

# Granulation of Mixed Fertilizers in Experimental Equipment and Determination of Storage and Drilling Characteristics of the Products

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Small-scale equipment was designed to ascertain the optimum granulating conditions of fertilizers and to determine the quality of the finished products. Results of the performance of the equipment on 10 different grades of mixed fertilizer are given. The moisture requirement for optimum agglomeration of mixtures decreased with increasing ammonium nitrate content and increased with increasing superphosphate content. Caking tendency of the granular products decreased with decrease in moisture content, increase in particle size, extension of curing time, and coating of the granules with synthetic calcium silicate. The period of time that granular mixtures remained drillable at 75° F. and 88% relative humidity increased with increase in superphosphate content. The small-scale granulation equipment provides an economical way of determining optimum processing conditions in a short time with small amounts of test materials.

FERTILIZER MATERIALS, in large quantities, and considerable time are often consumed in commercial plant-scale trials to ascertain the optimum conditions for granulating new fertilizer formulations and determining the quality of the finished products. The use of small-scale continuous, processing equipment for determining the agglomeration characteristics of mixtures, with supplementary equipment for measuring the drillability of the products, largely eliminates the undesirable features of plant-scale exploratory work on new formulations.

This paper describes the small-scale equipment, procedure, and processing conditions used in granulating several hundred pounds each of 10 different mixed fertilizers and gives the results of bag-storage and drillability tests on the products. This study is part of a broad research program on the preparation of high-analysis fertilizers of improved physical condition being conducted in cooperation with the Tennessee Valley Authority. Previous papers have included studies of the effects of formulation on granulation of mixed fertilizers (3), the relative effectiveness of granule coating agents (5), and the pilot-plant granulation of high-analysis fertilizers (4).

## Materials

Typical screen analyses of the solid raw materials used are given in Table I. In this paper the term mesh refers to the Tyler standard screen scale. The ordinary superphosphate (20% phosphorus pentoxide) was a den-cured material made with virgin acid. The triple superphosphate (48% phosphorus pentoxide) was made with furnace

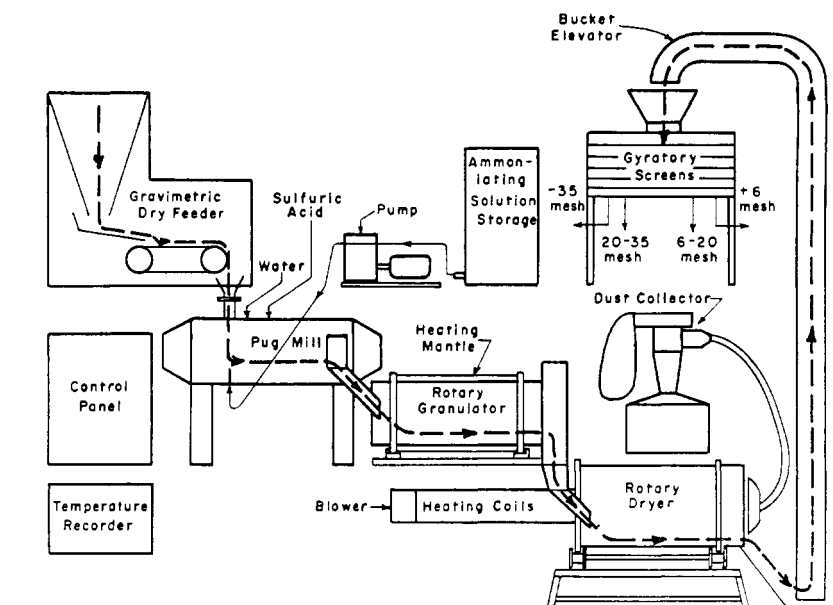


Figure 1. Small-scale continuous granulation equipment

acid in a funnel mixer and was pile cured. The potassium chloride (60.0% potassium oxide), ammonium nitrate (33.0% nitrogen), and by-product ammonium sulfate (20.0% nitrogen) were crystalline materials. The ammoniating solution contained 40.6% total nitrogen, 65% ammonium nitrate, 21.7% neutralizing ammonia, and 13.3% water. The sulfuric acid was a technical grade diluted to 90% concentration.

## Small-Scale Equipment and Procedure for Granulation

The small-scale equipment described here is intended as a research tool and not as a model for full-scale granulation plants. It consists of a continuous gravimetric feeder, ammoniating solu-

tion tank, solution pump, pugmill, rotary granulator, dryer, conveyor, and gyratory screens (Figures 1 and 2). The design permits measurement and regulation of the temperature and moisture content of the mixture during continuous processing.

The gravimetric feeder consists of a hopper, a vibratory feeder, and a constant-speed, constant-weighting, belt conveyor. The premixed dry materials are fed continuously to the inlet end of the pugmill through a chute.

Plastic tubing carries the ammoniating solution from a tank through a pump to the inlet end of the pugmill. Inserted in the line between tank and the pump is an ice-cooled trap to settle any foreign material and reduce vapor-

zation of ammonia in the line to the pump. The solution pump is a valveless, peristaltic-action pump, operating with a series of cam-operated fingers that press against the tubing in sequence, imparting positive unidirectional movement to the solution. The solution rate is adjusted by varying the size of tubing and cyclic frequency of the fingers. Ammoniating solution is introduced through two 1/4-inch stainless steel tubes, one below each shaft, extending through the bottom of the pugmill. Each tube is capped and has two 1/32-inch holes, one located 7/8 inch from the bottom of the pugmill directed toward its discharge end, and the other 1 5/16 inches from the bottom directed toward the side wall.

The function of the pugmill is to mix the premixed dry materials with ammoniating solution, sulfuric acid, and water to facilitate ammoniation of the superphosphate and distribute the plant nutrients and moisture throughout the material. The pugmill has a stainless steel lining and has inside dimensions of 10 × 20 × 9 1/4 inches. It has two horizontal counter-rotating stainless steel shafts each with 11 pairs of 2-inch blades spaced 2 inches apart at a 63° angle to the axis of the shaft. The last two pairs of blades on each shaft are reversed to discharge material through the side gate 3 1/4 inches above the bottom. Nominal retention time in the pugmill is 1.75 to 2 minutes at a throughput of 200 pounds per hour. In making some mixtures, the pugmill was air cooled by a current of air from a fan to prevent overagglomeration.

Water and sulfuric acid flow to the pugmill by gravity in fine streams from two constant head bottles. Sulfuric acid is added at a point 9 inches, and water, 12 inches from the inlet end at the top center of the pugmill. The rates are controlled by adjusting the hydrostatic heads and determined by weighing the liquid delivered per unit of time.

From the pugmill the mixture flows through a 2 1/2-inch inclined chute to the granulator. The granulator is a 12 × 36 inch steel cylinder containing 2-inch retaining rings and 1-inch longitudinal lifting flights. The temperature

in the granulator is controlled by heat supplied from an electric mantle around the cylinder. It was necessary to install a pneumatic atomizing nozzle in the inlet end of the granulator to obtain granulation of some mixtures. The rate of water addition is controlled by adjusting the water and air pressures in the pipe ahead of the nozzle. When water was sprayed in the granulator, a considerable amount of material stuck to the flights and retaining rings and formed an insulating coating on the cylinder wall which reduced rolling action of the material bed and decreased heat input. Occasional chunks of material breaking off the wall formed oversize and increased the heat input, which disturbed agglomerating conditions. To correct this, the flights and retaining rings were replaced by a scraper and a 3-inch retaining ring at the outlet end. A comparatively thin coating of hard dry material then formed on the wall, but temperature control and rolling action were improved. The granulator rotates at 16 r.p.m. and is slightly lower at the discharge end. Average retention time at a throughput of 200 pounds per hour is about 8 minutes with a 2-inch retaining ring and 11 minutes with a 3-inch retaining ring. The degree of agglomeration is controlled in the granulator by adjusting the temperature and/or water addition. The temperature of the mixture leaving the granulator is usually maintained at 194° to 212° F.

The granulator discharges through a 5-inch chute to the dryer. The dryer is a 20 × 42 inch steel cylinder with six 2-inch longitudinal flights. It rotates at 14 r.p.m. and is slightly lower at the discharge end. Air heated by electric coils may be blown through the dryer when necessary, and the degree of drying is varied by changing the retention time with the use of different size retaining rings at the outlet end. Owing to rapid heat transfer from the small amount of material produced, no cooler is necessary in this small-scale equipment. However, when artificial drying is not required, the dryer acts as a cooler. Products having high moisture contents were purposely made in order that variation in moisture

content of each material used in bag-storage tests could be obtained by further drying.

The granulated material from the dryer is lifted by bucket conveyor to a continuous laboratory-type gyratory screener equipped with 14 × 17 inch screens approximating 6, 20, and 35 mesh.

The exit gas from the dryer passes through a small cyclone dust collector.

Iron-constantan thermocouples and a 6-point potentiometer recorder are used to measure and record temperatures of the dryer-inlet air, ambient air, and the material in the pugmill, granulator, dryer, and solution tank.

Samples for the determination of moisture are taken of the dry mix, recycle, and material discharged from the pugmill, granulator, and dryer. For process control, a rapid determination of moisture is made by the infrared-lamp method described by Engelbrecht and Kumagai (2). The moisture content of all samples discussed in this paper was determined by the vacuum desiccation method (7) and reported on the dry basis.

### Granulating Conditions

Initial solid materials used in all of the mixtures shown in Table II were screened through a 10-mesh screen and thoroughly mixed together before entering the gravimetric feeder. Throughput of materials in the small-scale equipment was at the rate of 200 pounds per hour including recycle when it was used. Material used for recycle was obtained from a previous run, ground to pass a 10-mesh screen, mixed thoroughly, and then premixed with the dry feed.

Distribution of the ammoniating solution was more uniform than that of either acid or water in the pugmill. Good distribution of water added to the granulator mixture was obtained by directing a spray of atomized water on the rolling bed of material. Starting-up moisture requirement was higher owing to lower initial temperatures and water needed for initial wetting of equipment.

Temperature of the mixture in the granulator at the agglomeration point was kept fairly uniform by control of external heat input. Control was sometimes made difficult by change in heat transfer owing to build-up and breakdown of an insulating coating of material inside the granulator shell. However, once the moisture-temperature relationship for a mixture has been established, steady production of a uniform product can be realized.

### Control of Agglomeration

Control of agglomeration in the small-scale equipment other than by

Table I. Typical Screen Analyses of Raw Materials

Mesh <sup>a</sup>	Ordinary Superphosphate (20%), %	Triple Superphosphate (48%), %	KCl (60%), %	NH <sub>4</sub> NO <sub>3</sub> (33%), %	(NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> (20%), %
10-20	8	10	4	30	2
20-35	28	24	27	58	53
35-65	28	22	38	10	37
65-150	26	25	15	1	6
-150	10	19	16	1	2

<sup>a</sup> Tyler standard screen scale.

**Table II. Granulating Conditions and Analysis of Products**

Nominal Grade Sample No.	5-10-20 1	5-20-20 2	8-16-16 3	8-24-0 4	8-24-8 5	8-24-16 6	10-20-0 7	10-20-10 8	12-12-12 9	15-10-10 10
Formula, lb.										
Ammoniating solution, 40.6% N <sup>a</sup>	262	262	370	410	404	410	512	432	387	210
NH <sub>4</sub> NO <sub>3</sub> , solid, 33% N (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> , 20.0% or 20.5% N	...	...	42	...	...	...	...	75	193	306
Sulfuric acid, 90%	...	...	...	...	...	...	...	...	106	600
Superphosphate, 20% P <sub>2</sub> O <sub>5</sub>	...	...	...	...	...	...	...	...	(20.5%)	(20.0%)
Superphosphate, 48% <sup>b</sup>	1000	421	755	1105	639	163	1239	687	85	...
KCl, 60% K <sub>2</sub> O	...	658	352	540	734	932	317	547	600	260
Filler, dolomite	695	694	534	...	277	550	...	334	245	308
Subtotal	78	...	...	...	...	...	...	...	400	344
Water added to pugmill	2035	2035	2053	2055	2054	2055	2068	2075	2016	2028
Water added to granulator	...	57	...	...	139	...	...	...	...	...
Recycle, lb./formula wt.	128	90	...	110	...	44	120	...	...	63
Total	...	...	...	...	1027	...	...	...	900	...
Lb. NH <sub>4</sub> NO <sub>3</sub> (35% N) per formula wt.	2163	2182	2053	2165	3220	2099	2188	2075	2916	2091
Input degree of ammoni- ation, lb. NH <sub>3</sub> /20 lb. avail- able P <sub>2</sub> O <sub>5</sub> in formula	170	170	280	267	262	267	333	352	434	425
Moisture, % dry basis	5.7	2.8	5.0	3.7	3.7	3.7	5.6	4.7	4.8	4.6
Input <sup>c</sup>	10.1	10.7	5.1	13.2	8.3	6.1	13.1	5.2	2.9	5.5
Exit pugmill <sup>d</sup>	4.1	6.7	5.0	8.6	10.0	5.7	7.3	6.0	2.8	3.1
Exit granulator <sup>d</sup>	8.3	9.4	4.8	12.7	9.4	7.1	9.9	5.6	2.6	4.0
Exit cooler <sup>d</sup>	6.2	8.4	2.4	8.6	4.8	4.4	6.8	3.5	1.6	3.1
Temperature, ° F.										
Exit pugmill	154	158	171	190	135 <sup>e</sup>	145 <sup>e</sup>	205 <sup>e</sup>	127 <sup>f</sup>	169	126
Exit granulator	204	201	207	205	194	201	206	194	212	180
Screen analysis of cooler product,										
Tyler series, % +6 mesh	5	8	6	17	16	6	18	10	25	15
6-20 mesh	73	55	70	67	69	60	70	71	55	81
20-35 mesh	18	21	17	11	13	26	10	16	17	3
-35 mesh	4	16	7	5	2	8	2	3	3	1
Chemical analysis of 6-20 mesh, %										
Total N	5.1	5.2	8.1	8.2	7.9	7.8	10.4	8.9	11.9	15.0
NH <sub>3</sub> -N	3.6	3.5	5.4	5.8	5.6	5.3	7.5	5.7	8.1	11.3
Total P <sub>2</sub> O <sub>5</sub>	10.4	22.3	17.3	25.0	25.8	27.2	20.9	21.5	13.2	11.0
Available P <sub>2</sub> O <sub>5</sub>	10.3	22.0	16.9	24.7	24.9	26.7	20.5	20.9	12.9	10.8
W. S. P <sub>2</sub> O <sub>5</sub>	2.9	14.4	8.9	14.2	15.4	16.6	8.7	13.5	7.2	5.9
Available K <sub>2</sub> O	20.5	17.2	15.2	...	7.5	14.7	...	9.2	11.8	9.9
H <sub>2</sub> O, % dry basis	3.3	4.6	1.9	4.0	3.2	2.4	3.8	2.3	1.2	1.5

<sup>a</sup> 21.7% NH<sub>3</sub>, 65.0% NH<sub>4</sub>NO<sub>3</sub>, 13.3% H<sub>2</sub>O.

<sup>b</sup> 49% for 12-12-12.

<sup>c</sup> Calculated from water in dry feed, recycle, ammoniating solution, acid, and water added to pugmill and granulator.

<sup>d</sup> Determined by official AOAC vacuum desiccation method.

<sup>e</sup> Pugmill air cooled externally.

<sup>f</sup> Compressed air blown on mixture in pugmill.

adjustment of heat and moisture input was obtained by use of recycle material, change in formulation, or compressed air on the material in the pugmill. The additional heat of reaction and additional water supplied by the 85 pounds of sulfuric acid and 387 pounds of ammoniating solution used to granulate the 12-12-12 mixture (Table II, sample 9) required the use of recycle to avoid formation of a slurry in the pugmill. Although 45% of recycle (pound per pound formula weight) at 0.5% moisture was used, overagglomeration occurred at a moisture content of 2.8% at 158° to 169° F., indicating need for a greater amount of recycle. Sulfuric acid was omitted in formulation of other mixtures owing to low product yield, liberation of dense white fumes, and recycle requirement.

The 15-10-10 mixture (sample 10) was formulated with 6 units of ammonium sulfate to avoid formation of a

slurry in the pugmill without the use of recycle.

The 10-20-10 mixture (sample 8) overagglomerated in the pugmill at 6.0% moisture and 126° to 140° F. As no recycle was available, a stream of compressed air was blown on the mixture to reduce plasticity by reducing the temperature. Compressed air was used as a matter of convenience. Optimum agglomeration was obtained in the granulator at 194° F. and 5.6% moisture. Owing to removal of ammonia by the air stream, the nitrogen content in the product fraction was low, 8.9%.

#### Agglomeration Characteristics in Pugmill and Granulator

Because of the vigorous action of the pugmill and the thixotropic nature of some fertilizers, certain mixtures which appeared too wet in the pugmill were too dry in the granulator for optimum

agglomeration. An 8-24-8 mixture formulated the same as sample 5 was granulated without the use of recycle by adding 17 pounds of water per formula weight to the pugmill, which was the maximum amount possible without producing overagglomeration at 176° F. and 7.9% moisture. Although the temperature was increased to 212° F. in the granulator, the mixture was too dry for optimum agglomeration at 7.2% moisture. In another run, 50% of recycle used to granulate the 8-24-8 mixture reduced the pugmill temperature to 135° F. Water (139 pounds per formula weight) was added to the pugmill without resulting in overagglomeration, and optimum agglomeration in the granulator was obtained at 194° F. and 9.4% moisture. As this 8-24-8 mixture could have been granulated without recycle by introducing additional water in the granulator instead of the pugmill, a pneumatic atomizing nozzle was in-

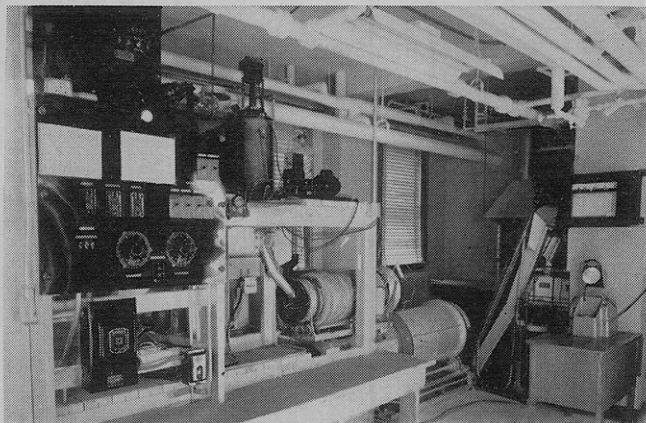


Figure 2. Small-scale continuous granulation equipment

Figure 3. Bag-storage piles

stalled in the front end of the granulator. Other mixtures that appeared wetter in the pugmill than in the granulator at higher temperatures and slightly lower moisture contents (without addition of water) were 8-24-16, 10-20-10, and 12-12-12. Agglomeration in the granulator resulted in rounder, smoother granules than agglomeration in the pugmill, and was easier to control because of external heat input.

**Effect of Formulation on Moisture Requirement.** Hardesty, Szabo, and Cummings (3) observed that, in general, an increasing proportion of soluble salt content decreases the moisture required for optimum agglomeration, rapidly, for the first increments of salt present, and less rapidly beyond about 10% salt content of the mixture. Ammonium nitrate was most effective in decreasing the moisture requirement, followed by ammonium sulfate and potassium chloride in that order.

The data in Table II indicate a general trend toward a decrease in moisture required for optimum agglomeration as the proportion of ammonium nitrate and other soluble salts in the mixtures increases and the proportion of superphosphates decreases.

#### Product Yield and Appearance

The yields of 6- to 20-mesh product given in Table II are for one pass through the equipment without crushing of oversize. The product yields varied from 55% for the 5-20-20 and 12-12-12 mixtures to 81% for the 15-10-10. The low granulation efficiency of the 12-12-12 was due mainly to overagglomeration of the mixture and poor distribution of solution phase in the pugmill. A product yield of 88% was obtained in another run with a 12-12-12 similarly formulated with substitution of ammonium sulfate and solid ammonium nitrate for the acid and part of the ammoniating solution, without the use of recycle or addition of water. The lower yield of 5-20-20 compared to

5-10-20 is attributed to the effect of the large quantity of triple superphosphate in the 5-20-20 mixture. The high product yield of 15-10-10 was obtained with the largest quantity of ammonium sulfate (600 pounds) in any formulation. The 15-10-10 product had the worst appearance, attributed to 306 pounds of solid ammonium nitrate in the mixture. The 12-12-12 had the second worst appearance owing to 193 pounds of solid ammonium nitrate in the formula and agglomeration difficulties in the pugmill. The other mixtures were about the same in appearance. The bulk densities of the 6- to 20-mesh mixtures varied from 53 to 63 pounds per cubic foot.

ammonium sulfate and a comparatively low ratio of ordinary to triple superphosphate. The content of total phosphorus pentoxide in the mixtures generally ran high owing to low values for nitrogen and potash, and also because the analyses of the ordinary and triple superphosphates usually were slightly higher than 20.0 and 48.0%.

#### Bag-Storage Tests

##### Preparation of Material for Storage.

The entire production of 6- to 20-mesh material was divided uniformly into six separate 80- to 85-pound lots, (designated A, B, C, D, E, and F in Tables IV and V), and treated as follows:

Lots	Adjusted Moisture Content, %	Other Conditions	Days Cured
A	0.2-1.5		2
B	1.2-4.7		2
C	2.6-8.3	Not dried	2
D	About 1.2-4.7	Treated with 3% synthetic $\text{CaSiO}_3$ having a mean particle size of $15\mu$	2
E	About 1.2-4.7	14- to 35-mesh	2
F	About 1.2-4.7		14

#### Distribution of Nutrients

The results of chemical analyses in Table II indicate the distribution of nutrients in 6- to 20-mesh product after one pass through the process. The potassium oxide content of the product of every mixture ran lower than that indicated by the formula, particularly in the 5-20-20 grade. The 5-10-20 and 5-20-20 mixtures were formulated to 20.8% potash. The ratio of potassium oxide in the product compared to potassium oxide content of the formula was highest for 5-10-20, 8-16-16, 12-12-12, and 15-10-10 mixtures and lowest for 5-20-20, 8-24-8, 8-24-16, and 10-20-10 mixtures. The first group of mixtures was characterized by a comparatively high ratio of ordinary to triple superphosphate and/or a quantity of ammonium sulfate in the formula, while the latter group had no

Each lot was individually mixed, sampled, divided into two 40-pound lots, and then placed in four-ply polyethylene-lined bags. The bags were constructed of one 50-pound natural kraft liner with 20-pound polyethylene coating, two 50-pound and one 60-pound natural kraft sheets, with wax under the end tape, oiled thread, and waxed filler cord. The tops of the bags were folded several times before stapling and the bags were then stored in an unheated frame building under prevailing atmospheric conditions (Table III). Two 40-pound bags, laid end to end, were placed on top of a 100-pound bag of sand and then eight more 100-pound sand bags were stacked on top. Figure 3 shows the type of construction of the storage piles. The piles were 15 inches apart to provide equal exposure of all test bags. A framework of 2 x 4 inch boards holds the piles in an

upright position. This type of construction ensures stableness of the pile and uniform weight on replicate test bags throughout the storage period.

After 3½ months of storage, the piles were taken down and the test bags dropped from waist-high on each edge and each side. The bags were then opened, samples for moisture analysis were taken at the stapled end and center of the bag, and the contents poured on a 3-mesh screen. Lumps remaining on the screen were weighed and their relative hardness was determined by measuring the length of time required to crush them through the screen. Bag-set was determined by observation of the rigidity of the bags when removed from storage.

**Results of Bag-Storage Tests.** The degree of caking is indicated by the percentage of lumps. Table IV shows that increasing the initial moisture content increased the caking tendency of the mixtures, except when the moisture content was increased from 3.4 to 6.2% in the 5-10-20 mixture. The decrease from 6% to 1% of lumps is attributed to sufficient solution-phase present in the mixture with 6.2% of water to dissolve some of the bonds between the granules. Drying the mixtures to 1.5% moisture, or below, eliminated any lumps except for 1% in the 10-20-10 mixture at 1.5% of water and reduced bag-set considerably.

Treatment of the mixtures with 3% of synthetic calcium silicate eliminated caking in five fertilizers, reduced lumps to 2% or below in four mixtures, and reduced bag-set of every mixture. The uncoated 10-20-0 mixture contained no lumps at 3.8% moisture.

Reducing the particle size from 6- to 20-mesh to 14- to 35-mesh increased the caking tendency of five mixtures considerably and of two mixtures (8-24-8 and 10-20-0) only slightly. The moisture contents of the different size granules of 8-24-8 and 10-20-0 fertilizers were not strictly comparable.

Extending the period of curing before storage from 2 to 14 days reduced the caking tendency of seven mixtures in varying degrees. The percentage of lumps in the 8-16-16 and 15-10-10

**Table IV. Results of Bag-Storage Tests**

Sample <sup>a</sup>	Storage Period Feb. 2, 1955 to May 11, 1956	Moisture Content, Dry Basis, %			+ 3 Mesh Lumps, %	Relative Hard- ness of Lumps	Bag-Set
		Before storage	After Storage				
			Stapled end	Center of bag			
5-10-20							
1A	Sept. 9-Dec. 21	1.2	1.0	0.9	0	...	Soft
B		3.4	3.4	3.2	6	Soft	Hard
C		6.2	6.3	5.9	1	Soft	Hard
D		3.6	3.4	3.2	0	...	Soft
E		3.3	3.0	3.0	22	Soft	Hard
F		Sept. 21-Jan. 3	3.2	3.0	3.1	1	Soft
5-20-20							
2A	Nov. 18-Mar. 2	1.3	1.3	1.0	0	...	Soft
B		4.7	4.0	3.5	16	Soft	Hard
C		8.3	7.6	7.0	18	Soft	Hard
D		5.0	3.8	3.8	2	Soft	Medium
E		4.4	3.6	3.6	36	Hard	Hard
F		Nov. 30-Mar. 14	4.6	4.2	4.0	8	Soft
8-16-16							
3A	Mar. 31-July 15	1.0	1.6	1.6	0	...	Soft
B		1.8	1.8	1.8	2	Soft	Medium
C		3.2	2.9	2.8	7	Soft	Hard
D		2.0	2.5	2.6	0	...	Soft
E		1.9	1.9	1.9	7	Soft	Hard
F		Apr. 12-July 27	1.9	2.1	2.2	3	Soft
8-24-0							
4A	Dec. 16-Mar. 30	1.5	1.5	1.4	0	...	Soft
B		4.1	3.9	3.7	9	Soft	Hard
C		8.0	7.8	7.5	13	Soft	Hard
D		4.1	4.1	4.0	0	...	Medium
E		4.0	3.6	3.5	25	Hard	Hard
F		Dec. 28-April 13	4.2	3.1	3.5	3	Soft
8-24-8							
5A	May 12-Aug. 26	1.4	2.2	2.1	0	...	Soft
B		3.1	3.8	3.6	14	Soft	Hard
C		5.2	5.4	5.3	16	Hard	Hard
D		3.5	4.2	4.2	<1	Soft	Soft
E		2.6	3.4	3.2	17	Medium	Hard
F		May 24-Sept. 9	3.4	3.5	3.5	13	Soft

mixtures cured 2 and 14 days were about the same. Bag-set of only one mixture was reduced with extension of curing time.

Other factors which may have some influence on the storage results are slight variation in processing conditions, size distribution in the product fraction of the various mixtures, variation in granule shape and bulk density, and variations in temperature and humidity during storage of different mixtures.

**Effect of Formulation on Caking Tendency.** A plot of moisture content vs. lumps was made for each mixture and from this graph the percentage of lumps in each mixture at 2.6% moisture was obtained. The 12-12-12 and 15-10-10 mixtures which had the most lumps (13 and 11%, respectively) contained the largest proportions of ammonium nitrate. The 10-20-0 and 8-24-0 mixtures which contained no potassium chloride had 0 and 4% of lumps, respectively. The correlation between formulation and caking tendency was not very good for the other mixtures which contained 4 to 10% of lumps.

**Change in Free-Moisture Content.** The average change in free-moisture content of the mixtures during storage was -0.15%. Some samples increased in free-moisture content as much as 0.7% when stored between May and August; others decreased in moisture as much as 1.3% (November to February). The exchange of moisture appeared to occur through the stitches of the bag closure and not through the side walls.

**Table III. Storage Conditions**

Period	Temperature, ° F. <sup>a</sup>			Relative Humidity, % <sup>a</sup>		
	Mean	High	Low	Average	High	Low
1955						
Feb.-March	39	58	13	64	98	29
Apr.-June	62	84	31	63	95	28
July-Sept.	77	93	55	72	95	41
Oct.-Dec.	48	79	19	63	90	30
1956						
Jan.-March	38	56	22	66	97	31
Apr.-May	57	82	33	62	92	31

<sup>a</sup> Recorded by hygrothermograph.

**Table IV. Results of Bag-Storage Tests (Cont.)**

Sample <sup>a</sup>	Storage Period Feb. 2, 1955 to May 11, 1956	Moisture Content, Dry Basis, %		+ 3 Mesh Lumps, %	Relative Hard- ness of Lumps	Bag-Set	
		Before storage	After Storage				
			Stapled end				Center of bag
8-24-16							
6A	June 23-Oct. 7	1.1	1.6	1.4	0	Soft	
B		2.5	2.7	2.5	7	Hard	
C		4.2	4.4	4.2	20	Medium	
E		2.6	2.8	2.6	34	Hard	
D		2.9	3.1	3.0	1	Soft	
F	July 22-Nov. 9 Aug. 3-Nov. 18	2.2	2.2	2.0	<1	Soft Hard	
10-20-0							
7A	Jan. 25-May 11	1.4	1.5	1.5	0	Very soft	
B		3.8	3.6	3.6	0	Soft-medium	
C		5.9	5.1	4.9	3	Soft	
D		4.0	3.6	3.6	0	Very soft	
E		4.2	3.6	3.8	3	Soft	
10-20-10							
8A	Mar. 4-June 20	1.2	1.5	1.5	1	Soft	
B		2.3	2.3	2.2	3	Soft	
C		4.1	3.4	3.6	10	Medium	
D		2.5	2.6	3.0	0	Soft	
F		Mar. 16-July 1	2.4	2.3	2.4	2	Soft
12-12-12							
9A	Feb. 2-May 17	0.2	0.4	0.4	0	Soft	
B		1.2	1.0	1.0	2	Soft	
C		2.7	2.5	2.5	13	Medium	
D		1.5	1.6	1.6	1	Soft	
F		Feb. 14-June 1	1.1	1.1	1.2	<1	Soft
15-10-10							
10A	Aug. 12-Nov. 28	0.8	0.9	0.8	0	Medium-hard	
B		1.5	1.3	1.3	1	Soft	
C		2.6	2.2	2.1	11	Hard	
D		2.1	2.1	1.9	0	Soft	
F		Aug. 31-Dec. 13	1.6	1.2	1.1	1	Soft

<sup>a</sup> A, B, and C designate different moisture contents.

D designates treatment with 3% of a synthetic calcium silicate with mean particle size of 15 microns.

E designates particle size 14-35 mesh; all other lots 6-20 mesh.

F designates cured for 14 days before bagging; all other lots cured for 2 days.

**Drillability Tests**

Any granular fertilizer that is dry, uniform, and lump-free upon removal from the bag can be expected to drill satisfactorily if used immediately. However, if a hygroscopic material is exposed to a high humidity before or during the drilling operation, the rate of application may change and the

flow may become irregular. Moisture absorption at the surface of the granule is the most important factor affecting the drilling rate of granulated fertilizers. In these tests, the effect of this factor was determined at a relative humidity of 86% to 90% at 74-76° F.

**Equipment and Procedure.** Drillability tests were conducted in a 16 × 24 foot room where the temperature and

humidity were controlled by an air-conditioning unit and humidifier. Several fans in the room provided adequate air circulation. Figure 4 shows the drillability testing equipment, which was constructed in cooperation with the Agricultural Engineering Research Division. The equipment consists of a drill-hopper mechanism mounted over a variable-speed endless belt divided into 6-inch sections for collecting the fertilizer during the drilling operation. A rotating-plate drill was used in the current test. The drill speed was set to conform to the manufacturer's recommendation for a vehicle speed of 3 miles per hour. The belt speed was about 1½ miles per hour. Six-inch sections of the belt moving at 1½ miles per hour collect an amount of fertilizer equal to that distributed over a foot of ground by a drill traveling at the rate of 3 miles per hour. Fertilizer was discharged from the hopper through an adjustable gate and flexible tube either onto the belt or into weighing pans as desired. During the current tests, the adjustable gate was set at a width of 9/32 inch. The drilling rates of the different mixtures as taken from the bag varied between 300 and 500 pounds per acre, based on 40 inches between rows.

Twenty-seven pounds of the fertilizer to be tested was placed in the hopper and drilled 8 minutes to allow for normal flow. Material discharged was returned to the hopper at intervals. With all the fertilizer in the hopper, the mixture was then drilled onto the moving belt with the outlet to the delivery tube clamped in a vertical position above the center of the belt. The material on 25 consecutive 6-inch sections was removed to individual containers and weighed. The material remaining in the hopper was then placed in trays 16 × 28½ × 2½ inches in size, and leveled to a depth of 5/16 inch. The trays were placed 5 inches apart in a rack which provided equivalent exposure of the material in all the trays. A 15-inch fan located 6½ feet from the rack circulated air over the trays. After 30 minutes of exposure, the fertilizer was stirred by hand to aid in uniform moisture absorption throughout the mass and after a 1-hour exposure, it was taken off the trays and mixed, and a sample was taken for the determination of moisture. The procedure of drilling, exposing, stirring, and drilling the fertilizer again was repeated until the material failed to drill or until the total exposure period was 5 hours. A material was called undrillable when it caked in the drill or when the observed flow was erratic. The drilling rate was determined by the total weight of material collected on 25 sections of the belt, expressing the rate on a dry basis. The relative drilling rate is defined as

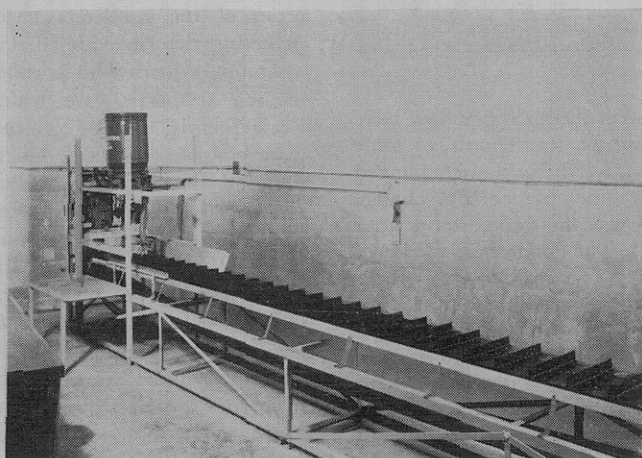


Figure 4. Drillability testing equipment

**Table V. Results of Drilling Tests**

Sample <sup>a</sup>	Initial Moisture, Dry Basis, %	Relative Drilling Rate after Exposure, %			Length of Time Material Remained Drillable, Hours	Range of Moisture Content, Dry Basis, Within Which Material Became Undrillable, %	
		1	2	3			4
5-10-20							
1B	1.9	99	94 <sup>b</sup>	89	c	3	9.3-11.8
D	2.2	89 <sup>b</sup>	88	83	c	3	9.4-12.1
E	1.7	88 <sup>b</sup>	82	74	c	3	9.0-11.6
5-20-20							
2B	3.8	99	99	c	..	2	8.8-11.5
D	4.1	92 <sup>b</sup>	88	82	c	3	11.3-14.0
E	3.7	87 <sup>b</sup>	75	c	..	2	8.3-10.5
8-16-16							
3B	1.9	101	81 <sup>b</sup>	c	..	2	7.3- 9.9
D	2.5	92 <sup>b</sup>	86	c	..	2	7.4-10.2
E	1.2	97	95 <sup>d</sup>	c	..	2	5.1- 7.7
8-24-0							
4B	3.7	97	91 <sup>b</sup>	81	65	4	12.2-13.8
C	7.7	97	91 <sup>b</sup>	72	c	3	13.0-14.6
D	4.2	108 <sup>b</sup>	115	111	96 <sup>e</sup>	5	14.4 <sup>e</sup>
E	3.8	98	87 <sup>b</sup>	86	77	4	11.8-13.4
8-24-8							
5B	3.5	95	88 <sup>b</sup>	c	..	2	9.0-11.7
D	0.8	94 <sup>d</sup>	86 <sup>b</sup>	65	c	3	8.7-11.2
E	0.9	102	95	84 <sup>b</sup>	c	3	7.5- 9.9
8-24-16							
6B	2.1	100	99	68 <sup>b</sup>	c	3	8.9-11.1
D	1.5	92 <sup>b</sup>	87	71	c	3	8.0-10.5
E	1.5	98 <sup>d</sup>	84 <sup>b</sup>	c	..	2	5.8- 8.3
10-20-0							
7B	3.5	106 <sup>d</sup>	96	75 <sup>b</sup>	c	3	11.5-14.1
C	5.3	106 <sup>b</sup>	102	c	..	2	10.1-12.7
D	3.4	94 <sup>b</sup>	89	76	65	4	13.7-15.6
E	3.8	92 <sup>b</sup>	92	83	c	3	10.9-13.4
10-20-10							
8B	2.2	98	96	c	..	2	7.7-10.5
D	2.8	90 <sup>b</sup>	90	c	..	2	7.9-10.6
12-12-12							
9B	1.3	89 <sup>b</sup>	c	...	..	1	4.4- 7.7
D	1.8	85 <sup>b</sup>	c	...	..	1	5.3- 8.3
15-10-10							
10B	0.6	99	c	...	..	1	3.4- 6.3
D	1.1	85 <sup>b</sup>	66	c	..	2	6.9- 9.9

<sup>a</sup> B and C designate different initial moisture contents.

D designates treatment with 3% of a synthetic calcium silicate with mean particle size of 15 microns.

E designates particle size 14-35 mesh; all other lots 6-20 mesh.

<sup>b</sup> Significantly different at 1% level.

<sup>c</sup> Material undrillable.

<sup>d</sup> Significantly different at 5% level.

<sup>e</sup> Material drillable after 5 hours of exposure.

the drilling rate, after exposure, expressed as a percentage of the original drilling rate, both on the dry basis.

**Results of Drilling Tests.** Table V shows the relative drilling rate and moisture content of the mixtures, before and after exposure, and the length of time the fertilizers remained in a drillable condition. An analysis of variance was made of the relative drilling rates before and after exposure to determine the level (5 or 1%) at which the rates were significantly different.

Comparison of lots B and C, samples 4 and 7, indicates that a higher initial

moisture content in this range may have shortened slightly the length of time before the relative drilling rate changed significantly. The percentage change in both rates was the same after 1 hour of exposure. The materials of lower initial moisture content remained drillable for longer periods of time, but became undrillable at about the same moisture content as the materials of higher initial moisture content. Drilling tests at intervals of 1 hour do not allow close comparison among different samples with respect to the exact time and moisture content at which they

become undrillable. However, a comparison of the drilling rates and moisture ranges of lots that fail to drill within the same hour gives an indication of their relative drillabilities and final moisture contents.

Comparison of B and D lots, samples 1 to 10, shows that the drilling rates of materials treated with 3% of synthetic calcium silicate changed significantly sooner than did the rates of the uncoated materials, except for the 12-12-12. The relative drilling rates of the coated mixtures were lower after 1-hour exposure than those of the uncoated mixtures except for the 8-24-0, indicating that the synthetic calcium silicate became sticky upon absorption of moisture and decreased flowability of the granules. Comparison of the coated and uncoated mixtures indicates that, at comparable initial moisture content, the coated materials remained in a drillable condition slightly longer and at slightly higher moisture contents than did the uncoated materials, presumably because of the high moisture-absorbing capacity of the synthetic calcium silicate.

Comparison of B and E lots, samples 1 to 7, indicates that at comparable initial moisture content, the drilling rates of the 14- to 35-mesh granules generally changed significantly, slightly sooner than did the rates of the 6- to 20-mesh granules. Comparison of these same lots indicates little difference, on the average, in length of time the different size granules remained drillable at comparable initial moisture contents, but indicates that the larger granules generally remained drillable at slightly higher moisture contents than the smaller granules.

The length of time that mixtures remained in drillable condition, allowing for differences in initial moisture content, generally increased with increase in superphosphate content and decreased with increase in soluble salt content of the mixture.

**Drillability of Mixed Fertilizers Compared with That of Ammonium Nitrate.**

To estimate the quality of drilling properties of the fertilizers tested by the procedure described above, it is necessary to compare them with a material whose drilling properties are widely known. Consequently, prilled ammonium nitrate consisting of 90% 10- to 20-mesh material at 0.2% moisture was exposed on trays for 20 minutes, after which it was undrillable at a moisture content of 1.5%. The relative drilling rate had decreased to 45% and continued to decrease with erratic flow. Any fertilizer that can be drilled uniformly after a 1-hour exposure has drilling properties superior to prilled ammonium nitrate and should drill satisfactorily. All of the mixed fertilizers described in this paper were drillable after at

least 1 hour of exposure, and many of them remained drillable after a much more severe exposure than would be normally expected in farm practice.

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#### Literature Cited

- (1) Assoc. Offic. Agr. Chemists, Washington, D. C., "Methods of Analysis," 8th ed., p. 7, 1955.
- (2) Engelbrecht, R. M., Kumagai, Rikio, *Com. Fertilizer* **92**, No. 1, 20-2 (1956).
- (3) Hardesty, J. O., Szabo, A., Cum-

- mings, J. G., *J. Agr. Food Chem.* **4**, 60-3 (1956).
- (4) Hein, L. B., Hicks, G. C., Silverberg, J., Seatz, L. F., *Ibid.*, **4**, 318-30 (1956).
- (5) Kumagai, Rikio, Hardesty, J. O., *Ibid.*, **4**, 132-5 (1956).

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## FERTILIZER TECHNOLOGY

# Production of Diammonium Phosphate by Continuous Vacuum Crystallization

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Diammonium phosphate was produced on demonstration scale in a vacuum crystallizer. The product was a coarse crystalline material containing 21% of nitrogen and 53% of phosphorus pentoxide. The process, developed earlier in pilot-plant work, consisted of feeding ammonia and furnace-grade phosphoric acid into a vacuum crystallizer. The crystal slurry was centrifuged and the crystals were dried. The mother liquor was maintained on the acid side, pH 6.5, which resulted in a low partial pressure of ammonia over the solution so that recovery of ammonia from vapors was unnecessary. The process was carried out in a vacuum crystallizer adapted from the production of crystalline ammonium nitrate. Only minor changes were required.

DIAMMONIUM PHOSPHATE as a fertilizer offers the advantages of high concentration of plant food (21% of nitrogen, 53% of phosphorus pentoxide) and favorable agronomic and physical properties. It can be used for direct application or for preparation of high-analysis fertilizers by either dry-mixing (6) or granulation (2) techniques. As diammonium phosphate is completely soluble in water, it can be used in irrigation systems and in liquid fertilizers.

The pilot-plant development for the production of diammonium phosphate fertilizer in a saturator or vacuum crystallizer from anhydrous ammonia and electric-furnace phosphoric acid has been described by Thompson and associates (7). A modification of the vacuum crystallization process that permits the use of impure, wet-process phosphoric acid has been described by Houston and coworkers (3).

In view of the potential advantages and uses cited above, TVA decided to produce diammonium phosphate from electric-furnace phosphoric acid as a part of its program for introducing and demonstrating the use of new and improved forms of fertilizer materials.

The purpose of this paper is to describe the diammonium phosphate production process as it was carried out in the TVA plant. Although the use of one type of crystallizer is described,

other types could be used satisfactorily, as shown by pilot-plant work (7). Crystallizers are in use currently in the fertilizer industry for making ammonium sulfate and other materials. The alternate use of such crystallizers for diammonium phosphate would provide flexibility for adjusting to changing market conditions.

#### Description of Process

The diammonium phosphate production facilities were located in the TVA ammonium nitrate plant. Much of the equipment that was to be used to produce diammonium phosphate originally was a part of the ammonium nitrate plant and now may be used in either process.

The TVA ammonium nitrate vacuum crystallization plant has been described by Saeman, McCamy, and Houston (5). It has five continuous vacuum crystallizers designed and fabricated by the Struthers-Wells Corp. Each unit has a production capacity of about 5 tons of ammonium nitrate per hour. The five crystallizers, which are operated in parallel, use a common surge tank and discharge to a common tank from which the crystal slurry is fed to two of three continuous centrifuges. One of the centrifuges has served as a stand-by.

The changes that were required to pre-

pare for the production of diammonium phosphate consisted of isolating one of the crystallizers from the ammonium nitrate circuit, installing piping for feeding ammonia and phosphoric acid, providing a separate surge tank, and installing drying and screening equipment. The stand-by centrifuge was used for dewatering the crystal slurry.

A flow sheet of the process is shown in Figure 1. Anhydrous gaseous ammonia, at 25 pounds per square inch of pressure, and electric-furnace phosphoric acid, about 78% concentration, were fed continuously to the crystallizer. The acid was fed at a predetermined rate and the ammonia was fed at such a rate as to maintain the pH of the mother liquor at about 6.5. The operating temperature was maintained at 125° F. Thompson and associates (7) found that, under these conditions, diammonium phosphate crystallized from the mother liquor with very little loss of ammonia in the vapors.

If 54% phosphoric acid had been used, vaporization of the water in the acid would have dissipated the heat liberated by the reaction of 2 moles of ammonia with 1 mole of phosphoric acid (7). With the more concentrated acid, water was added to the system to aid in dissipating the heat. This water was used to dissolve fines and the resulting solution was added to the surge tank (see Figure 1).