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THE SWIFT PROCESS of GRANULATION

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THIS paper describes a system of producing granular mixed fertilizer. The process differs in many important characteristics from those with which most manufacturers are familiar. The process was developed by Swift & Co. and is now used by it to produce both semi-granular and granular fertilizer. Blaw-Knox Co., Chemical Plants Division, has been licensed by Swift to design, fabricate, and erect equipment to produce fertilizer by this process, and Blaw-Knox is the licensing agent.

The research laboratories of Swift & Co.'s Plant Food Division several years ago* embarked on a program of research with the objective of developing a process and equipment with the following characteristics:

A. High rate of production.
B. Low investment cost.
C. Low labor cost.
D. Low raw material cost.
E. Production of high analysis grades.
F. Complete flexibility in the use of liquid and gaseous raw materials interchangeably with solid raw materials.
G. Use of the heat of reaction to produce a dry product.
H. Virtually complete elimination of nitrogen loss from gaseous ammonia or liquid nitrogen solutions.
I. Improved mechanical condition of the product.
J. Granulation of high nitrogen grades without drying.

Process And Equipment

THE Swift granulation process is adaptable to operation with a batch weighing system. This involves batch weighing, screening, and pre-mixing of the solid raw materials, and is the manner in which the process has been operated. Solid raw materials may also be supplied by continuous weighing, which would eliminate the need for pre-mixing. With the batch weighing system illustrated, dry materials are weighed and discharged to a batch hopper on a definite time cycle. This hopper feeds the two ton batch mixer on the same cycle, and each batch is discharged to the surge hopper. The feed screw to the Swift reactor is equipped with a variable speed drive. The feed screw speed is adjusted so that the pre-mixed material level in the surge hopper is maintained within minimum and maximum limits. In this way, the rate is controlled by the frequency of the preparation of batches, and a constant flow of pre-mixed dry materials is maintained to the reactor.

Liquid raw materials are supplied by pumping or by pressurized storage tanks, and are fed to the reactor through individual rotameters. The reactor is vented through a vapor stack, and the product discharged to storage or to drying, depending on the type of product being made.

The reactor is a rotating shell, 38 feet long by 5½ feet in diameter, set on a 3.75° slope (pitch of 13/16 inch/foot). The speed of rotation is 5.5 rpm. The first 5 foot section of the tube is equipped with spiral flights, which move the dry material rapidly into the lifting flight zone. The lifting flights are 24" long by 8" deep, and 12 are spaced around the inside circumference. The flight pattern is repeated at a 13° stagger to within 5 feet of the tube outlet.

The liquid feed discharges through nozzles directed toward the showering material, in the first 10 feet of the reactor. Hammers are provided to insure that no material sticks to the shell. Vapor is vented from the hooded discharge end by means of a gravity stack through the roof. Vapor tightness at the inlet end of the reactor is accomplished by overlapping the head ring with a stationary head sheet.

When sulfuric or phosphoric acid is reacted with ammonia in mixing fertilizer, a large volume of steam is developed. In a batch mixer, the escaping steam carries with it much of the unreacted ammonia vapor, resulting in substantial losses of nitrogen and local nuisance effects. In the Swift Process, ammonia that does not react in the feed zone travels the entire length of the reactor through a continuous curtain of falling solids, resulting in practically complete absorption of ammonia by the time the vapors reach the exit of the unit. Reaction of ammonia with superphosphates is promoted by the presence of water, and it is believed that the presence of water vapor is a factor in the high absorption efficiency. The spray method of feeding liquids into the solids permits the evaporation of large quantities of water, and the obtaining of a solids outlet temperature of from

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*In January 1950 application was made, by Charles Davenport and Walter Horn, for a patent covering the continuous process which was developed, and in November 1952, U. S. Patent No. 2,618,5477 was granted. The process is now in operation at a number of Swift's Plant Food Factories and more units are currently being installed.

200°-250° F through the use of raw materials, which develop a high heat of reaction. This results in a much drier product at the discharge of the reactor, and permits granulation with a relatively small "solution phase," as calculated by Hardesty's method.

Formulation

GRANULATION requires a minimum of about 175-300 pounds/ton of 60° Be sulfuric acid in order to provide enough heat. This amount of acid requires 2.0-3.3 units of nitrogen from free ammonia for neutralization. With a 5-20-20 grade, the degree of ammoniation may be as low as 3.0 lbs. NH_3 per unit APA. For grades containing 6% or more nitrogen, a normal degree of ammoniation (3.50-4.25 lbs. NH_3 per unit APA) can be used. To granulate grades containing 4% or less nitrogen, with economical raw materials, the water content must be increased, and the production rate is therefore decreased for the same size drying equipment.

The mechanism of granulation in this process apparently depends on the formation of a liquid phase containing solids, which have very high solubilities at elevated temperatures. The high temperature thus increases the liquid phase far beyond what would be expected from the amount of water in the formulation. Agglomeration occurs in and immediately following the acid stage of the reactor. Hardening and drying of the granules continues through contact with hot ammonia and water vapors, as the material passes to the exit end of the reactor.

Table I shows the heat developed in the ammoniation of normal superphosphate, triple superphosphate, phosphoric acid and sulfuric acid. The first three were calculated from the data of Hardesty and Ross, as published in *Industrial and Engineering Chemistry*, 1937. The heat of reaction of ammonia and sulfuric acid was calculated from heat of formation data.

TABLE I

Heats of Reaction

Reactants	End Products	BTU'S Per Unit Of N Reacted
NH_3 + Superphosphate	3.3 lbs.N/Unit APA	35,780
NH_3 + Triple superphosphate	3.25 lbs.N/Unit APA	37,669
NH_3 + H_3PO_4	$NH_4H_2PO_4$	46,214
NH_3 + H_2SO_4	$(NH_4)_2SO_4$	71,500

Table II illustrates a formulation that can be used in the Swift Process to produce a 12-12-12 grade fertilizer. The tabulation also shows, for the raw materials used, 1) the plant food analysis, 2) the free water contained, 3) the free ammonia which is contained in (plus) or which will react with (minus) each material, and 4) the approximate heat of reaction developed. The degree of ammoniation of the superphosphate is also shown.

TABLE II

12-12-12 Formulation

Raw Material	Weight/Ton Product Lbs.	Plant Food Analysis %	Free Water Lbs.	Free NH_3 Equivalent Lbs.	Approx. Heat of Reaction Btu/Ton
NH_3	119	82.2 N	......	+119	....
Solution	50	40.8 N	9	+ 13	....
H_2SO_4 (60° Bé)	332	....	73	— 89.5	264,000
$(NH_4)_2SO_4$	593	20.5 N	......	....	....
TSP	457	46.0 APA	28	— 37.0	58,000
NSP	149	20.0 APA	9	— 5.5	8,000
KC1	400	60.0 K_2O	......	....	....
	2100		119	0	330,000

Degree of Ammoniation=3.54 lbs. NH_3/Unit APA

To calculate the heat available for the evaporation of water, the specific heat of the dry mixture is estimated at about 0.25 Btu/lb. °F, based on the data of Hardesty and Ross. If the initial temperature is 70°F, and the final temperature 230°F, the heat available for evaporating water is 330,000 Btu/ton—(1981 pounds per ton) x (0.25 Btu/lb. °F) x (230° — 70° F)—(119 pounds per ton) x (1.0 Btu/lb. °F) x (230° — 70° F) = 232,000 Btu/ton. This heat is reduced by radiation losses from the reactor, and in practice it is found that evaporation amounts to 60-100 lbs. per ton, leaving a product containing 1% to 3% moisture.

Table III illustrates a formulation for making granular 5-20-20 by the Swift Process, with the same data. Deducting the sensible heat of solids and water in raising the temperature from 70°F to 230°F leaves 143,000 Btu/ton available for evaporation. About the same amount of water (60 to 100 lbs./ton) is evaporated, also leaving 1% to 3% moisture in the product.

Semi-granular material can be made over a wide range of grades such as 5-10-10, 5-20-20, 12-12-12, 10-20-10, etc., with economical raw materials.

For a very high analysis material, or when prices are favorable, phosphoric acid may be substituted for sulfuric acid, but it is necessary to use a stainless steel reactor to avoid corrosion. The use of phosphoric acid is well worked out since this material has frequently been used when triple superphosphate was unavailable or too high in cost.

Operation

THE process is convenient to operate, since it is normally put on stream in 10 minutes. This permits the operation of the process on a non-continuous basis, that is, the system may be operated for as many hours per day as are necessary to meet production requirements, and a variety of grades may be produced during one operating day.

TABLE III
5-20-20 Formulation

Raw Material	Weight/Ton Product Lbs.	Plant Food Analysis %	Water Free Lbs.	Equivalent Free NH_3 Lbs.	Approx. Heat of Reaction Btu/Ton
NH_3	97	82.2 N	......	+97	....
Solution	50	40.8 N	9	+13	....
H_2SO_4 (60° Bé)	185	....	41	—50	148,000
TSP	736	45.0 APA	44	—49.7	77,000
NSP	345	20.0 APA	21	—10.3	15,000
KCl	667	60.0 K_2O	......	....	....
	2080		115	0	240,000

Degree of Ammoniation=3.0 lbs. NH_3/Unit APA

The granules, as produced in the reactor, are hard and will retain their identity very well if the material is cooled (without drying) prior to stockpiling. Some evaporation takes place in cooling, of course. However, this method is not as reliable as drying, since the results vary somewhat with the conditions of production and the atmospheric humidity, temperature, etc.

In producing granular fertilizer by this process, the best method to assure the complete absence of caking is drying the material to 1% or less moisture content. In this case, no cooling is necessary, unless it is desired to bag or otherwise manually handle the material as it is produced. No caking results when the material is put in storage at a temperature of 200°F. This system also offers the option of using the dryer as a cooler by cutting off the fuel supply, and saving fuel costs when conditions are favorable for granulation.

Production Rates

USING the Swift Reactor only, without succeeding drying, cooling, or screening equipment, a production rate of 60 tons per hour may be obtained.

Using the Swift Reactor, followed by a cooler, a production rate of 60 tons per hour is also possible, depending on the cooler capacity and the required maintenance of granular form in storage.

For production of a completely granular fertilizer, the equipment consists of the reactor, dryer, screens, oversize crushing, and recycle conveying equipment. The factors limiting production are the capacities of the dryer and screen. Blaw-Knox is prepared to supply standardized units for the production of 15 or 30 tons per hour of —6 +20 mesh granular goods.

Products

Screen analysis of a sample of 12-12-12 taken at the reactor discharge shows:

Mesh Size	%
+ 4	20.8
— 4 +20	61.8
—20 +40	15.2
—40	2.2

which would certainly be satisfactory for sale in many areas without screening.

TABLE IV
Screen Analyses

Mesh Size	12-12-12 % (A)	12-12-12 % (B)	5-20-20 % (A)	5-20-20 % (B)
+ 6	5.8	1.8	1.0	0.5
— 6 +10	55.9	31.9	38.0	37.2
—10 +20	35.8	46.5	46.1	48.8
—20 +40	2.3	19.2	13.6	11.6
—40	0.2	0.6	1.3	1.9

Table IV shows two typical analyses of each of two products after screening, when made by the formulations given, and shows variations due to local operating conditions. Screen sizes are 5 mesh and 20 mesh. The amount of material recycled to obtain the particle size distribution shown may vary from 5% to 20%. Screening efficiency is low in some cases, since the production units are being operated at rates exceeding 250% of design capacity at times.

Ordinary 3-ply paper bags (without water-proof layers) are used, and no caking is experienced when granular material is made by drying to 1% moisture or less.

Operating Costs

WHEN this process is operated to produce a semi-granular material without drying and screening equipment as previously described, savings are made in operating costs over a batch system producing a pulverized material. A 2 ton batch mixer system has a capacity of 30 to 35 tons per hour; the same system with the addition of a Swift Reactor has a capacity of 60 tons per hour with the same number of operators.

Operating costs for the full granulation system compare with other granulation systems as follows:

1. Fuel—The cost of fuel for drying the product is greatly reduced since no sensible heat need be supplied to the fertilizer material, and the water to be evaporated is normally only 1% to 3% of the weight of the product.
2. Power—With the elimination of the cooler and its exhaust fan, the reduction in size of the dryer shell and exhaust fan capacity, and the reduction in the total number of pieces of operating equipment, the power costs are lower.
3. Maintenance — Maintenance cost is decreased not only by the reduction in the total number of pieces of equipment, but also in the elimination of the distributor for liquid which operates beneath a bed of material in the TVA process.
4. Labor—Labor costs for weighing and batch mixing are typical of the type installation which may be used. Operation of the ammoniation, granulation, and drying equipment does not require the full time attention of one man.
5. Royalties—When used by producers other than those who own the patent, there is an additional expense for royalty payments. The licensor has fixed this cost at

30¢ per ton of material produced for a period of 5 years.

Investment and Material Costs

THE Swift Reactor with only surge bin, feeder, and liquid flow indicators, can be furnished for about $25,000. Most manufacturers who are not now producing granular fertilizer will also require sulfuric acid and anhydrous ammonia unloading and storage equipment and possibly also nitrogen solution unloading and storage equipment.

Many combinations and variations of equipment are possible, depending on the degree of granulation desired and local conditions. Between the limits stated, a wide variety of costs of installation may be found.

The cost of materials will depend on location and may be easily calculated for granular 12-12-12 and 5-20-20 from the formulations previously given. Generally speaking, material costs are lower than for batch produced pulverized fertilizer, but in some cases are higher than other granulation processes, such as those using the TVA ammoniator. For many producers, these higher material costs and the royalty payments will be more than offset by the high production rate and flexibility of operation, the savings in nitrogen loss, and investment, operating, and bag costs.

Process Advantages

Semi-Granular

The advantages of this process using the Swift Reactor only, as compared with conventional fertilizer mixing, are a higher production rate with the same labor requirements, the use of lower cost raw materials, a lower nitrogen loss, lower moisture content in the product, less reversion of P_2O_5 and the production of a semi-granular fertilizer with the product particle size as high as 75% within the —4 +40 mesh range.

Granular

The Swift process for the continuous production of a granular mixed fertilizer provides:

1. Low investment cost.
2. High production rate.
3. Low rate material costs:
   Use of anhydrous ammonia and nitrogen solution, in place production of ammonium sulfate and/or ammonium phosphate, and low nitrogen loss.
4. Low operating cost:
   Labor, maintenance, fuel and power costs are all lower than in other comparable granulation systems.
5. Superior product condition:
   No caking of granular material, and use of non-water-proof bags.
6. Flexibility of operation:
   Semi-granular or complete granular production, and quick start-up and change of grades.
7. Proven operation:
   The process has been commercially proven with 4 plants currently in operation, more under construction, and more than 1 million tons of product sold to date.★★

Granulation of Mixed Fertilizers— Theory vs Practice

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IN attempting to incorporate the most desirable qualities in a granular mixed fertilizer, the manufacturer of the product is faced with the problem of bridging a wide gap between theory and practice. Theory, in this instance, is defined as an analysis of a given set of factors in their ideal relationships to one another. The ideal may often be at variance with practice, which is actual performance of the process or the product. However, practice is seldom improved without aiming, at least, in the direction of the ideal. For the purpose of discussion in this article, the following specifications are the author's conception of the *ideal* properties for a granular mixed fertilizer:

1. *Particle size;* 10-14 Tyler Standard mesh (screen openings 1.651-1.168 mm.)
2. *Particle shape;* spherical.
3. *Particle structure;* 95% of the particles to remain intact under a load equivalent to 100 lb./sq. in.
4. *Homogeneity;* each granule up-to-grade.
5. *Drillability;* product to be free of lumps and to maintain its original drilling rate for 1 hour when exposed to 88% relative humidity at 76° Fahrenheit.
6. *Fertilizing efficiency;* optimum delivery of nutrients to the growing plant.

Some of these specifications are beyond the realm of practical attainment, but none of them are beyond practical approach. For this reason, it seems desirable to give them more critical consideration.

Particle size

At present, there is no generally accepted definition of the particle-size limits between which a fixed proportion of a fertilizer should fall, for the product to be classed as granular. Manufacturing convenience and the attainment of improved physical condition determine largely the particle size. Examination of 28 representative samples of typical, commercial granular mixed fertilizer *(8)* showed an average particle-size distribution as follows:

Mesh (Tyler)	%
+6	12
6—8	21
8—14	38
14—20	11
20—35	11
—35	7

It is evident that the indicated ideal particle size (10-14 mesh) is well within the particle-size range of the average granular product. The granule size of the proposed ideal product is large enough to provide insurance against setting and caking, and yet small enough to permit reasonably uniform distribution at low applications, so that each plant is provided with a proportionate share of applied nutrients. For example, since the volumes of spheres are proportional to the cubes of their diameters, one 5-mesh granule (screen opening 3.962 mm.) is equivalent to fourteen 10-mesh granules (opening 1.651 mm.); therefore, if 5-mesh granules (5,700 granules/lb.) are applied at the rate of 150 lbs./acre with grain in rows 7 inches apart, approximately 11 granules are applied per linear foot, while, with 10-mesh granules, 154 granules are applied per linear foot.

Manufacture of the ideal granular product, whereby small moistened particles of raw materials are assembled into larger compacted granules, requires that the raw materials be considerably finer than 14 mesh. This may pose a difficult problem with respect to grinding of some raw materials, but a uniform granular product can be obtained only when the size of the prepared granules is several times that of the largest particles in the ingredient materials (4).

Particle shape

It is unreasonable to expect that every particle in a granular mixed fertilizer will be spherical. However, a large proportion of granules in the average product may be spheroidal in character. As the degree of sphericity of the particle is increased, the tendency toward caking becomes less. By virtue of fewer contacts per unit mass of particles, the free-flowing quality of the product improves, and there is less particle surface to be covered when coating agents are used.

Granules produced by spraying or prilling processes, such as prilled ammonium nitrate, usually have a high degree of sphericity *(10)* but the prilling process is not applicable to most mixed fertilizers. In the production of granular mixed fertilizers, the sphericity of the granule is increased by such processing expedients as fine-grinding of initial materials and compaction into firm granules by rolling the incipient agglomerates in a rotating cylinder during their slow congelation. The latter procedure requires accurate control of moisture and temperature relationships during processing *(5)*. Another way of obtaining more uniform sphericity of particles is to recycle crushed oversize material to the initial stages of the process, rather than incorporate it directly in the product.

Particle structure

Previous investigations *(4)* have indicated that at least 95% of the particles in most granular mixed fertilizers will remain intact under a load of 100 lb./sq. in. and that this granule-strength is sufficient to withstand the stresses imposed on the product by normal handling and storage. In this respect, most present-day granular products appear to approach the ideal fairly closely.

Field experiments, comparing the movement of nutrients from granular and powdered fertilizers *(11)*, have shown no appreciable difference in the rate of solution of inorganic nitrogen from granulated or powdered forms of 5-20-5 and 5-10-5 mixtures, band-placed in contact with the soil and between strips of Monel metal cloth. Inorganic nitrogen compounds, potassium chloride, and water-soluble phosphorus compounds, especially ammonium phosphates, dissolve out of the fertilizer quite rapidly, regardless of particle size *(7, 9, 11)*. While the percentage of available phosphorus in the residual substance of the fertilizer band was remarkably high after the fertilizer had been in the soil for a year *(4)*, there was no consistent relation between the size of the particle and the undissolved portion of the available phosphorus in the residue. Many mixed fertilizers, especially those formulated with ordinary superphosphate, retain their granular appearance after long periods in the soil, or even in water, owing to their content of less soluble constituents, such as calcium sulfate. However, this retention of granular appearance is not a reliable criterion of solubility or of the nutrient availability of the water-soluble constituents of the fertilizer. The residual granule, in such instances, usually is largely depleted of the most soluble nutrient constituents originally present *(7, 11)*.

Homogeneity

To expect that every granule in a granulated mixed fertilizer will be of the same composition is no more reasonable than to expect that they will be of the same shape. And yet, in proportion to the degree that homogeneity in composition is approached, segregation during storage, handling, and drilling of the fertilizer is decreased, and variations in sampling are reduced, so that analytical results on different samples of the product become more uniform.

Analyses of 28 granulated commercial mixtures *(8)* have indicated that N tends to be more uniformly distributed among the different size fractions of the fertilizer than either P_2O_5, which accumulates in the coarse fractions, or K_2O which accumulates in the fines. The greatest deviations from grade occur in the highest analysis mixtures. In mixtures averaging 41% total nutrient content, the P_2O_5 averages 1.9 units above grade in the coarse fraction and 1.5 units below grade in the fine fraction. The K_2O averages 2.9 units above grade in the fine fraction and 0.5 unit below grade in the coarse fraction. By comparison, nitrogen tends to be slightly below grade in both the fine and coarse fractions and above grade in the medium fraction with a maximum difference of only 0.5 unit. The difficulty of obtaining a representative sample of such products is probably no greater than that for non-granular products, except possibly in the case of mixtures formulated with raw materials containing high proportions of particles as large as those of the final granulated product.

In these exceptional cases of undesirable particle size of initial materials, the only apparent aid to greater uniformity in granule composition of the product is finer grinding and adequate blending of initial materials. Returning to the relation between particle size and volume, it is interesting to note that eight 20-mesh particles, sixty-two 35-mesh particles, or five hundred 65-mesh particles are equivalent to one 10-mesh granule. The tendency of granules in the final product to be more uniform in chemical composition increases with a decrease in particle size of the initial materials *(4)*.

Granulation of mixed fertilizer from slurry improves the uniformity of composition of the product, though it does not nullify the need for fine-grinding of initial solid ingredients.

Much remains to be accomplished with respect to improving grinding and blending equipment and physical and chemical specifications on raw materials used in mixed-fertilizer granulation plants, but there is every indication that materials producers and machinery manufacturers are giving serious attention to these problems. Research and industrial experience on these phases of the process should result in the production of granular mixtures with greater average uniformity in chemical composition.

Drillability

Many present-day, high-analysis, mixed fertilizers are formulated with high amounts of economical forms of nitrogen, for example, ammoniating solutions containing such hygroscopic materials as ammonium nitrate and urea. Granulation, drying, coating, and packaging in high-grade moisture-resistant bags makes possible the satisfactory use of many such products that otherwise could not be used because of poor physical condition. Segregation, caking, and moisture absorption during storage and transportation of the products are greatly reduced, and the drillability is thus improved. However, an additional factor to be considered in evaluating the ultimate drillability is the effect of moisture absorption during the drilling operation on distribution of the more hygroscopic products. This undesirable characteristic of the product, which may not be apparent at the outset of drilling operations, is as important to the consumer as those of segregation and caking, which are usually apparent when the bag is opened for distribution in the field.

Drillability testing equipment at Beltsville, Md. is designed in such a way as to make possible a comparison of the effect of moisture absorption on the drillability of different fertilizers during the drilling operation. This equipment was constructed in cooperation with the Agricultural Engineering Research Branch of the Department. It consists of a drillhopper mechanism mounted over a variable-speed, endless belt divided into six-inch sections for collecting the fertilizer at any desired rate of application. Comparative weights of fertilizer from different sections of the belt indicate the degree of uniformity obtained during the operation. The equipment is 20 feet in length and is housed in a constant temperature-humidity room.

Comparative drillability tests have been made on a number of experimental and commercial products, some of which are known to drill satisfactorily under rather severe field conditions. The results (unpublished data) indicate that a product which maintains its original uniform drilling rate for 1 hour at 88% relative humidity and 76° Fahrenheit will drill satisfactorily under severe field conditions insofar as the effect of moisture absorption on drillability is concerned. Many of the high-analysis mixtures tested, some of which contained as much as 785 pounds of ammonium nitrate per ton, met this requirement. A sample of grained ammonium nitrate, which ordinarily drills satisfactorily under normal atmospheric conditions in the field, did not meet requirements of this test.

Fertilizer Efficiency

While the ideal in fertilizer manufacture is to provide a product that will give optimum delivery of nutrients to the growing plant, the conditions for approaching such an ideal are dependent, not only on the physical and chemical characteristics of the fertilizer, but on variable requirement factors among different soils and crops. The fertilizer industry has had wide experience in formulating and processing fertilizers to meet such soil and plant requirements as have been well-defined by agronomic research and farm practice; *viz.* low-chlorine goods for tobacco, boron in mixtures for legumes, magnesium in some potato fertilizers, etc. Future developments might be devoted to the production of special mixtures with less soluble, slowly available forms of nitrogen and potassium for the purpose of avoiding germination and seedling injury and losses of these nutrients by leaching. Currently-marketed mixtures containing slowly available nitrogen from urea-formaldehyde reaction products are typical examples of such developments.

Considerable attention has been given in recent years to the forms and solubility of phosphates in mixed fertilizers. Analyses of 420 samples of mixed fertilizer marketed in various regions of the United States during the 1949-1950 fertilizer season *(1)* showed that the proportion of P_2O_5 soluble in water ranged from an average of 35.2% in the East South Central States to an average of 70.7 in the Mountain States. For the United States as a whole, the percentage of available P_2O_5 in water-soluble form in all mixtures ranged from 2.8 to 99.7%, with an average of 50.2%.

Water-soluble phosphates are usually more efficient sources of phosphorus for fast maturing, high-yielding crops grown on alkaline soils, such as predominate in the West, than are phosphates in which a major portion of the available P_2O_5 is insoluble in water. *(2, 3, 6, 12)*. The agronomic efficiency of water-soluble, as compared with water-insoluble, forms of phosphate is generally less affected by particle size of the fertilizer *(9, 12)*. Water-insoluble materials, such as dicalcium phosphate, are more effective in powdered form than in granular form when applied separately, but they become partially soluble and have greater agronomic efficiency in some granulated mixtures owing to their intimate contact with soluble salts in the granule *(13)*. There is growing evidence that, as the water-solubility of phosphorus falls below 40% of the total phosphorus content of the fertilizer, the value of the fertilizer becomes relatively less for fast maturing, high-yielding crops such as corn *(14)*. Granulated mixtures that meet the nutrient requirements of different soils and crops will be developed as the effects of fertilizer properties on agronomic efficiency become clearly defined.★★

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Pilot Plant Studies of Granulation of High Analysis Fertilizers

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Wilson Dam, Alabama

GRANULATION of high-analysis fertilizers has been studied by TVA on a pilot-plant scale for nearly three years. The objective of this project is to develop effective and economical methods and equipment for granulation as a means of improving the physical properties of high-analysis fertilizers. The results of the pilot-plant studies were reported in a paper presented at the 128th national meeting of the American Chemical Society(1). The present report briefly describes the pilot plant and presents in some detail the results obtained with one fertilizer, 5-20-20, since many manufacturers are particularly interested in this grade. Pilot-plant results for other grades are reviewed briefly.

The experimental work was done in a pilot plant that had a capacity of about three tons of granular product per hour. Figure 1 (page 143) is a diagram of the pilot plant.

Superphosphate, potassium chloride, recycled fines, and any other solid materials that were required were fed by volumetric feeders to a collecting belt and thence to the continuous ammoniator. No preparatory treatment of the materials was needed, unless they were caked or contained large lumps that might clog the feeders; in this case the materials were crushed to pass a 4-mesh screen. Finer grinding was not required and was not considered to offer any particular advantage.

The continuous ammoniator(2) is 3 feet in diameter by 3 feet long. It usually was rotated at 15 r.p.m. The bed depth in the ammoniator was 9 inches, and the retention time was about 3 minutes when operated at a throughput of 3 tons per hour. Ammonia or ammoniating solution or mixtures of the two were injected under the bed of material through a special distributor. Sulfuric acid, phosphoric acid, or steam was supplied through an adjacent distributor. Air for cooling and controlling granule size was supplied from a drilled pipe located over the bed of material. The drilled holes in the pipe were located so as to direct air jets onto the surface of the bed. When water was needed for granulation, it was either sprayed on the surface of the bed, or mixed with the ammonia or ammoniating solution in the pipe line leading to the ammonia distributor. The latter arrangement was preferable. A stream of air was drawn through the ammoniator to sweep out water vapor and fumes.

The material flowed from the ammoniator through a chute to the granulator, which was an open cylinder 24 inches in diameter by 6½ feet long. It usually was rotated at 20 r.p.m. From the granulator, the material flowed to a cooler, which was 3 feet in diameter by 24 feet long and contained flights. The flow of air in the cooler was countercurrent to the flow of solids. The temperature of the material leaving the cooler was about 100° F. In a few cases the cooler was used as a dryer with cocurrent firing.

A set of screens, a crusher, and conveying equipment were used to size the product at approximately minus 6 plus 28 mesh. The undersize was recycled to the ammoniator.

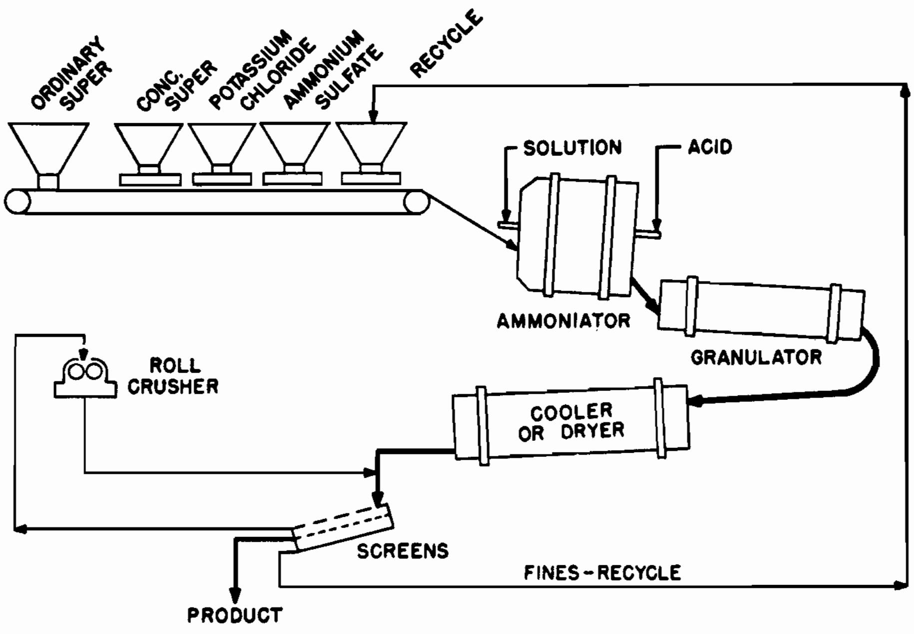

Figure 1
Flow diagram of TVA pilot plant for granulation of high-analysis fertilizer.

Factors Affecting Granulation

THE most important factor affecting granulation was the formulation. The formulation determined the heat of reaction and the amount and character of the soluble salts; these factors strongly influenced granulation. The best results were obtained when the formulation was self-granulating or nearly so. By a self-granulating formulation we mean that, when the constituents are brought together, the soluble salt content, the moisture content, and the temperature resulting from the heat of reaction are such as to impart a a suitable consistency for forming granules with only minor adjustment of the temperature or moisture content.

Poor results were obtained when the formulation was such that a large amount of water was required for granulation. Although it was possible to induce granulation at low temperatures by addition of large amounts of water, the granules often were weak and disintegrated during drying, which was required because of the high moisture content.

Poor results also were obtained when the formulations led to excessive agglomeration due to too much heat of reaction. The formation of too much oversize led to impaired efficiency of ammoniation and fume formation and interfered with efficiency of the subsequent cooling operation.

No method has been developed for predicting accurately the granulation characteristics of a formulation. It seems probable that the granulation obtained with a given formulation may vary depending on such factors as design of the ammoniator, heat losses from the ammoniator, effectiveness of temperature control in the ammoniator, physical properties of the superphosphate, and initial temperature of materials entering the ammoniator. For these reasons, it should be expected that some experimentation would be required in each plant to find the best formulation for good granulation of any particular grade.

In addition to the selection of a formulation that provides proper conditions for granulation, it is very desirable to provide operating controls to obtain the most efficient granulation. One effective and convenient method that was developed and used in the pilot plant was the use of air jets directed onto the bed of material in the ammoniator. The air jets cooled the material in the ammoniator and evaporated water from the surface of the granules, thereby decreasing the amount of oversize formed. Control of granulation by air cooling was effective when the formulation was such that too much agglomeration would occur otherwise.

Recycling of some of the cooled products was found to be effective in controlling overagglomeration. However, this method was not generally used in the pilot plant because it was inconvenient to handle the large amount of recycle that would be required for effective control. In most runs, only the fines normally produced were recycled, which amounted to 10 to 25 per cent of the product.

When the formulation was such that the extent of granulation was insufficient, additions of steam or water were sometimes effective in increasing it.

Production of Granular 5-20-20

JUDGING from comments that we have received from fertilizer manufacturers, 5-20-20 is an important grade that has given an unusual amount of difficulty in granulation plants. For this reason, several formulations for producing this grade were studied in the pilot plant. The formulations used and typical data obtained in these runs are shown in Table I. The formulations were calculated from actual feed rates; due to inaccuracies of the feeders, the proportions of the ingredients were sometimes appreciably different from the intended proportions. For this and other reasons, some of the formulations are not consistent with production of ongrade product.

In test J-1, ammoniating solution Y (26% free ammonia) was used to supply all of the nitrogen; water addition was the only means of granulation control. The heat released by the ammoniation reaction was not sufficient to permit granulation at low moisture content. It was necessary to add water to increase the moisture content to about 13 per cent to obtain granulation. The efficiency of granule formation was satisfactory, but the percentage of both oversize and undersize increased during drying, leaving only 42 per cent in the desired size range. After crushing the oversize, only 72 per cent was onsize. The dried granules were weak and dusty.

The need for artificial drying was eliminated and the stability of granules was improved by adding sulfuric acid to the formulation. The heat generated by the reaction of the acid with the free ammonia increased the temperature in the ammoniator. At the higher temperature, more liquid phase was formed by the solution of soluble salts, and no water addition was needed for granulation. This use of acid to promote granulation is illustrated by test J-2. Best operation was obtained when the acid rate was 143 pounds of acid per ton of product. Fifty-seven per cent of the granulator product was onsize. There was very little breakdown of particles in the cooler. Onsize material, including the crushed oversize, amounted to 80 per cent of the total product. The moisture content of the product was only 1.7 per cent even though artificial drying was not used.

During a part of test J-2 the acid rate was increased to 170 pounds per ton of product. Overagglomeration resulted, and operation was inferior at the higher acid rate. When the acid rate was reduced to 90 pounds per ton, there was too little granulation.

In test J-2 the degree of ammoniation was low, about 1 pound of ammonia per unit of P_2O_5. Consequently, the ammonia loss was very low, 0.1 per cent or less, and the water solubility of the P_2O_5 was very high, about 75 per cent.

The foregoing tests showed that operation with nitrogen solution as the source of nitrogen was much improved when sulfuric acid was added to supply heat by reacting with the free ammonia. Two additional tests were made to determine whether similar results could be obtained by adding part of the nitrogen as anhydrous ammonia. Thus, the amount of free ammonia available to react with the superphosphate was increased, and more heat from this source was released in the ammoniator. The nitrogen solution (solution X in these tests), anhydrous ammonia, and water required for granulation were all injected through the same distributor.

TABLE I.

Pilot-Plant Data for Production of Granular 5-20-20 Fertilizers

Test No.	J-1	J-2	J-3	J-4	J-5	J-6	J-7
Formulation, lb./ton of product							
Anhydrous ammonia	......	......	103	50	129	127	......
Nitrogen solution[a] (X, Y, or Z)	259(y)	252(y)	85(x)	180(x)	......	......	308(z)
Sulfuric acid (66° Be.)	......	143	92	......	139	......	123
Ordinary superphosphate	398	260	292	293	278	774	460
Concentrated superphosphate	675	741	807	810	769	......	......
Calcium metaphosphate	......	......	......	......	......	......	513
Phosphoric acid	......	......	......	......	......	380	......
Potassium chloride	690	669	628	630	643	645	641
Total	2022	2065	2003	1963	1958	1926	2045
Recycle, lb./ton	441	397	386	356	304	829	330
Means of granulation control	Water	Variation of acid rate	Water	Water	Water and air	Water	Water and air
Ammoniator temperature, °F.	140	206	235	176	207	176	166
Moisture content of product, %							
From ammoniator	12.6	4.4	3.7	7.5	5.9	9.0	......
Final product[b]	4.1[c]	1.7	2.0	4.5	3.9	......	0.6
Granulation, %							
Oversize	34	35	36	31	28	31	29
Onsize	55	57	63	65	70	58	66
Undersize	11	8	1	4	2	11	5
Onsize recovery after crushing oversize, %	83	88	81	87	91	86	91

[a] Composition of nitrogen solution:

Solution	Per Cent NH_3	Per Cent NH_4NO_3	Per Cent H_2O
X	21.7	65.0	13.3
Y	26.0	55.5	18.5
Z	16.6	66.8	16.6

[b] Product was cooled but not dried except as indicated.

[c] Product was dried.

TABLE II.

Pilot-Plant Data for Production of Various Grades of Granular Fertilizers

Grade	10-10-10	12-12-12	15-15-15	15-15-0	6-12-12	8-16-16	10-20-20
Formulation, lb./ton of product							
Nitrogen solution[a] (X, Y, or Z)	459(x)	431(x)	513(z)	485(x)	329(y)	386(y)	499(x)
Ammonium sulfate	96	206	574	494	......	......	......
Sulfuric acid (66° Be.)	116	126	......	129	......	......	93
Ordinary superphosphate		647	......	564	1265	1059	......
Concentrated superphosphate	......	252	306	390	......	......	854
Phosphoric acid (28 to 85% H_3PO_4)	......	......	256	......	......	211	......
Potassium chloride	338	383	472	......	414	522	620
Total	2085	2045	2121	2062	2008	2178	2066
Recycle, lb./ton	320	465	518	340	431	601	392
Means of granulation control	Air	Air	Air	Air	Steam	......	Air
Ammoniator temperature, °F.	202	199	144	190	187	183	197
Moisture content of product, %							
From ammoniator	2.8	2.4	3.9	4.2	7.3	4.7	2.9
Final product[b]	1.5	1.5	0.4[c]	2.6	4.2	1.4[c]	1.1
Granulation, %							
Oversize	20	29	54	37	37	26	30
Onsize	69	66	45	60	49	62	69
Undersize	11	5	1	3	14	12	1
Onsize recovery after crushing oversize, %	83	88	81	87	78	86	91

[a] Composition of nitrogen solution:

Solution	Per Cent NH_3	Per Cent NH_4NO_3	Per Cent H_2O
X	21.7	65.0	13.3
Y	26.0	55.5	18.5
Z	16.6	66.8	16.6

[b] Product was cooled but not dried except as indicated.

[c] Product was dried.

In test J-3, about 4 units of nitrogen were supplied as anhydrous ammonia, and slightly over 1 unit was supplied as solution. Ninety-two pounds of sulfuric acid were added per ton of product in order to reduce the degree of ammoniation to about 4 pounds of free ammonia per unit of P_2O_5. Good operation was obtained under these conditions. Almost two thirds of the material from the granulator was onsize, and there was practically no undersize. There was little breakup of particles in the cooler. Eighty-eight per cent of the product was onsize after the oversize was crushed. The moisture in the product was 2 per cent. Ammonia loss was only 1.8%.

In test J-4, about 2 units of nitrogen were supplied as anhydrous ammonia; the balance was added as solution. With these proportions the degree of ammoniation was 3.9, and no sulfuric acid was added. Water was added in the quantity that appeared to give the best granulation. Granulation was not quite as good as in the previous test but was considered satisfactory. Eighty-three per cent of the product was onsize after the oversize was crushed. The moisture in the product was 4.5 per cent. The ammonia loss was 3.0 per cent.

In a series of tests, of which test J-5 is typical, all of the nitrogen was supplied in the form of anhydrous ammonia. It was necessary to add sulfuric acid to prevent excessive ammonia loss and to add water to give enough liquid phase for agglomeration. In some of the tests, the water was added through an air-atomized spray on the surface of the bed of material in the ammoniator. Liquid ammonia and acid were distributed under the bed. Operation was poor because of frozen lumps which formed on the ammonia distributor and interfered with the rolling action of the bed. Much rodding was required. Fuming was severe. Granulation was fair but erratic. The ammonia loss was also erratic and reached as high as 15 per cent, although the degree of ammoniation was only 3.7 pounds of free ammonia per unit of P_2O_5. Increasing the acid rate from 158 to 179 pounds per ton did not improve operation.

In further tests, including test J-5, the water was premixed with the ammonia by teeing the water line into the ammonia line just upstream from the ammonia distributor. This change eliminated the formation of lumps on the distributor. The heat of solution of ammonia in the water raised the temperature in the vicinity of the distributor above the freezing point.

As illustrated by test J-5, operation under these conditions was very good. The material from the granulator contained only 2 or 3 per cent fines. The cooled product was 91 per cent onsize after the oversize was crushed. Loss of ammonia was only 1.5 per cent. There was no fuming when 5500 cubic feet of air and 190 pounds of water were used. There was only slight fuming when the air rate was reduced to 2800 cubic feet and the water rate was reduced to 140 pounds per ton of product.

In all of the tests in which all of the nitrogen was supplied as anhydrous ammonia, the moisture content was less than 5.0 per cent without artificial drying. The moisture ranged from less than 1 per cent up to about 4.5 per cent, depending largely on the input moisture content.

Several tests were made in which phosphoric acid rather than concentrated superphosphate was used to supplement the P_2O_5 from ordinary superphosphate. Nitrogen was added as anhydrous ammonia. Water, which was required for granulation, was added in an air-atomized spray on top of the material in the ammoniator.

Test J-6 is typical of operation with phosphoric acid and anhydrous ammonia. Operation was generally satisfactory except that frequent rodding was required to remove lumps which formed around the distributor. The lump formation was similar to that described earlier when anhydrous ammonia was used. It probably could have been prevented as before by premixing the water with the ammonia. The degree of ammoniation was 7.1, and the ammonia loss was negligible. Granulation was fairly good; 58 per cent of the granular product was onsize. The moisture content of the material from the granulator was 9 per cent. The material was not processed farther than the granulator. Artificial drying probably would have been required to decrease the moisture content enough to prevent caking in storage.

One exploratory test (J-7) was made in which calcium metaphosphate was used to replace the concentrated superphosphate in making granular 5-20-20. The production rate was 1 ton per hour. During the test the sulfuric acid rate was varied to find the amount that gave best granulation. About 120 pounds per hour appeared to be about optimum. Thirty-six pounds of water per ton were added to assist in granulation. Air was blown onto the surface of the material in the ammoniator at a rate of 3700 cubic feet per ton to reduce the formation of oversize.

Granulation was good in this test; 70 per cent of the material from the granulator was onsize. After crushing the oversize, 87 per cent of the cooler product was onsize. The ammonia loss was about 2 per cent. The screened product contained only 0.6 per cent moisture even though it was not dried artificially.

Production of Various Grades

TYPICAL pilot-plant data for production of several grades of granular fertilizer are shown in Table II. This work is described in detail elsewhere (1). The degree of granulation and strength of the granules were considered satisfactory for each of the tests shown in Table II. With the exception of those grades that were made with phosphoric acid, artificial drying was not required. Bag-storage tests indicated that the physical properties of the products were satisfactory, particularly when cured 7 days and coated with 2.5 per cent by weight of kaolin.

The effect of particle size of the solid raw materials was studied briefly during tests with 8-16-16. When the raw materials, potassium chloride and superphosphate, were ground to pass a 28-mesh screen, granulation was poor with large amounts of both oversize and undersize. Using unground raw materials in which 39 per cent of the superphosphate and

11 per cent of the potassium chloride were plus 28 mesh improved granulation considerably. Substituting granular potassium chloride (73% +28 mesh) further improved granulation. In a few tests with grades other than 8-16-16 the particle size of the potassium chloride had little effect on granulation.

A few tests were made in which 0.5 or 1.0 pound of an anionic surfactant per ton of product was added to the mixture entering the ammoniator. Some increase in the percentage of onsize granules was noted in some of the tests, but the effect was not conclusive.★★

References

1. Hein, L.B., Hicks, G. C., Silverberg, Julius, and Seatz, L. F. "Granulation of High-Analysis Fertilizers." October 28,1955. (Presented at the 128th National Meeting of the American Chemical Society, Minneapolis, Minnesota, September 1955
2. Yates, L. D., Nielsson, F. T., and Hicks, G. C. *Farm Chemicals* 117, No. 7, 38-48; No. 8, 34-41 (1954).

Granulation at the
Spencer ARK-MO Plant

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SINCE March 7, 1955, when the Ark-Mo Plant went through its shake-down run, much valuable information has been developed on the granulation of many grades of mixed fertilizers, using the most economical formulas, based on present supply of raw materials. Basically, plant design has proven sound, but experience has shown us where some changes will be beneficial. These changes are being recommended to other companies converting to granulation.

This discussion describes the present process and indicates desirable changes.

Solids

The flow diagram is shown in Figure I. In Figure II, the warehouse on the left is dry raw material storage; in the center, the process building; and on the right, a converted airplane hangar for finished product. Normal superphosphate, triple superphosphate, potash, and inerts are fed into the elevator in the center of the warehouse, which discharges onto a conveyor that connects with the process building. The conveyor that carries the finished product to storage may also be seen between the process building and the hangar.

Figure III is a picture of a model of the process building and equipment taken from the raw material side. The four storage hoppers (A) near the center are for the various raw materials. The hopper (B) at the left end of the building is for recycle storage. Each dry material and recycle is weighed continuously with gravimetric feeders and flows onto the common conveyor belt leading to the elevator on the right. The elevator discharges onto either or both of two ten-mesh screens. The rejects from the screens pass through either or both of two crushers and back into the base of the elevator. Actually in the operation at Ark-Mo, only one screen and one crusher have been used at the same time. We feel the extra ones are not needed.

Figure IV shows the finished product side of the model. The ten-mesh mixture of dry materials flows into a TVA-type ammoniator (C) which can be seen on the second elevation at the left in the picture. This cylindrical vessel is 7 feet in diameter and 8 feet long. There is a 27-inch retarding ring in the center and the ammoniation takes place in the front half.

Liquids

Ammoniating solutions, anhydrous ammonia, sulfuric acid, phosphoric acid, and water are either pumped or pressured through flowmeters, and are fed into the ammoniator through spargers. When ammoniating solution, anhydrous ammonia, and water are used in the same formula, they are mixed in a pipe ahead of the ammoniating sparger. A separate sparger is used for acid.

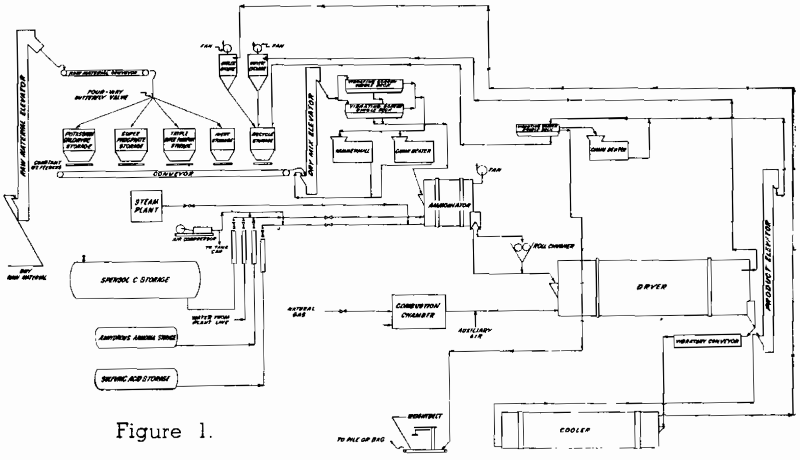


Figure 1.

Steam

Steam and acid are not used in the same formula, so the same sparger is used for either material.

The back half of the 7 foot by 8 foot vessel is used to firm up and round off the pellets formed in the ammoniator. The product then flows into the 8 foot by 40 foot dryer. The first three feet of this cylindrical vessel contain directional flights to move the material forward. The next three feet contain no flights, and the pellets are further firmed and rounded. The remainder of the vessel is equipped with lifting flights. A vibratory conveyor carries the product from the dryer to the 7 foot by 30 foot cooler. It then flows into an elevator that lifts it to a doubledeck screen. The +6 mesh material from the top screen goes through a crusher and back into the elevator. The material that flows over the bottom screen (14 or 20 mesh, depending on grade being made) flows onto the finished product belt. The fines going through the bottom screen and dust from the dryer and cooler cyclones drop into the recycle hopper.

A discussion on all the grades that have been made is not contained in this article, although some 30 different formulations have been made in the Ark-Mo plant. The two grades discussed below represent extreme conditions; that is, a grade that requires a great amount of liquids to supply the nitrogen, and one that requires a small amount, namely, 14-14-14 and 3-9-27. The 14-14-14

TABLE 1.

Ark-Mo Fertilizer Granulation Plant

Grade 14-14-14

FORMULATION	% Plant Food	#/Ton
Spensol C	37	762
Anhydrous		
Ammonium Sulfate		
Sulfuric Acid 66°Bé		194
Superphosphate	20	210
Triple Super	47	516
Potash	60	468
Filler		
Total		2160

FLOWS		
Production Rate	8	T/hr.
Recycle Rate	27	T/hr.
Total Throughput	35	T/hr.
Gas Consumed	2,100	CFH
Air Flow Dryer	14,000	CFM
Air Flow Cooler	15,000	CFM

TEMPERATURES	°F
Atmospheric	62
Burner Air	65
Ammoniator	180
Dryer Throat	420
Dryer Stack	195
Dryer Discharge	190
Cooler Stack	145
Cooler Discharge	
Finished Product	120

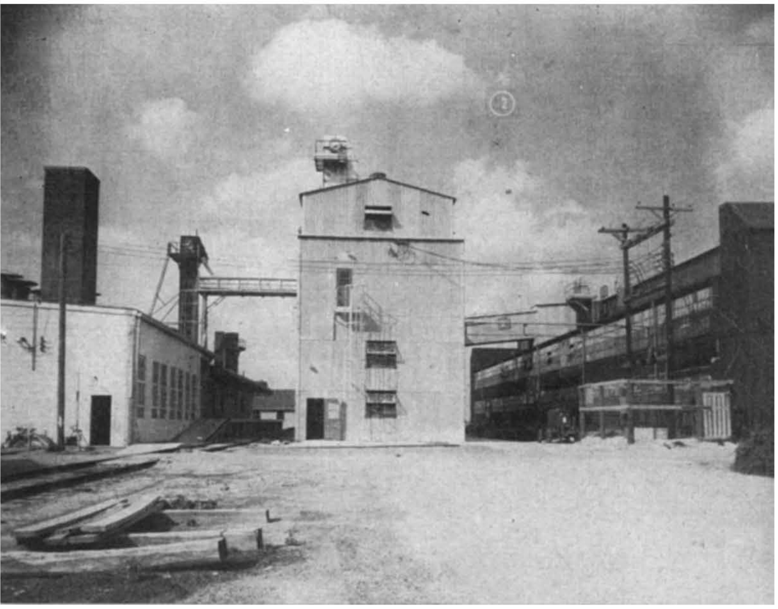
HUMIDITIES	#H_2O/#Dry Air	Dry Bulb	Relative %
Atmospheric	0.0094	62°F	79
Dryer Stack	0.052	175°F	18
Cooler Stack	0.026	140°F	29

SIEVE ANALYSIS (%)

	Ammoniator Exit	Cooler Exit	Finished Product	Recycle
Held on 6	38.6	28.4	4.7	0
Held on 14	58.8	52.7	92.4	9.0
Held on 20	78.3	75.5		51.1
Held on 40				87.2
Held on 65				

CHEMICAL ANALYSIS

	Ammoniator Feed	Ammoniator Exit	Finished Product
Moisture	1.80	3.00	2.40
N	11.14	13.46	13.71
Available P_2O_5	15.82	14.12	14.50
Insoluble P_2O_5	0.18	0.17	0.18
Total P_2O_5	15.00	14.30	14.68
K_2O	16.99	15.25	14.04



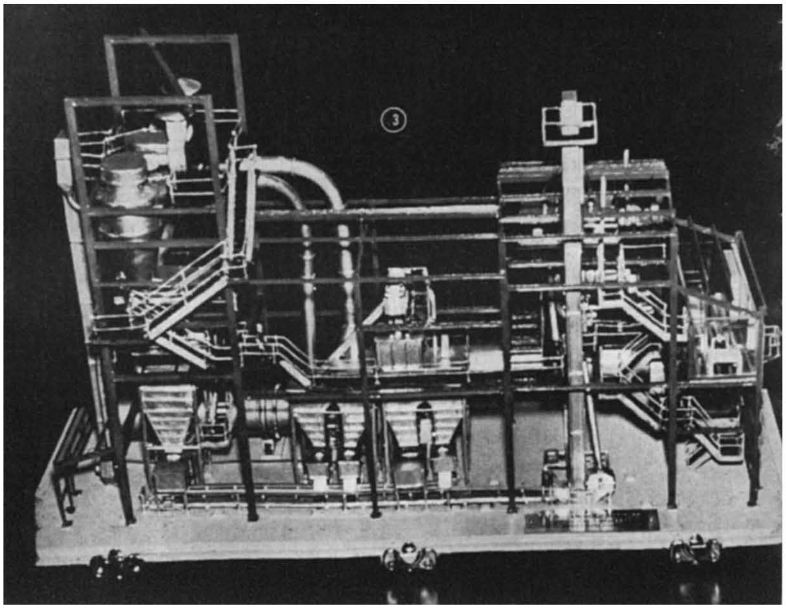


TABLE 2.

OPERATIONAL DATA
Ark-Mo Fertilizer Granulation Plant

Grade 3-9-27

FORMULATION	% Plant Food	#/Tons
Spensol C	37	109
Anhydrous	82.2	25
Ammonium Sulfate		
Sulfuric Acid		
Superphosphate	20	900
Triple Super		
Potash	0	900
Filler		110
Total		2044

FLOWS		
Production Rate	10	T/hr.
Recycle Rate	4.5	T/hr.
Total Throughput	14.5	T/hr.
Gas Consumed	3,500	CFH
Air Flow Dryer	10,650	CFM
Air Flow Cooler	4,095	CFM

TEMPERATURES	°F
Atmospheric	79
Burner Air	86
Ammoniator	205
Dryer Throat	410
Dryer Stack	197
Dryer Discharge	200
Cooler Stack	150
Cooler Discharge	121
Finished Product	120

HUMIDITIES	#H_2O/#Dry Air	Dry Bulb	Relative %
Atmospheric	0.019	79°F	89
Dryer Stack	0.094	190°F	21
Cooler Stack	0.024	130°F	25

SIEVE ANALYSIS (%)

	Ammoniator Exit	Cooler Exit	Finished Product	Recycle
Held on 6	9.1	14.8	1.5	0.0
Held on 14	61.0	60.0	65.0	0.0
Held on 20	22.4	19.6	33.0	21.0
Held on 40	7.1	5.6	0.5	65.0
Held on 65	0.4	0.0	0.0	14.0

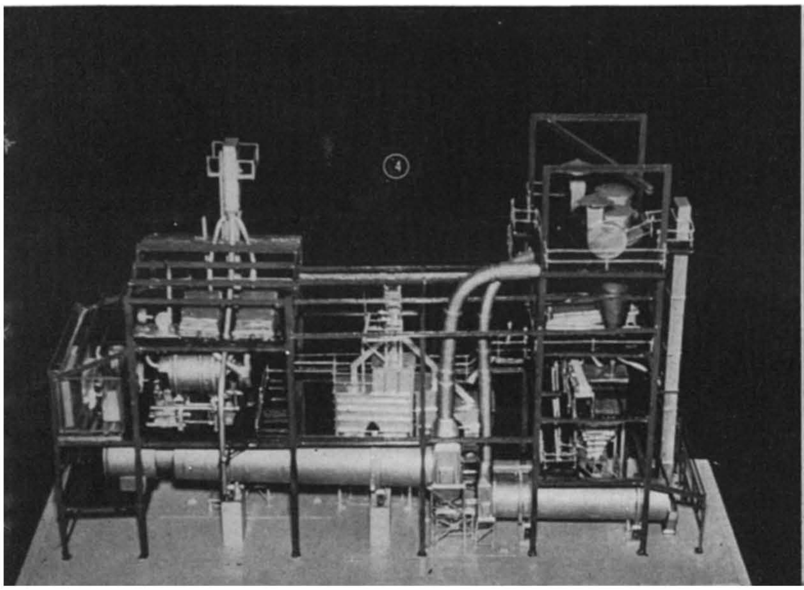
CHEMICAL ANALYSIS

	Ammoniator Feed	Ammoniator Exit	Finished Product
Moisture	4.60	4.68	1.48
N	1.39	4.81	* 4.74
Available P_2O_5	9.55	8.89	8.97
Insoluble P_2O_5	0.08	0.06	0.08
Total P_2O_5	9.63	8.95	9.05
K_2O	27.05	23.28	26.84

* Spensol requirement below flowmeter calibration. Corrected by installing smaller flowmeter.

formula has 956 pounds of liquids, whereas, the 3-9-27 has only 134 pounds of liquids. Formulas and operational data on these grades are shown in Tables 1 and 2.

The operational data on 14-14-14 shows that 27 tons per hour of recycle at an 8 ton per hour production rate was required. This dried and cooled recycle served to absorb the liquids, and thus prevent a slurry from being formed in the ammoniator. The finished product temperature was 120° F. With greater cooling capacity, the amount of recycle could have been reduced, because temperature of the recycle greatly affects the solution phase in the ammoniator. We now recommend that the cooler be the same size as the dryer. The data also show that the cooler exit has 28.4 per cent held on a 6-mesh sieve. With the present system, much of this material is broken up to finished product size. This causes too many jagged-shaped particles in the finished product. Furthermore, the particles being made from larger particles are not sufficiently dry to ensure proper mechanical condition. The +6-mesh material should be removed at the end of the dryer, crushed, and returned to the head of the dryer. This change eliminates the need for a double-deck screen, but it requires two single-deck screens instead. Two per cent of diatomaceous earth is added to 14-14-14 and other high nitrogen



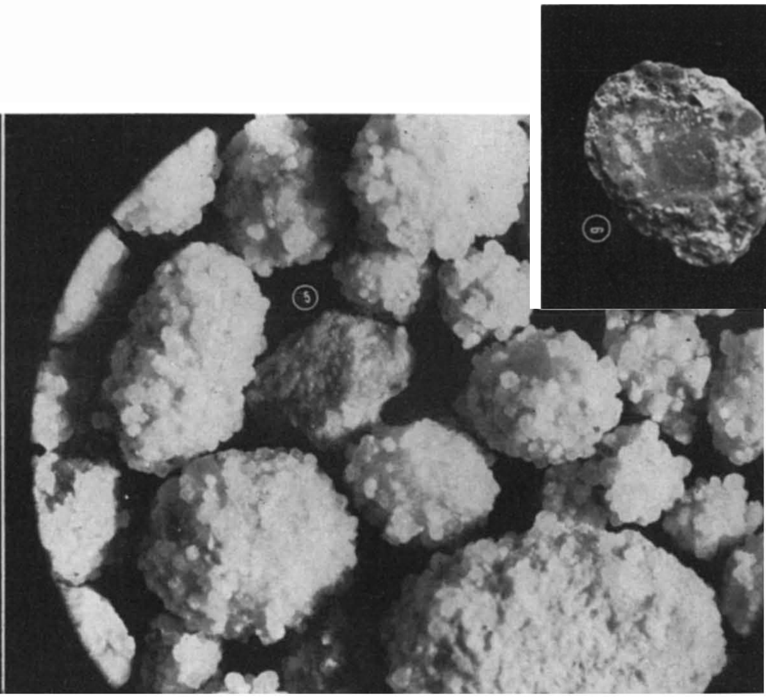


TABLE 3.

SIEVE ANALYSIS — RAW MATERIALS

60% Potash (Coarse)	Fraction	Acc.	60% Potash (Fine)	Fraction	Acc.
/6	0.0	0.0	/6	0.0	0.0
6/14	31.5	31.5	6/14	0.5	0.5
14/20	51.1	82.6	14/20	18.1	18.6
20/40	13.5	96.1	20/40	64.3	82.9
40/65	2.7	98.8	40/65	14.3	97.2
65/	1.2		65/	2.8	

TABLE 4

ANALYSIS SIEVE FRACTIONS

Grade	Particle Size	Total N	Total P_2O_5	K_2O	Free H_2O
3-12-12	Blank	3.0	11.2	12.5	1.0
	6/8	2.9	11.9	11.0	0.9
	8/14	2.9	11.0	13.2	0.8
	14/	3.1	11.2	13.7	0.9
3-9-18	Blank	2.9	8.9	17.9	0.6
	6/10	2.7	9.2	15.5	0.6
	10/16	2.9	8.7	19.1	0.6
	16/	3.2	9.0	18.2	0.6
3-9-27	Blank	2.7	8.1	27.6	0.7
	6/10	2.9	9.4	21.7	0.8
	10/16	2.4	7.7	29.3	0.6
	16/	2.7	7.9	29.0	0.6
10-10-10	Blank	10.0	10.9	10.8	0.8
	6/10	10.0	11.5	9.4	0.8
	10/16	10.1	10.9	12.6	0.8
	16/	10.4	10.8	11.8	0.7
14-14-14	Blank	13.3	13.7	13.8	2.6
	6/10	13.2	13.9	14.0	2.6
	10/16	13.1	14.6	13.4	2.4
	16/	12.2	13.7	15.4	2.2

grades as a parting agent to enhance mechanical condition.

The operational data sheet on 3-9-27 shows 4.5 tons per hour of recycle for a 10 ten per hour production rate. The only reason for this recycle is to remove the —20 mesh material from the finished product. Since the liquids in the formula are low, this recycle hinders granulation by competing with the other materials for the liquids present. In addition to the liquids shown in the formula, it is necessary to add 100 pounds of water as such per ton and 74 pounds of steam to bring about granulation. In this case, it would be better to recycle the hot fines, instead of cold fines.

A system of chutes in connection with the two single-deck screens will allow hot or cold recycle to be used, to suit the grade being made.

Coarse potash was used in this formula. The sieve analyses are shown in Table 3. As can be seen in the picture of a bisected pellet, Figure V, the potash particle is the nucleus. Figure VI contrasts granules made with fine potash, where the potash particles are sticking loosely to the surface.

Table 4 shows analyses of various sieve fractions. The 3-12-12, 3-9-18, and 3-9-27 were made with coarse potash, and the 10-10-10 and 14-14-14 with fine potash. Attempts to granulate high potash grades with fine potash have been unsatisfactory.

Summary

THE Ark-Mo Plant has successfully granulated 16-20-0, 13-39-0, 10-20-0, 14-14-14, 13-13-13, 12-12-12, 10-10-10, 8-8-8, 10-20-10, 8-24-8, 6-8-12, 4-12-4, 5-10-5, 3-12-12, 3-9-18, 3-9-27 and 5-20-20. All the nitrogen was derived from Spensol, or a combination of Spensol and anhydrous ammonia. In addition to using low-cost nitrogen, these high analysis materials allow more room for normal superphosphate, which often further reduces formulation costs.

We recommend the elimination of one raw material screen and one raw material crusher. Also, the cooler should be the same size as the dryer. The process should be changed to remove the +6-mesh material at the end of the dryer, which will replace the double-deck screen with two single-deck screens for finished product. A system of chutes should be provided to permit the use of either hot or cool recycle. Spencer Chemical Co. Technical service is available to assist in these problems.★★

TEMPERATURE & MOISTURE
Relationships in Granulation

*By Rodger C. Smith**

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THE search for some method of effectively reducing the problem of mechanical condition or bag set has been a long one. Of the many techniques and additives which have made their contribution to fertilizer manufacturing, the method known as granulation is now generally regarded as a practical and economical way of accomplishing good mechanical condition.

Within recent years, when those of us at Eastern States Farmers' Exchange, and other organizations began working on granulation techniques, there occurred serious questions on whether or not it was a practical answer to the problem of bag set. Virtually the only method of granulation being practiced was that used in an increasing number of European mixed fertilizer plants whereby mixed fertilizer is removed from the storage pile, placed in a batch mixer with sufficient water to accomplish agglomeration, and then discharged to a granulator . . . from which the material passes to drying and cooling facilities. Approximately 15-18% moisture is necessary to accomplish agglomeration with that method. Obviously, the important matter of cost causes serious objections to that method. Each step in that process represents additional capital and operating costs. Other objections are apparent.

Fortunately, when Eastern States and others in the United States commenced study of granulation, there was some basic work completed on the properties of common fertilizer materials, the ammoniation of superphosphates, and some of the factors important to granulation. Work had been done principally by W. H. Ross, J. H. Hardesty and their associates in the U.S. Bureau of Plant Industry, which is referred to in part in this article. At that time, ammoniation of superphosphate was common, and the studies by John Hardesty and others suggested the extent to which ammonia could be reacted with superphosphates of various concentrations and physical properties. Data was available on the heat resulting from the ammoniation reactions. Solubility of salts commonly used as fertilizer ingredients had been determined. This information led to the gradual recognition that use of ammonia as anhydrous or in nitrogen solutions not only could reduce the cost of nitrogen in the mixture, but would contribute to granulation in the following ways:

(1) Use of sufficiently high amounts of ammonia in the mixture increases the temperature of the batch substantially, and according to a fairly definite ratio.

(2) Large amounts of the salts contained in the mixture of ingredients are dissolved at the temperatures attainable through high ammoniation. These salts dissolved in the free water increase the solution phase of the mixture.

(3) Ammonium nitrate and urea are the most soluble of salts used commonly in mixed fertilizers. They have also the greatest increases in solubility with increases in temperature, which is of particular importance.

(4) During the drying and cooling steps, low moisture content and large amounts of salts in solution contribute to rapid crystallization of salts and, therefore, permanency of the granules formed during agglomeration.

From this very brief review, it is apparent that temperature and moisture relationships are extremely important considerations in granulation. They can be regarded as the keys to granulation, although much of our effort has necessarily concerned equipment to accomplish the several steps in granulation. Very appropriately, much of this conference concerns suitable equipment. In summarizing some of the temperature and moisture relationships, let us begin first with formulation and then consider the steps in the process individually.

Formulation

GRADES which are generally being granulated, contain 5% or more nitrogen. Granulation is facilitated directly with increasing nitrogen content, which in turn is directly related to the amount of the more soluble nitrogen salts contained. The relative solubilities in water at 68° F. of some common fertilizers are as follows:

	Weight Per Cent
Ammonium Nitrate	66.0
Calcium Nitrate, Anhydrous	56.4
Urea	52.0
Sodium Nitrate	46.7
Ammonium Sulfate	42.8
Diammonium Phosphate	40.8
Mono-ammonium Phosphate	27.2
Ammonium Chloride	27.1
Potassium Nitrate	24.0

(Crittendon: Allied Chemical and Dye Corporation)

As this table indicates, ammonium nitrate is the most water soluble fertilizer material, with urea high on this list. It is of importance, also, that the solubility of these two materials increases substantially with increase in temperature. Mono-ammonium phosphate and potassium nitrate respond similarly through a lower temperature range. With increases from 32° to 212° F., ammonium nitrate increases in percent solubility by weight from 54 to 89% and urea from 39 to 87%.

Eastern States Farmers' Exchange has used ammonium nitrate—ammonia solutions in granulation, with only limited experience with urea-ammonia solutions. Our observation indicates that smaller granules are formed with the urea-ammonia solutions. Attempts to formulate, using both ammonium nitrate and urea, have been discouraging in that the proportion of desirable granules has been reduced substantially.

The volume of the liquid phase depends upon the ingredients used, the concentration of the mixture and, as indicated above, the temperature of the fertilizer. Hardesty has reported that the heat developed when superphosphates are treated with 6 pounds of anhydrous liquid ammonia per 20 pound unit of P_2O_5 is equivalent to 1426 BTU per pound of ammonia absorbed for ordinary superphosphate, and 1589 BTU for triple superphosphate. Their work indicates that the heat evolved per unit of ammonia absorbed decreases somewhat with the degree of ammoniation. Their work further indicates that a variation in the moisture content of a superphosphate, from 3-15%, has little or no effect on the heat developed in its ammoniation. More recent work reported by Kumagai, Rapp and Hardesty, with controlled maximum temperatures of reaction from 122° F. - 190° F. and using 7 pounds ammonia per unit P_2O_5 in normal superphosphate, had negligible effect on percent of added ammonia absorbed. On the other hand, an increase in controlled maximum of reaction (from 149-216° F.) using 5 pounds ammonia per unit of P_2O_5 in triple superphosphate, increased the percent of added ammonia absorbed from 79.1% to 89.6%

It is our experience, and I believe that of others employing high ammoniation practices in batch mixers, that about 2.5 pounds NH_3 can be reacted with 1 unit P_2O_5 in triple superphosphate; and 6.0 - 7.0 pounds NH_3 can be reacted with 1 unit P_2O_5 in ordinary superphosphate. These rates of ammoniation suggest that essentially the following reactions occur when both triple superphosphate and ordinary superphosphate are present.

$$Ca\ (H_2PO_4)_2 \cdot H_2O + NH_3 \rightarrow CaHPO_4 + NH_4H_2PO_4 + H_2O$$

$$NH_4H_2PO_4 + CaSO_4 + NH_3 \rightarrow CaHPO_4 + (NH_4)_2SO_4$$

Use of nitrogen solutions in quantities to attain the above rates of ammoniation has resulted in mixer temperatures of about 160° F. with 5% nitrogen grades, and nearly 200° F. with 8 to 10% nitrogen grades. Higher temperatures in the mixer are desirable to increase the quantity of salts in solution. Common methods of providing additional heat are:

1. Reaction of either sulfuric or phosphoric acids with additional ammonia.
2. Addition of steam.
3. Addition of hot water.

Of these three methods, the inclusion of either sulfuric acid or phosphoric acid appears to be the most satisfactory. The cost of acid is approximately offset by use of additional nitrogen solutions or anhydrous ammonia as source of nitrogen in the formula, as compared with the cost of an equal amount of nitrogen in dry form. One pound of ammonia reacts with approximately three pounds of 100% equivalent acid, according to the following reactions:

$$2NH_3 + H_2SO_4 \rightarrow (NH_4)_2SO_4$$

$$NH_3 + H_3PO_4 \rightarrow NH_4H_2PO_4$$

$$NH_4H_2PO_4 + NH_3 \rightarrow (NH_4)_2HPO_4$$

Heat is developed, apparently, as a result of both the heat of reaction and heat of dilution. Hardesty and Ross have reported the heat developed in the neutralization of $H_3PO_4(2OH_2O)$ with NH_3 (liq.) to form $(NH_4)_2$HPO4 $(2OH_2O)$, to be 32,010 gram-calories.

With the use of either of these acids with anhydrous ammonia or a suitable nitrogen solution, temperatures of approximately 212° F. in the mixer can be attained. The evaporation of some moisture from the mix deters further rise in temperature.

Factors which contribute to high content of liquid phase can be summarized.

1. High analysis grades.
2. Ratios high in nitrogen.
3. Formulation with Ammonium Nitrate or Urea.
4. High ammoniation.
5. Reaction of an acid with additional ammonia.
6. Addition of steam or water as necessary, particularly in low analysis or low nitrogen grades.

Taking advantage of these factors in particular, it is possible to granulate at input moisture contents of approximately 6% with 8 or 10% nitrogen grades, and approximately 12% with 5 or 6% nitrogen grades. A balance between the amount of ammonium nitrate or urea, the moisture content and the temperature in the mixer is important. This balance will vary according to grade, equipment and sources of materials. As to materials other than nitrogen, let me state briefly that sources of superphosphates and muriate of potash or the use of sulfate of potash to replace muriate of potash do affect the

Air Ex-dryer	Fertilizer Ex-dryer	Fertilizer Ex-cooler	Moisture Ex-cooler
231-240°F	197°F	146°F	0.94%
221-230	187	140	1.27
211-220	191	144	1.59
201-210	172	139	1.66
191-200	175	134	1.77
181-190	161	125	2.21

balance between moisture and temperature necessary.

The composite of all materials introduced into the mixer determines largely the temperature and moisture relationships which occur, and the quality of granulation. However, construction and characteristics of equipment do have their effect on temperature and moisture, and the quality of granulation attained.

Granulator

IT is important to provide conditions which encourage agglomeration at the stage in the process when the temperature and moisture levels for a given formula encourage rapid crystallization and hydration. With formulas of high soluble salt content and low moisture discharge from the mixer at high temperature, granulation can be attained immediately following discharge from the mixer. With formulas of relatively low soluble salt content, and necessarily high moisture content, agglomeration is more gradual and related to the reduction of moisture content. Agglomeration may occur in a so-called granulator, the dryer or the cooler, depending upon the formula of the mixture and the characteristics of the equipment used.

A granulator essentially provides agitation or tumbling of the fertilizer during temperature reduction. A thin layer of material is important, the rate of production not exceeding 1000 pounds per square foot per hour of effective granulator surface. Either a rotary cylinder or mechanism providing horizontally vibrating surface can be used.

Dryer

A DRYER installation should attempt to accomplish at least the following:

1. Moisture reduction of the fertilizer.
2. Low fertilizer discharge temperature.
3. Maintain or increase agglomeration.

The quantity of heat, the volume of air and the direction of air flow principally determine moisture and temperature relationships in the dryer and affect agglomeration. Diameter and length of dryer and construction of flights also affect agglomeration. Temperature of the fertilizer must be maintained or increased to sufficiently high temperatures to evaporate water. Application of excessive heat can reduce the moisture content more than necessary, and result in unnecessarily high ex-dryer fertilizer temperature. Adequate volume of air is required to convey the evaporated moisture. Too large air volume can reduce drying efficiency, requiring excessive consumption of fuel. The fact that the discharge temperature of the fertilizer is sometimes less than the temperature of the fertilizer entering the dryer, leads to the occasional statement that fertilizer dryers are actually coolers.

These relationships can be illustrated by the average temperatures (see table below) on several weeks production of an 8% nitrogen grade at an Eastern States plant. Fertilizer exdryer temperatures, fertilizer excooler temperatures and fertilizer excooler moistures are summarized according to air ex-dryer temperatures.

If 2% moisture is assumed to be satisfactory, it can be concluded from this data that with this grade, an air ex-dryer temperature of about 200° F. is adequate in this particular dryer and with the given air volume. Higher ex-dryer air temperatures would increase both the fertilizer ex-dryer and ex-cooler temperatures.

A large dryer with substantial range of air volume and heat input offers advantages. Temperature and moisture conditions and mechanical features can be adjusted more satisfactorily in a large dryer to accomplish considerable agglomeration, the required drying and the partial cooling within the dryer. Co-current air flow is generally employed and assumed in this discussion.

Counter air flow in a dryer permits attaining lower moisture content with a given heat input, but results in substantially higher fertilizer ex-dryer temperatures, and is more likely to cause reduction of nitrates, with resulting evolution of nitrous oxide. In the summary above, there is less than 50° decrease in fertilizer temperature in the cooler. In an Eastern States granulation unit having counter-current air flow and similar air volume and fuel consumption, the fertilizer dryer discharge temperature ranges between 225° and 260° F, but the fertilizer decreases approximately 100° in the cooler. With these temperatures, agglomeration tends to occur in the cooler.

Cooler

REDUCTION of fertilizer temperature in the cooler is a result of transfer of heat from the granules to air, and the evaporation of limited amounts of moisture. The principles of the fertilizer rotary cooler, as first developed by Keenen and associates of the DuPont Co., are applicable today. With less moisture in granular fertilizer to evaporate in the cooler, there is less reduction of temperature of fertilizer per given volume of air. By study of air flow, air volume, air temperature, fertilizer temperature and fertilizer moisture, maximum efficiency of a cooler can be determined. Granulation has accentuated one factor and that is time. Increased time during which fertilizer is in the cooler encourages the transfer of heat to the surface of granules and then transfer to the air.

Typical Moisture and Temperature

IT may be helpful to summarize what can be considered approximate moisture and temperature levels at several locations in the process, which contribute to granulation. Formulation and other factors affect these figures.

Control

UNIFORMITY of granular product is important. An unsatisfactory granular product can be more objectionable than pulverized fertilizer. Lack of control of temperature and moisture levels is likely to

Approximate Temperatures & Moistures of Mixed Fertilizer During Granulation

Fertilizer in	8 - 10% N		5 - 6% N	
	°F	% H_2O	°F	% H_2O
Mixer	200	6-7	170	10-12
Granulator	190	5-6	165	9-11
Dryer discharge (co-current)	175	2-3	160	3-4
Cooler discharge	130	1.5-2.0	120	2.5-3.0

result in unsatisfactory granular fertilizer. Control methods and equipment which can be used include the following:

1. Cenco Moisture Balance—This inexpensive instrument enables moisture determinations within 15 minutes following sampling. Plant personnel can operate it. Use of this instrument at the plant provides opportunity for adjustments in formulation, dryer heat input and dryer air volume before a large tonnage too high in moisture is mixed and placed in the storage bin. It is important to correlate the determination with the method of moisture determination being used in the laboratory.

2. Potentiometer -- Opportunity to conveniently and accurately determine temperatures at strategic locations in the process is important. Single thermocouples, with or without recording instruments, can be installed. A satisfactory and economical method of obtaining continuous temperature records is by installation of a multi-record strip chart electronic potentiometer. With a six point recorder, thermocouples might be located to obtain temperatures of the following:

   a. Fertilizer — mixer or granulator.
   b. Fertilizer — ex-dryer.
   c. Air — ex-dryer.
   d. Fertilizer — ex-cooler.
   e. Air — ex-cooler.
   f. Air — ambient.

3. Electronic Proportioning Relay — With a uniform volume of air being exhausted through a dryer, the ex-dryer air temperature can serve as an accurate index of the fuel input necessary for satisfactory moisture reduction of a given grade. After obtaining sufficient temperature data on production of each grade in a given plant, it is possible to establish ex-dryer air temperatures by grade, which reflect satisfactory drying. Adjustment of fuel input can be done manually.

4. Control Laboratory — The control laboratory personnel can assist development and production personnel immeasurably during the development of a given process and the continued production of satisfactory granular fertilizer. Moisture determinations are included in the important information obtainable in the laboratory.

In final analysis, the most important method of control of temperature and moisture levels is through informed personnel. Plant supervisors and operating personnel who have adequate information on temperature and moisture relationships, and who are guided in the effective operation of their granulation equipment, have an advantage in the production of quality fertilizer.★★

Granulation with "Uramon" Ammonia Liquors

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COMPARATIVELY little has been done, or even attempted, using urea - ammonia - water solutions (UAL) in the granulation of complete fertilizers. Most of the process development, as well as the practice to date, has employed ammonium nitrate-ammonia-water solutions, often as the sole source of nitrogen, or supplemented with anhydrous ammonia, ammonium sulfate, sulfuric acid, or phosphoric acid.

The reasons for the lack of interest in or work with urea are probably twofold. First urea solutions have had for a considerable period, a somewhat limited production and availability, whereas the current and prospective supply situation of ammonium nitrate solutions, produced by a larger number of manufacturers and in much larger quantity, has been favorable. With the advent of new urea producers, and with more planned, this condition is rapidly changing.

A second and almost equally potent reason for the neglect of urea has been the generally held opinion that urea was not a suitable material in the granulating process, because of hydrolysis or decomposition under the high temperatures involved in the am-

TABLE 1

Materials and Analysis	Pounds Per Ton			
	8-16-16	10-10-10	5-20-20	10-10-10
UAL-B 45.5	335	444	220	330
R P Super 20	600	1000	540	1000
Triple Super 45	445	—	650	—
Ammonium Sulfate 20.6	—	—	—	275
Muriate of Potash 60	535	335	670	335
60° B Sulfuric Acid	170	330	25	180
	2085	2109	2105	2110

moniation and subsequent drying processes. The reasoning here was based on experience with urea in conventional fertilizers, in which urea is known to hydrolyze in long-continued hot storage. Since conditions are somewhat different in granulation processes, with lower moisture content of the finished product, and with cooling generally practiced, it was felt expedient to explore the behavior of urea; to ascertain if hydrolysis was a serious factor, and if so, where; and to determine the granulating characteristics of urea with respect to storage qualitites, granule hardness, and hygroscopic behavior.

This, then, is an account of tests, first in a pilot plant, and then in plant scale equipment, using Du Pont "Uramon" Ammonia Liquors A and B, hereinafter referred to as UAL-A and UAL-B. These tests were begun about a year ago, and are still continuing. They have been exploratory in nature, and no pretense is made of uncovering any new granulation technique. Most workers in the field will concede that granulation today is an art, more than a science, and that much remains to be learned.

Pilot Plant Work

IN the pilot plant tests, the equipment was essentially the same design and size as the TVA pilot plant, consisting of an ammoniator-granulator, a gas fired counter-current dryer, and accessories such as automatic feeders for dry materials and flow-rate meters or volumetric pumps for UAL, sulfuric acid, and water. Dry materials were screened to -10 mesh. Product was produced at a rate of about 1000 pounds/hour. Cooling was effected by spreading material in a thin layer with air movement provided by a fan.

Four formulas were tested, 8-16-16, 10-10-10, and 5-20-20, with all of the nitrogen from UAL-B; and a 10-10-10 with approximately 7¼ units from UAL-B and 2¾ units from ammonium sulfate. The formulas were calculated for absorption rates of 5 pounds ammonia-unit of phosphoric acid in 20% run-of-pile super, and 3 pounds/unit in 45% triple super. 60° B sulfuric acid (77% H_2SO_4) was added to neutralize the excess free ammonia, after allowing for the calculated absorption of the phosphoric acid, at the rate of 3.7 pounds 60° acid for each pound of free ammonia. In the 5-20-20, the acid was necessary only for the heat. As will be noted, the amount of sulfuric acid in the 10-10-10 with all N from UAL-B was considerable. Some variations were tried in the amount of water added. The exit temperatures from the ammoniator-granulator ranged from 150° to slightly over 200° F; and from the dryer 150° to 250° F, averaging 180° to 185° F. The through-put time was 10 minutes in the granulator, and 15 minutes in the dryer, or a total of 25 minutes. The formulas given on Page 45 allow for the evaporation of varying amounts of water, and would be calculated more exactly for plant-scale operation.

Samples for analysis to determine urea hydrolysis were taken from

TABLE 2

ANALYSES

No.	Description	Nitrogen %	Urea Nitrogen Calculated %	Found %	Recovery %
381	8-16-16 dryer 180°, cooled	8.00	3.55	3.53	00
382	8-16-16 dryer 180°, 16 hrs. 140°	7.33	3.25	3.42	105
391	8-16-16 ammoniator 185°, cooled	7.20	3.20	3.11	97
392	8-16-16 dryer 140° cooled	7.70	3.42	3.31	97
393	8-16-16 dryer 174° cooled	8.70	3.86	3.49	90
401	10-10-10 ammoniator 170° cooled	10.00	3.23*	3.37	104
402	10-10-10 dryer 195° cooled	10.55	3.23*	3.44	106
403	10-10-10 ammoniator 186° cooled	9.15	3.23*	3.47	107
404	10-10-10 dryer 180° cooled	9.60	3.23*	3.43	106
405	10-10-10 ammoniator 202° cooled	9.80	4.35	4.08	94
406	10-10-10 dryer 175° cooled	10.15	4.50	4.10	91
407	10-10-10 dryer 175° 30 min., 160°	10.25	4.55	4.26	94
411	5-20-20 ammoniator 155° cooled	5.83	2.59	2.45	95
412	5-20-20 dryer 180° cooled	7.00	3.11	2.97	96
413	5-20-20 ammoniator 155° cooled	4.85	2.15	2.10	98
414	5-20-20 dryer 180° cooled	5.20	2.31	2.23	97
415	5-20-20 dryer 150° cooled	4.93	2.19	2.13	97
416	5-20-20 ammoniator 155° cooled	4.65	2.07	1.93	93

*basis 7¼ units N from UAL-B

TABLE 3

	Screen Analysis — % +4	−4 +10	−10 +20	−20 +35	−35	Yield* %
8-16-16 no added water	2	25	26	26	21	52
8-16-16 plus 2% water	3	43	19	24	11	64
8-16-16 plus 4% water	28	47	11	9	5	76
10-10-10 7.25 units N from UAL plus 1.7% water	19	47	10	13	11	69
10-10-10 all N from UAL plus 3.8% water	18	47	15	12	8	74
5-20-20 plus 6.7% water	1	29	23	22	25	52
5-20-20 plus 7.7% water	5	47	16	17	15	66
5-20-20 plus 9% water	11	39	19	20	11	65
5-20-20 plus 10.2% water	8	28	14	24	26	47

*Sum of (—4 +10) (—10 +20) and ⅔ (+4)

TABLE 4

6-24-12	— UAL-B	Lbs./Ton N	P_2O_5	K_2O	H_2O	NH_3
803	"Conc." super 48.5		388.6		8	—19
270	UAL-B 46.5	125.5			47	+85
462	R P super 20.0		92.4		42	—23
405	Muriate 60.0			243	6	
170	60°B H_2SO_4				39	—46
2110		125.5	481	243	142	—3
—110					—110	
2000					32	

both ammoniator and dryer, and cooled to air temperature, about 85° F, as rapidly as possible by air current from a fan. One sample from the dryer was kept at 160° F for 30 minutes. Another sample was stored at 140° F for 16 hours. The hot storage samples showed no greater urea hydrolysis than the cooled samples, and in none was the hydrolysis significant, or an important factor, as indicated in the following analyses (Table 2).

In these trials the desired or permissible particle size was considered to be -4, through 4 mesh, and +20, retained in 20 mesh. The fines, -20 are normally recirculated in the granulator, although they were not in most of the grades tested here. The oversize, +4 are normally run through a crusher, recovering about ⅔ in the desired particle size. The aim is to have as few fines as possible, and likewise to have a few oversize, since large granules are difficult to dry. Satisfactory granulation and yield was achieved for all grades, but the 5-20-20 was the least satisfactory. The following figures indicate the screen analyses and yields (Table 3)

No great technical difficulties were encountered in these tests. The granules were smooth, almost spherical, and in hardness and resistance to degradation were considered quite satisfactory.

Plant Scale Test

FOLLOWING the pilot plant tests, a plant scale test was made in a process closely resembling the TVA process, the essential units being an ammoniator-granulator, a counter-current dryer, and a cooler. Only one formula, 6-24-12, was tried with UAL-B, as follows:

Initial pressure of UAL-B was 25-30 p.s.i. Air was applied to increase to 50 pounds, and later to 70 p.s.i. No difficulty was experienced in "flashing" in the recording rotameter. A small amount of water, varied to suit the optimum granulating conditions, was added to the above formula through a suitable flow-meter. Granulation of 6-24-12 with UAL-B was begun at an initial rate of 15 tons/hour without difficulty. However, as soon as the fines were recycled, trouble was experienced in maintaining the proper moist "plastic" conditions in the mixer, resulting in increased fines and a progressively lower and lower yield of granular product. The difficulty was corrected by temporarily suspending the recirculation of fines, and by reducing the production rate to 10 tons/hour, then gradually increasing the rate to 15 tons/hour with re-introduction of the fines. When the proper equilibrium was established, granulating proceeded satisfactorily at the normal rate.

Once "on stream," the yield of the 6-24-12 with UAL-B was considered satisfactory, as indicated by screen analyses as shown in Table 5. (Yield is taken as the sum of the fractions -4 to +20, plus ⅔ of this +4 fraction.)

TABLE 5

		Screen Fractions, in Per Cents									
		+4	-4 +6	-6 +8	-8 +10	-10 +14	-14 +20	-20 +35	-35	Total	% Yield
6-24-12	UAL-B	2	13	21	24	7	10	19	4	100	76

Temperatures in the granulator-mixer were 180°-190°, and cooler exit material 124° F. Air temperature was 32° to 45° F. The cooler was not as effective as desired, since the heat transfer from granules is slower than from the conventional fine fertilizer, and because the low moisture content of the dried granules does not afford appreciable latent heat cooling. Increasing the air flow from the present rate of 5000 c.f.p.m. to 7500 or 10,000 would probably suffice. The efficient cooling of granular goods may well deserve some further attention to design on the part of equipment manufacturers.

Temperatures of the product as dumped in storage 11 a.m. were 114° to 120° F. Later in the day, 3 p.m., temperatures as low as 102° F. were observed. Over night, the exterior of the pile cooled, and the interior showed no temperature rise. The product was very free flowing, and free from pile set. Later observations showed less pile set than had previously been experienced with a similar grade formulated with an ammonium nitrate solution. A bag storage test was made with product less than one hour after granulation, piling 14 bags high, product temperature estimated at 100° to 105° F. At the end of three weeks there was comparatively little set. The slight form set was easily broken up, and the product was free flowing, free from

TABLE 6

		Lbs./Ton				
12-12-12		N	P_2O_5	K_2O	NH_3	H_2O
340	UAL-A 45.5	154.7			125	85
70	Urea 45	31.5				
316	Ammon. Sulfate 21.0	63				
412	R P super 19.5		81		(	
					(—52	70
372	Conc. super 45		167		(	
400	Muriate 61			244		
270	60° H_2SO_4				—73	62
2180		249.2	248	244	0	217
—180						–180
2000						37

TABLE 7

		Lbs./Ton				
5-20-10		N	P_2O_5	K_2O	NH_3	H_2O
230	UAL-A 45.5	105			84	57
960	R P super 19.5		187		(	
					(—78	124
500	Conc. super 45		225		(	
333	Muriate 61			204		
27	CaO-MgO					
100	60° H_2SO_4				—27	23
2150		105	412	204	—21	204
—150						—150
2000						54

TABLE 8 **Properties of "Uramon" Ammonia Liquors**

Total N	UAL-A 45.5	UAL-B 45.5	UAL-C 37.0
Composition			
Urea	32.5	43.3	26.5
Ammonium Carbamate	10.5	15.0	8.6
Ammonia	32.2	24.1	26.3
Water	24.8	17.6	38.6
	100.0	100.0	100.0

Total N	UAL-A 45.5	UAL-B 45.5	UAL-C 37.0
Vapor Pressure 100° to 104° F, p.s.i.	64	50	22
Salting Out Temp. F	5°	34°	6°
Lbs. Water/Unit of N	11.0	7.8	21.0
Lbs. Water/5 Lbs. NH_3	3.4	2.9	6.5

hard lumps. Granule hardness was judged to be quite satisfactory.

In another plant scale test, two grades, a 12-12-12 and a 5-20-10, were tried with UAL-A. The process was again a modified TVA design, with ammoniator-granulator and co-current dryer. There was no cooler. In the 12-12-12, 7.5 units of N were derived from UAL-A and 1.5 units from solid urea, this combination being equivalent to 9.0 units from UAL-B. In the 5-20-10, all of the nitrogen was derived from UAL-A, and a small amount of calcium oxide and sulfuric acid was included to provide additional heat. The formulas are listed in Table 6.

Granulation of the 12-12-12 was started at 10T/hour, and after one hour stepped up to 15 T/hour. Product from the ammoniator was estimated at 200 to 225° F, and had a moisture content of 5.8 to 7.1%. Product from the dryer had a temperature of 195° F, and a moisture content of 0.6% to 0.9%. Yield was estimated at 60 to 67%, —6+20 mesh. In general, good granulation was secured, but some difficulty was experienced in maintaining the optimum granulating conditions in the ammoniator-granulator. Control of water added was not as good as could be desired, and the amount of water in the formulation appeared to be too close to the critical amount to allow adjustment by added water. The product in storage was very free flowing, and had good granule characteristics. The surface of the pile cooled rapidly, but the interior appeared to increase in temperature (over 160° F) with a distinct odor of ammonia. Rehandling and moving the product brought down the temperature so that no further free ammonia could be detected. It was quite apparent from this test that to make and store this grade successfully cooling is necessary.

The 5-20-10 grade proved to be difficult to granulate with UAL-A (as it had been with other solutions), and repeated runs varying the amount of water produced erratic granulation, the material becoming alternately too dry or too wet. It was concluded that for this grade, the amount of urea (71.5 lbs./T) was insufficient for good granulation.

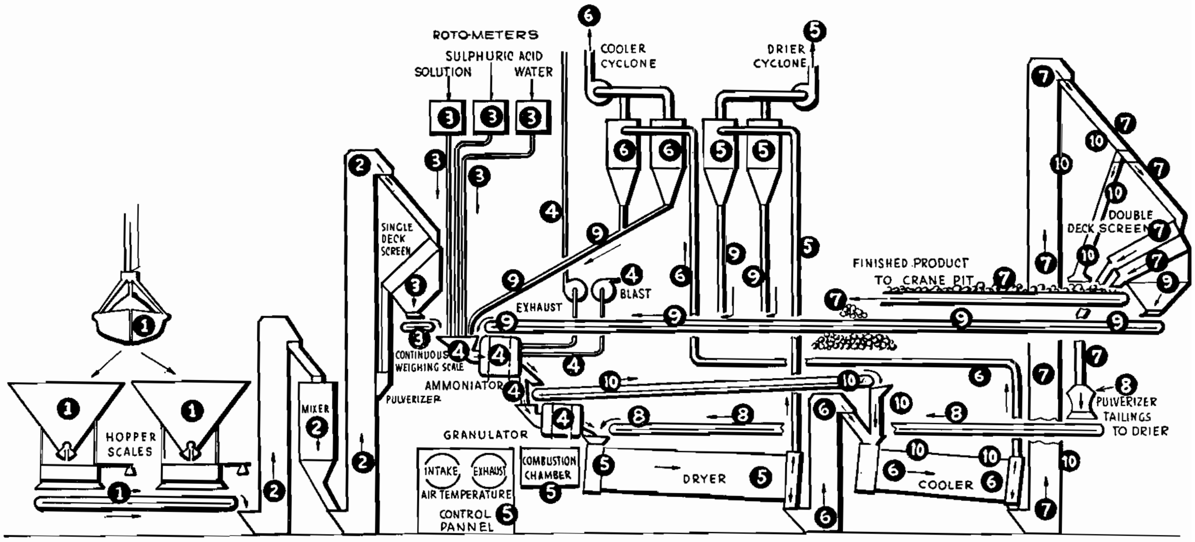
In another plant test, UAL-B was used in completely different equipment making 5-20-20 formulated with all N from UAL-B; a 5-20-20 with 3 units from UAL-B and 2 units from anhydrous ammonia, plus sulfuric acid; and a 12-12-12, 7.2 units from UAL-B and 4.8 units from ammonium sulfate, plus sulfuric acid. Granulation was satisfactory, but no data or analyses are available.

As indicated at the beginning, the work reported here was concerned principally with the question of urea hydrolysis under conditions of high ammoniation rates and granulation. The properties of urea do not preclude its consideration in granulating processes. From the standpoint of solubilities, urea is close to ammonium nitrate, and therefore affords a good agglomerating and adhesive effect. Only slight modification in formulating might be necessary in some cases to utilize UAL in any process now employing nitrate solutions.

In conclusion, we believe that this exploratory work has shown:

(1) that urea is stable in granulating processes involving high temperatures, provided the product is cooled before storage.

(2) that urea solutions afford satisfactory granulation from the standpoint of yield, desirable granule size, hardness of granule, appearance, free-flowing qualities, and comparative freedom from subsequent reactions that may induce excessive pile set or caking in bag storage.★★



Key to Flow Diagram

(1) Supply Hoppers and Batch-Weighing Equipment—Cranes equipped with 2½ yard (approximately 2½ ton) clam-shell buckets pick up the raw materials from bins in the crane-bay and load the overhead large supply hoppers. From these hoppers the required weight of each material called for in the formula is weighed into hopper scales and dumped on conveyor belts which empty into a vertical bucket elevator to lift the materials into the mixer for blending.

(2) Mixing and Screening Unit—All of the dry materials are assembled by batch method and thoroughly blended through a Sackett gravity 1-ton mixer. From the mixer each blended batch is dumped into a vertical bucket elevator and lifted to a single-deck Tyler-Niagara 4'x8' screen. The screen (4½ mesh) throws out all of the foreign material, sends the over size to a Sackett #15 pulverizer for recycling and conditions the batch for ammoniation. From the screen the blended conditioned batched materials dump into a supply hopper directly over a continuous belt weighing machine.

(3) Solid and Liquid Proportioning Equipment—The continuous operation begins at this stage. The batch blend of solid ingredients is converted to an accurately controlled Poidometer belt scale receiving its feed continuously from the supply hopper directly above. Rotometers and flowraters control continuously the flow of liquid nitrogen solutions, sulphuric acid, and water. The dry materials flow from scalebelt into the ammoniator and the liquids feed into the ammoniator under a deep cascading bed of solid materials.

(4) Continuous Ammoniator and Granulator—The rotary ammoniator and rotary granulator are T.V.A. developed and patent pending equipment. The ammoniator is 7 feet in diameter and 7 feet long, rotates at 8 revolutions per minute and is equipped with 15 H.P. variable speed motor. The granulator is 6 feet in diameter and 8 feet long also rotates at 8 revolutions per minute and equipped with 10 H.P. variable speed motor. Vapors, principal moisture generated by the chemical reaction in the ammoniator, are forced through a stack into the atmosphere by a suction fan equipped with 5 H.P. motor. Sometimes the mixture becomes too wet for rolling, and when this happens air is blown on the bed of material in the ammoniator for some drying. The fan used is equipped with 3 H.P. motor. Most of the granulation takes place in the ammoniator, and the granulator is used for additional rolling of the product.

(5) Drying Equipment—The damp granular product flows from the granulator by chute directly into the dryer. The rotary dryer is 6 feet in diameter and 50 feet long and is driven by a 50 H.P. motor. A co-current 15 million B.T.U. capacity Natural Gas-Fired combustion chamber feeds the hot gases thru the dryer. The first several feet of the dryer has directional flights to move the material away from intake of dryer, and from this point lifting flights move the material to the discharge end. Combustion air is furnished by a fan equipped with 20 H.P. Motor. Moisture is removed from the dryer through a stack to the atmosphere. Dual cyclones recover the fine dust. A 50 H.P. motor is used to drive the cyclone fan.

(6) Cooling Equipment—Material from the dryer empties into a vertical bucket elevator, which feeds the cooler. The rotary cooler is 6 feet in diameter and 40 feet long, and is driven by a 40 H.P. motor. A counter-current air flow is used for cooling. The cooler flights are similar to the drier flights. Additional moisture during the cooling process is removed through the stack to the atmosphere. Dual cyclones recover the fine dust. A 40 H.P. motor is used to drive the cyclone fan.

(7) Product Classifying Equipment—The cooler discharges onto a conveyor flowing the material into a vertical bucket elevator which lifts the product to a double deck 4' x 15' classifying screen. This screen gives us 3 classifications: Oversize, fines, and finished product. The oversize passes through a large pulverizer, equipped with 15 H.P. motor, and is returned via a belt conveyor to the dryer for reworking. The fines are then returned via a belt conveyor to the ammoniator and the desired product flows on a belt conveyor to storage pit for the crane to pick up to store to desired bin.

(8) Crushed Oversize—Pulverizer and belt conveyor taking the crushed over size to the dryer.

(9) Fines Return for Granulation—Fines from dryer and cooler cyclones and from classifying screen flow on conveyor belt back to the ammoniator for reworking.

(10) Ammoniating and Cooling Powdered Mixed Goods—Conventional powdered mixed goods is batch-weighed and the dry materials required for the formula desired are assembled and blended similar to the granular dry materials. The T.V.A. ammoniator is used for blending the dry materials with the required nitrogen solution. The granulator and dryer are not needed, therefore, this equipment is by passed. The finished ammoniated powdered mixed goods flows from the ammoniator onto a conveyor belt emptying into the cooler. From the cooler the product flows onto a conveyor belt to a vertical elevator by passing the classifying equipment used for the granular product delivering directly to the belt conveyor to take the product to the crane pit to be picked up by crane to deliver to desired bin storage.

AMMONIATION and GRANULATION AT FERTILIZER MANUFACTURING COOP.

By Albert Spillman

Fertilizer Manufacturing Cooperative
Baltimore, Md.

THIS business of granulation is no "Utopia". It takes a great deal of hard work, continuous research and experimenting to continue improving manufacturing methods, formulation and quality.

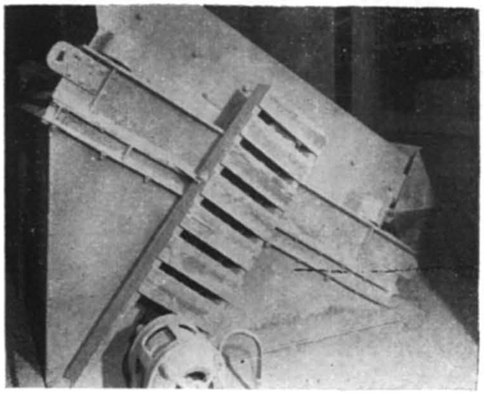
Although it was anticipated, Fertilizer Manufacturing Cooperative, Inc., had its problems getting started. For several weeks all kinds of difficulties were encountered, Viz: Material from the ammoniator was too wet, too dry, too much oversize, too many fines, too much recycle, chute plugups, pulverizer plugging, screen blinding, too much heat, too little heat, high moistures, not enough cooling, ammonia fumes, cyclone dusting, etc., etc. During this shakedown period, day by day, we were learning more and more about the behaviour of our equipment, finding trouble spots, eliminating previous operating errors, stationing our crew where needed, and getting straightened around on formulation.

Keeping accurate records of operations from day to day, during the startup period, was the real key to getting us started on full production by April 1st, 1955. We are continuing to keep records of each day's operation, showing grade granulated, formula used, water in materials, water added at the ammoniator, ammoniation rate, moisture driven off by drier heat and sulfuric acid, cubic feet of gas consumed per ton of finished product, temperatures, moistures from finished product, cooler, ammoniator, granulator, recycle fines, and oversize; also screen analyses before classifying, finished product, oversize, fines from classifying screen and cyclones. N.P.K. analyses are made once per shift, and the other tests are made periodically through the shift. This control and permanent record work, I am sure, has helped us to stay on the right track as much as any of the other major controls used in our process.

Since starting on full production, April 1, 1955 to June 30, 1955, one eight-hour shift per day, FMCI manufactured 14,000 tons of granular mixed goods. Four grades were made during this period. Most of the tonnage was 10-10-10 and 8-16-16 and a small tonnage of 6-12-12 and 6-18-18. Nitrogen solution carrying 22.2% free ammonia was used in the 10-10-10 and 8-16-16, and nitrogen solution carrying 16.6% free ammonia was used in the 6-12-12 and 6-18-18. Since July 1st FMCI has improved its production operation, using 22.2% free ammonia in 6% nitrogen formulas. Types of nitrogen solution used may have to vary from time to time, depending on the formula desired.

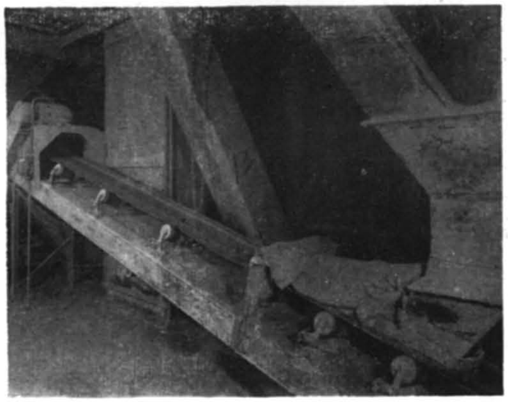
FMCI had no difficulty granulating the higher nitrogen grades 10-10-10 and 8-16-16 by adjusting the water added if the material from the ammoniator was too dry, or by not adding water, or taking out 25 lbs. to 50 lbs. of nitrogen solution and substituting sulfate of ammonia, if the material from the ammoniator was too wet. Lower nitrogen grades 6-12-12 and 6-18-18 were more difficult to granulate. We got nowhere with these grades until we added 100 lbs. of 60° Baumé sulfuric acid to give additional heat to help plasticity and also added considerable water. Since July 1st we have experimented with a coarse potash, almost 100% through 10 mesh and about 85% on 20 mesh, and find we can do a better granulating job with 6% nitrogen mixtures by using a combination fine and coarse or all coarse potash. We have done considerable experimenting with "Minco

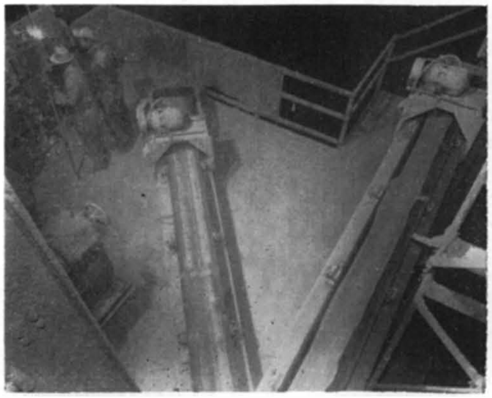
The 4′ x 8′ Tyler-Niagara single deck screen for screening the dry material batch before entering T.V.A. Ammoniator.



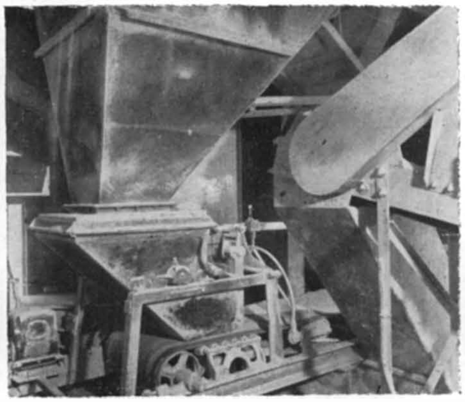
Twin dryer cyclones and the double deck Tyler 5′ x 15′ vibrating classifying screen.

Conveyor takes the fines from the cyclones and fines from the classifying screen back to the ammoniator for reworking.

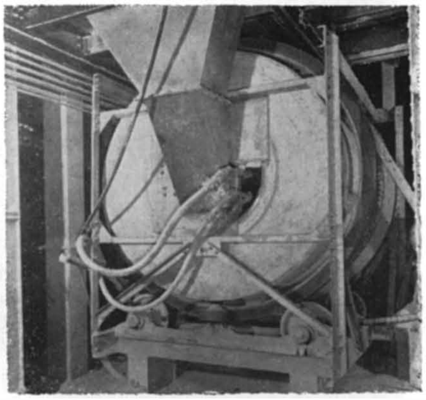




Another view of the ammoniating floor showing conveyor on right, which takes the finished screened product to pit for cranes to carry back to storage and conveyor.



A surge hopper carries the screened dry material and empties into a continuous poidometer for delivery to T.V.A. ammoniator on floor below.



The intake to the TVA ammoniator, showing piping for ammonia, sulfuric acid and water. The dry batch flows into the hopper from the floor above.

Conditioner", a fine clay dolomitic limestone and find this material helped granulation.

Most of the 14,000 tons of granular made from March 15th to June 30, 1955 was shipped within 48 hours after manufacture, and in some cases the fertilizer was shipped direct from production with no trouble on condition. However, several complaints on severe packing of 6-18-18 shipped during hot weather, July and early August were encountered. I believe we could have eliminated these complaints had the mixture been dusted with a fine clay at time of bagging. We plan to do considerable experimenting by dusting the mixtures with a fine clay at time of shipment to find out what improvement we can expect in mechanical condition when the goods are stored for long periods.

At the present we are on a one shift basis, working 8 hours per day. After each shift all of the equipment is thoroughly cleaned. This procedure is very important to keep operating with practically no "down time." Granulation at present is more of an art than a science. The man assigned to watch the continuous ammoniator must stay on the job at all times. He is the most important of the entire crew. All of the controls for feeding the dry material, solution, sulfuric acid, water, return fines and oversize belts are near the ammoniator easily accessible to the operator. This procedure permits the ammoniator operator to make the necessary changes within seconds.

As we approach the end of our first year of granular production, substantially over 40,000 tons of granulated mixed goods have been made in the following 7 grades: 6-12-12, 6-12-18, 6-18-18, 8-16-16, 8-16-16 carrying 3 units magnesium from sulfate of potash magnesium, 10-10-10 and 10-20-10.

In general, the process as shown in Figure 1 has given a very good account of itself in that the hourly production estimates made by the equipment manufacturer have been completely fulfilled, and in most cases have been substantially exceeded.

The flow diagram (Pg. 40) shows the ammoniating, granulating, drying, cooling and classifying equipment. The entire unit, building changes, foundations, all engineering, design and equipment was engineered and built by the A. J. Sackett Sons Co., Baltimore, Md.★★

THEORIES and PRACTICES in AMMONIATION

by Elmer C. Perrine

Allied Chemical and Dye Corp.
Nitrogen Division
New York, N. Y.

AMMONIA is the lowest cost nitrogen. Thus from the economic angle alone, it behooves management to use as much of this product as practical — whether it be ammonia contained in the ammoniating solutions or as anhydrous ammonia. In addition to the price advantage, many persons believe that much of the good condition of ordinary fertilizer is achieved through high rates of ammoniation, and that extreme rates are almost mandatory in granulation. Also the heat of the ammoniation reaction is desirable in more cases than in granulation alone. We are not unmindful of the possibility of reversion in the P_2O_5 and of some other disturbing features related to high as well as to low rates of ammoniation.

If anhydrous ammonia alone could supply all of the nitrogen required in all of our formulas, the problems would be greatly simplified. It so happens that the second lowest cost nitrogen is derived from chemicals that are logically in combination with anhydrous ammonia. This combination forms the very popular ammoniating solutions. The makers of these solutions are constantly trying to meet the demands of performance that are being put on their products.

One of the most significant developments in the fertilizer industry in recent years has been the doubling of the old long-time rate of 3 pounds of ammonia per unit of P_2O_5 in 20% superphosphate. As more suitable ammoniating media are developed and some of the shortcomings are circumvented, the demands focus more on getting a few more pounds of ammonia into the superphosphate. This is just about the number one problem; and it is through an understanding of the theories and practices of ammoniation that this problem can be solved.

Ammoniation can be effected a little more readily with nitrogen solutions than with anhydrous ammonia . . . this discussion, however, deals with ammonia regardless of its source. The principles remain the same although it is necessary to vary the techniques a little in handling anhydrous ammonia. "Superphosphate" in this article refers to a normal 20% product. The same principles apply to the ammoniation of triple superphosphate, but the practical limit of ammoniation seems to be about half that for the 20% material. It should be noted too that the presence of many large particles reduces the ammonia take-up seriously, as shown in a study on particle size by the USDA.

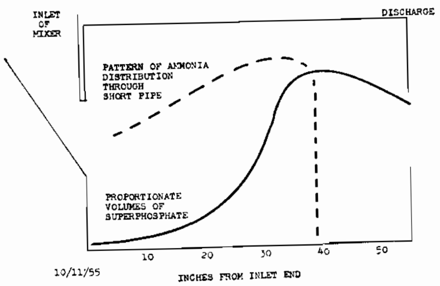
About the same amount of ammonia can be added to a given amount of superphosphate, whether the superphosphate is in a nitrogen-phosphorous base or in such dilute formula as the 3-12-12. It is necessary only to provide a little mixing of the dry ingredients prior to introducing the ammonia into complete goods.

Normal superphosphate has such a high affinity for ammonia that almost any scheme that brings the two materials into reasonable proximity for a short time will result in 2.5 to 3.0 pounds of ammonia being taken up per unit of P_2O_5. With 20% superphosphate the theoretical limit is 9.6 pounds of ammonia plus about one pound of each three pounds of free acid present. Until 6 or 7 years ago, ammoniating arrangements were such that in the initial steps, most of the ammonia was taken up by less than half of the superphosphate. Some operators were never converted to modern techniques, and an apalling number have gone back to the old methods because of their failure to understand the newer developments.

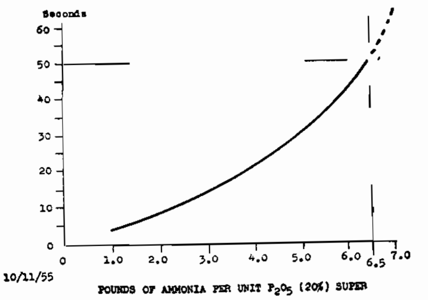
The reactions at the lower levels are almost instantaneous and permanent. Today, however, we are attempting to make all of the superphosphate do its share of the work.

Superphosphate probably takes up ammonia beyond 3 pounds per unit of P_2O_5 more slowly than before this point is reached. The gas will react more slowly with dry materials than will the liquid from which this gas escaped. We certainly must provide means for reacting this gas when we go beyond 3 pound rates of ammoniation at the tons per hour rates that pay off. This requires intimate contact, combined with either continual or repeated exposure of the superphosphate to the gas. As the task of reacting the gas is the most serious of the ammoniating problems, it is advisable to generate the gas only as fast as it can be taken up in the reactions.

PATTERN OF DENSITY OF MATERIALS IN ROTARY MIXER



TOTAL TIME IN SECONDS
REQUIRED TO APPLY 6.5 POUNDS OF NH_3 PER UNIT P_2O_5 (20%) SUPER



These production problems are almost the same with both nitrogen solutions and anhydrous ammonia. They differ only in degree and not nearly as much in degree as is generally supposed.

These principles are more easily understood when applied to the conventional rotary batch mixer. The TVA type of continuous ammoniator uses different means to implement the principles. But the principles will help in getting the very best performance regardless of the type of ammoniator used.

All of the finely screened materials should be in the mixer before ammoniation is started. It is usually best to use the full capacity of the mixer. This will assure better contact with the ammonia and provide for maximum "scrubbing" of the gas by the showering mass. We have found no improvement in ammonia take-up nor in condition of the goods when the superphosphate is ammoniated in the mixer prior to admitting the balance of the materials. Some operators attempt to ammoniate the mass collectively, or the superphosphate separately, as the materials are sliding into the mixer. How could an ammonia distributor pipe be drilled to successfully achieve this feat?

The distribution of the mass in the rotary mixer has probably become constant after one revolution of the mixer. You can watch a small glass mixer and see this. Most continuous ammoniators rightly provide for some mixing ahead of the ammoniator to conserve its capacity for the important function of getting the ammonia into the superphosphate.

It is possible to locate the holes in the ammonia distributor pipe with a pattern that will introduce the right amount of ammonia for the amount of superphosphate that exists along every inch of the length of the mixer. And it is extremely important to do this. In the conventional mixer this means a pipe that extends through the entire length of the mixer. In some cases, it is necessary to offset a portion of the end to clear the movements of the discharge door. In some mixers, it means cutting off some of the width of the extremely wide flights to allow clearance between the flights and the discharge door. Some mixers are so long that it is difficult to support the pipe.

All of these problems are being met, and quite easily in most mixers. But it would pay to throw away even a costly new mixer that can not be made to accomodate the higher ammoniation rates . . . for that is indeed a costly mixer. Were it not that stainless steel does the job perfectly well, it would be profitable to make a good ammonia distributor pipe even of gold or platinum for the savings in the cost of nitrogen can run over $200 per operating day.

Actually, this distributor pipe is a metering device or manifold whether it is so recognized or not. And it had better be a good one. It contends with requirements as exacting as many of the manifolds in the chemical industry. Both types should be given the same close attention that they deserve.

For years enough has been known about the requirements of a distributor pipe to justify making the next one of stainless steel. We admit that there is room for improvement. The holes of an ordinary steel pipe soon corrode, thus destroying any ability to correctly meter the ammonia to the desired points in the desired volume. So then you have to reduce the amount of low-cost nitrogen that can be used, buy a dozen gas masks, and start on the hunt for that nitrogen that the state chemist failed to find in your fertilizer.

The problems of gas are greatly reduced when the ammoniating medium is applied in its liquid state in such a manner that every particle of superhosphate can be contacted at least once by the liquid. It is quite conceivable that in batch rotary mixing the particles are returned several times for repeat baptisms of ammonia, thus effecting an accumulative, or multi-stage ammoniation. It does not require much imagination to believe that this is also happening in the TVA unit, and just as effectively.

Judged by the speed by which moderate rates of ammoniation are performed with good equipment, there is much evidence to indicate that as much as 4 pounds of ammonia can be taken up from the solution while it is still in solution. The duration of the act of ammoniation for these moderate rates need not exceed 25 or 30 seconds.

Atomizing sprays have been tried. If it were practical to retain the fogging effect that is so easily demonstrated outdoors, we would be ammoniating at much higher rates. But the very heavy showering curtains of fertilizer blanket any known atomizing spray. The effect is that of a very few holes in a pipe and those very poorly placed.

One continued slot for the length of the pipe with additional short slots to effect a good pattern was tried. This failed, probably because the very thin sheets of liquid lacked driving power for penetration. The ammonia was deposited in a thin layer on the surface of the thick curtains of material. If time were of no concern, this, as well as many other discarded methods, would suffice.

Much more superphosphate is contacted in a short time through the modern distributor pipe by its 12 to 30 powerful streams located strategically, each of which can penetrate the heavy loose curtains and disperse inside for yet more contact. "It delivers the milk along the street as called for by the concentration of milk drinkers along the street."

If no scale or rust can enter the system, the holes in the pipe may be 3/16 inch drilled in one row on one side of the pipe. This size of hole permits twice the number of points of contact that will result from ¼ inch holes. There is no justification for using ordinary steel in such a vital place as the distributor pipe. Where any ordinary steel exists elsewhere in the system, the holes in the pipe should be ¼ inch, even though the pipe itself be of stainless steel.

Even the most ardent supporter of the principles which have been outlined will concede that gas is a certainty at 5 pounds of ammonia per unit of P_2O_5 in 20% superphosphate and probably at a lower level. At the popular high rates of ammoniation and of plant production the problems of gas take on magnitude. No mixer is tight enough to hold the gas if provisions are not made to take care of it inside the mixer as it is generated. Any successful high rate ammoniating system surely does generate gas. The unsuccessful ones release it to the atmosphere before it can be reacted.

There are two factors which make it necessary to add ammonia at rates above 4 or 5 pounds progressively more and more slowly. One: since the mass of the material into which the ammonia is injected is getting hotter and hotter, a higher portion of the ammonia is being transformed into gas. The reaction of gas with the dry super is slow. Two: the reaction of any form of ammonia with superphosphate is apparently slower as the reactions reach the upper practical limits.

Cascading superphosphate through the confines of a machine in the manner as is done in the rotary mixer is a very fine means of scrubbing ammonia out of the atmosphere, whether that atmosphere contains 1% or 99% ammonia. Even the more efficient of the present modern systems do not give wholly uniform contact between the super particles and the ammonia while the ammonia is in the liquid stage. The use of gas, unintentional and troublesome as it is, does permit evening up the irregular or spotty work of the liquid. Gas seems to be the most feasible method of getting uniform exposure.

Provisions For Reacting Ammonia Gas

WITH the liquid stage accounted for, we can now turn our attention to the problems of scrubbing the ammonia gas from the atmosphere inside the mixer. The mixer, it must be remembered, is essentially an open or at best a non-pressure machine. Conditions are such that there is a vaporizing action in one sense, and there can be an equal scrubbing action if operations are geared to it. The problem is to limit the production of gas to the ability of the cascading superphosphate to pick up the gas. This takes time. And there is time for this action. Some mixers are under partial suction from a ventilating system, others from the dryers connected by closed chutes. Under these conditions, the suction will pull the ammonia gas off before it has had time to react fully. Losses have been detected at more than half a unit of nitrogen, to say nothing of air pollution.

There is time for a good job of ammoniation if we force the system at its top speed during the early part of the cycle when the ammonia take-up is in fact very rapid. Beyond 6.5 pounds any further take-up is so slow that it may be uneconomical.

See chart "Total Time in Seconds——"

There is too much tendency to rob the ammoniating cycle of much of its valuable time by improper use of time. The foreman is thinking of only the most obvious point in the mixing cycle when he summarily cuts down on the ammoniating time by forcing all of the ammonia into the mixer at a uniform rate. Some operators will continue to mix the mass for as much as a minute after the ammonia has been dumped unceremoniously into the mixer. Most of the gas is forced out of the mixer as fast as it is made and it can never be recalled no matter how long the mixer is subsequently run. It takes something more elaborate to get nitrogen out of the free air.

Only half of the time that is absolutely wasted in this after-mixing is necessary to accommodate the gas as it is being generated. It is acceptable practice to start discharging the mixer immediately after all of the ammonia has been added, if it has been added correctly. The goods are being mixed in the meantime. Good practice requires no more than 50 or 60 seconds total elapsed time for mixing and ammoniating as these operations are simultaneous.

At 6.5 pound rates the first four pounds of ammonia per unit of (20%) superphosphate can be taken up at the average rate of one pound in 5 seconds. The last half pound is likely taken up at a rate of a pound in 20 seconds, or only one fourth as fast.

Continuous Ammoniation

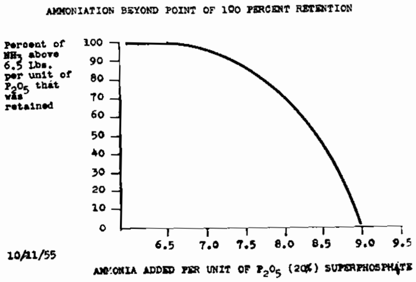
THE TVA type of continuous ammoniator is a marvelous machine. It is a great boon to granulation, where its characteristics are rendering great service in a branch of the fertilizer industry that has long needed such a device. Practically every phase of granulation can be performed better continuously than batchwise. There has always been the serious problem of converting from perfectly good batch ammoniation to the logical continuous steps of granulating, drying, and cooling.

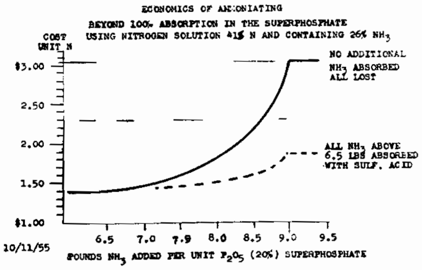
It can be reasoned that the TVA type of ammoniator functions at as high a rate of ammoniation as the rotary without regards to the foregoing principles, particularly as concerns the handling of gas. It is clear that a very effective accumulative ammoniation is achieved and that quite a long pipe is required to provide both intimacy and time of contact. The requirements of the distributor pipe in the TVA ammoniator are sharply defined. This is demonstrated by the poor results that are obtained from some of the locally contrived pipes. Changes in the excellent original design brought about by corrosion or abrasion will destroy the original ammoniation pattern. Several fine papers have been presented to the industry by persons who developed the TVA ammoniator, and by those who have put it to the crucial tests of practical use. It is in tribute to them that we dwell briefly on this machine and not in any failure to recognize its great worth. They are doing excellent work in ammoniation as well as in granulation. Most of the failures in the TVA ammoniators that have come to our attention stem from the same causes that produce failures in many good devices, that is the men who operate and maintain them.

Excessive Application of Ammonia

WHERE local conditions permit, there has grown up a new concept of ammoniation. It may be called "excessive ammoniation" or, where the operator understands what is transpiring, as most of them now do, it can be termed "calculated loss."

In equipment that can be operated to yield essentially 100% reten-





tion of 6.5 pounds of ammonia input per unit of 20% superphosphate, it is possible to hold as much as 8 pounds when 9 pounds are applied. This involves a loss of one pound of ammonia. It means that 8 pounds is about all of the ammonia that it is practical to hold without the aid of acid. The addition of 10, 11 or even 12 pounds fails to give any more than 8.0 pounds retention. How much ammonia is taken up as gas in this scheme?

Acid—Its Use and Mis-Use

MOST of the mixers, including the TVA continuous, are capable of retaining 100% of an ammonia input of 6.0 to 6.5 pounds of ammonia. Finely divided triple superphosphate is reckoned at about half this rate while the large particle sizes in some triples limit the take-up still further. Any assist from acid before the full potential of the superphosphate is satisfied is like a healthy man using a crutch. Beyond the point of 100% retention in the super, sulfuric acid is used at the rate of about 3 pounds of acid for each one pound of ammonia, unless there is some valid reason for using more acid for additional heat in granulation. It is too easy to control fumes by using acid. Formulae that can justify no more than 40 pounds of acid are observed to degenerate through poor maintenance of equipment to such a degree that three times as much acid is being used.

These theories and practices may be discounted heavily by some since the methods have permitted us to reach only two thirds of the way to complete ammoniation and have been stalemated on this plateau for 6 or 7 years. Nevertheless, acceptance of these principles doubled the rates of ammoniation overnight. Somebody will eventually discover more potent medicine and break through this barrier of 6.5 pounds.

Until then, these simple principles are leading to the use of a very good amount of ammonia in many plants.★★

Driers and Coolers in Making Fertilizer

By G. Halldorson

IN the fertilizer industry, the dryer and cooler, which is of the greatest interest because of capacity, simplicity, compactness and low first cost, as well as general economy in operation, is the rotary direct fired cocurrent dryer and the countercurrent rotary cooler.

Because of the heat sensitivity of most fertilizers the temperatures to which they can be subjected, in a dry state, are rather low. This limits the temperature of the drying air, which can be used in a countercurrent dryer, and thereby the heat which a certain amount of air can carry. It follows that for a certain dryer volume, fan size and dust collector size, the capacity of a counter current dryer is much lower than in a cocurrent dryer of comparative size, since in a cocurrent dryer a much higher temperature can be used without damaging the wet fertilizer, entering at the same end as the hot air. While a film of moisture encases each particle, the actual material temperature stays at around 200 degrees, or near the wet bulb temperature, even if the temperature of the air is over 1000 degrees.

There may be certain cases, where plasticity in the wet state makes high temperature objectionable. Such materials may call for different kind of a heat treatment. However they are then the exception to the rule.

The reasons for a selection of a countercurrent cooler, over a cocurrent one, are basically the same, in that a certain amount of air will absorb more heat when flowing through such a cooler since there is no limitation to how much the material may be cooled. On the other hand, the object is to arrive at the lowest possible material temperature at the discharge end. The only objection to a counter current cooler is dusting at the feed end, but this problem can be solved. Mr. Halldorsson reviewed the effects of "tilting" the drier or cooler, diameter size, material-air contact time, etc. He said that there is a limit to both the diameter and the degree of tilting. Because of road and railway regulations it is difficult to ship larger diameter than 11 to 12 feet. With excessive uphill tilting the material tends to back out of the inlet end or stay too long in the first hot part of the dryer. Both these considerations have been accounted for in the first Fertilizer Dehydro-Mat dryer installed some moths ago in Joplin, Iowa.

Mr. Halldorsson, who is the inventor and has obtained a U.S.A. patent on this new type of rotary dryer, licensed to Edw. Renneburg & Sons Co., Baltimore, described the functioning of this dryer, which has created considerable interest in industrial circles and between engineers and received wide publicity.

When discussing dryer capacities

Mr. Halldorsson described how formulas in the *Chemical Engineer' Handbook* would come up with a Volumetric Heat Transfer Coefficient of around 4 to 5 in several cases he had checked, where actual experience in the industry would show a figure of 15, or three times as much capacity for both coolers and dryers.

With the first Dehydro-Mat dryer operating in the Fertilizer Industry, coefficient from 25 to 30 and even 35 have been been obtained. The coefficient will of course vary for different particle sizes and materials.

In the past, almost without exception, dryers and coolers of this type have been mounted in an inclined position intended to help move the material towards the discharge end . . . and in concurrent units this movement corresponds to both the force of the air flow and gravity. In a counter current unit, however, this tilting was logical in order to overcome air resistance. It is not necessarily logical for the concurrent unit.

On the contrary, when trying to obtain the greatest possible drying capacity of a certain dryer or dryer volume, where the inlet air temperature has already reached a maximum, the limits are reached when the air flow has become so great that it takes the material too rapidly through the dryer, resulting in a too short drying time, and a wet material being discharged.

One way to overcome this difficulty is to increase the length of the unit, thereby increasing the time of contact. This, however, is uneconomical, since increased air velocities increase the material velocities at an accelerating rate. Another means of increasing dryer capacity is to increase the dryer diameter, which is an economical and space saving method. The third possibility is to adapt a horizontal position, or even a backwards or uphill degree of tilting, making the material drop backwards into the air stream, thereby increasing the drying time.

Through the realization of the importance of the effect of the air velocity upon the material movement, this dryer is designed with cross sections of different diameters. In an ideal dryer, the material should move fast through the hot inlet zone, where surface moisture is quickly evaporated, the inlet zone is therefore narrow, with a small cross sectional area. The air velocity therefore is very high at this point and moves the material with a considerable force. After the material leaves this hot section of the dryer, it enters a section with gradually decreasing temperatures, where it needs to stay for considerable time in order to dry through. Therefore, this section has a large diameter and cross sectional area, with low air velocities and low material velocity. Thus the material stays in long enough to be completely dried. It is finally discharged through a narrow section. Tires and gear can be mounted on the narrow sections, and breaching can be limited in size and cost.

Finally, the whole unit is mounted horizontal or in an uphill position.

With a 34 foot long Dehydro-Mat throughout, capacities of as much as 70 tons are reported with a steady average production of 40 tons base material per hour, 24 hours a day.

In discussing coolers Mr. Halldorsson mentioned that for every percent of moisture evaporated, based on material weight, a temperature drop of approximately 30° could be expected. Finally he described a new type of fertilizer cooler which he has designed, and which is expected to be non-dusting. While it is a counter current unit, it has a co-current inlet end. Patents are in process of being applied for. He also described a possible combination Dryer-Cooler, where the cocurrent principle is adapted to the dryer end, and the counter current principle to the cooler end, ideal for both processes.

The first of these new type coolers is now being built for the Davison Curtis Bay Plant, who are also installing a Dehydro-Mat dryer. Another Dehydro-Mat is starting up in Perry, Iowa. In Texas, three Dehydro-Mats are being started on ammonium nitrate.

When discussing dryer specifications Mr. Halldorsson suggested that it would be practical always to refer to actual dryer capacities in terms of throughput, including recycle, rather than in terms of base material.

MORE ON DRIERS

By E. Leister

Continuous granulation-ammoniation at two Virginia-Carolina plants was described by E. Leister, Edw. Renneburg & Sons Co., Baltimore, who outlined the combination operation, giving details on each unit. "A wide range of flexibility can be built into a continuous combination unit," reported Mr. Leister, as he listed the following advantages:

a. Any retention time can be had for the varying grades of material, by the use of retaining rings or the variable inclination feature.

b. Speed of rotation can be increased easily, or decreased by the use of variable speed drive, or by changing a V-belt sheave.

c. Bed depth can be controlled, which can have a great effect on the loss of ammonia.

d. The unit is designed to permit use of any of the following: anhydrous ammonia, nitrogen solutions, sulfuric acid, phosphoric acid, steam, hot or cold water, compressed air, fresh air.

The ammoniator-granulator was described as a cylindrical shell 7′ in diameter and 14′ long. This, according to Mr. Leister, is the size required for 20 ton per hour capacity in most grades of fertilizer. "Base" materials and fines are fed into the first, or ammoniating-section. Acid is fed into the second half of the ammoniating section. From here the material spills into the granulation section. Granulation can be increased or assisted when hot or cold water (in a fine misty spray) is added to the rolling material in the granulation section.

Mr. Leister gave complete details of the Renneburg ammoniator-granulator, listing operation data and technique. He reported several features of the unit which allowed for better granulation and more complete ammoniation. One practice he suggested to the production group was: screening the product before cooling . . . then cooling only the finished product. "This," he said, "offers the advantage of recycling hot fines, and a system in which the product will be cooled to a lower temperature." The practice also offers the possibility of eliminating the cooler dust collector. "The big advantage would be to a fertilizer manufacturer who has a small cooler, and wants to produce granular fertilizer at a capacity above that of the cooler. He can save the cost of a cooler, if his cooler is not required to do the unnecessary job of cooling the fines and oversize."

Mr. Leister reported that considerable success has been obtained with the use of a vertical disintegrator to produce a more uniform particle size by impact mixing. The disintegrator, he said, is able to delump the mix, and impact mix the various ingredients, and yet produce a minimum of fines . . . thus a uniform product is obtained, which should result in better ammoniation, and possibly better drying action.

A continuous combination ammoniator-granulator, said Mr. Leister, permits the nitrogen to be introduced into the materials at a slow rate, thus a high total ammoniation can be reached with relatively little loss of nitrogen and fumes.

A Fertilizer Graining Agent

By M. W. Thomas

THE production and properties of a natural colloidal kaolinitic kaolin were reviewed by M. W. Thomas, Thos. Alabama Kaolin Co., who told his audience that the product has been found a useful and economical agent in pelletizing, or as a graining agent in the production of fertilizer . . . or as a diluent in insecticides. The material in crude form, said Mr. Thomas, is very pure, containing a small percentage of iron . . . less than 1 percent, and averaging approximately ½ percent. It is low in alkalies, soda, and lime, and contains no mica.

The chemical analysis of kaolin shows it to be 99.6% a pure kaolinite . . . and the x-ray pattern gives a true kaolinite pattern. Properties are as follows:

bulk density (lbs./cu. ft.)
  loose: about 20 lbs.
  packed: about 41 lbs.
porosity: 130%
specific surface: cm^2-22,700
specific gravity: 2.62
particle type: crystal rectangular plates and crystal aggregates
limiting particle size: .01 to 50 microns
absorption of moisture from air when exposed in open vessel at average room temperature with "Tako" dried to absolute zero:
  24 hrs., 9/10 %
  7 days, 1 %

The kaolin is mined by selective methods and transported to a processing plant, where the crude ore is air-dried, passed through mills and grinding equipment which transform the fine material to a product with a flat rectangular surface. This form of the product is reported to be particularly suitable to blending in the insecticide field, and to pelletizing in the manufacture of fertilizer. The finished product generally has a free moisture content of .7 to .8%.

In discussing its storage properties, and moisture content, Mr. Thomas reported that in a test case, "Tako" (tradename for kaolin processed by Thos Alabama) stored for a period of one year showed no change in flowing properties . . . had not caked or packed. The moisture content increased only slightly, from .7% to 1%. He reviewed also the production methods of mining and processing "Tako" for distribution, and presented data on physical properties and particle size.

COMMENTS ON SCREENS

Wayne King

THE ROLE OF POTASH

Edwin Kapusta

In connection with particle size, Wayne King, W. S. Tyler Co., Baltimore, presented some factual information on the effective use of screens in production of fertilizers. With the increasing interest in granulation . . . and granular fertilizers, production men at the Round Table presented several questions to Mr. King on determining product size, and optimum angles of screen installation.

Edwin C. Kapusta, U. S. Potash Co., New York, presented some ideas on the role of potash salts in the ammoniation and granulation of mixed fertilizers. "Although dating back in origin many years," said Mr. Kapusta, "the greatest progress in the development and application of granulation processes has been made in the past few years. The popularity of granulation is evident by the large number of plant conversions and construction of new units currently taking place in the industry. It is estimated," he continued, "that over 100 mixed fertilizer plants either have installed or are installing some form of granulation equipment. Manufacture of granular mixed fertilizers may approximate up to 15 percent of the total domestic production of mixtures in the coming fertilizer year."