

Figure 1. Approximate locations of granulation plants

Plant Practice in Granulation

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Granular fertilizer tonnage has tripled in the past two years, necessitating radical changes in technology for the mixed fertilizer industry. The authors contacted 135 plants to find out how the change-over was accomplished

GRANULATION is the major current development in the mixed fertilizer industry. It is probably the most far-reaching in effect of any of the innovations which have come into the industry since its beginning in this country. Ammoniation of superphosphate was also a major development, but it involved only the use of another raw material in existing equipment, with little change in operation. Granulation requires new equipment, new operating skills, and a basically different type of operation.

The trend toward granulation is due to several factors. From the producer's viewpoint, granulation reduces operating cost by allowing use of more low-cost nitrogen solution without troubles from physical condition. Moreover, use of nitrogen solution gives a higher analysis product, thereby reducing handling and shipping cost per unit of plant food. From the consumer's viewpoint, granular

material is less dusty, less apt to cake, easier to handle, and can be applied with more uniformity. These factors have led to very rapid growth in production of granular mixtures in the past six years.

In the initial phases of any major change in an industry, wide variation in practice is normal. In time, however, a standardized practice usually develops. Granulation of mixed fertilizers is still in the first phase; there is wide variation in type of plant, operating technique, and type of product. The survey reported in this paper has been made with the objective of bringing together the many variations and evaluating them. The granulation processes covered are those commonly used by the mixed fertilizer industry. Complex slurry-type processes that produce granular fertilizers such as nitric phosphate and ammonium phosphate-sulfate are not included.

Although published material has been reviewed, the principal sources of information for this survey are personal contacts—either visits or correspondence—involving a total of about 135 granulation plants.

Status of the Industry

Widespread use of granular mixed fertilizers is a relatively new development. However, granular materials have been used to a limited extent for 30 years or more. Materials such as granular cyanamide and sodium nitrate were available in the 1920's, and production of granular superphosphate—by the Oberphos process—began in 1929-30. The first production of granular mixed fertilizers in this country appears to have taken place in 1935-36 (39). No major trend toward granulation developed, however, until well after World War II. During the war, granular ammonium nitrate came into widespread use and

its popularity no doubt was a major factor in building up consumer interest in other granular products. By 1950, the first of what might be called modern granulation plants was put into operation (1). Since then, new plants have been built at a rapid rate.

The present survey indicates that there are 171 granulation plants in this country, either in operation or being installed. This figure, which should be considered only an approximation, includes as granulation plants only those in which the product is agglomerated and either screened or otherwise treated to remove both fines and oversize. The survey does not include plants that make a semigranular product by achieving some degree of agglomeration followed by screening to remove oversize, crushing the oversize, and returning the crushed material to the product. Such products usually contain a large proportion of fines. Some producers are making such a semigranular fertilizer as a first step in progressing from the usual powdered, nongranular product to a fully granulated one.

The approximate locations of the granulation plants are shown in Figure 1. Of the 171 plants, 162 are located in the central and eastern regions. The greatest concentration—over 50 plants—is in the Midwest section consisting of Iowa, Missouri, Illinois, Indiana, and Ohio. Granulation is moving eastward and southward from this area, the frontiers now being the Ohio and lower Mississippi Rivers. The Southeast has shown little interest in granulation thus far, because of the generally low analysis of fertilizers, and because the low price level in the area discourages new ventures. In the past year or so, however, granulation plants have been built in Mississippi and North Carolina, and it appears that interest is growing throughout the Southeast.

A rough estimate of current production of granular fertilizers is 3 to 4 million tons per year. This estimate is based on the number and capacity of the granulation plants, published figures on capacity versus actual production, and industry estimates as to the ratio of granular to total production in various regions. In a previous survey, Scholl *et al.* (32) reported that consumption of granular fertilizers was 1.3 million tons for the year 1954-55. Thus it appears that consumption of granular fertilizers has increased nearly threefold in the past two years. As an example of the increase which has taken place, producers estimate that at least 75% of the fertilizer now consumed in Texas, Oklahoma, and Kansas is in granular form; in 1954-55, the figure for this region was 37%.

Continuing growth in production of granular mixtures seems assured as analyses continue to increase, and producers move more and more to lower cost formulations that require granulation. Moreover, competitive pressure often leads to granulation of grades that have acceptable physical condition without granulation. The attractive appearance of the granular product is a strong sales factor which tends to force producers into granulation even though it may not be dictated by economics.

The most popular nutrient ratios in granular fertilizers in 1954-55 (32) were 1:1:1 and 1:4:4. The most popular granular grades in these ratios were 12-12-12 and 5-20-20, each with about twice the tonnage of the grades next in popularity, 10-10-10 and 3-12-12. Although no data were collected in the current survey as to the quantity of various grades, it appears that 12-12-12 and 5-20-20 are still the leading grades of granular fertilizers.

In many areas in which both granular and nongranular mixtures are sold, granular mixtures sell at a premium of \$1.00 to \$2.00 per ton. In other areas particular grades are available in only one form, either granular or nongranular, and comparisons cannot be made.

Mixing of Granular Materials

The simplest way to obtain a granular product is to dry-mix granular raw materials. There is some produc-

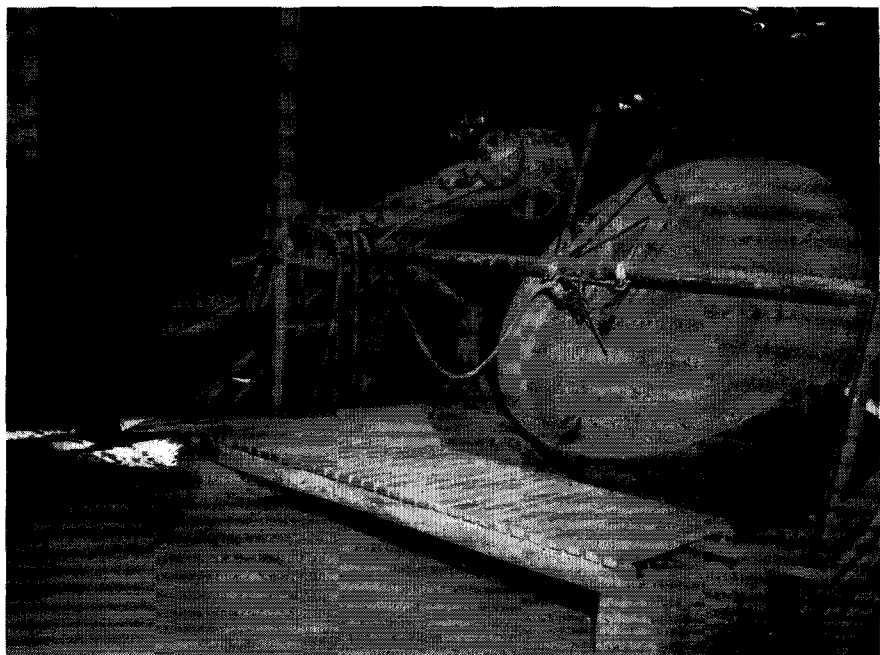
tion of this type, mainly by bulk blenders who normally apply the blend to the field from a spreader truck. The main problem is segregation of constituents. Transfer of the blend directly from the mixer to the truck is desirable, since intermediate storage in piles aggravates segregation. When blends of this type are bagged, segregation is not a serious problem if the bags are filled directly from the mixer. However, the product is relatively unattractive and has not gained wide acceptance except for no-nitrogen grades. These are often prepared by blending granular superphosphate with granular potash, even by companies that have granulation facilities. The usual granulation methods are not applicable to such grades, and it is difficult to produce a strong granule.

In areas in which base goods are used because of the multiplicity of grades, it may be desirable to granulate the base and blend it with granular nitrogen and potash materials to get the final product. This practice has not developed much as yet, but appears likely to grow as granulation moves into the Southeast.

Wetting and Drying

The earliest process for making a homogeneous granular product, as opposed to a blend of granular materials, involved wetting the mix with sufficient water to cause granulation and then removing the water in a dryer. This process was used in early plants in this country, and is still

Figure 2. Pan granulator of Commonwealth Fertilizers & Chemicals, Ltd., Melbourne, Australia



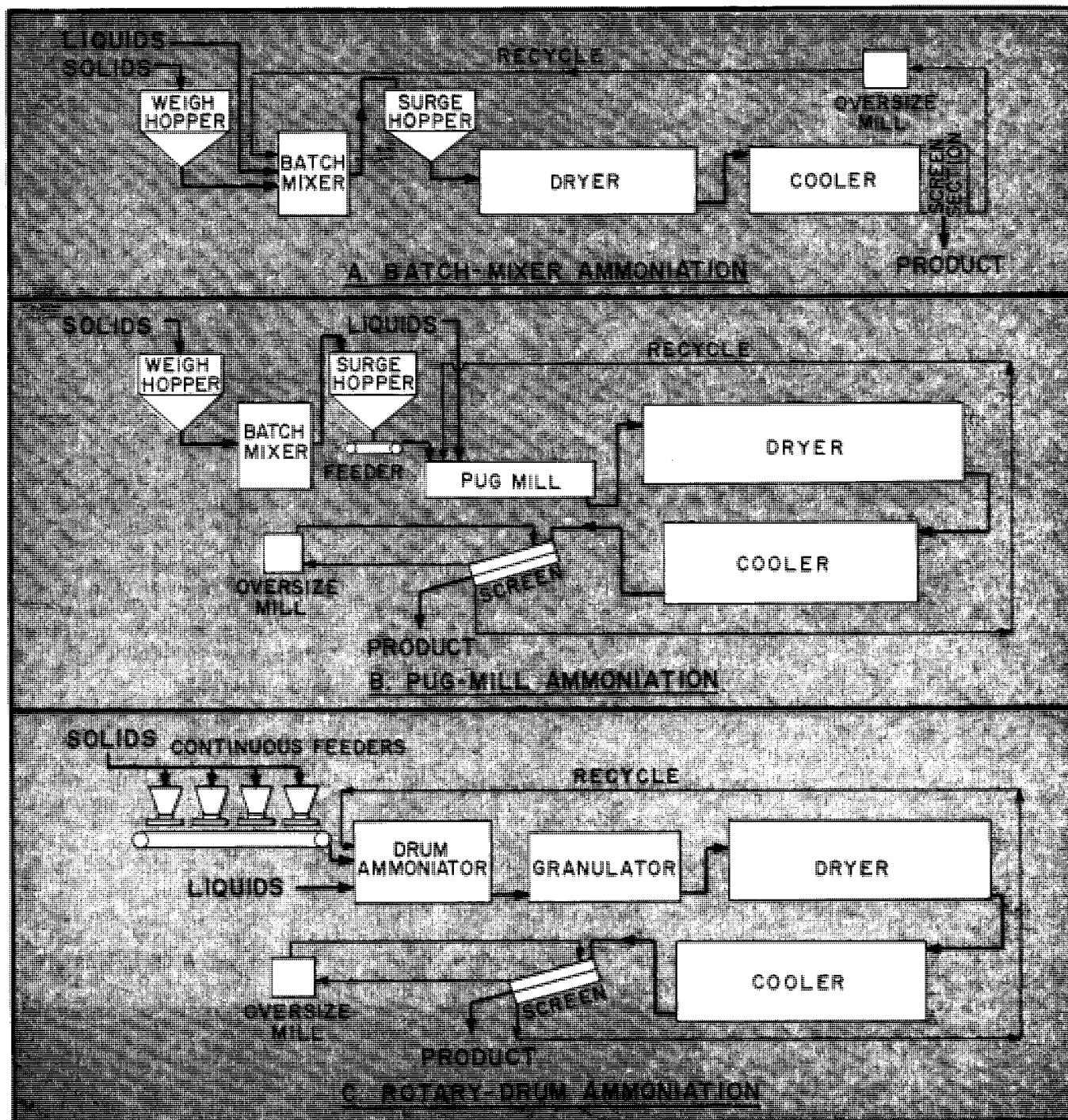


Figure 3. Flow sheets for granulation processes

widely used abroad. Normally an unammoniated mixture is used. When ammoniation is practiced, the ammoniation and granulation steps are often discontinuous, that is, no attempt is made to take advantage of the heat of ammoniation to promote granulation. Even when the ammoniated mix goes directly to the granulator, the main reliance is still on water (often added in the granulator), since no special effort is made to formulate a self-granulating mixture as in current practice in this country.

Various types of equipment are used in the wet-and-dry method. For continuous operation, both rotary drums and "pan granulators" are used. The latter (Figure 2) is a rotating pan inclined at an angle (40, 4). The larger granules gravitate to the rim of the lower edge of the pan where they are discharged; the fines are carried to the top of the pan and then slide through an area where they are wetted by water sprays. Thus the pan gives a combined classifying and granulating action which is said to

promote uniformity of granule size. The pan granulator appears to be satisfactory for wet-and-dry granulation and for simple spray-type ammoniation. However, tests at TVA have indicated that it is not especially promising for attaining high degrees of ammoniation (13).

The wet-and-dry method has fallen into disfavor in this country because of high drying cost and the relatively low strength of granules so produced. The present survey revealed no producers who rely on this method to

granulate the general run of grades. Its principal use is for no-nitrogen or very low-nitrogen grades and for normal superphosphate. Considerable quantities of these fertilizers are granulated, especially in the northern areas. Difficulty in obtaining granules of satisfactory strength was reported. Another difficulty is that free acid in superphosphate reacts with potassium chloride to cause release of acidic gases, and corrosion of equipment results. Most operators reported adding lime or limestone to avoid this and also to prevent attack on bags in storage.

In work at TVA (13), granular no-nitrogen grades have been made in a continuous rotary drum by replacing part of the superphosphate with phosphate rock and sulfuric acid. The heat of reaction and the plasticity of the fresh superphosphate formed gave good granulation at a low moisture content. Granules of satisfactory strength were obtained. Application of this method to the manufacture of granular superphosphate is being studied.

Modern Granulation Practice

The feature that best distinguishes modern granulation from the former wetting-and-drying practice is the technique of devising formulations that granulate readily, as opposed to the previous practice of imposing granulation as an extra step on formulations used for powdered material. The new practice involves the use of soluble salts in conjunction with high temperature to develop the proper volume of liquid phase with minimum use of water.

Other distinguishing features found in the majority of plants are the integration of the ammoniation and granulating steps and the use—when needed—of materials such as anhydrous ammonia, sulfuric acid, and phosphoric acid to promote granulation in the ammoniator by providing additional heat of reaction. In a typical plant, most of the granulation takes place in a continuous ammoniator, and the formulation is carefully selected to provide the right combination of liquid phase and temperature level. In other plants, most of the granulation is made to take place in the dryer or cooler by using a relatively high dryer temperature. These plants are especially dependent on formulations that contain highly soluble and easily fusible salts, so that granulation can take place at a low moisture level.

The types and arrangement of equipment used in modern granulation plants vary widely. The basic units

are the ammoniator, granulator, dryer, cooler, and screens. However, many plants lack one or more of the last four items. All have an ammoniator, and the type of ammoniator is the basis used in this survey for classifying plants. The principal types are batch mixers, pugmills, and continuous rotary drums (TVA type). There are other types, but they are used in relatively few plants. Simplified flow-sheets for the three major types are shown in Figure 3; the equipment arrangements represent majority practice for each, except for dry materials feeding in the pugmill and rotary drum processes. Both batch and continuous weighing are shown, but batch weighing is the prevalent method in both processes.

Batch Mixer. Since most producers of nongranular fertilizers have batch mixers, many of them have tried to use this equipment to make granular products, thereby minimizing new investment required for granulation. Three general methods have been used: (1) the batch mixer has been used for ammoniation and granulation, usually followed by either a rotary cooler or a handling system that provides some cooling; (2) the batch mixer has been used for ammoniation only, followed by a dryer—where granulation occurs—and a cooler (Figure 3A); and (3) the batch mixer has been used only for mixing solid raw materials, followed by a continuous ammoniator and a dryer or cooler, or both (Figure 3B).

Obviously the first scheme involves the least capital outlay and, for that reason, many companies have tried it, some with considerable success. Converting a batch mixer to granular production requires some modifications. Formulations are selected to provide the proper amount of liquid phase for granulation with such low moisture content that drying is not required. Good results have been reported in some cases in which either granular superphosphate or granular potassium chloride was used to control granulation. The granular raw material provided nuclei upon which other raw materials formed a coating.

The companies most successful with granulation in the batch mixer regard the detailed information about their methods as confidential. Other companies have had poor results and abandoned the method.

The second method, use of the batch mixer for ammoniation only, is widely used. Granulation takes place in a dryer operated at a relatively high temperature. In both methods, however, a major difficulty is that most batch mixers are not well suited to high degrees of ammoniation and use of large proportions of nitrogen solu-

tion and acid. Therefore, considerable operating difficulty has been encountered, and in many cases the mixer has been modified in an effort to improve operation.

One of the major problems is corrosion of the mixer when formulations contain large proportions of acid. Flights are most affected, but shell corrosion also is encountered. In contrast to continuous ammoniators with submerged injectors, batch mixers offer less opportunity for fast neutralization of the acid, and free acid apparently reaches the metal surfaces. Much less corrosion is encountered with nonpotash grades, indicating that hydrochloric acid fume may be the major corrosive agent. Stainless steel flights can be used to reduce corrosion, but the more common practice is to reduce the amount of acid.

Another problem is handling of the sticky mixtures often encountered in granulation. Scrapers cannot be used because of the flights. Hammers are used on the shell, instead, but these are often ineffective. Most producers minimize the problem by limiting the amount of either ammoniating solution or acid. The former increases the cost of formulation because a more expensive solid nitrogen material must be used to replace the solution. The major practice is to reduce the acid, which also helps decrease corrosion. In this operation the low temperature in the mixer prevents much granulation at that point, so that granulation must take place in the dryer. A sufficiently high temperature is used in the dryer to fuse the soluble salts (principally ammonium nitrate) in the mix and, thereby, produce enough liquid phase for granulation.

In efforts to reduce loss of ammonia from batch mixers, producers have made various changes in the method of introducing liquids. The simplest one is to relocate the solution inlet pipe so as to inject the solution as low in the mixer as possible. This allows the solids to give a limited scrubbing effect, and reduces loss. Another method is to use downcomers from a manifold to give better distribution over the length of the mixer. In some cases a horizontal distributor set close to the flights is used. Still another method is to alter the flights so as to locate the liquid distributors deeper in the solids. This usually involves cutting down the flights, which helps absorption but may interfere with proper discharging of the mixer. A variation of this is to notch the flights and insert a downcomer in the notch. Producers have reported some of these changes effective in obtaining efficient ammonia absorption at degrees comparable to those used in continuous mixers.

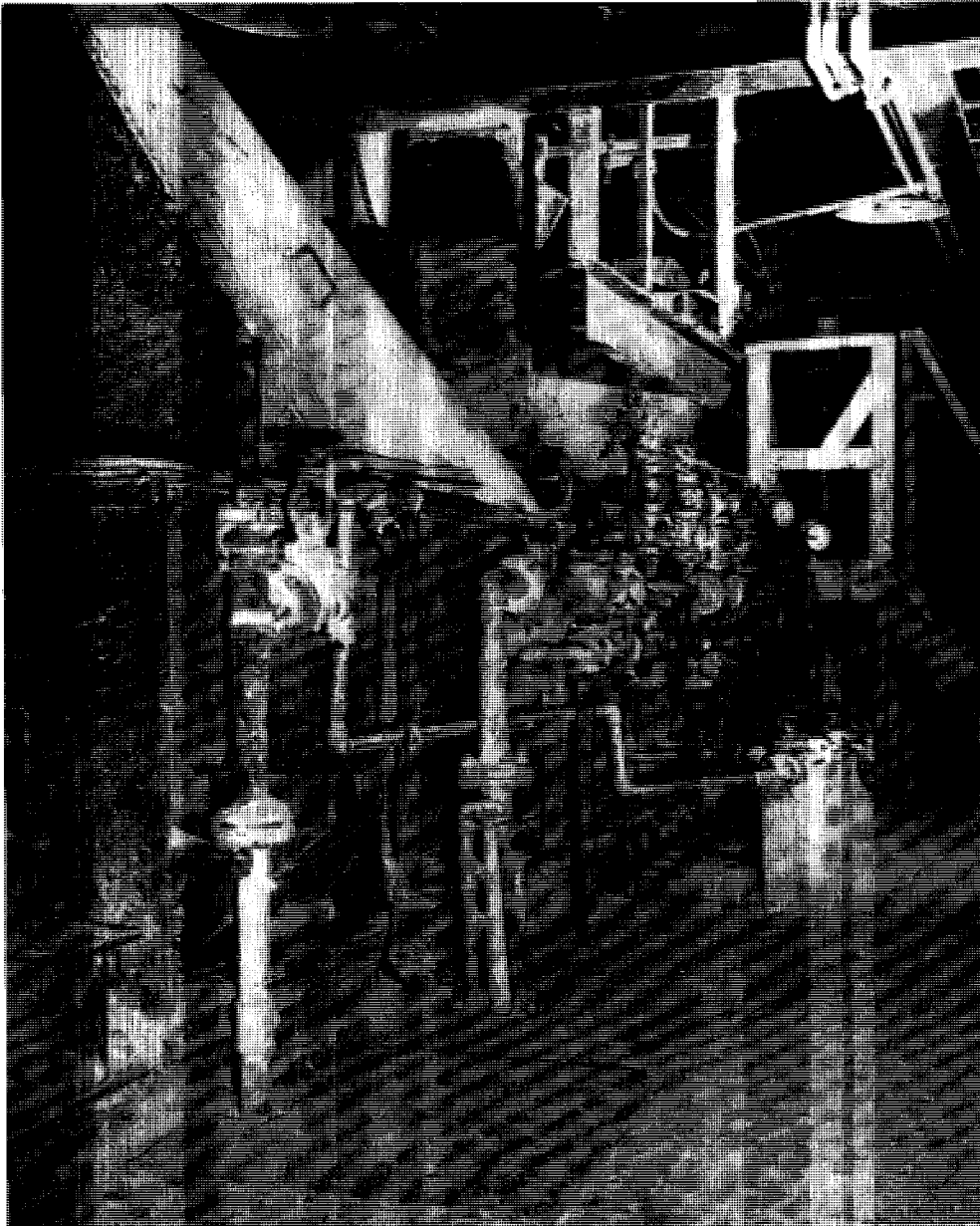


Figure 4. Pugmill ammoniator of Davison Chemical Co.

Pugmill. Because of the shortcomings of batch mixers, there has been a general trend to submerged injection of liquids in continuous ammoniators. One type of ammoniator used for this is the pugmill. Reynolds (31) has described operation in some of the plants equipped with pugmill ammoniators (Figure 4).

Most pugmills are of the twin-shaft type. A few ribbon mixers are also in use and these have been classed with the pugmills. Solid raw materials and recycled fines are normally fed into the inlet end of the pugmill and liquids are injected under the bed. In some cases provision is made for adding the fines at different points along the length of the unit. Advantages claimed by those who favor the pugmill over other types are:

- The kneading action gives a harder, stronger granule

- Operation more closely approaching the slurry state can be maintained without sticking or serious reduction in ammonia absorption. This tends to give a product with more uniformity in composition between granules
- More variation in operating conditions can be tolerated, which is said to be quite important with inefficient operating personnel. The positive action of the pugmill will move out mixes that might stick in other types of ammoniators

Disadvantages reported for the pugmill are:

- Maintenance costs and power requirements are generally higher
- The fume problem on the operating floor tends to be severe because it is difficult to provide effective fume removal

- Proper distribution of liquids under the bed is more difficult
- Unless a separate granulator is provided, the operator cannot see the material well until it enters the dryer. Thus, he has less time to adjust operating conditions

Distribution of liquids appears to be one of the more troublesome problems in operating a pugmill. If the distributors are placed lengthwise under the bed they must clear the paddles and thus are in a relatively inactive zone. In this location, material tends to cake over the holes or slots in the distributor. As an alternative, distributors can be inserted from the side or top between the paddles. This seems to give better operation but may produce local wet spots and consequently poor ammonia absorption. In several plants either compressed air or a rodding arrangement is provided to clear stoppages in the injectors.

Rotary Drum. The continuous rotary drum ammoniator with submerged liquid injection has been developed at TVA (26). Use of the TVA-type ammoniator in granulation has been described in numerous papers, both by TVA (13, 14, 15, 28) and by industry (8, 11, 12, 33, 34). The basic unit consists of an open, slightly inclined rotary cylinder with retaining rings at each end, and with a scraper or cutter mounted inside the shell (Figure 5). A rolling bed of solid material is maintained in the unit, and liquids are introduced through horizontal, multiple-outlet distributor pipes set lengthwise of the drum under the bed. Most of the drums in use range between 5 and 8 ft. in diameter and 5 and 15 ft. in length. Some plants have units quite close to the original TVA design. In others, variations have been made in an effort to improve operation or reduce plant cost.

One of the main variations is in design of the liquid distributors. Most plants use the TVA-type distributor set lengthwise under the bed, but some have installed a downcomer arrangement to avoid interference with the rolling action of the bed by the distributor, and difficulty in removing the distributor for maintenance. These downcomers vary widely. Special nozzles, flattened tube ends, and other combinations are used to inject the liquids, and various spacings of acid and solution downcomers are used.

The TVA-type distributors in use also vary widely—in length, difference in length between acid and solution distributors, position, type of openings, spacing of openings, direction of injection of liquid, and material of construction. Some operators stress

the importance of locating the distributors fairly high in the direction of rotation, while others locate them in the lower part of the bed. Some use slots in the tubes but most use drilled holes. Many use a uniform spacing rather than a graduated one. In some long ammoniators, two sets of distributors in series are used. All combinations of direction of injection are used; the only consistency is that the acid is usually injected after the solution and at an angle diverging to some extent from that of the solution. Little information is available on the relative effectiveness of the various arrangements, except that there is some agreement that impinging of the acid and solution streams on one another aggravates fuming.

Most solution distributors are made of stainless steel and most acid distributors are made of black iron. The life of these distributors depends on the grades of fertilizer produced and their formulations. The usual life of black iron acid distributors corresponds to the production of about 2000 tons of granular fertilizer. Corrosion of the sulfuric acid distributor occurs mostly on its exterior surface and around the holes through which the acid discharges. The corrosion is attributed mainly to hydrochloric acid which forms when the sulfuric acid contacts potassium chloride. Daily inspection, with replacement before the holes enlarge appreciably, is a helpful practice. Building up a lip or ring around the holes by welding is said to increase distributor life considerably. Use of Hastelloy C acid distributors for sulfuric acid is increasing; most of those who have tried this alloy believe that its longer life makes it worth the extra cost. Other alloys are also in use for sulfuric acid, and plastic-coated distributors are used successfully in some plants. Stainless steel is often used for phosphoric acid distributors.

Optimum ammoniator conditions for 1:1:1 ratios (the so-called "flat" type) are quite different from those for low-nitrogen ratios (the "steep" type, such as 1:4:4). Most of the change in going from one type to the other is accomplished by changing the formulation. However, some have used the design of the ammoniator to assist in this. In some cases different distrib-

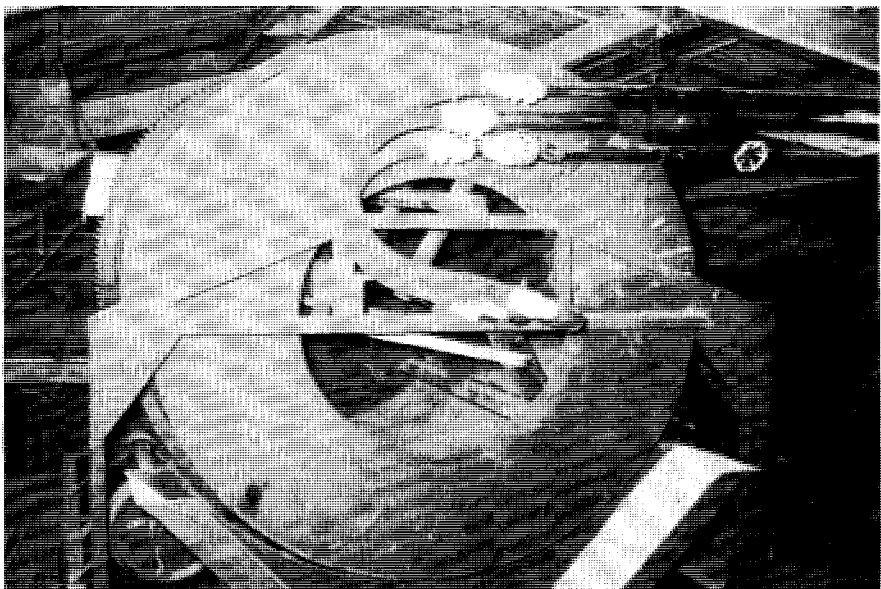
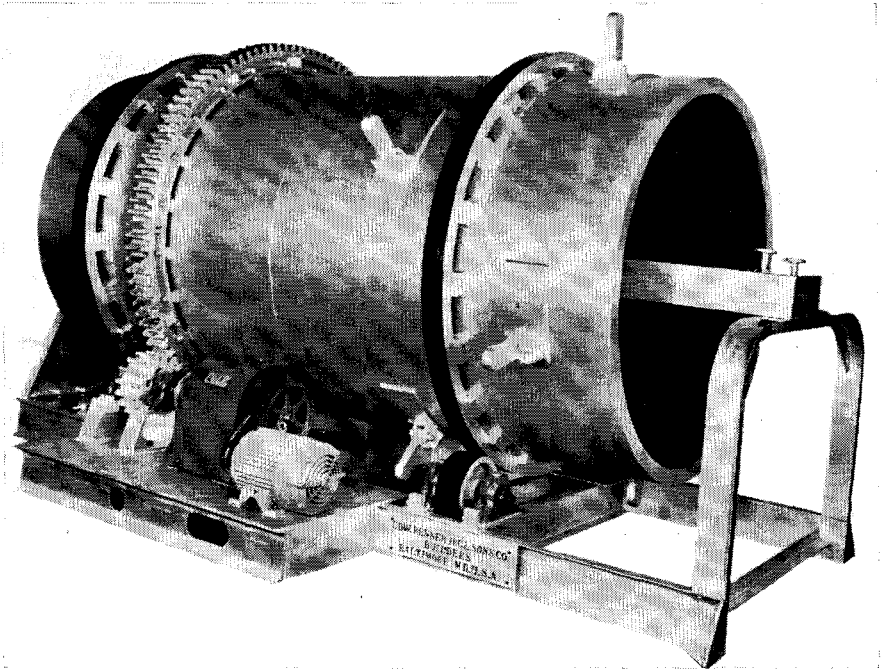
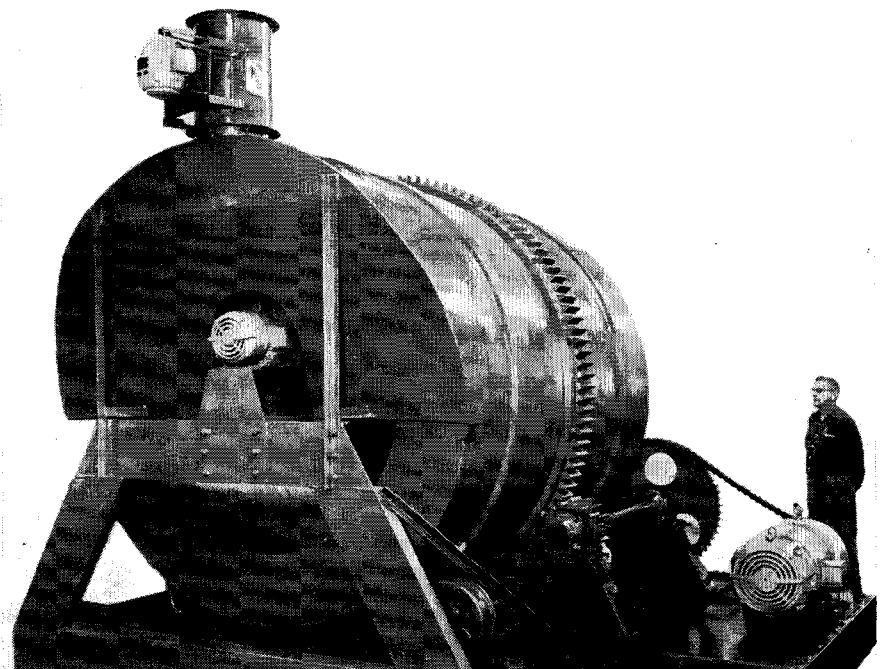


Figure 5. TVA-type drum ammoniators. Top: 8 by 8 ft. unit with fume hood, made by Stedman Foundry & Machine Co., Inc. Center: Combination ammoniator-granulator made by Edw. Renneburg & Sons Co. Bottom: D.M. Weatherly Co.'s 7 x 10 ft. unit showing liquid feed arrangement

utors are used for flat and steep grades. For the steep grades the distributors are relatively short in order to concentrate the heat of reaction in a "hot spot," thereby promoting granulation with a minimum addition of water. For the flat grades the distributors are made longer to spread out the heat of reaction and minimize over-agglomeration, which tends to occur when there is a high concentration of water-soluble nitrogen salts. Most plants, however, have a single set of distributors; the length is that deemed most appropriate for the majority of grades made.

Pilot-plant studies by TVA have confirmed the advantage of using longer distributors for flat grades; better control of granulation, improved absorption of ammonia, and less fume

were obtained with longer distributors. The pilot-plant data did not show any advantage for use of a short distributor in making steep grades.

Miscellaneous Types. The granulation processes described to this point are widely used. However, some companies have developed their own processes for use in their own plants. One of these on which published information is available is the Swift process (10, 18). A continuous rotary drum is used, but operation is similar to that of a batch mixer in that the drum has flights, and the liquids are sprayed on, rather than injected under, the bed. The scrubbing action of the solids in the long (38 ft.) drum is reported to give good ammonia absorption. Hammers are used on the drum to prevent sticking. Most of the nitro-

gen is supplied as ammonium sulfate, either added as such or produced *in situ*. One of the main advantages claimed is a high rate of production.

A continuous rotary drum is also used in the Stauffer plant at Los Angeles (6). The unique feature of this process is preneutralization of sulfuric acid with ammonia in a "bazooka" tube. This is a tube containing baffles to aid in mixing. The tube discharges the hot ammonium sulfate solution directly into the rotary drum where it is mixed with superphosphate and other constituents. The main drum is followed by a granulating drum.

All of the processes described thus far have been of the solids type as opposed to the slurry type. The latter is widely used in the production of nitric phosphates and ammonium

Choosing the Most Economical Source of P₂O₅

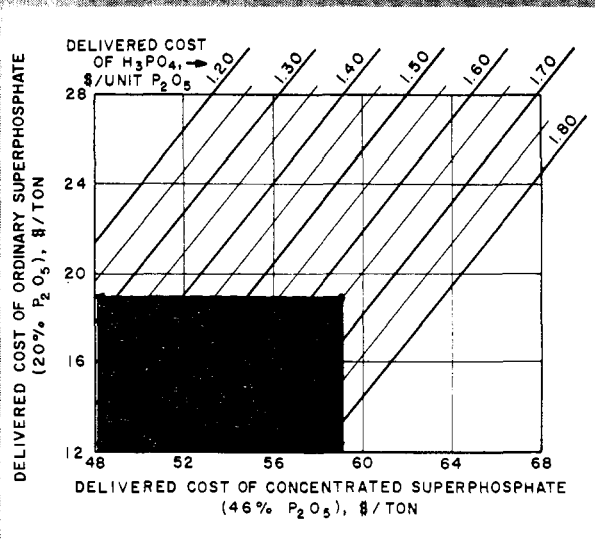


Figure 6

Figures 6, 7, and 8 compare the cost of concentrated superphosphate vs. DAP or phosphoric acid in various formulations. In the example shown by the red block in Figure 6 (where ordinary superphosphate is \$19 a ton and concentrated super is \$59), phosphoric acid would be more economical in 5-20-20 formulations if it is less than \$1.65 per unit of P₂O₅. Figure 7 shows that for 12-12-12, phosphoric acid at less than \$1.60 per unit of P₂O₅ is more economical than ordinary and concentrated super at \$19 and \$59 a ton, respectively. For a 12-12-12 formulation, Figure 8 shows that DAP at \$113 a ton is more economical. These charts are based on costs of raw materials as follows: ammonium sulfate, \$38 a ton; sulfuric acid, \$23 a ton; nitrogen solution, \$1.20 per unit f.o.b. and \$4.00 per ton of freight. These costs have a minor effect on the relationship shown.

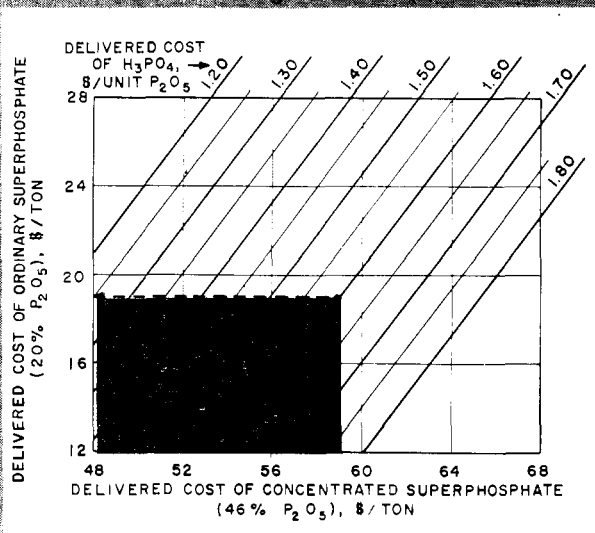


Figure 7

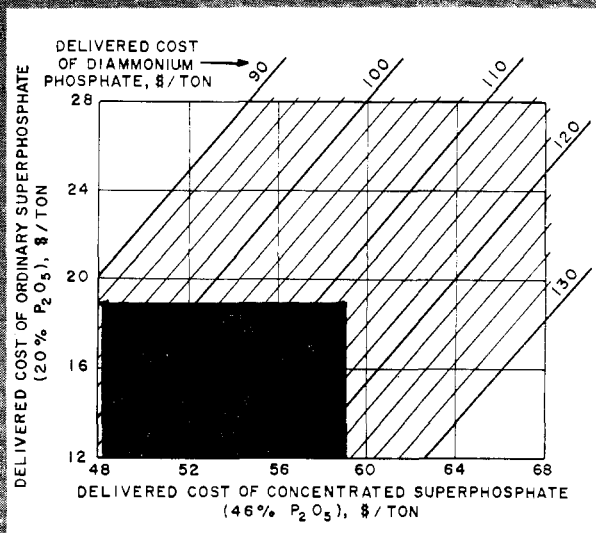


Figure 8

phosphate-sulfate but is not widely used for granular mixed fertilizers. In the only plant reported to be using such a process (24), solid and liquid raw materials are mixed in proportions to give a slurry, which is then mixed with wet solid recycle, and granulated in a pugmill. Advantages claimed are wide flexibility in formulation and superior absorption of ammonia.

Status of Various Types. An approximate distribution of plants among the various types is given in Table I. Process requirements vary so with grade that no one type of plant can give optimum operation for all conditions. Each plant type has advantages in respect to some specific problem, but each has shortcomings in regard to other problems. Submerged injection in a rotary drum appears to offer the best compromise, and this has become the most popular process; it accounts for 51% of the plants in this survey. Batch mixers occupy second place (27%), mainly because retaining the batch mixer reduces the cost of converting to granulation. The pugmill is in third place with 13%; it gives good operation but power and maintenance requirements and liquid-injection problems have held back its use.

Granulation plants that use batch mixers for ammoniation usually are relatively small, so that they account for a smaller percentage of the production of granular fertilizer than their numerical percentage. A rough estimate, based on published capacity figures, indicates that about 63% of the production of granular fertilizers is by plants using rotary-drum (submerged injection) continuous ammoniators. 19% is from batch mixer plants, 12% from pugmill plants, and 6% from miscellaneous or unknown types. Granular fertilizer producers who use batch mixers for ammoniation tend to add continuous ammoniators as soon as they can. Because of this the position of the batch mixer probably will recede in time.

Mixing and Feeding Materials

Most plants that have converted to continuous ammoniation and granulation have retained their old weighing systems and batch mixers, using the latter to mix solid materials. The weighing systems vary, as in nongranular plants, from use of a cart or tractor shovel on a scale to systems with feed-and-weigh hoppers. The mixed batch ordinarily passes to a surge hopper, from which it is fed continuously to the ammoniator. Some plants use an automatic gravimetric feeder for this but many merely weigh on a time cycle and manually adjust the

Table I. Types of Granulation Plants

Type	Number
Batch mixer	46
Pugmill	22
Rotary drum (submerged injection)	88
Miscellaneous	11
Unknown	4
TOTAL	171

feed from the surge hopper. Some have tried both methods and found the latter to be more dependable.

Plants which still use a batch mixer for ammoniation usually pass the ammoniated mix to a surge hopper that feeds the dryer. Rough manual control of the feed to the dryer is adequate since no proportioning is involved. Some are reported to omit the surge hopper and dump the batches directly into the dryer, a practice which gives very inefficient drying. New plants, as compared to remodeled plants, usually have automatic continuous feeders on each raw material feed hopper (Figure 3C).

Liquids usually are fed through rotameters, but in some cases they are fed by filling and emptying a measuring tank in the line on a time cycle. Some plants have had difficulty in metering anhydrous ammonia and have installed a heat exchange system to avoid vaporization in the meter.

Control of Granulation

Effective control of granulation, a problem in many plants, is important because it affects productive capacity of the plant. Obviously, if much of the product must be recycled, output is decreased, and operating cost per ton of product is increased. In some grades, excessive recycle may adversely affect granulation and cause still further increases in recycle rates. Control of granulation is particularly important in those plants in which granulation occurs mainly in the ammoniator, since failure to control granulation effectively may result in fuming and poor absorption of ammonia.

One reason for difficulty in controlling granulation is the large number of grades produced. Each grade presents a different control problem. Frequent change from one grade to another causes problems, because the plant must be adjusted to the new conditions, and because there is not time enough for operators to become proficient in granulating any one grade.

The most widely accepted theory is that granulation occurs when the liquid phase of the mixture reaches the

proper proportion, which usually is in the range of 20 to 30%. The liquid phase consists of water plus dissolved fertilizer salts. The amount of salts that dissolves in a given amount of water depends on the quantity and kind of salts, and on the temperature. Most operators agree that increasing the temperature to obtain the proper amount of liquid phase is more effective in making strong granules than is increasing the water content. In extreme cases, the liquid phase may be made up almost entirely of fused salts with very little water.

The liquid phase theory is useful and generally valid, but no method of quantitative application has been found that can be used with confidence for all formulations. One reason for this situation is that it is impossible to predict the solubility of the complex system of salts involved in fertilizer manufacture. Also, chemical reactions and granulation take place concurrently so the proportion of liquid phase may change as the mixture progresses through the process. In addition, the cementing action of new chemical compounds formed during ammoniation may play an important part in granulation.

Methods used for control of granulation may be divided into two classes, basic and operating controls. The basic controls are predetermined operating conditions which are not subject to controlled variation during a run on one grade. Operating controls are those which may be varied from minute to minute by the operator to maintain granulation at an optimum or, at least, satisfactory level. As an illustration of these two types of controls, selection of the formulation is a basic control, but the operator may vary the proportion of sulfuric acid within certain limits as an operating control. Ideally, the basic controls should establish conditions near enough to those required for good granulation that the operating controls are fully adequate to deal with accidental disturbances or uncontrolled variables.

Basic Controls. Selection of formulation is the most common and important basic control. The formulation determines the kind and amount of soluble salts and the minimum water input. It also determines the heat of reaction, and hence it has a strong influence on the temperature attained in the ammoniator.

For high-nitrogen grades, such as 12-12-12, the most economical formulations, from the viewpoint of raw materials cost, usually result from the use of nitrogen solution plus enough sulfuric acid to react with the ammonia in excess of that absorbed by the superphosphates. However, such

formulations have a strong tendency to overgranulate because of the large heat of reaction and amount of liquid phase. The principal preventive measure is to add ammonium sulfate to the formulation, and thereby reduce the quantity of nitrogen solution and sulfuric acid. The usual practice is to add somewhat less ammonium sulfate than would be required to prevent overgranulation. The remaining tendency to overgranulate is dealt with by operating controls.

The choice of nitrogen solution also is important in formulating for high-nitrogen grades. Ideally, the solution should contain only enough free ammonia to react with the superphosphates. This would obviate the need for sulfuric acid and minimize overagglomeration by reducing the heat of reaction. Such solutions are available but are not widely used because they have relatively high salting-out temperatures.

In low-nitrogen grades, such as 5-20-20, it is usually difficult to get enough heat and liquid phase for good granulation when nitrogen solutions are used. The general practice is to increase the heat of reaction by using anhydrous ammonia and sulfuric acid instead of nitrogen solution. The remaining deficiency in granulation tendency is dealt with by operating controls.

Most plant operators have found or selected formulations for most grades that provide satisfactory granulation. However, many are not entirely satisfied with their formulations and are trying to improve them to get better granulation or reduced formulation cost. Some plants modify the formulation by using more sulfuric acid at start-up to bring the equipment quickly to operating temperature. In some cases, formulations used in winter differ from those used in summer.

Coarse or granular potash is widely used as a basic control to aid granulation of low-nitrogen, high-potash grades. The majority of manufacturers report that granular potash is helpful, or even essential, for good granulation of 1:4:4 ratios, but unnecessary or harmful for 1:1:1 ratios. Most operators have definite ideas about the optimum particle size of the potash, but few reported actual data to support their opinions. Potash producers have been forced to supply granular potash in several different particle size specifications to meet the varying demands of their customers. The particle sizes of granular or coarse potash materials currently offered are given in Table II. Recent studies by TVA indicated that potash in the size range of 6 to 16 mesh is the most effective in promoting granulation of

4-16-16 with the least sulfuric acid or water. However, good granulation was obtained with increased water or acid when only part of the potash was in the 6- to 16-mesh range. When the plus 10-mesh fraction of a commercial granular potash was removed by screening, the remaining (-10 mesh) material was less effective in promoting granulation than the original material. Satisfactory granulation was not obtained with any amount of water or acid when all the raw materials were finer than 16-mesh.

Granular triple or ordinary superphosphate was found in TVA tests to be effective in promoting granulation, and some use of these materials for this purpose is reported in industry. In some instances, granular dolomite is used to promote granulation.

Use of high recycle rates to control granulation may be classed either as a basic or operating control, depending on whether or not the amount of recycle is under the control of the operator during operation. For instance, in preparation for a run on a grade in which the need for a high rate of recycle is anticipated, a relatively coarse screen may be installed to throw an increased proportion of the throughput into the recycle circuit. In some plants, chutes may be rearranged to recycle the crushed oversize to the ammoniator to provide for an anticipated need for increased recycle. If these changes can be made during operation as the need arises, they may be classed as operating controls.

Operating Controls. The principal operating controls for granulation are addition of water, addition of steam, use of cooling air in the ammoniator, minor variations in sulfuric acid rate, variation in recycle rate, and control of dryer temperature.

When granulation is expected to occur in a countercurrent dryer by partial fusion of the mix, the principal control of granulation is by control of the dryer temperature. This method seems to be simple and effective. The dryer usually is fired with an open flame, and the operator controls granulation by turning the flame up or down. This method usually is used with formulations that provide a large proportion of ammonium nitrate; the resulting mixes are readily fusible at a moderate temperature and a low moisture content. A common disadvantage of this type of operation is the formation of fume in the dryer. Another disadvantage is the difficulty in providing a readily fusible mix for low-nitrogen grades.

In those plants in which granulation is expected to occur mainly in the ammoniator, the most common method for control of granulation of low-nitrogen grades is to vary the

amount of water or sulfuric acid. Facilities for heating the water are available in some cases. Some plants use steam and consider it very helpful. Several plants are considering installation of steam facilities, but some that were already equipped reported steam to be of little use. In pilot-plant studies by TVA (13), steam was found to be effective in obtaining granulation of some grades at low moisture content. Hot water was only slightly better than cold water.

Since recycle is deleterious to granulation of most low-nitrogen grades, most operators try to keep the amount of recycle as low as possible when making these grades. This can be done only by maintaining high granulation efficiency or accepting a wider size range in the product. To minimize the deleterious effect of recycle, a few plants are equipped to recycle the fines without cooling.

Operating controls for high-nitrogen grades include the use of cooling air in the ammoniator, variation of sulfuric acid rate, and use of more recycle than would normally be needed to dispose of fines.

Only a few plants are equipped to use cooling air on the bed of material to control overgranulation. The consensus was that air is effective in controlling minor fluctuations but not effective as a primary control.

Reduction in the sulfuric acid rate to correct overgranulation is usually effective. However, the normal formulations for high-nitrogen grades contain only enough sulfuric acid for absorption of the ammonia, so any considerable reduction may result in excessive loss of ammonia. Nevertheless, reduction in acid rate is regarded by many as the best choice of the control methods. No additional investment is required to use the method, and ammonia loss is usually not very great because reduction in acid feed rate is only temporary.

Use of recycle to control granulation is rather common, although it is difficult to get a clear picture of the effectiveness of this control. Many plants have a surge bin and feeder from which recycle can be fed at a controlled rate. Overgranulation or undergranulation may be corrected at least temporarily by increasing or decreasing the recycle feed rate. Some operators claim that good permanent control of granulation of high-nitrogen grades can be maintained by operating with a recycle rate high enough to give incomplete granulation, thereby producing the increased amount of fines required to maintain the high recycle rate.

Some plants are equipped to provide positive control of the recycle rate by (1) narrowing or widening

the particle size range of the product, thus diverting more or less of the throughput to recycle, (2) recycling crushed oversize to the ammoniator rather than to the screens, or (3) recycling some of the onsize product. These systems have the advantage that positive prevention of overgranulation is provided regardless of the liquid phase content of the formulation. Also, more (in some cases, all) of the product consists of the original round granules, and less or none of the product is in the form of irregular particles obtained when the oversize is crushed. Use of a large proportion of recycle to control granulation may decrease the net production rate or overload one or more of the production units. It may also decrease the

retention time in the ammoniator sufficiently to impair absorption of ammonia.

Any of the recycle control systems adds to the cost of the plant and the complexity of operation, and some plants operate quite well without such control. However, many in the granulation field are convinced that this method best controls the self-accelerating effect of accidental variations in granulation.

When most of the granulation is expected to occur in a granulator following an ammoniator of the pugmill type, or in a cocurrent dryer, granulation is usually controlled by controlling the plasticity of the mix leaving the ammoniator. Methods for controlling plasticity of the mix are generally

similar to those used in connection with granulation in continuous ammoniators.

Formulations

The formulations used in production of granular fertilizers vary widely, because of differences in process and local differences in costs of raw materials. Also, it is likely that many plants have not had sufficient experience to determine optimum formulations.

Two of the most popular grades of granular fertilizers are 12-12-12 and 5-20-20. Some of the formulations used in producing these grades are shown in Tables III and IV.

Table II. Particle Size of Granular Potash Currently Available

Percentage Retained on Screen, Cumulative^a
Company

Screen mesh (Tyler)	A					F		G	H
	Type 1	Type 2	B	C	D	E	Type 1		
6		0						Trace	
8	0	3.6				0		10	1.1
10	0-10	35.9	1.0	4.5	8		16	91	14.8
14		71.7	12.6	33.4	18		58	97	42.6
20	65-85	89.5	72.6	73.6	45	80.8	82	98	79.0
28	85-95	96.6	99.6	94.5	72		96		94.6
30						95.1		93	
35	95-100	98.8		97.7	88		99		98.4

^a Typical or average analysis. Information supplied by producers in July 1957. ^b 18 mesh.

Table III. Formulations for 12-12-12 Granular Fertilizers

Lb./Ton of Product

Material	Swift process (18)	Davison's Trenton process (31)	Spencer and ArkMo (2)	Diammonium phosphate (17)	TVA pilot plant (13)	Batch mix-countercurrent dryer	Phosphoric acid	Urea solution (19)
Nitrogen solution ^a	50	407	649 ^b	445	500	586	446	340 ^c
Anhydrous ammonia	119
Ammonium sulfate	593	400	...	136	200	96	293	316
Urea	70
Diammonium phosphate	166
Ordinary superphosphate	149	281	729	775	513	697	761	412
Triple superphosphate	457	415	207	...	311	214	...	372
Phosphoric acid (75% H ₃ PO ₄)	168	...
Muriate of potash	400	397	400	408	408	400	400	400
Sulfuric acid (100% basis)	260	130	138	139	139	56	62	210

^a Solution containing 41% N and 22.2% ammonia, except as noted. ^b 37% N. ^c 45.5% N (urea-ammonia solution).

Table IV. Formulations for 5-20-20 Granular Fertilizers

Lb./Ton of Product

Material	Swift process (18)	Davison's Trenton process (31)	Spencer and ArkMo (2)	TVA pilot plant (13)	Calcium metaphosphate (29)	Phosphoric acid	Urea solution (19)
Anhydrous ammonia	97	56	88	129	...	125	...
Nitrogen solution ^a	50	146	76 ^b	...	297 ^b	...	220 ^c
Ordinary superphosphate	347	377	740	278	460	902	540
Triple superphosphate	736	703	546	769	650
Calcium metaphosphate	510
Phosphoric acid (75% H ₃ PO ₄)	412	...
Muriate of potash	667	646	668	643	662	667	670
Sulfuric acid (100% basis)	144	140	...	129	119	...	20

^a Solution containing 41% N and 22.2% ammonia, except as noted. ^b 37% N. ^c 45.5% N (urea-ammonia solution).

In the production of 12-12-12 granular fertilizer, most or, in some cases, all of the nitrogen is supplied as solution. Because of the large proportion of liquids, the principal problem in controlling granulation is prevention of overgranulation. Most plants use some ammonium sulfate to minimize plasticity and prevent overgranulation. For the plants surveyed, the amount of ammonium sulfate in 12-12-12 formulations varied from 100 to 700 pounds per ton, and averaged about 350. In those plants in which unlimited recycle can be used to control plasticity, no ammonium sulfate is necessary. However, so much recycle is required that the plant's production capacity may be seriously reduced. Also, the hygroscopicity of the product is increased when all of the nitrogen is derived from solution.

Formulation cost usually increases

as the amount of ammonium sulfate is increased, partly because ammonium sulfate is usually more expensive than other forms of nitrogen. Moreover, because of its low analysis, increased use of ammonium sulfate leaves less room in the formulation for superphosphates; less ordinary superphosphate and more concentrated superphosphate must be used. The increased proportion of concentrated superphosphate increases formulation costs. For these reasons, most plants try to use no more ammonium sulfate than is necessary for control of plasticity. Control of plasticity is often poor in 1:1:1 grades, as evidenced by widespread difficulty with fume and loss of ammonia.

Sulfuric acid is required in most 12-12-12 formulations for ammonia absorption. The amount used (Table III) was found to vary from about 50 to 250 pounds per ton (100% basis).

Increasing the amount of sulfuric acid decreases the degree of ammoniation of the superphosphates, which should help prevent excessive ammonia loss. However, increased amounts of sulfuric acid raise the reaction temperature and, therefore, the plasticity of the mix. This effect often results in poor ammonia absorption as well as fume. For this reason, increasing the sulfuric acid input does not always have the desired effect of improving nitrogen recovery.

Formulations used for 4-16-16 and 5-20-20 commonly include anhydrous ammonia as the principal source of nitrogen. It is usually the only source of nitrogen for 4-16-16. About half of the plants were found to use ammonia as the only nitrogen source for 5-20-20; others use a combination of ammonia and solution; a few use only solution. Sulfuric acid is normally used in amounts ranging from 30 to 230 pounds (100% basis) per ton. Often the amount used is in excess of that needed for ammonia absorption. The excess acid is used to obtain sufficient granulation or to supply sufficient heat for drying when the plant does not have a dryer.

The degree of ammoniation appears to be generally higher in granulation plants than in those producing powdered goods. The majority of the plants surveyed use degrees of 6 and 4 lb., respectively, for ordinary and concentrated superphosphate (expressed as pounds free ammonia per unit of P_2O_5). A few reported that inventory checks over a long period indicated too much ammonia loss at this level and therefore they have reduced it somewhat. Some of the batch mixer plants use little acid or ammonium sulfate in high-nitrogen mixes, a practice which results in a high degree of ammoniation and loss of part of the ammonia. Input degrees of ammoniation as high as 10 lb.—with retention of 8 lb.—have been reported. Although the ammonia loss is expensive, it is offset partially—or perhaps fully in some situations—by the elimination of most of the cost for acid, and by increased use of ordinary superphosphate and low-cost nitrogen solutions.

Phosphoric acid, either wet-process or electric-furnace grade, is used in several plants as a replacement for some or all of the concentrated superphosphate in standard grades. Phosphoric also replaces some or all of the sulfuric acid. Most operators felt that a phosphoric acid formulation rather than a conventional one gives as good or better operation. Several mentioned that phosphoric acid is economically attractive, because it provides room for more ordinary superphosphate in the formulation. This

Figure 9. Countercurrent dryer-cooler arrangement widely used with batch mixers. Burner is on platform at right. Blue Valley Equipment Mfg. & Engineering Co.

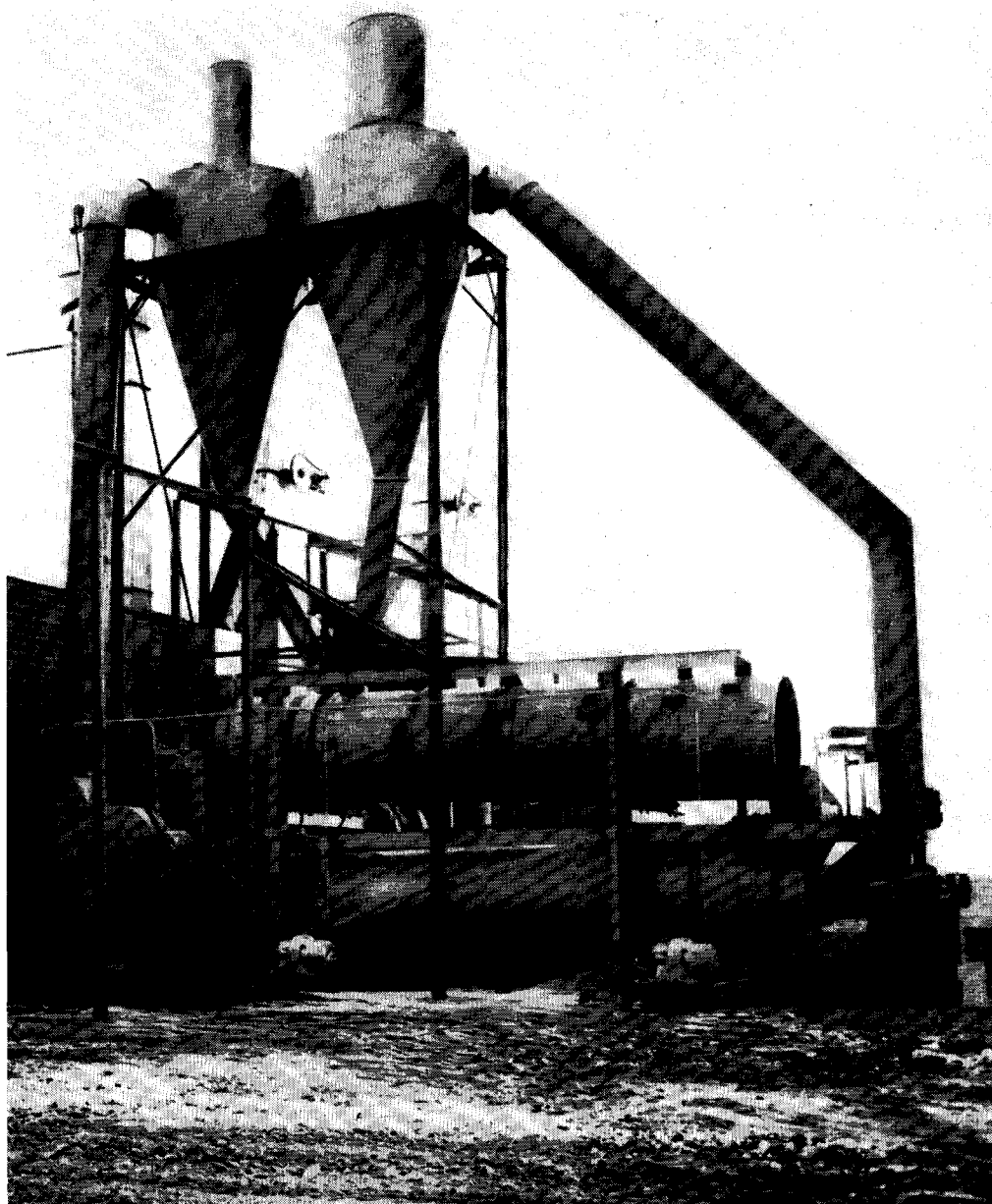


Table V. Alternate Formulations for 12-12-12 and 5-20-20 Granular Fertilizers

Raw material	Analysis	Formulation, Lb./Ton				
		12-12-12			5-20-20	
		(A) Conventional (with CSP)	(B) With diammonium phosphate	(C) With phosphoric acid	(D) Conventional (with CSP)	(E) With phosphoric acid
Superphosphate						
Ordinary	20% P ₂ O ₅	537	788	761	293	902
Concentrated	46% P ₂ O ₅	300	761	...
Phosphoric acid	54% P ₂ O ₅	168	...	412
Diammonium phosphate	21% N, 53.5% P ₂ O ₅	...	160
Nitrogen solution X	41% N	494	436	446
Ammonium sulfate	20.5% N	195	146	293
Sulfuric acid	66° Bé.	162	158	62	140	...
Potassium chloride	60% K ₂ O	400	400	400	667	667
Anhydrous ammonia		125	125

NOTE: All formulations are based on no overages, 3% loss of free ammonia, and 2% loss of availability in superphosphates. The 12-12-12 formulations assume product moisture of 1.5%; the 5-20-20 formulations assume product moisture of 3%.

is a particularly important advantage for companies that make their own ordinary superphosphate.

The economics of using phosphoric acid in 5-20-20 and 12-12-12 granular fertilizers has been evaluated for the formulations shown in Table V. These formulations are believed typical of those used in industry. The charts, Figures 6 and 7 (see page 820), compare the relative economics of the phosphoric acid and conventional formulations for these grades. For instance, the cost of formulating 5-20-20 with phosphoric acid would be lower than that for the conventional formulation if the phosphoric acid cost were less than \$1.65 per unit of P₂O₅ and if ordinary and triple superphosphate cost \$19 and \$59 per ton, respectively. The cost of formulating 12-12-12 with phosphoric acid would be lower than that for the conventional formulation if the phosphoric acid cost were less than \$1.60 per unit.

In a few instances phosphoric acid was found to be in use in making unusually high-analysis grades such as 14-14-14 or 15-15-15.

Some companies are using or have tried diammonium phosphate in formulations for granular fertilizers. When used in standard grades such as 12-12-12, diammonium phosphate, like phosphoric acid, has the advantage of leaving room for more ordinary superphosphate. It has the additional advantages of providing a drier formulation and decreasing the amount of reaction and, hence, the ammoniator temperature.

The economics of using diammonium phosphate in 12-12-12, for the formulations shown in Table V, is compared with that of a conventional formulation in Figure 8 (see page

820). The chart shows that, for example, the diammonium phosphate formulation is more economical when the diammonium phosphate cost is less than \$113 per ton, if the costs of ordinary and concentrated superphosphates are \$19 and \$59 per ton, respectively.

Several plants use diammonium phosphate to make unusually high-analysis grades such as 16-20-0, 15-15-15, 15-30-15, and 13-39-0. Some operators expressed the opinion that granular products made with diammonium phosphate had better physical properties than the same grades made with other formulations. However, some companies reported difficulty with some grades because of disintegration of granules in storage.

A few plants use calcium metaphosphate as an ingredient in granular fertilizers, particularly in low-nitrogen grades. Advantages reported are improved granulation, low moisture content of the products, and good physical properties.

Use of Granulator

As discussed previously, granulation occurs primarily in either the ammoniator or the dryer, depending on the process used. For this reason, most granulation plants do not have a granulator as a separate unit. The following tabulation shows the practice in 97 granulation plants:

TYPE OF GRANULATOR	NUMBER OF PLANTS
Separate unit	21
Section of ammoniator	8
Section of dryer	9
None	59

None of the batch-mixer plants and only about a fourth of the pugmill plants were found to be using a granu-

lator or granulating section. However, 60% of the plants that use a rotary-drum ammoniator have a granulator or granulator section.

The function of a granulator in most plants is to provide a rolling action that improves the shape, firmness, and uniformity of agglomerates that have formed in the ammoniator. In a few instances water is sprayed on the material in the granulator to cause additional agglomeration. However, in most plants the granulator merely provides some additional retention time during which the granules are subjected to a rolling action. The need for this in improving granule strength is greatest in low-analysis, low-nitrogen grades.

Most of the separate granulators used are plain drums, normally with a few ribs to promote the rolling action. A few plants have altered the drum design to increase the rolling action.

When a section of the ammoniator is used as a granulator, a retaining ring separates the two sections. Bed depth in the granulator section usually is shallower than that in the ammoniator section.

When a section of the dryer is used as a granulator, it usually consists of a portion of the feed end, with flights omitted.

The granulator is considered desirable by some because of either the effect on granulation or the beneficial effect in firming up granules, or because it gives a surge capacity which allows the operator more leeway in controlling the process. The opposite view is that any delay between ammoniator and dryer allows the mix to cool, and thereby reduces dryer efficiency and degree of granulation in the dryer. It appears that good operation can be

obtained without a granulator but that there are advantages in having one. Whether or not the advantages are worth the additional cost is a question difficult to answer.

Drying

The main differences in drying practice are in the type of solids-gas flow and the size of the drum. In a typical group of 41 plants, about 60% of the dryers were of the cocurrent type, involving parallel flow of combustion gas and solids. The remainder were of the countercurrent type. In addition to these, there are a few cross-flow dryers in use.

The cocurrent dryer has the advantages of less decomposition of product during drying and less critical nature of the inlet air temperature. Another advantage, as pointed out by Leister (23), is that less air flow is required because the air leaves the dryer hot, and therefore can hold a relatively large amount of moisture without danger of condensation. Nielsson (25) states that case-hardening of the granules takes place in cocurrent dryers and that this is beneficial in making high-nitrogen grades because it minimizes overagglomeration in the dryer.

One of the arguments for the countercurrent dryer (Figure 9) is that there is less trouble with sticking at the inlet. Formulations containing large amounts of ammonium nitrate tend to develop a large amount of solution phase and therefore tend to stick in the hot zone at the inlet to cocurrent dryers. In contrast, the coolest zone in the countercurrent dryer is at the inlet. By the time the material reaches the hot zone most of the water is gone and the ammonium nitrate produces less solution phase because of the increase in melting point as it becomes drier.

Simpler construction at the inlet end is another point in favor of countercurrent dryers. However, the main reason for their popularity appears to be that they work better in conjunction with batch mixers. In most of the batch mixer plants granulation takes place in the dryer, a function which is best fulfilled by the countercurrent type. The case-hardening effect in cocurrent operation is a handicap when granulation must be accomplished in the dryer (25). Moreover, the relatively high temperature level required for granulation in the dryer is more easily obtained in the countercurrent type.

From a mathematical analysis, Leger (22) has concluded that: (a) the product is drier at the dryer exit in cocurrent operation than in counter-

current; (b) the product from a cocurrent dryer is cooler and therefore more easily screened or further cooled; and (c) granule growth in the dryer is more rapid in countercurrent operation.

Most dryers range from 5 to 8 ft. in diameter and from 30 to 50 ft. in length. A 5-foot by 30-foot size is widely used in batch mixer plants, producing about 10 tons per hour; in continuous plants, producing at 20 to 30 tons per hour, the usual size is 7 or 8 ft. in diameter by 40 ft. or more in length.

Temperature of the product issuing from cocurrent dryers is usually in the range of 160° to 200° F. In countercurrent operation the temperature may be considerably higher, usually ranging between 225° and 285° F.

There have been some efforts at recovering heat and reducing dust loss by using air from the cooler as combustion air in the dryer. Some trouble was reported in connection with this from decomposition of dust in the burner.

In some granulation plants the dryer is omitted. Such plants usually have a rotary continuous ammoniator and a rotary cooler. Water input is limited by using nitrogen solutions of low water content, or anhydrous ammonia, or combinations of these materials. The formulations selected develop enough heat of reaction for drying. Much of the moisture content of the granules is evaporated in the cooler.

There is considerable difference of opinion in the industry as to the advisability of omitting the dryer. Some plants seem to do well without one. Others, however, have had so much trouble that they have given up and installed a dryer. From the economic viewpoint, formulation cost is increased by operating without a dryer, since more acid must be used to produce the increased heat of reaction needed for drying. Moreover, the extra acid dilutes the product and necessitates a higher proportion of concentrated superphosphate. Most of the producers that do not have dryers use from 190 to 230 pounds of sulfuric acid (66° Bé.) per ton for 5-20-20, and 200 to 275 pounds for 12-12-12, a considerably higher level than that used in most plants that have dryers. The increase in formulation cost varies considerably from area to area because of differences in cost of the raw materials. Costs that appear applicable in many areas of the Midwest are, per ton: acid (66° Bé.), \$23; normal superphosphate (20% P₂O₅), \$20; and triple superphosphate (46% P₂O₅), \$60. On this basis, estimates indicate that the increase in formulation cost in operating without a dryer would range from \$0.60 to \$0.75 per ton for

the lower level of acid use listed above, and \$1.50 to \$1.80 for the higher level. Drying costs are estimated to be about \$0.60 per ton for a large plant (40,000 tons per year) and \$1.00 for a smaller one (15,000 tons). From this it is evident that if the higher level of acid is used for comparison, a dryer should be quite economical in a large plant (70% or more return on \$50,000 investment). For the small plant the return is much lower but still appears attractive. If the lower level of acid is used, however, the dryer would show an operating loss in the small plant and little or no return in the larger one.

Since the amount of acid used in operating without a dryer is such an important consideration, an effort was made to determine whether the high level is really necessary. Successful operation at the lower acid level, yielding products with good physical properties, was observed in the present survey. This indicates that a dryer is not desirable except in cases of unusually high acid cost or low differential between ordinary and concentrated superphosphate costs. There are additional factors involved, however, which are somewhat difficult to evaluate. For example, operation without a dryer involves (1) closer attention and greater skill of operators in accomplishing three separate operations—ammoniation, granulation, and drying—in the same piece of equipment; (2) a probable increase in fume because of the higher temperature in the ammoniator; and (3) difficulty in reducing the moisture content of some grades—mainly those of low analysis—to an acceptable level. On the other hand, (1) formulations that are used in plants that do not have a dryer usually contain less ammonium nitrate and the products are less hygroscopic; (2) elimination of the dryer reduces the amount of material in process, thereby facilitating change from one grade to another and reducing starting-up and shutting-down time; and (3) the dust loss, evolution of fume, and fines formation associated with drying are avoided.

Cooling

Practically all granulation plants have coolers, even if they have no dryers. In most plants the cooler is a little smaller than the dryer. Many plants have trouble due to insufficient cooling, either because of underdesign of a new unit or because the cooler is a second-hand unit designed for some other operation. Inadequate cooling capacity seems to be one of the most widespread problems in the industry.

Most coolers are of the simple rotary-drum type with countercurrent flow of air and solids. A few plants have units of special design, involving such features as cross flow, cocurrent flow at feed end, and combination of drying and cooling in the same unit.

Screening

Most plants are equipped with standard double-deck screens. The major exception, as practiced in some of the batch-mixer, counter-current-dryer plants, is use of a trommel-type screen attached to the end of the cooler to reject oversize. No screen is used for fines in this case, dependence being placed on semifusion in the dryer and the use of a high air rate through the cooler and dryer to remove fines.

Screen mesh sizes vary widely. The size adopted usually represents a compromise between the desire to maintain a high production rate and the need for producing an attractive, closely sized product. Some producers vary particle size with the season. When the demand for fertilizer is low, they size closely to make a more attractive product; when it is high, as in the spring season, they broaden the size range to get more production, or even omit the fine screen and produce a semigranular product.

The results of a survey of screen sizes used in a typical group of plants are shown in Table VI. The most popular particle size range is 6 to 20 mesh, which gives a good production rate and is well received by customers in most areas. However, there are several plants which size closer than this for competitive reasons. The closest sizing found was between 8 and 12 mesh. There appears to be a general trend to close sizing, especially in those areas where there is competition with the closely sized products made in slurry processes.

A variable sizing practice is used in a few plants, to control either production or granulation. For the former different sizes of screens are used according to the grade being produced. A wider size range is used for low-nitrogen grades—which have less tendency to cake—and the production rates of these grades are thereby increased. Varying of size to control granulation is practiced in some of the plants that control recycle rate. When more or less recycle is required, the size range is increased or decreased to provide the proper amount of fines. Size range may be changed by an arrangement at the screen that allows either diverting the coarser part of the fines to the product or the

finer part of the onsize into the recycle.

The product is normally screened after the cooler. However, a few plants screen just after the dryer and in one plant (24) wet oversize is screened out before the dryer and recycled to the granulator. When the screening is carried out after the dryer, the fines are recycled while still hot. The purpose is to economize on fuel, increase the capacity of the cooler, and increase the temperature level in the ammoniator. Use of hot recycle is especially helpful in making low-nitrogen grades, since the necessary temperature level in the ammoniator is relatively difficult to obtain. For high-nitrogen grades, hot recycle normally is undesirable, since there is adequate heat of reaction in the ammoniator. Considerable difference of opinion was found as to the feasibility of screening before cooling. Difficulties encountered include blinding of screens, inadequate crushing of hot oversize, and condensation of moisture in recycle hoppers and conveyors. However, some plants reported satisfactory operation.

Another variation is screening out oversize before cooling, after which it is crushed and recycled to the dryer. Much of the crushed oversize is in the proper size range and normally passes into the product. When the oversize is screened out and crushed after the cooler, as in normal practice, the centers of the large particles may be incompletely dried and therefore unsuitable as product. Moreover, the onsize from crushed oversize tends to have a jagged, irregular surface rather than the smooth rounded shape desired. Screening after the dryer and

redrying the crushed oversize helps in these problems. In the usual installation, in which all screening is done after the cooler, some help in this problem can be obtained by recycling the crushed oversize through the cooler. This gives some further drying and tends to smooth the surfaces of the particles. Crushed oversize in batch mixer plants usually is recycled to the mixer without any screening.

Although these variations in screening practice are advantageous and feasible, many of them involve some additional equipment and are found in relatively few plants. Most plants follow the standard flowsheets shown in Figure 3.

The general problem of fines recycling, or of disposing of excess fines produced during start-up, is avoided in some instances by selling the fines separately as nongranular material. The main objection to this practice is that the fines tend to differ in composition from the onsize product.

Dust and Fume

Probably the most pressing problem related to granulation is atmospheric pollution resulting from dust and fume emission. This is a particularly serious problem for plants near residential areas.

The distinction between dust and fume is important and sometimes not fully appreciated. "Dust" is mechanically entrained small particles of fertilizer materials that become suspended in the air in handling or in passage through the dryer or cooler. "Fume" consists of much smaller particles. It may form in the ammoniator by chemical reaction of hydrochloric acid and ammonia in the vapor phase. Fume also may be formed by vaporization in the dryer of certain volatile salts at high temperature, followed by condensation.

Dust control is relatively simple. The dust cyclones in most plants are fairly effective. Any dust escaping from these units may be collected in a simple water scrubber.

Fume is very difficult to collect. Cyclone collectors and simple water scrubbers are not very effective, and equipment that will collect fume efficiently is very expensive. One installation that was reported to remove fume effectively was described at the Fertilizer Round Table meeting in 1956. This was a water scrubbing unit in which a large number of spray nozzles were installed. The nozzles operated at a water pressure of 500 p.s.i. The high pressure was said to be quite important in obtaining a high efficiency. Other types of scrubbers have been reported to give good total

Table VI. Particle Size of Granular Fertilizer Produced in a Typical Group of Plants

Screen mesh (Tyler)	Number of plants
3-14	1
4-12	1
4-20	6
5-12	1
5-14	1
5-20	1
5-30	1
6-10	1
6-12	2
6-14	4
6-16	4
6-18	1
6-20	10
6-30	1
8-12	1

efficiency for fume and dust removal, but the available data do not indicate the effectiveness for fume removal as distinguished from dust removal.

In view of the difficulty and expense of fume collection, prevention of fume appears to be the most promising approach. Fume formed in ammoniators is generally thought to consist principally of ammonium chloride. This opinion is confirmed by analyses, which show a large percentage of ammonium chloride. The usual explanation of the cause of fume formation is that sulfuric acid reacts with potassium chloride to form gaseous hydrogen chloride, which then reacts with gaseous ammonia to form ammonium chloride.

The feasibility of avoiding fume formation in the ammoniator has been demonstrated in the TVA pilot plant and in some commercial plants, although it is not always clear what factors were important in preventing fume. In the authors' opinion, the primary causes of fuming in the ammoniator, in the approximate order of importance are (1) poor control of granulation with consequent overgranulation or "mud" formation; (2) poor distribution of acid due to either poor design, improper placement, or corrosion of the distributor; (3) overloading or underdesign of the ammoniator; and (4) unsuitable formulations.

Even distribution of sulfuric acid is an important factor and depends on maintaining a properly designed acid distributor in a good state of repair. This may be difficult because distributors corrode rapidly. The usual black iron distributors are often in poor condition and discharge too much acid in some spots.

Conditions that lead to fuming in the ammoniator are also likely to cause poor absorption of ammonia. The combination of ammonia loss and ammonium chloride loss may cause a serious shortage of nitrogen in the product. The widespread nature of this problem is illustrated by the prevalence of nitrogen deficiencies in 1:1:1 ratio fertilizers, as shown by published reports of analyses by state chemists. These nitrogen deficiencies occurred most frequently in 10-10-10 and 12-12-12 grades. In several states, about two-thirds of the samples of these grades contained less than the guaranteed nitrogen content, and in about half of the samples the nitrogen deficiency exceeded the tolerance allowed by state law. (In arriving at these figures, fertilizers made by nitric phosphate or ammonium phosphate processes were excluded; in these materials, nitrogen deficiencies were relatively rare.)

Several methods for decreasing or

eliminating fuming in the ammoniator, other than careful control, have been tried or suggested. One method is to neutralize the sulfuric acid before it comes in contact with the potash. This has been done by adding the potash near the discharge end of the ammoniator, at which point the sulfuric acid already has been neutralized. This arrangement is reported to give fume-free operation and satisfactory granulation. However, the method would entail major alterations in most plants.

Some companies reported experiments with addition of the sulfuric acid to the ammonia or ammoniating solution in the ammonia distributor. Very good operation was obtained, but corrosion of the usual type of distributor was severe. The method is not known to be in commercial use. Neutralization outside the ammoniator is carried out in at least one plant (6), but this practice presents some difficulty in retaining the heat of neutralization, which is needed for granulation of some grades, and in distributing the thick ammonium sulfate or ammonium sulfate-nitrate slurry in the ammoniator.

Decreasing the concentration of sulfuric acid, usually from 66° to 60° Bé., is of some help in avoiding fume. However, dilution adds to the water content of the formulation, which complicates the production of high-nitrogen grades.

Formulation without sulfuric acid is one means of avoiding fume. Apparently no fume from the ammoniator has been encountered when using phosphoric acid. One company reported use of phosphoric rather than sulfuric acid for the sole purpose of decreasing fume problems. This method may be attractive for some grades when phosphoric acid is not too expensive. Since the use of phosphoric acid introduces more water into the system, however, some complications might occur in production of high-nitrogen grades.

Elimination of sulfuric acid from formulations for 1:4:4 ratios by using ammoniating solutions plus steam to supply the necessary heat—rather than anhydrous ammonia and sulfuric acid—has been successful in some plants.

If the fume originates in the dryer, an obvious means of preventing it is to avoid excessive temperatures in the dryer. However, in those plants in which granulation is obtained in a countercurrently fired dryer, lowering the temperature may preclude good granulation. Bridger (3) has discussed nitrogen losses in drying mixed fertilizers. Some mixtures lost 8 to 14% of their nitrogen when dried at a product temperature of 200° F.

Other mixtures did not lose an appreciable amount of nitrogen at temperatures as high as 280° F.

Physical Condition of Product

Physical condition of the product is a serious problem in many plants, even though the main purpose of granulation is to improve condition. The increased proportion of hygroscopic materials, ammonium nitrate or urea, in granular fertilizer formulations may offset the advantage gained by granulation. Also, it is likely that farmers are less tolerant of poor condition in granular than in nongranular fertilizer, especially if they pay a premium for the granular material. However, the widespread demand for granular fertilizers indicates that their physical properties are in general quite good.

Factors affecting the physical condition of granular fertilizers include moisture content, chemical composition, curing time, particle size and shape, amount and type of coating agent (if any), climatic conditions, type of bag, and duration and conditions of storage. Several articles have discussed these factors (13, 20, 30).

Moisture content is generally regarded as the most important factor affecting caking properties. Most manufacturers aim at 1% or less moisture in granular fertilizers of 1:1:1 ratio and less than 3% in those of 1:4:4 ratio. These levels seem to be generally satisfactory, although there are exceptions. A somewhat higher moisture level may not result in poor condition of some grades if other factors are favorable. For instance, the addition of about 2% of a good conditioner or coating agent may offset any caking tendency caused by a slightly higher moisture content. On the other hand, very high-analysis 1:1:1 ratios may cake even at 1% moisture if they are not conditioned.

Few producers use a coating agent to condition granular fertilizer. Some use of coating agents on high-analysis grades was noted.

The high temperature in some dryers partially fuses the granules and as a result a very hard surface is obtained upon cooling. This is said to give improved condition at any given moisture level, but no confirming quantitative data appear to be available.

Many producers limit the ammonium nitrate or urea content of their products in an effort to avoid caking in storage or poor drillability due to moisture absorption during use. This practice is more prevalent in the more humid areas. Others rely on thorough drying and use of moisture-resistant bags to get the product to the user in good condition.

Product Composition

Keeping the product on grade is a problem in many plants. Perhaps the most serious problem of this type is the frequent shortage of nitrogen in 1:1:1 ratios as discussed previously.

Some producers have difficulty with high-potash grades such as 5-20-20 because of non-uniformity of analysis. If coarse potash is used, the fines are low in potash and high in other ingredients. If fine or nongranular potash is used, the fines are high in potash. In either case, segregation of fine and coarse fractions of the product in the storage pile may cause some shipments to be off-grade. In those plants which produce a closely sized product, variations in analysis due to segregation are usually not important. When the size range is wide the problem may be acute, especially in the case of semi-granular products.

Operating Hazards

Fires in storage piles have long been a hazard in the industry; however, granulation has brought with it a process hazard known as the "flash fire." This is a burning of gases over the mix in the ammoniator; the fire usually goes out when the liquid feed is turned off. The main contributing factors are the use of acid to develop a high temperature level in the ammoniator and the high nitrate content in many mixtures. The fires seldom have had serious consequences, but the potential hazard is a source of concern to plant operators.

The mechanism of ignition and burning is not known. However, the causes of the fires seem to be well established. The general consensus is that faulty operation of equipment is the prime cause and that with good operation any normal combination of granulating conditions can be used with safety. Specific contributing factors are:

- Accidental stoppage of solid feed, which permits raw acid and ammoniating solution to come together without the diluent effect of the solids.
- High local concentration of acid caused by failure of the acid distributor, increase in size of the holes in the distributor, or plugging of the distributor holes.
- Inadequate operation of fume exhaust system.
- Use of high concentrations of sulfuric acid. Those using 78% acid reported less trouble than those using 92%. However, the majority of producers use the latter, many

without any difficulty. Mild explosions, or "puffs," have occurred in some instances, especially with use of 98% acid.

Fires are normally associated with increase in bed plasticity, which interferes with proper rolling action of the bed and consequently results in poor reaction of ammonia with superphosphate. Excess fuming normally results. The fumes are generally orange in color and appear to be easily ignited.

The use of urea rather than ammonium nitrate in ammoniating solutions is claimed to reduce or eliminate the fire hazard.

Perrine (27) has treated the subject of ammoniator fires at length.

Fires also occur in dryers and in storage. Caked material in dryers has been known to catch fire and burn itself out. Overheated lumps of material appear to be a special hazard (9). Apparently continuing decomposition inside a large lump raises the temperature to the ignition point.

Overheated lumps in the product appear to contribute also to the formation of fumes, which are sometimes an operating hazard as well as a public nuisance. Lumpy material has been reported to give off chlorine or a chlorine compound.

Plant Capacity

Capacities of most granulation plants range from 10 to 40 tons per hour, depending on equipment size, degree of drying, closeness of sizing, amount of solid nitrogen used, and other factors. Batch mixer plants generally produce at the 10-tons-per-hour level; most continuous mixer plants can produce 20 to 30 tons per hour. Rates as high as 40 tons per hour are rare, and normally are attained only with low-nitrogen grades which do not have to be dried to a low moisture level.

Granulation Costs

Investment. A great many granulation units have been installed in existing plants; for these the conversion cost has depended largely on how much of the old equipment was retained. Many have retained the weighing equipment and the batch mixer, using the latter to pre-mix the dry materials before feeding to a continuous ammoniator. The least expensive installation, however, is that in which the batch mixer is retained as the ammoniator. Plants of this type, in which the added equipment consists of surge hopper, feeder, dryer,

cooler, and cyclones, have cost from \$30,000 to \$50,000 installed.

At the other extreme is a completely new plant of the largest size. Plants of this type have cost from \$225,000 (5) to \$357,000 (12). A complete breakdown of construction costs has been published for one of the plants (2).

Operating Cost. Stanfield (35) has estimated operating cost in a large granulation plant at \$4.63 per ton of 10-10-10 fertilizer, exclusive of raw materials and bagging cost. The main difference in comparison with a plant making powdered fertilizer is in the cost of operating the dryer, cooler, and screens. In addition, the powdered goods plant can be operated at a somewhat higher production rate. A rough estimate indicates about \$0.90 per ton additional manufacturing cost for granulation in a 40,000-tons-per-year plant, exclusive of raw materials cost. It should be noted that this is on the basis of an equal degree of mechanization in common parts of the plants. Many of the older powdered goods plants are not as well mechanized as granulation plants, which are all relatively new. For this reason the operating cost in a new granulation plant may be no higher than that in an old powdered goods plant.

Granulation plants usually use quite different formulations from those in powdered fertilizer plants. Formulations for granular fertilizers are designed to facilitate granulation, whereas formulations for powdered goods are designed to obtain satisfactory physical condition without granulation. The difference in formulation cost is quite important and varies with the grade. Some of the factors which enter into the comparison are:

- Dryers and coolers are not used in most powdered fertilizer plants. Therefore, ammoniation must be kept at a lower level in these plants because otherwise the warm, moist product would tend to undergo P_2O_5 reversion in the pile.
- When high-nitrogen grades are made in nongranular form, only limited use can be made of nitrogen solution, and relatively large amounts of ammonium sulfate are used. This makes the formulation cost higher for nongranular fertilizers in the high-nitrogen grades.
- When low-nitrogen grades are made in nongranular form, little or no ammonium sulfate is used and sulfuric acid is not required. When made in granular form, sulfuric acid is usually used. Therefore, the nongranular formulation cost usually is lower for low-nitrogen grades.

The effect of these factors is shown in:

Table VII. Both formulation and operating costs are higher for granular 5-20-20 than for nongranular 5-20-20, so this grade is more expensive to produce in granular form. In the case of 10-10-10, the lower formulation cost more than offsets the higher operating cost, so this grade can be produced more economically in granular form than in nongranular.

The cost of producing granular 5-20-20 could be reduced somewhat by using a cheaper formulation such as solution plus anhydrous ammonia without acid. However, this type of formulation is little used in industry, even though it gave good results in the TVA pilot plant. The general tendency in industry is to add some acid in making low-nitrogen grades, no matter what the source of nitrogen. It should be noted that although the granular 5-20-20 shows a cost disadvantage, it has the advantage of better physical condition.

It should be emphasized that any cost disadvantage in granular production is offset to some extent by the fact that granulation normally allows an increase in grade for any particular plant food ratio. Thus, the cost per ton of plant food in granular products may compare much more favorably than the cost per ton of product. The main disadvantage for granulation is in production of low-analysis grades—4-16-16 for example—which have to be granulated for competitive reasons.

For grades containing more than 10

units of nitrogen, common practice is to use some ammonium sulfate. The cost of formulation can be reduced by using solutions only, but the larger amount of water and ammonium nitrate introduced makes the mix sticky, and cuts production by requiring a higher recycle rate. Available information indicates that for 12-12-12, production would be cut by about half if no sulfate were used. The producer therefore has to choose between a larger plant to maintain production, and use of a higher-cost formula. Table VIII gives the order of magnitude of the saving to be expected by using all solution. The indicated saving is almost \$2.50 per ton, from which would be subtracted any additional cost for operating larger ammoniating, drying, and cooling units. Some net advantage is indicated, but the total saving would depend on the tonnage of grades containing more than 10 units of nitrogen. Because of insufficient tonnage of these grades, or perhaps because of lack of additional capital, the great majority of producers have chosen high production rate with ammonium sulfate rather than low formulation cost without it.

Agronomic Considerations

A considerable amount of work was done in the early days of granulation on the relative agronomic value of granular and nongranular fertilizers. Much of this was reported in the

period 1935-38 (36). The work was generally inconclusive. In many cases no differences were found; a few superior results were reported for granules when mixed with the soil, but occasional decreases in yield were reported with localized application.

Modern research on the problem has associated water solubility of the phosphorus with the effect of particle size. For example, Terman *et al.* (38) found a pronounced interaction between water solubility and granule size on acid-to-neutral soils. For early growth response, the smaller granules gave better results for low water-soluble fertilizers, but the larger granules did better when water solubility of the phosphate was high. This was corroborated by Lawton *et al.* (21), who tested 12-12-12 fertilizers containing various proportions of water-soluble phosphate on soils of varying pH. When the fertilizer was mixed with the soil, granules were inferior to powder when water solubility was low and superior when it was high. In banded placements, response was about the same. It was concluded that (1) all granular material should contain 40 to 60% of the phosphate in the water-soluble form, (2) powdered material should have the same restriction if banded, and (3) if powdered fertilizer is mixed with the soil, water solubility is not important. It may be concluded from this that producers of granular fertilizer should be more concerned with water solubility than those who make the powdered type.

When ordinary superphosphate is the source of phosphorus in mixed fertilizers, water solubility of the phosphorus varies inversely with the degree of ammoniation. In the past few years the degree of ammoniation has increased significantly in both powdered and granular products. The increase has been most pronounced in granulation plants, however, since the dried, cooled product can hold more ammonia without serious P_2O_5 reversion. As has been noted, the general level of ammoniation is 5 to 6 lb. per unit of P_2O_5 for ordinary superphosphate and 3 to 4 lb. for concentrated superphosphate. Hignett (16) has shown that at these levels the respective water solubilities of ammoniated ordinary and concentrated superphosphate are 25 to 35% and about 50%. On this basis, the water solubility of granular fertilizers produced by current methods appears to be fairly close to Lawton's minimum (21).

The practice in some plants of over-ammoniating and accepting some loss to get a high degree of ammonia retention may present a problem in water solubility. As much as 7 to 8

Table VII. Comparison of Production Cost for Granular and Nongranular Fertilizer

Material ^a	Amount, Lb./Ton Product		Cost, \$/Ton of Product	
	Granular	Non-granular	Granular	Non-granular
5-20-20				
Nitrogen solution		146		3.87
Anhydrous ammonia	125	25	5.60	1.10
Ammonium sulfate		100		1.90
Sulfuric acid (66° Bé.)	140		1.61	
			7.21	6.87
	Advantage for nongranular		\$0.34	
10-10-10				
Nitrogen solution	488	150	12.90	3.97
Ammonium sulfate		690		13.10
Ordinary superphosphate	1020	659	10.20	6.59
Concentrated superphosphate		158		4.74
Sulfuric acid (66° Bé.)	158		1.82	
			24.92	28.40
	Advantage for granular		\$3.48	
Net advantage ^b				
5-20-20	\$1.24 for nongranular			
10-10-10	2.58 for granular			

^a Only those materials included for which there is a difference in amount used. Costs assumed, per ton: solution (41% N; 22.2% NH_3), \$53; anhydrous ammonia, \$90; sulfuric acid, \$23; ammonium sulfate, \$38; ordinary superphosphate \$20; and concentrated superphosphate \$60. These appear typical for many sections of the Midwest.

^b Assuming \$0.90 operating cost for granulation.

pounds of ammonia is retained by this practice, and, according to Hignett's data, this would give a water solubility of less than 20% when ordinary superphosphate is the source of phosphate. For high-analysis grades, however, water solubility may be somewhat higher. These grades contain some concentrated superphosphate, and solubility of the latter increases at high degrees of ammoniation.

New Developments

Granulation is a relatively new practice, and improvements are constantly being made. Some of these have already been discussed in connection with current problems. Further innovations in granulation techniques are likely to be adopted in the future.

There is a distinct possibility that standard granulation plants will be adapted for production of grades and use of processes which up to now have been restricted to large slurry-type plants. The main products involved are ammonium phosphate-sulfate, nitric phosphate, and ammonium phosphate-nitrate. Work at TVA (37) has shown that the latter two products can be made economically in standard granulation plants equipped with TVA-type ammoniators. D. M. Weatherly Co. (7) is also applying the rotary drum ammoniator to production of ammonium phosphate-sulfate.

Another development is use of highly concentrated furnace phosphoric acid (76% P₂O₅) in granulation processes. This acid, developed at TVA, shows promise of improving granulation by supplying more heat of reaction and producing a drier product.

A further possibility for rotary drum ammoniators is a one-step process for producing granular superphosphate. Phosphate rock and acid are fed to the ammoniator, and acidulation and granulation take place in the same piece of equipment.

An integrated process for producing superphosphate and granular fertilizer in a continuous operation has shown some promise in tests at TVA (41). By the use of more acid and finer rock than is normally used the phosphate rock is sufficiently converted in an hour or so to allow immediate use in the granulation process. Standard denning procedures are used in the acidulation.

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Table VIII. Formulation Cost for 12-12-12

Material	Amount		Cost, ^a	
	Lb./Ton of Product With sulfate	Without sulfate	\$/Ton of Product With sulfate	Without sulfate
Ammoniating solution	494	649 ^b	13.10	15.50
Ammonium sulfate	195	...	3.70	
Ordinary superphosphate	537	729	5.37	7.29
Concentrated superphosphate	300	207	9.00	6.21
Potassium chloride	400	400	7.60	7.60
Sulfuric acid (66° Bé.)	162	138	1.86	1.59
			40.63	38.19

^a Costs assumed, per ton: solution (41% N; 22.2% NH₃), \$53; ammonium sulfate, \$38; ordinary superphosphate \$20; concentrated superphosphate, \$60; potassium chloride, \$38; and sulfuric acid, \$23. These appear typical for many sections of the Midwest.

^b 37% N (16.6% NH₃); cost per unit of N assumed to be same as for 41% N solution.

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