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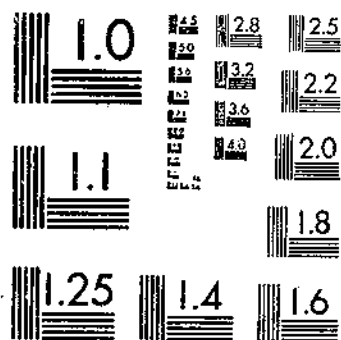
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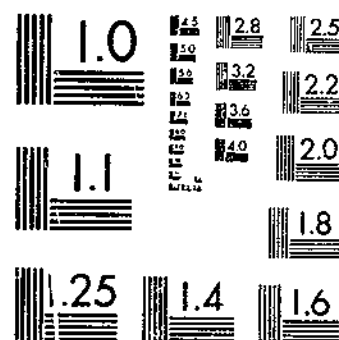
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MICROCOPY RESOLUTION TEST CHART  
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MICROCOPY RESOLUTION TEST CHART  
NATIONAL BUREAU OF STANDARDS-1963-A



**UNITED STATES  
DEPARTMENT OF AGRICULTURE  
WASHINGTON, D. C.**

# Preparation of Ammonium Nitrate for Use as a Fertilizer

By WILLIAM H. ROSS, *principal chemist*, J. RICHARD ADAMS, *chemist*, J. Y. YEE, *associate chemist*, COLIN W. WHITTAKER, *chemist*, and KATHARINE S. LOVE, *assistant chemist*, Division of Soils, Fertilizers, and Irrigation, Bureau of Plant Industry, Soils, and Agricultural Engineering, Agricultural Research Administration<sup>1</sup>

## CONTENTS

	Page		Page
Summary .....	2	Factors in caking tendency .....	29
Fertilizer materials containing		Moisture .....	29
ammonium nitrate .....	3	Particle size .....	32
Nitrogen Solution .....	3	Pressure during storage .....	34
Lennasalpeter .....	3	Temperature and duration of	
Cal-Nitro .....	4	storage .....	36
Preparation .....	4	Conditioners .....	37
Monocrystalline ammonium		Large-scale tests on storage	
nitrate .....	4	behavior .....	42
Granular ammonium nitrate ..	5	Sprayed and grained nitroge-	
Physical properties .....	8	nous materials .....	42
Crystal form .....	8	Effect of conditioners .....	45
Microscopic examination .....	11	Effect of location .....	48
Apparent density .....	11	Monocrystalline ammonium	
Hardness of particles .....	12	nitrate .....	54
Solubility .....	13	Moisture absorption in stor-	
Determination of moisture con-		age .....	55
tent .....	14	Final inspection .....	55
Hygroscopicity .....	16	Effect of bags on storage be-	
Temperature and humidity re-		havior .....	58
lations .....	17	Bag-storage tests .....	59
Location and humidity rela-		Importance of bag recognized	
tions .....	19	by industry .....	66
Rate of moisture absorption ...	24	Drillability .....	66
Method of measurement .....	24	Tests in air-conditioned room.	
Moisture-holding capacity ....	25	Field tests .....	76
Effect of water repellents ....	25	Literature cited .....	78

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**A**MMONIUM nitrate from a number of ordnance and industrial plants in the United States and Canada became available as such for use as a fertilizer in the spring of 1943. The material first offered for sale proved to be unsatisfactory, owing to its marked tendency to cake and to its property of absorbing moisture from the air under humid conditions. When it was originally placed on the market its behavior was such as frequently to render it completely undrillable and entirely unsuitable for separate application in the field. Despite these drawbacks ammonium nitrate has certain advantages that make it desirable for use as a fertilizer. It is a cheap source of nitrogen and its agronomic value as a fertilizer has been amply demonstrated in numerous field tests in comparison with other nitrogenous materials. It was therefore recognized that any improvement that could be made in its mechanical properties would supply a pressing need, in view of the shortage of nitrogen that prevailed during 1942 and the increased general demand for fertilizers that developed in 1943. The War Production Board accordingly requested the Division of Soils, Fertilizers, and Irrigation to undertake investigations on the production of satisfactory ammonium nitrate fertilizer.<sup>3</sup> Investigations were at once undertaken in cooperation with producers in this country and in Canada, and the results may be summarized as follows.

### SUMMARY

Ammonium nitrate has been produced commercially by the methods of crystallizing, graining, and spraying. Each particle of monocrystalline ammonium nitrate as obtained in the Oslo-Krystal process consists of a single crystal, whereas the particles of sprayed or grained material consist of aggregates of crystals. The grained ammonium nitrate is about twice as porous as the monocrystalline material and the sprayed material about five times as porous. These differences in porosity have a marked effect on the capacity of the different types of ammonium nitrate to hold absorbed moisture and on their tendency to cake under varying climatic conditions.

A slight change in moisture content has a greater effect on the caking of ammonium nitrate than a corresponding small change in any of the other factors that affect its tendency to cake. The most effective conditioning agents for reducing the caking of ammonium nitrate are Celite, Dicalite, Kittitas and other forms of kieselguhr, kaolin and other types of clay, plaster of paris, and tricalcium phosphate.

The most effective water repellents for decreasing the rate of moisture absorption from the air are mixtures of paraffin with petrolatum or asphaltum and of petrolatum and paraffin with asphaltum or rosin.

Granular ammonium nitrate that has been treated with 0.5 to 1.0 percent of an effective water repellent plus 3.0 to 5.0 percent of a suitable conditioning agent or with 3.0 to 5.0 percent of a suitable conditioning agent alone will remain in a drillable condition for prolonged periods in piles at least 12 bags high, provided it is dry when stored and the bags are sufficiently moistureproof to keep it dry throughout the storage period. Bags now on the market are capable

<sup>3</sup> The work was supported in part by a grant from the Office of Production Research and Development, War Production Board.

of maintaining properly conditioned ammonium nitrate in a satisfactory mechanical condition for at least a year in the most humid sections of the country.

Tests under carefully controlled conditions with standard farm equipment indicate (1) that the drillability of any given material increases with the uniformity of its particle size; (2) that the sprayed ammonium nitrate now on the market is more drillable than the grained material, owing to differences in particle size; and (3) that granular ammonium nitrate that has been treated with a water repellent and a conditioning agent is more drillable under conditions of high humidity than when treated with a conditioning agent alone. Although a double treatment with a water repellent and a conditioning agent greatly improves the drillability of ammonium nitrate under conditions of high humidity, the use of a water repellent is not considered necessary in sections where the prevailing humidity is relatively low.

## FERTILIZER MATERIALS CONTAINING AMMONIUM NITRATE

Ammonium nitrate cannot be considered a new fertilizer material except when used directly as such. Its consumption as a fertilizer in the United States during the past 15 years varied from a minimum of 1,000 tons in 1932 to a maximum of 425,000 in 1943-44. Until recently it was not marketed as such but in association with one or more other materials to form the products known as Nitrogen Solution, Leunasalpeter, and Cal-Nitro.

### NITROGEN SOLUTION

Nitrogen Solution is the trade name given to a series of solutions prepared by dissolving anhydrous ammonia in solutions of ammonium nitrate of the desired concentration. These solutions are used only in the preparation of mixed fertilizers or in the ammoniation of superphosphate for the preparation of base goods (33).<sup>4</sup> The four grades of solution that have thus far been placed on the market vary in the proportion of free ammonia to ammonium nitrate. The solution with the highest content of free ammonia and the lowest content of ammonium nitrate has the highest vapor pressure and can be cooled to the lowest temperature before the "salting out" of ammonium nitrate takes place. A solution of this kind is best adapted for use during the coldest season of the year. The other Nitrogen Solutions are adapted to more general use and are marketed according to the demands of the fertilizer manufacturer.

### LEUNASALPETER

Leunasalpeter is the trade name given to a double salt of ammonium nitrate and ammonium sulfate (11, p. 432). Between 1926 and 1931 it was imported from Germany in relatively large quantities for use in mixed fertilizers, but importations ceased in 1932. It has never been produced in this country, and the economics of fertilizer production and consumption are such that there is little likelihood that it will be produced in the United States at any time in the near future.

<sup>4</sup> Italic numbers in parentheses refer to Literature Cited, p. 78.

### CAL-NITRO

Cal-Nitro (A-N-L) is the trade name of a fertilizer material containing ammonium nitrate. It was first produced in Germany, where it was known also as Kalkammonsalpeter. A similar product manufactured in England is known as "Nitro-Chalk" (11, p. 431). Cal-Nitro containing 20 percent of nitrogen is now being produced in this country, where it is sometimes sold under the trade name of A-N-L fertilizer (37). In the production of this material, finely divided dolomite is mixed with a hot solution of ammonium nitrate to form a slurry that is sprayed from the top of a granulating tower. The granules that collect at the bottom of the tower are first cooled, then dried in a rotary drier, treated with about 5 percent of a conditioning agent, and stored in an air-conditioned warehouse in which the humidity of the air is continuously maintained below the hygroscopic point of the material.

### PREPARATION

The first experimental plant for the production of synthetic ammonia was put in operation at Ludwigslafen, Germany, in 1910. Methods for the oxidation of byproduct ammonia to nitric acid were developed somewhat earlier. The first of these oxidation plants was built at Westphalia, Germany, in 1909. Synthetic nitric acid, however, was first produced at Notodden, Norway, in 1905 by the arc process (5). Methods for the production of synthetic nitric acid and for the oxidation of ammonia to nitric acid were thus known before the development of the synthetic ammonia process. The production of ammonium nitrate from synthetic ammonia and synthetic nitric acid immediately followed the commercial production of synthetic ammonia.

### MONOCRYSTALLINE AMMONIUM NITRATE

The first ammonium nitrate to contain synthetic nitrogen was that produced at Notodden, Norway, from synthetic nitric acid and byproduct ammonia (7). This process consisted in neutralizing the nitric acid with ammonia, concentrating the resulting solution in vacuum evaporators, discharging the concentrated solution into trough-shaped crystallizers that oscillated about their horizontal axis to facilitate the formation of pure crystals without enclosure of mother liquor, and separating the crystals from the mother liquor by use of centrifugals. Subsequent improvements in the process have related particularly to modification of the crystallizing equipment, with a view to the formation of larger sized crystals (22).

The latest development in the preparation of monocrystalline ammonium nitrate is known as the Oslo-Krystal process; the product is commonly referred to as Krystal ammonium nitrate. In the operation of this process the solution from neutralizers containing 80 to 85 percent of ammonium nitrate at 75° to 80° C. is mixed with mother liquor of 68-percent concentration and pumped to a vacuum evaporator at 61-mm. pressure. The discharge from this evaporator is passed at 58° to 60° to a second evaporator operating at 10- to 15-mm.

pressure. The bottom of this evaporator connects with a crystallizing tank. The supersaturated solution entering this tank from the second evaporator is uniformly conducted upward through a dense suspension of crystals. The classifying action in the crystallizing tank keeps the fully grown crystals suspended in the bottom layer, the smallest crystals in the top layer, and the intermediate sizes suspended between. The saturated solution leaving the crystallizing tank is mixed continuously with the solution from the neutralizers, the mixture is again supersaturated to the desired extent, and the cycle is repeated.

The fully grown crystals in the bottom of the crystallizing tank are pumped as a suspension to centrifugals in which they are separated from the mother liquor. The liquor is returned to the system, while the centrifuged salt containing 0.8 to 1.0 percent of moisture is further dried in a steam-heated rotary drier to a moisture content of about 0.1 percent.<sup>5</sup> This process for producing monocrystalline ammonium nitrate has been used on a pilot-plant scale by the Military Chemical Works, Inc., Pittsburg, Kans., and the Tennessee Valley Authority, Wilson Dam, Ala. Monocrystalline ammonium nitrate has been produced by other methods on a commercial scale by the Welland Chemical Works, Ltd., Welland, Ontario, and the Consolidated Mining & Smelting Co. of Canada, Ltd., Trail, British Columbia. The crystallizing processes formerly used by these concerns have since been replaced by methods of spray granulation.

### GRANULAR AMMONIUM NITRATE

Ammonium nitrate can be granulated (20) by the methods of shredding, rotary drying, graining, and spraying. The methods of shredding and rotary drying are adapted to the granulation of ammonium nitrate in a finely divided condition, as that produced by crystallization. The other methods of graining and spraying are adapted to the granulation of ammonium nitrate in the form of a hot concentrated melt of the salt. Three methods are now in use for the concentration of a solution of ammonium nitrate to give a melt of the material in a form suitable for granulation. These are known as the Hi-Pan evaporation (15, 27, 28), vacuum evaporation (1), and film evaporation (10) methods. Any one of the three might be used as a preliminary step in the preparation of ammonium nitrate for granulation by either graining or spraying, but in actual practice the Hi-Pan evaporation method is commonly used in the granulation of ammonium nitrate by graining, while one of the other two methods is used in the spraying process.

The methods of graining and spraying give the most uniform product with respect to both size and shape of granule. The average particle size of the sprayed material as produced at present is somewhat larger than that of the grained material. Either of these methods produces a satisfactory particle size for use in mixed fertilizers, but the larger particle size of the sprayed material gives it an advantage for use as a top dressing.

<sup>5</sup> SPENCER, K. A. PRODUCTION OF A SATISFACTORY FERTILIZER GRADE AMMONIUM NITRATE. War Prod. Bd. Proj. Rpt. 122, 34 pp., illus. 1944. [Processed.]



### SHREDDING

In the shredding method of granulating ammonium nitrate the solid material to be granulated is moistened with water, if necessary, and pressed through a perforated plate or other device. Means are provided for cutting the filaments of material formed in this way into suitable lengths as they issue from the shredding device. The process is better adapted to the preparation of materials in the form of large than of small pellets. The cylindrical pellets obtained by this method can be given a more spherical shape by rolling them while soft in a revolving drum. Although ammonium nitrate has been granulated by shredding on a pilot-plant scale, this method has not given sufficient promise to justify commercial development.

### ROTARY DRYING

Rotary drying is similar to the shredding method, in that it is applicable only to the granulation of finely divided solid materials. In the granulation of finely divided monocrystalline ammonium nitrate, the material is first treated with steam to raise the temperature of the mass and to increase its moisture content to 5 to 10 percent. The moisture necessary for granulation decreases with increase in the temperature of the material. The treated material is then rolled in a rotary drum until granulation takes place. A very uniform product can be obtained in this way, but the granules first formed have a tendency to break down in the process of drying. This can be largely prevented by subjecting the moist granules to a preliminary cooling and by recirculating a part of the dried material through the drier for the purpose of quickly reducing the average moisture content of the mass. Larger granules can be produced by this method than have so far been prepared by either the graining or the spraying method. Although this method is particularly adapted to the granulation of superphosphate (18) and mixed fertilizers containing ammonium nitrate (30), it is not recommended for the commercial granulation of ammonium nitrate as such.

### GRAINING

In the process of graining ammonium nitrate, a fusion of the salt containing 1 to 2 percent of water, such as that produced by Hi-Pan evaporation, is run into a circular shallow vessel equipped with mechanical plows and jacketed for cooling with water or heating with steam. The rotation of the plows agitates the material during the process of crystallization. This prevents the formation of a solid mass, as the moisture is eliminated by the heat that is first developed by the crystallization of the material and later supplied, after a preliminary cooling, by steam or hot water in the jacket of the graining apparatus. The size and uniformity of the particles of ammonium nitrate granulated by graining vary with the concentration or percentage of moisture present and the rate of drying. When these factors are properly adjusted and controlled, the uniformity of the product is second only to that produced by spraying.

This method of graining has long been used in the production of ordnance ammonium nitrate. It has the disadvantage that it oper-

ates as a batch process. A continuous method of graining has recently been developed in this country on a pilot-plant scale. In the operation of this process, use is made of the standard graining kettle, but after such modifications as will permit continuous withdrawal of the grained ammonium nitrate.

#### SPRAYING

In the spraying process a spray of molten ammonium nitrate is allowed to fall in a tower through a counter-current flow of cool air. In their fall the sprayed liquid droplets congeal as spherical particles of more or less uniform size. The molten solution of ammonium nitrate may be subdivided into droplets (1) by allowing it to flow by gravity into a vessel provided with a screened or perforated bottom (25); (2) by directing a jet of air at right angles to a stream of the liquid as it issues from a suitable opening (24); (3) by allowing it to flow into a perforated cylinder that rotates at such speed that the liquid breaks up into small droplets as it issues from the perforations (6); (4) by delivering it to a rapidly revolving surface in the form of a plate (21), cup (39), horizontal cylinder (12), or other device; or (5) by passing it under pressure through a spraying nozzle (1, 38).

Although various devices have been used on an experimental scale to produce a spray of fused ammonium nitrate, the rotatable horizontal cylinder and the spraying nozzle are the only devices now being used on a commercial scale in the United States and Canada for this purpose.

In the method that makes use of a rotatable horizontal cylinder (12), the fused material to be sprayed is fed to the cylinder through a box containing a weir that is adjacent to, but does not touch, a horizontal line along the surface of the cylinder. On overflowing the weir the liquid makes contact with the ascending side of the rotating cylinder, whereby a film is distributed on the drum surface from which it is thrown tangentially by centrifugal force. The cylinder is heated with steam to maintain the material in a molten condition. A shield is provided to intercept that part of the spray stream that leaves the cylinder at an angle below the horizontal. The sprayed particles are allowed to drop through a tower against a counter current of air for a distance of about 85 feet. This process is now being used in the preparation of Cal-Nitro. The product so produced is not surpassed by any other fertilizer material with respect to uniformity in size and shape of granules.

The spraying nozzle is used in the granulation of such fusible materials as sodium nitrate (16) and urea (38), as well as of ammonium nitrate (35, 36). The nozzle can be so directed in the spraying tower that the spray leaves it in (1) a horizontal, (2) an upward, or (3) a downward direction.

The downward direction has the disadvantage that the particles produced when the nozzle is in this position possess an initial velocity when starting to fall and therefore require a higher tower for an equal cooling effect. When the sprayed particles leave the nozzle in an upward direction they must reverse their direction before starting to fall. This prolongs the time that they remain in suspension in the air and permits the use of a lower tower. Thus the time taken for a

droplet to rise  $A$  feet and then fall  $B$  feet is equivalent to that required for a straight fall of  $X$  feet, when  $X = A + B + 2\sqrt{A + B}$ . If  $A = 9$  feet and  $B = 40$  feet, then  $X = 63$  feet. This shows that a 40-foot fall when the droplets first rise to a height of 9 feet is equivalent to a fall of 63 feet when the material is sprayed in a horizontal direction.

The height of the tower in any spraying process must be such that the droplets become sufficiently congealed in their fall to withstand the shock to which they are subjected when striking the bottom of the tower. Large droplets take a longer time to cool than small ones, and the height of the tower must therefore be increased with increase in the size of the sprayed granules.

The congealed granules that reach the bottom of the tower pass through rectangular hoppers to endless belts that convey them to the drying equipment. Fans are located at either the bottom or the top of the tower for forcing or withdrawing air through the tower.

The method of granulating a fertilizer by spraying the fused material into the top of a tower has always been referred to in the publications of this Division as the spraying process, and the particles of granular material are spoken of as granules. The process has also been called the shotting method. In Canada it is known as the prilling method, the particles are referred to as prills, and the product prepared in this way is sold under the trade name of Nitraprills. The particles of a granular fertilizer are also occasionally spoken of as pellets, but this term would seem to apply more properly to the particles of feeding stuffs produced by the shredding method. The product known as Nitraprills is now being produced by the Welland Chemical Works, Ltd., Welland, Ontario; Alberta Nitrogen Products, Ltd., Calgary, Alberta; and the Consolidated Mining & Smelting Co. of Canada, Ltd., Trail, British Columbia. Grained ammonium nitrate is now being produced by the Tennessee Valley Authority, the Hercules Powder Company, and by ordnance plants in different parts of the United States.

In this publication the individual particles of Krystal ammonium nitrate are referred to as monocrystals.

## PHYSICAL PROPERTIES

The principal properties of ammonium nitrate that affect its use as a fertilizer are its crystal form, hardness of particles, solubility, caking tendency, and hygroscopicity.

### CRYSTAL FORM

Ammonium nitrate differs from other fertilizer materials in that it is capable of undergoing several crystalline modifications, the transformation of one from another being accompanied by measurable volume and thermal changes. According to Hendricks, Posnjak, and Kracek (20), ammonium nitrate is cubic above  $125.2^{\circ}\text{C}$ .; tetragonal between  $125.2^{\circ}$  and  $84.2^{\circ}$ ; orthorhombic between  $84.2^{\circ}$  and  $32.3^{\circ}$ ; orthorhombic pseudotetragonal between  $32.3^{\circ}$  and  $-18^{\circ}$ ; and tet-

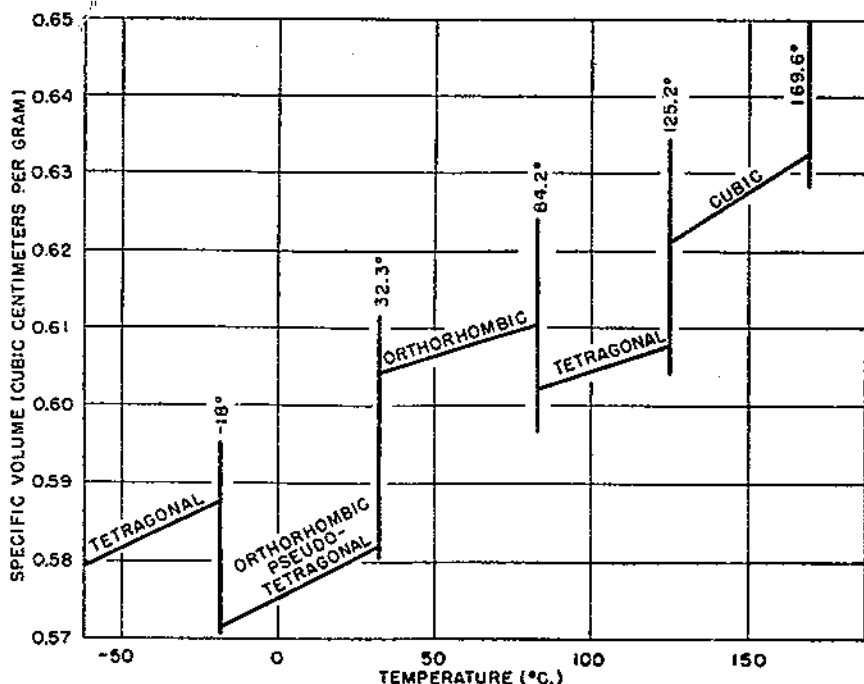


FIGURE 1.—Crystal transitions and volume changes in ammonium nitrate as a function of temperature.

agonal below  $-18^{\circ}$ , as shown in figure 1. The rate (32) at which these transformations take place as the material is cooled down from  $100^{\circ}$  or heated up from  $10^{\circ}$  is shown by the curves in figure 2. These curves show that when the orthorhombic form is maintained at  $28^{\circ}$ , it will undergo more or less complete transformation into the pseudotetragonal form in about 90 minutes; and that at a temperature of  $34^{\circ}$  the pseudotetragonal form will be largely converted into the orthorhombic form again in about the same length of time.

The effect of one transformation on the appearance of monocystals is indicated by the photomicro-

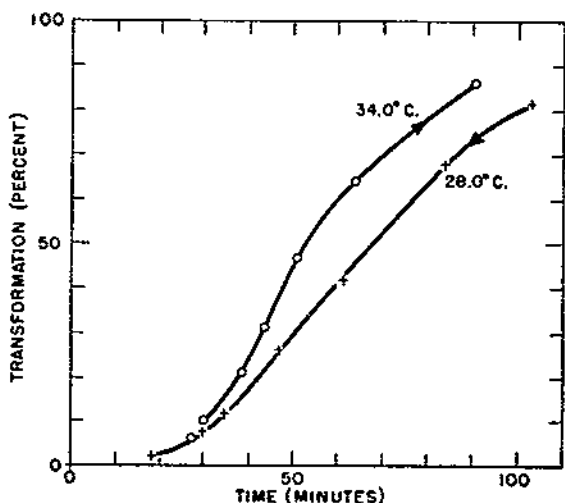


FIGURE 2.—Rate of transformation of the two orthorhombic forms of ammonium nitrate.

graph shown in figure 3. The clear crystals on the left were dried at a temperature below 32° C.; the opaque crystals on the right were dried at 70° and cooled to room temperature.

In its preparation by either the graining or the spraying methods, ammonium nitrate is cooled over a range of temperature that is capable of bringing about several transformations in the form of the crystal. It is doubtful, however, whether any subsequent change in crystal form takes place during ordinary conditions of storage. Summer temperatures frequently exceed 32.3° C. (90.2° F.), but tests made under commercial conditions of storage indicate that the temperature of any considerable quantity of the material when stored in sections of the country where most fertilizers are used is not likely

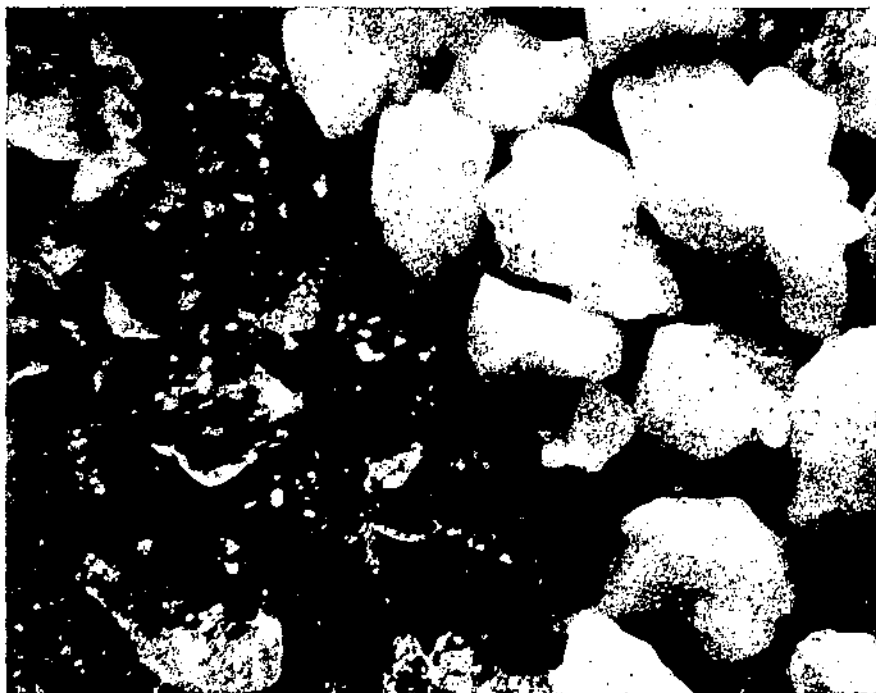


FIGURE 3.—Monocrystalline ammonium nitrate: Clear crystals on the left dried below 32° C.; opaque crystals on the right dried above 32° C.  $\times 16$ .

TABLE 1.—Effect of crystal transformations on the crushing strength of a sample of ammonium nitrate granules prepared by the rotary drying method

Test	Treatment	Moisture content	Crushing strength at 25° C.
		Percent	Pounds per square inch
No. 1	Heated to 50° C. for 1½ hours, cooled to 25°	.37	165
No. 2	Reheated to 32° C. overnight, cooled to 25°	.62	200
No. 3	do	.62	195
No. 4	Reheated to 40° C. overnight, cooled to 25°	.62	118
No. 5	do	.62	60

to reach 32.3° C. during the time that the surrounding air exceeds this temperature.

It would be expected that repeated fluctuations of the temperature of a granule of ammonium nitrate above and below the 32.3° C. transition point would tend to weaken it. This was found to be the case as shown by the data in table 1. It was also found that repeated fluctuations of temperature above and below 32.3° C. increased the tendency of a mass of ammonium nitrate granules to cake under pressure. This is in agreement with what might be expected, inasmuch as the applied pressure would tend to break up the weakened granules and thereby cause them to be compressed together into a solid mass. It may be concluded, therefore, that repeated changes in the crystal form of ammonium nitrate would increase its tendency to cake under pressure, but such changes as do actually take place under commercial conditions of storage are considered to be of little more than academic interest.

### MICROSCOPIC EXAMINATION

A microscopic examination of sprayed, grained, and monocrystalline ammonium nitrate as produced by Alberta Nitrogen Products, Ltd. (ANPL), the Tennessee Valley Authority (TVA), and the Military Chemical Works, Inc. (MCW), respectively, was made by S. B. Hendricks, of the Division of Soils, Fertilizers, and Irrigation. His findings may be summarized as follows:

1. Each particle of monocrystalline ammonium nitrate consists of a single crystal, whereas the granules of the sprayed and grained materials consist of aggregates of crystals.
2. Particles of monocrystalline ammonium nitrate of 10-mesh size contain more voids than those of 16-mesh, indicating that the void space in the material increases with the size of the crystals.
3. Most of the void space in monocrystalline ammonium nitrate that has been air-dried at a humidity below its hygroscopic point is filled with air rather than water.
4. Determination of refractive indices with the petrographic microscope showed that the monocrystals examined were all orthorhombic pseudotetragonal and that they had not passed through the 32.3° C. transition point.

### APPARENT DENSITY

The apparent densities of the three types of ammonium nitrate—sprayed, grained, and monocrystalline—are given in table 2. The apparent density of the particles in bulk represents the ratio of the weight of the material required to fill a liter cylinder to the weight

TABLE 2. Apparent density of ammonium nitrate of different types

Type of material	Producer	Apparent density of -	
		Particles in bulk	Average particles <sup>1</sup>
Sprayed <sup>2</sup>	ANPL	0.883	
Do, <sup>2</sup>	do	.859	1.77
Grained	TVA	.987	1.65
Monocrystalline	MCW	.973	1.59

<sup>1</sup> Determined by S. B. Hendricks.

<sup>2</sup> Old production.

<sup>3</sup> Current production.

of the same volume of water. The cylinder was filled by adding small increments of the material while the cylinder was gently tapped. Each value given in the table represents the mean of five determinations.

The apparent density of the average particle was determined by noting the density of an organic liquid that will hold the greater part of the particles in suspension. The absolute density of ammonium nitrate is 1.72 (34). The values obtained for the apparent density of the average particle of sprayed, grained, and monocrystalline ammonium nitrate were 1.56, 1.65, and 1.69, respectively. These data indicate that the ANPL sprayed particles contain on the average 10 percent of voids, the TVA grained particles about 4 percent, and the MCW monocrystalline particles less than 2 percent.

### HARDNESS OF PARTICLES

The crushing strengths of ANPL granules and of MCW crystals of ammonium nitrate were determined by means of the apparatus described by Hardesty and Ross (17). Under this method of testing it was found that the granules of such nonplastic materials as superphosphate and mixed fertilizers showed a definite crushing strength that varied with their size, shape, and moisture content. All crushing-strength tests with ammonium nitrate were accordingly made with dry uniform samples of known particle size. The granules and crystals of ammonium nitrate were found to differ from those of the other materials tested in that they exhibited plastic properties and frequently failed to show a definite crushing point.

Crushing-strength tests were made with old and current ANPL granules and with MCW crystals dried below and above 32° C. The numbers of particles of each of these four samples that had to be tested in order to obtain 50 that showed a definite crushing strength were 64, 55, 128, and 117, respectively. These tests indicate that the MCW crystals are even more plastic in their properties than the ANPL granules.

The results obtained in these crushing-strength tests are given in the third column of table 3. The values obtained indicate that the sprayed granules of the ANPL current production are considerably harder than the old-production granules and that MCW crystals dried below 32° C. are harder than those dried at a temperature above the transition point. Accurate data with the MCW crystals were hard to obtain, however, owing to their plasticity and irregular shape and to the fact that their resistance to crushing is less in the direction of their longitudinal axis than in the transverse direction. The TVA

TABLE 3.—Crushing strength and break-down in tumbling test of dry ammonium nitrate granules and crystals

Type of material	Producer	Crushing strength of particles, 8 to 10 mesh	Particles having definite crushing point		Break-down in tumbling test	
		Pounds	Percent		Percent	
Sprayed, old production.....	ANPL	1.30	89		3.95	
Sprayed, current production...	do.	2.47	90		.95	
Monocrystalline, dried below 32° C.	MCW	3.78	39		7.65	
Monocrystalline, dried above 32° C.	do.	1.03	43		0.15	

granules were too small to be conveniently used in these crushing-strength tests.

The percentage of break-down that occurred when the materials were subjected to a tumbling test is shown in the last column of table 3. The test consisted in tumbling 200 grams of 5- to 14-mesh samples in 16-ounce bottles end over end for 48 hours and then determining the proportion of each material that passed the 14-mesh sieve. The test indicated that the MCW crystals have the greatest tendency to break down under the conditions of the test and the ANPL current-production granules the least.

Ammonium nitrate granules are compared in crushing strength with those of other well-known materials and mixtures in table 4.

TABLE 4.—Relative crushing strength at 30° C. of dry granules of various fertilizer materials and mixtures

Fertilizer material	Method of granulation	Crushing strength of single granules, 6 to 8 mesh	Crushing strength in mass
		Pounds	Pounds per square inch
Ammonium phosphate	Rotary drying	12.0	460
Ammonium nitrate <sup>1</sup>	Spraying	2.8	75
Do. 1	do.	4.9	190
Potassium nitrate	do.	0.6	230
Sodium nitrate	do.	4.0	120
Mixed fertilizer	Rotary drying	0.4	220
Do. 1	do.	3.7	110
Superphosphate <sup>2</sup>	do.	5.5	180
Do. 1	do.	1.5	35

<sup>1</sup> Old production.

<sup>2</sup> Current production.

<sup>3</sup> Granulated to give hard dense granules.

<sup>4</sup> Granulated to give soft porous granules.

Most of the granules used in the test were between 6 and 8 mesh. The crushing strength of spherical granules of smaller diameter was calculated on the basis that this factor varies directly with the square of the diameter. The results show that ammonium nitrate granules range in crushing strength between those of other materials and mixtures and that they are sufficiently strong to withstand reasonable handling and to support the loads to which they would normally be subjected in storage (17).

### SOLUBILITY

A comparison of the solubility of ammonium nitrate at different temperatures with that of several other fertilizer salts (table 5) shows

TABLE 5.—Solubility of fertilizer materials in water  
(grams per 100 grams of water)<sup>1</sup>

Temperature	Ammonium nitrate	Sodium nitrate	Ammonium sulfate	Potassium chloride	Potassium sulfate	Urea
°C.						
0 (32)	118.3	73.0	70.0	27.6	7.35	55.0
20 (68)	192.0	87.7	76.4	34.0	11.11	70.0
40 (104)	267.0	103.5	81.0	40.0	14.70	100.0
60 (140)	321.0	123.0	88.0	45.5	18.17	132.0
80 (176)	580.0	148.0	95.3	51.1	21.40	
100 (212)	871.0	178.0	103.3	56.7	24.10	

<sup>1</sup> From Alberton Seldell (21a).



that at ordinary temperatures the solubility of ammonium nitrate exceeds that of any of the other materials listed. Its solubility is also influenced by temperature to a greater degree than that of any other fertilizer material. Temperature changes, on the other hand, have relatively little effect on the solubility of some materials, as ammonium sulfate. The high solubility of ammonium nitrate and the marked effect of temperature on its solubility are important factors influencing its tendency to cake, as discussed later in greater detail.

### DETERMINATION OF MOISTURE CONTENT

A slight change in the moisture content of soluble fertilizer salts has been shown to have a greater effect on their mechanical condition than a corresponding small change in any other factor that affects their tendency to cake (3). It was felt that this should hold particularly true for ammonium nitrate, because of its high solubility and wide variation in solubility with temperature. An accurate method for the determination of moisture in ammonium nitrate became imperative therefore if any progress was to be made in improving its mechanical condition.

It is known that ammonium nitrate undergoes slow decomposition at 100° C., and it did not seem, therefore, that the official method (4) was adapted to the determination of moisture in a material of this kind. With a view to finding a suitable method, sets of standard ammonium nitrate samples were prepared and submitted to a number of laboratories in which determinations for moisture in this material are known to have been made. The samples were accompanied by the request that they be analyzed by such methods as are considered best suited for the determination of their moisture content. The standard samples prepared for this collaborative study are given in table 6 and the results in table 7.

Standard sample ANPL No. 1 was selected from a commercial shipment that had been stored in a dry place. Sample ANPL No. 2 was also selected from a commercial shipment, but its moisture content was increased by spraying with water while it was being rolled in a rotating drum. Sample WCW No. 3 was collected at the plant before it was dried to the point desired for shipment. TVA samples Nos. 4 and 5 were taken from commercial shipments that had been stored in bags for a period of 3 months at 55° F. at a relative humidity of 80

TABLE 6.—Standard ammonium nitrate samples<sup>1</sup>

Source and sample No.	Type of granules	Treatment	Moisture in sample
Alberta Nitrogen Products, Ltd.: ANPL No. 1	Sprayed	None	Original.
ANPL No. 2	do	0.5 percent WP + 3.5 percent Kittitas.	Added.
Welland Chemical Works, Ltd.: WCW No. 3	do	None	Original.
Tennessee Valley Authority: TVA No. 4	Grained	do	Absorbed.
TVA No. 5	do	1.0 percent PRP + 4.0 percent Cellite.	Do.
Military Chemical Works, Inc.: MCW No. 6	Monocrystalline	None	Original.
MCW No. 7	do	do	Do.

<sup>1</sup> Results of analyses shown in table 7.

TABLE 7.—Moisture in standard ammonium nitrate samples

Collaborator	Method	Moisture in samples <sup>1</sup> —						
		ANPL No. 1	ANPL No. 2	WCW No. 3	TVA No. 4	TVA No. 5	MCW No. 6	MCW No. 7
Alberta Nitrogen Products, Ltd.	2 hours in oven at 70° C.	Percent 0.13	Percent 2.97	Percent 0.74	Percent 0.75	Percent 0.92	Percent 0.17	Percent 0.75
Do.	Fisher (14)							
Hercules Powder Co.	2 hours in oven at 75° C.	.09	2.52	.72	.70	.99	.09	.85
Solvay Process Co.	2 hours in vacuum at 80° C.	.11	2.70	.50	.57	.85	.11	.44
Division of Soils, Fer- tilizers, and Irrigation.	2 hours in vacuum	.09	2.67	.89	.68	.83	.07	.71
Do.	at 80° C.							
Consolidated Mining & Smelting Co., Ltd.	20 hours in oven at 70° C.	.08	2.91	.74	.75	.78	.08	.66
Welland Chemical Works, Ltd.	Air at 70° C. through sample for 4 hours.	.07	2.89	.80	.79	.80	.08	.60
Do.	4 hours in oven at 60° to 85° C.	.08	2.75	.72	.65	.80	.03	.55
Do.	Dry air at 65° to 70° C. through sample for 40 min- utes.	.07	2.69	.78	.61	.70	.06	.33
Military Chemical Works, Inc.	5 hours in oven at 70° C.	.09	2.59	.64	.47	.71	.11	.47
Do.	2 hours in oven at 100° C.	.08	2.76	.71	.50	.68	.09	.42
Eastern States Farm- ers' Exchange.	Air at 60° C. through sample for 3 hours.	.09	2.73	.78	.58	.77	.09	.47
Do.	5 hours in oven at 100° C.	.11	2.79	.76	.61	.81	.09	.43
Tennessee Valley Au- thority.	Dry air at 60° C. through sample for 6 hours.	.07	2.72	.75	.78	.81	.08	.67
Do.	Dry air at 70° C. through sample for 5 hours.	.08	2.68	.91	.83	.79	.10	.60
Do.	20 hours in oven at 70° C.	.09	2.84	.82	.71	.83	.09	.73
Mean.		.09	2.75	.75	.67	.82	.09	.58

<sup>1</sup> See table 6 for type of granules and treatment.

percent. Samples MCW Nos. 6 and 7 were prepared on a pilot-plant scale; No. 6 was dried as completely as was considered practical under commercial conditions, and No. 7 was represented as being only partially dried. The moisture in samples ANPL No. 1, WCW No. 3, and MCW Nos. 6 and 7 represents original moisture that had not been eliminated by drying, while that in samples ANPL No. 2 and TVA Nos. 4 and 5 represents moisture that had been added or absorbed subsequent to drying.

The results reported by the collaborators for the moisture in the samples and the methods used in the determination of moisture as shown in table 7 show good agreement in general, considering the hygroscopic nature of the samples submitted for analysis. The results obtained with the Fisher method (14) show good agreement with the average values, but this method is not considered to be adapted to general use in fertilizer laboratories for the reason that it gives water of hydration in addition to free moisture in materials and mixtures containing water of hydration. The other methods used by the collaborators may be considered as adaptations of the following three methods: (1) Drying in an air oven at 70° C. for 20 hours; (2) drawing air at 70° C. through the sample for 2 hours; or (3) drying in a vacuum at room temperature over Anhydron for 20 hours.

Moisture was determined by these three methods in samples ANPL No. 2, WCW No. 3, TVA No. 4, and MCW No. 7. The results obtained are given in table 8, which shows that the first two procedures

TABLE 8.—Rates at which standard samples of ammonium nitrate decrease in weight on drying by different methods

AIR OVEN AT 70° C.

Time of drying (hours)	Loss in weight of sample			
	ANPL No. 2	WCW No. 3	TVA No. 4	MCW No. 7
	Percent	Percent	Percent	Percent
6.....	2.83	0.66	0.66	0.48
12.....	2.88	.72	.73	.59
18.....	2.90	.74	.75	.65
24.....	2.88	.74	.75	.67
30.....	2.92	.76	.78	.68

AIR FLOW AT 70° C.

1.....	2.82	0.58	0.73	0.41
2.....	2.87	.72	.77	.57
3.....	2.90	.82	.81	.71
4.....	2.90	.85	.81	.75
6.....	2.93	.89	.83	.78

VACUUM OVER ANHYDRONE AT ROOM TEMPERATURE

6.....	2.79	0.36	0.65	0.28
12.....	2.80	.36	.69	.28
18.....	2.82	.38	.70	.28
24.....	2.85	.40	.73	.29
30.....	2.85	.39	.73	.29

give closely agreeing results for moisture in all samples tested and that the method of drawing air at 70° C. through the sample for 3 hours is equivalent to 24-hours' drying in an air oven at the same temperature. The air-flow method has the advantage, therefore, of being more rapid than drying in an air oven and for this reason it is better adapted for use in control work.

The table further indicates that samples ANPL No. 2 and TVA No. 4, which contain only added or absorbed moisture, can be completely dried in a vacuum over Anhydron in 24 hours, but that this method of drying does not eliminate within the same period all the original moisture in such samples as WCW No. 3 and MCW No. 7. The results indicate that a part of the original moisture in ammonium nitrate is present as occluded moisture and that this moisture is more difficult to remove than absorbed moisture.

The moisture in monocrystalline ammonium nitrate, which drying in vacuum over Anhydron failed to remove, amounts to about 0.35 percent of the weight of the crystal. The data in table 2 indicate that nearly 2 percent of the volume of MCW crystals is void. It must follow, therefore, that the greater part of the void space in the crystals is filled with air rather than water.

### HYGROSCOPICITY

A material is said to be hygroscopic when it absorbs moisture from the air at ordinary temperatures and humidity. This occurs when the pressure of water vapor in the air exceeds that of the saturated

solution of the material. The relative hygroscopicity of the more common fertilizer materials as determined by Adams and Merz (2) is indicated in table 9, which shows that calcium nitrate is the most

TABLE 9.—*Relative hygroscopicities of soluble fertilizer materials*

Fertilizer material	Humidity of air in equilibrium with saturated solution of material at—						
	10° C.	15° C.	20° C.	25° C.	30° C.	40° C.	50° C.
	Percent	Percent	Percent	Percent	Percent	Percent	Percent
Calcium nitrate.....	55.9	55.9	55.4	50.5	40.7	35.4	48.4
Ammonium nitrate.....	75.3	69.8	66.9	62.7	59.4	53.5	74.1
Sodium chloride.....	76.6	77.0	77.0	75.5	75.2	74.7	67.3
Sodium nitrate.....	78.0	78.8	77.1	74.4	72.4	70.1	71.3
Ammonium chloride.....	79.5	79.2	79.3	70.0	77.2	73.7	77.8
Ammonium sulfate.....	79.8	79.3	81.0	81.8	79.2	78.2	77.8
Urea.....	81.8	79.0	80.0	75.8	72.5	68.0	62.5
Potassium chloride.....	88.3	89.2	88.7	83.4	81.0	81.2	80.0
Potassium nitrate.....	97.0	95.6	92.3	92.0	90.5	87.3	85.0
Monocalcium phosphate.....	97.8	97.0	91.7	91.9	91.6	90.3	88.2
Monocalcium phosphate.....	97.9	98.8	94.1	95.0	93.7	94.5	94.0
Monopotassium phosphate.....	98.0	98.4	96.2	95.4	92.9	92.9	92.6
Potassium sulfate.....	99.1	99.7	98.5	98.8	99.3	95.7	95.8

hygroscopic, ammonium nitrate is next, and potassium sulfate is the least hygroscopic of the soluble fertilizer materials.

#### TEMPERATURE AND HUMIDITY RELATIONS

The temperature and humidity relationships that influence moisture absorption by ammonium nitrate can best be understood by reference to figure 4. The curves in the figure show (1) the variations in the pressure of saturated aqueous vapor in the air (absolute humidity, or 100 percent relative humidity) at different temperatures between 0° C. (32° F.) and 50° C. (122° F.); (2) the aqueous vapor pressures for the same temperature range at 60, 65, 70, and 75 percent saturation (relative humidities); and (3) the vapor pressures of saturated solutions of ammonium nitrate at different temperatures between 0° and 50° C. (2). The ammonium nitrate curve cuts the curves corresponding to relative humidities of 75, 70, 65, and 60 percent at temperatures of 10°, 15°, 21.5°, and 28.5° C., respectively.

This means that at these temperatures ammonium nitrate is in equilibrium with the water vapor in the air at the relative humidities indicated, or, in other words, that the pressure of the water vapor in the air at each specified temperature and humidity is equal to that of a saturated solution of ammonium nitrate at the same temperature. If the water vapor in the air at any given temperature exceeds that of a saturated solution of ammonium nitrate at the same temperature, moisture will be absorbed. If the conditions are reversed, however, so that the water vapor in the air is less than that of a saturated solution of ammonium nitrate, a drying of the material will take place as a result of loss of moisture to the surrounding air.

The curves in figure 4 further show that at temperatures below freezing, the pressure of saturated or partially saturated water vapor in the air is very small compared with that in the air at summer temperatures. It would be expected, therefore, that ammonium nitrate would exhibit little tendency to absorb moisture from the air at temperatures below freezing, even when the relative humidity approaches 100 percent.

A saturated solution of ammonium nitrate has a vapor pressure of 18.9 mm. of mercury at 30° C. (86° F.). The aqueous vapor pressure of the air at the same temperature and a relative humidity, for example, of 50 percent is only 15.8 mm. Ammonium nitrate will therefore not absorb any moisture from the air under these conditions. If the temperature were to drop to 20° C. (68° F.) without change in the water vapor in the air, the aqueous vapor pressure would then remain the same at 15.8 mm., but the relative humidity would have increased to 90 percent. If the ammonium nitrate were not allowed

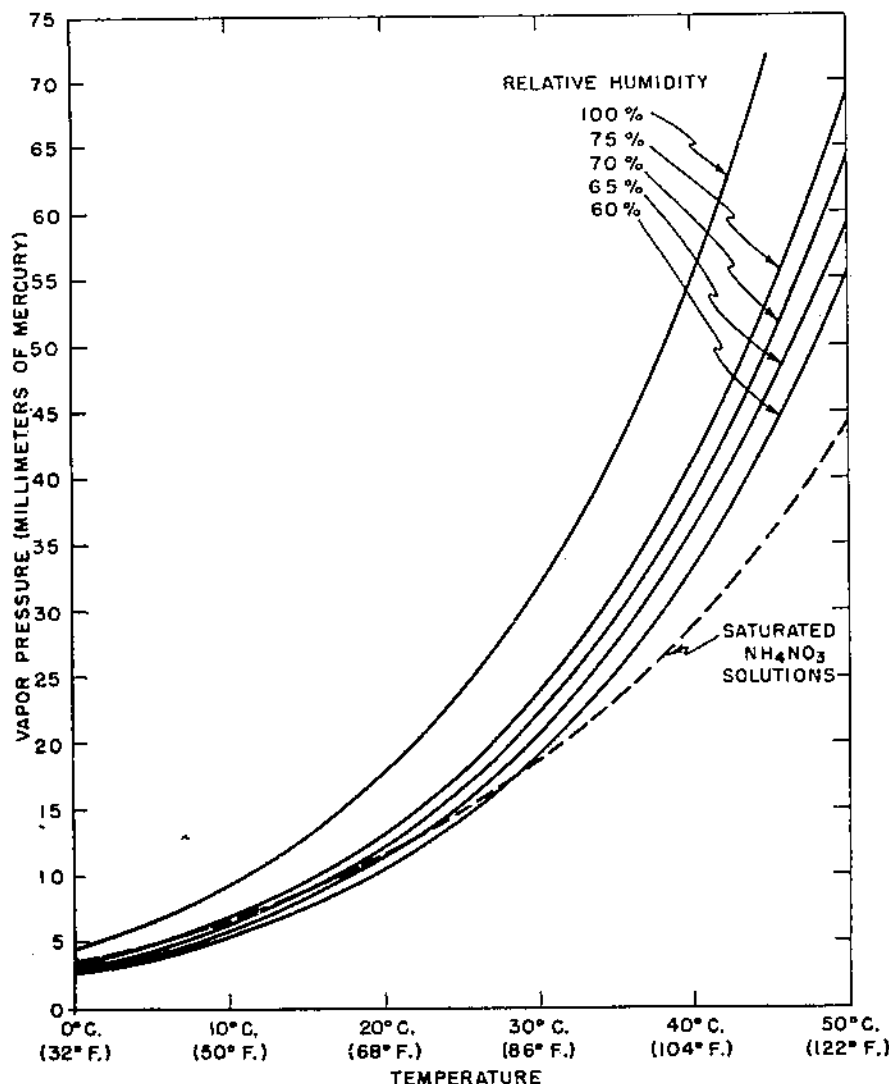


FIGURE 4.—Variations with temperature of (1) the aqueous vapor pressure in air corresponding to various relative humidities; and (2) the vapor pressure of saturated ammonium nitrate solutions.

to cool during the same interval, the vapor pressure of its saturated solution would still remain at 18.9 mm. and no absorption of moisture would take place, as the relative humidity of the surrounding air is increased to 90 percent. If the temperature of the ammonium nitrate is allowed to decrease with decrease in the temperature of the air, however, the vapor pressure of its saturated solution at the lower temperature of 20° C. will be only 11.7 mm. as compared with an aqueous vapor pressure of 15.8 mm. in the surrounding air, and a marked absorption of moisture will then take place.

If ammonium nitrate were to be granulated by the spraying method in a very humid section of the country, it might be expected that the air in the spraying tower would have to be dried if absorption of moisture by the sprayed material is to be prevented. That this is not necessary is shown in figure 4. According to the figure the vapor pressure of a saturated solution of ammonium nitrate at 45° C. (113° F.) is 35 mm. This is higher than that represented by the pressure (32 mm.) of water vapor in the air at 30° C. and a relative humidity of 100 percent. It follows therefore that if sprayed ammonium nitrate is not cooled below a temperature of 45° C. in the spraying tower it will not absorb any moisture at air temperatures below 30° C., even at a relative humidity of 100 percent. It should not be necessary therefore to dry the air passed through the tower in the process of spraying ammonium nitrate, provided the nitrate is collected at a temperature 10° to 15° C. above that of the surrounding air.

These observations show that ammonium nitrate can be stored without danger of moisture absorption (1) by maintaining the relative humidity of the air below that at which ammonium nitrate absorbs moisture, or (2) by maintaining the ammonium nitrate at a temperature about 10° C. above that of the surrounding air.

The relative humidity of the air below which ammonium nitrate does not absorb moisture varies from 75 percent at 10° C. (50° F.) to 52 percent at 40° C. (104° F.). The pressure of water vapor in the air at 10° C. and a relative humidity of 75 percent amounts to about 7 mm. At 40° C. and the same relative humidity the pressure of water vapor in the air is about 41.5 mm., or nearly six times as great. This explains why ammonium nitrate absorbs moisture more rapidly during hot weather than in winter and why the relative humidity alone gives little or no indication of the behavior of ammonium nitrate with respect to moisture absorption. To know this the temperature also must be specified. Inasmuch as the temperature varies greatly in different sections of the country and in the same section at different seasons, the behavior of ammonium nitrate in different localities and at different seasons may vary greatly, even if the relative humidity were to remain unchanged.

#### LOCATION AND HUMIDITY RELATIONS

Data on the manner in which ammonium nitrate is likely to behave under normal conditions in different parts of the country are given in table 10. This table shows for each month (1) the average monthly temperature over a period of 20 years for the places listed (13); (2) the average monthly aqueous vapor pressure over the same period; and (3) the difference between the average aqueous vapor pressure

TABLE 10.—Average monthly temperature for 20 years, average aqueous vapor pressure (V. P.) in the air, vapor pressure of saturated solution of ammonium nitrate, and the difference between the latter two at the average monthly temperature, for different places in the United States

Place	January				February				March			
	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>
	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury
Atlanta, Ga.	41.8	5.44	5.25	0.19	41.2	4.90	5.15	-0.25	52.1	7.29	7.35	-0.06
Baltimore, Md.	33.1	3.66	4.03	-0.37	31.3	3.25	3.81	-0.56	42.2	4.95	5.40	-0.45
Bismarck, N. Dak.	7.8	1.30		(?)	8.4	1.42		(?)	22.8	2.44		(?)
Boston, Mass.	28.1	3.12	3.45	-0.33	26.4	2.60		(?)	36.3	4.06	4.46	-0.40
Buffalo, N. Y.	25.4	3.00		(?)	21.6	2.54		(?)	31.6	3.71	3.84	-0.13
Cincinnati, Ohio.	32.4	3.91	3.93	-0.02	30.1	3.40	3.68	-0.28	43.0	5.23	5.54	-0.31
Columbia, S. C.	41.3	5.66	5.79	-0.13	43.9	5.33	5.70	-0.37	54.8	7.62	7.98	-0.36
Denver, Colo.	31.1	2.29	3.78	-1.49	29.7	2.24	3.60	-1.36	37.6	2.82	4.65	-1.83
Des Moines, Iowa.	21.4	2.57		(?)	21.6	2.51		(?)	35.5	3.91	4.85	-0.94
Detroit, Mich.	24.5	3.00		(?)	21.4	2.59		(?)	32.6	3.84	3.96	-0.12
Fort Worth, Tex.	46.5	5.59		-0.61	45.0	5.31	5.90	-0.59	55.4	6.91	8.14	-1.23
Indianapolis, Ind.	28.5	3.51	3.50	-0.01	26.5	3.15		(?)	39.7	4.85	4.97	-0.12
Little Rock, Ark.	41.8	5.31	5.36	-0.05	41.1	4.80	5.22	-0.42	53.0	7.56	7.55	0.01
Madison, Wis.	17.3	2.29		(?)	16.9	2.18		(?)	30.6	3.53	3.73	-0.20
Meridian, Miss.	46.2	6.86	6.15	-0.71	45.4	6.20	5.98	-0.22	56.1	8.60	8.30	-0.30
Montgomery, Ala.	46.9	6.71	6.27	-0.44	47.1	6.45	6.31	-0.14	57.6	9.09	8.68	-0.41
Nashville, Tenn.	38.7	4.88	4.82	-0.06	37.3	4.47	4.60	-0.13	49.5	6.60	6.78	-0.18
New Orleans, La.	52.8	8.69	7.50	-1.19	52.9	8.41	7.54	-0.87	62.0	11.46	9.88	-1.58
Norfolk, Va.	40.0	5.13	5.02	-0.11	38.0	4.67	4.70	-0.03	47.7	6.71	6.43	-0.28
Northfield, Vt.	14.4	2.03		(?)	13.4	1.83		(?)	25.9	3.00		(?)
Parkersburg, W. Va.	32.1	4.05	3.60	-0.46	29.7	3.61	3.60	-0.01	42.3	5.33	5.43	-0.10
Raleigh, N. C.	39.8	4.83	4.98	-0.15	39.0	4.42	4.56	-0.14	49.9	6.60	6.87	-0.27
San Francisco, Calif.	49.8	7.32	6.85	-0.47	52.0	7.62	7.53	-0.09	52.6	7.54	7.45	-0.09
Savannah, Ga.	48.7	7.11	6.92	-0.19	48.6	6.93	6.60	-0.33	57.3	9.88	8.59	-1.29
Seattle, Wash.	38.6	5.54	4.80	-0.74	42.3	5.59	5.42	-0.17	44.7	5.66	5.84	-0.18
Shreveport, La.	47.2	6.45	6.33	-0.12	47.0	6.15	6.28	-0.13	57.5	8.74	8.73	0.01
St. Paul, Minn.	13.4	1.85		(?)	14.0	1.91		(?)	29.0	3.10	3.50	-0.40
Tampa, Fla.	57.8	10.29	8.73	-1.56	58.2	10.29	8.82	-1.47	64.8	12.60	10.72	-1.88
Wichita, Kans.	31.1	3.35	3.75	-0.43	35.8	3.30	4.38	-1.08	43.7	4.83	5.68	-0.85

TABLE 10.—Average monthly temperature for 20 years, average aqueous vapor pressure (V. P.) in the air, vapor pressure of saturated solution of ammonium nitrate, and the difference between the latter two at the average monthly temperature, for different places in the United States—Continued

Place	April				May				June			
	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>
	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury
Atlanta, Ga.	58.8	8.53	8.98	-0.45	68.8	11.63	12.01	-0.38	74.9	14.99	14.23	0.76
Baltimore, Md.	52.6	6.50	7.45	-0.95	64.0	10.01	10.45	-0.44	71.7	13.54	13.03	.51
Bismarck, N. Dak.	42.7	4.39	5.48	-1.09	54.0	6.71	7.79	-1.08	63.7	10.16	10.37	-.21
Boston, Mass.	45.9	5.51	6.08	-.57	56.9	6.96	8.50	-1.54	65.6	11.23	10.96	.27
Buffalo, N. Y.	42.5	5.21	5.45	-.24	54.6	8.18	7.93	-.25	64.6	11.38	10.65	.73
Cincinnati, Ohio.	53.0	6.60	7.55	-.95	64.6	9.96	10.65	-.69	72.6	13.13	13.30	-.17
Columbia, S. C.	61.0	8.89	9.58	-.69	70.2	12.09	12.65	-.56	75.5	15.85	14.45	1.40
Denver, Colo.	46.7	3.68	6.28	-2.65	55.1	5.33	8.07	-2.74	64.4	7.06	10.57	-3.51
Des Moines, Iowa.	49.5	5.99	6.78	-.79	61.1	9.30	9.61	-.31	70.1	12.75	12.46	.29
Detroit, Mich.	45.1	5.54	5.92	-.38	57.7	8.59	8.70	-.11	66.8	11.79	11.79	.00
Fort Worth, Tex.	62.8	9.22	10.10	-.88	69.7	12.55	12.33	.22	79.7	14.78	16.16	-1.38
Indianapolis, Ind.	50.8	6.71	7.05	-.35	62.8	9.80	10.10	-.30	71.2	12.83	12.85	-.02
Little Rock, Ark.	60.7	9.09	9.50	-.41	69.3	12.90	12.19	.71	76.6	15.90	14.90	1.00
Madison, Wis.	44.2	3.18	5.76	-.58	56.6	8.13	8.42	-.29	66.6	11.89	11.27	.62
Meridian, Miss.	62.2	10.31	9.93	.38	70.1	13.18	12.48	.70	76.3	16.74	14.75	1.99
Montgomery, Ala.	63.2	10.16	10.23	-.07	72.1	13.31	13.17	.14	78.0	16.71	15.44	1.27
Nashville, Tenn.	67.4	7.98	8.62	-.64	67.7	11.40	11.63	-.23	75.3	14.96	14.37	.59
New Orleans, La.	66.8	12.80	11.35	1.45	73.9	15.98	13.85	2.13	79.3	18.97	15.98	2.99
Norfolk, Va.	55.3	8.31	8.11	.20	65.2	11.99	10.84	1.15	72.5	15.80	13.33	2.47
Northfield, Vt.	39.5	4.67	4.94	-.27	53.4	7.65	7.65	0.00	61.6	10.49	9.75	.74
Parkersburg, W. Va.	51.7	6.86	7.26	-.40	63.3	10.34	10.25	.09	70.4	13.92	12.57	1.35
Raleigh, N. C.	57.4	7.85	8.62	-.77	67.6	11.63	11.60	.03	74.0	15.31	13.88	1.46
San Francisco, Calif.	54.1	7.77	7.77	0.00	54.6	8.33	7.93	.40	66.0	8.92	8.28	.64
Savannah, Ga.	62.8	10.97	10.10	.87	71.4	14.96	12.92	2.04	76.9	18.44	15.00	3.44
Seattle, Wash.	49.2	6.27	6.73	-.46	54.4	7.47	7.90	-.43	65.8	8.59	8.08	.51
St. Louis, Mo.	63.8	10.54	10.40	.14	71.7	14.22	13.02	1.20	78.4	17.37	15.60	1.77
St. Paul, Minn.	45.4	4.93	5.98	-.105	57.3	7.62	8.60	-.98	66.7	11.05	11.31	-.26
Tampa, Fla.	68.6	13.34	11.95	1.39	75.0	16.36	14.25	2.11	78.3	19.28	15.57	3.71
Wichita, Kans.	55.1	6.91	8.06	-.115	64.6	10.52	10.64	-.12	73.8	13.82	13.82	0.00



TABLE 10.—Average monthly temperature for 20 years, average aqueous vapor pressure (V. P.) in the air, vapor pressure of saturated solution of ammonium nitrate, and the difference between the latter two at the average monthly temperature, for different places in the United States—Continued

Place	July				August				September			
	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — Vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — Vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — Vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>
	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury
Atlanta, Ga.	76.0	17.17	14.63	2.54	75.5	17.22	14.46	2.76	71.3	14.22	12.88	1.34
Baltimore, Md.	76.4	15.82	14.80	1.02	74.3	15.54	13.99	1.55	68.1	13.16	11.78	1.38
Bismarck, N. Dak.	69.0	11.33	12.08	— .75	66.1	10.52	11.13	— .61	55.5	7.49	8.15	— .66
Boston, Mass.	71.3	13.02	12.80	1.03	68.8	13.39	12.01	1.38	62.5	11.20	10.00	1.20
Buffalo, N. Y.	70.3	13.82	12.53	1.29	68.5	13.00	11.90	1.10	62.6	10.97	10.04	.93
Cincinnati, Ohio	76.4	14.01	14.80	— .79	74.8	14.58	14.19	.39	68.2	11.99	11.53	.46
Columbia, S. C.	77.8	17.88	15.37	2.51	76.2	18.19	14.73	3.46	72.0	15.31	13.11	2.20
Denver, Colo.	68.8	8.56	12.02	— 3.46	68.7	8.33	11.98	— 3.65	60.5	6.10	9.44	— 3.34
Des Moines, Iowa	74.6	14.38	14.11	.27	72.6	14.20	13.37	.83	63.5	11.20	10.30	1.00
Detroit, Mich.	71.6	13.51	13.00	.51	69.2	13.11	12.15	.96	62.5	10.92	10.02	.90
Fort Worth, Tex.	82.8	15.98	17.48	— 1.50	81.9	16.24	17.01	— .80	75.7	14.12	14.53	— .41
Indianapolis, Ind.	75.0	14.55	14.26	.29	73.5	14.12	13.55	.57	66.0	11.58	11.09	.49
Little Rock, Ark.	79.3	18.29	15.97	2.32	78.5	18.11	15.65	2.46	72.8	15.09	13.45	1.64
Madison, Wis.	71.1	13.31	12.82	.49	68.6	13.11	11.91	1.17	60.8	10.77	9.53	1.24
Meridian, Miss.	77.8	18.59	15.38	3.23	77.3	18.75	15.16	3.59	72.8	16.26	13.45	2.81
Montgomery, Ala.	78.9	18.62	15.81	2.81	78.3	19.05	15.55	3.50	74.6	16.07	14.11	1.96
Nashville, Tenn.	77.8	18.87	15.39	3.51	76.7	16.87	14.93	1.94	70.9	13.72	12.74	.98
New Orleans, La.	80.3	20.37	16.33	4.01	80.1	20.73	16.28	4.45	77.4	18.90	15.20	3.70
Norfolk, Va.	76.8	18.41	14.97	3.47	75.7	18.31	14.53	3.81	70.7	15.31	12.68	2.66
Northfield, Vt.	66.1	12.83	11.13	1.70	61.7	11.71	9.78	1.93	54.3	9.40	7.86	1.51
Parkersburg, W. Va.	74.1	15.77	13.92	1.85	72.3	15.01	13.27	1.77	65.6	12.45	10.95	1.50
Intleigh, N. C.	76.8	17.53	14.97	2.56	75.2	17.42	14.35	3.07	70.0	14.45	12.43	2.02
San Francisco, Calif.	56.2	9.55	8.33	1.22	56.9	9.91	8.48	1.43	59.1	9.86	9.07	.79
Savannah, Ga.	78.7	20.01	15.73	4.31	78.1	20.29	15.48	4.81	74.3	18.29	14.00	4.29
Seattle, Wash.	63.5	9.88	10.30	— .42	63.0	10.05	10.17	— .11	58.2	9.19	8.83	.36
Shreveport, La.	80.2	18.87	16.33	2.54	80.0	18.90	16.29	2.61	74.9	16.05	14.23	1.82
St. Paul, Minn.	71.2	12.75	12.55	.20	68.3	12.09	11.85	.24	59.6	9.55	9.20	.35
Tampa, Fla.	79.3	20.52	15.98	4.54	79.2	20.83	15.95	4.88	77.3	19.71	15.17	4.54
Wichita, Kans.	77.8	15.44	15.37	.07	77.2	14.94	15.13	— .19	68.7	11.99	11.98	.01

TABLE 10.—Average monthly temperature for 20 years, average aqueous vapor pressure (V. P.) in the air, vapor pressure of saturated solution of ammonium nitrate, and the difference between the latter two at the average monthly temperature, for different places in the United States—Continued

Place	October				November				December			
	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>	Average temperature	Average aqueous vapor pressure of air	Vapor pressure of saturated solution of $\text{NH}_4\text{NO}_3$	Vapor pressure of air — vapor pressure of saturated $\text{NH}_4\text{NO}_3$ solution <sup>1</sup>
	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury	°C.	Mm. of mercury	Mm. of mercury	Mm. of mercury
Atlanta, Ga.	60.3	9.58	9.39	0.19	50.5	6.86	7.00	-0.14	42.1	5.46	5.37	0.09
Baltimore, Md.	56.2	8.59	8.33	.26	45.1	5.54	5.93	-.39	35.3	3.91	4.33	-.42
Bismarek, N. Dak.	42.2	4.83	5.40	-.57	25.7	2.74		(?)	14.8	1.73		(?)
Boston, Mass.	52.6	7.77	7.45	.32	41.8	5.21	5.33	-.12	31.8	3.56	3.86	-.30
Buffalo, N. Y.	51.2	7.47	7.13	.34	39.7	4.98	4.97	.01	29.4	3.40	3.60	-.20
Cincinnati, Ohio	55.4	7.98	8.13	-.15	44.0	5.38	5.73	-.35	33.8	4.01	4.10	-.09
Columbia, S. C.	61.1	10.21	9.62	.59	51.2	6.81	7.13	-.32	44.1	5.69	5.73	-.04
Denver, Colo.	48.7	4.09	6.63	-2.54	38.8	2.79	4.84	-2.05	29.7	2.21	3.62	-1.38
Des Moines, Iowa	51.4	7.09	7.18	-.09	37.5	4.42	4.63	-.21	25.2	2.00		(?)
Detroit, Mich.	50.5	7.34	7.00	.34	38.4	4.88	4.77	.11	27.7	3.35	3.40	-.05
Fort Worth, Tex.	64.6	10.31	10.64	-.33	54.8	7.72	7.99	-.27	43.8	5.38	5.68	-.30
Indianapolis, Ind.	53.6	7.57	7.70	-.13	41.4	5.05	5.25	-.20	30.4	3.61	3.70	-.09
Little Rock, Ark.	61.0	10.26	9.58	.68	51.4	6.99	7.19	-.20	41.7	5.13	5.32	-.19
Madison, Wis.	48.2	6.65	6.52	.13	35.0	4.27	4.27	.00	22.6	2.79		(?)
Meridian, Miss.	60.5	10.82	9.45	1.37	51.3	7.98	7.17	.81	45.2	6.55	5.94	.61
Montgomery, Ala.	63.6	10.97	10.33	.64	54.0	8.00	7.78	.22	46.7	6.55	6.23	.32
Nashville, Tenn.	58.5	8.99	8.90	.09	48.0	6.20	6.48	-.28	39.0	4.83	4.86	-.03
New Orleans, La.	68.3	13.74	11.85	1.89	59.8	10.77	9.25	1.52	52.6	8.48	7.45	1.03
Norfolk, Va.	60.1	10.77	9.33	1.44	49.7	7.16	6.83	.33	41.4	5.36	5.25	.11
Northfield, Vt.	43.6	6.17	5.64	.53	31.7	3.86	3.83	.03	19.2	2.40		(?)
Parkersburg, W. Va.	52.8	8.03	7.50	.53	39.7	5.38	4.97	.41	33.3	4.11	4.05	.06
Raleigh, N. C.	58.7	9.60	8.95	.65	48.5	6.35	6.50	-.15	40.5	4.98	5.11	-.13
San Francisco, Calif.	58.7	9.25	8.95	.30	55.1	8.38	8.25	.13	50.4	7.16	6.98	.18
Savannah, Ga.	64.9	12.88	10.73	2.15	55.9	9.22	8.25	.97	49.0	7.26	6.68	.58
Seattle, Wash.	52.2	8.18	7.36	.82	46.0	6.81	6.09	.72	42.1	5.89	5.38	.51
Shreveport, La.	63.7	11.10	10.36	.74	55.0	9.26	8.02	.25	46.3	6.15	6.16	-.01
St. Paul, Minn.	47.6	6.10	6.40	-.30	32.0	3.61	3.88	-.27	19.2	2.29		(?)
Tampa, Fla.	71.4	15.80	12.92	2.88	63.9	12.45	10.43	2.02	58.0	10.54	8.77	1.77
Wichita, Kans.	56.4	7.87	8.36	-.49	44.0	5.23	5.73	-.50	32.5	3.51	3.94	-.43

<sup>1</sup> Minus sign indicates ammonium nitrate will dry out under average conditions; plus value that ammonium nitrate will absorb moisture.<sup>2</sup> Average temperature below freezing.

and that of a saturated ammonium nitrate solution at the average monthly temperature. A minus sign for this differential value indicates that ammonium nitrate will dry out under the average conditions prevailing at the place indicated, while a plus value indicates that it will absorb moisture. The larger the plus value the faster the rate at which moisture will be absorbed.

The table shows that at such places as Tampa, Fla., New Orleans, La., and San Juan, P. R., moisture will be absorbed by ammonium nitrate under normal conditions every month in the year, while the opposite will usually hold true for every month as a whole at Denver, Colo., Fort Worth, Tex., and Wichita, Kans. In most places ammonium nitrate will show a gain in moisture absorbed in summer, but not in winter.

### RATE OF MOISTURE ABSORPTION

A material begins to absorb moisture from the air when the pressure of the water vapor in the surrounding air exceeds that of the vapor pressure of its saturated solution. The rate at which moisture is absorbed will vary with the differential between the two vapor pressures and with such other factors as the surface area of the material exposed to the air, the movement of the air with respect to the material, and other factors. It may thus happen that a sample of one material may absorb moisture at a faster rate than another, even though its hygroscopicity may actually be less than that of the second material. A study was accordingly undertaken of methods for reducing the rate at which ammonium nitrate absorbs moisture by coating it with various materials with a view to reducing its exposed surface area.

### METHOD OF MEASUREMENT

In making this study it was imperative that all comparative measurements of rates of moisture absorption be made under such conditions that all factors affecting rates of moisture absorption be kept constant excepting the one under test. It was felt that the method and apparatus developed by Yee and Davis (30) complied with these requirements in the most satisfactory way. It was accordingly used in all subsequent tests on the value of various coating agents for reducing the rate at which ammonium nitrate absorbs moisture from the air. The apparatus used in this method consists of a metal humidity chamber provided with an aluminum top having six equally spaced holes through which the samples are introduced into the chamber. The samples are placed in 2-inch shallow dishes with flat bottoms and vertical sides. The dishes are supported by baskets suspended within the chamber. A saturated salt solution with an excess of the solid salt is contained in a shallow glass vessel in the bottom of the chamber. Continuous circulation of the air within the chamber is maintained by means of a fan operating at a speed of 350 r. p. m. The entire apparatus is placed in a constant-temperature room at 30° C.

In making a determination, 4-gram samples of the material or materials to be tested are weighed into the sample dishes, the surface of the material in the dishes is made as smooth as possible, and the dishes are then placed in the humidity chamber. At the end of any desired

interval (1 or 2 hours), the sample dishes are taken out of the humidity chamber and weighed with the covers on to determine the increase in weight. They are then replaced in the chamber for another like interval before reweighing. The increases in weight for the various time intervals are calculated as moisture-absorption rates in percentage of the sample. A relative humidity of 72.4 percent was found most convenient for determining the rate of moisture absorption of variously treated ammonium nitrate samples. This humidity was obtained by use of a saturated solution of sodium nitrate.

### MOISTURE-HOLDING CAPACITY

Saturated solutions of all three types of ammonium nitrate—sprayed, grained, and monocrystalline—have the same vapor pressure at a given temperature and all absorb moisture at essentially the same rate when exposed under the same conditions in a humid atmosphere, as shown in table 11. The particles of granular ammonium nitrate,

TABLE 11.—Effect of conditioners on the rate at which ammonium nitrate of different types absorbs moisture at 30° C. and a relative humidity of 72.4 percent

Type of material	Particle size	Conditioner added		Moisture absorbed in—		
		Percent of $\text{NH}_4\text{NO}_3$	Kind	2 hours	4 hours	6 hours
	Mesh			Percent	Percent	Percent
Sprayed.....	10-20	0	None.....	11.85	23.68	34.10
		4.0	Dicalite.....	12.36	23.79	34.14
		4.0	Kittitas.....	12.66	24.30	34.80
		4.0	Cellite.....	11.02	23.38	33.70
		4.0	Tricelcolum phosphate.....	12.16	23.60	33.00
Grained.....	10-40	0	None.....	12.21	23.17	33.08
		4.0	Dicalite.....	11.02	22.78	32.80
Fine crystals.....	-40	0	None.....	11.01	22.79	32.60
		4.0	Dicalite.....	12.65	24.62	35.27
Coarse crystals.....	10-20	0	None.....	11.50	22.94	32.77
		4.0	Dicalite.....	11.03	21.30	30.63
		4.0	Dicalite.....	11.11	20.78	30.02

as already explained, are somewhat porous for the reason that they are made up of aggregates of crystals. The particles of monocrystalline ammonium nitrate on the other hand are nonporous and are incapable of holding any appreciable quantity of moisture except on their surface. There is therefore a difference in the quantity of moisture that the granular and monocrystalline materials will absorb before they become dripping wet. Thus the solution phase in a sample of monocrystalline ammonium nitrate containing 1.5 percent of moisture will tend to settle out in the bottom of the container on standing. This quantity of moisture will not separate out as a solution phase from granular ammonium nitrate. Figure 5 shows that the MCW crystals have become joined together by a greater volume of solution than the ANPL granules, although both types of material were exposed to the same humidity for the same length of time.

### EFFECT OF WATER REPELLENTS

The hygroscopicity of a dry mixture of ammonium nitrate with an inert material is the same whether the mixture contains little or much

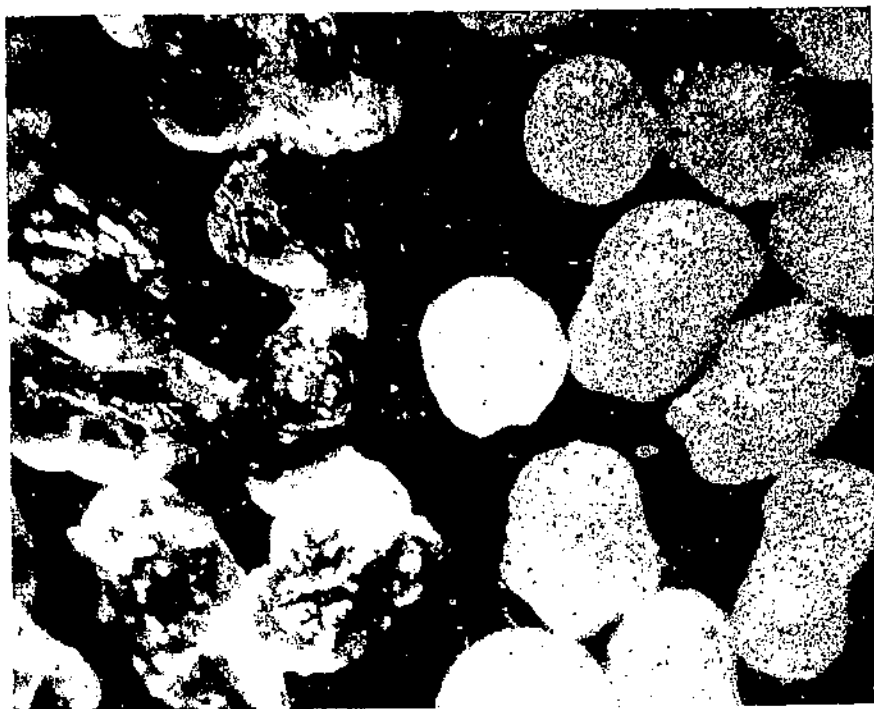


FIGURE 5. Sprayed granules (current production) on right, monocryystals on left. A greater volume of surface solution joins the monocryystals than the granules after both have been exposed to the same humidity for the same length of time.  $\times 10$ .

ammonium nitrate. The rate at which such a mixture absorbs moisture at humidities above the hygroscopic point of ammonium nitrate is not the same for all proportions of ammonium nitrate but increases with increase in the content of the ammonium nitrate until the concentration of the nitrate is sufficient to form a saturated-solution layer over the exposed surface of the sample within the period of the test. When this occurs no further increase in the rate of moisture absorption with increase in the proportion of the ammonium nitrate will take place.

In view of the foregoing, it would be expected that admixture of ammonium nitrate with a relatively small proportion of a conditioning agent in the form of a finely divided, water-insoluble material would have little effect in reducing its rate of moisture absorption. That this is actually the case is shown by the data in table 11. The table shows that materials such as kaolin, the different types of kieselguhr, and tricalcium phosphate have little or no value in reducing the rate at which ammonium nitrate absorbs moisture from the air at a humidity above its hygroscopic point. A material such as phosphate rock that contains calcium carbonate may react with the ammonium nitrate to increase rather than decrease its rate of moisture absorption.

The opposite effect was found to be true, however, in the case of such materials as petrolatum, paraffin, and rosin, which act as water repellents and are capable of forming a more or less impervious layer over the crystals or granules of ammonium nitrate. The value of these coating agents as retardants of moisture absorption by ammonium nitrate was studied by Krase, Yee, and Braham (23) about 20 years ago. Petrolatum was reported in this investigation to be more effective as a coating agent than various mixtures of paraffin and rosin.

In the method of coating described by Cairns (8) grained ammonium nitrate is treated with 0.1 to 0.2 percent of petrolatum, followed by the addition of sufficient rosin-paraffin mixture in the proportion of 80 to 20 to make the total mixture of petrolatum and the rosin-paraffin mixture equivalent to 1 percent of the product.

The results obtained in the present investigation with water-repellent coating agents are given in tables 12 to 14. In making these tests the coating material or mixture was heated to fusion and sprayed onto the ammonium nitrate while it was being rolled in a cement mixer. During the treatment the ammonium nitrate was maintained at a temperature of about 80° C. When coating small batches, it was found convenient to dissolve the coating agent in carbon tetrachloride. The resulting solution was then poured over the ammonium nitrate at a temperature of about 50° C. while it was being rolled in a small drum. The temperature of the material was then increased to a maximum of about 80° C. to drive off the carbon tetrachloride and leave a uniform coating of the water repellent on the surface of the granules. The concentration of the carbon tetrachloride was such that the desired coating of the water repellent was obtained when sufficient carbon tetrachloride was added to wet the entire mass. The results obtained with the two methods of applying the coating agent agreed very closely.

The results given in table 12 indicate that the most efficient coatings consist of combinations of equal parts of paraffin with petrolatum or

TABLE 12.—Rate of moisture absorption by coated and uncoated granular ammonium nitrate (10- to 20-mesh) at 30° C. and a relative humidity of 72.4 percent

Water-repellent coating agent	Amount added in percent of $\text{NH}_4\text{NO}_3$	Moisture absorbed in—		
		2 hours	4 hours	6 hours
		Percent	Percent	Percent
None.....	0	11.86	23.68	34.18
Petrolatum.....	.2	10.19	11.77	12.76
Do.....	.5	4.56	5.95	6.32
Do.....	1.0	2.50	2.93	3.29
Paraffin.....	.2	5.46	9.16	11.28
Do.....	.5	5.39	8.60	11.02
Do.....	1.0	4.75	8.07	9.87
Beeswax.....	1.0	2.82	5.21	7.55
Petrolatum + rosin.....	.2	8.56	10.23	10.02
Do.....	.5	6.43	7.98	8.22
Do.....	1.0	2.28	2.64	2.87
Paraffin + rosin.....	.2	4.60	8.05	9.01
Do.....	.5	4.02	6.70	8.50
Do.....	1.0	3.77	6.39	8.10
Paraffin + asphaltum.....	.5	.47	.51	1.43
Do.....	1.0	.36	.58	.78
Petrolatum + rosin + paraffin.....	.2	1.05	1.60	2.18
Do.....	.5	.49	.74	.92
Do.....	1.0	.34	.51	.69

TABLE 13.—Effect of water-repellent coatings on the rate at which ammonium nitrate of different types absorbs moisture at 30° C. and a relative humidity of 72.4 percent

Type of ammonium nitrate	Particle size	Water repellent added <sup>1</sup>		Moisture absorbed in—		
		NH <sub>4</sub> NO <sub>3</sub>	Kind	2 hours	4 hours	6 hours
	Mesh	Percent		Percent	Percent	Percent
Sprayed.....	10-20	0	None.....	11.86	23.68	34.16
		1.0	WP.....	.29	.38	.47
		.5	PRP.....	.49	.74	.92
		1.0	do.....	.34	.51	.69
Grained.....	10-40	0	None.....	11.92	22.78	32.80
		.5	PRP.....	.52	.75	.95
		1.0	do.....	.40	.59	.77
		0	None.....	12.65	24.62	35.27
Fine crystals.....	-40	.5	PRP.....	.37	.58	.74
		1.0	do.....	.19	.34	.46
		0	None.....	11.03	21.30	30.93
		.5	PRP.....	.67	1.04	1.33
Coarse crystals.....	10-16	1.0	do.....	.23	.39	.50

<sup>1</sup> Components of water repellent added in equal proportion: WP = paraffin-petrolatum; PRP = petrolatum-rosin-paraffin.

TABLE 14.—Effect of varying the proportions of petrolatum, rosin, and paraffin (PRP) on the rate at which granular ammonium nitrate (10- to 20-mesh) absorbs moisture at 30° C. and a relative humidity of 72.4 percent

PRP added		Moisture absorbed in—		
Percent of NH <sub>4</sub> NO <sub>3</sub>	Proportion of components	2 hours	4 hours	6 hours
		Percent	Percent	Percent
0.....		11.86	23.68	34.16
0.5.....	1:1:1	.40	.65	.85
	1:1:1	.34	.51	.69
	2:1:1	.44	.70	.89
	1:2:1	.34	.58	.81
1.0.....	1:1:2	.23	.45	.63
	4:1:1	.40	.64	.84
	1:4:1	.34	.63	.87
	1:1:4	.55	.90	1.21

asphalt or of petrolatum and paraffin with rosin or asphalt. The coating agent most commonly used in Canada for the commercial treatment of ammonium nitrate consists of a mixture of equal parts of paraffin and petrolatum. In the United States preference is given to a mixture of petrolatum, rosin, and paraffin. This PRP mixture, as it is commonly called, is about equally effective in reducing rate of moisture absorption by all types of ammonium nitrate (table 13), and the effectiveness of the mixture is not changed very much by a four-fold change in the proportion of any one of its components (table 14).

The addition of a conditioning agent to the petrolatum-rosin-paraffin coating nullifies to some extent the effect of the water repellent in reducing rate of moisture absorption by ammonium nitrate, as shown in table 15. In the tests in which Dicalite, plaster of paris, and tricalcium phosphate were directly compared, Dicalite without exception had nullified the effect of the water repellent to the greatest extent and tricalcium phosphate to the least extent. The extent to which a conditioning agent nullifies the effect of a water-repellent

TABLE 15.—Effect of a double coating of a water repellent and a conditioning agent on the rate at which granular ammonium nitrate (10- to 20-mesh) absorbs moisture at 30° C. and a relative humidity of 72.4 percent

Water repellent (PRP) added		Conditioning agent added		Moisture absorbed in—		
Percentage of $\text{NH}_4\text{NO}_3$	Proportion of components	$\text{NH}_4\text{NO}_3$	Kind	2 hours	4 hours	6 hours
		Percent		Percent	Percent	Percent
0		0	None	11.86	23.08	34.16
		0	do	.40	.05	.85
0.5	1:1:1	3.0	Cecil soil colloid	4.25	7.54	9.87
		3.0	Volclay	6.82	11.73	10.08
		3.0	Kaolin	3.76	6.76	8.70
		3.0	Tricalcium phosphate	2.62	3.98	4.02
1.0	1:1:1	0	None	.34	.51	.09
		4.0	Dicalite	2.80	4.82	0.04
		4.0	Plaster of paris	1.85	2.29	0.17
		4.0	Tricalcium phosphate	.46	.81	1.15
1.0	1:1:2	4.0	Dicalite	2.20	4.15	5.99
		4.0	Plaster of paris	1.87	2.34	2.69
		4.0	Tricalcium phosphate	.39	.74	1.08
1.0	1:1:4	4.0	Dicalite	2.50	4.39	6.20
		4.0	Plaster of paris	2.19	2.89	3.48
		4.0	Tricalcium phosphate	.71	1.32	1.88

coating varies not only with the conditioning agent used but also with the composition of the coating agent.

The rate at which ammonium nitrate absorbs moisture when coated with PRP (1:1:1) decreases slightly with time of storage, but the reverse is true when the PRP coating is followed by a second coating of a conditioning agent. Different conditioning agents differ in the rate at which they tend to nullify the effect of the coating agents. Tricalcium phosphate not only has a small initial effect on the PRP coating, but the rate at which its effect increases with time of storage is also relatively small.

### FACTORS IN CAKING TENDENCY

The principal factors that affect the caking of fertilizer materials are: (1) Moisture content, (2) particle size, (3) storage pressure, (4) temperature and duration of storage, and (5) treatment with conditioning agents.

The effect of these different factors in promoting caking varies greatly with different fertilizer materials. Thus, time of storage and applied pressure have little effect on the caking of well-cured superphosphate and monoammonium phosphate (3), but both factors have a marked effect on the caking of sodium nitrate, ammonium nitrate, and other materials of relatively low fusibility. The effect of the different factors mentioned on the caking of ammonium nitrate was determined by the method of Adams and Ross (3), which consists in subjecting a sample of the material to a pressure of 12 pounds per square inch for 7 days at 30° C. in a cylindrical bomb of special design. The bomb is 2 inches in internal diameter and has a total weight of  $23\frac{1}{4}$  pounds.

### MOISTURE

Slight changes in moisture content, as previously stated, have a greater effect on the caking of ammonium nitrate than a correspondingly small change in any of the other factors that affect its tendency



to cake. This result might be expected for the reason that the solubility of ammonium nitrate and the variation of its solubility with temperature exceeds that of any other fertilizer material. A decrease in moisture content or a lowering of the temperature will, therefore, result in a greater deposition of crystals than will take place for a like change in any other fertilizer material. The knitting together of the crystals that accompanies their deposition is a characteristic of the material undergoing recrystallization, but it usually causes the material to cake into a solid mass. A gradual increase in moisture content on the other hand without an accompanying drop in temperature has the opposite effect—the crystals that were deposited are redissolved and the hardness of the original cake is decreased.

While the presence of moisture may reduce caking under certain conditions, it greatly interferes at all times with the drillability of the material. It is also true that a given drop in temperature or humidity will cause a greater deposition of crystals in moist than in dry ammonium nitrate and will correspondingly increase the degree of caking of the moist material. Owing to the effect of these climatic factors on the caking of ammonium nitrate, it was expected and tests showed that its tendency to cake varies greatly in different localities.<sup>6</sup>

The effect of a decrease in moisture content on the caking of ammonium nitrate is shown by the data in table 16. In making the tests

TABLE 16.—Effect of change in the moisture content on the caking of ammonium nitrate when the temperature (30° C.), time of storage (7 days), and the applied pressure (12 lbs. per square inch) remained constant

Type of ammonium nitrate	Particle size	Moisture content			Crushing strength of briquets
		Initial	Final	Decrease during storage	
	Mesh	Percent	Percent	Percent	Pounds per square inch
Small crystals.....	-40	0	0	0	22
		.20	0	-.20	50
		.40	.01	-.31	325
		.60	.01	-.59	387
Sprayed.....	10-20	1.40	.51	-.89	500
		0	0	0	3
		.20	0	-.20	5
		.40	.01	-.39	42
Large crystals.....	10-16	1.40	.31	-1.09	500
		.25	.14	-.11	35
		.71	.38	-.33	92
		.62	.38	-.24	95

represented by the data in the table samples of ammonium nitrate containing varying quantities of moisture were stored in the caking bomb in a constant-temperature room at a relative humidity below 60 percent. Partial drying therefore took place during the 7-day storage period. The results given in the table show that the extent of caking increased with loss of moisture when all other factors were kept constant and that a relatively slight loss of moisture may cause a marked change in the degree of caking.

<sup>6</sup> ADAMS, J. R., LOVE, K. S., and ROSS, W. H. STORAGE EXPERIMENTS WITH VARIOUSLY CONDITIONED AMMONIUM NITRATE FERTILIZERS. Div. Soil and Fert. Invest. Res. Rpt. 26, 16 pp. 1944. [Processed.]

It becomes evident therefore that a study of the effect of various factors on the caking of ammonium nitrate is possible only when the moisture content is kept absolutely constant throughout the period of the test. This was accomplished by using only dry material in the study of the other factors affecting caking and by storing the caking bombs in a constant-temperature room having a relative humidity below the point at which the ammonium nitrate absorbs moisture from the air.

A comparison of the data in table 16 with the results given in tables 17 and 21 shows (1) that different types of ammonium nitrate show different degrees of caking when other factors are the same; (2) that granular ammonium nitrate cakes less than the monocrystalline material; and (3) that treatment with conditioning agents reduces caking of ammonium nitrate even when dry.

TABLE 17.—*Effect of particle size on the caking of dry ammonium nitrate when subjected to an applied pressure of 12 pounds per square inch in a dry place for 7 days at 30° C.*

Type of ammonium nitrate	Particle size	Crushing strength of briquets
	Mesh	Pounds per square inch
Sprayed.....	8-10	0
Do.....	10-20	14
Do.....	40	90
Grained.....	10-20	6
Do.....	40	18
Small crystals.....	40	70
Large crystals.....	10-16	35

Owing to variations in their physical properties some differences in the caking tendency of different types of ammonium nitrate would be expected. As previously explained ANPL granular material is somewhat porous, while the MCW material is essentially nonporous. Consequently, when a limited quantity of water (1.0 percent or less) is sprayed onto ANPL ammonium nitrate while it is being rolled in a drum, a considerable proportion of the water is absorbed and held within the granules. A subsequent lowering of the temperature will cause deposition of crystals within as well as outside the granules. To the extent that this deposition of crystals takes place on the surface of the granules it induces caking, but to the extent that it occurs within the granule it tends to increase the hardness of the granule. The crushing strength of a granule or crystal is weakened, however, by a repeated gain and loss of moisture and by fluctuations in temperature above and below a transition point.

In the case of the nonporous monocrystalline ammonium nitrate, all absorbed moisture must remain on the surface of the particle (fig. 5) and any deposition of crystals as a result of a lowering of the temperature or loss of moisture will be limited to the surface of the particles. Climatic changes following the absorption of a limited quantity of moisture is, therefore, likely to cause a greater degree of caking in monocrystalline than in granular ammonium nitrate of the same particle size.<sup>7</sup>

<sup>7</sup> See footnote 6, p. 30.

## PARTICLE SIZE

An increase in the particle size of ammonium nitrate, as well as of other fertilizer materials, lessens their tendency to cake by decreasing the points of contact between the particles and their surface area per unit weight. The farther apart the centers of two adjoining particles are, the easier it is to break them when they are joined by a given deposition of material. A material will therefore cake less if the particles are large rather than small and if the particles are spherical rather than irregular in shape. The treatment known as granulation, especially when it leads to the formation of spherical particles, is particularly effective in decreasing the points of contact between particles and therefore it affords one of the most effective means for decreasing the caking of soluble salts.

The ANPL ammonium nitrate first used in this investigation was prepared, as previously explained, by spraying a melt of the material containing about 4 to 5 percent of moisture. In the subsequent drying of the sprayed material, small crystals were deposited on the surface of the granules, as shown in figure 6, *A*. These projections on the granules increased the points of contact between them, with the result that the product prepared in this way exhibited a greater tendency to cake than the current material (fig. 6, *B*). The granules of this latest production are not only more uniform and spherical than those of the earlier product, but the material as a whole also shows less tendency to cake and is more free flowing. The type of granules produced by the graining method is shown in figure 7.

The monocrystalline ammonium nitrate used in this investigation (fig. 3) was not so uniform in shape as the sprayed material. Having therefore more points of contact between its particles, it exhibited a greater tendency to cake than was true of the granular material of similar particle size when all other factors were the same. This is indicated by the data in table 17, which show that large crystals of ammonium nitrate cake less than small crystals, while the sprayed ammonium nitrate in turn shows less tendency to cake than the monocrystalline material of the same mesh size.

It should be emphasized that these and all subsequent tests were made with the materials in the form in which they were originally produced. The surface uniformity of the particles of all types of ammonium nitrate is seriously impaired when they are subjected to repeated drying following absorption of moisture, and the areas of contact between them is greatly increased, as shown in figure 8.

The screen analyses of the different types of ammonium nitrate now on the market and of other inorganic nitrogenous materials are shown in table 18. The data in the table show that the average particle size of the current production of ANPL sprayed ammonium nitrate is somewhat larger than the old-production material and that this in turn is considerably larger than the commercially grained ammonium nitrate now on the market. The Champion Brand sodium nitrate does not differ greatly in particle size from the current production of sprayed ammonium nitrate, and the particles of both in turn are somewhat larger than those of the Arcadian sodium nitrate.

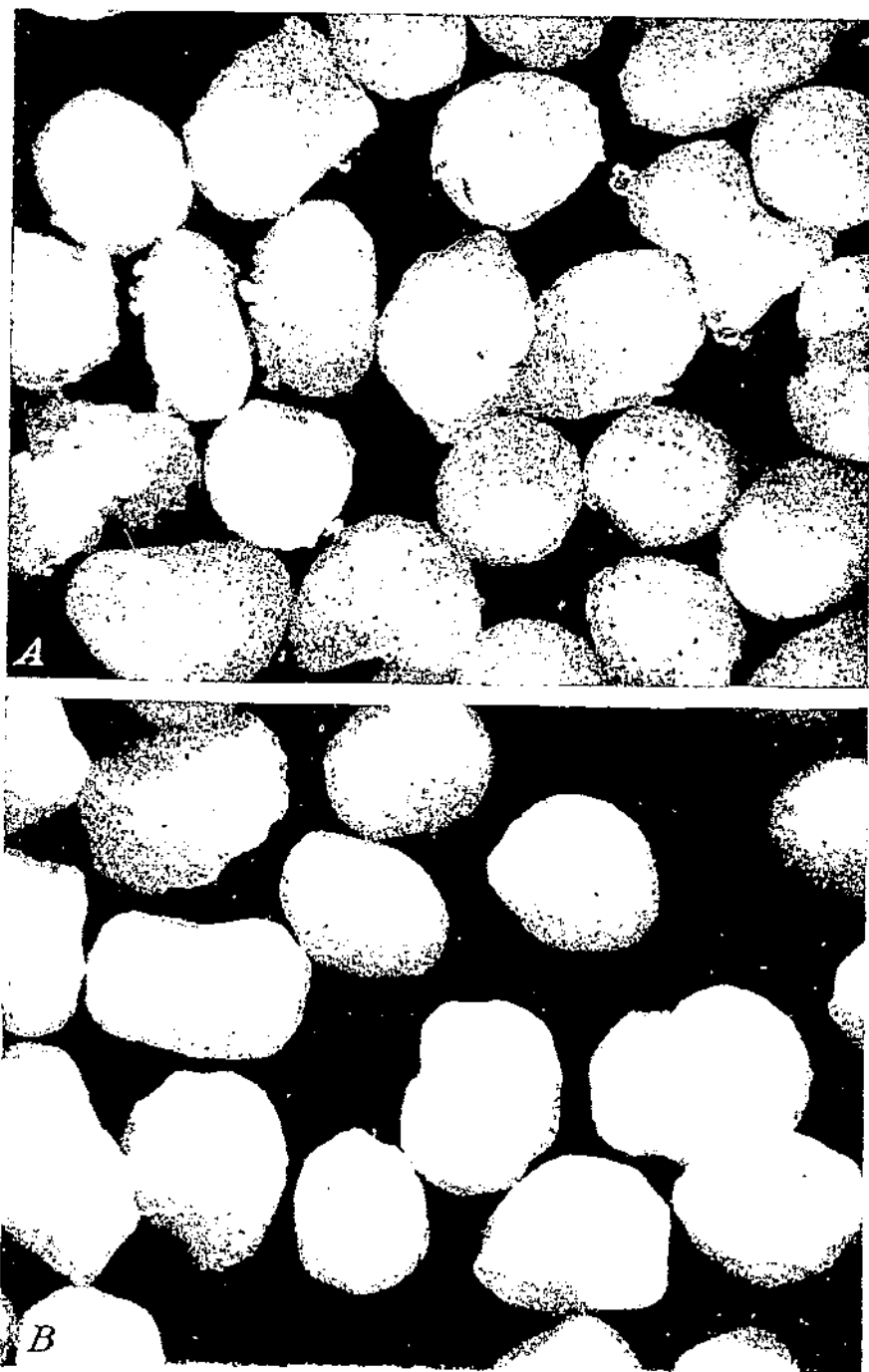


FIGURE 6. Sprayed ammonium nitrate granules: *A*, Old production, and *B*, current production, 14 to 16-mesh,  $\times 10$ .

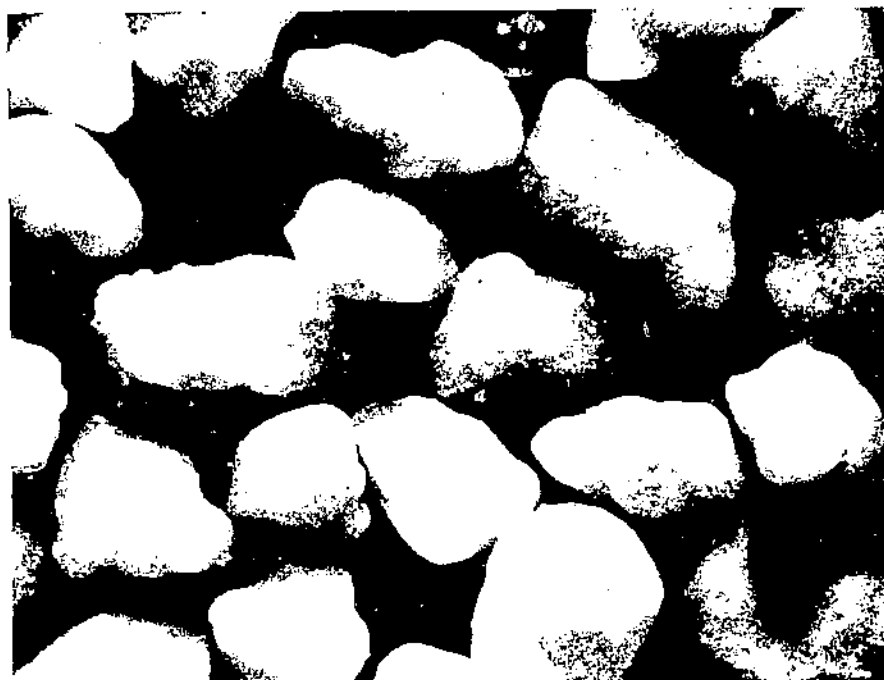


FIGURE 7. Granulated ammonium nitrate, 20 to 40 mesh, U. S. 10.

TABLE 18. *Screen analyses of typical samples of nitrogenous materials from different sources*

Fertilizer grade	Fertilizer type	Screen fraction, mesh				
		10	20	40	60	100
		Percent	Percent	Percent	Percent	Percent
Sulfuric acid	Concentrated, Bragg's	96	97	94	94	94
H <sub>2</sub> O	Water	4	3	6	6	6
C <sub>12</sub> H <sub>22</sub> N <sub>2</sub> O <sub>4</sub>	Stearic acid	0.1	0.8	—	—	—
Ammonium nitrate	—	11	12	2	17	—
H <sub>2</sub> O	—	26	22	22	3	—
H <sub>2</sub> O	Free water	—	4	28	—	11
H <sub>2</sub> O	—	—	—	20	12	—
H <sub>2</sub> O	—	22	17	14	—	—
Ammonium sulfate	—	—	—	—	—	—
Ammonium phosphate	—	—	—	—	—	—
Ammonium phosphate	—	—	—	—	—	—

ANPL, acid production.

ANPL, acid production.

Concentrated batch process.

Continuous process, batch developed continuously.

### PRESSURE DURING STORAGE

Applied pressure or pressure incident to the storage of a fertilizer material increases its tendency to cake by compacting the particles, thereby increasing their cohesion and causing a closer knitting of crystals that may be deposited from the solution phase as a result of

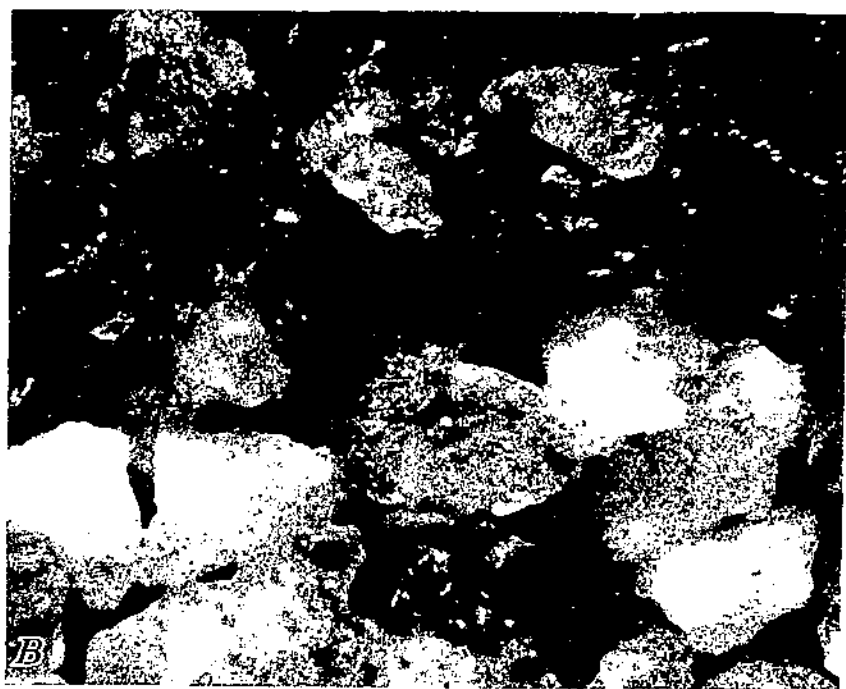
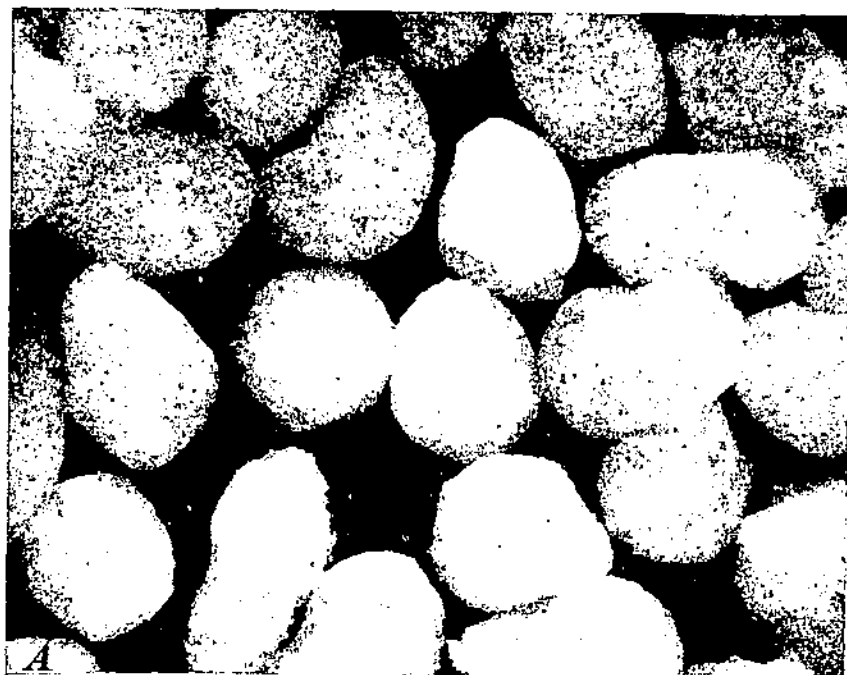


FIGURE 8. Effect of drying, following slight absorption of moisture: A, Sprayed granules, current production; and B, monocrystals, 14- to 16-mesh,  $\times 16$ .

a drop in temperature or humidity. If the moisture content is increased without change in temperature, re-solution of the deposited crystals may take place and the degree to which the material is caked may actually decrease, as previously explained. This will not prove true, however, if the pressure to which it is subjected is sufficient to squeeze out absorbed moisture and crush the moist granules into a solid mass.

Ammonium nitrate differs from most other fertilizer materials in that it exhibits plastic properties and will therefore cake under moderate pressure in a perfectly dry condition. The application of pressure to a plastic material increases its tendency to cake (1) by making its particles conform to the shape of their neighbors, thus increasing the surface area of contact between them, and (2) by causing the crystal particles to enmesh each other, whereby they tend to adhere to each other even though no recrystallization has taken place.

The data given in table 3 indicate, as already explained, that mono-crystalline ammonium nitrate is somewhat more plastic than the granular material. The plasticity of all types tends to decrease with decrease in temperature. Ammonium nitrate under pressure in storage might therefore be expected to show a greater caking tendency during summer storage than in winter.

The results of variations in pressure on the caking of ammonium nitrate show that the crushing strength of the briquets becomes greater with increase in the pressure to which they were subjected during formation (table 19). While applied pressure has thus a marked effect in increasing the caking of ammonium nitrate, it has little effect in increasing the caking of such materials as monoammonium phosphate and superphosphate (2).

TABLE 19. *Effect of pressure on the caking of dry ammonium nitrate when stored in a dry place for 7 days at 30° C.*

Test	Applied pressure	Crushing strength of briquets	
		Sprayed granules, 10-20 mesh	Fine crystals, -10 mesh
	Pounds per square inch	Pounds per square inch	Pounds per square inch
No. 1.	1	0.4	10.0
No. 2.	2	0.5	30.5
No. 3.	4	2.8	45.5
No. 4.	8	5.1	55.7
No. 5.	12	16.5	100.0

#### TEMPERATURE AND DURATION OF STORAGE

An increase in temperature increases the solution phase of most soluble fertilizer salts and affects their caking in a manner similar to an increase in the moisture content. A drop in temperature has the effect of causing a crystallization of salts from the solution phase. The effect of temperature on solubility is much greater for ammonium nitrate than for ammonium sulfate (table 5). It would be expected, therefore, that a temperature change would have a greater effect on the caking of ammonium nitrate than of ammonium sulfate, and this is known to be true.

It would be expected also that a change in temperature would have a greater effect on the caking of ammonium nitrate when moisture is present than when it is dry. Table 20 shows this and also that the tendency of ammonium nitrate to cake increases somewhat with increase in temperature even when perfectly dry. This result should follow, for the reason that an increase in temperature has a marked effect in increasing the plasticity of ammonium nitrate.

The effect of time of storage on the caking of ammonium nitrate when the material is dry and all other factors are kept constant is also shown in table 20. The data show that under the conditions of the test the degree of caking increased with the time of storage. The

TABLE 20.—*Effect of change in time or temperature of storage on caking of granular ammonium nitrate (110- to 20-mesh); moisture, 9.06 percent; applied pressure, 12 pounds per square inch*

Temperature for 7-day storage		Crushing strength of briquets	Time of storage at 30° C. (in days)	Crushing strength of briquets
		Pounds per square inch		Pounds per square inch
0	32°	7	7	22
4	40°	15	14	56
16	50°	45	30	175
21	70°	105	90	230

caking that takes place in dry ammonium nitrate is due in large measure to its plastic properties, as already explained, and a gradual increase in caking with time of storage is therefore to be expected. This may not hold true, however, when the storage period is accompanied by changes in such factors as temperature, pressure, and moisture content. The influence of time of storage on caking will then represent the algebraic sum of the combined effects of the three factors mentioned. Thus, the time of storage has little effect on the caking of ammonium nitrate when the applied pressure is decreasing while the temperature and moisture content are increasing.

#### CONDITIONERS

The method of improving the mechanical condition of a fertilizer by mixing it with a finely divided water-insoluble material is known as conditioning. This treatment is particularly effective in the case of such soluble materials as potassium nitrate and potassium chloride, which are relatively nonhygroscopic and have a marked tendency to cake in a finely divided condition. When a finely divided, water-insoluble material is mixed with a fertilizer of this kind, the fine particles of the insoluble material adhere to and form a coating over the more sticky surfaces of the soluble particles, and its mechanical condition is thereby greatly improved (26).

Among the materials that have been used as conditioning agents are peat and other organic ammoniates, lime, magnesite, limestone, dolomite, kieselguhr, kaolin, talc, phosphate rock, clay, bentonite, plaster of paris, and tricalcium phosphate.

The organic ammoniates have long been used as conditioners in mixed fertilizers, but their use as conditioning agents for ammonium nitrate is not to be recommended, because of the fire hazard. Such basic materials as lime and magnesite react with ammonium nitrate



to evolve ammonia, and they are therefore not suited as conditioning agents for this material. Finely divided limestone also reacts slowly with ammonium nitrate to form calcium nitrate, and as a result its use as a conditioning agent tends to increase rather than decrease the tendency of the ammonium nitrate to cake. Finely divided dolomite and phosphate rock are also unsatisfactory, because of the calcite that they are likely to contain. The materials used in this investigation as conditioning agents for ammonium nitrate have therefore been limited to kaolin and other claylike materials, varying types of kieselguhr from different sources, plaster of paris, and tricalcium phosphate.

An application of a relatively small quantity (3 to 5 percent) of any one of these materials as a coating to ammonium nitrate has a very marked effect in reducing its tendency to cake. This is shown by the data in table 21 and by the results obtained in the large-scale storage tests, described on pages 42 to 58.

TABLE 21.—*Caking tendency of coated and uncoated dry ammonium nitrate of different types when subjected to an applied pressure of 12 pounds per square inch in a dry place for 7 days at 39° C.*

Type of ammonium nitrate	Particle size	Treatment		Crushing strength of briquets
		Water repellent	Conditioner	
	Mesh			Pounds per square inch
Sprayed	10-20	None	None	14
		1.0 percent WP	do	2
		None	3.5 percent Kittlas	1
		1.0 percent WP	do	1
Grained	10-40	None	None	10
		1.0 percent PRP	do	10
		None	4.0 percent Calcite	2
		1.0 percent PRP	do	2
Small crystals	-10	None	None	100
		do	3.5 percent Kittlas	25
		1.0 percent WP	do	20
		None	None	35
Large crystals	10-16	do	3.5 percent Kittlas	15
		1.0 percent WP	do	15

WP = paraffin petrolatum; PRP = petrolatum rosin paraffin.

The effectiveness of different conditioning agents in reducing the tendency of ammonium nitrate to cake varies with such factors as the size, shape, hardness, and frictional characteristics of its particles. The particle sizes of some of the conditioning agents used in these tests are shown in table 22. According to the data in this table tri-

TABLE 22.—*Average particle size of typical conditioning agents for ammonium nitrate*

Conditioning agent	Shape of particle	Average diameter of particle	Surface area
			Square meters per gram
		Microns	
Tricalcium phosphate	Irregular granules	0.020	100
Dewey soil colloid	Flat hexagonal plates	.027	87
Cecil soil colloid	Small hexagonal plates	.017	50
Knofinite	Flat hexagonal plates	.15	15
Bested Veilay	Irregular granules	.15	15
Dicalite	Small diatom forms	1.0 to 2.0	4
Calcite	do	1.0 to 2.0	4
Kittlas	do	1.0 to 2.0	4

calcium phosphate had the smallest particles of any of the materials listed and the diatomaceous earths had the largest. Numerous tests have shown that tricalcium phosphate is more effective as a conditioner for ammonium nitrate than any of the other materials, but the diatomaceous earths are more effective than kaolin. It would seem therefore that while the value of a material as a conditioner for ammonium nitrate increases with decrease in the size of its particles, it does not always follow that the conditioner with the smallest particles makes the most efficient material for this purpose.

Certain types of clay and of kieselguhr have the property of absorbing moisture from the air at ordinary temperatures and humidity. That the quantity so absorbed will vary with the relative humidity is shown by the data in table 23 and the curves in figure 9. Thus, Volclay at a temperature of 30° C. will absorb 10.5 percent of its weight of moisture at a relative humidity of 25.0 percent, 15.5 percent at a relative humidity of 50.0 percent, and 24.0 percent at a relative humidity of 90.0 percent (19). If Volclay that had been in equilibrium with air at 90.0 percent relative humidity were used as a conditioner for ammonium nitrate, which was then stored in a moistureproof bag, the humidity of the air in the bag would remain at 60.0 percent when the temperature is in the neighborhood of 30°. At this humidity the Volclay would evolve about 7.0 percent of moisture, which would be absorbed by the ammonium nitrate. If any drop in temperature subsequently took place, the tendency of the ammonium nitrate to cake would nullify to some extent the effectiveness of the conditioner.

If the Volclay had been in equilibrium with air at 25.0 percent relative humidity before being applied to the ammonium nitrate, it would then have a moisture content of only 10.5 percent. Inasmuch as the humidity of the air in the moistureproof bag would remain at 60.0 percent as before, the added Volclay would absorb moisture, if present, from the ammonium nitrate until it reached equilibrium with a relative humidity of 60.0 percent. The moisture absorbed by the Volclay under these conditions would total about 7.0 percent. The accompanying decrease in the moisture content of the ammonium nitrate would result, as before, in an increase in the tendency of the material to cake.

TABLE 23.—*Moisture contents of various conditioning agents in equilibrium with different relative humidities at 30° C.*

Conditioning agent	Moisture absorbed at 30° C. and a relative humidity of —					
	10 percent	25 percent	40 percent	50 percent	70 percent	90 percent
	Percent	Percent	Percent	Percent	Percent	Percent
Bentonite, Wyoming Volclay	6.00	10.50	11.50	13.50	16.00	21.00
Dicalite, 650V		.07	.12	.19	.38	.69
Dicalite, 625V		3.03	4.05	5.20	8.18	16.55
Dicalite, 675V		.01	.01	.03	.12	.45
Kittans, O.S. 127		1.80	2.89	4.02	7.21	11.43
Celite, C-20114		1.41	2.21	3.17	5.91	9.02
Celite, C-20115		.07	.10	.19	.27	.50
Celite, C-20116		1.53	2.43	3.29	6.03	8.95
Celite, C-20117		.09	.30	.42	.77	1.41
Cecil soil rollard		1.67	2.12	2.64	3.93	8.38
Kaolin		.28	.53	.42	.78	1.10

Inasmuch as an active conditioning agent has the property of absorbing a limited quantity of moisture from the air at a humidity below that at which ammonium nitrate takes up moisture, it might be expected that a lot of ammonium nitrate that had been treated with a conditioning agent of this kind might show a slightly higher moisture content than when it had not been treated or when treated with an inactive conditioning agent. This has been observed to be the case in commercial storage tests. This activity on the part of many materials that are otherwise suited as conditioning agents for

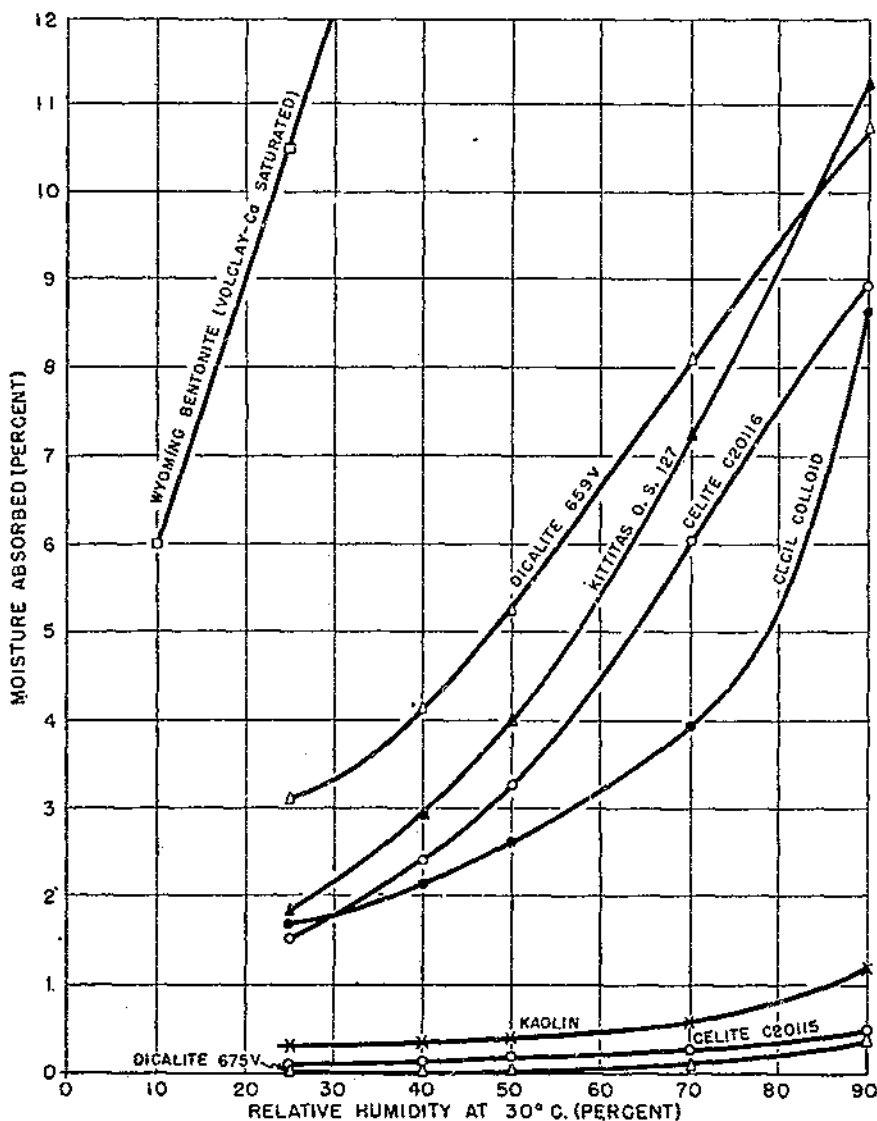


FIGURE 9.—Moisture content of typical conditioning agents in equilibrium with varying relative humidities at 30° C.

ammonium nitrate can be quickly destroyed by heating to a temperature of about 500° C.

A treatment of this kind, however, would add greatly to the cost of the conditioning agent. A more practical procedure for eliminating any exchange of moisture between the conditioning agent and the ammonium nitrate consists in drying the conditioner to a moisture content that is in equilibrium with ammonium nitrate or with a relative humidity of 60 percent at 30° C. The Kittitas curve in figure 9 shows that this material, for example, is in equilibrium with a relative humidity of 60 percent at 30° C. when its moisture content is 5.5 percent. Kittitas that has been dried to this moisture content and is prevented from further change by storage in moistureproof bags, when necessary, will therefore neither increase nor decrease the moisture content of the ammonium nitrate to which it is applied as a conditioning agent.

The curves in figure 9 further show that Dicalite 659V and Celite C-20116 are in equilibrium with ammonium nitrate at moisture contents of 6.6 and 4.4 percent, respectively. Conditioning agents such as Dicalite 675V, Celite C-20115, and kaolin, which do not absorb any appreciable quantity of moisture at ordinary humidities, do not have to be stored in a moistureproof bag or other enclosure to prevent absorption of moisture.

The use of a conditioning agent that will absorb moisture from ammonium nitrate might prove advantageous, provided it will dry out the ammonium nitrate and not give up the absorbed moisture again at a lower humidity. This can be accomplished by use of plaster of paris as a conditioning agent. It was found that when 2.5 pounds of this material was rolled for a few minutes with 50 pounds of granular ammonium nitrate in a cement mixer and the resulting coated product was stored for a few days in a closed container, automatic drying of the ammonium nitrate took place from an initial moisture content of 0.75 percent to 0.04 percent. Plaster of paris by itself cakes quite hard, but the cake formed when 5 parts of the material is diluted with 95 parts of ammonium nitrate should be rated as soft. The gypsum formed by reaction of the plaster of paris with the moisture in the ammonium nitrate retains the moisture with which it combines and serves as a good conditioning agent. In subsequent tests it was found that varying percentages of moisture can be removed from the ammonium nitrate in this way up to the maximum occurring in the freshly sprayed or grained material. The process could probably be of practical application only if provision could be made for storing the treated material in bulk in an air-conditioned warehouse. Such caking as would take place during the bulk storage would be automatically broken up in the process of bagging the material for shipment.

Storage tests indicate that when a conditioning agent is properly applied it is effective in reducing the caking of all types of ammonium nitrate. Such conditioning agents as Kittitas, Celite, Dicalite, or kaolin seem to adhere equally well to granular or monocrystalline ammonium nitrate, although the distribution of the coating agent is likely to be more uniform on spherical particles than on those that are irregular in shape. The quantity of conditioning agent usually applied to sprayed or grained ammonium nitrate is in the neighbor-

hood of 3.0 to 4.0 percent. Applications in excess of 5.0 percent are likely to undergo segregation. This was indicated in laboratory tests in which sprayed and monocrystalline ammonium nitrate samples of 8 to 14 mesh were coated with 6.0 percent of Kittitas and then shaken for 15 minutes on a 60-mesh screen. The quantity of the conditioning agent that remained on each material at the end of the test was approximately 3.5 percent of the weight of the sample. Slight agitation quickly reduced the conditioning agent adhering to the samples from 6.0 to 5.0 percent, but the agitation required to reduce it to 3.5 percent exceeded that to which the materials would normally be subjected under ordinary conditions of handling and storage.

### LARGE-SCALE TESTS ON STORAGE BEHAVIOR

The data presented in the first part of this bulletin were based on the results of laboratory experiments. The results indicate that the tendency of ammonium nitrate to absorb moisture from the air can be decreased by coating the granules with a water-repellent material, as a petrolatum-rosin-paraffin mixture, and that its tendency to cake can be greatly reduced by treating the dry material with 3 to 5 percent of a suitable conditioning agent and keeping it dry throughout the storage period. It was recognized, however, that the behavior of ammonium nitrate in storage is subject to so many factors that conclusions respecting the relative value of different treatments should not be adopted as final until the laboratory experiments were supplemented by large-scale storage tests. These were carried out under commercial conditions. The ammonium nitrate materials used in the tests were supplied by Alberta Nitrogen Products, Ltd., Calgary, Canada, and the Tennessee Valley Authority, Wilson Dam, Ala. The material from Calgary was granulated by spraying, while that from the Tennessee Valley Authority had been prepared by the graining process. Both materials were subjected to various coating and conditioning treatments for use in the storage tests.

### SPRAYED AND GRAINED NITROGENOUS MATERIALS

The first storage tests were made to compare the caking tendency of the sprayed and grained ammonium nitrate then on the market with that of Cal-Nitro and sodium nitrate. The tests were made in the fertilizer warehouse of the Division of Soils, Fertilizers, and Irrigation, at Beltsville, Md. This storage test began on August 20, 1943. Each bag contained 100 pounds of material, and the bags of each lot were piled nine high. The piles were arranged in rows 4 feet apart and 1 foot apart in the row. The bags were retained in an upright position by 2- by 4-inch supports placed on both sides of each pile. The materials used in the test were as follows:

1. Chilean nitrate of soda, Champion Brand.
2. Arcadian nitrate of soda.
3. Cal-Nitro.
4. Calgary granular ammonium nitrate diluted with 2 percent of coarse limestone.
5. Same material as 4, conditioned in the Division with 2 percent of Dicalite.
6. Same material as 4, conditioned in the Division with 4 percent of Dicalite.

7. TVA ammonium nitrate coated by the producers with 1 percent of a petrolatum-rosin-paraffin mixture plus 4 percent of kieselguhr.
8. Same material as 7, coated by the producers with 1 percent of petrolatum plus 4 percent of kaolin.

None of these materials were in a caked condition when placed in storage. Those that were caked when received were crushed, screened, and rebagged, if necessary, before being used in the caking tests. All materials were stored in the bags in which they had been originally shipped.

A final inspection was made of the caked condition of these materials on November 20, 1943, 3 months after they had been placed in storage. The drill test, the drop test, and the crushing-strength test were used in comparing the caked condition.

*Electric hammer-drill test.*—In this method, the hardness of a material is determined by the time taken to drill a 1-inch hole into a bag of the material to a depth of 5 inches. The drill was operated at 106 volts.

*Drop test.*—In this test a bag of the material is dropped four times, once on each side and on each edge, from a height of 3 feet. The proportion of the material that will not pass a 4-mesh screen after four drops of the bag is taken as a measure of the caked condition of the whole.

*Crushing-strength test.*—After each bag is dropped, tests are made by hand to determine the hardness of the lumps that remain in the bag. These are classified as follows: Soft (S), breaks under finger pressure; medium hard (M), breaks under hand pressure; hard (H), does not break under hand pressure but readily breaks under foot; and very hard (VH), difficult to break under foot. Following the drop test a bag of material will normally contain lumps of varying size and hardness. Different individuals therefore are likely to assign a different hardness rating to the same material when making the crushing-strength test. To insure a reasonable degree of accuracy in making this test it is important to select lumps of uniform size.

The results obtained in these caking tests are given in table 24, which shows that all three methods agree fairly well in comparing the caked condition of different bags of the same material, as Chilean nitrate of soda or either one of the TVA products. They do not agree so well, however, when used to compare the mechanical condition of such different materials as the coated and uncoated Calgary ammonium nitrate. It has been noted that ammonium nitrate that has undergone serious caking will not again cake as hard when crushed, screened, and exposed for a second time to the same conditions of storage. The uncoated Calgary product had undergone a previous and much more severe caking at the time of its shipment, which may explain the brittle condition of the material at the time of the storage test.

The degree of caking of the granular Chilean nitrate of soda was shown to be considerably less than that of the Arcadian nitrate of soda and less than for any of the ammonium nitrate samples. The data also indicate that an application of 2 percent or less of a conditioning agent to ammonium nitrate may actually increase rather than decrease its caking tendency.

TABLE 24.—Caking tests with different nitrogenous materials after 3-months' storage (Aug. 20 to Nov. 20, 1943) at Beltsville, Md.

Position of bar in pile, top to bottom	Chilean nitrate of soda			Arcadian nitrate of soda			Cal-Nitro			ANPL $\text{NH}_4\text{NO}_3$ , uncoated		
	Time of drilling	Lumps		Time of drilling	Lumps		Time of drilling	Lumps		Time of drilling	Lumps	
		Weight	Hardness <sup>1</sup>		Weight	Hardness <sup>1</sup>		Weight	Hardness <sup>1</sup>		Weight	Hardness <sup>1</sup>
Top.....	Seconds (2)	Pounds (2)		Seconds	Pounds		Seconds	Pounds		Seconds	Pounds	
Second.....	(2)	(2)		8	18	M	(2)	0		15	0	
Third.....	(2)	(2)		12	12	M	15	4	S	21	1	S
Fourth.....	(2)	(2)		16	11	M	25	1	M	31	1	S
Fifth.....	(2)	(2)		6	13	M	22	13	M	36	1	M
Sixth.....	(2)	(2)		11	11	M	29	5	M	31	1	H
Seventh.....	(2)	(2)		16	7	M	40	14	M	33	1	
Eighth.....	(2)	(2)		14	11	M	31	15	M			
Bottom.....	9 17	0 0		15	10 7	M	46	16	H			

Position of bag in pile, top to bottom	ANPL $\text{NH}_4\text{NO}_3$ , plus 2.0 percent Dialite			ANPL $\text{NH}_4\text{NO}_3$ , plus 4.0 percent Dialite			TVA $\text{NH}_4\text{NO}_3$ , plus 1.0 percent PRP plus 4.0 percent Celite			TVA $\text{NH}_4\text{NO}_3$ , plus 1.0 percent petrolatum plus 4.0 percent kaolin		
	Time of drilling	Lumps		Time of drilling	Lumps		Time of drilling	Lumps		Time of drilling	Lumps	
		Weight	Hardness <sup>1</sup>		Weight	Hardness <sup>1</sup>		Weight	Hardness <sup>1</sup>		Weight	Hardness <sup>1</sup>
Top.....	Seconds	Pounds		Seconds	Pounds		Seconds	Pounds		Seconds	Pounds	
Second.....							(2)	(2)		(2)	(2)	
Third.....				3	0		1	0		2	0	
Fourth.....	24	8	M	4	1	S	2	0		3	0	
Fifth.....	47	20	M	7	1	S	6	0		7	0	
Sixth.....	46	33	H	12	1	S	9	1	S	11	3	S
Seventh.....	60	34	H	23	14	M	11	1	S	13	2	S
Eighth.....	61	32	H	32	15	M	35	2½	M	11	10	S
Bottom.....							58	16	H	19	19	M

<sup>1</sup> S = soft; M = medium; H = hard; VH = very hard.<sup>2</sup> Drill plunged through material before instrument was turned on.<sup>3</sup> No caking; bags not dropped.

Samples were withdrawn from two bags in each pile for moisture determinations. The results, given in table 25, show that Cal-Nitro and the TVA samples contained more moisture than any of the other materials. Had their initial moisture content been as low as in the other materials, the extent to which they caked in the storage test would no doubt have been considerably less.

TABLE 25.—Moisture content of nitrogenous materials used in storage test

Fertilizer material	Position of sampled bag from top of pile	Moisture content
		Percent
Chilean nitrate of soda.....	Bottom.....	0.09
Aracadian nitrate of soda.....	Seventh.....	.01
Do.....	Eighth.....	.01
Cal-Nitro.....	Third.....	.49
Do.....	Sixth.....	.54
ANPL ammonium nitrate:		
Uncoated.....	Fourth.....	.07
Plus 2.0 percent Dicalite.....	Third.....	.08
Do.....	Fifth.....	.08
Plus 4.0 percent Dicalite.....	Third.....	.11
Do.....	Fifth.....	.05
TVA ammonium nitrate:		
Plus 1.0 percent PKP plus 4.0 percent Celite.....	Third.....	.31
Do.....	Fourth.....	.35
Plus 1.0 percent petroleum plus 4.0 percent kaolin.....	do.....	.23
Do.....	Sixth.....	.22

#### EFFECT OF CONDITIONERS

The second storage tests were undertaken to compare the caking tendency of variously treated lots of sprayed ammonium nitrate as supplied by Alberta Nitrogen Products, Ltd. In this test, set up at Norfolk, Va., the bags of each lot were piled 10 high. The piles were arranged in rows as in the previous storage test and the bags were retained in an upright position by means of narrow boards that extended from pile to pile between the fifth and sixth bags. This method of supporting the piles in an upright position proved satisfactory for the valve-closure bags. It does not suffice, however, to support bags that are closed with a wire tie, for the reason that bags of this type are more round in shape than those having sewn or valve closures.

The 16 lots of ammonium nitrate used in this storage test were shipped from Calgary, Alberta, Canada, on August 30, 1943, and were placed in storage at Norfolk on September 14. The lots coated with 0.5 to 1.0 percent paraffin followed by a second coating of 3.0 to 5.0 percent of Kittitas were superior in mechanical condition to any of the other lots at the time of unloading the car in which they were shipped. The greatest degree of caking was observed in the lot that had been treated with 2.0 percent of limestone.

In examining this shipment at the time it was unloaded, it was observed that some of the bags, such as lot 16, had a hard crust just inside the bag. This crust, which adhered somewhat to the inner lining of the bag, varied from about  $\frac{1}{2}$  to 2 inches thick. Lot 16, as shown in table 26, was treated with 1.0 percent of Kittitas and was loaded into the bags at a temperature of 51° C. A saturated solution of ammonium nitrate at this temperature has a vapor pressure of 45



TABLE 26.—*Mechanical condition of different lots of ANPL ammonium nitrate after 4-months' storage (Sept. 14, 1943, to Jan. 16, 1944) at Norfolk, Va.*

Lot <sup>1</sup> No.	Treatment		Bagging temper- ature	Moisture content at—		Estimated number of bags suited for direct application
	Water repellent	Conditioner		Time of bagging	Final in- spection	
			<sup>°</sup> C.	Percent	Percent	
11.....	1.0 percent paraffin.....	5.0 percent Kittitas.....	42	0.20	0.15	10
2.....	do.....	3.0 percent Kittitas.....	40	.10	.14	10
3.....	0.5 percent paraffin.....	do.....	38	.17	.12	10
8.....	None.....	4.0 percent Kittitas.....	40	.12	.10	10
7.....	do.....	4.0 percent bentonite.....	47	.21	.07	10
4.....	do.....	2.0 percent Kittitas.....	40	.08	.02	9
5.....	do.....	do.....	60	.08	.10	8
6.....	1.0 percent paraffin.....	1.0 percent Kittitas.....	37	.16	.01	6
10.....	0.5 percent paraffin.....	do.....	37	.19	.08	6
12.....	None.....	2.0 percent Kittitas.....	35	.23	.01	5
9.....	0.5 percent paraffin.....	do.....	37	.20	.07	5
14.....	None.....	2.0 percent bentonite.....	48	.18	.15	4
17.....	do.....	2.0 percent Kittitas.....	51	.17	.20	3
15.....	do.....	do.....	60	.21	.08	2
16.....	do.....	1.0 percent Kittitas.....	51	.18	.06	0
13.....	do.....	2.0 percent limestone.....	50	.22	.01	0

<sup>1</sup> 10 bags in each lot.

mm. of mercury. Water has a vapor pressure of 45 mm. of mercury at a temperature of 36°. It follows, therefore, that if the temperature of the outside air was below 36°, which it undoubtedly was at the time the bags were filled with ammonium nitrate at 51°, moisture would be distilled from the ammonium nitrate and be condensed on the interior lining of the asphalt-laminated bags in which it was shipped. This condensed moisture would cause a partial solution of the granules in contact with it. When the bags were later shipped to a warmer climate, the condition with respect to temperature gradient in the bag would in a measure be reversed. Redistillation of moisture from the inner lining of the bag to the ammonium nitrate within the bag would then take place, with resulting recrystallization of the ammonium nitrate that had gone into solution. That a crust will form within the inner lining of a bag as a result of a fluctuation in temperature as described was confirmed by laboratory tests.

Lot 10 of the same shipment was loaded into the bags at a temperature of 37° C. The vapor pressure of a saturated solution of ammonium nitrate at this temperature is equivalent to about 25 mm. of mercury, the same as that of water at about 25° C. While the outside temperature at the time the bags were loaded is not known, it is certain that the initial temperature gradient within this lot of bags was less than in the case of lot 16. Lot 10 also differed from lot 16 in that it was coated with 0.5 percent paraffin as well as 1.0 percent of Kittitas. It would be expected, therefore, that this lot would show little or no more caking in the area adjacent to the inner lining of the bag than in the central part, and no caked area was found.

The formation of a thin crust within the inner lining of the bag offers the disadvantage that it is difficult to break up without leaving a residue of small hard lumps throughout the mass of ammonium nitrate. These lumps tend to clog up the delivery tubes of the fertilizer distributor and thereby interfere with the drillability of the material. It was apparent that the formation of this crust could be

readily prevented by lowering the bagging temperature. When this was done, no crust was observed in subsequent shipments.

The final inspection of these different lots was made on January 16, 1944, at the end of a 4-month storage period, when the degree of caking was determined by the drilling, drop, and crushing-strength tests. In making these tests it was assumed that any bag of material that had a drilling time of less than 30 seconds or that contained only soft lumps after the drop test was suited for direct application in the field. By thus comparing the results obtained in the three types of tests it was possible to determine the number of bags in each pile that was suited for direct use as a fertilizer and to arrange the different lots of material in the descending order of their mechanical condition (table 26).

The results obtained in these final storage tests agree on the whole with laboratory observations made in the interval between the setting up of the storage tests and their final inspection. The least degree of caking occurred in the lots that had been treated with 0.5 to 1.0 percent of paraffin plus 3.0 to 5.0 percent of Kittitas. All 10 bags in each pile of these lots were considered to be suitable for direct application in the field. The lots that had been treated with 4.0 percent of Kittitas or of bentonite were also found to be in a satisfactory condition throughout. The observations made indicate, however, that the use of 2.0 percent or less of such a conditioning agent as Kittitas is not sufficient to prevent caking of ammonium nitrate in storage. This holds true whether or not the material is coated with wax before the addition of the conditioning agent.

The moisture contents of the different lots at the time of bagging were determined by the producer, while the final moisture contents were determined by this Division. The differences between the two sets of determinations are small and probably within the limits of experimental error. No appreciable quantity of moisture thus appears to have been either absorbed or lost during the storage period. The differences observed in the caking of the different lots would, therefore, seem to be a fairly good indication of the relative effectiveness of the different treatments.

The manner in which caking occurred in these storage tests, as well as in the Beltsville storage tests previously described, may be considered as being of four types, as follows: (1) A condition of uniform hardness throughout the bag, (2) a caked core surrounded by a protecting layer of uncaked material, (3) a hard crust just inside the bag surrounding a core of uncaked material, and (4) more or less detached lumps of varying hardness interspersed through material of a relatively less degree of caking.

A variation in the hardness of the type of caking outlined under the first type can be readily detected by all three methods of caking. Owing to the protective action of the uncaked part outlined under the second type as well as in that outlined under the fourth, the caked part is not so likely to break readily in the drop test as when the caking occurs as a crust on the outer section of the contents of the bag. A material caked as outlined under the second or fourth type is therefore likely to show a higher proportion of lumps in the drop test than one caked under the third type, even though the hardness and degree of caking is the same in both cases. The hammer-drill test

tends in a sense to offset the drop test, in that it gives low results where the latter gives high results, and vice versa. Thus the drop test tends to give a low proportion of lumps for hard, brittle materials and a high proportion of lumps for materials that cake as outlined under types 2 or 4. The hammer-drill test, on the other hand, tends to give high results for hard, brittle materials and low results for those that cake as outlined under 2 or 4.

In the drop test, allowing the bag to fall four times from a height of 3 feet is considered equivalent to the handling to which it would normally be subjected in the process of being shipped from the plant or warehouse to the farmer's field. The extent to which the material remains in a caked condition after the drop test is, therefore, a better indication of its mechanical condition as judged by the farmer than any hardness test, irrespective of how accurate the hardness test may be. The hammer-drill test was accordingly omitted in all subsequent storage tests.

#### EFFECT OF LOCATION

In the large-scale storage tests previously described no appreciable change in the moisture content of the ammonium nitrate was observed throughout the storage period. It was recognized, however, that the behavior of the material might have been very different had the storage tests been made in sections of the country where the humidity is such as to cause absorption of moisture by the ammonium nitrate. An extended series of tests was accordingly undertaken in which ammonium nitrate was placed in storage under commercial conditions at six different locations in some of the more humid sections of the country (table 27). Storage tests were also set up at Beltsville, Md., in cold-storage rooms under controlled conditions of temperature and humidity.

The materials used in these storage tests were a sprayed ammonium nitrate (ANPL) produced in Canada, a grained domestic product (TVA), and a mono-crystalline product (MCW) prepared by the Oslo-Krystal process. A list of the treatments to which each of the materials was subjected in their preparation, and their mechanical analysis are given in table 28.

The test materials were shipped from the producing plants directly to the storage sites. Insofar as possible any set that had occurred during transit was broken up before the different lots were put in storage. The bags of each type of material were then stacked close together in a row 7 piles long and 12 bags high on boards laid on the floor. There was an air space between each row, but the bags in each row were in contact and thus supported each other. All materials were placed in storage in the bags in which they were shipped.

Three inspections were made of the materials stored at New Orleans, Raleigh, Stoneville, and Wilson Dam, and 2 at each of the other locations with the exception of Pittsburg, Kans., where only 1 inspection was made. In making these inspections the bags of 1 pile of each lot of material were removed and subjected to the drop test. The results obtained in this test are given in table 29 for the materials stored at the different locations under commercial conditions and in table 30 for the materials stored in the cold-storage rooms. These tables

TABLE 27.—Location, storage condition, and storage and inspection dates of large-scale storage tests

Location	Cooperating organization	Condition of storage	Date of storage for—		Date of inspection	
			ANPL ammonium nitrate	TVA ammonium nitrate	First	Second <sup>1</sup>
Baltimore, Md.....	Fertilizer Manufacturing Cooperative, Inc.	Open concrete areaway on the shore of the bay.....	Dec. 27, 1943	Dec. 10, 1943	<sup>1944</sup> Mar. 20	<sup>1944</sup> May 25
New Orleans, La.....	Armour Fertilizer Works.....	Large frame building with concrete floor.....	Dec. 23, 1943	Dec. 29, 1943	Mar. 1	May 12
Raleigh, N. C.....	North Carolina Agricultural Experimental Station.	Second floor of brick dairy barn.....	Jan. 5, 1944	Jan. 5, 1944	Mar. 13	May 16
Stoneville, Miss.....	Mississippi Agricultural Experimental Station.	Small corrugated iron building with concrete floor.	Jan. 2, 1944	Jan. 2, 1944	Feb. 25	May 9
Wilson Dam, Ala.....	Tennessee Valley Authority.....	Large hollow-tile building with concrete floor.....	Jan. 9, 1944	Jan. 9, 1944	Feb. 22	May 4
Beltsville, Md.....	Bureau of Plant Industry, Soils, and Agricultural Engineering.	Second floor of cinder-block building.....	Jan. 7, 1944	Dec. 31, 1943	June 20	-----
Room A.....	do.....	85° F.; uncontrolled humidity.....	do.....	do.....	Do.	-----
Room 2.....	do.....	55° F.; relative humidity, 80 percent.....	do.....	do.....	Do.	-----
Room 13.....	do.....	35° F.; relative humidity, 75 percent.....	do.....	do.....	Do.	-----
Room 18.....	do.....	55° F.; relative humidity, 60 percent.....	do.....	do.....	Do.	-----
Room 23.....	do.....	75° F.; relative humidity, 65 percent.....	do.....	do.....	Do.	-----
Pittsburg, Kans.....	Military Chemical Works, Inc.....	Frame barn without floor.....	Mar. 17, 1944	Mar. 17, 1944	May 30	-----

<sup>1</sup> Final inspections were made between Nov. 30 and Dec. 9, 1944, at New Orleans, Raleigh, Stoneville, and Wilson Dam (see table 36).

TABLE 28.—*Treatments and screen analysis of lots of ammonium nitrate used in storage tests in different locations*

Lot	Treatment		Screen analysis (mesh)				
	Water repellent <sup>1</sup>	Conditioner	+ 10	10-14	14-20	20-40	-40
			Percent	Percent	Percent	Percent	Percent
ANPL:							
No. 1	None	None	14	38	26	17	5
No. 2	0.5 percent WP	do	14	31	29	21	2
No. 3	None	3.5 percent Kittitas	13	39	28	17	3
No. 4	0.5 percent WP	do	15	38	27	15	5
TVA:							
No. 1	None	None	1	4	26	58	11
No. 5B	1.0 percent PRP	4.0 percent Celite	1	4	23	55	17
No. 6C	do	4.0 percent kaolin	1	8	28	53	10
No. 7C	do	4.0 percent plaster of paris	0	10	31	51	8
No. 8C	do	4.0 percent soapstone	1	4	23	55	17
No. 9C	do	None	1	4	23	55	17
MCW:							
No. 1	None	do	22	57	14	6	1
No. 2	0.2 percent PRP	2.0 percent Dialite	11	70	16	3	0
No. 3	2.0 percent Premix	None	14	59	17	8	1
No. 4	0.2 percent PRP	3.0 percent Dialite	11	70	16	3	0
No. 5	do	4.0 percent Dialite	11	70	16	3	0

<sup>1</sup> WP = paraffin-petrolatum; PRP = petrolatum-rosin-paraffin; and Premix = 1 part of PRP plus 3 parts of Dialite mixed before applying.

TABLE 23.—Caking test with variously treated lots of ammonium nitrate in storage tests at different locations,<sup>1</sup> showing weight<sup>2</sup> and hardness<sup>3</sup> of lumps formed

## FIRST INSPECTION

Lot	Baltimore, Md.		New Orleans, La.		Raleigh, N. C.		Stoneville, Miss.		Wilson Dam, Ala.		Beltsville, Md.	
	Weight	Hardness	Weight	Hardness	Weight	Hardness	Weight	Hardness	Weight	Hardness	Weight	Hardness
	Pounds		Pounds		Pounds		Pounds		Pounds		Pounds	
ANPL No. 4.....	0	S	0	S	0	S	0	S	1	S		
ANPL No. 3.....	1	S	1	S to M	0	S	1	S	1	S		
TVA No. 5B.....	0	S	5	S to M	1	S to M	4	S	2	S		
TVA No. 7C.....	3	S			4	S to M	8	S	5	S		
TVA No. 6C.....	4	S to M	28	S to H	4	S	9	S to H	12	S to M		
TVA No. 8C.....			24	S to H					10	S to M		
ANPL No. 2.....	37	M to H	21	S to M	20	S to M	29	S to M	17	S to M		
ANPL No. 1.....	18	S to H	26	S	21	S to M	26	S	27	S		
TVA No. 9C.....			47	S to VH					20	S to H		
TVA No. 1.....	30	S to H			38	M to H	42	M to H	43	H to VH		

## SECOND INSPECTION

ANPL No. 4.....	0	S	0	S	0	S	0	S	0	S	3	S
ANPL No. 3.....	0	S	1	S to M	0	S	1	S	1	S	2	S
TVA No. 5B.....	1	S	5	S to M	1	S to M	4	S to H	2	S	4	S to H
TVA No. 7C.....	10	S			4	S to M	8	S	5	S	25	S
TVA No. 6C.....	6	S	28	S to H	4	S	9	S to H	11	S to M	13	S
TVA No. 8C.....			24	S to H					10	S to M		
ANPL No. 2.....	36	S to H	21	S to M	20	S to M	29	S to M	17	S to M	55	S to M
ANPL No. 1.....	28	M to H	26	S	21	S to M	26	S	27	S	62	S to H
TVA No. 9C.....			46	S to VH					20	S to H		
TVA No. 1.....	26	S to H			38	M to H	42	M to H	42	H to VH	86	M to H

<sup>1</sup> See table 27 for storage and inspection dates.<sup>2</sup> After 1 drop of bag at Beltsville; 4 drops at all other locations.<sup>3</sup> S = soft; M = medium; H = hard; VH = very hard.

TABLE 30.—Caking tests with variously treated lots of ammonium nitrate after 5½-months' storage (Dec. 31, 1943, to June 20, 1944) under controlled conditions at Beltsville, Md., showing weight<sup>1</sup> and hardness<sup>2</sup> of lumps formed

Lot	Room 13 (35° F.; 75 percent relative humidity)		Room 18 (55° F.; 60 percent relative humidity)		Room A (85° F.; uncontrolled relative humidity)		Room 23 (75° F.; 65 percent relative humidity)		Room 2 (55° F.; 80 percent relative humidity)	
	Weight	Hardness	Weight	Hardness	Weight	Hardness	Weight	Hardness	Weight	Hardness
	Pounds		Pounds		Pounds		Pounds		Pounds	
ANPL No. 4	0	S	0	S	0	S	0	S	0	S
ANPL No. 3	1	S	1	S	0	S	0	S	1	S
TVA No. 5B	0		0	S	0	S	7	S to VII		
TVA No. 7C	2	S	9	S	6	S	21	S to II		
TVA No. 6C	3	S	3	S	3	S	14	S to M		
ANPL No. 2	58	S to II	64	S to II	57	S to M	47	S to II	36	S to M
ANPL No. 1	42	S to II	41	S to II	48	II	50	S to VII	40	II
TVA No. 1	44	S to II	60	S to II	41	S to M	71	M to VII		

<sup>1</sup> After 1 drop of bag.

<sup>2</sup> S = soft; M = medium; II = hard; VII = very hard.

give the average weight of lumps per bag for each pile of material. This value does not show the variation in the degree of caking in the different bags of a pile of any type of material, but it does afford a convenient means for comparing the relative degrees of caking of the different lots. The bags were piled 10 high in the cold-storage rooms and 12 high at all other locations. In making the caking tests the bags at all locations except Beltsville, Md., were dropped 4 times. This standard test brings out the difference between the badly caked materials and those that do not set very much, but it is too drastic for differentiating between materials that show very little caking. To bring out the finer distinctions, the materials stored in the fertilizer house and in the cold-storage rooms at Beltsville, Md., were dropped only once before screening.

The data in tables 29 and 30 show also the range of hardness of the lumps in each pile of the various materials. The sprayed ammonium nitrate (ANPL) contained a small quantity of hard lumps of a coating or conditioning agent or what appears to be scrapings from the equipment used in the preparation of the material. Lumps of this kind would interfere with the drillability of what might otherwise be a product of excellent mechanical condition. These lumps are now being removed by screening at the point of production and they were not found in subsequent shipments.

The unconditioned ammonium nitrate and the ammonium nitrate that had been treated only with a water repellent caked to an objectionable degree. While the lumps in these materials were soft in some cases, this was due to the fact that moisture had been absorbed during storage and there had been no opportunity for the material to dry out again. The ANPL lots that had been conditioned with Kittitas stood up better under storage than any of the others. In only one case did the average weight of lumps per bag exceed 1.0 percent, and in all cases the lumps were soft except for the small quantity of hard lumps that were introduced, as already explained, in the process of manufacture. The TVA products suffered from the presence of a large proportion of fines, as shown in table 28. These tended to increase the caking of the material and the hardness of the resulting lumps.

The relative degree of caking of the different lots of ammonium nitrate is further shown by the data in table 31. This table gives the estimated number of bags in each pile suited for direct application in the field. All the bags in each pile of ANPL Nos. 3 and 4 were considered to be suited for direct application. This held true for all the locations at which the storage tests were made. TVA No. 5B remained in as good a mechanical condition as either ANPL No. 3 or ANPL No. 4 at some, but not all, locations. This TVA material was decidedly superior to TVA Nos. 6C and 7C. The storage tests at all locations indicate very clearly that unconditioned ammonium nitrate is not suited to the fertilizer trade.

On the basis of the observations made at the various locations at which granular ammonium nitrate had been stored, it would seem that the different lots may be rated in decreasing order, as shown in table 32.



TABLE 31.—*Mechanical condition of variously treated lots of ammonium nitrate in large-scale storage tests at different locations*

[Piled 10 bags high in storage rooms at Beltsville, Md.; 12 bags high at other locations]

Lot	Estimated number of bags in pile suitable for direct application at time of final inspection at—										
	Balti- more, Md.	New Orleans, La.	Ra- leigh, N. C.	Stone- ville, Miss.	Wilson Dam Ala.	Belts- ville, Md.	Beltsville, Md.				
							Room 13	Room 18	Room A	Room 23	Room 2
ANPL No. 4	12	12	12	12	12	12	10	10	10	10	10
ANPL No. 3	12	12	12	12	12	12	10	10	10	10	10
TVA No. 2B	12	7	12	12	12	12	10	10	10	8	
TVA No. 7C	10		11	12	11	5	10	10	10	4	
TVA No. 6C	12		11	4		12	10	10	10	5	
TVA No. 8C		3				7					
ANPL No. 2	2	2	2	0	4	6	0	0	1	1	3
ANPL No. 1	0	1	1	1	1	0	0	2	0	0	0
TVA No. 9C		0			4						
TVA No. 1	0		0	0	0	0	2	1	1	0	

TABLE 32.—*Rating in decreasing order of various treatments on lots of granular ammonium nitrate*

Lot	Water repellent	Conditioning agent
ANPL No. 4	0.5 percent petrolatum-paraffin mixture	3.5 percent Kithins.
ANPL No. 3	None	Do.
TVA No. 5B	1.0 percent petrolatum-rosin-paraffin mixture	4.0 percent Celluc.
TVA No. 7C	do.	4.0 percent plaster of paris.
TVA No. 6C	do.	4.0 percent kaolin.
TVA No. 8C	do.	4.0 percent soapstone.
ANPL No. 2	0.5 percent petrolatum-paraffin mixture	None.
ANPL No. 1	None	Do.
TVA No. 9C	1.0 percent petrolatum-rosin-paraffin mixture	Do.
TVA No. 1	None	Do.

## MONOCRYSTALLINE AMMONIUM NITRATE

The storage tests with several lots of MCW monocrystalline ammonium nitrate were initiated at a later date than those with the granular materials. These lots were supplied as experimental material with the request that they be incorporated in the storage tests. The results obtained are given in table 33. In making these tests the bags of material stored at Beltsville, Md., were dropped only once, while those stored at New Orleans, La., and Pittsburg, Kans., were subjected to the standard four-drop test. The unconditioned MCW material at Pittsburg and at New Orleans contained no lumps after the drop test, while similar material at Beltsville showed decided caking. The lots of material at Pittsburg and New Orleans contained in excess of 0.30 percent of moisture, however, and it was felt that this was sufficient to lubricate the surface of the crystals and thus permit them to slip apart. The material stored at Beltsville, on the other hand, contained only 0.07 percent of moisture, or essentially the same as the initial moisture content. No re-solution of interlocked crystals could, therefore, have taken place, and a caked condition would have resulted because of the plastic nature of the material. The results obtained with the monocrystalline material further indicate that the conditioning agents used were not so effective in reducing the caking of large crystals as of granular materials.

TABLE 33.—Caking tests with variously treated lots of MOW monocrystalline ammonium nitrate after storage at different locations, showing weight<sup>1</sup> and hardness<sup>2</sup> of lumps formed

BELTSVILLE, MD., MAR. 14 TO JUNE 26, 1944

Arrangement of bags in pile from top to bottom	MCW No. 1		MCW No. 2		MCW No. 3		MCW No. 4		MCW No. 5	
	Weight	Hardness	Weight	Hardness	Weight	Hardness	Weight	Hardness	Weight	Hardness
Third.....	Pounds 10	S			Pounds 30	S				
Fourth.....	17	S			36	M				
Fifth.....	6	S							29	M
Sixth.....	3	S	19	S	51	S				
Seventh.....	1	S	27	S						
Eighth.....	10	S	40	S	48	S				
Ninth.....	5	S	39	S					35	M
Tenth.....	9	S	13	S						

NEW ORLEANS, LA., MAR. 1 TO MAY 12, 1944

Fifth.....	0		2	S			15	M		
Sixth.....	0		0	S			10	S		
Tenth.....	0		2	S			18	M		
Eleventh.....	0		15	S			16	M		

PITTSBURG, KANS., MAR. 17 TO MAY 30, 1944

Second.....	0		0		13	S				
Seventh.....	0		3	S	10	S to M				
Eleventh.....	0		9	S to M	2	H				

<sup>1</sup> After 1 drop of bag at Beltsville; after 4 drops at New Orleans and Pittsburg.<sup>2</sup> S = soft; M = medium; H = hard; VH = very hard.

## MOISTURE ABSORPTION IN STORAGE

Moisture determinations were made on each lot of material at the beginning of the tests and again at each inspection. A representative sample for analysis was obtained in an open bottle by passing it back and forth under the screen as the material was being screened during the drop test. The sample was always taken from the bag next to the bottom of the pile. The results obtained are given in table 34 for the lots stored at the different locations and in table 35 for those stored in the cold storage rooms at Beltsville, Md. The tables show (1) that for any given lot of material the moisture absorbed increased with increase in the humidity of the location in which it was stored; and (2) that for any given location the moisture absorbed by the TVA materials exceeded that absorbed by the ANPL materials. The relatively high absorption of moisture by TVA No. 7C was no doubt due to hydration during storage of the plaster of paris used as the conditioning agent.

## FINAL INSPECTION

A final inspection of the ammonium nitrate samples stored at Wilson Dam, Ala., Stoneville, Miss., New Orleans, La., and Raleigh, N. C., was made during the period between November 30 to December 9, 1944. The materials had been in storage for a period of about 11 months. Considerable variation was found in the moisture content of the materials since the previous inspection during May 1944.

TABLE 34.—Gain in moisture content of variously treated lots of ammonium nitrate during storage<sup>1</sup> at different locations

Lot	Baltimore, Md.		New Orleans, La.		Stoneville, Miss.		Raleigh, N. C.		Wilson Dam, Ala.		Beltsville, Md.	
	Period	Gain	Period	Gain	Period	Gain	Period	Gain	Period	Gain	Period	Gain
ANPL:	Days	Percent	Days	Percent	Days	Percent	Days	Percent	Days	Percent	Days	Percent
No. 1	150	0.05	141	0.60	128	0.72	132	0	116	0.13	165	0.03
No. 2	150	.08	141	.38	128	.14	132	.05	116	.11	165	.02
No. 3	150	.12	141	.53	128	.30	132	.14	116	.33	165	.04
No. 4	150	.26	141	.67	128	.21	132	.20	116	.27	165	.23
TVA:												
No. 1	167	.23			128	.77	132	.06	116	.35	172	.08
No. 5B	167	.22	135	1.44	128	.81	132	.11	116	.54	172	.14
No. 6C	167	.27	135	1.75	128	.70	132	0	116	.50	172	.13
No. 7C	167	.33			128	1.38	132	.56	116	.89	172	.19
No. 8C			135	2.31					116	.63		
No. 9C			135	1.24					116	.50		
MCW:												
No. 1			72	.31			74	2.30			97	0
No. 2			72	.51			74	2.16			97	0
No. 3							74	2.08			97	0
No. 4			72	.22							97	0
No. 5											97	0

<sup>1</sup> See tables 27 and 33 for dates of storage and inspection.<sup>2</sup> Stored at Pittsburg, Kans.

TABLE 35.—Moisture content of variously treated lots of ammonium nitrate after 5½-months' storage under controlled conditions at Beltsville, Md.

Lot	Initial moisture content, Dec. 31, 1943	Moisture content at end of storage period, June 20, 1944				
		Room 13 (35° F.; 75 percent relative humidity)	Room 18 (55° F.; 60 percent relative humidity)	Room A (85° F.; uncontrolled humidity)	Room 23 (75° F.; 65 percent relative humidity)	Room 2 (55° F.; 80 percent relative humidity)
ANPL:	Percent	Percent	Percent	Percent	Percent	Percent
No. 1	0.12	0.05	0.05	0.17	0.32	0.74
No. 2	.13	.09	.07	.39	.25	.69
No. 3	.25	.31	.42	.32	.79	.71
No. 4	.19	.31	.32	.29	.60	.90
TVA:						
No. 1	.10	.11	.09	.24	.13	1.10
No. 5B	.17	.25	.33	.34	1.20	1.50
No. 6C	.12	.16	.14	.22	.60	1.37
No. 7C	.25	.26	.34	.58	2.24	1.45

<sup>1</sup> Moisture content after storage of 2½ months, Dec. 31, 1943, to Mar. 14, 1944.

While most of the bags showed a slight increase in moisture content, a few showed a decrease. It was further observed that the bags that had absorbed sufficient moisture during the humid season to become wet showed such a marked increase in hardness at the time of the final inspection as to make it difficult to pry them apart. This was to be expected, in view of the marked drop in temperature and humidity that had occurred since the previous inspection. It has been shown that caking occurs in ammonium nitrate when crystallization takes place from the solution phase as a result of a drop in temperature or a drying out of the material. Both factors operated between the previous and the final inspections. A marked increase in caking was therefore to be expected in the case of the materials stored in the more permeable bags, and this was found to be the case, as shown in table 36.

TABLE 36.—Caking tests with variously treated lots of ammonium nitrate in final storage tests at different locations<sup>1</sup>

Lot	New Orleans, La.			Raleigh, N. C.			Stoneville, Miss.			Wilson Dam, Ala.		
	Moisture content	Lumps		Moisture content	Lumps		Moisture content	Lumps		Moisture content	Lumps	
		Weight <sup>2</sup>	Hardness <sup>3</sup>		Weight <sup>2</sup>	Hardness <sup>3</sup>		Weight <sup>2</sup>	Hardness <sup>3</sup>		Weight <sup>2</sup>	Hardness <sup>3</sup>
	Percent	Pounds		Percent	Pounds		Percent	Pounds		Percent	Pounds	
ANPL No. 4	1.62	1	S	0.29	0	S	0.53	1	S	0.82	0	S
ANPL No. 3	1.65	8	S to M	.46	0	S	.36	8	S to M	.57	1	S
TVA No. 5B	3.44	33	S to H	.59	5	S to H	1.43	34	M to H	1.08	11	S to H
TVA No. 7C				1.24	30	S to H	1.40	84	S to H	1.24	32	S to H
TVA No. 6C	1.72	70	M to H	.68	40	S to H	.15	74	H	.78	49	S to H
TVA No. 8C	4.06	64	M to H							1.02	41	S to H
ANPL No. 2	1.05	36	S							.44	59	M to H
ANPL No. 1	2.10	81	S	.05	74	M to H	.07	95	S to H	.20	78	M to H
TVA No. 9C	1.51	90	VH		92	M to H	.04	97	H	.75	92	M to H
TVA No. 1					98	VH	.13	98	VH	.33	98	M to VH

<sup>1</sup> At the end of a period of approximately 11 months.<sup>2</sup> After 1 drop of bag.<sup>3</sup> S = soft; M = medium; H = hard; VH = very hard.

The results of the final inspection emphasize the necessity of thoroughly drying the ammonium nitrate before bagging and storing in moistureproof bags in a dry building. They further indicate that if the material contains any considerable percentage of moisture it is inadvisable to carry it in storage from the hot humid weather of summer into the cool dry weather of late fall or winter. Only the ANPL ammonium nitrate that had been treated with 0.5 percent of a petrolatum-paraffin mixture plus 3.5 percent of Kittitas, and with 3.5 percent of Kittitas alone (samples ANPL Nos. 4 and 3), remained in a satisfactory condition for direct application in the field at the end of the 11-month storage period. The condition of the other materials would place them in essentially the same order as was done at the time of the second inspection (see table 32).

### EFFECT OF BAGS ON STORAGE BEHAVIOR

In the study of the hygroscopicity of different types of ammonium nitrate, it was found that the uncoated ANPL and TVA materials differed but little in their rates of moisture absorption (table 11). It was further found that coating either product with a waxlike or water-repellent material greatly reduced its rate of moisture absorption, but that the addition of a conditioning material, such as kieselguhr, more or less nullified the effect of the water repellent. This nullifying effect increased with time of storage until, after a period of several months, there was little difference in rate of moisture absorption between ammonium nitrate treated with wax and kieselguhr and the same material treated with kieselguhr only.

Of the types of ammonium nitrate used in the storage tests, lot ANPL No. 4 had been treated with 0.5 percent WP + 3.5 percent Kittitas while lot ANPL No. 3 had been treated with 3.5 percent Kittitas only. The results given in tables 34 and 35 show that the initial effect of the wax coating in reducing rate of moisture absorption was entirely destroyed during the storage period. It will be noted also that both lots of ammonium nitrate contained a little higher percentage of moisture at the end of the storage period than the untreated ammonium nitrate. This may have been due in part to moisture absorbed by the Kittitas itself, as explained on pages 39 to 41, and to the fact that the conditioned materials did not cake, although the untreated ammonium nitrate did cake and was therefore less subject to movement of air through the mass.

The data in tables 34 and 35 show further that when there is a positive differential in vapor pressure between the outside and inside of the bags in which they are stored, TVA ammonium nitrate absorbs moisture more rapidly than the corresponding ANPL material under the same conditions of temperature and humidity. This was particularly noticeable in the materials stored in room 2 at Beltsville, Md. According to the data in table 34 the TVA materials absorbed more moisture in 2½ months than the ANPL in 5½ months, although the conditions of storage with one exception were the same for both types. The one exception was the quality of the bags used. Both types of material were stored in the same bags in which they were shipped. The package used in shipping the ANPL ammonium nitrate was a 5-ply multiwall paper bag with 1/90-pound asphalt-laminated layer

and valve closure. The TVA material was shipped in a burlap bag with pleated paper lining attached to the inside of the burlap with asphalt and closed with a wire tie.

It was observed in storage tests in humid areas that the burlap bags frequently became dripping wet, while the paper bags with an asphalt-laminated layer remained relatively dry. The ammonium nitrate in the wet burlap bags was found to be undrillable, whereas the material in unbroken asphalt-laminated multiwall paper bags of good quality was usually in suitable condition for direct application in the field.<sup>8</sup> These observations indicate that the moisture-resistant bags used in these large-scale storage tests differ greatly in their permeability to water vapor and in the protection that they afford to ammonium nitrate stored under humid conditions. A study was accordingly undertaken of the relative permeability to water vapor of the different types of bags that are now being used or might be used in the shipment and storage of ammonium nitrate.

### BAG-STORAGE TESTS

In making bag-storage tests the bags were filled with granular ammonium nitrate that had been conditioned with 3.5 percent of Kittitas to prevent caking. The tops of the bags were closed with plywood clamps bolted together in such way as to insure against any leakage of water vapor through the closures. This method of closure afforded a convenient means for opening and closing the bags for the removal of samples. The bags were stored in an air-conditioned room that was maintained at a temperature of 35° C. (95° F.) and a relative humidity of 80 percent. An Indiana sampler was used to withdraw samples from different parts of each bag for moisture determinations at intervals of 1 month. The results obtained are given in table 37.

The results show (1) that ammonium nitrate in a new burlap bag with asphalt-attached creped and pleated paper lining will absorb more than twice as much moisture in 1 month under the conditions of the test as a paper bag containing one 30-30-30-pound asphalt-laminated layer; (2) that the permeability to water vapor of multiwall paper bags with one 30-30-30-pound asphalt-laminated layer is at least twice as great as that of bags containing two 30-30-30-pound asphalt-laminated layers of the same quality; (3) that the permeability of the asphalt-laminated layers in certain makes of bags may be greater than that in other bags of the same type that contain a less weight of asphaltum; and (4) that there is very little difference as a rule in the permeability to water vapor of burlap bags with double asphalt layers and multiwall paper bags containing two asphalt-laminated layers.

The difference in water vapor pressure between the outside and the inside of a bag of ammonium nitrate under the conditions of the test is equivalent to about 10.3 mm. of mercury ( $\frac{2}{3}$ ). The corresponding differential in pressure that would prevail in the case of bags of ammonium nitrate stored in commercial warehouses will differ greatly in different localities and in the same locality at different seasons of the year, as shown in table 38. If it be assumed that the transmission

<sup>8</sup> ROSS, W. H. AMMONIUM NITRATE IN PUERTO RICO. Div. Soil and Fert. Invest. Res. Rpt. 17, 24 pp. 1944. [Processed.]

TABLE 37.—Moisture absorbed by granular ammonium nitrate stored in multiwall paper bags and in burlap bags of different types at 95° F. and a relative humidity of 80 percent

## MULTIWALL PAPER BAGS

Bag No.	Type of bag	Moisture absorbed after—	
		1 month	2 months
		Percent	Percent
P-1	5-ply, with one 25-25-25-pound A-L-L <sup>1</sup>	1.25	
P-2	do.	1.33	
P-3	5-ply, with one 30-30-30-pound A-L-L	1.67	
P-4	do.	1.50	
P-5	6-ply, with two 25-25-25-pound A-L-L	.44	1.07
P-6	do.	.23	.87
P-7	do.	.52	.73
P-8	do.	.41	.73
P-9	6-ply, with two 30-30-30-pound A-L-L	.55	.09
P-11	do.	.83	1.71
P-12	do.	.57	1.31
P-13	do.	.60	1.25
P-14	do.	.65	.92
P-15	do.	.81	1.37
P-16	do.	.76	.98
P-17	6-ply, with one 30-100-30-pound A-L-L	.33	.65
P-18	do.	.17	.51
P-19	6-ply, with two 30-60-30-pound A-L-L	.12	.25
P-20	do.	.07	.25

## BURLAP BAGS

B-1	Burlap, with asphalt-attached single-pleated paper lining on inside.	3.20	
B-2	Burlap, with asphalt-attached single-erinkled paper lining on inside.	2.81	
B-3	30-pound erinkled kraft, 90-pound asphalt, 40-pound 10-ounce burlap, 90-pound asphalt, 30-pound erinkled kraft. <sup>2</sup>	.70	1.12
B-4	do.	.70	1.06
B-5	40-pound creped kraft, 55-pound asphalt, 40-pound 10-ounce burlap, 55-pound asphalt, 40-pound creped kraft. <sup>2</sup>	.38	.56
B-7	40-pound 10-ounce burlap, 55-pound asphalt, 40-pound creped kraft, 55-pound asphalt, 40-pound creped kraft. <sup>2</sup>	.34	
B-8	do.	.33	
B-9	do.	.57	1.00
B-10	do.	.47	.90
B-11	do.	.58	1.17
B-12	do.		.79
B-13	Burlap, with asphalt-attached single-erinkled paper lining on inside.	1.69	

<sup>1</sup> Asphalt-laminated layer.<sup>2</sup> From outside to inside.

TABLE 38.—Average monthly differential in vapor pressure between the outside and inside of ammonium nitrate bags stored in different localities for the periods indicated

Period of storage (1944)	Place of storage					
	Baltimore, Md.	Raleigh, N. C.	Memphis, Tenn.	Norfolk, Va.	Savannah, Ga.	New Orleans, La.
	Mm. of mercury	Mm. of mercury	Mm. of mercury	Mm. of mercury	Mm. of mercury	Mm. of mercury
Jan.-Mar.	-0.46	-0.28	-0.05	0.12	0.60	1.21
Apr.-June	-.20	.24	.04	1.27	2.12	2.10
July-Sept.	1.32	2.55	2.44	3.31	4.47	4.06
Oct.-Dec.	-.18	.69	.03	.63	1.23	1.48
Yearly average	.10	.65	.77	1.33	2.10	2.23

of water vapor through a bag is proportional to the time of storage and to the differential vapor pressure between the inside and the outside of the bag, then the period of storage in an air-conditioned room at 35° C. and a relative humidity of 80 percent that will be equivalent in water vapor transmission to a storage period of 1 year (29) at each of the places listed in table 38 will be as follows:

Storage of 1 year at:	<i>Equivalent storage period (days) in room at 35° C. (95° F.) and a relative humidity of 80 percent</i>
Baltimore, Md. ....	4
Raleigh, N. C. ....	23
Memphis, Tenn. ....	27
Norfolk, Va. ....	46
Savannah, Ga. ....	73
New Orleans, La. ....	78

These results indicate that the 2-months' storage period to which the bags were subjected in the air-conditioned room at 35° C. and a relative humidity of 80 percent is equivalent, with respect to moisture transmission, to storage for the greater part of a year in the most humid sections of the country. It is also apparent that a storage period of 1 month under the conditions stated is equivalent to 1 year or more in such places as Memphis, Raleigh, Baltimore, and all points north (29).

The role played by bags in the storage of ammonium nitrate in humid areas was further demonstrated in a second series of tests. In these tests, granular ammonium nitrate that had been conditioned with 3.5 percent of Kittitas was stored in the air-conditioned room at 35° C. and a relative humidity of 80 percent for a period of 2 weeks. The temperature was then further increased and the humidity was lowered to 40 percent for 2 days to cause a partial drying out of the material through the bags. Although all bags contained the same material and were treated in exactly the same way, their contents showed a marked variation in caking that was closely correlated with the permeability of the bags to water vapor.

Thus the material in the bags that permitted a relatively high absorption of moisture and a corresponding loss of moisture during the drying period showed a marked degree of caking at the end of the test. Little or no caking, however, was found in bags that prevented any appreciable transmission of water vapor in either direction throughout the period of storage. This experiment shows conclusively that the degree of caking that ammonium nitrate undergoes when stored in humid sections of the country depends in large measure on the permeability to water vapor of the bags in which it is stored.

#### PERMEABILITY TO WATER VAPOR

The results obtained in the bag storage tests depended on the leakage or permeability to water vapor of the bags as a whole rather than on that of the walls alone. A determination of the permeability to water vapor of the walls of bags of different types was made by means of a modification of the water-vapor permeability cell as described by Carson (9). The permeability cell used in this determination is shown in cross section in figure 10. The cell consists of an



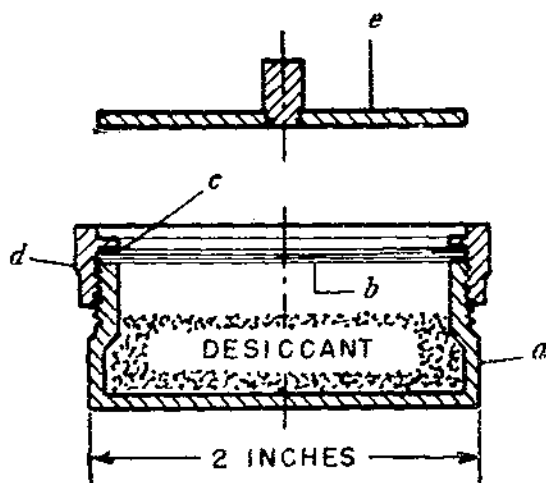


FIGURE 10.—Water-vapor permeability cell.

aluminum dish *a* with threaded top containing a desiccant; a bag sample that is cut in the form of a circular disk *b*; a brass ring *c*, 1/16 inch in width, that serves as a bearing for disk *b*; an open cap *d* that is capable of being screwed onto the dish *a* and makes contact with it through the medium of the ring *c* and the disk *b*; and a cover *e* for closing the cell while it is being weighed. The permeability cell when assembled is 2 inches in

diameter and 1 1/4 inches high. The construction of an aluminum cell of this kind is such that a sample cut from a bag in the manner described by Ross and Yee (21) can be quickly mounted in place to give an airtight connection and as quickly detached without the necessity of having to attach and remove a wax seal, as described by Carson (9).

When making a test, about 8 grams of Anhydron is placed in the bottom of the dish *a* (fig. 10), the bag sample and brass ring *c* are centered in place, and the open cap *d* is screwed on tightly to make an airtight connection through the waxed edge of the bag sample that serves as a gasket. The entire assembly with the cover removed is then placed in a humidity chamber of the type described by Yee and Davis (40). The humidity chamber is provided with a fan and contains a saturated solution of potassium sulfate. It is placed in a constant-temperature room at 30° C. A saturated solution of potassium sulfate gives a relative humidity of 96.3 percent at this temperature (2). The differential in water vapor pressure between the outside and the inside of the bag sample under these conditions is equivalent to 30.7 mm. of mercury.

The permeability of the bag sample under this differential of vapor pressure was determined every day for 7 days. The gain in weight during the first 3 days while the bag sample was being saturated with moisture was discarded. During this period the gain in weight was excessive. A constant gain in weight was generally maintained during the rest of the experiment. The permeability of the sample was calculated from the average gain in weight during the last 4 days. It was expressed in terms of grams of water passing through a 100-square-inch area of the sample for 24 hours at 96.3 percent relative humidity and a temperature of 30° C. To offset irregularities in the bag, tests were made with six samples taken from different parts of the bag and the average taken for the true permeability.

The ordinary kraft layers of multiwall paper bags have little or no moisture-proofing value. This property as a rule is confined to the

asphalt-laminated layers alone. Some of the new wet-strength layers used in some types of bags, however, seem to retard the passage of moisture through the wall to a slight extent. For this reason all layers from each bag were used in the tests.

The results obtained by this method of determining the relative permeability to water vapor of bags of different types are given in table 39, in comparison with the corresponding data in table 37. The

TABLE 39.—Resistance of bags of different types to water-vapor transmission, as determined by permeability and storage tests

Bag and No.	Number and weight of asphalt layers	Permeability expressed in grams of water per 100 square inches per 24 hours <sup>1</sup>	Moisture absorbed per month in storage test <sup>2</sup>
With single asphalt layer:	Number/Pounds		Percent
P-17.....	1 400	0.87	0.33
P-1.....	1 25	1.50	1.25
P-4.....	1 30	1.50	1.50
B-13.....		1.98	1.69
B-2.....		2.59	2.81
B-1.....		2.65	3.20
With double asphalt layers:			
P-10.....	2 60	.42	.12
P-9.....	2 30	.45	.55
B-11.....	2 55	.65	.58
P-15.....	2 30	.71	.81
B-7.....	2 55	.73	.31
P-7.....	2 25	.71	.52
B-5.....	2 55	.86	.38
B-9.....	2 55	.83	.57
P-5.....	2 25	.87	.44
P-13.....	2 30	1.00	.69
B-3.....	2 90	1.01	.70

<sup>1</sup> B = bushing bags; P = multiwall paper bags.

<sup>2</sup> At 30° C. (86° F.) and a relative humidity of 95 percent.

<sup>3</sup> At 35° C. (95° F.) and a relative humidity of 80 percent.

data in table 39 show that the two methods of testing place the bags in the same order when they differ considerably in their resistance to water vapor. Thus, both methods of testing show that bags with a single asphalt layer are about twice as permeable to water vapor as those with two asphalt layers. The two methods of testing, however, do not always place the bags in the same order when the differences in their resistance to water vapor are not great. This is due to irregularities in different parts of the same bag and to imperfections in the bottom and longitudinal seams. Owing to these variations in bags of the same and of different types, the storage test is considered more accurate for each bag as a whole than the laboratory permeability test. It has been observed that when bags show irregularities in different parts of the same bag, the results obtained in duplicate tests and with the two different methods of testing do not show so close agreement as do results with bags that are of more uniform construction.

#### LIGHT-PENETRATION TEST

While storage in an air-conditioned room under carefully controlled conditions is the most reliable method for comparing the relative permeability to water vapor of bags of different types, the results thus obtained can be approximated very closely and much more rapidly by a method recently developed in this laboratory. This method

consists in placing a part of the bag or of an asphalt-laminated layer over a ground-glass window in a box that contains a bright light. When different bags are examined in this way in a darkroom they often present a very different appearance, as shown by photographs (fig. 11). These photographs were made by exposing, over a printing box, a sensitive film placed in contact with a section of a burlap bag or an asphalt-laminated layer in a printing frame.

An examination of the data in table 37 shows that the results obtained in the storage tests are in good agreement with what might have been expected from the quality of the bags, as indicated by the illustrations in figure 11.

New bags were used in all the storage tests, and the only handling to which they were subjected was that incident to the filling and the closing of the bags. The primary object of the storage tests was to

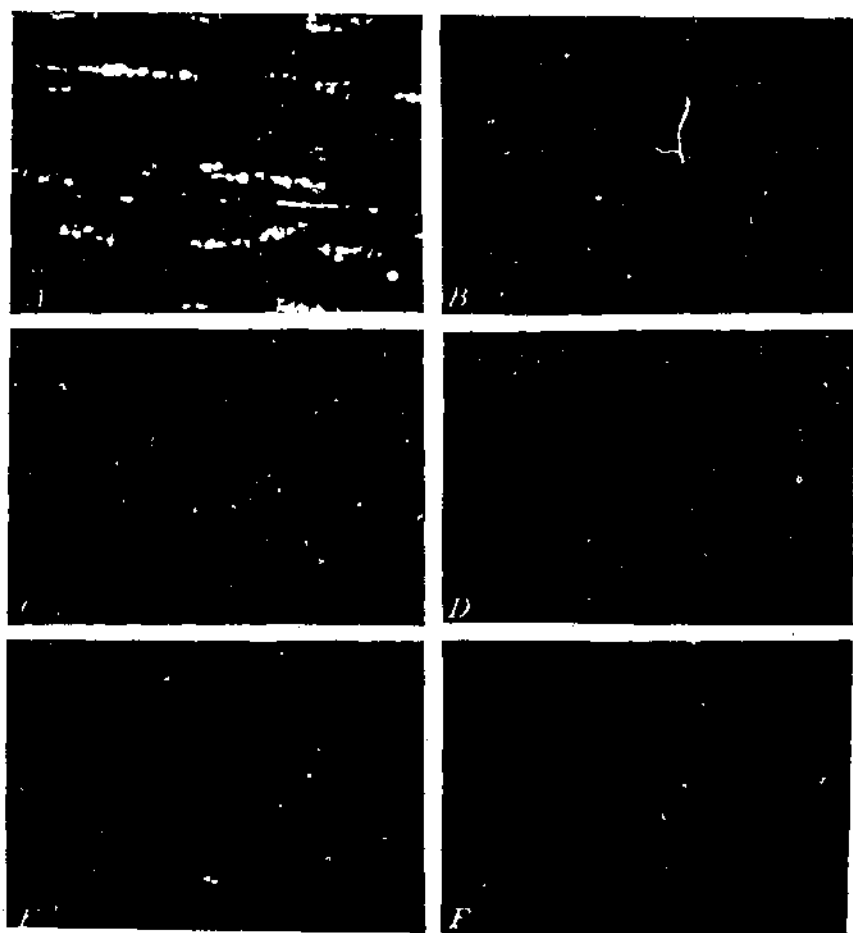


FIGURE 11.—Light transmitted through bags of different types (see table 37): A, No. B-1; B, No. P-1; C, No. P-3; D, No. P-5, crumpled; E, No. B-5; and F, No. P-7.

compare the permeability to water vapor of bags of different types and makes. It was thought inadvisable to make tests with used bags, inasmuch as there was no way of knowing the relative degree of handling to which different bags had been subjected. Photographs made of bags indicate that their permeability to water vapor could be impaired by moderately rough handling. A paper bag that was originally of better quality than another might, therefore, show less resistance to water vapor, owing to greater crumpling of the asphalt-laminated layers as a result of rough handling. Thus, it was felt that storage tests made with used bags might lead to wrong conclusions respecting the relative merits of different makes of bags.

#### BAG SEAMS AND CLOSURES

The different methods used by the manufacturers in closing the bottom and sides of the burlap bags used in the storage tests are as follows: Cemented center seam with folded and sewn bottom; folded and sewn bottom and side; and cemented center seam with sewn bottom and overlapping cemented tape of the same material as the bag.

All makes of the multiwall paper bags used in the storage tests, with one exception, were constructed with gusseted sides and sewn bottoms. The one exception was one constructed in the form of a sack with cemented center seam and pasted bottom. The walls of these 5-ply multiwall bags with two asphalt-laminated layers were of very good quality, but the bags examined were defective in that a water-soluble paste was used in closing the bottom. For this reason openings frequently developed in the bottom of the bags, thus permitting leakage of water vapor.

All the paper bags with self-forming gussets had cemented center seams. The different methods used in closing the bottom of the bags were as follows: Sewn with cemented bound-over creped-paper tape; sewn with cemented bound-over creped-paper tape and wax-dipped; sewn with cemented bound-over creped-paper tape plus a second paper tape covering the needle thread; and sewn with cemented bound-over creped-paper tape plus a second paper tape covering the needle thread and wax-dipped.

Two methods have so far been used in closing bags used in the shipment and storage of ammonium nitrate. These consist of wire ties and valve closures. Valve bags have proved satisfactory, provided the valve is properly constructed and closed. If not properly closed some of the ammonium nitrate will flow through the opening in the valve and spread over other bags in the same pile. In humid areas the spilled material will liquefy and cause deterioration of the outer layers of the bags with which it comes in contact. Little or no leakage takes place from valve bags that are properly closed. Any leakage that takes place from standard valve bags is due to carelessness or inexperience on the part of workmen.

Wire ties for ammonium nitrate bags have the disadvantages that (1) they tend to slip off and cut the necks of bags; (2) they rust and break, thereby allowing the ammonium nitrate to flow over and destroy other bags in turn; (3) they give a more or less rounded shape to the bags, thus increasing the difficulty of maintaining piles in an upright position; and (4) they frequently permit some leakage of

water vapor through the closure. These disadvantages are so serious that the use of wire ties for ammonium nitrate bags is now being replaced by valve or sewn closures.

### IMPORTANCE OF BAG RECOGNIZED BY INDUSTRY

The importance of the part played by bags in the storage of ammonium nitrate is now recognized by all the leading ammonium nitrate producers. Steps are being taken to replace bags of the type represented by bag No. B-1, listed in table 37, with multiwall paper bags having two asphalt-laminated layers or their equivalent in burlap bags. Bags that meet the following specifications, reading from the inside to the outside, are considered satisfactory for service in the storage of ammonium nitrate:

#### Five-ply multiwall paper bag:

Consisting of—

One 75- to 90-pound asphalt-laminated layer.

Two 50-pound natural kraft paper layers.

One 75- to 90-pound asphalt-laminated layer.

One 70-pound low-stretch crepe-paper layer.

Sewn bottom and valve top, wax-dipped.

Waterproof glue in seams.

Asphalt-laminated tuck-in sleeve, or wax-dipped sewn closure.

#### Burlap bag:

Consisting of

30-pound crinkled kraft paper.

55-pound asphalt.

30-pound crinkled kraft paper.

55-pound asphalt.

40-pound 10-ounce burlap.

Cemented center seam.

Sewn and taped over bottom.

Sewn closure, with bound-over crinkled paper tape, wax-dipped.

In the case of the burlap bag the central crinkled kraft and the outer burlap layer may be interchanged without having any appreciable effect on its permeability to water vapor. For overseas shipment an extra layer, such as 40-pound natural kraft, is added, preferably on the inside of the multiwall paper bag.

The resistance of asphalt-lined bags to water vapor can be greatly impaired by filling the bags with ammonium nitrate at too high a temperature. This injury to ammonium nitrate bags is now being prevented by cooling the material to 40° C. (104° F.), or below, before transferring it to the bags. Filling the bags too full or using bags of unusually stiff walls increases the tendency of ammonium nitrate to cake.

### DRILLABILITY

At ordinary summer temperatures untreated ammonium nitrate may be freely exposed to the air without danger of moisture absorption, provided the relative humidity remains below 60 percent. This humidity is above the monthly average for all seasons in certain sections of the country, but it is below the average prevailing in the more humid sections of the Southeast, where most fertilizers are used. Exposure of untreated ammonium nitrate in areas of high humidity is therefore likely to result in rapid absorption of moisture, particularly during the hot humid months of the summer season.<sup>9</sup>

<sup>9</sup> WHITTAKER, C. W., LUNDSTROM, F. O., YEE, J. Y., and others. DRILLABILITY OF VARIOUS TYPES OF AMMONIUM NITRATE FERTILIZERS. Div. Soil and Fert. Invest. Res. Rpt. 25, 26 pp., illus. 1944. [Processed.]

It has been shown that certain water-repellent coatings will reduce for short periods the rate at which ammonium nitrate absorbs moisture under humid conditions, but that for adequate protection under commercial conditions dependence must be placed on the bag rather than on any treatment with a water-repellent. When ammonium nitrate is to be distributed in the field, however, it must be removed from the bag in which it was stored and shipped. Any treatment that would afford protection during the relatively short period required to distribute it in the field might therefore be expected to have a beneficial effect on its drillability. An experimental study was accordingly undertaken of the effectiveness of different treatments on the drillability of ammonium nitrate under conditions of high humidity. The tests<sup>10</sup> were made (1) under controlled conditions in a constant-temperature, constant-humidity room, and (2) in the field.

### TESTS IN AIR-CONDITIONED ROOM

#### MATERIALS

The materials used in the drillability tests, together with their screen analysis and moisture content, are listed in table 40. The different lots of ammonium nitrate were supplemented with a sample of Chilean nitrate of soda, which served as a standard. The average particle size of the latest type of ANPL ammonium nitrate (samples Nos. 7 and 8) is somewhat larger than the ANPL material used in the storage tests (samples Nos. 2, 3, 4, and 5) and the average particle size of the latter in turn is considerably larger than any of the TVA materials. The differences in the particle size of the Chilean nitrate of soda and the WCW and MCW ammonium nitrate were not great. All samples used in the tests had an initial low-moisture content, and all were passed through a coarse mesh screen to remove abnormal particles and lumps.

#### PROCEDURE

The drillability of the different materials listed in table 40 was studied: (1) By repeated passage of each at regular intervals through standard commercial fertilizer-distributing equipment with exposure to controlled conditions of temperature and humidity, referred to as cyclic-exposure tests; (2) by passing each material through the same equipment after exposure in thin layers under controlled conditions of temperature and humidity for varying periods of time, referred to as static-exposure tests; and (3) by determining the uniformity of distribution before and after exposure, referred to as uniformity-of-distribution tests.

Except when otherwise noted, all tests under controlled conditions were made at 75° to 77° F. and a relative humidity of 80 to 82 percent in an air-conditioned room that housed the fertilizer distributors

<sup>10</sup> All the drillability tests were made with equipment designed and furnished by the Division of Farm Power and Machinery, of this Bureau, and operated under the supervision of L. G. Schoenleber and G. A. Cummings.

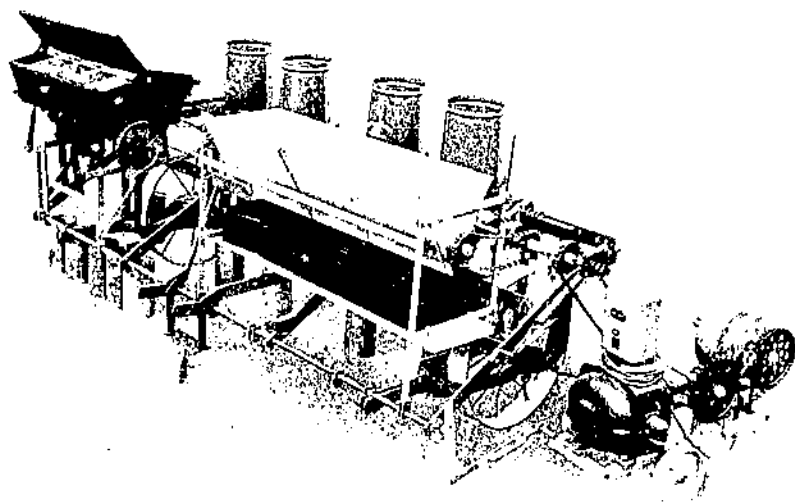


FIGURE 12.—Assembly used in cyclic- and static-exposure tests.

(fig. 12) and other equipment used in the tests. These conditions are not the most severe that fertilizers may encounter, but they may be considered typical of conditions that frequently occur in the more humid areas where fertilizers are widely used.

#### EQUIPMENT

Three common types of fertilizer-distributing mechanisms or feeds in which distinctly different mechanical principles are employed were used in the cyclic- and static-exposure tests.

- (a) Gravity feed, consisting of a series of adjustable openings in the bottom of a hopper, above which a reel-type agitator rotates. This type is commonly used for broadcast applications.
- (b) Rotating-plate feed, consisting of a shallow circular pan with rim wall at the periphery that serves as the bottom of the hopper. The fertilizer is permitted to remain at a regulated depth on the outward section of the plate and is removed by a small plow. Row applications are often made with this type of mechanism.
- (c) Star-wheel feed, consisting of a rotating horizontal circular plate with projections or fingers extending beyond the periphery. The star wheel is located immediately above the hopper bottom, and as the wheel rotates the fingers pass over an opening in the bottom of the hopper where the fertilizer is dispensed. An inclined plate known as the back plate shields a section of the feed wheel as well as the delivery opening, thus preventing uncontrolled flow of fertilizer from the hopper. The material is carried between the fingers of the feed wheel and the quantity dispensed depends on the rotating speed of the feed wheel. In some cases, an adjustable gate in the back plate above the fingers is used to augment the rate of adjustment. This type is typical of that used in grain drills.

TABLE 40.—Materials used in drillability tests

Sample	Treatment		Screen fractions (mesh)					Moisture content
	Water repellent <sup>1</sup>	Conditioner	+10	10-14	14-20	20-40	-40	
Nitrate of soda, Champion Brand:			Percent	Percent	Percent	Percent	Percent	Percent
No. 1	None	None	35	27	19	14	4	0.02
ANPL ammonium nitrate:								
No. 1	do	do	14	38	26	17	5	.10
No. 2	0.5 percent WP	do	14	34	20	21	2	.10
No. 3	None	3.5 percent Kittitas	13	39	23	17	3	.28
No. 4	0.5 percent WP	do	15	38	27	15	5	.20
No. 5	None	2.8 percent Celite	3	68	21	7	1	.22
No. 6	0.42 percent WP	2.9 percent Celite	33	47	15	4	1	.28
No. 7	0.62 percent PRP	do	33	47	15	4	1	.31
No. 8	1.0 percent PRP	2.0 percent tricalcium phosphate	4	45	25	21	5	.18
No. 9	do	5.0 percent Dicalite	3	41	25	22	9	.12
WCW ammonium nitrate:								
No. 1	None	4.7 percent Celite	10	68	15	5	2	.13
No. 2	0.88 percent OP	5.0 percent Celite	12	62	16	9	1	.24
No. 3	0.80 percent OP	5.0 percent plaster of paris	5	61	21	11	2	.18
TVA ammonium nitrate:								
No. 1	None	None	1	4	26	58	11	.02
No. 3	1.0 percent petrolatum	4.0 percent kaolin	0	8	35	49	8	.13
No. 5B	1.0 percent PRP	4.0 percent Celite	1	4	23	55	17	.09
No. 6C	do	4.0 percent kaolin	1	8	28	53	10	.03
No. 7C	do	4.0 percent plaster of paris	0	10	31	51	8	.18
MCW ammonium nitrate:								
No. 1	None	None	22	57	14	6	1	.04
No. 2	0.2 percent PRP	2.0 percent Dicalite	11	70	16	3	0	.07
No. 3	2.0 percent Premix		14	59	17	8	1	.05

<sup>1</sup> WP = paraffin-petrolatum; PRP = petrolatum-rosin-paraffin; OP = oil-paraffin; and Premix = 1 part PRP plus 3 parts Dicalite mixed before applying.



Five identical dispensing mechanisms of each type were assembled to form a unit. The three units were mounted on a common frame and driven by a single power unit so that five materials could be tested simultaneously in each of the three types of mechanism. Each unit could be individually controlled by means of suitable clutches on a central countershaft, and interchangeable drive sprockets permitted independent changes of the speed of the rotating part of each unit as required to obtain the desired delivery rates. The hoppers were adjusted to deliver approximately 100 pounds of an average mixed fertilizer, with due consideration given to the usual spacing of dispensing units on each type of machine represented. The size of the delivery opening and the speed of the rotating parts were the same for each of the five mechanisms constituting a hopper unit.

For the uniformity-of-distribution tests, use was made of equipment developed by the Division of Farm Power and Machinery of this Bureau (fig. 13). A carriage consisting of a row of 25 metal compartments, each 1 foot square, is moved along a track under a fertilizer distributor at a constant rate while the distributor is likewise operated at a constant rate. The compartments have sloping sides that converge to a small funnellike opening. The fertilizer that falls into each compartment passes into a container under the mouth of the



FIGURE 13.—Equipment used in uniformity-of-distribution test.

funnel. This equipment makes it possible to collect and weigh the quantity of fertilizer distributed on each successive square foot. It was used indoors but, because of its size, not in the air-conditioned room. Star-wheel and rotating-plate dispensers were mounted on this machine. These were similar to those used in the cyclic- and static-exposure tests, except that the star wheel was equipped with a mechanical knocker that tended to clean each finger as it passed over the opening where the fertilizer is dispensed.

#### CYCLIC-EXPOSURE TESTS

The same quantity of each of five materials selected for testing was placed in separate hoppers of each of the distributing units, and the dispensing equipment was then operated for an accurately measured interval of sufficient length to permit the discharge of an adequate sample into tared cans. The discharged materials were weighed at once without removal from the room and the receptacles replaced under the hoppers. The operation of the dispensing units was then continued until the hoppers were empty, except for small quantities of material in the corners. This routine was repeated twice at 2½- to 3-hour intervals. Each material was thus exposed to the temperature and humidity of the room for 5 to 6 hours and was passed through the dispensing units three different times.

The results obtained in these tests are shown in table 41, where the quantities delivered in unit time are expressed as a percentage of that delivered before the material had been exposed to the humid atmosphere. The gravity-feed hopper gives the most severe test, but unfortunately this is less consistent than that afforded by either of the other two types of hoppers.

The results of the tests reported in table 41 indicate that no one of the three types of distributing equipment used was sufficiently accurate to measure small differences in drillability between the different materials. The data do indicate, however, that the drillability of ammonium nitrate is (1) increased by treatment with a petrolatum-paraffin or a petrolatum-rosin-paraffin mixture and a suitable conditioning agent, and (2) that so treated it is equal to or superior in drillability to that of untreated granular Chilean nitrate of soda.

While a soft water-repellent coating, as petrolatum or oil, may have a marked effect in reducing the rate at which ammonium nitrate absorbs moisture, it decreases its drillability by increasing the tendency of the particles to stick together. This is particularly noticeable in the case of TVA sample No. 3, which was treated with 1.0 percent of petrolatum. The use of petrolatum alone as a water repellent was discontinued in the fall of 1943.

#### STATIC-EXPOSURE TESTS

In making the static-exposure tests 1 gallon of each material to be studied was placed in each of four cloth-bottomed trays and exposed in the air-conditioned room for 3, 6, and 9 hours. The layer of material in the trays was about half an inch thick and was actually exposed to the air on both upper and lower surfaces, since the cloth bottom of the tray offered little resistance to the passage of atmospheric mois-

TABLE 41.—Relative drillability, on basis of 100 for first period, of nitrate of soda and of variously treated lots of ammonium nitrate, as indicated by cyclic-exposure tests with gravity-feed, rotating-plate, and star-wheel distributors

Sample	Treatment		Gravity feed in—		Rotating plate in—		Star wheel in—	
	Water repellent <sup>1</sup>	Conditioner	Second period	Third period	Second period	Third period	Second period	Third period
Nitrate of soda, Champion Brand:			Percent	Percent	Percent	Percent	Percent	Percent
No. 1.....	None.....	None.....	80	76	97	95	98	94
ANPL ammonium nitrate:								
No. 1.....	do.....	do.....	22	0	95	94	83	51
No. 2.....	0.5 percent WP.....	do.....	53	36	92	96	92	87
No. 3.....	None.....	3.5 percent Kittitas.....	73	65	92	96	97	97
No. 4.....	0.5 percent WP.....	do.....	100	103	92	98	92	93
No. 5.....	None.....	2.8 percent Celite.....	96	40	93	90	99	96
No. 6.....	0.42 percent WP.....	2.9 percent Celite.....	108	84	102	102	99	99
No. 7.....	0.62 percent PRP.....	do.....	87	81	99	100	101	103
No. 8.....	1.0 percent PRP.....	2.0 percent tricalcium phosphate.....	61	25	85	78	97	93
No. 9.....	do.....	5.0 percent Dicalite.....	129	104	106	109	101	103
WCW ammonium nitrate:								
No. 1.....	None.....	4.7 percent Celite.....	135	130	97	105	97	94
No. 2.....	0.88 percent OP.....	5.0 percent Celite.....	75	27	102	74	101	97
No. 3.....	0.80 percent OP.....	5.0 percent plaster of paris.....	80	21	95	59	95	88
TVA ammonium nitrate:								
No. 1.....	None.....	None.....	21	5	98	89	98	94
No. 3.....	1.0 percent petrolatum.....	4.0 percent knollin.....	11	0	90	74	94	55
No. 6C.....	1.0 percent PRP.....	do.....	97	63	121	112	96	94
No. 7C.....	do.....	4.0 percent plaster of paris.....	76	64	120	117	97	95
MCW ammonium nitrate:								
No. 1.....	None.....	None.....	59	40	96	96	94	93
No. 2.....	0.2 percent PRP.....	2.0 percent Dicalite.....	59	30	96	90	94	101
No. 3.....	2.0 percent Premix.....	do.....	91	93	96	94	94	100

<sup>1</sup> WP = paraffin-petrolatum; PRP = petrolatum-rosin-paraffin; OP = oil-paraffin; and Premix = 1 part PRP plus 3 parts Dicalite mixed before applying.

TABLE 42.—Relative drillability, on the basis of 100 for zero hour's exposure, of nitrate of soda and of variously treated lots of ammonium nitrate as indicated by static-exposure tests with gravity-feed, rotating-plate, and star-wheel distributors

Sample	Treatment		Gravity feed in—			Rotating plate in—			Star wheel in—			Moisture in sample after exposure of—		
	Water repellent <sup>1</sup>	Conditioner	3 hours	6 hours	9 hours	3 hours	6 hours	9 hours	3 hours	6 hours	9 hours	3 hours	6 hours	9 hours
Nitrate of soda, Champion Brand:			Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent
No. 1.....	None.....	None.....	43	38	25	88	83	84	97	87	84	0.5	1.2	1.3
ANPL ammonium nitrate:														
No. 1.....	do.....	do.....	0	0	0	95	57	1	36	23	2	2.0	2.7	5.0
No. 2.....	0.5 percent WP.....	do.....	53	39	9	94	93	86	82	78	39	1.0	2.5	3.3
No. 3.....	None.....	3.5 percent Kittitas.....	5	0	0	83	13	0	60	4	0	1.7	4.3	5.9
No. 4.....	0.5 percent WP.....	do.....	9	0	0	90	65	3	52	22	1	2.3	4.1	5.2
No. 5.....	None.....	2.8 percent Celite.....	2	0	0	82	68	3	60	7	1	2.4	4.3	6.8
No. 6.....	0.42 percent WP.....	2.9 percent Celite.....	40	24	3	92	89	51	82	70	18	2.6	4.2	5.8
No. 7.....	0.62 percent PRP.....	do.....	41	33	23	82	93	84	83	84	77	2.5	4.3	4.8
No. 8.....	1.0 percent PRP.....	2.0 percent tricalcium phosphate.....	15	10	8	78	75	78	80	73	63	1.0	2.4	3.3
No. 9.....	do.....	2.0 percent Dialite.....	70	8	9	99	82	85	97	78	65	1.2	2.5	3.7
WOW ammonium nitrate:														
No. 1.....	None.....	4.7 percent Celite.....	16	13	0	94	91	59	83	85	45	2.6	3.3	5.8
No. 2.....	0.88 percent OP.....	5.0 percent Celite.....	13	8	0	70	71	73	68	66	55	2.0	3.4	4.3
No. 3.....	0.80 percent OP.....	5.0 percent plaster of paris.....	2	0	0	72	69	30	67	17	12	1.3	2.2	4.1
TVA ammonium nitrate:														
No. 5B.....	1.0 percent PRP.....	4.0 percent Celite.....	0	0		58	50		14	8			2.2	
No. 6C.....	do.....	4.0 percent kaolin.....	5	0	0	90	10	6	30	3	0	1.1	2.2	2.7
MCW ammonium nitrate:														
No. 1.....	None.....	None.....	26	22	(?)	75	67	(?)	74	67	(?)	1.1	1.6	1.5
No. 2.....	0.2 percent PRP.....	2.0 percent Dialite.....	24	12	10	85	74	76	81	74	64	1.0	2.1	2.0
No. 3.....	2.0 percent Premix.....	do.....	74	47	18	96	93	85	99	96	78	.3	1.0	2.0

<sup>1</sup> WP = paraffin-petrolatum; PRP = petrolatum-rosin-paraffin; OP = oil-paraffin; and Premix = 1 part PRP plus 3 parts Dialite mixed before applying.

<sup>2</sup> Moisture lost from sample by dripping.

<sup>3</sup> Passed through dispenser partly by liquid flow.

ture. The four trays were placed in various random positions in special frames made for supporting them, and these were so arranged that each would receive about the same quantity of circulating conditioned air. At the conclusion of the exposure, the four trays of each material were emptied into a small concrete mixer and mixed to insure uniform moisture content throughout. A small sample for moisture determination was taken at the conclusion of the mixing.

The exposed materials were then passed through the dispensing units, care being taken to use the same hopper unit for the same material in each test. The quantities delivered per unit time with each dry material were previously determined in triplicate, and the average of these values was used in calculating the relative drillability of the different materials. In testing the exposed materials, the delivery was continued over a longer period in many cases in order to obtain a reliable sample of material and to average out minor variations. Such data were calculated back to the time intervals found convenient in testing materials with good drillability—2 minutes for the gravity-feed distributor, 4 minutes for the rotating plate, and 8 minutes for the star wheel.

Eighteen materials were selected for the static-exposure tests. The quantities delivered on zero exposure are given the value of 100 in table 42, and those delivered after exposure are expressed as percentages of that value.

The results obtained in the static tests are in general agreement with those reported for the cyclic tests. They show, as before, that ammonium nitrate treated with a petrolatum-paraffin or petrolatum-rosin-paraffin mixture and a suitable conditioning agent is more drillable than the untreated material and at least as drillable as the untreated Chilean nitrate of soda. The static test was carried out under more severe conditions than the cyclic tests as regards moisture absorption. It might be expected, therefore, that treatment of the ammonium nitrate with a water repellent might have a more beneficial effect on drillability under these conditions than a coating of a conditioning agent. The results given in the table show that this is the case.

#### UNIFORMITY-OF-DISTRIBUTION TESTS

In the uniformity-of-distribution experiments both the rotating-plate and the star-wheel types of fertilizer-dispensing units were employed. After the material to be tested was placed in the hopper the dispensing mechanism was operated until feeding became uniform, then the train of compartments was passed under the dispensing unit at the rate of  $1\frac{1}{2}$  miles per hour. Each compartment was brushed down to make certain that all material flowed into the collecting receptacle. The results were recorded in the order in which the compartments passed under the dispensing unit. When studying the effect of exposure to high relative humidity on the uniformity of distribution, the materials were first exposed in the trays for 8 hours, as in the static tests. All tests were made in duplicate, 2 series of 25 weights each being taken for each material tested. The series of compartments were always started under the star-wheel hopper at the same point in the delivery cycle in order to eliminate so far as possible any variation in the operation of the equipment. The data

TABLE 43.—Uniformity of distribution of nitrate of soda and of variously treated lots of ammonium nitrate from rotating-plate and star-wheel hoppers after 3-hours' exposure at 75° to 77° F. and a relative humidity of 80 to 82 percent

Sample	Treatment		Rotating-plate hopper				Star-wheel hopper				Moisture content
	Water repellent <sup>1</sup>	Conditioner	Average quantity delivered		Variation coefficient		Average quantity delivered		Variation coefficient		
			Dry	After exposure	Dry	After exposure	Dry	After exposure	Dry	After exposure	
Nitrate of soda, Champion Brand:			Grams	Grams	Percent	Percent	Grams	Grams	Percent	Percent	Percent
No. 1.....	None.....	None.....	6.75	4.76	13.2	23.7	1.20	0.62	27.2	70.7	0.50
ANPL ammonium nitrate:											
No. 3.....	do.....	3.5 percent Kittitas.....	2.77	2.38	12.8	20.3	.44	.27	48.3	78.4	2.88
No. 8.....	1.0 percent PRP.....	2.0 percent tricalcium phosphate.....	3.21	2.40	13.1	18.1	.67	.35	31.0	54.4	1.12
No. 9.....	do.....	5.0 percent Dicalite.....	2.95	2.76	15.0	13.8	.46	.42	44.6	37.4	1.38
TVA ammonium nitrate:											
No. 6C.....	1.0 percent PRP.....	4.0 percent kaolin.....	5.05	2.35	15.0	119.8	1.00	.25	21.6	95.0	1.35
MCW ammonium nitrate:											
No. 1.....	None.....	None.....	3.03	3.01	12.3	51.3	.50	.46	51.3	107.9	.86
No. 2.....	0.2 percent PRP.....	2.0 percent Dicalite.....	3.31	2.54	13.5	20.9	.50	.52	60.2	64.3	1.06
No. 3.....	2.0 percent Premix.....		3.16	2.81	16.4	17.3	.52	.52	54.5	62.7	.78

<sup>1</sup> WP = paraffin-petrolatum; PRP = petrolatum-rosin-paraffin; and Premix = 1 part PRP plus 3 parts Dicalite mixed before applying.

were evaluated by use of means and coefficients of variation computed for each series of 50 observations. The distributing mechanisms used were similar to those of the static and cyclic tests and hence subject to about the same variations, but because of the large number of observations made in each test the results are considered relatively more reliable.

The results obtained in the uniformity-of-distribution tests indicate that all the materials when dry were dispensed from the rotating-plate hopper with about the same degree of uniformity (table 43). Greater variations occurred in the case of the star-wheel hopper. The data show that of the materials tested the ANPL and MCW ammonium nitrate that had been treated with a PRP mixture and a conditioning agent were distributed most uniformly after exposure and that the most irregular distribution occurred in the case of the corresponding TVA material. These results indicate that when other factors are the same the uniformity with which ammonium nitrate can be distributed increases with increase in the size of its particles.

This was further demonstrated in static-drillability tests with two lots of untreated ANPL ammonium nitrate of 6 to 10 mesh and 20 to 40 mesh. The proportion of the coarser mesh material that was delivered from the gravity-feed, rotating-plate, and star-wheel distributors after an exposure of 6 hours amounted to 22, 84, and 41 percent, respectively, of the unexposed samples. The corresponding delivery rates for the finer mesh material were 2, 61, and 23 percent, respectively.

## FIELD TESTS

### EQUIPMENT AND MATERIALS

The field tests were made by hand distribution and with a 7-foot International Harvester broadcast-fertilizer distributor. The hopper of the distributor was partitioned into four compartments to permit the simultaneous testing of four materials. The distributor was pulled at a uniform rate by a small tractor.

The dispensing mechanism of this distributor consists essentially of a reciprocating mesh-type plate, the meshes of which are filled by the gravitational flow of the material through closely spaced cross slots in the hopper bottom. The charge of fertilizer in the meshes is carried horizontally to delivery slots in a lower deck that forms a closed channel and supports the mesh plate. The delivery slots are offset far enough from those in the hopper bottom to prevent any uncontrolled flow of the material. A reciprocating agitator working immediately above the slots in the hopper bottom serves to keep the slots uniformly filled. When the fertilizer leaves the delivery slots it falls onto a spreader board that scatters the individual streams of fertilizer and also serves as a windshield to prevent the material from blowing about.

The materials selected for the field tests were Chilean nitrate of soda, Champion Brand; ANPL ammonium nitrate samples Nos. 4 and 5; and TVA ammonium nitrate sample No. 6C (table 40). All materials were taken directly from the bags, which were opened only as used. The distributor was set to deliver 120 pounds per acre of the TVA sample. Rates obtained with the other materials differed

somewhat from this, owing to differences in their volume weights and flow properties.

### PROCEDURE

In the machine tests the distributor was driven at a rate of about  $1\frac{1}{2}$  miles per hour over a piece of meadowland where the ground was hard and rough. Both the hand and machine tests were made between 9:30 a. m. and 2:30 p. m. on a day when the temperature varied from  $72.5^{\circ}$  to  $81.0^{\circ}$  F. and the relative humidity from 95 to 68 percent. In spite of the moderately severe conditions of the test, all materials were dispensed with apparent uniformity throughout the period of the test and no clogging of the mechanism was observed.

### RESULTS

All of the materials, especially the nitrate of soda and the TVA ammonium nitrate, tended to accumulate on the spreader board to some extent and to fall off in lumps at intervals when more jolting occurred than usual. The TVA material tended to bridge in the hopper after about the first hour and the fertilizer adhering to the board appeared wetter than the other materials. The ANPL sample No. 4 appeared wetter on the distributing board than ANPL sample No. 5, and the No. 4 sample together with TVA sample 6C appeared to be slightly wetter in the dispensing mechanism and in the hopper than any of the other materials. Small drops of liquid appeared on the parts of the mechanism handling these two materials.

The quantities and screen analyses of the different materials adhering to the spreader board after  $4\frac{1}{2}$ -hours' operation of the distributor are given in table 44.

TABLE 44.—Quantity and screen analyses of the different materials adhering to the spreader board after  $4\frac{1}{2}$ -hours' field operation of distributor

Material	Treatment	Quantity on board	Screen analyses			
			Material from board		Fresh material	
			+80 mesh	-80 mesh	+80 mesh	-80 mesh
		Grams	Percent	Percent	Percent	Percent
Nitrate of soda, Champion Brand.						
No. 1	None	321	74	26	93	7
Ammonium nitrate:						
ANPL No. 4	3.5 percent Kulfas.	31	13	87	96	4
ANPL No. 5	0.5 percent WP plus 3.5 percent Kulfas.	41	50	50	95	5
TVA No. 6C	1.0 percent PRP plus 4.0 percent kaolin.	178	86	14	99	1

The higher proportion of fines in the material adhering to the distributing board in relation to that in the fresh material indicates that the fine fractions in the original material were responsible for most of the accumulation and that this would be largely eliminated in materials that do not contain fine fractions.

All of the materials could be distributed satisfactorily by hand under the conditions of the test. The finer grained TVA sample was not so easily handled as the granular materials of larger particle size.



This material also tended to blow about more than the others. None of the materials took up enough moisture during the test period to interfere with hand distribution.

The behavior of the materials in the field tests is in general agreement with that observed in the drillability tests made under controlled conditions. The results as a whole indicate that the drillability of a material increases within limits with increase in the size of its particles and that a sample of ammonium nitrate that has been treated with a double coating of an effective water repellent and a suitable conditioning agent is more drillable under humid conditions than one that is untreated or treated with a conditioner only. It would seem, therefore, that ammonium nitrate intended for use in the more humid areas of the South should preferably be coated with a water repellent as well as a conditioning agent, but that the use of a water-repellent coating would be unnecessary in the treatment of sprayed ammonium nitrate intended for use in the central and northern sections of the United States.

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