company, of Orangeburg, S. C.

An intermediate pump tank of approximately 1000 gallon capacity is equipped with liquid level float switches. These float switches activate a small capacity ammonia compressor and a solonoid valve located in the liquid line from the tank car to the pump tank. When the pump tank reaches low level the small compressor is turned on and the liquid lines solonoid valve is opened. The compressor takes vapor from the vapor space in the storage tank and discharges to the vapor space in the tank car. The slight differential pressure attained in this way is sufficient to move liquid into the pump tank from the tank car. When the high level is reached in the pump tank the compressor is cut off and the solonoid valve closed.

The pump that is used to move liquid anhydrous ammonia to process is a turbine type manufactured by Corkens, Inc., Oklahoma City, Oklahoma. The same comments as to pump suction lines and recycle lines previously mentioned apply here.

A good differential pressure relief valve with the additional built in feature of ability to devaporize the pump lines is also supplied by Corkens, Inc.

An additional feature in the piping which has proven beneficial is a heat exchanger. The simple arrangement which we employ is constructed of a jacket fabricated from extra-heavy, 8 inch, black iron pipe, approximately 10 feet long. Three passes of 1¼ inch extra-heavy, black iron pipe are made inside this jacket. The heat exchanger is placed in the lines in close proximity to the meter. Liquid anhydrous ammonia comes from the pump, through the 1¼ inch line and then to the meter. A manually operated control valve is located downstream from the meter. The anhydrous ammonia expanding through this regulating valve goes to the jacket of the heat exchanger where it cools the incoming liquid. Again vaporization is not detrimental after metering. A Fischer-Porter metal tube Flowrator is used here also for metering.

As previously stated there are several ways of imparting the energy necessary for handling these liquids but adherence to the general principles outlined are necessary for successful metering.

Joe C. Sharp

I am interpreting the wording of this question as having reference to continuous flow. Both anhydrous ammonia and pressure solutions can be handled in conventional flow meters. They may be either pumped or pressured to the meter.

Solutions are most commonly moved by air pressure from the storage tanks or tank car; air is applied to the tank at a sufficiently high pressure to effect transfer through the system. When using different solutions through the same flow meter, a correction factor must be applied to the meter to compensate for the variations in specific gravity or accuracy will be penalized. This can be done mathematically, or factor tags can be secured for the meter. Since a variation in the specific gravity of anhydrous ammonia occurs with temperature, correction factors should also be applied when extreme accuracy is desired.

Air pressure cannot be used with

anhydrous ammonia because of the hazard involved in obtaining an explosive air-ammonia mixture. Pressure to transfer ammonia is created with heat. When more than that supplied by ambient temperatures is necessary, it may be obtained by the use of a compressor or an auxiliary heater utilizing an electrical heating element or steam coil. Pumping anhydrous can be accomplished when the storage tank has a bottom discharge outlet.

In metering anhydrous ammonia, a simple heat exchanger cooling the upstream side will help prevent bubbling in the meter. The control valve must be downstream from the meter for both anhydrous ammonia and ammoniating solutions, or the drop in pressure through the valve will cause bubbling.

What Are Some of the Proven Scrubber Systems for Controlling Effluents from Getting into the Atmosphere? Can Fumes Be Collected Without Expensive Scrubbers?

G. F. Sachsel

THIS question again cannot be answered in a straight-forward way. I put down here that what this country needs is Socony-Mobile economy run for scrubbers. That's kind of a flippant way of saying that it's difficult to set up standards for scrubbers that will allow a clear-cut choice for each problem. Of course, ideally everybody wants a low investment, a low operating cost, and the best possible performance including—at least sometimes—the recovery of what goes out the stack in a usable form, at least the solids that go out. Such recovery is only occasionally accomplished with scrubbers. There are other dust collecting methods that give you a better chance of getting your solids back out. Some of the general characteristics of scrubbers and the charts showing some fume particle sizes and the performance range of scrubbers and other gas cleaning equipment are shown in Figure 1 which in essence represents data that have been presented in a variety of places including particularly the Handbook on Air Pollution by McCabe and others.

The top half of this chart shows the characteristics of some fumes and the bottom half shows approximate operating ranges of various kinds of collection equipment, including scrubbers and other collectors. The bottom scale, if you can see this, on its lower side has particle size in microns, metric unit, and on the upper side of this same line is a Tyler screen mesh. Now the microns start out on the left hand side at 1,000; right in the middle go to 1.0 micron; and way over on the right hand side goes to .001 of a micron. In that range between .01 and .001, you will see only one item listed and that's diameters of gas molecules over here. This is the point at which you quit. Nobody has found a dust collector that will catch gas molecules as far as I know. The Tyler screen mesh that goes with this is shown on the top section of this bottom line and the first number you can see on the left is a number 35. 35 mesh and its' between 100 and 1,000 microns, a little more than half way to the left of that interval. So take a rough guess, I don't know the exact number in most handbooks, this would be equivalent to, oh, something like six or seven hundred microns there. One standard number that may be easy to remember is 325 mesh. That corresponds to 44 microns. Most sieve analyses go just about that far; that is, you can tell what is 44 microns or less. After that you have to use different ways of finding out what the particle size is. You may notice that industrial aerosols as they're called here —that's a fancy name for fumes— range all over the map. Ground limestone is listed, for instance, in the range of, oh, about 20 or 25 microns to sizes that get up to the 900 micron. You have some minus 325 mesh and you go up in particle size about 35 mesh. In that range, of course, pulverized coal and stoker fly ash and some foundry dusts and cement dusts are listed there. You don't have too much trouble, ordinary gravity settling chambers and the like will bring a lot of your material down. Things get a little more complicated when you get down below 10 microns where you are pretty well in the sub-sieve range. Reading from the top to the bottom, this thing shows sulphuric acid missed, sulphur trioxide missed. which goes over down below one micron, pigments which travel any where from ten on down to a tenth of a micron, and a good bit of the pulverized coal fly ash which goe down below ten microns and th smallest that's listed here is about one Foundry dust can go down to on micron, and a variety of alkali fume

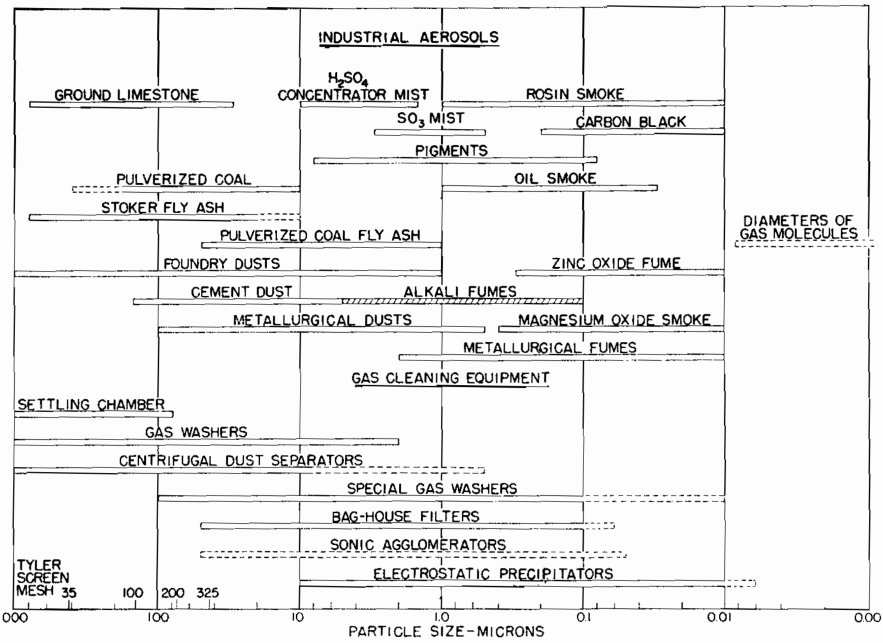

FIGURE 1

may start at about eight microns and go on down all the way to a tenth. Metallurgical dusts similarly will go down beyond one micron. Now metallurgical fumes which are, among other things, such things as metallic oxide particles actually are only partially in this range and go way on down to a hundredth of a micron. Things like zinc oxide fumes and magnesium oxide smoke and carbon black as well as rosin smoke which you find over toward the right hand side there—I read from bottom to top—run all the way down to a hundredth of a micron, and the largest of these particles are listed to be somewhere between a tenth and one micron. It is in this range, I think, that the scrubber finds a good bit of application. When you go to the bottom of this chart you will find that starting from the top, settling chambers are considered to be good until about a little under 100 microns or 200 mesh. Gas washers of various kinds will go on from 1,000 microns, which is in the 20-odd mesh or higher size range, on down into the one to ten micron range where we are in sub-sieve again. Centrifugal dust separators are shown to be pretty good down to ten microns and then you notice a dotted section there which indicates that some special designs will collect smaller particles. The type of thing we have here are the small diameter multiple cyclones, etc. Then we have special gas washers and I'll have a few comments on some of those. They do cover from about the 100 to 200 mesh range and go on down to a tenth of a micron with occasional cases where you can collect fairly well down to a hundredth of a micron. Bag house filters cover a similar range. Sonic agglomerators, which are not particularly economical at the latest writing as far as I know, are indicated approximately covering a smaller range. I think the reason the whole bar is started there is because at the time these charts were made up, which was probably about five or six years ago, there weren't too many performance data available to really evaluate that. Electrostatic precipitators, as you can see, are probably the best thing there is when you get to real small particle sizes. They start at the ten micron subsieve size and good performance is indicated all the way down a hundredth of a micron and maybe a little bit beyond. As a matter of fact, the way this chart reads, it looks like they would catch just a smidgen of gas particles if the gas has a large enough molecular weight. I don't know whether anybody's ever checked for that.

All scrubbers depend on contacting the gas stream that carries the solids with water. They differ in the way they convey the gas stream, what kind of accelerations they give to the gas stream and the particles in it, and the way the water is introduced. Again, I largely relied on the Handbook of Air Pollution by McCabe and similar references to give a really quickie survey on what kinds there are, and I'll just go through this briefly because I think Bob Sibley has some data on this. Water is the most common liquid used. Things like oils and caustics are used at times. Wetting agents may be used at times to improve contacting. They do accentuate frothing, increase in tremen sometimes. As a matter of fact the people who collaborated in air pollution handbook were a little bit skeptical about wetting agents. The simplest way was some kind of a tower, an empty one as a matter of fact, without packing anything into which a spray of liquid while the gas passes simply in opposition to the falling drops of liquid. These things can be used as coolers and primary cleaners for blast furance gas and the like. They are satisfactory for moving coarse dust if high efficiencies are not required. Then there are scrubbers that use the cyclonic motion and the centrifugal acceleration you can give to a particle which tends to fling it out toward the wall and combine this with a water spray going in the same general direction. Probably not the only one, but one example that a number of you are probably familiar with is the Pease-Anthony cyclone spray scrubber. The figures I have here for cost probably have to be revised. I'm using an index, but around 52. For mild steel construction this type of unit ran from about $1.50 per cfm (cubic foot per minute) of gas handled for the 500 cubic foot per minute capacity range down to $.40 per cfm for units larger than 25,000 cfm. Corresponding figures for stainless steel at that time were $2.25 and $.80. You almost doubled your costs there.

Another device is a so-called venturi scrubber — again a Pease-Anthony version as well as other exhibits of that. In this case scrubbing liquid is introduced at low pressure at the throat of a venturi which is a nozzle, a convergent divergent nozzle. If anybody's looked at pictures of rockets recently as you probably have, you see them there all the time. And the gas goes through at pretty good velocities, 200 to 500 feet per second. The Handbook indicated at the time that initial cost for mild steel ranged from about $2.00 per cubic foot per minute in the lower range of around 500 cfm, to $.50 in larger units in the range of 25,000 cfm. You can see the venturi scrubber generally looks a little more expensive than the cyclone scrubber.

There are a number of deflector washers that use deflector plates to capture and also help disperse spray droplets. In general this type in which there is no real atomization is designed for removing particles larger than the one to five micron range.

I might just briefly indicate a couple of experiences that we've had. These have indicated that the Pease-Anthony type or other types of venturis and with some modifications the cyclonic scrubbers will seem to do a good job on acidulation fumes. Ordinary spray towers and impingement scrubbers, where you let the air hit the water surface and run under a baffle or something of that type, seem to be less effective. Joe Reynolds here last year reported a rather special case where they were trying to catch some ammonium chloride out of their Trenton process—pugmill, I believe—and he indicated that a rather simple scrubbing device which really approached a spray tower type of a unit, a closed chamber, seemed to catch the stuff. The one difference here was that they did use very high pressure water. This is something that has been debated a lot. Our air pollution people at Battelle feel that if you have a choice, it is usually cheaper to put a lot of energy into the water than to put it in the air stream; that is, take high water and low gas pressure drop. If water won't wet the fume particles, wetting agents can be tried, but another fluid such as an oil may actually be better. This is a rough one because you immediately face losses of your scrubbing liquid and there's a question of how far you can go with it.

There is a relatively new scrubber that's been described recently and is supposed to be quite effective which relies on a foam chamber principle. It has a downward flow of air into a foam chamber and uses a screen in the air screen probably primarily to get some turbulence in that air stream. There are some hazards connected with it. If the foam should collapse, the pressure drop would be quite significant because you will get quite a volume of liquid. There is also a little bit of question how large the foam chamber has to be to get effective gas liquid contact since the bubbles in this foam tend to be large compared with the droplets of a high pressure butter spray.

Mr. R. Sibley: Mr. Chairman, gentlemen, the preceding speaker has just about taken both phases of my talk, so I'll just have to give excerpts from part of it which might be pertinent. The question has been divided into two parts. First, what are some of the proven scrubber systems to control effluents from getting into the atmosphere? And the second part is, can fumes be collected without expensive scrubbers?

Scrubbers are employed by the fertilizer industry for two main purposes: One, the prevention of atmospheric pollution by effluent dusts, noxious vapors; and, two, recovery of valuable materials from effluent gas streams. In actual practice these purposes are frequently combined. Thus, a scrubber may be used for preventing atmospheric pollution by the recovery of dust which is returned to the process. The most important constituents recovered by scrubbers from fertilizer plant effluent gas streams are: One, fluorine compounds evolved from acidulation of phosphate rock. Two, ammonia from ammoniation and neutralization operations. And, three, product dusts from drying and cooling operations. The most common types of scrubbers used in the fertilizer industry have been discussed briefly—I might name them again and perhaps elaborate on one or two. The centrifugal types have been discussed. Spray scrubbers have been touched upon. These consist of many forms of chambers where gas is brought into contact with a spray liquid. Venturi type scrubbers. The inertial scrubber. The inertial scrubber works on the principle of directing the gas-laden—the dust-laden gas stream toward liquid film in such a manner that the inertia of the dust particle is carried into the liquid, whereas the gas stream is diverted in another direction. This impingement principle is employed in a number of ways. For example, by placing baffles inside of chambers and applying a continuous wash over the baffles, or by passing gasses up through a flooded perforated sieve-type tower tray having impingement plates over the openings, or by forcing gases through a flow constriction downward at high velocities into a water reservoir. Pack towers. Pack towers are designed to spread the collecting liquid over a large surface area and to impede the flow of gas as little as possible. The principal application is in removal of soluble vapors rather than the removal of solid particles which tend to flood this device.

The second part of the question: can fumes be collected without expensive scrubbers? The general answer to this general question is, no. Specifically, it has been mentioned that scrubber costs vary from 25c to up over $1.50 per cfm. To those of us in business in the Los Angeles basin, having to conform with strict air pollution regulation laws, spending the money to install a scrubber may mean whether you are going to continue in business or not. This condition is going to become more common across the nation. Actually the installation of a scrubber in many ways can pay for itself. In pay-off periods for your initial capital investment may be as short as several months. Normally it runs from one to three years. Attractive savings can be had for the increase in yield, recovery of raw materials, by-products, or finished materials.

DISCUSSION

Mr. Sachsel: This is in the nature of a warning which our air pollution people at Battelle give everybody from bitter experience. It's quite a job to find out what the particle size of your effluent is; that is, how big or small the stuff is because the business of sampling it and having in your sample a particle size range that you can measure that actually represents what's in that gas stream is quite tricky because many sampling methods cause some agglomeration to take place before you get a chance to look at the sample and you can fool yourself in thinking you've got nothing below ten microns in your dust and therefore a certain collector will work like a charm only to find out when you put it in that you've still got 20 per cent of the stuff coming out and the only conclusion you can come to is that you've really got minus ten micron stuff in there and you just didn't catch it when you sampled it or you didn't recognize it. So, and this is the hard part because it's not a very straightforward or cheap proposition to get a really reliable measurement on what you've got in your gas steam. Things like cascade impactors which are becoming increasingly available. I think there are some commercial models out. Probably one of the relatively speaking, simpler ways of getting a reasonably good particle size distribution, you've still got to watch your gas stream with moistures in it, you've got to prevent its condensation, particularly if your scrubber is not designed to condense it in the right places because it may fool you about particle size and it may cause agglomeration even in a impactor type unit where you actually separate particle size fractions in various compartments and therefore avoid the tricky business of getting the stuff onto a slide, for instance, which would be the microscopic way, and continuing and thereby trying to determine what your particle size range is. That is, make sure you know that because if you don't you might be very unpleasantly surprised.

Mr. Spillman: The next question is what experience has anyone with a dust collector for hygroscopic materials? With bags plugging up with dampish dusts? This question will be answered by Mr. Cizek of Eastern States Cooperative.

Mr. Rodger Smith: We are very sorry that it hasn't been possible for Lou Cizek, Manager of the Cambridge plant, to attend this meeting. He didn't consider it possible for him to attend for the reason that the particular unit to be described was delayed in its installation and only came into production last week Thursday and he is very much concerned with getting the new equipment into ful operation and this week getting second shift organized at the plant Since we have virtually no experienc with the unit, I'll only describe i rather briefly.

The particular unit comes unde the classification just shown on th slide by the representative of Battelle of bag house filters. Very simply the installation consists of the air from the dryer—that's the one the question refers to here—from the dryer being exhausted and exhausted through a settling chamber and an enclosure consisting of two parts or two sides, each side of which contains 84 orlon bags in a vertical position six and a half inches in diameter, 80 inches long. And the air flow is downward through these orlon tubes and is exhausted through the walls of the cloth tubes. It is in two parts in order that according to a time cycle, the air can be directed from one side and the bags on that side go through a shaking cycle to remove the dust which has collected there. According to the data on this slide which you've just seen, this type of mechanism should collect the dust in the range of about 60 microns down to a tenth of a micron. It is for the purpose of collecting dust and not fumes. This whole unit of the settling chamber plus the housing for the orlon bags is contained in a heated room, heated with a furance for the purpose of maintaining the temperature, the X drier air above the dew point until after it has passed through the cloth bags and out of the whole unit and exhausted to the stack. Obviously that is for the purpose of avoiding the moisture in the air getting down to the dew point until after it passes on beyond this unit where, of course, it could cause very serious and rapid plug up of the whole mechanism. We're exhausting through the unit above 12,000 cubic feet of air. Although we have virtually no experience with it, I'm sorry to say, we have every confidence that it will work.

Let me comment just further on this whole problem. I think what I have to say here is entirely in agreement with what the two gentlemen who have just talked much more extensively on this subject had to say. We regard installation of equipment of this sort as something which you do when there is public pressure or for other reasons you consider it necessary to do a very complete job of removal of dust. If you do not have that situation where you are possibly confronted with a shutdown, I believe it is much more practical to attempt to avoid the problem up to some reasonable degree by applying just basic principles, many of which or most of which have been described in the last couple of days here. In other words, if we can use ammoniators, speaking in terms of continuous ammoniators, which are of sufficient size and particularly sufficient diameter to give depth of bed and they are sufficient size in the pipes, as far as your pipes are constructed, so that there is good distribution of the acid in the solution, we're going to reduce a great deal of the fumes and particularly the more undesirable nitrous oxide fumes at that point. A large part of the problem can be avoided right there. And then an equally important factor is the proper relationship between the size and particularly the diameter of dryers and coolers in relation to the amount of air which is found necessary to accomplish drying or cooling or removal of moisture and heat from the dryers and coolers. I think there is a lot of work which indicates and it goes way back to that first work done by Dr. Keenen of Du Pont, if we can have dryers and coolers of sufficient diameter so that the velocity of air in a dryer or cooler does not exceed approximately 300 feet per minute, the pick-up of material is not going to be excessive and we're not going to be confronted with the serious problem of dust particles being exhausted out into the atmosphere. That is entirely within reason to meet that situation. For example, with a six-foot diameter shell, you can stay within 300 feet per minute and handle about 8500 cubic feet of air. And if it's an 8-foot diameter shell, it is possible to go up to about 15,000 cubic feet of air and still be in the range of 300 feet per minute. Taking those basic precautions of adequate size of continuous ammoniator and sufficient depth and close supervision of the pipes and installing dryers and coolers, in most plants and many locations you can avoid much more costly installations of some of this filtering or other equipment. I think we can afford to make quite an expenditure for perhaps new installation of driers and coolers in order to avoid the problem at the beginning rather than trying to correct it after we have cleared the problem and, of course, we will at the same time be doing a better job of drying and cooling at the same time.

Mr. Spillman: Mr. Sibley, would you be kind enough to give us the commercial names of some of the proven scrubbers.

Mr. Sibley: This can be a little ticklish because I'm bound to leave some of them out. I hope I don't offend anybody. It will be strictly unintentional and due to ignorance. The Doyle scrubber has been mentioned for the Dorr-Oliver process. Centrifugal types. The Pease-Anthony or P-A cyclonic scrubbers are mentioned made by Chemico. Whiting Company make the Hydroclone or Type S. There are a number of scrubbers that can be called the multi-wash type scrubber offered by Ducon, Schneibly, and Pangborn. These have seen actual service and appear to be quite satisfactory. Chudicurting make a line of educators and equipment for spray chambers. The venturi type scrubber, Pease-Anthony here again make a venturi scrubber. Johnson-Marsh make a type of venturi. The inertial scrubbers include one made by the Joy Manufacturing Company just recently come out called the Joy Microdine which has just applied in the fertilizer industry with unknown results at the present time to me. The American Air Filter puts out the Rotoclone Type N which is a fine inertial scrubber. Also, the National Dust Company have a hygrofilter. Mechanical type has been missed. This involves mechanically driven rotor wheel which can be combined in a fan for impinging particles on water sprayed bleeds. The American Air Filter Rotoclone Type W is of this type and it's quite popular. Ducon and Schneibly are incorporating this principle in their spray fans and are combining them with their centrifugal type scrubbers.

Mr. Spillman: Thank you, Mr. Sibley. Are there any other questions? We're sure sure to have some questions on this because it's a very important thing. A good many of us would be granulating today at certain of our plants but until we are positively sure that we can build a plant and not have to shut down, we just can't go ahead. As far as I know now, I don't think we have a scrubber that is doing the job 100 per cent as some people want it to do. Any further questions?

Advances in the Liquid Fertilizer Field and Davison's Liquid Fertilizer Plant at Wakarusa, Indiana

J. E. Reynolds, Jr.

BEFORE the status of liquid fertilizer projects can be determined, a review of the achievements of the past three (3) years should be made. The liquid fertilizer operations have advanced from insufficiently tried principles based on stereotyped techniques to proven methods based on experience and a better understanding of the chemicals being mixed. Several improved systems for manufacturing liquid fertilizers are available using batch or continuous methods. Facilities for cooling the liquid mixed fertilizers are most often incorporated in the design of a plant. Instrumentation has also entered the demands of the liquid fertilizer producer.

Simultaneously with the advance for more effective means to produce liquid fertilizers, achievements were being made in the methods for storing and distributing this product. The liquid fertilizer plant has been able to increase its sales volume and territory beyond the normal 30-mile radius through the installation of liquid bulk tank storage in the fringe area. Arrangements for custom application and convenient terms for leasing transport and application equipment encourages the use of liquid fertilizer without requesting the dealer to purchase the equipment.

Liquid fertilizer formulation practices have become more flexible through the use of combinations of anhydrous ammonia or aqua ammonia, urea-ammonia solutions, and urea-ammonium nitrate solutions. Research to permit the use of wet process phosphoric acid and higher strengths of phosphoric acid is continuing. Some manufacturers are using a partially purified wet process phosphoric acid to achieve formula savings. The entry of superphosphoric acid into the liquid fertilizer field will enable the production of higher analyses fertilizers which can more nearly compete in total plant food content with solid mixed fertilizers.

Of specific interest to Davison has been its liquid fertilizer plant at Wakarusa, Indiana. This plant was constructed the Spring of 1956 primarily for market research into the potentials of liquid fertilizers and the methods of distribution. (1) The Wakarusa plant provides for the production and storage of aqua ammonia and neutral complete liquid fertilizers, as well as storage for bagged and bulk dry fertilizers. The main building houses the dual reactors and accessories for producing neutral complete liquid fertilizers, and storage for the dry raw materials and bagged dry fertilizer

(2) Dry granulated fertilizer is stored in thirty (30) ton capacity bulk hoppers which are filled by a conveyor through the top, and emptied through the bottom. A small portable conveyor elevates the granulated fertilizer from the hoppers into bulk trucks. All liquid storage tanks and the converter-cooler are located outside the building.

(3) Aqua ammonia is prepared by combining anhydrous ammonia and water in a "converter." The aqua ammonia is continuously pumped to outside storage tanks for later sale or mixing, or to applicating and transport tanks for direct application. The converter is designed to mix and water-cool aqua ammonia, and to be used interchangeably for cooling neutral liquid fertilizers.

(4) Neutral complete liquid fertilizers are formulated using aqua ammonia, phosphoric acid, urea, and muriate of potash. Grades such as 5-10-10, 8-8-8, 8-24-0, and 14-7-7 are produced at the rate of 15 tons per hour. The actual mixing of these grades is accomplished by reacting balanced proportions of aqua ammonia and phosphoric acid under noncorrosive conditions controlled by a pH instrument. The liquid fertilizer being formulated is continuously cooled by pumping the liquid through the converter.

The sales area serviced by the Wakarusa plant is confined to a radius of 50 miles with most of the sales in the 30-mile zone.

(5) Liquid fertilizer transport and applicating equipment is leased to the dealer to encourage the use of liquid fertilizer without requesting them to purchase the equipment. The equipment available for leasing includes distribution tanks for mounting on trucks, nurse tanks mounted on trailer frames, and application equipment complete with booms and injector facilities.

The usage of liquid fertilizer should increase during the next three or four years for the following reasons:

1. The increasing number of liquid fertilizer plants make for convenient delivery and service. Localized sales effort in our trade area accentuates the interest in promoting liquid fertilizer.

2. Liquid fertilizer is easily handled as pumps substitute for hand labor.

Of course, there are problems which must be corrected to permit further expansion. Foremost in these problems is the need for overcoming the limitations of producing liquid fertilizer grades which contain fewer units of plant food than is possible in granulated dry fertilizer. This limitation is caused by the crystallization of higher analyses liquid fertilizers. Habits and traditions of using dry fertilizers also influence the farmers' consideration in accepting this new product.

DISCUSSION

Mr. Slack: What are the major technical and economic features of liquid fertilizers vs. granulated of the same analysis? At least thaat's the version of the question I have here. Well, this has been covered in publications and I'm sure most of you are familiar with these major factors. The primary one, the reason so many people are in the field, of course, is that investment is much lower. I think most of the liquid plants are probably run under $50,000 or not much more than that. I just wanted to cover the granulation plant cost, to make 20 tons an hour is a sort of a moot question. People have different ideas as to what a granulation plant will cost, but most will agree it is considerably more than that. The main economic drawback to the liquid mix, of course, is that you can't use ordinary superphosphate. Having to use phosphoric acid as the source of the P_2O_5 is expensive, but the manufacturing cost is lower, just how much lower is another question difficult to evaluate. I've seen cost estimates showing the overall cost lower, and

I've seen others that showed it to be higher, that is comparing the liquids to the solid and granulated type. So you can pick whatever answer you like best. However, there is the practical matter, the selling price of the two types seems to be about the same, as near as I have been able to obtain information on it. Two of the factors difficult to evaluate as Mr. Reynolds covered—at least he covered one of them—is convenience of use, the actual saving probably and application on the farm once you are equipped to distribute the liquid. And there is the complete water solubility for the liquid fertilizer which is another moot question in the industry. Finally, the last question is what is the present status of the complete liquid fertilizer industry and is there a definite trend toward increasing production and consumption? I'm sure the question that they had in mind was the usual one as to what is the future of liquid fertilizers and are they going to replace solids and all that. Well, each of us has his own crystal ball and I guess one is as good as another. It would be nice for someone to tell us just what is the present status of the new trend in the field, but I can't do it and I don't think anyone else can. All we can do is consider what the growth has been and decide what future trend it might indicate. In 1955 the number of plants as best could be estimated was 72. The recent count we made as of 1957 was 165. So we have doubled the number of plants in two years. Of course, those of you who know this industry, sometimes it is difficult to decide whether to call an installation a plant or not, so those are pretty rough figures. Production from these plants, I don't have any for 1955; for 1953 and 1954, the Department of Agriculture estimated 27,000 tons which isn't very much. The present estimate for this year is on the order of 250,000 tons. Again, that's more of a guess than anything else. If it is approaching an accuracy, however, that is a ten-fold increase in three or four years. So percentagewise that is impressive. However,—I mean, percentage of growth—but percentage of production of liquid as compared to solids it is still very small, not more than a drop in the bucket compared to total production in this country.

Dr. Sauchelli: Do you have any idea where they are concentrated?

Mr. Slack: In the Middle West and the West Coast, of course, where the industry started.

Dr. Sauchelli: Do you have any questions you want to ask Mr. Slack?

Mr. Gordon: Mr. Slack, in the use of metallic ions as crystallizing point depressant, are they in the form of dissolved metallic salts or in the form of dispersed colloidal suspension?

Mr. Slack: They are dissolved. We used the salts but we didn't use enough to exceed the solubility. I imagine there are a minimum, of course, present in most fertilizer materials and this is not the phosphate rock directly. We found if you take wet acid and ammoniate it and filter off the precipitate, the resulting ammonium phosphate solution is much more difficult to crystallize than if you started with furnace acid. That's familiar, I am sure, to most people here.

Question: That .1 of one per cent chromium was the metal but in the form of sulphate?

Mr. Slack: This was the oxide, I believe.

Question: Chromium oxide .1 of one per cent in the liquid fertilizer.

Mr. Slack: Yes.

What Suitable Drepressants Are Known That Could Be Used to Lower the Freezing or Salting-Out Point of Liquid Fertilizers?

A. V. Slack

THE problem of depressing the salting-out temperature of liquid fertilizers can be approached from either the thermodynamic or kinetic standpoint. For the former, an additive is used which alters the solubility relationships in the system involved and produces a permanent depression in salting-out temperature. Usually a relatively large amount of the additive is used. For example, if about half the orthophosphoric acid normally used is replaced with pyrophosphoric acid the crystallization temperature is lowered considerably.

For the kinetic approach, dependence is placed on delaying crystallization rather than permanently preventing it. In some cases, however, the delay may be so long as to be considered permanent for practical purposes. The mechanism involved is either a thickening of the solution to slow down diffusion of ions to crystal surfaces or a blocking of crystal surfaces to retard crystal growth.

At TVA we have tested various additives such as hydrophilic colloids (agar, dextrin, cornstarch, etc.), surfactants, dyes, and metallic ions. All of these have been reported to retard crystal growth under some circumstances. The metallic ions were found to be the most effective, with chromium, iron, and aluminum the best of those tested. Small amounts of these caused a delay in crystallization for several days in solutions stored at temperatures as much as 27° F. below their normal crystallization temperature.

Two drawbacks to use of such additives were found in TVA work. One is that although fair results were obtained with solutions from which ammonium phosphate crystallizes, much poorer results were obtained when a potash salt was the crystallizing phase. The other is that dependability of the effect was poor, i.e., duplicate samples gave widely varying results as to the length of time that crystallization was delayed.

A depressant was placed on the market some months ago. The producer claims that it will reduce the salting-out temperature of 10-10-10 to 0° F., which is a depression of 50° to 60° F. We have no information on its composition or effectiveness.

(2) What are the major technical and economic features of liquid fertilizers versus granulated of the same analyses?

One of the major economic factors in comparing liquid mixed fertilizers with the granulated solid type is the investment required to get into business. A complete liquid mixed fertilizer plant can be built for less than $50,000, while a granulation plant of the same capacity probably would cost $250,000 or more.

The main economic drawback to

liquid mixes is that ordinary superphosphate cannot be used. Producers of granular fertilizer can be used relatively large amounts of this relatively low priced phosphate sources and thereby gain in advantage in raw material cost. However, manufacturing cost is lower for the liquids because of lower manpower and maintenance requirements. Depreciation charges are also lower and a lower markup per unit of production is necessary to obtain a reasonable return on investment. These two factors—raw material cost and manufacturing cost—appear to about cancel each other out, so that selling price for liquids is usually close to that for solids.

Two of the factors involved in the comparison are difficult to evaluate. The phosphate in liquid fertilizers is completely water soluble whereas in most granulated fertilizers it is not. The higher solubility for the liquid may be an advantage in some situations but in others it is not particularly advantageous. The liquid mixes are also more convenient to use and require less labor for application on the farm. Neither of these advantages is very amenable to cost evaluation.

(3) What is the present status of the complete liquid fertilizer industry, and is there a definite trend toward increasing production and consumption?

There are at present approximately 165 plants making liquid mixed fertilizer in this country. Current production is estimated roughly at 250,000 tons per year. In comparison total production of mixed fertilizers over the country is about 15 million tons. Thus the liquid mixes have not as yet attained a place of any significance. However, continuing growth is evident. There were only 72 plants in 1955 as compared to 165 now and new ones are reported frequently. Production in 1953-54 was reported to be about 27,000 tons as compared to the current estimated figure of 250,000 tons.

Advantages and Disadvantages of TVA Cone Mixer.

Allen S. Jackson

WHEN any cone mixer is installed, whether it is put on an existing den or on a completely new den, the normal problems of rock handling and measuring and liquid control will appear. These usually present very small problems that are easily solved.

Early cone designs would allow only a very narrow rate of operating range, usually in the neighborhood of from flooding rate to a rate of feed of 5% less than flooding. Any reduction of rate beyond that would not give proper mixing. Modern cones, however, have good mixing with operating ranges from flooding to approximately 50% of flooding.

The use of strong acids present a problem in operating the cone. Heat of dilution raises the temperature high enough in the cone to cause large quantities of steam to be evolved. While this steam could certainly be controlled with proper fume hoods, the operators visibility in the cone is greatly reduced. Uniform results are not as easy to obtain with 93% or 98% acid as it is with 78% acid.

The life of the cone liner is quite variable. We normally expect a liner made of 10 gauge mild steel to last between 20,000 to 40,000 tons when used on a box or continuous den. On Sturtevant type dens, we expect the mild steel liner to last 5,000 to 10,000 tons. Starting and stopping the cone is much more damaging to it than operating. In order to improve the start-stop life of a cone liner, we are experimenting with liners that have been Teflon lined, lead lined, coated with various Epoxy compounds, liners that are made entirely with reinforced plastics and various rubber coatings for steel cones. We are hoping to find a cone coating that will allow us to reach a 100,000 ton life for the liner under all conditions.

When a given quantity of acid, water and phosphate rock are mixed together, no matter what means of mixing are used substantially, the same end product as far as analysis goes, will result. The cone gives some advantages over pan type mixing in that mixing techniques will always be the same. The time going through the cone is always the same. In cones all of the gasses are involved from the resulting mix in the den, none in the cone. The gasses percolating through the hardening bed of super tends to make it a very light, honey-combed mass. Because of the short retention time in the cone, the mix going to the den is very fluid. Dens must be kept tight to contain the super. Apparently because of lack of stirring. the mix gives a feature to the finished super that is both good and bad. Super made in cones tends to have a very fine particle structure. Each individual particle of rock appears to retain its own identity. The resulting super because of its small particle size, is very easy to ammoniate. The small particle size, however, can make the super rather dusty to handle, which can be a nuisance in handling of it in both the mill, and if not granulated, in the field.

The main advantage of a cone super mill is that the mixing machinery is easier to maintain and has a much lower initial cost than any other conventional means of making super. Normal replacement of worn parts of the mixing unit, weighing and feed equipment, is estimated to be about 1¢ to 2¢ per ton.

F. T. Nielsson

1. *Advantages.* To realize fully the advantages of a cone mixer, the cone should be engineered by someone who has a record of experience with cone mixers, like the FESCO group of Atlanta. The cone mixer has the following advantages in comparison with any other type of mixer:
   - (a) Less initial equipment investment.
   - (b) A lower investment for structural supports and installation.
   - (c) No operating power for mixing.
   - (d) Exceptionally low maintenance.
   - (e) No moving parts.
   - (f) Small space requirements.

   In common with other types of continuous mixers, the cone has the following advantages over a batch mixer:
   - (g) Less personnel required for operation.

(h) Fume recovery is better using the exhaust system designed for a given tonnage batch system.

(i) Acid can be diluted over a substantial range within the mixer. However, 66° Bé acid should not be diluted within the mixer as some operators have found out to their sorrow.

2. *Drawbacks*. Some of the drawbacks are those associated with any continuous mixer. These are:

(a) Feeder calibrations must be made accurately and checked periodically.

(b) The entire ground rock system must be engineered to prevent flooding.

(c) Some spent acids may be difficult to meter properly on a continuous basis.

Some drawbacks are inherent with the cone, and the seriousness of these drawbacks depends upon the type of den used with the mixer.

(d) The cone is not amenable to provide variation in rock-acid proportions.

(e) The cone requires a fluid discharge and mixing is hindered as net acid concentration is increased.

(f) Maintenance is a function of stop and start operation because of acid drip from the nozzles. Less maintenance per ton is required when the cone is used with box or continuous dens than when it is used with Sturtevant dens.

(g) The cone is so simple that apparently anyone should be able to build his own cone. However, there are certain nozzle sizes and cone dimensions that must be used over a given range of tonnage if good results are to be obtained. The ability of amateur to build their own cones is a drawback if their unsatisfactory results cast doubt as to the efficiency of the cone mixer in general.

I'D like to just summarize. We've tried to indicate the advantages and disadvantages of a TVA cone mixer by comparing it as a piece of continuous equipment against other continuous equipment and also by comparing it as a piece of continuous equipment against batch equipment so that when you compare the cone mixer in comparison with any other type of super-mixer, it appears that it has less initial equipment investment because the thing isn't very heavy, you have less equipment for structural supports and installation. You've got the pump acid to assist them anyway, this cone mixer has no moving parts so you have no operating power. You have exceptionally low maintenance. You've got no moving parts, you don't have much space. And actually those of you who have been to TVA and have seen a cone mixer—I guess it's pretty old hat now, but I know years ago when people used to come down there when I used to work there it was the biggest surprise to take a man up on the floor and show him this little cone, on triple especially, and the stuff went in and the stuff went out and the man just stood there and it was just like a tuba; I mean, it came in here and it went out there and people couldn't believe it until they saw it. So it is simple and on triple not too much trouble. With super, now, the big thing I do believe, though, is that if you are interested in the cone mixer, although I'm mentioning the advantages and disadvantages here, that you ought to get one from somebody who has built one. Allen Jackson's outfit has put in a lot of cone mixers and they know what they're doing. A lot of other people have tried to put in cone mixers and you can get hurt awfully quickly.

In common with other continuous mixers, I mean any continuous mixer of which the cone is an example, has some advantages over a batch mixer and these are that there is less personnel required for operation, the fume recovery is going to be better. If you have a fume recovery system for a 20 ton an hour for a batch operation where you're working alternately, you know that you've got to handle about 40-ton per hour peak whereas if it's a 20-ton per hour system, you're just handling 20 tons per hour.

In any continuous mixer you can dilute acid over a fairly wide range. The only thing that you can't do is you cannot dilute 66° acid into a cone directly. Some people have tried it and two things happen. First, the heat of reaction or heat of solution, is so great that you get a pretty violent reaction when you're pouring in water through some nozzles and 66° acid through other nozzles. The other thing is that due to the heat formed and also probably due to the fact that 66° acid isn't diluted all the way before it mixes with the rock you get a superphosphate that has practically no conversion.

Now, the drawbacks of the cone mixer. Some of the drawbacks are the kind that you have with any continuous operation. One of them, of course, is that you've got to calibrate your feeder and if you're in continuous operation with ammoniators you know you have continuous feeder trouble. You're apt to get into the same kind of trouble with any type of continuous system. The other thing is that a continuous rock feeding system is something that has to be really engineered, and again some of this is old hat to a lot of people, but until you've seen rock dust flood into a continuous operation, you just don't know what trouble is, because if it starts flooding you might as well go home and call somebody else and start all over again and start engineering from the beginning. And then also some spent acids may be quite difficult to meter properly on a continuous basis. Now those are troubles that any continuous system will have. The cone mixer has some inherent disadvantages of its own and the seriousness of the disadvantage depends on what kind of bin you're using. The cone initially was designed for triple super. With sulphuric acid you're neutralizing two hydrogen ions. With phosphoric acid you're neutralizing only one hydrogen ion. Therefore you use twice as much phosphoric acid on a pound basis as you do sulphuric acid. Therefore you have twice as much liquid when you make triple as you do when you make super, something people don't realize, and because you have twice as much liquid you have a much more fluid mix and this cone mixer having no moving parts in it, just depending on spin of a fluid, if it's fairly fluid you get a lot of spin. Now as you go to sulphuric acid and your fluidity decreases, it stops, slows down, and you're in trouble. I mean, you just can't mix mud by itself, you just can't spin enough. So that's the big trouble with the cone mixer, that if you think that some day you're going to be in an operation where you're going to try the effect of 60 Baumé acid all by itself, you had better take it easy before you start thinking about that in connection with a cone mixer because you can take any rock grind and any acid concentration and you can blend it physically, mechanically, but here you're depending upon fluidity and we do know that as you cut down the concentration of acid you're going to decrease the fluidity and when you do that you're going to be in trouble in a cone mixer. Also, because you have to have a fluid concentration, you can't run quite the acid concentration that you can in a batch mixer. Then also there's maintenance, you get acid drip from the nozzle, so the more times you stop and start this acid drip eats on your cone a little bit. If you have a bin that takes only eight hours to fill you're stopping and starting once in eight hours. If you have a Sturtevant bin and filling it every hour or so, why, you have more acid dripping and you have a little more maintenance.

The big trouble with the cone mixer—this is just a personal observation—is that it's so simple that apparently anybody ought to be able to build his own mixer. The trouble is that people go ahead and build their own mixer and don't realize that there is a little bit of theory involved in it. Not too much, but a little bit, and they build the mixer and it doesn't work and they think it is no good. That's why I said back in the beginning you ought to talk to somebody who has put in some mixers because I know an actual example of how that can occur. I used to be down in Alabama and some big consulting company came down and wanted to know all about the TVA cone mixer. So I took them out and showed them and explained a little bit of theory and the next thing I knew I was hired by a private company and I happened to be in a triple plant that this consultant had built. And I was put on a train and told to go down to Florida and see what was wrong with this triple operation since it wouldn't work. The cone mixer wouldn't mix. And I looked at the blueprints on the train and I couldn't figure out anything wrong, and I got down there and the big trouble was that this man had seen a 40-ton an hour cone, took the whole thing down there and put it in an operation which was running at 20 tons an hour. The important thing he didn't realize is that the acid has to wet the inside of the cone. If the acid dribbles and the rock hits the cone you don't get a mixture. You have to have an acid swirl, the rock falls in the middle of the swirl, comes out, and you have a mixing section there. At TVA they had ⅝ inch nozzles; he put in ⅝ inch nozzles. All I had to do was to tell him to put in ⅜ inch nozzles and it worked. As I say, there are some simple things like that. It looks so simple that you think, anybody can do it. But before you go ahead think about it. I've heard of some other people who tried to copy cones. Oh, there is no need of our getting in an engineer designer for this, they say, there is nothing to this. Well, it's a little bit more complicated than that, and you've got to know what you're doing.

A. S. Jackson: I would like to show you one of the design changes that we made on a cone to increase the range of operation. When we first started using cone mixers three years ago we found that we could operate from the rate that the cone would begin flooding to somewhere between three and ten per cent less than the flooding rate. After that the rock dust began to show up in the bin, mixing was very incomplete and completely inadequate. When we started working with continuous bins, people particularly with these lightweight bins would let their cutters wear out; they then must slow down the operation of the bin to continue to run, they begin to have trouble then with high insoluble, high free acid—just really inadequate mixing. If the velocity of the liquids or the swirl of the cone mixer were proportional to the distance away from the center of the cone, the shape of the swirl would be a parabola or something like that. If there were nothing to slow down the swirl the shape of the swirl would be a hyperbola. Actually the shape of the swirl or the mix in the cone is somewhere in between. The center of the cone discharge is never filled by the solid liquids. It may be filled by a little foaming right in the surface. Well, as you slow down the rate of running on this type of cone, you're dropping the level of your swirl, you open up a larger hole to pass the rock through. The change that we made was rather obvious and extremely simple. We set the footed elbow down so as to make the rock or the liquid take the curve. It caught all of the rock coming through the cone and gave it one more twist. The ranges of the cone now is wider, if you make one for 40 tons per hour, it will operate very effectively down to 20 tons per hour. When we were using straight bottoms, if we were operating at 40 tons per hour, it might operate as low as 38 or 37 but not lower. As Frank Nielsson said, when you are running continuously a mild steel cone will fool you on the length of time that it will last. The original cones we made were made of 16-gauge black iron, purely for trial. After the shape was determined we were going to stainless or Hastelloy or some other material. We had comments from the very best people, people familiar with phosphate much more so than we were, that this cone would last anywhere from thirty minutes to two or three days. Well, it ran about 6,000 tons. So the next cones we made we stayed with mild steel about ten-gauge. We got life on those cones of up to 40,000 and 50,000 tons on a mild steel ten-gauge cone. Now this is using 60° acid. It will not last that long with strong acid. When we started putting cones on existing bins such as the Sturtevant we ran into trouble with the dripping of the acid as Frank told you, and the deterioration of the cones between batches. We found that the cone that should last 30,000 or 40,000 tons now would last five thousand tons. Well, that ceases to be very economical because the cone liner of mild steel costs $70 or $80. We started experimenting first with various paints, various coatings. We started out I believe it was a polyvinyl paint. That lasted maybe thirty minutes. We tried sprayed teflon. We tried the teflon films that were bonded to steel. The teflon films would last fine but we couldn't keep it bonded, and for this bonding we had a professional coating company do it. We did not try to do it ourselves. We have tried all plastic cones. They are reinforced with glass fiber and various types of plastic. They are in between —they do better than steel on Sturtevant bins. They do not do as well as mild steel on continuous bins. We have a cone now in use that is mild steel that is coated with an epoxiresin. It is on a Sturtevant bin. We expect the results of that one to be somewhat between a little better than mild steel would normally last and not as good as mild steel would last on a continuous bin. There are lead cones being made. One of our customers has made the prettiest lead cone I have ever seen. I expect the lead cone will give the best result. I do not know; it is just being tried. We are still experimenting with other compounds. Some of the rubber companies are coating them for us with various rubbers, and we don't know yet. I would like to speculate just a second on the use of cones on continuous bins. I have seen several bins that were normal pugmill bins or pan mixer bins that have been converted to cones (makes diagram on blackboard). Assuming that is a normal Broadfield type of bin in which the pugmill generally discharges very close to the rear. We feel—we have not tried it, now, but we feel that the cone mixer should be placed nearly half-way up the bin, so that it will form its own pile running to the rear on the surface and then coming forward in the pile. The idea of this arrangement is simply to delay this very, very fluid mix as it hits the bed with the cones right on the back with the pile being formed abruptly. These seals must be kept very tight to make it really satisfactory. We believe this will relieve it a little bit. But then we introduce one more problem. How do you start the thing. The wet super is extremely liquid as it is and will just come right off the end. In the second step we propose to incline the bin just a little bit to help start it. It will not help running, it will just help start it. Bins even with the cone on the back now are often started by building a dam of cubic super in it. But it's a lot of trouble and we would like to avoid it.

Mr. Spillman: I would like to make one comment about the lead cone. We have one. We've used it about six weeks. We took it out a week ago. This was on a Sturtevant bin, therefore, intermittent operation.

Dr. Sauchelli: What was the material?

Mr. Spillman: Lead.

Mr. Jackson: Hard lead or soft lead?

Mr. Spillman: Hard lead.

Mr. Jackson: We have had several people's suggestions in talking about lead goods. They suggested soft lead with a minimum of animo will be the best lead. On Sturtevant type bins, I'm sure that's true. If it is a very high steady production unit then erosion may become a factor, the lead may be too soft, but I don't know. It probably would have to be varied with particular lead.

Question: On this cone that you put on your Broadfield bin—in other words you just put the elbow to the back and set the cone alongside of that curve from the back?

Mr. Jackson: The direction of the elbow, as far as the ejection of the liquid is concerned, makes no difference. It comes right straight back. We've looked at it inside the bin for 85 seconds and we can see it. But how far down I don't know. A long bin up to a third of the way; a short bin maybe half the way. But I feel that it should be inclined to facilitate the starting, even though it is an existing bin, you can still build a dam. They are awfully hard things to keep from leaking if you don't do something like that.

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Novemer 6-7-8, 1957

PROCEEDINGS

OF THE

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1958

Held at the
MAYFLOWER HOTEL
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