1956

PROCEEDINGS OF THE

FERTILIZER INDUSTRY ROUND TABLE

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FOREWORD

DURING the past 15 years a remarkable change has occurred in the pattern of fertilizer manufacture and in the nature of fertilizer materials. The new technology practiced in the fertilizer industry coupled with the rapid expansion of fertilizer usage in the Middle West and Pacific regions have made our industry one of the largest segments of the nation's huge chemical industry.

We are living in a world of change in which the search for more efficient and more economical sources of materials and technologies never ends. Changes in sources of raw materials and demands for more concentrated complete fertilizers have created new problems in both the factory and on the farm. All these changes are emphasizing the need for the closest, friendliest cooperation between industry and government research personnel concerned with the production and efficient use of fertilizers.

This friendly cooperation is reflected in the contributions made at the 1955 and 1956 Fertilizer Industry Round Tables. In the following pages is information on the latest advances in fertilizer technology reported by government and industry personnel. To one who is familiar with the old-time attitude of industry's reluctance to talk about improvements or advances in manufacturing techniques, it is truly remarkable to see this friendly exchange of information at these Round Tables by various units of industry with competitors and government agencies. Of the many changes taking place in our time this certainly is one that strikes me as most significant; it heralds a new era of greater progress and development in the fields of manufactures and agnonomy. No one can ever get so far ahead of his group as to think he has a monopoly of the knowledge and arts of his industry. To succeed as a leader he must bring the majority of the industry up to his level and then start a new round of advances and so on. The new fertilizer industry deals with engineering problems, synthesis of ammonia, urea, ammoniating solutions, nitric and phosphoric acids, statistical quality control techniques, high gas temperatures, heats of reaction, flow integrators, electric pulses and all the many other concepts of modern technology. The old-time superintendent familiar with the simple, manually-operated fertilizer plant would be amazed and bewildered were he to visit the clean, efficient, odorless fertilizer facilities of today.

Granulation and ammoniation are perhaps two of the most significant changes in fertilizer manufacture. The Round Table discussions concentrated on these subjects and the problems directly and indirectly associated with them. The Proceedings constitute an excellent contribution to the development of these practices.

A word of explanation: in these Proceedings have been included two papers from overseas which although not actually presented at the Round Table we thought sufficiently pertinent to be included. The paper by Dr. Bernard Raistrick replaces that given by his associate, Mr. E. P. Hudson, covering the same developments. Mr. Hudson's paper had been published elsewhere. We are grateful to the publishers of *Agricultural Chemicals* for assembling, printing and issuing the collected papers as Proceedings.

The Executive Committee thanks authors and all others who participated in the Round Table for their generous and loyal support.

VINCENT SAUCHELLI,
Chairman

Fertilizer Industry Round Table Proceedings

1956

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the Changing Technology of Granulation and Ammoniation

THE development of the TVA continuous ammoniator has been described by Yates, Nielsson, and Hicks *(14)*. Pilot-plant studies of the use of the continuous ammoniator with auxiliary equipment to produce granular, high-analysis fertilizers were described by Hein, Hicks, Silverberg, and Seatz *(2)*. Subsequent papers reported the results of further pilot-plant work on producing granular fertilizers using diammonium phosphate *(4)*, calcium metaphosphate *(11)*, and urea-containing ammoniating solutions and solid urea *(10)*.

These previous papers have shown that granular fertilizers can be made from a large variety of formulations and raw materials by using the TVA continuous ammoniator and accessory equipment. This report deals with new combinations of raw materials and methods that are currently being studied in the continuous ammoniator pilot plant, and with other new information pertinent to ammoniation and granulation.

Ammonium Phosphate-Nitrate and Ammonium Phosphate-Urea

ONE current project is the production of granular, high-analysis fertilizers from phosphoric acid and ammoniating solution. The fertilizer products made from these materials contain ammonium phosphate and either ammonium nitrate or urea, depending on the composition of the ammoniating solution. The advantages of the process are (1) unusually high analysis of the products and (2) high water solubility. Estimates show that the process is economically quite attractive when phosphoric acid is available at a favorably low price.

Several methods of carrying out the reaction between ammoniating solution and phosphoric acid and of granulating the product were studied in the laboratory. The continuous ammoniator method was selected for pilot-plant development. One advantage of this method is that the equipment in many granulation plants could be adapted to use this method.

The pilot plant was the same as that described previously *(2)* with a few modifications, which are mentioned later. Ammoniating solution and phosphoric acid were fed into the ammoniator through appropriate distribution. Potassium chloride, if desired, and recycle were fed through volumetric feeders. Granulation was controlled by the proportion of recycle. The amount of recycle required varied from about 50 to 75% of the throughput, depending on the grade. When electric-furnace phosphoric acid was used, some grades were not sufficiently plastic to granulate properly. This difficulty was overcome by premixing some phosphate rock with the phosphoric acid. The amounts of phosphate rock used were such as to supply 5 to 10% of the P_2O_5 content of the product; 5% was sufficient for satisfactory granulation. Similar improvement in granulation was obtained by adding enough concentrated superphosphate to supply 15% of the P_2O_5. The concentrated superphosphate was added along with the other dry raw materials. The availability of the P_2O_5 in the product was the same (about 99%) when using the phosphate rock—acid mixture as when using separate addition of concentrated superphosphate.

When wet-process phosphoric acid was used, it was not necessary to add phosphate rock or superphosphate to ensure satisfactory granulation. However, it might be economically advantageous to do so in making some grades.

When phosphoric acid was used without phosphate rock addition, the acid was distributed under the bed in the ammoniator through a drilled pipe adjacent to the ammonia distributor. The acid distributor was the same length as the ammonia distributor and was drilled to give the same distribution pattern. When phosphate rock was premixed with the acid, the mixture could not be fed through a drilled pipe; it was distributed on the surface of the bed through an open-end pipe which was moved back and forth across the length of the bed by a mechanical device at a rate of 30 passes per minute.

The granular product leaving the ammoniator contained 3 to 6% mois-

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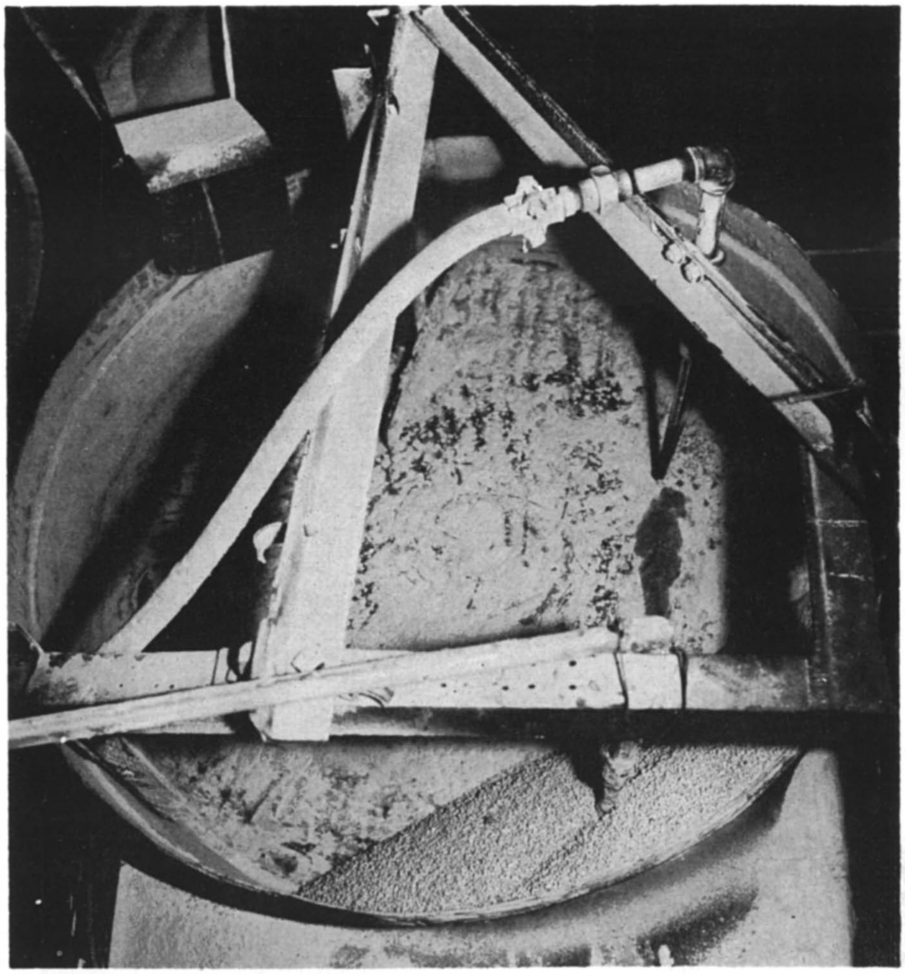

Fig. 1. Experimental Pan Granulator

ture. It was dried in the rotary dryer to less than 1% moisture. The dried product was cooled and screened to separate plus 6-mesh material as oversize and minus 12, 20, or 28 mesh as undersize. The undersize and crushed oversize were recycled to the ammoniator. Usually the amount of oversize and undersize was less than the recycle requirement, so it was necessary to crush some of the onsize product to get enough recycle.

TABLE I

Ammonium Phosphate-Nitrate and Ammonium Phosphate-Urea

	15-30-15	16-22-16	16-48-0
Formulation, lb./ton			
Nitrogen solution	630[a]	967[b]	334[c]
Anhydrous ammonia	—	—	220
Ammonium sulfate	100	—	—
Concentrated superphosphate	180	—	316
Phosphoric acid (80% H_3PO_4)	915	780	1410
Potassium chloride	500	557	—
Recycle, % of throughput	48	74	80
Moisture content, %			
From ammoniator	6.6	3.6	4.2
From dryer	0.8	0.8	0.7
Temp., ammoniator product, °F.	170	149	101
Granulation, %			
Oversize	42	23	21
Onsize	51	62	56
Undersize	7	15	23

[a] Ammonia—urea—amonium carbamate solution (UAL-B).
[b] Ammonia—ammonium nitrate solution (Spensol C).
[c] Ammonia—ammonium nitrate solution (Spensol A).

Bag-storage tests of 6 months' duration were made with some of the products. When the products had been dried to less than 1% moisture, cured for a few days, and conditioned with 2% kaolin, no caking and little or no bag set occurred. Bulk-storage tests of conditioned 16-22-16 product were made in which a few tons were held in an open bin in a well-ventilated building throughout the summer. No caking or deterioration in physical properties was observed.

Data for typical pilot-plant tests for production of 15-30-15, 16-22-16, and 16-48-0 are shown in Table I. Other grades that have been made in the pilot plant include 17-17-17, 11-22-22, and 7-28-28. A small proportion of sulfuric acid was used in addition to phosphoric acid in making 17-17-17.

Nitric Phosphate

TVA has developed several nitric phosphate processes through the pilot-plant stage. The results of this work have been published *(3, 5, 7, 8, 9, 13)*. Also, similar processes are used in Europe. The fertilizer industry showed considerable interest in the nitric phosphate processes a few years ago during the sulfur shortage. Less interest has been shown since the sulfur supply has become adequate. Only two nitric phosphate plants were built, and one is now under construction.

One of the main advantages of the nitric phosphate process is the low cost of raw materials, which may be 20 to 30% less than that of similar grades of fertilizers made by conventional processes *(12)*.

In explaining the fertilizer industry's reluctance to adopt the nitric phosphate processes, the following disadvantages have been pointed out: (1) the equipment required is much more expensive than that used in the usual fertilizer manufacturing plant, (2) entirely new plants would be required, since little use could be made of existing plants, and (3) the nitric phosphate processes are not adaptable to the manufacture of as large a variety of grades as most manufacturers deem necessary, and the equipment is not adaptable to make use of other processes.

Recently, however, exploratory tests made in the TVA continuous ammoniator plant showed that this equipment can be used instead of the slurry-type ammoniation and granulation steps of the original process. Use of the TVA ammoniation-granulation method would eliminate most of the disadvantages of the nitric phosphate processes. The equipment cost would be reduced by 50% or more, and operating costs would be decreased. Many existing granulation plants could be adapted to use the nitric phosphate process by addition of a relatively inexpensive acidulation unit and other minor changes. The equipment would be versatile enough that it could be used alternately for nitric phosphate and conventional processes.

In the exploratory tests, phosphate rock was acidulated with nitric and phosphoric acid. The acidulation was carried out batchwise in two stainless steel tanks. A continuous acidulation system will be used in fur-

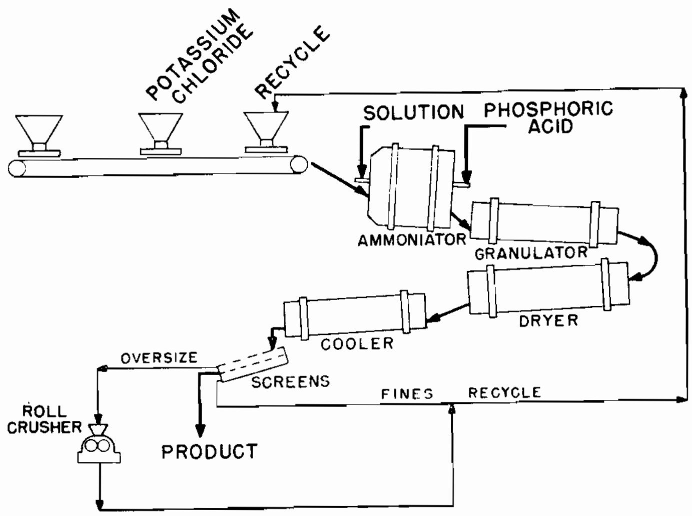

Figure 1
FLOW DIAGRAM OF TVA PILOT PLANT FOR PRODUCTION OF AMMONIUM PHOSPHATE-NITRATE

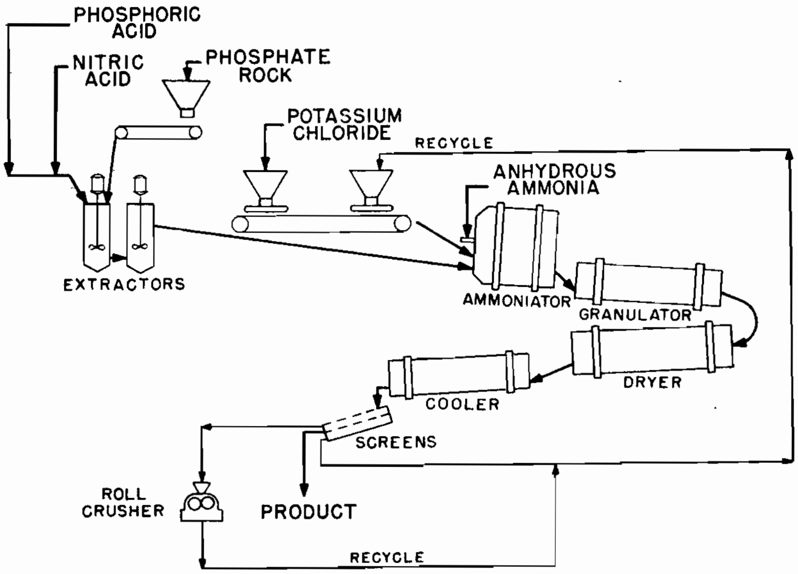

Figure 2
MODIFIED NITRIC PHOSPHATE PROCESS

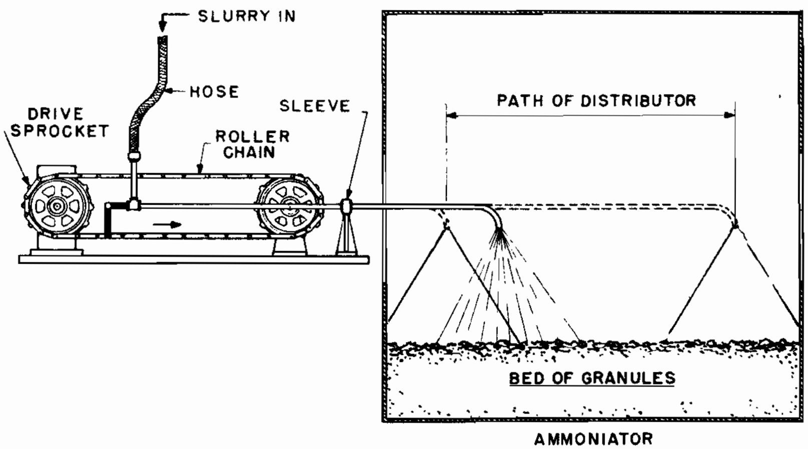

Figure 3
SLURRY DISTRIBUTOR FOR CONTINUOUS AMMONIATOR

ther tests. The acidulated slurry was distributed on the surface of the bed of material in the continuous ammoniator from an open-end pipe, which moved back and forth across the length of the bed at a rate of 30 passes per minute. Potassium chloride and recycle were fed to the ammoniator through the usual dry feeders. Anhydrous ammonia, in gaseous form, was fed under the bed through a distributor similar to that used in previous work for ammoniation of superphosphates. The ammonia was used in gaseous form to increase the heat of reaction and thereby assist in evaporating water.

Table II shows data for a pilot-plant test in which a 14-14-14 nitric phosphate fertilizer was produced using nitric and phosphoric acids for acidulation. Operation of the pilot plant was very good. Ammonia recovery was satisfactory. Availability of P_2O_5 in the product was 98%. Granulation was controlled by the amount of recycle. About 82% of the product had to be recycled to control granulation. This amount of recycle was high because (1) the nitric acid concentration was low (51% HNO_3) so an excessive amount of water was introduced with the acid and (2) the pilot-plant facilities were not adequate for crushing the recycled products and, consequently, much of it was recycled without crushing.

14-14-14 Data

Oversize (+6 mesh)	43
Onsize (—6 +20 mesh)	50
Undersize (—20 mesh)	7

TABLE III

Pilot-Plant Data for No-Nitrogen Grades Using Phosphate Rock[a]

Grade	0-14-14	0-14-14	0-20-20	0-26-26	0-48-0
Formulation, lb/ton product					
Phosphate rock	438	888	420	163	266
Concentrated superphosphate	—	—	574	771	1432
Ordinary superphosphate	720	—	—	—	—
Sulfuric acid (94% H_2SO_4)	326	613	281	—	—
Phosphoric acid (78% H_3PO_4)	—	—	—	148	309
Potassium chloride	466	452	657	888	—
Water	150	140	100	—	—
Steam	—	—	50	125	360
Recycle, % of total feed	—	48	—	8	21
Temp., ammoniator product, °F.	148	162	162	157	187
Onsize recovery after crushing, %	89	72	87	84	88
Moisture content of product, %	6.0	4.1	3.7	4.3	4.4
Net conversion of P_2O_5 in rock,[a] %	95	94	98	92	98

[a] After 1 to 7 days of curing.

The granulation efficiency was good; about 50% of the product was in the 6- to 20-mesh size range. The granules were round and hard. The product leaving the ammoniator contained about 3% moisture; it was dried to less than 1% moisture.

It is believed that the amount of recycle can be reduced to about two thirds of the throughput for some formulations. The process should be quite advantageous economically to manufacturers who can adapt their present plants to use it, if they can obtain nitric acid at a reasonable price.

Granular No-Nitrogen Grades

In the granulation of fertilizer grades that contain nitrogen, the reaction of ammonia with superphosphates or acid plays an important part in providing conditions that are favorable to granulation. This reaction is not available as an aid to granulation of no-nitrogen grades. Granular no-nitrogen grades are usually made by granulating superphosphates or mixtures of superphosphates with potash salts. In our first experiments we sought to granulate these materials by using the continuous ammoniator as a rotary granulator. Water or water and steam were added in sufficient quantity to cause granules to form. The granulation efficiency was fairly good, but the granules were not very strong. Also, the high percentage of moisture required for granulation made the drying step difficult and expensive.

TABLE II

Data for Production of Nitric Phosphate Fertilizer (14-14-14) in Continuous Ammoniator Pilot Plant

Formulation, lb./ton	
Phosphate rock	481
Phosphoric acid (75% H_3PO_4)	241
Nitric acid (51.7% HNO_3)	1222
Ammonia	176
Potassium chloride	509
Recycle, % of throughput	82
Temp., ammoniator product, °F.	154
Moisture content, %	
Ammoniator product	3.3
Dried product	0.9
P_2O_5 availability, %	98

TABLE IV

Granulation of Mixed Fertilizers in a Rotating Pan and in TVA Ammoniator-Granulator

Grade Type ammoniator-granulator	10-20-20[a] Pan	 Rotary drum
Granulation, %		
Oversize (+6 mesh)	37	30
Onsize (—6 +28 mesh)	57	69
Undersize (—28 mesh)	6	1
Onsize after crushing oversize, %	85	91
Degree of ammoniation	3.7	3.9
Ammonia loss, %	19.2	2.8
Ammoniator product		
Temperature, °F.	175	197
Moisture, %	3.8	2.9

[a] Raw materials: Nitrogen solution (21.7% NH_3, 65% NH_4NO_3), concentrated superphosphate, potassium chloride, and sulfuric acid.

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

Data for typical pilot-plant tests are presented in Table III. In making 0-20-20, concentrated superphosphate, phosphate rock, sulfuric acid, and potassium chloride were the raw materials. The amount of phosphate rock and sulfuric acid was such as to form about 700 pounds of superphosphate per ton of product. The acid: rock ratio was about 0.63 pound of H_2SO_4 per pound of rock, which is believed to be about the proportion generally used in making superphosphate. The granulation efficiency was satisfactory; about 61% was onsize, and the remainder was mostly oversize. After crushing the oversize, 87% was onsize. The conversion of the P_2O_5 content of the phosphate rock to an available form was 98% when analyzed 3 days later. The product as discharged from the cooler contained 3.7% moisture; after curing 1 week in an open bin, the moisture content was 3.2%. It was then bagged and stored; after 6 months there was no caking and very little bag set.

(Table III)

In a similar test with 0-14-14, about half of the superphosphate was supplied as cured material and half was formed from phosphate rock and acid. Granulation was controlled readily by controlling the amount of water. No recycle was required.

In another run on 0-14-14, all of the superphosphate was derived from phosphate rock and acid. About 50% recycle was required to control granulation. Granulation was controlled by water addition and recycle at such level as to provide about 50% fines (-16 mesh) for recycle. The P_2O_5 availability was 95% after 7 days' storage in an open pile.

Phosphate rock and phosphoric acid were used in making granular 0-26-26 and 0-48-0. The acid : rock ratio was somewhat less than that usually used in making concentrated superphosphate. About one third of the concentrated superphosphate was formed in the process, and the remainder was supplied as cured material from the TVA plant. Steam was added in the amount required to obtain good granulation. Granulation was quite good, and conversion of phosphate rock to an available form was quite satisfactory after only 1 day. In other runs, granular 0-48-0 was made entirely from phosphate rock and phosphoric acid; about 40% recycle was used to aid in granulation

The granules, after cooling, contained about 4% moisture and appeared to have satisfactory physical properties. They were hard, free-flowing, and resistant to abrasion.

Further work on the use of phosphate rock and acid to make granular superphosphates and phosphorus-potassium fertilizers is in progress.

Ammoniation and Granulation in a Pan Granulator

AN inclined, rotating pan has been used extensively to pelletize various materials such as iron ore and raw materials for Portland cement production. In some foreign countries the inclined pan granulator is used to granulate superphosphate or mixed fertilizer. It has been reported that both ammoniation and granulation have been carried out in the pan granulator.

The advantage claimed for the pan granulator is that a classifying action takes place such that the largest granules are discharged continuously over the rim of the pan, and the small granules and fines are retained for further build-up. This action is said to result in efficient, uniform granulation.

A small pan granulator, 3 feet in diameter was built to study granulation of ammonium nitrate. Good granulation was obtained by spraying hot, concentrated ammonum nitrate solution on fine, crystalline ammonium nitrate. As much as 85% of the product was obtained in the size range of 6 to 16 mesh.

Tests were then made to determine whether the pan granulator would be useful for ammoniation and granulation. In these tests, concentrated superphosphate and potassium chloride were fed continuously to the pan through a chute. Nitrogen solution and sulfuric acid were fed through distributors submerged in the material in the pan. The proportions of these materials were such as to make a 10-20-20 fertilizer.

After several attempts, fairly good granulation was obtained. Figure 1 is a photograph of the pan granulator. The pan was not rotating when the picture was made, because it was difficult to get a clear picture with the pan in motion. Table IV shows comparative data for the production of 10-20-20 in the pan granulator and in the TVA continuous ammoniator. The results are generally poorer with the pan granulator; the ammonia loss was high, and the granulation efficiency was not as good. The submerged distributors tended to interfere with the classifying action of the pan. When acid was applied by spraying it on the surface of the bed rather than by a submerged distributor, severe fuming took place.

(Figure 1 appears on Page 6, Table 4 appears on Page 7.)

The tests are not considered to be conclusive. It is quite possible that most of the difficulties experienced in the test could be overcome by modifying the design of the pan and the arrangement of the distributors. However, on the basis of these tests, it was considered unlikely that the pan granulator would have any significant advantage over the cylindrical continuous ammoniator.

Use of Coarse Potash

IT has become rather common practice to use coarse potassium chloride as an aid to granulation of low-nitrogen, high-potash grades that are otherwise difficult to granulate. Some questions have been raised as to what size range of potassium chloride particles is most effective in aiding granulation. In an attempt to shed some light on this question, a run was made in the pilot plant in which four sizes of potassium chloride were used.

The fertilizer produced in these runs was 4-16-16. The raw materials used were nitrogen solution, anhydrous ammonia, ordinary and triple superphosphates, sulfuric acid, and potassium chloride. Granulation was controlled by varying the sulfuric acid in the range of 100 to 180 pounds per ton of product.

Four sizes of potassium chloride were used. The coarsest was mostly 6 to 10 mesh, the next was 10 to 16 mesh, the third was 16 to 48 mesh, and the finest was mostly finer than 48 mesh. The most efficient granulation was obtained when using the 10- to 16-mesh potash; 70% of the product was in the desired size range of 6 to 16 mesh, 22% was oversize, and 8% was undersize. Very nearly as good results were obtained with the 6- to 10-mesh potash. Distinctly poorer results were obtained with the 16- to 48-mesh potash, and very poor results were obtained with the minus 48-mesh potash. The granulation efficiencies were 63, 70, 49, and 28% for the 6- to 10-, 10- to 16-, 16- to 48-, and minus 48-mesh potash sizes, respectively. The amount of sulfuric acid required to obtain as good granulation as could be obtained with each potash size increased from 107 to 179 pounds per ton of product as the potash size decreased.

It is concluded that the most effective potash particle sizes for promoting granulation of 4-16-16 are 6 to 10 mesh and 10 to 16 mesh; and that potash smaller than 28 mesh is relatively ineffective. In other runs, mixtures of fine and coarse potash were used. The granulation efficiency was about as high with the mixtures as when all coarse potash was used. This may indicate that only a limited number of nuclei are needed to promote granulation.

A question has been raised as to whether the use of granular potash in making granular fertilizer may increase the caking tendency of the product. Bag-storage tests were made of several grades made with granular potash or with nongranular potash. There was no indication that the particle size of the potash affected bag set or caking to any appreciable extent.

Caking of Granular Fertilizers

GRANULAR fertilizers are much less subject to caking during storage than nongranular mixtures of the same formulation and moisture content. However, granulation is not an infallible panacea for caking problems. Research by USDA *(1, 6)* has shown that many factors are involved in caking of granular and non-granular fertilizers. These factors include size and shape of granules, moisture content, curing period, formulation, and amount and kind of conditioner added, if any.

In the course of TVA's work on granulation of high-analysis fertilizers, the experimental products have been subjected to bag-storage tests. In some of these tests, caking has occurred. Microscopic examination of some of the caked products showed that the bonds between granules consisted of crystals of soluble salts that had formed on the surface of the granules during the storage period and had knitted the granules together. The crystals that formed the bond were identified as one or more of several compounds, such as potassium nitrate, monoammonium phosphate, and a urea-ammonium chloride double salt, and depended on the composition of the fertilizer granule.

It is pointed out that products that did not cake were prepared from the same formulations by better granulation, more thorough drying, longer curing, conditioning, or a combination of these factors.

Further work is being done to get a better understanding of the mechanism of caking and methods of preventing it.★★

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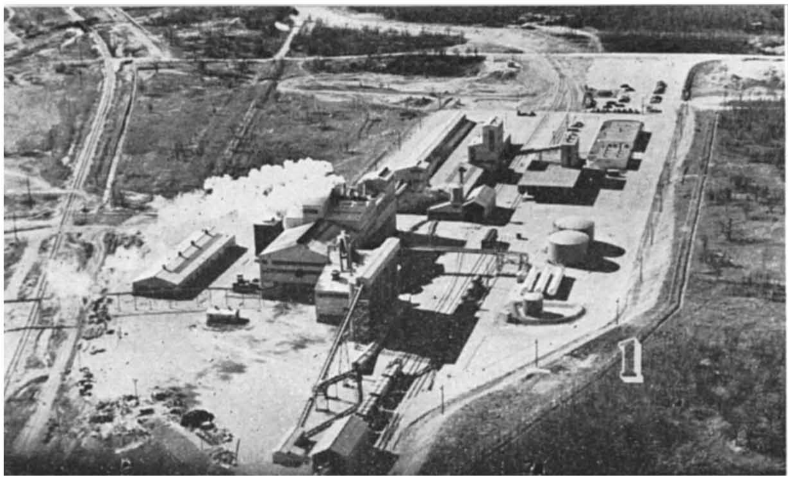
A high analysis product, and high water solubility are two advantages of the TVA process for granulation and ammoniation.



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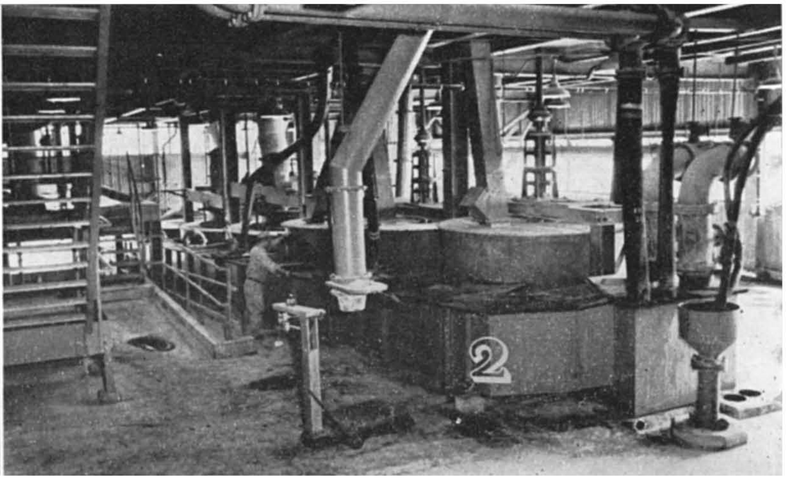

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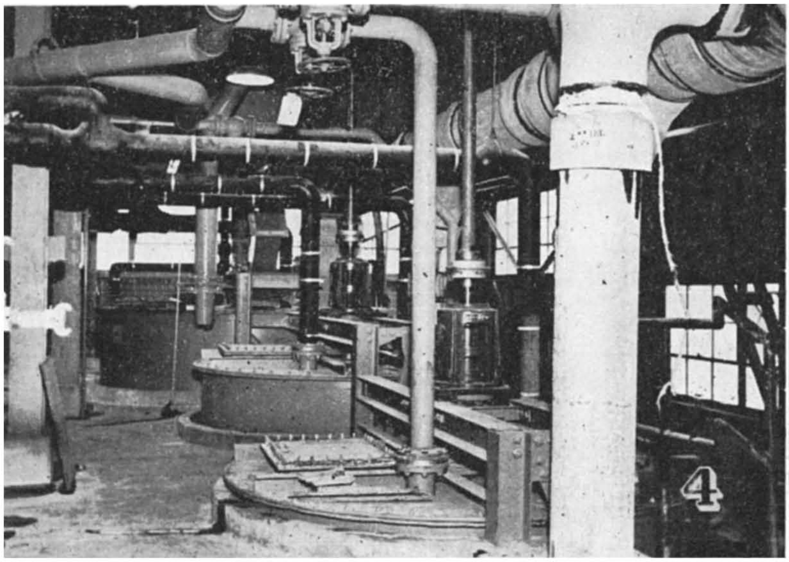

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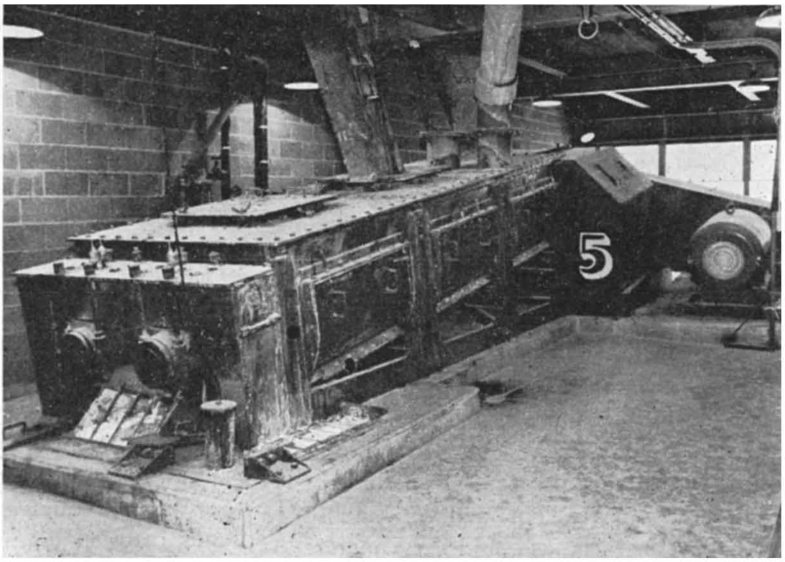

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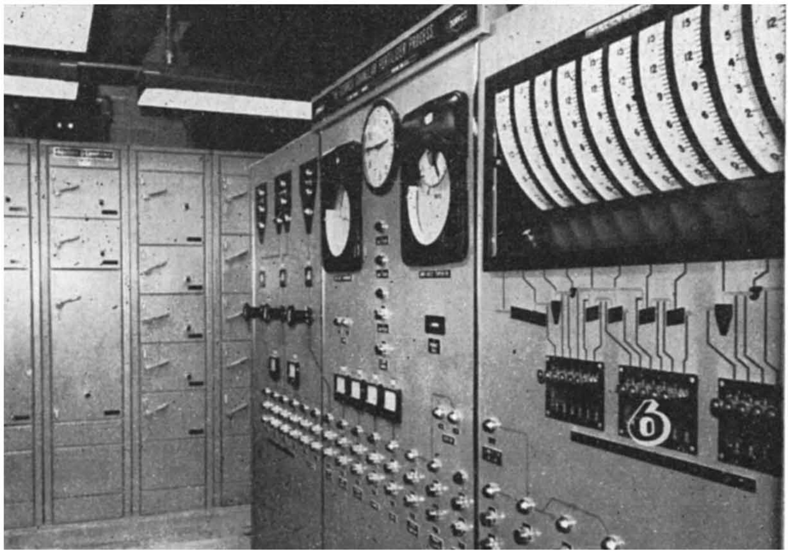

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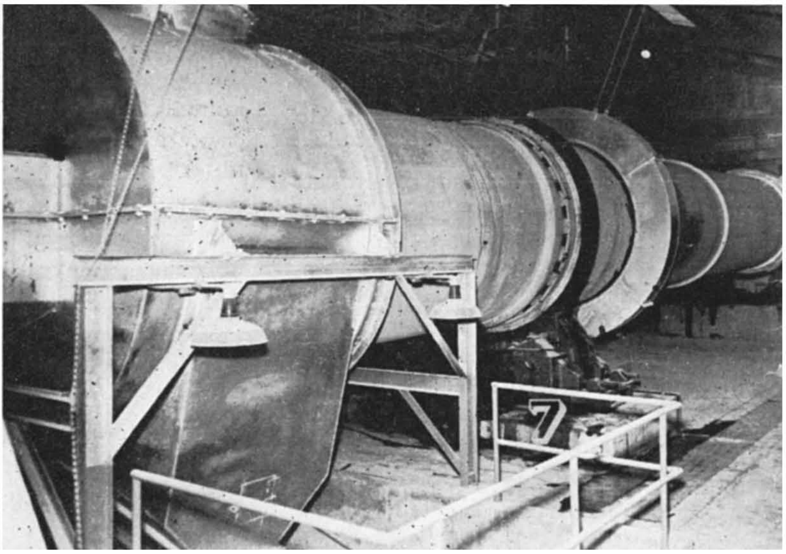

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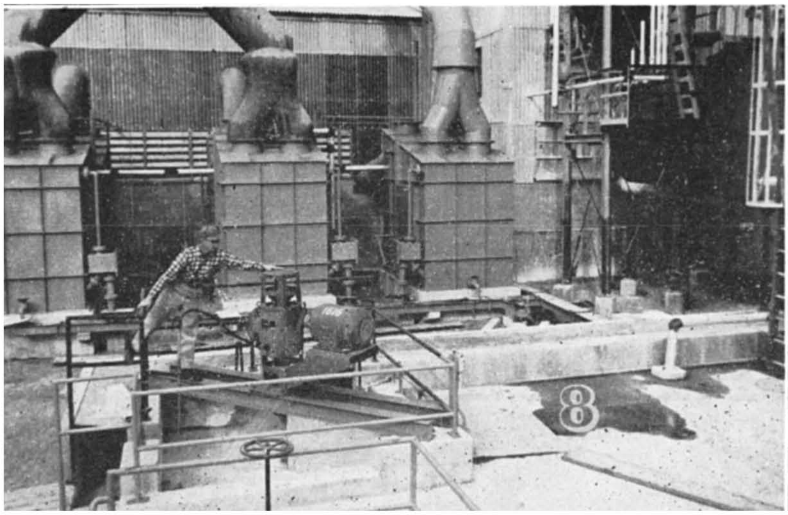

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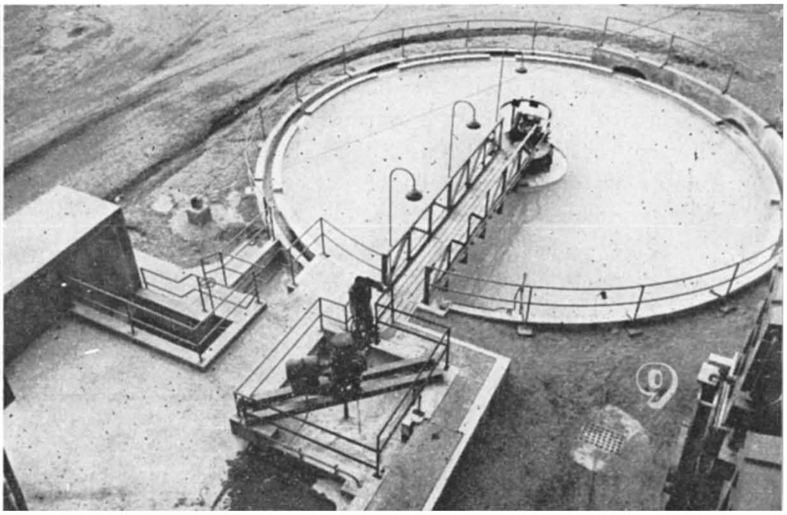

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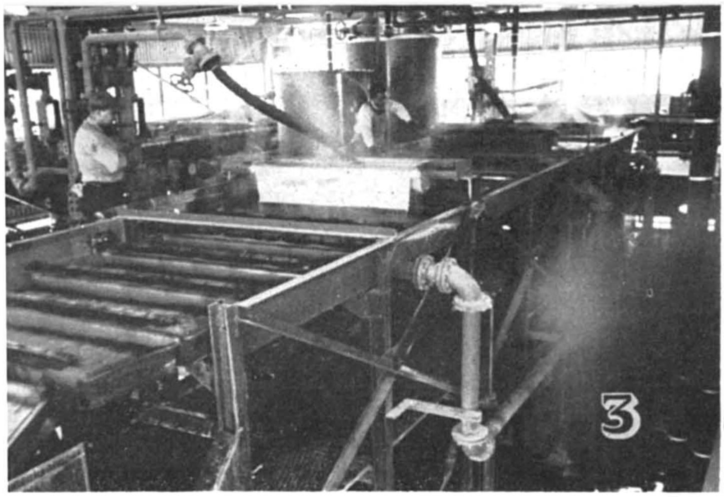


A DORR-OLIVER AMMONIUM PHOSPHATE PLANT

(1) Birdseye view of the plant. The sulfuric acid pipeline can be seen running parallel to the railroad track.

(2) Phosphoric acid production section — line of agitated reaction vessels

(3) A traveling pan filter in the phosphoric acid production section.

(4) The ammonium phosphate fertilizer section, a line of agitated reaction vessels.

(5) The blunger, where pellets are coated with ammonium phosphate slurry in the ammonium phosphate fertilizer section.

(6) The blunger control station of the ammonium phosphate fertilizer section.

(7) The ammonium phosphate fertilizer section's eight by 72 foot drier.

(8) Impingement type wet scrubbers in the background. In the foreground, a flash mixer for process water effluent and milk of lime.

(9) Process water treatment plant flash mixer and clarifier.

(10) Storage building in the new Dorr-Oliver plant.

THE Missouri Farmers Association's new fertilizer plant at Joplin, Missouri, is a high analysis pelletizing unit with annual capacity of seventy-thousand tons of complete fertilizers. Principal chemical compounds manufactured are phosphoric acid, ammonium phosphates, and ammonium sulfate. The phosphoric acid is produced by the wet process.

The plant, which went on stream November, 1954, was designed by the Dorr-Oliver Co., Stamford, Conn. Merritt-Chapman & Scott Corporation of New York were the constructors.

By Walter R. Horn

Missouri Farmers Association
Joplin, Mo.

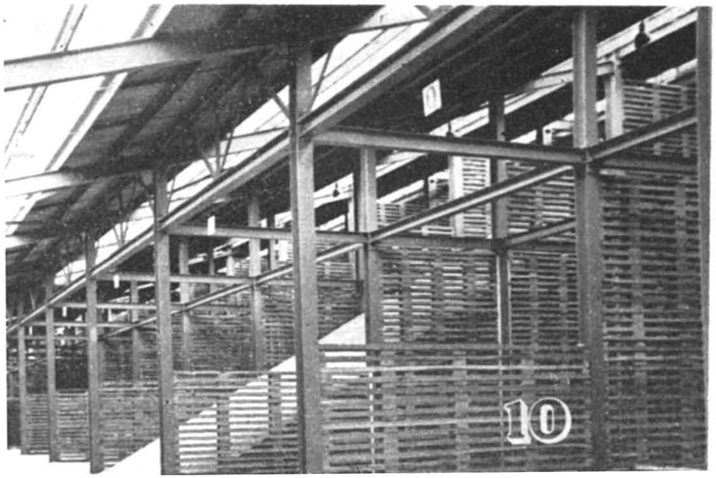
The plant consists of six primary sections. These sections are as follows: Rock Unloading and Grinding, Phosphoric Acid Production, Fertilizer Production, Product Storage, Bagging and Shipping, and Water Treatment.

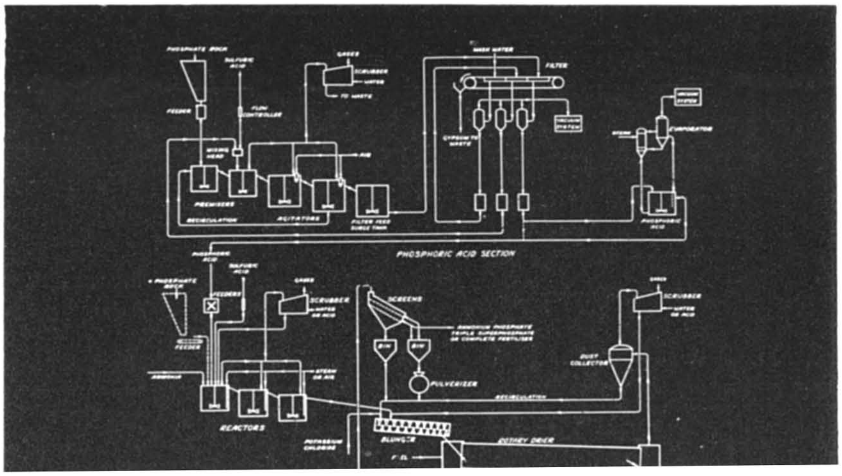
Rock Unloading and Grinding

THE rock unloading and grinding section consists of the necessary conveyors, silos, grinding mill, and a dust pump. Belt conveyors are used exclusively. The silos are constructed of glazed tile. The grinding mill is a 66-inch high side roller mill. The rock is ground to 60% minus 200 mesh. The dust is conveyed to either the phosphoric acid production section or the fertilizer production section through a pipe in an air stream by the dust pump.

Phosphoric Acid Production

THE ground phosphate rock, a weak phosphoric acid which is the second filtrate from the filters, and sulfuric acid are reacted in rubber lined vessels with violent mechanical agitation. The reaction products are phosphoric acid and gypsum. Sulfuric acid, (66° Bé), is conveyed





through approximately 4,000 feet of regular steel pipe line from the new Eagle-Picher zinc concentrate roasting and contact-sulfuric acid plant at Galena, Kans. The acid is pumped across the state line from Kansas to Missouri. The slurry resulting from the reaction of the rock, the weak phosphoric acid and the sulfuric acid is pumped to the filters, where the two reaction products are separated, and the gypsum is washed continuously and countercurrently before it is discharged from the filters. The gypsum is then slurried with water and pumped to the disposal pond.

The vacuum filters are known as traveling pan filters, which very aptly describes them. The filter consists of a number of stainless steel pans fastened to a rubber conveyor belt. Slots in the pans match open slots in the belt so that the acid which drains from the slurry and through the filter cloth in the pans enters the receiving system through these slots.

The first filtrate from the filters is pumped to the storage tanks where the phosphoric acid is either concentrated before using in the fertilizer section for the production of ammonium phosphates, or it is concentrated for shipment as such. Two single effect evaporators, using steam and operating under a very reduced pressure, are used for concentrating from 32% P_2O_5 to various concentrations up to 55% P_2O_5. This is equivalent to 76% ortho-phosphoric acid. The use of the Dorrco recirculation system minimizes the difficulty connected with the precipitation of calcium sulfate and various fluosilicates on the sides of the flash chamber, and the tubes of the heat body. Two-stage steam jet ejectors are used with each evaporator to maintain a reduced pressure of about 1.5 psia.

Ammonium Phosphate Fertilizer

THE main equipment of the fertilizer production section is the reaction agitators where anhydrous ammonia is added to phosphoric acid and to mixtures of phosphoric acid and sulfuric acid to make monoammonium phosphate, diammonium phosphate and ammonium sulfate. Other essential equipment in this section is a pelletizing unit, large swing hammer pulverizers, a package water tube boiler, a large rotary drier, an acid tower, and impingement type wet scrubbers.

In this section, a variety of high analysis fertilizer grades are produced by varying the relative amounts of monoammonium phosphate, diammonium phosphate and ammonium sulfate. Various ratios of $N:P_2O_5$ can be produced such as $1N:4.5P_2O_5$ with monoammonium phosphate, $1N: 2.5 P_2O_5$ with 20% mono-80% diammonium phosphate mixtures, and ratios very low in P_2O_5 such as $1N:1P_2O_5$ with ammonium phosphate-ammonium sulfate mixtures. The popular 11-48-0 and 16-20-0 can be made. In addition to these two, the following grades are produced: 19-19-0, 19-38-0, 13-39-0, 16-48-0, 14-14-14, 14-28-14, 12-36-12, and 10-20-20.

The reaction of the acids and the anhydrous ammonia results in a great deal of heat. Temperature is controlled by the evaporation of moisture in the slurry, and supplemented with low pressure air when necessary. The agitated reaction vessels are vented through an acid tower. Dilute sulfuric acid is circulated through the tower to remove any escaping ammonia from the vapors. A portion of this circulation is continuously drained to the reaction system and makeup sulfuric acid is added.

The slurry is withdrawn from the third reaction vessel to a mechanical mixer with two horizontal shafts, each fitted with intergeared paddles. This piece of equipment is called a blunger. The hot slurry meets a large excess of pelleted product. A size separation of this product has just taken place on double deck Tyler Hummer vibrating screens. This product to the blunger is principally the oversize and undersize from the screening operation. The oversize is first sent through a hammer mill. Mixing of the slurry and the fine pellets is rapid. Each pellet receives a coating of the slurry resulting in the general build-up of particle size. The material gravitates from the blunger to the rotary drier.

In the drier the thin coating is dried and the pellet hardened. Each pellet receives a number of coatings by repeating the cycle until it is of proper size to be separated by the screens as product. Each pellet is built-up of multiple coatings. The flow of product and gasses in the drier is concurrent because temperature is critical.

From the drier, the product is elevated to the screens where the size separation takes place. The production amount of the product size goes to storage, and the remainder with the undersize and oversize repeats the cycle by returning to the blunger.

Product Storage
Bagging and Shipping

THE product is carried by overhead covered belt conveyor to the product storage building where the material is transferred to a reversible shuttle conveyor for discharge to any of the fifteen bays. The bays are separated by bookshelf partitions. In addition to fifteen bays for product storage, there is a bay for the storage of potash. The capacity of each bay is about one thousand tons. Transfer of the product from storage is by scoop trucks to two continuous bucket elevators. Each elevator has in closed circuit a double deck vibrating screen and a cage mill. The screened product gravitates to two conveyor belts which move the material across the plant trackage to the bagging and shipping building.

Fertilizer is shipped, bulk or bagged, by boxcars or trucks. Valve packers bag the product in 80 pound bags. Fork-lift trucks are used for conveying the bagged material from either the packers or from the adjustment bag storage.

Water Treatment

THE total demand for water is approximately 1200 gpm. Most of this demand is within the process itself. For practical and economic reasons, it was decided to have a water treatment plant, and thereby reduce the raw water intake to about 350 gpm. The principal equipment in this section is a lime slaker, milk of lime feeder, a Dorrco clarifier, and a redwood cooling tower.★★

developments in—

Granulation and Ammoniation

by Elmer C. Perrine

Nitrogen Division,
Allied Chemical & Dye Corporation

DISCUSSION of the development of many industries reaches far back into their history for some foundation of understanding. When arranging developments in order of importance, one realizes that often events which appeared as trifles in original concept may later have a profound influence on an industry.

Although the granulation of fertilizer covers nearly a generation, some of the main barriers to progress have been breached only recently and other serious obstacles remain. Granulation is very closely associated with ammoniation. Among other things, the successful continuous ammoniator has been a very important link in the long detour around the problems of converting from even good batch ammoniation to the subsequent stages of granulation which are best performed continuously. As long ago as 1938 a batch practice of granulation was greatly improved when the ammoniating medium was distributed evenly enough to about double the ammonia take-up, thereby imparting more heat to the mass at an early stage in the process. This early heat is accepted as a very important feature today.

The basic simplicity of many systems has probably limited development because passable results are obtained through the bare-handed skills of the better operators. The conspicuous contribution that manual skills have made to the industry has given rise to the statement that granulation is more an art than a science.

As we welcome the high promise of science and technology, we still recognize the great importance of the skilled operator who will long remain the best ally the technician has. A few days struggle with even the most instrumented equipment will demonstrate the value of skilled operating personnel. As in most good things that come to stay, science and technology are rapidly entering the industry.

The attaining of desirable physical condition in fertilizer was the original justification for granulating and this is still a very important reason. The trends in farming and the just demands of the consumer have increased the problems of fertilizer manufacturing. It is interesting to conjecture over the status of the industry if more effort had been expended to improve the machinery for fertilizer application. As fertilizer is further improved, the limitations of application equipment will be brought more into light, and some relief from the troubles over condition of fertilizer may be forthcoming from this angle. Beyond a certain perfection in a product, further improvements in the end results are often more economically achieved through improved means of using the product. Attempting to solve all of the problems of applying fertilizer by granulating it may prove to be quite costly.

Some practices that had their origin in local usages are being rather widely adopted in the industry. For instance, at present prices it is economically justifiable in some cases to add to 1,000 pounds of normal superphosphate as much as 30 pounds of ammonia, through nitrogen solutions, beyond the point of 100 percent retention, knowing that only some 20 of these additional 30 pounds will be retained by the (normal) superphosphate. This calculated loss is fairly predictable and is sometimes referred to as excess ammoniation. It is quite frequently practiced in rotary batch mixers. Some operators are actually doing this in rotary batch and in continuous ammoniators without recognizing it. Any gain in ammonia take-up beyond these figures in present equipment is made at the cost of so much lost ammonia that the use of acid is often resorted to. Thereafter the trend is toward excess acid rather than excess ammonia.

The inadequacy of equipment and skills in some cases has resulted in the use of as much as 75 or 80 pounds more sulfuric acid than is indicated by the ammoniation requirements alone. Under these circumstances, and in equipment that is marginal at best, it now seems probable that much of the granulation was dependent on the use of large amounts of sulfuric acid.

We now observe that acid serves an important function in granulation beyond those of ammonia takeup and heat.

It can become costly, however, to resort to great amounts of any acid to perform functions that can also be performed by mechanical equipment, low cost fuel, or through control of the chemical and physical actions of the normal ingredients of the formulae.

Granulation and drying are greatly simplified when the mass enters the dryer at 200 degrees F or higher, but such temperatures are difficult to reach in the ammoniation and more difficult to hold for any time with the moisture content at a desired level. This is a potent statement for it justifies large amounts of ammonia and acid, it questions any delay between mixer and dryer and endorses the new commercial practice of using steam or hot water as the source of added water. Enough steam would be required in many cases that the local or state laws or insurance regulations would demand that a licensed engineer be in attendance.

One very important development is the realization reflected in later design and operation that regardless of the processing ahead of the dryer, the volume of acceptable granulation through the dryer is substantially lower than when the task is that of merely removing the moisture. This fact has been painfully forced upon equipment manufacturers and operators alike. Of the many efforts to increase the effectiveness of the dryer in granulation, the most obvious one of raising the heat in the dryer is less rewarding than some others which become quite involved. Even though there may appear to be quite complete granulation before the material enters the dryer, the need for drying in such a manner as to retain this condition does place a load on the dryer above its mere moisture-removing capabilities.

Many systems, intentionally or from lack of any other provision, require that practically all forming of granules be effected in the dryer. Some systems further burden the dryer by requiring that it convert the wide pulsations of batch operation to continuous. This will reduce the effectiveness of the first 5 or 10 feet of the dryer's length. The extreme difficulty of delivering highly ammoniated batches of fertilizer through a sealed feed uniformly in a continuous flow to a dryer has added greatly to the popularity of continuous ammoniators. Much of the effectiveness of even these devices is too often lost through faulty delivery into the dryer, usually from large air leaks. In some cases for reasons of economy or lack of space, no combustion chamber is provided and some portion of the dryer is used to mix the cold air with the very intense heat of the burners. When all of these factors prevail in the same dryer, which may also be quite short, there is not much space or capacity left to influence granulation or to hold that which may have been started elsewhere.

Some dryers are now 60 or 70 feet long and regularly yield a quality product with several formulae at fair tonnage. Some co-current (parallel) flow dryers are so large that conservative tonnage is handled at such low temperature of product (120°F discharge) that no cooler is required and screens are present only as insurance against the system running amuck.

The indiscriminate recycling of fines as fast or as slow as they are made has caused untold misery, since any fluctuations throw the system into erratic performance. Even locally improvised arrangements for regulating the amount of fines that return to the process are very rewarding.

Quite a few systems employ two rotary dryers and two coolers with one continuous or batch ammoniator. Unless the dryers particularly are operated as individual units, serious consequences will quite probably result. After six months the operators of several of these systems did not know the screen size analysis nor the moisture content of the discharge material of each of the dryers, although good data had been collected on the composite product. As the product from one unit reached the conveyor belt, it was found to contain about twice the moisture of the product from the other dryer. The composite samples from the discharge end of the belt contained a fairly safe amount for the local conditions, but only if it had been uniform throughout the storage pile. Serious disintegration took place in the large pile.

At the cost of some tonnage there is a trend toward reducing the number of large particles in the finished product. Few plants now use a 3 mesh screen and some are tentatively going to 6 mesh, and 8 mesh is being considered in some cases as the largest size permissible. In the total screened sample some granulated fertilizer that contained only 1.5% moisture was found to have nearly 4% in the largest screened particles. This may contribute to bag setting, granule disintegration and to faulty performance through the farmers' equipment.

Most granulated fertilizers contain from 20 to 35% of their weight in the large particles that are subject to suspicion. The simple, direct approach of using less water to avoid forming the coarse particles usually results in excess fines, another source of trouble in making and in selling the product. The present practice of milling the oversizes is probably the most suitable, but the equipment for doing this will be quickly overloaded if its burden is greatly increased.

In one plant all ingredients were ground to quite a fine mesh ahead of mixing to control the final size of the particles. It is probably because several factors in addition to the size of the original ingredients have their influence on final particle size that this practice is not widely used.

High analysis goods are inviting serious consideration of phosphoric acid as far as it can serve a purpose. In the field not enough of this has been practiced to be conclusive regarding its influence on granulation. The evidence so far is that it does not detract from the effectiveness of the usual granulating system.

Fumes and dust are the subject of much work in the industry and they are as much a concern from the standpoint of economic losses as of immediate safety and air pollution. Some plants face the expenditure of nearly as much for dust control as was originally spent for granulation.

Fires in fertilizer mixers continue to be a serious concern although it is gratifying to learn of the success many have had in controlling them once it was realized that control is possible.

In batch operations considerable control of fires as well as of dust and fumes is obtained by adding the muriate of potash after any acid is added and neutralized by the ammoniating medium. Some manufacturers use anhydrous ammonia separately but in the same formula with nitrogen solution and acid. Here the acid in batch operation can be prevented from acting on either the muriate of potash or the ammonium nitrate by withholding them from the batch until the anhydrous ammonia has neutralized the acid. Thereafter the potash is added and ammoniation with the nitrogen solution may proceed in the normal manner.

Some things which now are barely discernible in the firmament will doubtless be recognized as important developments perhaps even before this group meets again. Of the developments discussed, some of the following crept inauspiciously into the granulating industry while others have enjoyed the fanfare of a visiting fireman.

Consideration of the characteristics of materials that influence the granulating potential including the formation in the mix of additional products such as through the use of acid and possibly variation of temperatures.

Improving storage and application properties through more uniform particle size with emphasis on reducing the amount of coarse sizes and more uniform moisture content. Coating is under consideration in some cases.

Continuous operations including recycling, sampling and ammoniation.

Excess ammoniation at a calculated loss of ammonia without the use of acid.

The use of acid beyond the requirements for neutralizing the uncombined ammonia for the added heat and other influences on granulation.

Control of fires related to the use of acid.

Fumes and dust involving economic loss as well as air pollution.

Refinements of control by personal skills and instrumentation, particularly in the double granulating units which use only one mixer and ammoniator.

Means for delivering the highly ammoniated mass to the dryer at maximum temperatures by direct delivery from the ammoniator to the dryer, if necessary.

The use of materials for purposes other than for plant foods such as acid, steam and hot water.

Provision for drying under conditions that are more favorable to granulation than has existed where the dryer has been operated almost exclusively to obtain maximum drying capacity.

Overcoming a Caking Tendency in GRANULATED FERTILIZERS

by Bernard Raistrick

Research Manager
Scottish Agricultural Industries
Edinburgh

OVERCOMING the problem of caking in granulated fertilizers by practical manufacturing procedures was the subject of a recent talk* in London by Dr. B. Raistrick, Director of Research, Scottish Agricultural Industries, Edinburgh, Scotland. A brief review of the main points of his address follows. Some of these findings may prove helpful to operating personnel in our own country.

The problem was analyzed along these lines:

A. Study the nature of caked fertilizers and the mechanism of caking by laboratory techniques.

B. Study the manufacturing processes. Assemble the facts of operation such as temperatures, rates, analyses, moisture, etc.

C. Investigate the possible use of additives.

Important findings from this laboratory approach:

(a.) *The cause of bridging or binding of particles.* The chemical causing granules to bind and cake is crystalline ammonium chloride. These crystals formed a bridge and occurred during storage by a recrystallization from traces of solution on surfaces. Careful studies proved that ammonium chloride tends to concentrate on the surfaces of granules (This has also been reported by other investigators, and confirmed by work at U.S.D.A., Beltsville, Maryland, V.S.) Explanation: saturated fertilizer solution diffuses to surface of granules during the drying process. Also, ammonium chloride has a tendency to "creep." To prove this, one of the analyses having a strong tendency to caking was extracted at room temperature with different amounts of water kept small so as not to completely dissolve any ion. The chemical analysis for some of the ions in the saturated solutions gave NH_4^+, 0.85, Cl^-, 0.41, K^+, 0.047, PO_4^-, 0.041 and SO_2^-, 0.23 gram. And ammonium chloride needle crystals formed in great volume from such solutions.

(b.) *Humidity conditions inside a bulk pile.* 800 tons of a granulated 7.5-8-10 fertilizer with average mois-

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*Proceedings No. 38, The Fertilizer Society, London, W.C.I. This paper was condensed for *Agricultural Chemicals* by Vincent Sauchelli.

ture content of 3-4%, were stored in bulk in an area 50′ x 50′, height about 13′. A metal tube extended from the middle of pile to the surface. The tube was used to withdraw air from the pile and measure its temperature. After five weeks, the temperature near center of pile was 25-28°C (77-82.4F) and the dew point was 20-22°C (68-71.6F). It was observed that water condensed wholesale in the apparatus outside the pile when air was drawn up the tube into the apparatus.

(c.) *Operating conditions fluctuated.*

In an 8-hour shift it was observed operating conditions could vary widely. Example: temperature of product coming out of co-current drier varied from 76° to 98°C (168.8 to 208.F) with one sudden increase to 128°C (226°F) and moisture content ranged from 0.8% to 4.6% or more. (Moisture content determined by drying at 100°C in a fan equipped oven for 4 hours.)

(d.) *Additives gave poor results.*

Additives were tested at ¼%, 1% and 5% included magnesia, Fullers earth, talc, lime, diatomaceous earth, dicalcium phosphate and aluminum stearate.

At moisture contents of 2 to 5% range, the additives did not prevent caking. With a moisture content below 2%, the fertilizer was less hard especially when kept apart from a higher moisture product.

(e.) *Influence of ammoniation in caking.*

Ammoniated at rate of 2.2 lb. NH_3 per 100 lbs. super, fertilizer behaved similarly as to caking as non-ammoniated product.

At the end of these preliminary tests, Dr. Raistrick summarized his impressions: "caking was probably due to recrystallization forming ammonium chloride bridges between adjacent granules. This recrystallization occurred because of the presence of moisture, and maybe because at pressure points (and fertilizers set hardest when under load) salts will dissolve in otherwise saturated solutions and deposit at points of lower stress."

f. *Relation between vapor pressure and caking tendency.*

Moisture content of fertilizer is the most important single factor in determining caking behavior.

Moisture studies:

> Decided that most reliable and convenient method of expressing moisture activity is to measure the water vapor pressure over the granules and to express it as relative humidity. This relative humidity was then correlated with propensity to cake. Devised special method to measure the humidity of the air in equilibrum with fertilizer.

After much study and testing, it was decided that "an immediate solution to the problem of caking of NPK fertilizers involved reducing the relative humidity to less than 30% and this in turn meant reducing the moisture content of normal products to about 1% or below. . . . The exact safe maximum moisture content varies considerably with composition, and especially with superphosphate content: for example, 12-4-12 has a low super content and therefore needs more thorough drying. It is because of the effect of this variation in composition that relative humidity was chosen as a better guide to safety than moisture content. The moisture limit is in the neighborhood of 1% when the super content of the granules is in the range of 30-50% and is higher or lower when the super is respectively above or below this range.

(g.) *Influence of fines on caking tendency.*

Belief is current that a high fines (or dust) content leads to bad caking properties. Experiments showed that, in their case at least, this belief is not warranted. Good screening alone is no answer to the caking problem. Other good reasons exist of course for removing fines.

(h.) *Influence of temperature on caking.*

Belief that efficient cooling of granules is essential to prevent caking is not substantiated by tests. Large-scale tests were made to test this belief. If moisture content is kept low, cooling has no influence. If moisture content is high, cooling does not prevent caking. Other excellent reasons exist however, for cooling product prior to bagging.

(i.) *Influence of condition of paper bags on caking.*

Condition of paper bags for packing becomes of great importance in the prevention of caking during storage. Punctures of the bituminous ply or cracks in this interlayer caused by rough handling of filled bags induced lumps of caked product around these weak spots in the bitumastic interlayer.

(j.) *Thorough drying on full scale plant.*

By the end of these several series of long time tests, faith was strong that thorough drying is the means of preventing caking of all fertilizers all the time, and this means the *whole production* has to be dried to below 1% moisture content: it is not enough that *the average* moisture should be below 1%. To assure success it is absolutely essential that the process operation be maintained under steady conditions with a minimum of stoppages. A thoroughly dried product was produced by reducing through-

Moisture content most important single factor!

Condition of paper bag an important consideration.

Thorough drying one means of preventing caking.

High fines content does not lead to caking.

Efficient cooling not essential to prevention.

put, by subjecting the plant to one control overriding all other, as for example, maintaining a steady ex-drier temperature of solids of about 120-125°C (248°-257°F).

Observation: overwhelming evidence that moisture contents were often very high or very low for the hour or two before and after breaks in production. For complete success in producing material of low moisture content and to produce it consistently: occasional jumps in moisture due to changes in operating conditions may not be tolerated.

Previously mentioned throughput was reduced. However, it is realized that a regular operation can not stand a reduction in throughput while burning the same amount of fuel as a means of producing low-moisture fertilizer. Means had to be found of reducing thermal load or drier to offset increased load on drying to ½-1% instead of 2-5%. This was accomplished by reducing the amount of fines recycle through an improvement in the efficiency of granulation. Fines put a heavy load on drier because they are re-wetted and have to be re-dried and they do not agglomerate easily, making granulation more difficult in proportion as fines increase.

The effect of fines is cumulative.

(k.) *Improvement of efficiency of granulation.*

Efficiency of granulation is important because it reduces the amount of fines to be recycled. Thus, reduction permits thorough drying, while maintaining high throughput rate and keeping drier capacity down.

Definition of efficiency: the percentage of material in the size range 1 to 3.4 mm. (5-16 mesh B.S.S.) and applies to material coming out of drier, that is, prior to screening.

(l.) *The gaseous effluent.* The problem of atmospheric pollution can be divided into 3 headings: I fume; II dust, and III acidity and acridity. Dust and acidity can be handled to some degree by a water irrigated tower; fume is very difficult to reduce, and is believed to be the worst offender. Fume consists chiefly of sub-micron size ammonium chloride particles — ammonium fluoride and hydrogen chloride are probably present also.

Since high drier temperatures are involved in drying fertilizer to low moisture, volatilization of ammonium chloride will increase.

Ammonium chloride begins to volatilize in appreciable quantities at 180°C (365°F). Fume could be formed from the particles of fertilizer dust suspended in hot drier gases, such as exist at inlet to drier. Some ammonium salts could conceivably be volatilized from over-heated material on the drier feed chutes of co-current driers.

Using a 1500 c.f.m. Venturi scrubber achieved more than 99% chemical efficiency, and about 75% visual efficiency in the removal of fume.

It is possible also to volatilize ammonium salts at the fairly low temperature caused by the heat of reaction between ammonia, sulfuric acid, muriate of potash and superphosphate.

Final comment.

Dr. Raistrick does not claim he has the answer to the caking problem for all plants. He does claim that by adhering to the 30% relative humidity in the drying specification they have produced 400,000 tons of non-caking granulated fertilizers, and this was accomplished at all times. After choosing this specification, it was up to process development and process control to work out the ways and means. "The granulation plant," he adds, "must be operated continuously in balance, with a minimum of stoppage, both planned and unplanned, which might cause deviations from the chosen conditions. The characteristics of the raw materials, and the quantity of granulation water used must be controlled as far as possible to give steady operating conditions."★★

SAMPLING of FERTILIZERS and MATERIALS

By J. A. Archer
International Minerals & Chemical Corp.

DURING the last few years, the demand for high analysis regular, semi-granular, and granular fertilizers has greatly increased, and thus quality control, of which sampling and analysis is an important part, is becoming increasingly important as the materials used and mixtures produced become more concentrated.

It is important that the operator know, at all times, the composition of his raw materials, base piles, and the composition of his finished product. The composition of his raw materials base pile is determined by three consecutive steps, namely: the taking of samples, their preparation and their analysis.

The analysis of an improperly drawn or improperly prepared sample is worse than no analysis at all, since the analysis of such a sample is mis-leading. Therefore, it is necessary that the man who is assigned to the job of taking and preparing samples be fully trained, and that his sample room be equipped with the necessary sampling tools. Some typical sampling instruments are described below:

SPECIAL SAMPLING CUP

Base production of regular or granular goods can best be sampled with an automatic sampler. However, if the automatic sampler is not available, a representative sample can be taken with this special sampling cup. The sample is taken by passing the special cup through the stream of material as it drops from a transfer belt. The cup should be passed in a direction parallel to the head drum and should cut the entire stream. The cup is useful also for sampling superphosphate which is shipped in bulk, and for sampling mixed goods which are shipped in bulk, provided the goods are moved to box car or truck by means of transfer belts.

The DOUBLE TUBE SLOTTED TYPE SAMPLER is commonly used for Sampling:

1. Bagged incoming raw materials
2. Regular or granular mixed goods shipments

TABLE 1.
Analysis Comparison—Exchange Samples

	State Laboratory Working Sample			IM&CC Laboratory Working Sample		
	Nit.	A.P.A.	Potash	Nit.	A.P.A.	Potash
Exchange Sample No. 1						
State Analysis	8.40%	21.74%	9.44% (1)	10.64%	20.34%	9.20% (3)
IM&CC Analysis	10.75	21.37	9.05 (4)	10.95	21.05	9.11 (2)
Exchange Sample No. 2						
State Analysis	10.84	20.89	8.51 (1)	8.46	21.73	10.30 (3)
IM&CC Analysis	8.50	21.88	10.10 (4)	8.90	21.07	10.08 (2)
Exchange Sample No. 3						
State Analysis	6.47	15.70	4.94 (1)	6.52	16.29	5.32 (3)
IM&CC Analysis	6.45	16.20	5.75 (4)	6.70	16.18	5.78 (2)

(1) Initial analysis reported by State.
(2) State furnished IM&CC an unground sample—IM&CC Analysis.
(3) IM&CC furnished State a portion of ground working sample (2).
(4) State furnished IM&CC with ground sample—IM&CC Analysis.

The TUBE SAMPLER is Suitable for Sampling:

1. Incoming bulk raw materials
2. Base production when conveyed to storage in bulk
3. Regular mixed goods shipped in open mouth bags
4. Bulk mixed goods or superphosphate shipments

MODIFIED SOIL AUGER

The sampling of a free flowing granular stock pile is somewhat of a problem, due to the fact that the goods will not stay in the sampling instrument. This problem can be largely overcome by using a modified soil auger. The modification simply consists in adding a shaped steel plate to one side of the auger to retain the sample when the auger is withdrawn from the pile. It is well to have an indicator mark on the handle of the auger so the sample man will know the position in which to hold the auger when it is withdrawn from the pile.

SAMPLE CONTAINER

The sample man should have a supply of rugged, light weight sample containers. Sketch (following page) shows an inexpensive container which we have used for some time. We find it is well received by the plant people. It is constructed of ½″ plywood with suitable cover and carrying strap. The container is 10″ wide, 20″ long and 8″ deep, and holds about 4 gallons of sample.

SAMPLE PREPARATION TABLE

The sample man should have a convenient work table for preparing the samples. Shown (next page) is a very useful inexpensive sample table. It is ruggedly constructed. The table has a smooth masonite top 7′ x 3′6″, and a plywood chute at the end of the table. The chute is used for disposing of sample rejects. This is a convenient feature of the table and is an aid to the sample man in keeping his room clean.

TABLE 2.
Analysis of Plant Granular Samples Reduced to Laboratory Size by Different Methods
(Plant Size = approximately 3 gallons)

		Plant sample reduced to ½ pint size by riffling method			Plant sample reduced to ½ pint size Quartering Method			Plant sample reduced to ½ gal. size by riffling method Then ½ gal. sample reduced to ½ pint size by Quartering Method			
		Column No. 1			Column No. 2			Column No. 3			Average of columns 1-2-3
		Sample No. 1	Sample No. 2	Var.	Sample No. 1	Sample No. 2	Var.	Sample No. 1	Sample No. 2	Var.	
	N	9.49%	9.53%	.04	9.17%	9.45%	.28	9.29%	9.45%	.16	9.40%
10-20-10	APA	20.59	20.70	.11	21.37	20.93	.44	21.15	20.37	.78	20.58
	K_2O	10.30	10.37	.07	10.00	10.20	.20	10.08	10.42	.34	1023
			Ave.	.07		Ave.	.27		Ave.	.43	
	N	14.70	14.80	.10	14.00	14.52	.52	14.95	14.60	.35	14.60
15-15-0	APA	17.49	17.30	.19	18.29	17.60	.69	16.78	17.28	.50	17.46
	K_2O	—	—		—	—		—	—		—
			Ave.	.15		Ave.	.61		Ave.	.43	
	N	10.20	10.20	.00	10.35	10.35	.00	10.10	10.30	.20	10.25
10-10-10	APA	11.12	11.54	.42	11.23	11.26	.03	11.46	10.89	.57	11.25
	K_2O	8.64	8.65	.01	8.95	9.03	.08	8.62	9.10	.48	8.81
			Ave.	.14		Ave.	.04		Ave.	.42	

Stock Pile Sampling

Now let us look at the problem of stock pile sampling. Shown in figure 5 as sketch 1 is a cone shaped pile. The pile should be sampled by drilling 2 holes equal distance apart around the pile, in section B, and by drilling 6 holes equal distance apart around the pile in section C. It can be shown mathematically that if a pile is sampled as indicated, the resulting analysis of the sample should be representative of the entire pile or of the face section of the pile, depending on the depth to which the sample holes are bored.

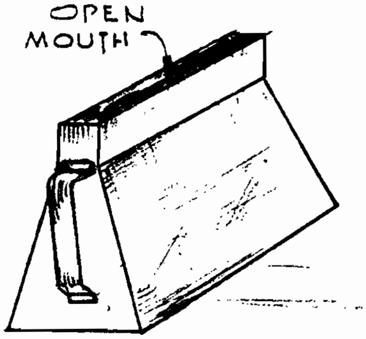
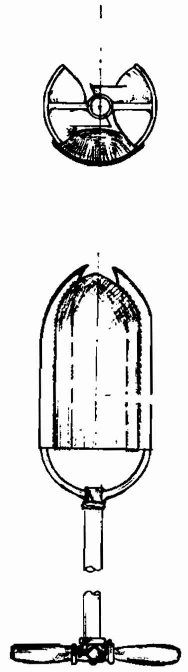


Fig. 1



Left: Modified Soil Auger

Fig. 2

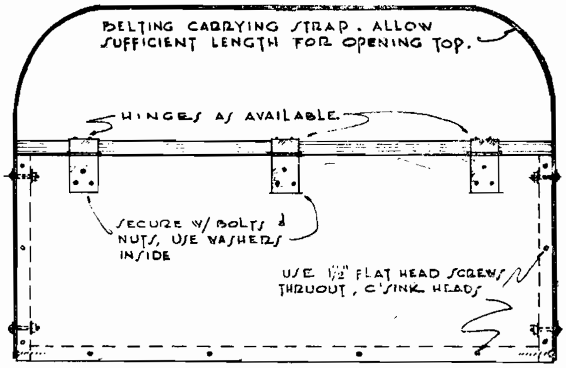


Fig. 3. Sample Container

A number of tests were made in an attempt to prove the above method of stock pile sampling. The tests were made by taking a sample from a shovel as the pile of superphosphate was moved to base manufacture. These tests indicated that the analysis of the sample taken from the tractor varied in A.P.A. analysis ½ to 1% from the analysis of the sample taken from the pile. The comparison indicated that the analysis of the sample taken from the shovel was more correct than the analysis of the sample taken from the pile. Therefore, there is a definite need for developing a method for stock pile sampling.

Comparative screen analyses of samples taken with a 1" diameter and ⅝" diameter Indiana type slotted, tubes were made on 12-12-12, 10-10-10, 5-20-20, 4-24-12, 5-10-15 and 5-10-10. In all instances, except possibly the 4-24-12 and the 5-10-10, samples taken with the smaller diameter tube contained too great a proportion of fines. Since all size particles do not necessarily have the exact same analysis, this data indicates that the ⅝" tube which is commonly used may not be large enough to sample granular fertilizer. Therefore, the analysis of a sample taken with the ⅝" diameter tube may be misleading.

Comparison — Exchange Samples

THE data presented in Table 1 indicate there is a problem in dividing a granular sample. Looking at the data—exchange sample No. 1 (1) is the initial analysis reported by the state. You will note that the nitrogen report is 8.40%. On the other hand, the IM&CC laboratory analysis of a sample which the state furnished indicated 10.95% nitrogen. This was a granular sample and the sample sent us was unground. A portion of our ground sample (2) was sent to the state and the state reported 10.64% nitrogen (3) which confirmed our findings. In view of these variances, we asked the state to furnish us with a portion of a ground sample and the analysis of this sample is reported as (4) with the nitrogen well above 10.00%. Exchange samples Nos. 2, 3, 4, 5 and 6 show somewhat similar results, but perhaps not as great as shown under exchange sample No. 1. I believe that it is necessary to grind a granular sample before it can be reduced in size and divided.

Analysis of Plant Granular Samples Reduced to Laboratory Size

IN view of the results of the exchange samples reported, it was decided to make a study of the various methods of reducing the size of

(*Continued on Page* 20)

Fig. 4. Sample preparation table

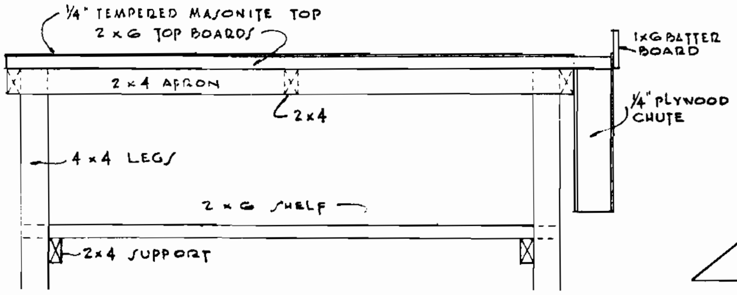
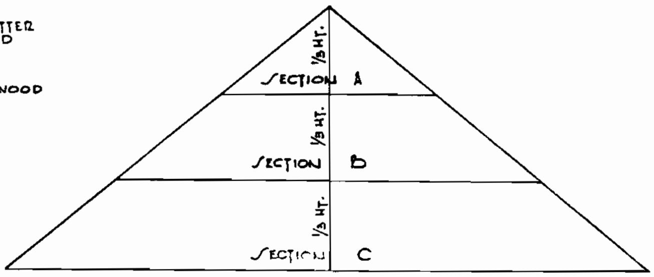


Fig. 5. Stock pile sampling



a granular sample. The analysis reported in column 1 (Table 2) represents the analysis of a sample of about a 3 gallon size which was reduced to ½ pint size by the standard riffling method. The results under column 2 represent the analysis of a 3 gallon size sample reduced to ½ pint size by the standard quartering method. The results under column 3 represent the analysis of a 3 gallon size which was reduced to ½ gallon size by riffling, followed by quartering the ½ gallon size sample to ½ pint size. The work was in duplicate and the variance between duplicate sampling and analysis is indicated in column under *variance.* This shows a grand average variance of .12 for the riffling method, a grand average variance of 0.32 for the quartering method and a grand average variance of .31 for combination of the two methods. The results shown under columns 4, 5, and 6 are the analyses of samples prepared as in columns 1, 2, and 3, except the entire sample was ground 85% to pass 20 mesh before the size of the sample was reduced. It is observed that the variances in these samples is much less than the variances of the samples which were reduced without grinding. In fact, the variances on these samples are well within the limits of the analytical accuracy. The results reported in this chart indicate that if a sample is first ground its size can be reduced by riffling, quartering or a combination of the two methods.★★

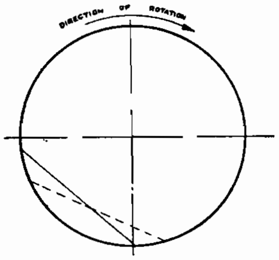
GRANULATION of MIXED FERTILIZERS

by Vincent Sauchelli

Davison Chemical Co.
Baltimore, Md.

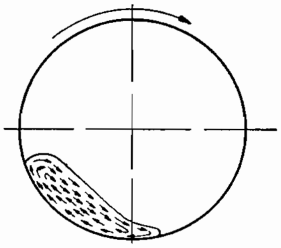
A MOST interesting set of experiments on the control of the size of granules in the manufacture of granulated mixed fertilizers was described by A. T. Brook (Messrs. Fisons Ltd., Great Britain) at the Sept. 24-26, 1956, I. S. M. A.* Conference, held at Lausanne, Switzerland. Some of the highlights of this investigation are given in the following all-too-brief digest of the presentation.

The research attempts to relate the results of the batch, pilot-plant unit with continuous granulation in full-scale, rotary tube granulators. The fertilizer materials used in formulation comprised normal and triple superphosphates, sulfate of ammonia, and muriate of potash — all requiring the addition of water for granulation and hence subsequent drying to effect hardness in the granules.** The workers realized that there is a wide difference in the characteristics of a full-scale continuous operation and those of the experimental batch units. However, they also realized that owing to the many variables involved in this kind of study it was not practicable to conduct the investigation with commercial units.



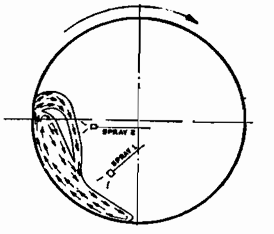
SEE-SAW MOTION WITH MASS RELATIVELY STATIC.

FIGURE 1.



ROLLING MOTION WITHOUT CASCADING DUE TO LOW SPEED OF ROTATION.

FIGURE 2.



IDEAL CASCADING MOTION RESULTING FROM CORRECT SPEED OF ROTATION.

FIGURE 3.

First essentials

a. Raw materials should be uniform as to particle size and quality; for example, moisture content. It is not possible to get a product of good quality if the mixture varies in constitution from minute to minute.

b. *Segregation*—Granulation by addition of water starts as soon as the mix is placed in the test granulator. In commercial practice, however, handling of the materials after mixing and before they reach the granulator results in some segregation, the amount depending upon the size range of the raw materials. Segregation may also occur in the granulator when tumbling begins and before the water is added.

c. *Uniformity in rate of flow* In a commercial unit, water is added at a more or less uniform rate of flow at one or more fixed points. To insure uniform wetting, the rate of passage of the mixture beneath the sprays should also be uniform. Otherwise, some portion of the materials may become more wetted than adjacent portions and become lumpy and less mobile due to its failure to move as fast as it should. Thus it will form a sort of weir at that point and get still wetter.

Finally, the material behind

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*International Superphosphate Manufacturers Ass'n Conference, Sept. 24-26, 1956.

**The British fertilizer industry does not utilize ammonia solutions to ammoniate superphosphates as is customary in American practice. V.S.

it builds up a head to a size so that it is able to push the temporary obstacle away. The subsequent rate of flow of the dry material is temporarily increased. Gradually normal flow becomes re-established. But meanwhile, the harm has been done, in that some portion of the mix has been overwetted, some not enough and the result is oversize and fines. The same situation may arise if the raw materials have not been thoroughly mixed. The result here is non-uniform progress through the granulator tube.

d. *Uniform wetting*
Materials having very fine particles may suffer from "slippage," that is, the material refuses to be "gripped" or lifted by the rotary action of the granulator. What develops is described as a see-saw motion of the mass, up and down the shell, but in which the constituents of the mass are relatively at a standstill. If the bed of material in the tube becomes static or stops cascading uniformly beneath the spray, some parts will become wetted more than others with the result that granulation suffers. If the tube does not turn rapidly, it will produce a rolling motion minus the cascading effect. Hence, we see how interdependent are uniform rate of flow and uniform wetting in a continuous tube granulator.

Correct rolling in the tube

Wetted fertilizer materials in a tube granulator tend to stick in the corners of lifters. After building up for some time they will often fall away in large lumps, again promoting oversize. The larger the tube the less the centrifugal force and lifting action for a given peripheral speed. Hence, it is that a small diameter shell rotating at high speed and having ample inclination favors the wetting process more so than a shell of large diameter rotating slowly, which produces a deep bed of material and prevents the uniform wetting of all particles.

Some observations based on experiments in a batch granulating test unit

The tests were run to determine the effect on granulation and granule size of the following factors: speed of rotation of drum; growth of granules in drier; time of retention in the granulator; water requirements of different mixtures; particle size of sulfate of ammonia; addition of fines to raw materials; sprays versus jets for addition of water; and positioning of water sprays.

Because in the United Kingdom fertilizers based on superphosphates are sold on their water-soluble P_2O_5 content, manufacturers there allow the superphosphate to "cure" for at least 2 weeks before using it for granulation. As a consequence, the super is cold and less plastic, but it does attain its maximum water solubility which is not affected by subsequent granulation and drying if temperatures are kept reasonably low. Continental practice is to granulate with super fresh from the den when it is hot, moist and plastic. It may not be necessary then to add water or to use heat for drying the granules.

a. *Speed of drum rotation*:

The speed of movement of particles has to be such as to distribute the spray water uniformly within the mass while producing sufficient shearing action to break down oversize particles.

It is believed that agglomeration of particles in a rotating tube follows definite laws and responds to correct conditions.

b. *Growth of granules in drier*:

Increase in temperature and moisture content will promote the agglomeration of particles in mixtures based on superphosphates. Granulation of cold, raw materials is accomplished by adding water with subsequent drying in a concurrent drier. The art of granulation requires experience and keen observation to enable the operator to judge when he has the correct degree of agglomeration ex-granulator. Differences in formulations will influence the growth of granules in the granulator; growth occurs also in the drier.

c. *Time of retention in granulator*:

If the rolling time in the granulator is varied, what effect does it have on granule formation? The results of numerous tests showed: granule size depended entirely on the rolling action in the drier and was independent of the amount of rolling obtained during the wet stage immediately following the wetting process, provided the spray water is correctly distributed during the wetting phase. The researchers found that a retention period of 2½ minutes in the granulator was adequate.

d. *Water requirements of different mixtures*:

The addition of water is one of the few controls used by the operator to regulate the size of granules. Most operators fail to appreciate the critical nature of this sensitive control and over-correct for water addition. Different formulations require different amounts of water. To illustrate how sensitive this control can be, the investigators cite this one among many examples: the initial water content of the 0-19.5-19.5 mixture was 8.25%; the size range of the final product was to be within the 1.5mm to 4mm size; the final granulated product obtained after adding 7.15%, 7.6% and 8% respectively of water showed efficiencies of 66%, 88% and 39% respectively; that is, for each of the water additions, the per cent of granules falling within the mentioned size range was as shown. In other words, the differences in the amounts of water added in this test represented only 10 gallons per hour on a 10-ton-per-hour plant basis out of a total addition of water of 170 gallons per hour, yet the efficiency in building up the desirable range in size of granules was so variable.

In another test using a 9-9-15 mixture with similar differences in the addition of water (or 1 gallon per ton of product), fairly wide variations in water could be made without significantly affecting the percentage of granules within the required range of 1.5 to 4 millimeters. This shows the influence of the superphosphate, the lower the proportion the lower the sensitivity to additions of variable amounts of water.

e. *Effect of particle size of sulfate of ammonia*:

The effect of particle size of sulfate of ammonia on granule size when granulating with cold water was found to be of major importance. The reasons advanced by the investigators for this effect are: fine soluble salts generally require more water addition for a given degree of agprove the rolling characteristics, with resulting improvement in range size of the granules. Tests with large particle size potash salt and superphosphate containing particles up to 2 mm size confirmed this observation.

It is essential that good rolling characteristics be obtained immediately after the addition of water. Raw materials having particles less than 1 mm size tend to slump at this stage rather than roll. Slumping prevents good agglomeration and reduces the efficiency of producing granules within the desirable range of 1.5 to 4 mm size.

f. *Adding recirculated fines to raw materials*:

It is common practice to return fines to the mix with fresh raw materials. This alters the size grading of the final mixture. The fines influence granulation in two ways: they reduce slightly the net amount of water required for granulation and they provide coarser particles to the raw feed which promotes better rolling in the granulator. Plant difficulties with fines were traced to *variations* in fines, that is, ratios of raw materials. To avoid the problems associated with recycling fines it was found necessary to mix these fines thoroughly with the raw materials and then control artificially the rate of addition to the raw materials so as to maintain a constant ratio.

g. *Effect of sprays versus jets for addition of water*:

In a few tests it seemed that using an open water jet in place of sprays improved the granulation. Further tests were made in the experimental unit to check on these observations. It was then found that the jet water increased the amount of oversize granules. Investigation in full-scale units confirmed this: where jets had proved superior to sprays, the spray had been placed too far from the rolling bed of material. This caused the spray mist to spread to the uncovered surface of the granulator shell. The rolling material tended to stick to the wetted shell surface and eventually it broke away in large lumps which formed oversize. By placing the sprays close to the rolling material, the spread of the spray mist to the shell surface was prevented, giving superior results over the open jet.

When the spray plays on the material which is cascading as it should, the water has a better chance to penetrate and distribute itself throughout the falling curtain of material (Fig. 3, Spray 2, p. 20.)

Comparison of Indiana Sampler, Slotted Single Tube and Riffle

By C. W. Schneider and M. D. Sanders
Swift & Co.—Plant Food Div.

As part of a general program of sampling mixed plant food shipments, some study was devoted to attempting to determine the relative effectiveness of several simple sampling devices that might be used for bagged goods.

4-80 lb. bags at equal intervals from shipments of 5 to 10 tons ea. (100 to 200 bags)
Grades sampled were 3-12-12, 4-16-16, 5-20-20, 10-10-10, 12-12-12, 0-20-20.
Products were semi-granulated; individual bags were sampled as follows:
Odd numbered bags were sampled, lying flat, twice, by probing opposite diagonals with a ¾" single tube slotted tryer inserted upside down and turned right side up before withdrawal. Each probe was handled as a separate sample.
Even numbered bags were similarly sampled in duplicate with a double tube, ¾" "Indiana Sampler."
After probing, each bag was split on a large riffle, each original half being split down to approximately 1-qt. size, providing duplicate samples from each riffled bag.
All samples so taken were ground to pass 20 mesh through a Mikro Sample Mill, quartered by riffling to 4 oz. and analyzed without replication.

Differences between duplicates with the same sampling device were computed and the standard deviations of these differences (s_d) were calculated. (The difference is always the first sample minus the second).

Application of the "F" test revealed that the only standard deviation significantly greater than the others by 95% odds or better was that of the single tube probe for potash.

Average Difference — Tubes vs. Riffle

To determine whether the average differences ($\bar{d}$) between sampling devices were signficant, averages of duplicates with a trier were compared with corresponding averages of riffle duplicates on the same bag with the "t" test.

	P_2O_5	K_2O
Single tube—riffle $\bar{d}=$	+.16	+.15
Double tube—riffle	+.10	+.18

(Values for nitrogen were obviously insignificantly different).

Two of the above values (+.16 and +.18) were significant at 95% or better odds. However, these average differences are not really of considerable magnitude.

The confidence limits dependent on a minimum of ten bags between

n = Number of Samples
Values of Standard Deviations (S_d)

	N		P_2O_5		K_2O	
Single tube	.144	n = 14	.220	n = 16	.360	n = 16
Double tube	.151	n = 14	.221	n = 16	.210	n = 16
Riffle	.196	n = 28	.159	n = 32	.189	n = 32

the various instruments, were calculated according to accepted procedure with the following results provided values from 10 or more bags were averaged for each device.

N & P_2O_5	Max. Diff.	Odds
Either trier vs. riffle	.5%	90%
	.25%	70%
K_2O		
Single tube vs. double tube or riffle	.75%	90%
	.4%	70%

This means, for example, that we would not expect differences averaged from 10 or more bags greater than .5% between either trier and riffle for N & P_2O_5 90% of the time. In one of ten such comparisons such a value might be exceeded.

Conclusions

1. Application of the "F" test reveals that the only standard deviation of differences between duplicates significantly greater than the others by 95% or better odds was that of the single tube probe for potash.
2. The average difference between the average of duplicates with one device and that with another device was significant in only two cases, and the magnitude for these was not great.
3. The maximum difference 90% of the time expected between the average analysis of 10 or more bags with one device and that with another device would be less than 0.5% for N & P_2O_5 and less than .75% for K_2O 9 times out of 10.
4. This study casts some doubt on the precision of the single slotted tube, but did not, of course, take into consideration different sized tubes or fertilizer other than that having a wide size range.

Economics of Automatic Sampling

By P. E. Cook

Denver Equipment Co.

TODAY practically all progressive companies recognize the importance of quality control, and have some type laboratory testing facilities.

Yet, regardless of the investment in buildings, equipment or technical personnel, the results of their testing can be no more accurate than the samples on which their tests are based.

If there is ever going to be the remotest connection between sampling, testing and economics, there must be a method of obtaining an accurate sample from which test results can be projected.

It is absolutely impossible to manually sample material in motion with any consistent degree of accuracy. Individual sampling techniques vary from day to day. Regardless of personnel training or standardized procedure, the sampling technique will vary from one individual to another. I want to emphasize, it is absolutely impossible to manually sample with any consistent degree of accuracy or reliability. The answer to this problem of "economics" in sampling is a mechanical sampler that will do what it is told to do, when it is told to do it and will do it in exactly the same way each and every time.

The purpose of sampling is of course to secure a small amount of material representative of the entire larger amount. Material which is not in motion is difficult to sample with any predictable accuracy.

Material in motion offers an ideal condition for sampling. The deviations from quality would appear in the sample in the proportional degree they were present in the main stream.

In order to secure such a sample the cutter would be in continuous motion and the sample would normally be too large for testing purposes without secondary sampling.

In manual sampling the human element tends to meet volume requirements, yet lacks consistent accuracy, both in cutter travel, speed and manner of sampling. Mechanical sampling follows the set procedure regardless of existing conditions and results can be depended upon as having the sample taken exactly in the same manner at each cut.

Now, disregarding the factor of reliability, what is the cost factor? Is there an economic advantage to automatic sampling over manual sampling?

Let us assume the quality control engineer wants a sample taken each 5 minutes. To do so would require a man's full attention. But he cannot spare a man full time and modifies his decision to sample each 30 minutes. This means an operator must stop what he is doing, take the sample, dispose of the sample, then resume his work. For illustration, we will estimate a sampling cycle as 10 minutes or 20 minutes for the two samples per hour. That is 20 minutes each hour to stop work twice, take the two samples, dispose of same, and get back on the job.

As an individual item manual sampling does not seem large—16½¢ per sample if you can get a man at the minimum wage. But even at this rate it amounts to $660 per shift per year or nearly $2000 per year for 3 shift operation.

Now compare this cost with an automatic sampler. Using a cost of $600 for the sampler and $600 for installation we have a cost of $1200. Amortized over 5 years, the cost per year is only $240 for full time operation. On the basis of 2 samples per hour, each sample cut mechanically would cost 2¢ as compared with 16½¢ for manual sampling.

Location of the sampling unit, the amount of sample and the frequency of the sampling cut must come from the quality control engineer.

A fertilizer plant flowsheet is given in the Industrial and Engineering Chemistry plant process series. The solid raw materials are Potash, Normal Super Phosphate, Triple Super Phosphate, inerts and recycle material.

Possible location for automatic samplers in a fertilizer plant might include one on the raw materials before they are put in storage bins. Another sampler might be between the ammoniator and the dryer. A third sampler could be placed ahead of the bagging department.

In a flaked ammonium nitrate plant it is possible that a sampler might be desired ahead of the cooling belt, one on the screen oversize material and one on the material going into the bagging hopper. If coating agents are supplied to specifications, it might be desirable to have a sampler ahead of the batching scale.

Agreement between producer and customer on how the material is to be sampled in determining whether or not it meets specifications is important.★★

Raw Materials Symposium

A REVIEW of raw materials used in processing granulated fertilizers included comments by R. D. Taylor, National Potash Co., Joseph Sharp, Spencer Chemical Co., J. O. Hardesty, and W. L. Hill, both of the USDA.

The physical nature of the raw materials is largely responsible for the compliance of the product to the requirements of: free flowability, uniformity of composition, and ease of distribution," remarked R. D. Taylor. Continuing, he reported that particle size is a particularly important factor.

"As shown by Mr. Hignett and others, increasing the potash particle size does help granulation efficiency, particularly high potash grades. The extent is dependent to a considerable degree upon the efficiency of the ammoniator and the preceding equipment as mixers. The more positive the mixing action, the less the effectiveness of coarse potash as compared with fine potash. Mr. Taylor completed his observations with comments on the relative merits of coarse and fine potash in making granulated fertilizer.

Effects of phosphorus on granulation and in processing mixed fertilizers were described by W. L. Hill, USDA, while John O. Hardesty, also of the USDA, reviewed "Particle Size Effect of Potassium Chloride on the Processing of Granular Mixed Fertilizers." Mr. Hardesty said in part:

"Observations by the fertilizer industry corroborated by experimental work at the laboratory of the USDA, Beltsville, Md., indicate that potassium chloride in some types of mixed fertilizer appears to resist agglomeration with other ingredients during the granulation process. This difficulty is less apparent in high-nitrogen than in low-nitrogen mixtures. High concentration of salts, such as ammonium nitrate or urea, in the solution phase is characteristic of high-nitrogen mixtures and is one of the most important conditions favoring agglomeration of potassium chloride with other ingredients of the mixture.

"Such high-nitrogen mixtures as 8-16-16 and 10-10-10 usually can be granulated without difficulty. On the other hand, low-nitrogen, high-potash mixtures, such as 4-16-16 and 5-20-20, are often difficult to granulate, largely because the low proportion of nitrogen in such mixtures does not permit the use of sufficient amounts of highly-soluble nitrogen carriers. These mixtures may have inadequate plasticity owing to low concentration of salts in the solution phase. As a result, the potassium chloride complement of the mixtures, which is the least amenable to agglomeration of any of the materials present, is not fully incorporated in the granule. Thus a considerable proportion of it appears as individual particles of potassium chloride in the granular product.

"When the potassium chloride in a 5-20-20 mixture is finely-divided, (96% passing a 35-mesh screen; Tyler Standard Screen Scale) a high proportion of potassium chloride tends to accumulate as fines in the granulator product during processing, often causing excessive recycle loads and interfering with production of on-size product. When the potassium chloride in the initial mixture is composed of coarse granular material, (100% passing a 6-mesh and 92% remaining on a 20-mesh screen) a high proportion of the individual granules of this salt accumulates in the on-size fraction of the product, allowing possible segregation and poor nutrient distribution. When the potassium chloride in the initial mixture is composed of medium-size granules (92% in the range of 10 to 35 mesh) the quantity of fines, or recycle material, obtained during processing is reduced appreciably, as compared with that obtained when finely-divided potassium chloride is used in the initial mixture. Plant nutrient distribution in the on-size product is fairly uniform.

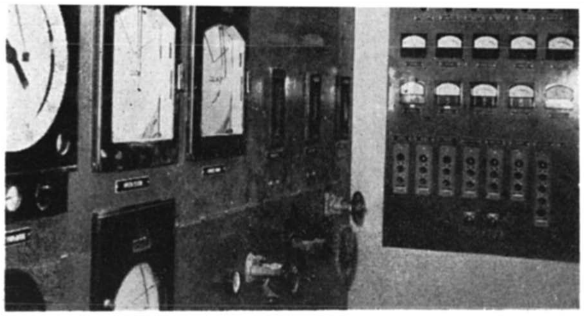
"Industry observations and results of laboratory tests indicate that granular potassium chloride, intended for use in fertilizer mixtures to reduce the amount of recycle fine fraction in the granulated product, preferably should pass a 10-mesh screen."

Comments on Bagging

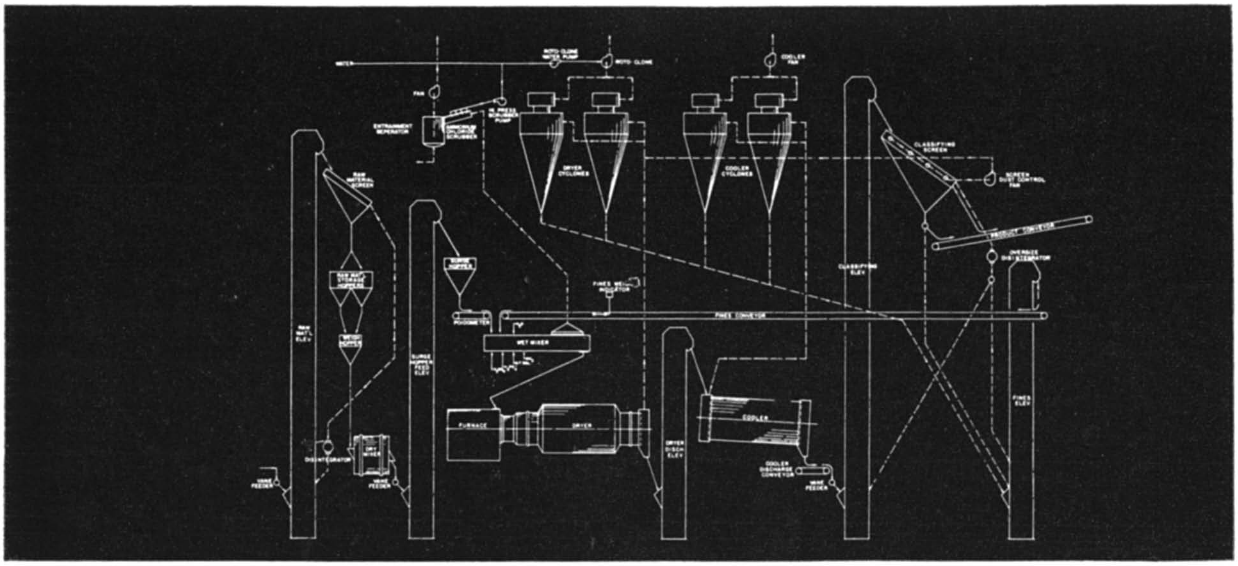
"What's in the bag?" asked R. J. McDonald of Round Table members, as he pointed out that a bag offers two prime functions. "A bag is not an end product by itself, but is rather a means of transporting a manufacturer's product from the production plant to the market place. More than this, a properly designed bag of quality is a valuable aid to a manufacturer in selling his product for maximum profit."

"A particularly valuable service provided by bag suppliers is brand design and printing. Leaders in the bag industry maintain art departments which are staffed by trained and skilled artists who have an expert knack for designing customer's brands for the greatest eye appeal. Printing presses for reproducing these special designs on bags are unique, as are certain means for making plates and other steps in the total process. Research is being conducted at all times to improve known techniques and existing equipment."

DAVISON'S TRENTON CONTINUOUS PROCESS FOR GRANULATION



Top photo shows operating control room with automatic temperature control equipment.



By J. E. Reynolds, Jr.
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THE Davison-Trenton Process had its beginning the spring of 1953 at our Trenton, Missouri plant. During the past three years, the process has exhibited its adaptability to keep pace with the economical advantages of many different types of formulation practices. A recent report (1) presented the theories and practices of ammoniating and granulating fertilizers by the Trenton process. Development data, formulation data, and operating experiences were given also at that time. We now propose to take this group, the Fertilizer Industry Round Table, on an actual plant tour showing photographs which reveal the machinery and equipment used in the process.

To follow the flow of materials through, we start with the raw material supply system. Ingredients are fed separately into an elevator, which feeds a single deck screen. The screened dry ingredients are discharged into separate storage hoppers

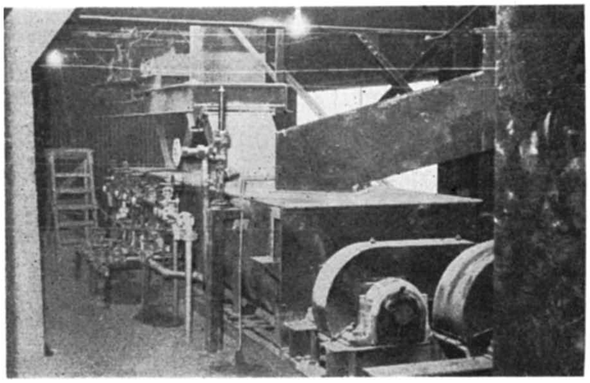
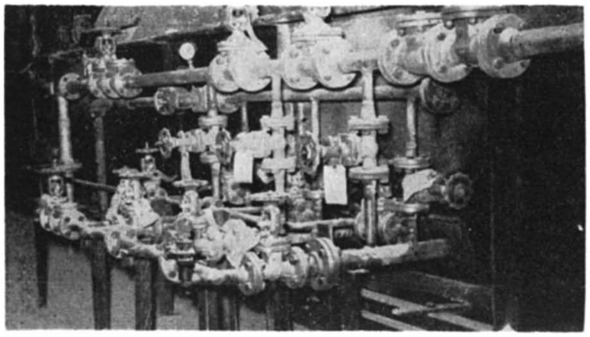
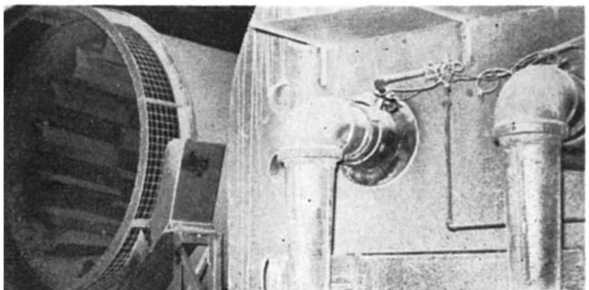


Photo at left shows pug mixer with liquid distribution piping arrangement to the left.



Liquid distribution piping arrangement which introduces all liquids from one side of the pug mixer.



Burners and the furnace of the dryer and the discharge end of the cooler.

above a single weigh hopper. The batch is weighed collectively into a single weigh hopper, and is then discharged into a two (2) ton rotary mixer. The blending achieved in the rotary mixer is established on a time cycle set for maximum retention in the mixer.

The blended dry batch is then elevated to the surge hopper. Constant flow is maintained from the surge hopper by means of a poidometer weigh belt discharging directly into a pug mixer. (The pug mixer is designed and arranged to permit thorough mixing of the dry materials and all recycle portions prior to coming in contact with the submerged liquids.)

The product from the wet mixer is discharged through a chute to a concurrent dryer. The dryer shown in this flow sheet is a DehydrO-Mat, but we also have several straight tube dryers. Sufficient heat and air is available to dry the heat sensitive-low free moisture grades, as well as the low nitrogen-high free moisture grades. The material from the dryer goes to the cooler, usually by means of an elevator. The larger coolers reduce the temperature of the product approximately 80-100°F. prior to passage across the double deck classifying screen. The boot of the classifying elevator is equipped with a rotary vane feeder to control the even flow of material to the elevator, and primarily to provide a seal for any potential "blow back."

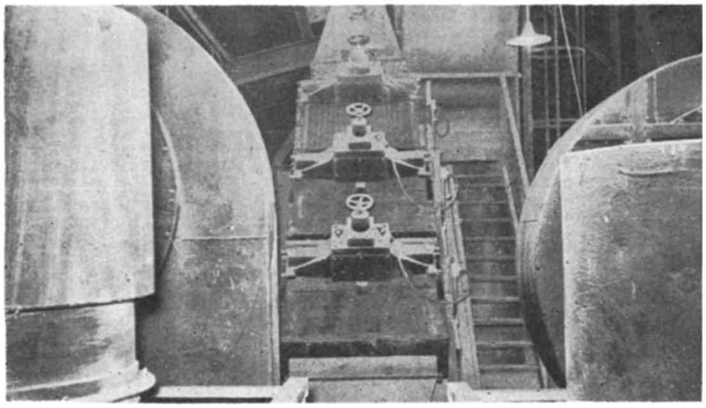
The classifying screen is a 4'x15' W. S. Tyler double deck hummer screen, equipped with three panels of U. S. Standard 6 mesh top deck screens, and three panels of U. S. Standard 16 mesh bottom screens. A dust control fan applies suction to the screen hopper to remove dust and heat to the dryer cyclones. This same fan also applies suction to other dust control points, such as the point of discharge from the recycle or fines elevator.

The oversize particles are cracked by a cage mill, or pulverizer, which returns the material across the screen to recover the acceptable particles. Approximately 60-70% of these particles will be recovered as product on this second time across the screen. The minus 16 (U. S. Standard) mesh particles are returned to the pug mixer via the fines elevator and conveyor belt. The closely sized acceptable particles are discharged onto the product belt which takes the material to storage, or a portion to storage and a portion to the recycle system. Cool, dry granules are returned to the wet mixer only for control of the wetness, temperature, and particle size formation necessary in the production of the highly concentrated mixed fertilzer grades. All recycle portions are combined on a conveyor belt which discharges into the dry raw materials feed end of the pug mixer. A continuous weigh indicator dial reports the total rate of recycle returned.

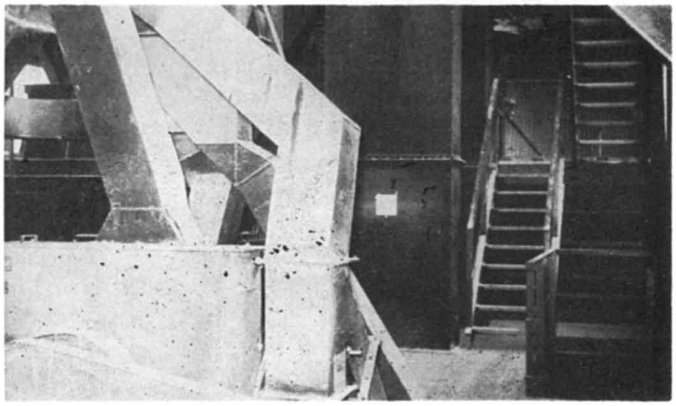
Dust collectors remove entrained dust from the dryer and cooler air streams. A wet fan arrangement is provided in the dryer air system to remove the excessively fine dust particles which might escape the dryer cyclone. This warm effluent liquid is returned to the wet mixer as make up water for the process. The transfer of heat from this warm water to the materials in the pug mixer assists in granulating the grades normally experiencing low reaction temperatures. The small solid particles from the cyclone are returned by chutes to the wet mixer by means of a recycle elevator conveyor system.

Figure 1 shows the operating control room. The automatic temperature control equipment for regulating the input of heat to the dryer is shown at the left. This control is designed for maintaining a desired dryer exhaust air temperature which is within 20-30°F. of the dryer product temperature. As less BTU's are required, and as the exhaust air temperature goes above the selected exhaust air control temperature, the input of heat is reduced. Conversely, if more heat is expended during passage through the dryer, and the dryer exhaust air temperature is reduced, the temperature controls activate the fuel supply to obtain additional heat. The next two recording charts on the top are for maintaining a record of the formulated nitrogen solution and anhydrous ammonia. The two bottom instruments on the left are for indicating the total rate of recycle to the system, and the recording instrument provides a record of the natural gas consumption. Other indicating flowraters are available on this panel for sulfuric acid, phosphoric acid, and water. The panel on the back wall has starter buttons for the pumps, poidometer, and the wet mixer; ammeters for such motors as the wet mixer, dryer fan, cooler fan, disintegrators, elevators, etc.; running lights for the major pieces of equipment; bindi-

The classifying screen, below, utilizes American Air Filter Rotoclone to remove particles smaller than can be removed by the conventional cyclone



Oversize particles from the classifying screen are "cracked" in the pulverizer, below, and returned through the cycle.



cator lights connected to the wet mixer surge hopper; emergency stop buttons for the burners.

The pug mixer is shown in figure two. Dry raw material flows from the poidometer, and the recycle return chutes are noted in the top left of this picture. The liquid distribution piping arrangement is shown on the left side of the pug mixer. A water flowrater is in close proximity to the wet mixer discharge control point. Slight fan suction is maintained on the top of the wet mixer.

The positive action agglomeration features typical of a double shaft pug mixer accomplish the ammoniation and granulation steps simultaneously. The pug mixer design, and the control of the paddle arrangement are planned to permit mixing and retention of the liquids and the solids. The pug mixer is used as the ammoniator-granulator, due to its many flexible features. It is thought that in addition to the positive action of the paddles, the ability to introduce large amounts of liquids at many submerged locations and to control the wetness economically, the ability to adopt many types of formulation practices, and to return the recycle portions wherever desired in this flat top mixer, are some of the advantages possible with this type of mixer.

A liquid distribution piping arrangement is shown in figure 3. This system introduces all of the liquids from one side of the pug mixer. The header provides for all nitrogen liquids to enter through the larger pipes at the top, and the acids to enter through the pipes nearer the shell of the mixer. Anhydrous ammonia enters the pipe near the pug mixer feed end, and the nitrogen solution enters the pipe at the other end. By valves we can regulate the number of points of introduction for either liquid. This same practice is followed for bringing the acids, sulfuric and phosphoric, into the manifold from opposite ends and valving off to control the number of distribution points. Water can be introduced into the nitrogen manifold for making an aqua ammonia solution. Air blow out connections are provided to keep the pipes open, if they become momentarily clogged. Premixing the ammonia containing liquids and the acids is accomplished in the pipes near the mid-point of this mixer.

The burners and the furnace of the dryer and the discharge end of our cooler are shown in figure 4. The materials flow from the wet mixer into the dryer; into an elevator; and back through a cooler located parallel to the dryer. A straight tube dryer and the "DehydrO-Mat" designed for large volumes of air and a large BTU output capacity are employed. The cooler is equipped with an extension of heavy wire cloth for the purpose of screening out any large object which could damage the oversize disintegrator. A magnetic pulley is incorporated also into the design of the conveyor belt feeding the classifying elevator.

The product from the cooler is brought by elevator to the classifying screen shown in figure 5. This screen is a standard 4'x15' W. S. Tyler double deck hummer screen equipped with equivalent 6 and 16 mesh (U. S. Standard) Ty-Rod screen cloths. The dryer fan and the top of one of the two dryer cyclones are shown on the left. In this installation American Air Filter Rotoclones are used to remove particles smaller than can be removed by the conventional cyclone. The cooler fan and one of the two cooler cyclones are shown on the right.

The oversize particles from the classifying screen are brought by gravity through a chute to the pulverizer shown in figure 6. These particles are "cracked" and then returned to the classifying elevator, or are returned to the recycle elevator in the case of the grades formulated with a high ratio of liquids to solids. These latter grades require additioned recycle portions for control of wetness, particle size, and reaction temperatures. The chute on the front left is the oversize chute to the cage mill; the chute immediately behind, is directed to the right and goes down in the product chute; and the next chute behind this is the fines chute, which takes the material to the recycle elevator, or to the product belt, in the case of a "semi-granulated" grade. The classifying elevator is in the background with the mixed raw materials elevator parallel and behind. The stairs on the right come from the second or pug mixer—control room floor. The first flight of steps up goes to the raw material hoppers, and provides an access to the wet mixer surge hopper which feeds the poidometer. The next flight of steps up leads to the raw materials and classifying screens, the dryer and the cooler fans, and the tops of the dryer and the cooler cyclones.

The Davison Trenton Process may be adapted to formulas which use large amounts of nitrogen solutions, phosphoric acid, sulfuric acid, anhydrous ammonia and water.

Formulating and Operating Practices

THE Davison-Trenton Process is characterized by the ability to conform to many types of formulating practices, which is extremely desirable due to the changing economic conditions of our industry. This process is adaptable to formulas which use large amounts of nitrogen solutions, phosphoric acid, sulfuric acid, anhydrous ammonia, and water, either individually or in combination with one or more other liquids. We control the complete liquid phase by means of recycle control to achieve a non-slurry condition, but to still achieve a satisfactory penetration of the liquids into the solids.

Reference

(1) Reynolds, Jr., J. E., Alfrey, N. K., and Rose, G. W. "Davison's Trenton Process For Ammoniating and Granulating Fertilizers." Presented at the 130th National Meeting of the American Chemical Society, Atlantic City, New Jersey, September 1956.★★

Packages—Packaging and

MATERIALS

OVER the years the packaging of inorganic chemicals and fertilizers has evolved from 200 lb. burlap bag units to the present trend toward 50 lb. paper units. While many manufacturers of fertilizers and chemicals have been aware of the costs of packaging, they have not given the subject the attention it deserves until recent years.

As in all industries lately, more attention is currently being given by fertilizer manufacturers to reducing costs and increasing production. Recent low profit margins realized in this industry have brought all costs sharply into focus. Manufacturers have made great strides as far as production efficiency is concerned, and the advent of granulation — started some years ago — has caused plant renovations not only in the granulating process, but in the handling of raw materials, batching and mixing as well.

Manufacturers in all industries are now finding that rapid efficient production is advantageous, but that it is only as efficient and economical as their ability to package and ship their product. We in our industry have run up against the same problem. We have increased the speed of our bag making machines considerably, but have realized only recently that we must remove the bags, pack them and ship them at the same rate of speed.

It is only during the last 25 years that materials handling has become recognized as a field unto itself. Materials handling equipment, however, is by no means a recent invention. Conveyors, lift trucks, hand trucks, and overhead cranes have of course been used for many years. The reason that materials handling tools and equipment have gone without the attention they deserve is because, in earlier periods, the supply of unskilled labor was plentiful and wages were at a low level, while the capital expenditures required for machinery were relatively high. This combination did not make the economies of materials handling particularly attractive. Since World War II, however, there has been a complete change in the labor supply market. In the major portion of the country the supply of unskilled labor has been critically short, and from all indications it will continue to be short for at least the next ten years.

Unskilled or semi-skilled labor is normally used to package and move materials. Pay rates for this type of labor have moved ahead considerably faster than the salaries earned by more skilled workers. Moreover, everyone is finding it increasingly harder to get people who will do a decent day's work in handling materials.

Industrial construction costs have increased over those of prewar days more than in any part of our economy. However, the efficiency of conveyors, bagging equipment, lift trucks, and so on, permits better use of expensive warehouse and plant space. This in turn allows substantial savings in capital outlays for construction. In fact, the cost of capital required to purchase materials handling and bagging devices is below the historical average for many years.

Because of rising costs of materials and labor, the cost of bagging materials has necessarily risen. Within the past three years the cost of manufacturing paper has increased over 20%, while the basic cost of paper itself has increased barely 5%. There is not much hope, therefore, of reflecting economies through lower pricing of packaging materials themselves. One of the few avenues open to cost reduction is through more efficient handling of the manufactured material. This can be effected by increasing the tonnage packaged and moved while using the same number of men. It must be borne in mind that packaging and materials handling equipment add only to the cost of a product but not to its value. Engineers estimate that this cost seldom represents less than 20%, and is often as high as 30% of total manufacturing outlays. This equipment can reduce the ultimate cost of the product to the manufacturer, therefore increasing his profit margin.

Let us talk specifically about the fertilizer industry. In the packaging of fertilizer there are two materials which can be considered; 1) burlap bags and 2) paper shipping sacks, commonly known as multiwall bags. There are four primary types of multiwall bags that can be considered:

1. Sewn Open Mouth
2. Sewn Valve
3. Pasted Open Mouth
4. Pasted Valve

Factors that must be considered in the final decision of bag type to be used are as follows:

1. Product characteristics
2. Economy
3. Filling & weighing
4. Package appearance & merchandising attraction

Now let us consider No. 1—Is the material hydroscopic? Does it set up? What is its flowability? I am sure that all of you are familiar with these terms and are aware that the products we are discussing are practically all hydroscopic. This in turn affects whether the material will set up or harden. Consequently, it is necessary to have some moistureproof or waterproof barrier construction in the bag being used. Flowability, or the ability of the material to flow readily also affects the construction of the bag to be used. Sifting of material from the bag is a problem with which we are frequently faced. If the material is granular and flows readily,

HANDLING COSTS

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a much tighter type closure is needed than is used ordinarily.

Now let us consider point No. 2—Naturally all of us are keenly aware of costs of our packaging materials. The greatest danger is in false economies; that is, trying to use a lighter basis weight than is practical, or, in some cases, doing away with the moisture barrier. While lighter basis weights may work satisfactorily for local shipments, their value in the over-all marketing of your product is debatable. If you inventory one type bag for truck shipment, another type for local shipment, and still another type bag for long hauls or railroad shipments you offset any savings effected by lower basis weight—not to mention the loss of customer good will through broken and torn bags. In the bag industry we check constantly factors affecting bag wear, and try to devise new types of construction which will result in greater economies for our customers. One successful shipment, however, does not always prove the case. I feel strongly that one should approach the reduction of basis weights with extreme caution. Economies can be effected by carload purchases, by proper warehousing and inventories, and by proper sizing of bags for the material being shipped. Economies along these lines will lead to the least troubles and greater savings in the long run.

Filling and closing is one of the functions by which the greatest savings in cost can be effected. Up until three years ago (with the exception of valve packers) the industry was using manually operated gross weighing scales which depend entirely upon the operator for rate of speed and accuracy of weight. Today there are various types of high speed, automatic accurate bag filling* devices.

Basically, as far as filling and weighing machines are concerned, there are two types of machines from which to make a choice: The open mouth type, using sewn open mouth or pasted open mouth bags, and the valve type, using sewn or pasted valve bags. There are quite a few factors to be considered regarding these two types of baggers and bags:

1. Cost of the bag
2. Cost of filling and weighing
3. Their carrying ability as far as your product is concerned.

Let us take the first item which is the cost of the bag itself. Naturally, sizes and constructions will vary to some extent; however, picking at random a printed bag constructed of 1/90 asphalt sheet, 1/50 asphalt sheet, and 1/60 multiwall kraft,—size 15 x 5 x 33—generally the cost of this bag will run approximately $90.00 in carload quantities. The cost of closing thread and materials for this size bag would be $1.26 per thousand, giving a total cost of $100.16. A sewn valve bag made from the same materials, 15 x 5 x 32, would cost approximately $108.50 without insert or tuck-in sleeve. The open-mouth bag thus offers a saving in bag costs of $8.34 per thousand bags, not including the cost of the insert or tuck-in sleeve. If a tuck-in sleeve is used, the cost would go up $4.00 per thousand for the valve bags. Naturally, we must take into consideration the fact that the sewn open mouth bag must be closed by the purchaser. This additional operation is offset, however, by increased bagging production.

Another factor to consider is that a bag of greater capacity must be used in order to increase the rate of production. This not only results in high costs, but has a direct effect on sifting, for the looser the pack the greater the probability of sifting through the valve. This probability is greatly increased when packing granular materials, since a loose pack is more likely to permit the granules coming between the valve and the bag. This in turn allows the material to sift.

It is our opinion that the sewn open mouth bag allows for greater economy, greater speed of production, and a positive closure. Valve bags have the advantage of not requiring a sewn closure or sewing operator. However, this advantage, in my opinion, is not sufficiently great to offset the disadvantages which I have already outlined. I realize that many plants are now equipped with valve packers. Naturally, they want to effect as many economies as possible without changing their equipment. I can only state that during the past two years there have been a tremendous number of changes in plants of this type from valve packing to open mouth packing.

Everything that I have said about the differences between the two types of sewn bags also applies to some extent to the same bags using pasted bottoms and closures. Let me, however cite the approximate cost of these bags. The pasted open mouth bag 20″ x 29″, bottom 5″, of the same construction and same capacity, costs approximately $103.60 per thousand. The pasted valve bag, same construction, 20″ x 24½″, bottom 5″, valve 5″, amounts to $105.11. Strange as it may seem, the pasted open mouth and pasted valve bags—at least as far as I have been able to determine—are still higher in cost than the sewn open mouth bag.

All in all, the combined high speed production and bag economy offered by sewn open mouth bags offers cost savings from 3¼ to 9½ percent over the other basic types for 50 lb. units, and from 4½ to 8¼ percent for 80 lb. bags.★★

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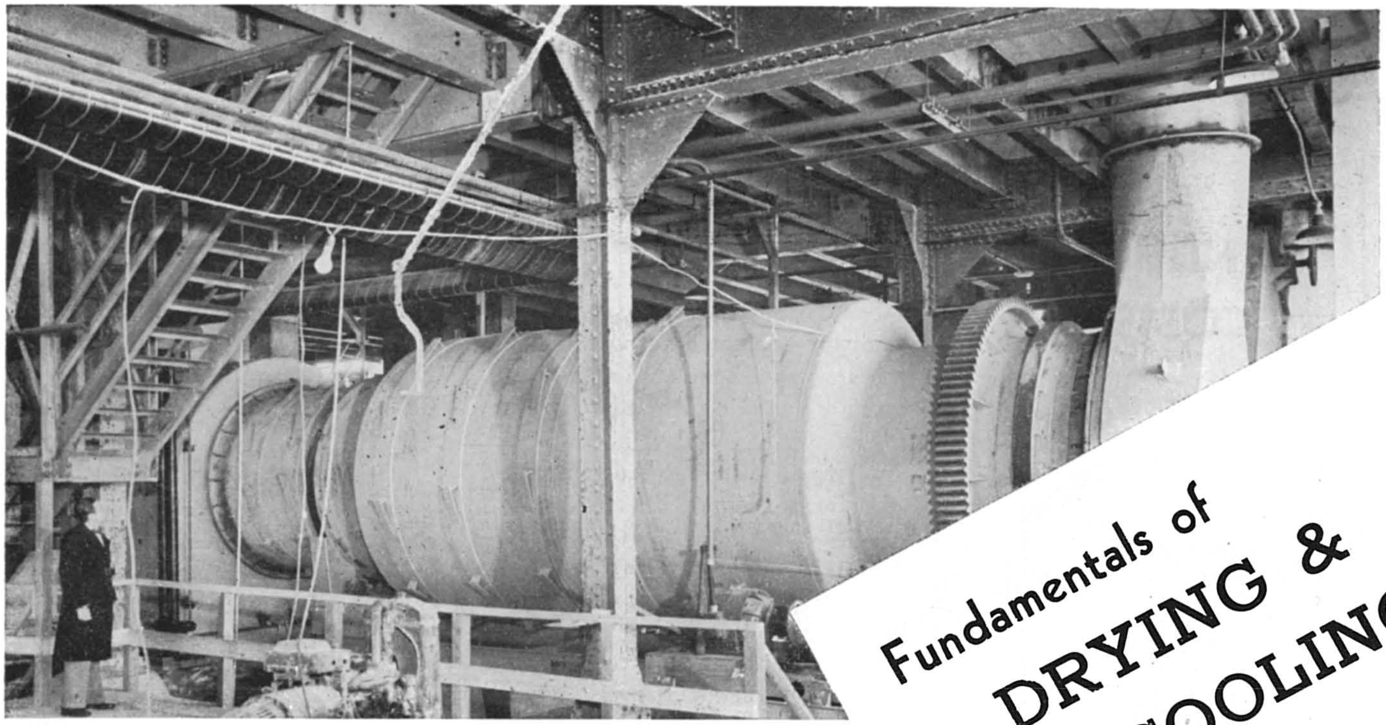
*Bagging Equipment, October Agricultural Chemicals, pp. 42-45, 1956.



Fundamentals of DRYING & COOLING

DRYING of fertilizer is done to facilitate and speed up the process of manufacture and to preserve the material in its final stage. But whatever the reasons for drying, the performance of the drying equipment may be the keystone on which depends the success or failure of your granular fertilizer plant. There are various forms of dryers on the market, any one of which may possess advantages over others when operating under definite conditions. This report deals only with the more important types of dryers employed in the drying of granular fertilizers.

An interchange of heat is necessary in all fertilizer drying operations. Heat is required to warm up the fertilizer undergoing treatment and to evaporate the moisture. Air is the medium generally employed for conveying heat to the material because first, it can operate on each particle directly; second, it readily lends itself to accurate control; and thirdly, it provides a convenient means of carrying away the evaporated water. When passing thru the dryer the air gives up the necessary amount of heat for heating and evaporating the water, and in so doing suffers a fall in temperature. It must, however, leave the dryer at a sufficiently high temperature to enable it to retain its water vapor, without the danger of reaching the saturation point. In practice it is seldom allowed to exceed 50 to 75% saturation at the exit of the dryer. For the drying process to continue, the air must be removed continually, *and the quicker it is removed, the more rapid is the rate of drying.*

The temperature to which the air may be heated at entry to the dryer will depend mainly on the nature of the grade of fertilizer being treated, its initial moisture content, the extent to which it is desired to reduce the moisture and the amount of heat of reaction. If the last traces of moisture are to be removed, the fertilizer will gradually rise in temperature and, in the case of countercurrent dryers, the material may have to be heated almost to the temperature of the entering air, in which event, the air may only be heated to the highest temperature which the fertilizer can stand without injury. If, however, the fertilizer may still retain a certain amount of moisture in its dried state, it will not be necessary to raise its temperature to that of the incoming air, and accordingly the air may be admitted to the dryer at a higher temperature, as in the case of parallel flow drying. As a general rule, it may be stated that the higher the temperature of the incoming hot air compatible with the grade of the fertilizer being dried, the more efficient will be the dryer.

The amount of water to be removed varies considerably with different grades of fertilizer. Some wet mixes may have a water content of 14%, or it may be as low as 4%. As for the final moisture content, most grades are considered dry at 1½% of water, or even higher. The initial and final water content have an important influence upon the time and cost of drying.

When wet ammoniated fertilizer starts to dry, the moisture in the outer

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layers moves to the surface, where it is removed by evaporation. Since the moisture content of the outer layers is being reduced, a general diffusion toward the surface is set up from the relatively wet to the relatively dry portions, and this diffusion extends gradually into the interior of the body of each fertilizer granule. The rate of this movement (diffusion) depends on *the rate of evaporation, the temperature of the material, its texture and structure, its water content, and the way in which the moisture occurs throughout the material.*

It is quite evident that the drying of granular fertilizer really involves two simultaneous processes, namely (1) evaporation at or near the surface of the granule and (2), diffusion of the moisture to the surface of evaporation. Evaporation or drying will therefore be limited by the rate at which the moisture can diffuse to the surface. But with the diminishing water content, the rate of diffusion, and hence also rate of evaporation is slowed down.

Gas or oil direct fired rotary fertilizer dryers will evaporate from 2 to 6 pounds of water per cubic foot of dryer volume. Evaporation capacity increases to some extent with increasing moisture content of the fertilzer feed and with increasing allowable moisture content of the final product.

Temperature

HEAT increases the rate of evaporation as well as the diffusion of moisture to the surface. An upper limit to the temperature which may be employed is set by the formulation and the grade of fertilizer. The higher the amount of water a grade of fertilizer contains, the higher the drying air temperatures may be.

A certain quantity of heat has to be supplied to the dryer per hour to dry a given tonnage. That heat employed usefully goes to evaporate the moisture in the goods. In a plant showing a low thermal efficiency, this portion of heat usefully employed may be only a small fraction of the total heat supplied. The rest is lost by radiation, high exhaust air temperature, air leaks, in heating up the fertilizer, in transporting equipment, and in the entire drying system itself. The thermal efficiency of a dryer is measured by the ratio of heat required to evaporate the moisture from the material, to the total heat supplied.

Humidity

THE amount of moisture in the air influences not only the drying rate, but also the extent to which the fertilizer will dry. At each temperature and pressure a cubic foot of air is capable of holding a definite maximum quantity of water in the form of vapor. In this condition the air is said to be saturated, or to possess a degree of saturation or a relative humidity of 100%. If this same cubic foot of air contains less vapor, as for instance only ¾ or ¼ of the saturation amount, at the same temperature and pressure, these conditions are expressed by saying that it has a relative humidity of 75% or 25% respectively. In the first case, the air would be "moist," in the last case "dry."

It is important to note that raising the temperature of moist air, lowers its relative humidity. Thus air at 75% humidity and at 60 degrees F. has a relative humidity of only 11% when raised to 120 degrees F. Drying cannot, of course, take place in already saturated air. Reducing the degree of saturation of the drying air increases the rate of drying.

During the first stages of drying, the temperature of the material is approximately at the wet bulb temperature of the surrounding air (well below the boiling point of water). As drying proceeds, fertilizer temperature gradually rises, and towards the discharge end approaches that of the drying air temperature. This is an important practical point when dealing with grades of material which have an upper temperature limit which cannot be passed without causing injury to the material. This point must also be considered very seriously when drying in a counter-current flow dryer.

Air Velocity

THE rate of evaporation is increased by an increase in the velocity of the air passing over the surfaces of the granular material, because the rate of diffusion of the vapor lying at the surface of the fertilizer, into the surrounding air, is increased. Therefore, the amount of heat brought to each fertilizer particle in a unit time is increased also.

As stated previously, air forms a convenient vehicle for carrying heat to the wet fertilizer and for removing the vapor produced by evaporation.

Obviously, this heat so conveyed must be distributed uniformly throughout the cascading, showering fertilizer. Moreover, the vapor must be swept away effectively from all surfaces of evaporation. This clearly entails a correctly designed lifting flight system for uniform distribution and active air circulation. If the hot air takes a direct path from the furnace thru the dryer and out into the atmosphere, the internal air to material circulation is likely to be defective, and if it escapes with low humidity the thermal efficiency of the operation will be low.

Retention Time, Flight Arrangement and Inclination

ROTARY dryers usually run with 10 to 15% of their volume filled with fertilizer, and under these conditions the dryer usually can be made to hold the material long enough to accomplish the removal of internal moisture. If the holdup or retention in the dryer is not great enough, the time of passage thru the dryer may be too short to achieve the desired degree of removal of internal moisture, and its capacity will be less than it should be. Time of passage is defined as holdup divided by feed rate.

The slope of rotary dryer shells varies from 0 to ¼" per ft. The slope is adjusted usually to give a holdup of 15% after the diameter, length and speed of rotation have been fixed. In some cases of parallel flow operation, negative slopes have been used.

The direct rotary dryer is usually equipped with flights on the interior surface of the shell for lifting and showering the wet material thru the hot gases during passage through the cylinder. These flights may be extended continuously through the entire length of the dryer, or rows of them may be offset or staggered every 3 to 4 ft. The slope of the flights depends upon the handling characteristics of the fertilizer. For free-flowing granular material, a radial flight with as much as a 90 degree lip is used. For sticky materials, a straight radial flight without any lip is used. Intermediate types have been designed to give maximum showering action of the fertilizer as it passes from feed to discharge during rotation of the dryer unit. In other words, when the fertilizer changes characteristics during drying, the flight design is changed along the dryer length. Spiral flights are used for the first few feet at the feed end of the dryer to accentuate a forward motion of the fertilizer into the dryer before normal flight action begins. When parallel air flow is used, flights are often left out of the last few feet of the dryer to prevent excessive dust carryover in the exhaust gases.

Parallel and Counter Flow Drying and Cooling

WHEN the fertilizer travels thru the dryer parallel with the drying air, there is a rise in material temperature from the feed end to the discharge end. If the air is to be exhausted at the highest humidity possible, then the drying of the product as it approaches the discharge end will be retarded, whereas this is the region in which it should be accelerated. If the humidity of the exhaust air is reduced in order to speed up drying, the thermal efficiency of the drying process will be lowered.

A favorable feature of the parallel flow dryer is the relatively small amount of air required to carry off the moisture, because it leaves the dryer at a higher temperature.

In the countercurrent flow dryer the highest material temperature occurs at the discharge end. On the other hand, the condition of highest relative humidity exists at the material feed end. The fertilizer is more likely to leave the dryer in a hotter condition and a relatively large amount of air will be required to prevent condensation at the discharge end. This condensation could take place on the surfaces of the relatively cold material entering dryer at this end.

The hot gas flow can be parallel or counter-current. Parallel flow is especially suitable for heat sensitive chemical fertilizers, since the high initial rate of evaporation maintains a solids temperature well below 212 degrees F., and instantly cools the surrounding gases. The high allowable inlet air temperature and the low product discharge temperature make thermal efficiency relatively easy to attain. However, extremely low moisture contents cannot be obtained in parallel flow dryers because the product leaves the dryer shell in contact with the vapor laden exhaust gases.

From calculations based on equipment sized for a typical plant producing 20 tons per hour of finished granular fertilizer (40 tons throughput), it can be shown, for example, that approximately twice the quantity of air is required where counter-current drying is used as contrasted with that needed for a concurrent (or parallel flow) dryer installation. Since twice the amount of air is required in the case of counter-flow drying 20 tons per hour, the drying unit must be larger to compensate for the increased air velocity.

On the other hand, almost three times the quantity of air is required to do the same amount of cooling in a parallel flow cooler as compared with a counter-current cooler. Therefore, a larger parallel flow cooler would be needed.

Phases of Drying

THE DehydrO-Mat by Edw. Renneburg & Sons, was designed for drying heat sensitive materials at rates that vary as the charge moves through the unit from the throat to the discharge end. Like other concurrent flow adiabatic dryers, this unit uses high temperature gas in contact with the wet charge to effect rapid, initial drying and then continues with steadily decreasing gas temperature and increasing humidity as heat flows into the charge from the gas, and as water evaporates from the charge into the gas stream.

As long as there is moisture diffusing to the surface of the material fast enough to keep the surface damp, the fertilizer remains at the bulb temperature of the gas, even though the actual gas temperature is hundreds of degrees above that of the fertilizer. Only when the rate of evaporation from the surface of the fertilizer exceeds the rate of diffusion from the interior of the particles does the surface become dry and its temperature start moving up. This is the principle upon which all parallel flow dryers depend to keep the fertilizer from being overheated during the most of its travel. For the final reduction of moisture to "commercial dryness," the gas should have lost so much temperature that it will not harm the dry product. The Renneburg dryers use this principle to an advantage because of the varying diameter throughout their length. It is a recognized fact that the movement of wet charge should be rapid during the initial contact with very hot gas, because less time is needed to evaporate water when there is a large temperature difference. Rapid movement of the fertilizer is of further advantage in constantly exposing new surfaces to the hot gas and in improving heat transfer by greater gas velocity over the surface of material.

Higher temperatures and air flows can be used in the constant rate drying section because moisture evaporation holds the particle temperature at the wet bulb point. The drying rate will be high, so the conveying can be fast. Lower temperature will be used in the falling rate section, where the product temperature will approach that of the heating medium. The rate of drying in the falling rate section will be relatively independent of the air velocity, so lower air flow rates can be used. Since air flow rate will be lower, the bed depth can be increased, and since drying will be slower, in the falling rate section, the holdup time should be increased.

In some dryers, efforts have been made to slow down the movement of the solids at the falling rate period of the drying cycle by changing the pitch of lifting flights. However, we felt that the increased mass of gas resulting from the addition of water vapor was certain to pickup and convey more and more partially dried material of decreased weight. As a result, much of the fertilizer would be transported by the relatively high velocity gas, and thus its retention time in the conventional dryer would be reduced in the zone where longer time is really needed. The conventional solution in the straight shell dryer has been to design the entire dryer larger to meet the low velocity requirements in this section.

The idea adopted for the DehydrO-Mat Dryer was to increase the shell diameter in this section and thereby reduce the gas velocity here to the desired rate—that is, to size the various parts of the dryer to conform to the theory of drying. After planning for suitable gas velocities throughout the length of the unit, we decided that we could set the dryer horizontal rather than sloping toward the discharge end as most units are arranged.

Recent Developments In Furnace Design

CONVENTIONAL dryer furnaces normally require the installation of insulation and refractories which add considerably to the overall cost of the dryer installation. In the larger fertilizer plants insulating refractories often cost as much as $4,500.00 to $6,000.00. A double shell refractoryless, gas-fired steel combustion chamber developed by Edw. Renneburg offers considerable savings over the conventional furnace. Sizes of the unit range up to approximately 15,000,000 BTU per hour capacity.

DehydrO-Mat Cooler

Patents Pending

THE DehydrO-Mat cooler is a counter-current unit, similar in external appearance to the dryer, but operating in a very different manner.

Air is used to introduce the material into the feed end of the cooler in such a way that the dust-spilling problem, so often experienced in straight shell counter-current units, is eliminated. This is achieved by a design employing a concurrent or parallel flow of air and material for a short section of the cooler feed end.

Because of the big diameter section following the feed section, a large volume of air can be drawn against the flow of material before dusting occurs or material movement is obstructed. At the narrow discharge end, fine material cannot leave except through the rotating unloading chute or valve.

The unloading, and thereby the hold-up time, can be controlled by (1) adjusting the speed of rotation or (2) adjusting the overflow of the material into the unloading chute by the regulating valve.

Although we have designed and manufactured both parallel flow and also counter-current types of coolers, from our experiences we have found that the counter-current cooler has far greater capacity and is considerably more efficient than the parallel flow or concurrent unit. For example, in one granular fertilizer plant using a DehydrO-Mat counter-current cooler approximately 35 feet long we have cooled up to 40 tons or more per hour of finished granular fertilizer of various grades of materials. The product entered the cooler from the dryer at 240° F and 2½% moisture and was discharged from the cooler at 110° F to 120° F with 1½% moisture in the finished product.

Single Air Handling System for Both Drying and Cooling

SOME of the advantages of a single air-handling system for both drying and cooling are as follows:

1. Because the same air is being used for both cooling and drying, only one air-handling system (collector, ductwork, fan and drive) is required.
2. The heated air from the cooler is no longer wasted but put thru the dryer, resulting in fuel savings.
3. Cuts air pollution problem in half.
4. This system is particularly attractive where screening takes place ahead of cooling the finished product.
   (a). Send hot fines to ammoniator/granulator;
   (b). Cooler required to cool only the finished product; and
   (c). Greater cooler capacity.

To summarize, after consideration of all of the factors, we are of the opinion that the drying and cooling of ammoniated, granular fertilizers can be best accomplished in parallel flow dryers and counter-current coolers.★★

A Fertilizer Equipment Bulletin is offered by Edw. Renneburg & Sons, Baltimore, giving further details and illustrations of their coolers, furnaces, dryers, etc.

Drying a granulated fertilizer really involves two simultaneous processes: evaporation at or near the surface of the granule; and diffusion of the moisture to the surface of evaporation.

MORE COMMENTS ON DRYING

by F. T. Nielsson

International Minerals & Chemical Corp.

THE fertilizer industry generally is a large volume producer of a low cost material. Therefore processing and equipment costs must be kept low. With the advent of granulation, a series of manufacturing steps has been added to the process that cannot but help increase the cost of manufacture. Of course we hope that in most grades, reduced costs of new raw materials will help overcome the additional processing cost. The most expensive ingredient in the fertilizer formula is nitrogen, and the cheaper forms of nitrogen have poor physical condition in that they tend to absorb moisture from the air and result in caked products. Caking can occur from the purely physical phenomenon of compression of moist material into solid lumps. It can occur also from the chemical reactions between ingredients that result in new crystal formation with added bonding area.

However, both types of caking begin at a given moisture level, and caking accelerates as the moisture content of the product increases.

I believe that low moisture is the ultimate answer to the caking problem. Powdered fertilizer will not cake at low enough moisture contents, and granular material will cake if the moisture is high enough.

Of course each grade and formulation require a given moisture content for freedom from caking. But for high nitrogen goods like 12-12-12 or 10-10-10, good condition can be had if the moisture is 1% or less, while in low nitrogen goods like 4-16-16, 5-20-20, or 5-10-10 a moisture content of about 1.5 to 2.0% usually is satisfactory.

To reach moistures of from 1 to 2%, with low cost materials I believe artificial drying is necessary, and the drier becomes the most important piece of equipment in the granulating process if we consider physical condition as of utmost importance.

The rotary drier is the simplest piece of equipment for lowering the moisture content of fertilizer materials. It consists of a cylindrical chamber rotating on its horizontal axis with the discharge end slightly lower than the feed end. Because contamination is not a problem, flue gases are led directly through the cylinder, and very rapid evaporation of water is realized. Lifting plates, or shelves are fastened usually to the inside of the cylinder running the length of and parallel to the axis of the cylinder. The rotation of the cylinder continually elevates the material and throws it through the current of hot gas, and the inclination of the shell moves the charge forward.

Usually the drier is a single shell, supported on steel tires that rest on rollers held in suitable bearings. It is driven by a gear fastened to the shell which engages a pinion keyed to a driving shaft.

We have a choice of passing the hot gas in a direction opposite to the passage of the charge, (counter-flow or counter current,) or passing the hot gas in the same direction as the charge, (parallel flow or concurrent.)

You have heard of the advantages of counter current drying in a straight tube and of parallel flow drying in a DehydrO-Mat.

I will discuss parallel-flow drying in a straight tube. I prefer the word parallel to "con" because "con" can be misunderstood to be counter.

You know now that a counter-current drier theoretically is more efficient. However this efficiency cannot be fully realized in fertilizer drying, because of possible overheating of the material, and some grades are heat sensitive, that is, the materials will decompose if heated to too high a temperature. In a parallel flow drier, the hot gases contact material that is wet, and the evaporation of the water from the particles cools the particles. Regardless of the gas temperature, the particle temperature will be below 200+°F as long as water can evaporate freely from the surface of the particle. As the water content of the solids drops, the temperature of the solids can increase, but by then they have progressed down the tube where the gas temperature is lower.

So parallel flow drying is somewhat of a guarantee against overheating.

Because of the high gas temperature in contact with moist particles, parallel flow results in case-hardening, just like searing a steak. Case-hardening is a good thing on high analysis nitrogen goods like 12-12-12 because it minimizes the possibility of further agglomeration in the drier with resultant excessive oversize. On the other hand, case-hardening is not desirable on low nitrogen goods like 5-10-10, where granulation is difficult and the added agglomeration in a drier minimizes fines. Furthermore, in plants that do not have a granulating section, either as part of the ammoniator or as a separate piece of equipment, granulation takes place in the drier. Because of the case-hardening feature of parallel-flow, it is not the preferred type drier if a substantial portion of the granulation is to take place in the drier. Therefore, the relative percentage of high and low nitrogen grades and the presence or absence of a granulator will determine whether you should install a parallel-flow or a counter-current drier.

Now for some side comments—

Have the drier equipped with staggered rows of flights. I believe a flight running the full length of the drier is not desirable, because there is always the possibility of granules rolling along the flights without spilling over. Drying takes place when the particle falls through the gas stream, so the more times the particle falls, the more efficient the drying action.

In parallel-flow, the hot gas enters at the feed end. The feed chute enters the drier also at the feed end.

If the hot gas surrounds the feed chute, various troubles result. Corrosion is increased and the half of the feed chute facing the flame must be of stainless steel. Sticking of the feed to the hot chute is accelerated, and when sticking occurs, it is difficult to remove the crust because it has to be done through the feed chute opening.

On the other hand, if the feed chute enters the drier alongside the hot gas duct, the chute can be fabricated of mild steel or even rubber belting. Furthermore, rapping devices can be installed on the chute to minimize caking.

Next, provide automatic temperature control for the drier. Have the flame governed by the exit stack gas temperature. Then regardless of the variations in feed rate and feed moisture (as long as they are within the capacity of the drier) you will have a uniform moisture content in the product. Then provide about a 4-foot section near the feed end free from flights to permit some final hardening of granules in a drying atmosphere before they begin to be lifted up and dropped from the flights. Flight action occurring too early will break-up the granules.

Finally, do not make the drier too small. For normal recycle (between 25-50%) and with normal formulations that result in a drier feed of about 5-8% moisture, the drier should be about 8 feet in diameter and 50 feet long for a 20 ton per hour production rate. The exhaust fan should be capable of handling about 30,000 CFM of hot gas.

A drier 8 by 50 allows the material to remain in it for from 20 to 30 minutes. This retention time is necessary if drying is to be done with fairly low inlet gas temperatures, below 90° F.

Summarizing,

1. The more dry your product, the longer will it stay in satisfactory condition.
2. For low nitrogen goods with no preliminary granulating step use a countercurrent drier.
3. For high nitrogen goods with a preliminary granulating step, use a parallel flow drier.
4. Do not let the hot gas surround the feed chute.
5. Provide a short smooth walled section near the feed end.
6. Do not skimp on the size of your drier.★★

Screening in the Fertilizer Industry

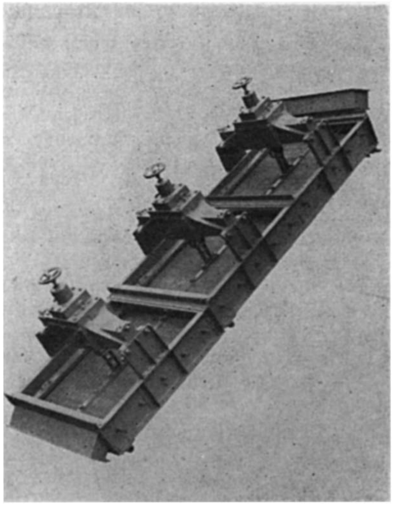
by Wayne King

W. S. Tyler Company

A DETERMINATION of the quantity of fines or oversize is obtained by sieve analysis of the final product. In actual fertilizer production, oversize is separated from the final product and sent through the crusher, while fines are returned through the granulator. Selection of screen sizes, therefore, for these separations, is an important step in plant operation.

A laboratory screen analysis gives a complete analysis of particle size distribution of the product, and offers a means of determining particle size range for separation.

After determining the range of granular distribution desired, it is suggested (1) a sieve be selected which determines the point of separation for the top size and for fines removal, (2) one sieve selected directly preceding or coarser than the sieve selected, and (3) one sieve on the next finer size position; in addition, at least one intermediate sieve should be included in the series.



The Tyler Screen

Chart 1 gives the entire picture on extent of granulation, from the point of view of particle size measurement. Particles retained on each sieve are shown in vertical bars to indicate percentage of depth of each bar. The chart aids determination of the economical top size point of separation. The desired point of separation for removal of fines may also be determined by the same procedure.

The rejected top size is in closed circuit through a crusher, and this closed circuit is effected by return through the ammoniator, the dryer, the cooler or direct to classifier screen. The fines recycle is returned through the ammoniator. The sieve analysis shows granulation efficiency and should be taken as frequently as required, i.e., depending upon processing problems, where the granulation efficiency could vary considerably, especially between grades.

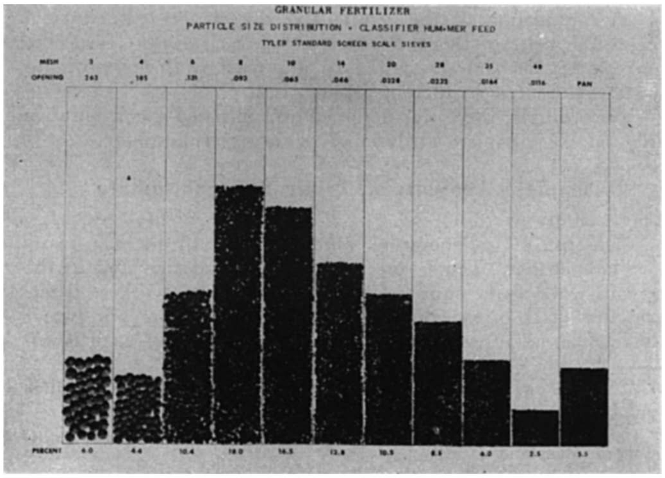


Chart I—Particle size distribution—Classifier Hummer Feed

Volumetric Analysis of Triple Superphosphate

A REPORT OF COLLABORATIVE STUDIES

By H. L. Marshall and M. H. Replogle***

Analysis of Triple Superphosphate

In past years, laboratories have had difficulties in securing comparable values for phosphorus, particularly in high phosphate materials. This discrepancy was brought to the attention of the Chemical Control Committee of the National Plant Food Institute with the request that they ascertain the cause of variance when it was assumed all laboratories were using the Official Method of the A.O.A.C. for analyzing the material.

The first step was to make a survey by questionnaire of many industry laboratories to find out the minute details of the methods each used to analyze superphosphates and mixed fertilizers in their respective laboratories. Eighteen replied: each followed the Official Method in a general way: However, variance occurred in: sample preparation, digestion (acid ratio and time), filtration (paper grade and type of filter), precipitation and precipitate washing; titration of the phosphomolybdate was fairly uniform.

From the questionnaire data and personal conferences with those who have worked in developing analytical methods, information was developed by which a collaborative program was prepared. The first phase of the study was designed to ascertain the most effective moisture procedure, the effect of three variations of acid digestion, and the appropriate size of sample to use for molybdate precipitation. A uniform prepared sample of triple superphosphate was obtained and forwarded to twenty two collaborators along with minutely detailed instructions.

The return from the collaborative work showed:

1. Moisture via vacuum was 2 percent or more, less than 100°C.
2. Fair uniformity of moisture at 100°C was attained.
3. No significant variances were developed by varying the ratio of HCl to HNO_3 for perchloric acid—HNO_3.
4. Collaborators had less individual deviation using a 0.10 gm. sample.
5. Collaborators showed significant individual deviations using a 0.05 gm. sample.
6. Results using 0.05 gms. sample uniformaly were 0.20 percent higher.
7. Variations between collaborators were as much as 1.5 percent, although the majority were within 0.40 percent.

The results of the preceding study indicated that the deviations might lie in the technique of precipitation. To test this theory, a pure, completely soluble salt (monopotassium phosphate) was used. Identical samples and detailed instructions for precipitation and titration were sent to the previous collaborators.

Results: the salt contained more P_2O_5 than theoretical owing to unexpected impurities. Subsequent analysis proved this true.

The returns from the collaborative work on the monopotassium phosphate showed:

1. Significant deviations, between replicates by the same operator on different days.
2. Deviations between collaborators greater than in the preceding study. Spread between high and low was larger, although the majority when averaged were in the same range.
3. The use of 0.05 gm. sample probably accounts for the deviation, and indications are that more experience on the part of the laboratory workers with this size of sample may reduce the deviation.
4. The question of precipitation and titration was not wholly resolved.

A capitulation of the results and experiences gained from the several series of this collaborative study clearly set forth the approximate mechanics required to approach uniformity of analytical values from different laboratories.

Accordingly, there was designed and initiated a collaborative study on the complete analysis of triple superphosphate.

Complete Anaylsis of Triple Superphosphate

Experimental:

The list of collaborators was expanded to include many more laboratories. Ten laboratories that had participated in the previous work were requested to do supplemental work to determine the effect of variations in technic in moisture and water soluble determinations. To evaluate the precision of each laboratory with respect to time, each collaborator was requested to run duplicates at the same interval.

Sample Source and Preparation:

1. Triple Superphosphate ground to—35 mesh and bottled at the Davison Chemical Company plant, Barton, Florida.

Instructions to Collaborators:

A. General
  1. Run analyses in duplicate.
  2. Report each duplicate value regardless of variance: it may help in the evaluation of the overall data and be of significance to you. In case of mechanical errors, repeat immediately.
  3. Make 2 series of analysis. Report all values just as you obtain them. Each series to be *exactly 7 days apart.*
  4. To help evaluate the data, include in your report under comments, the weather conditions at time of making each analysis; i.e. outside temperature, laboratory temperature, and humidity conditions.

B. Detailed—Primary Results
  a. Moisture
    Sample Weight
      2 gms. if 5 cm. dia. dish used
      5 gms. if 10 cm. dia. dish used
    Electric Oven
      100°C; 5 hours
    Vacuum dessicator
      Room temperature
      20 in. of vacuum
      18 hours

Note: If electric oven is used, place dish as close to thermometer or regulator as possible. Oven or dessicator load should not be such as to saturate air or dessicant. Under "Comments" in your report please give type of oven and whether air is circulated through the oven or not.

  b. Total P_2O_5
    1. Sample weight: 1.0 gram.
       (Duplicates if desired. Report all values.)
    2. Put sample in 400 ml. beaker or 500 ml. volumetric flask.
    3. Digestion
       10 ml. HCl—30 ml. HNO_3.
       Digest 15 minutes after the acid mixture starts to boil. Cool by adding 150-200 ml. cold distilled water.
    4. Making to volume
       500 ml. volumetric flask.
       If digestion is made in beaker, transfer to volumetric flask by water washes of 50 ml. each.
    5. Filter solution into Florence Flask (500 ml.) through a Whatman #2 or #12 filter paper.
    6. Aliquot
       A. 250 ml. wide-mouth Erlenmeyer or 400 ml. beaker or glass.
       B. From filtered volumetric solution take a 25 ml. aliquot.

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(1) A special study sponsored by the Chemical Control Committee National Plant Food Institute

* Olin-Mathieson Chemical Company, Baltimore, Maryland

** Davison Chemical Company, Div. W. R. Grace & Company, Baltimore, Maryland

7. Proceed with precipitation and titration as under Section E.

c. Water Soluble P_2O_5
   1. Sample weight—1.0 gm.
   2. Transfer sample to preformed 9 cm. #2 Whatman filter paper in a funnel.
   3. Wash with successive washings via gravity until total wash volume is 225 ml. (Be sure each wash is through the filter before the next wash). Collect washings in a 500 ml. volumetric flask to which 5 ml. HNO_3 has been added.

Summary

The Chemical Control Committee of the National Plant Food Institute in early 1955, noting that differences existed throughout the country in the analysis of high P_2O_5 goods, such as triple superphosphate, decided to do something about this.

The differences were felt to be due to variations in the interpretation of the official A.O.A.C. analytical methods. The Committee set out to study through collaborative work the effect of such differences in the mechanics of the methods.

The results of the three studies conducted from May 1955 through August 1956 show that:

1. Chemists' interpretations of the official procedures differ widely.
2. Collaborators differed in results, even when using specific mechanics.
3. A criterion for accuracy in the analysis of triple superphosphate does not exist at present.
4. Precision of test varied from collaborator to collaborator.
5. Various modifications of the specific mechanics of the official methods may be feasible to reduce the degree of variability among chemists and to imimprove precision in results.
6. Statistical methods in the analysis of collaborative studies are useful to describe more clearly the progress of such work. The diagrams and charts used by the quality control engineer can be utilized to show the results of such studies.

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   4. After washing, make to volume and mix well.
   5. Aliquot
      A. 250 ml. wide-mouth Erlenmeyer or 400 ml. beaker or glass.
      B. From volumetric solution take a 25 ml. aliquot.
   6. Proceed with precipitation and titration as under Section E.

Note: Please report time required to wash sample with water.

d. Citrate Insoluble P_2O_5
   1. Residue from water washes introduce into 250 ml. Erlenmeyer containing 100 ml. of neutral ammonium citrate solution at 65°C.
   2. Place in water bath maintained at 65°C.
   3. Agitate continuously or intermittently for 1 hour.
   4. Filter solution after digestion using suction or fast gravity filter. Use Whatman #2 or equivalent.
   5. Wash citrate residue with successive washing of 65°C. distilled water until total filtrate volume approximates 350 ml.
   6. Return residue and filter paper or pad to digestion flask.
   7. Add 10 ml. HCl—30 ml. HNO_3 to residue in flask.
   8. Digest by boiling 15 minutes.
   9. Cool by flooding with distilled water to 150 ml. volume.
   10. Transfer residue solution to 250 ml. volumetric flask by washing.
   11. Make to 250 ml. volume and *mix well.*
   12. Filter through dry Whatman #2 or #12 filter paper with Florence Flask.
   13. From filtered volumetric solution take a 50 ml. aliquot.
   14. Proceed with precipitation and titration as under Section E.

e. Precipitation and titration of P_2O_5
   1. Preparation of aliquot for precipitation
      a. Add 15 ml. 30% HN_4NO_3.
      b. Add 2 ml. of HNO_3.
      c. Adjust volume to 75 ml. with water.
   2. Precipitation (Use ammonium molybdate adjusted with HNO_3)
   (a) To adjusted aliquot add:
      1. Total and water soluble, 40 ml. molybdate soln.
      2. Citrate insoluble, 20 ml. molybdate solution.
   3. Digestion
      Stirring (a) continuously, room temp. 30 minutes.
      (b) intermittently or 40°C., 30 minutes.

Note: Report stirring method under "Comments."

   4. Filtration
      Suction:
      Moore-Shimmer or Hirsch Funnel — Paper pulp; Whatman #2, or equivalent layer of paper pulp or asbestos.
   5. Precipitate washings
      10 washings of 15 ml. distilled water each washing, using washings to complete transfer from precipitation container.
   6. Transfer washed precipitate to original precipitation container, using distilled water to assist transfer of any precipitate clinging to filtration funnel.
   7. Adjust final volume in original aliquot container to 100 ml. minimum with distilled water.
   8. Titration
      a. Dissolve preciptate with standard caustic solution, using 2 ml. excess.
      b. Let stand 10 minutes—closed, if practical.
      c. Add 0.5 mi. of 1% phenolphthalein indicator.
      d. Back titrate with standard acid to colorless, white or ph 8.0.

Calculate P_2O_5 from amount of standard caustic used.

Note: Restandardize caustic solution with potassium acid phthalate during analytical test. Report normality.

f. Special—Supplemental Results

In addition to the above primary outlined tests you are requested to run the following analyses. These data will assist in pointing up moisture mechanics and evaluating the effect of varying the technic on the water soluble and citrate insoluble values.

   1. Moisture
      Sample—same as General Instructions
      Electric Oven
      100°C.
      5 hours
      Circulated Gas
      2 liters per minute

Note: The oven should be provided with an opening in the top for ventilation. Insert a 1/8" copper tube in the top and extend to the bottom of the oven. Connect the copper tubing to an inert gas supply, (Nitrogen or carbon dioxide.)

During the test allow inert gas to be discharged into the bottom of the oven at the rate of two liters per minute. The circulation of gas will sweep out the moisture vapors released from the sample and also aid in maintaining a more uniform temperature throughout the oven.

Report results as *supplemental* under the primary results.

   2. Water Soluble P_2O_5.
      a. Sample weight: 1.0 gm.
      b. Transfer to 150 ml. beaker.
      c. Add 100 ml. water.
      d. Let stand 0.5 hour.
      e. Stir every 10 minutes while standing.
      f. Transfer to 9 cm. Whatman #2 filter paper. (Gravity filter).
      g. Wash beaker and filter with successive washings to total volume of 225 ml. in a 500 ml. volu-

metric flask to which 5 ml. HNO_3 have been added.

h. Follow General Instructions from this point on. Report values as *supplemental* underneath primary results.

3. Citrate Insoluble

a. Proceed as under General Instruction Section D above.

Note: Please report C. I. values on Report Sheet as supplemental underneath primary results.

Results:

The returns from the collaborators were tabulated and subjected to statistical analysis. Response was complete from all laboratories. Tables (1 and 2) and control charts* (1 to 20) were prepared from the data.

Statistical Methods Used:

1. Analysis of variance
2. Quality control technic using range statistics to estimate collaborator precision.
3. Multivariable charts.
4. Control charts.

Item 1.—The statistical method used for data analysis is known as the Complete Factorial Model. The detailed explanation of the mechanics of the analysis of variance for factorial experiments is covered in the referenced texts 1, 2, 3, and 4 and is not detailed in the scope of this paper.

Item 2.—The quality control technic used are the familiar type used by the quality control engineer in the analysis of process control data. The methods for such analysis are more fully covered in references 4, 5, and 6.

A brief explanation of the technic used in the studies in this paper is in order. This may best be illustrated through an example from Study No. 1.

Calculation of test precision using range. Results from Collaborator No. 1.

1. Using Aqua Regia
2. .10 gm. aliquot
3. Analysis in triplicate in one day.

Results — Total P_2O_5

Test 1 — 48.09%
Test 2 — 48.04%
Test 3 — 48.10%
Average — 48.08%
Range — 0.06%

Calculation of standard deviation σ of individual tests.

1. Equation

$$\sigma = \frac{R}{d_2^*} \quad \text{where}$$

σ = standard deviation
R = range or difference in 3 tests
$^*d_2$ = factor equal to 1.693 for three results
* From A.S.T.M. Manual, p. 63. (reference 3)

2. Calculation

$$\sigma = \frac{.06\%}{1.693} = .035\%$$

3. Precision is expressed as three standard deviations or 3 x .035% = 0.105%
4. Precision of all collaborators is pooled over all estimates of precision weighted by number of results where necessary.

Control charts designed to show the state of control between collaborators as well as their internal precision.

1. Average of the three results are plotted consecutively starting with Collaborator No. 1 and continuing over all collaborators.
2. Range is plotted for individual collaborators using the difference obtained from the highest to lowest value.

Construction of multivariate charts designed to illustrate conclusions from analysis of variance in graphic form.

1. Chart is constructed by plotting the highest and lowest value obtained by each collaborator in the form of a large X. This form is the choice of the writer; other forms may be used.

General:

1. A significant difference between collaborators is found in this study for all items analyzed.

2. The difference exists in primary as well as supplemental results.

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*Some of the charts are reproduced in publication of this article. Interested readers may check directly with authors for further details.

NPFI Study No. 3
Triple Superphosphate
Summary of Collaborators' Data - Avg'd. Over 2 Days

Table I

Collab. No.	Moisture		Total P_2O_5		Insoluble		Available (1)		Water Soluble		Free Acid	
	Avg.	Precision $3\sigma_R$	Avg.	Precision $3\sigma_R$	Avg.	Precision $3\sigma_R$	Avg.	Precision $3\sigma_R$	Avg.	Precision $3\sigma_R$	Avg.	Precision $3\sigma_R$
1	3.81	.189	49.06	2.769	2.14	1.53	46.92	4.29	40.10	8.769	1.96	1.923
2	5.65	.438	47.56	.291	2.58	.102	44.98	.387	32.28	13.785	2.02	.349
3	3.98	.105	48.15	.438	2.28	.072	45.87	.219	41.73	.146	2.31	.175
4	4.16	.351	48.21	.219	2.38	.219	45.84	.438	42.01	1.748	1.85	.219
5	3.48	.729	48.35	.438	1.73	.219	46.63	.363	43.08	.874	2.38	0
6	5.56	.510	47.72	.699	2.21	2.04	45.51	.495	42.08	.233	2.66	.102
7	3.82	.321	47.63	.174	1.18	2.214	46.45	2.247	39.57	6.382	2.41	.044
8	3.27	1.923	47.51	.684	1.96	.117	45.55	.756	41.46	.423	3.05	.801
9	4.59	.321	48.02	.510	2.03	.540	45.99	.918	40.61	2.550	7.28	3.21
10	3.88	.801	47.69	.306	2.26	.363	45.43	.669	41.70	2.113	1.50	.160
11	3.85	.438	47.56	.219	2.84	.147	44.73	.189	42.40	.437	4.96	.393
12	3.80	.261	48.15	.438	2.69	.087	45.46	.495	42.45	.219	2.50	.146
13	2.51	.294	48.60	1.065	.59	0	48.01	1.065	40.75	.798	.028	0
14	4.21	.378	48.13	.537	1.73	.816	46.40	1.239	42.20	2.291	2.82	.423
15	4.17	.027	48.05	.267	2.18	0	45.87	.267	41.40	.532	—	--
16	4.54	.132	48.23	.234	2.07	.102	46.16	.174	43.05	.612	2.33	.102
20	4.66	1.296	47.80	.495	1.96	.159	45.84	.627	41.73	.685	2.55	1.064
21	3.37	.336	48.35	.438	1.96	.291	46.39	.438	42.33	.291	2.87	.175
22	4.11	.351	47.75	1.167	2.20	.351	45.55	.933	42.70	1.457	2.54	.466
23	4.48	.204	48.09	.276	1.63	.159	46.46	.261	42.21	.583	2.28	.146
24	4.78	.030	48.14	.291	1.78	.102	46.36	.276	40.96	.583	2.41	.073
25	4.44	1.092	48.10	.363	2.23	.159	45.87	.378	42.68	1.238	2.98	.481
26	4.48	.276	47.90	.861	2.39	.177	45.56	.867	40.97	2.62	6.97	.248
27	4.67	.102	48.25	.291	2.29	.219	45.96	.510	42.09	.495	2.64	.102
28	4.84	.147	47.65	.267	2.83	.147	44.82	.219	39.12	.437	2.37	--
Overall Avg.	4.24	.456	48.01	.531	2.11	.357	45.91	.750	41.27	1.98	2.91	.466

Note: (1) Calculated from primary total P_2O_5 results.

NPFI Study No. 3
Triple Superphosphate
Summary of Collaborators' Data

Table II

Primary vs. Supplemental Tests Avg'd. Over 2 Days

	Moisture						Citrate Insoluble				Available				Water Soluble			
	Primary		Supplemental				Primary		Supplemental		Primary (1)		Supplemental (1)		Primary		Supplemental	
	Oven On Air No Circ.		Oven Forced Draft		Vacuum Oven													
ollab.	Avg.	Pre. $3\sigma_R$	Avg.	Pre. $3\sigma_R$	Avg.	$3\sigma_R$	Avg.	Pre. $3\sigma_R$	Avg.	Pre. $3\sigma_R$	Avg.	Pre. $3\sigma_R$	Avg.	Pre. $3\sigma_R$	Avg.	Pre. $3\sigma_R$	Avg.	Pre. $3\sigma_R$
7	3.82	.321			2.22	1.341												
12	3.80	.261			2.32	.276												
20	4.66	1.296			2.97	.306	1.96	.159	1.90	.030	45.84	.627	45.90	.495	41.73	.685	41.29	.583
21	3.37	.336	3.92	.261			1.96	.291	2.17	.102	46.39	.438	46.19	.510	42.33	.291	42.33	.146
22	4.11	.351	4.17	.174			2.20	.351	2.01	.408	45.55	.933	45.74	.932	42.70	1.457	42.65	.583
23							1.63	.159	1.82	.540	46.46	.261	46.27	.816	42.21	.583	42.22	.291
24							1.78	.102	1.65	.438	46.36	.276	46.50	.423	40.96	.583	42.16	.350
25							2.23	.159	2.24	.102	45.87	.378	45.86	.452	42.68	1.238	42.61	---
26	4.48	.276	4.47	.363	2.50	.291	2.39	.177	2.36	.072	45.56	.867	45.54	.787	40.97	2.62	41.80	.874
27	4.67	.102	4.70	.423	1.78	.102	2.29	.219	2.36	.087	45.96	.510	45.89	.277	42.09	.495	42.65	.291
28	4.84	.147			2.37	.072	2.83	.147	2.39	.174	44.82	.219	45.26	.262				
verall vgs.	4.22	.386	4.32	.306	2.36	.396	2.14	.196	2.10	.216	45.87	.501	45.91	.550	41.96	.994	42.22	.390

Note:
(1) Calculated from primary total P_2O_5 results.

3. A significant difference was found for collaborators' results between the first and seventh day of analysis, with the following exceptions:
   a. Supplemental moistures, oven method, using air circution.
   b. Primary results—Total P_2O_5.
   d. Primary and supplemental results—water soluble P_2O_5.

The extent of such differences are tested using the analysis of variance shown in Tables 5a through 6b.

4. Collaborators could not agree in analysis within one day over all items analyzed. The only exceptions were in the following analyses:
   a. Primary results—Total P_2O_5.
   b. Primary results—Available P_2O_5.

Conclusions:

Specific

A. Moisture

1. Collaborators differed widely in moisture using the oven method with no air circulation. Average results over the two days ran from a high of 5.65% to a low of 2.51%: see Table I. Since careful preparation was used for all samples sent to a collaborator, the only conclusions which can be derived are:
   a. Moisture technics differ perhaps in types of furnaces, sample placement, quantity, etc.
   b. Laboratory atmospheric conditions differ widely.
   c. Care in handling samples when received differed widely with reference to (1a) above. Comments received from collaborators indicate differences in technic, equipment and sample placement. With reference to item (1b) above, Table VII has been prepared to show the differences existing in temperature and relative humidity inside and outside the labs.

   Item (1c) is based on conjecture, only.
2. The use of forced air in ovens gave slightly higher results as shown in Table II, for the collaborators running the supplemental as well as the primary results.
3. Vacuum ovens give lower moistures as previously found in the brief results in Study No. 1. The comparison as shown below was averaged over all collaborators and days.

Oven method		*Vacuum Method*
No air	Air Circ.	
4.22%	4.32%	2.36%

4. Moisture precision varied among the collaborators with the various test methods ranking from lowest to highest variability from Table II as follows:

Method	*Precision*
Oven—air circ.	0.306%
Oven—no air circ.	0.386%
Vacuum	0.396%

The precision over all collaborators for the official method is 0.456% as shown in the table I summary.

5. Difference in moisture levels existed between days for all methods of determination. The differences are as follows:

Moisture by days
Averaged over collaborators

Method	*1st Day*	*7th Day*	*No. Collab.*
oven—no air	4.21%	4.28%	25
oven—air circ.	4.30%	4.33%	4
Vacuum	2.26%	2.46%	6

The difference found over all collaborators using the various methods is quite small even though the analysis of variance verifies such differences as being significant. A question of what is a practical difference needs to be resolved by the committee.

B. *Total P_2O_5*

1. Collaborators differed significantly in total P_2O_5. The high, averaged over the two days, was found for collaborator No. 1 at 49.06% P_2O_5. The low was found for collaborator No. 8 at 47.51%. (See Table No. I.)
2. No difference recorded in average level between days. For over all collaborators the average total P_2O_5 was 48.01% both days.
3. The test precision in Study No. 3 was better than in Study No. 2, but not as good as in Study No. 1. This tends to verify the previous conclusion reached in Study No. 2 that precision suffers over a period of time. A comparison of the precision in the three studies is shown below:

Precision over all collaborators (1)

0.05 gm. Aliquots		3	*Sample Used*
Study No. 1 (2)	0.092	0.277	Triple super
Study No. 2 (3)	0.340	1.02	"Pure" salt
Study No. 3 (4)	0.177	0.531	Triple super

Notes: (1) Single analysis
(2) Overall methods of digestion run in one day
(3) Run over 3 day period
(4) Run over 2 day period

C. *Citrate Insoluble P_2O_5*

1. Collaborators differed widely in citrate insoluble primary results running from low of 0.59% for Collaborator No. 13 to a high of 2.58% for Collaborator

No. 28. Results were averaged over the two days of test. (See Table I.)

2. The difference between results found on the two days averaged over all collaborators was as follows:

Citrate Insoluble
Averaged over all collaborators

	1st day	*7th day*	*No. Collabs.*
Primary results	2.15%	2.07%	25
Supplementary results	2.12%	2.08%	9

While the differences are found significant in the analysis of variance, the extent of the difference may not be considered critical by the committee.

4. Precision of citrate insoluble was found to be 0.357% over all collaborators in the primary results. (See Table I.)
5. The precision of supplemental results was not as good as the primary results: shown by the comparison in Table II. The small difference is not significant.
6. The wide variety of washing times used by the collaborators is shown in Table VIII with the levels of citrate insoluble and water soluble found by each collaborator.

   Time of washing varied from a low of 29 minutes for Collaborator No. I to a high of 265 minutes for Collaborator No. 2. Such practice illustrates the committee's objectives of revealing differences in the individual interpretation of the mechanics of the official method.

D. *Available P_2O_5*

1. Using the primary total P_2O_5 and the primary citrate insoluble P_2O_5, the available P_2O_5 for each collaborator's results was calculated.
2. Collaborators differed in available P_2O_5 from a low for Collaborator No. 11 of 44.73% to a high for Collaborator No. 13 of 48.01%. The 3.28% difference is certainly of a serious nature, considering the tolerance limits for available P_2O_5 in various states. The effect on product price to the producer and consumer of triple superphosphate is unquestionably of a serious nature. (See Table I.)
3. The supplemental available P_2O_5 results were calculated using the total P_2O_5 from the primary results of collaborators and the supplemental results for citrate insolubles. A small difference in average available P_2O_5 is shown. (See Table II.)
4. The difference found between days on available P_2O_5 is shown below:

% Available P_2O_5
Averaged over all Collaborators

	1st Day	*7th day*	*No. Collabs.*
Primary results	45.88%	45.94%	25
Supplemental results	45.90%	45.92%	9

The differences were not found significant in the analysis of variance as shown in Table 5a and 5b.

5. The precision of available P_2O_5 was found to be 0.75% over all collaborators for the primary results. (See Table I.)

   The precision of the supplemental results does not show improvement over the primary results for the special collaborators participating in this work. (See Table II.)

E. *Water Soluble P_2O_5*

1. Extreme differences among collaborators existed in the determination of water soluble P_2O_5. Collaborator No. 2 was low with 32.28% P_2O_5, while Collaborator No. 5 was high with 43.08% P_2O_5. (See Table I.)
2. Precision of water soluble testing leaves much to be desired. The precision over all collaborators was 1.98% P_2O_5. (See Table I.)
3. An improvement in agreement as to water soluble is noted in the results from the collaborators running the supplemental results. This group was higher in the water soluble results than all collaborators on primary results and they agreed very well in the supplemental program. (Collaborator No. 20 gave the lowest results in this work. (See Table II.)
4. Precision of test was noticeably improved in the supplemental work as shown in Table II. This indicates that prior leaching and stirring of the sample for extraction of the water soluble may be a practice which could in general improve method mechanics for high analysis goods.
5. The difference between days averaged over all collaborators was found as follows:

Percent Water Soluble P_2O_5
Averaged over all Collaborators

	1st Day	*7th day*	*No. Collabs.*
Primary results	41.41%	41.13%	25
Supplemental results	42.22%	42.21%	8

F. *Free acid as H_3PO_4*

1. Collaborators differed widely in level of free acid. Collaborator No. 9 reported a high of 7.28%. Such results may only indicate inexperience with the acetone extraction method or may indicate the need for spelling out operating instructions in more detail. (See Table I.)
2. Precision of test varied widely among collaborators. The average precision over collaborators was found to be 0.466%
3. The difference in results for the two days is shown below:

   Percent Free Acid—Averaged over all Collaborators

   1st Day..............................3.02%
   7th Day..............................2.79%

   (The collaborators in general found lower free acid in the sample on the seventh day.)
4. The free acid method used in the study is used by laboratories doing extensive superphosphate analyses. Many laboratories used the water soluble extract for free acid. So far no real finite method other than ether extraction has been developed.

Acknowledgments:

The scope of the work covered in this paper is not limited to the work of the authors. Many people are concerned in such an effort.

To enumerate all would occupy an excessive amount of copy. The collaborators are listed. The authors and the Chemical Control Committee express their thanks for the splendid cooperation displayed.

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To Mr. V. Sauchelli, Davison Chemical Company, whose advice and leadership in the program was invaluable, the authors' thanks are extended.

To all who gave of their time and talent to this study, thanks are extended for aid in the studies.

List of Collaborators

American Agricultural Chemicals	New York
Armour & Company	Atlanta, Ga.
“ “	Bartow, Fla.
“ “	Jacksonville, Fla.
“ “	Nashville, Tenn.
“ “	Wilmington, Del.
Eastern States Farmers Exchange	York, Pa.
Olin-Mathieson Chemical Corporation	Pasendena, Texas
I. P. Thomas Company	Paulboro, N. J.
Smith-Douglass	Norfolk, Va.
Virginia Carolina Company	Richmond, Va.
Wiley and Company	Baltimore, Md. (Analytical Laboratory)
Consolidated Mining Company	Canada
Canadian Industries, Ltd.	Canada
Gascoyne and Company	Baltimore, Md. (Analytical Laboratory)
Baugh Chemical Company	Baltimore, Md.
Federal Chemical Company	Nashville, Tenn.

Davison Chemical Company	Baltimore, Md.
" " "	Bartow, Fla.
G.L.F.	Ithaca, N. Y.
International Minerals & Chemical Corporation	Eastport, Ga.
Olin-Mathieson Chemical Company	Baltimore, Md.
Wilson Toomer Fertilizer Company	Jacksonville, Fla.
Swift & Company	Chicago, Ill.
Guanos-y-Fertilizentes	Cuautitlan, Mexico

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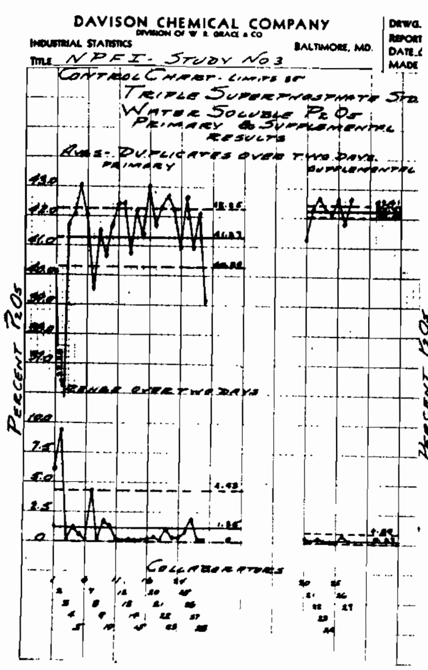

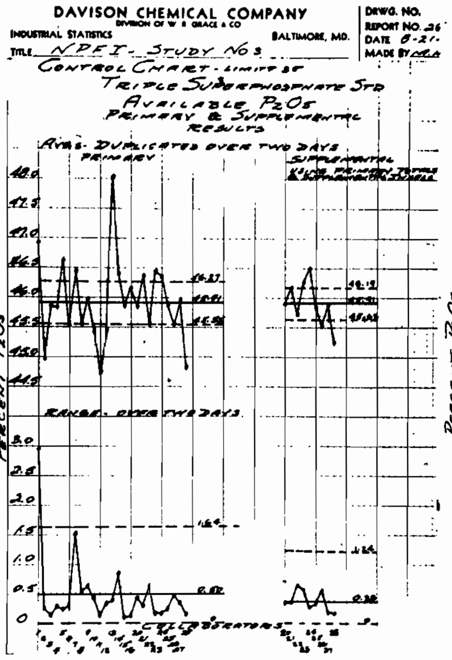

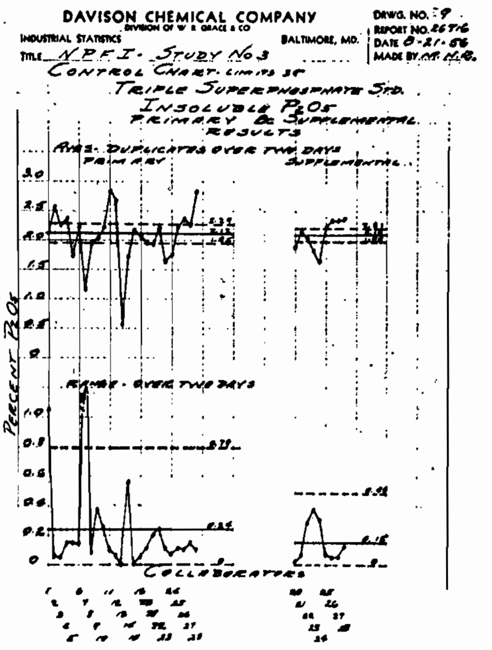

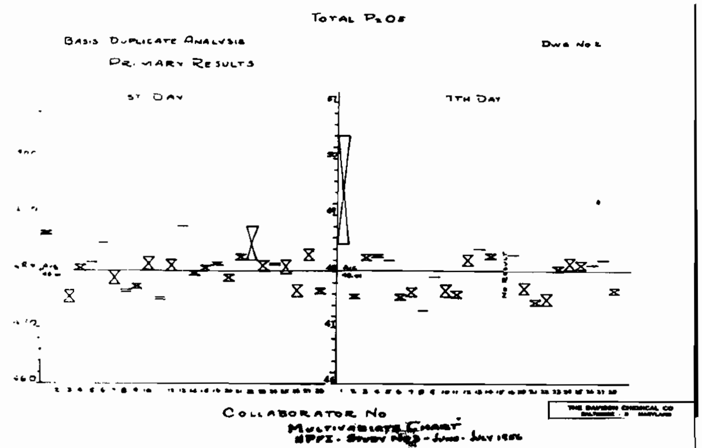

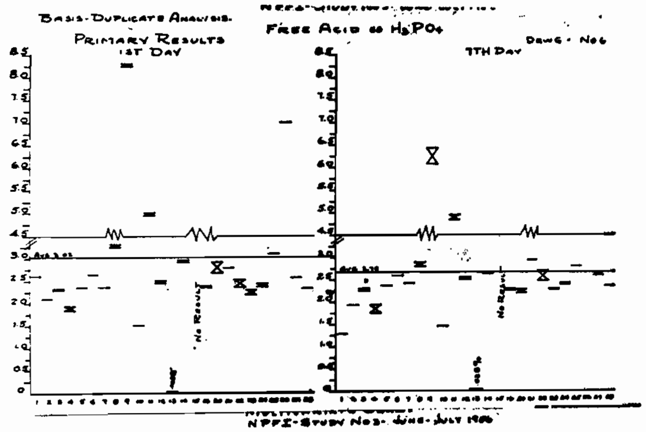

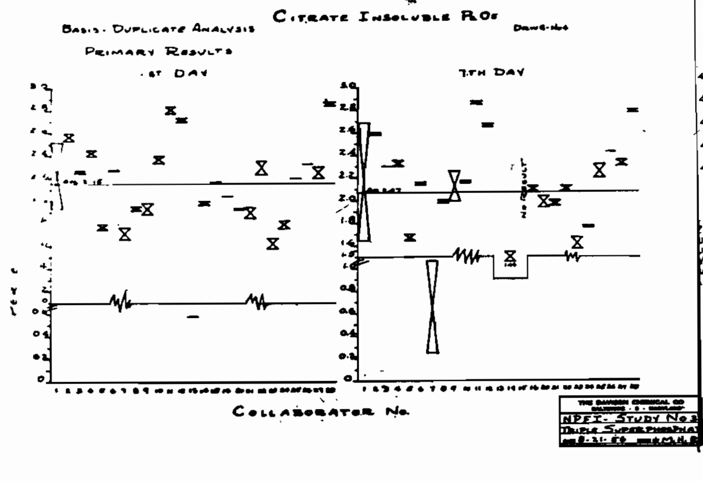

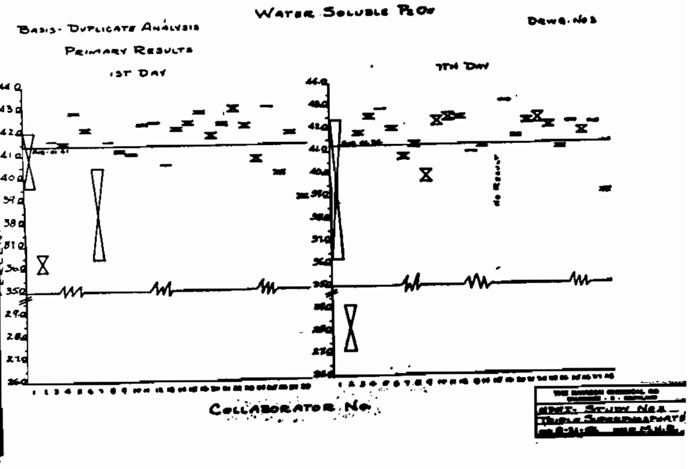

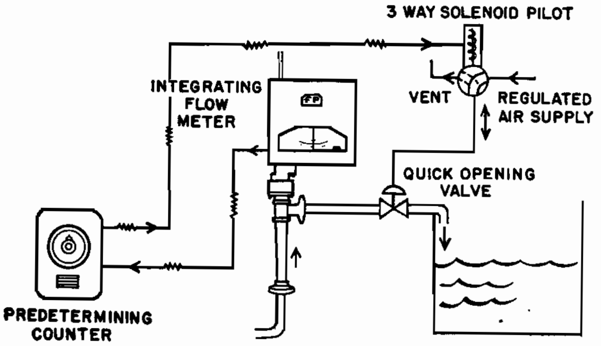

The single component batch control system measures the flow rate and volume of fluid passing through.

PROPORTIONING FERTILIZER INGREDIENTS

by Donald J. Tricebock

Fischer & Porter Co.

THE combination of a dust problem, continuous handling and mixing of highly corrosive chemicals, and the need to produce a satisfactory dry product create a not too simple materials handling problem for the fertilizer manufacturer. Ultimate customers do not often go to the trouble of making a chemical analysis of products, but from a quality viewpoint as well as that of the regulatory bodies in the fertilizer industry, it is essential that manufacturers produce a product that meets high quality standards. To do this, it is essential that each and every one of the ingredients involved in the production of fertilizer be properly measured, controlled, and proportioned one to the other. There are numerous ways of doing just these things, some of which are discussed below.

In a very simple form of continuous flow control, is a device which senses the actual flow rate of fluid in a pipe line. Having sensed this flow rate it becomes necessary to convert the value determined into some other useful measuring means.

In most cases simply holding a single stream to a constant value is not nearly enough control. It is necessary that several different materials be mixed in certain proportions one to the other. One method of doing this is by batch control. This system measures the rate of flow of fluid passing through it at any time and computes the total volume of fluid by means of a flow integrator, which is built right into the instrument. This integrator, besides keeping account of how much fluid has passed through the flow meter, also provides an electrical signal each time a certain amount passes through. For example, the flow integrator can be set to provide one electrical pulse for every ten gallons of liquid, or it can be set to provide one pulse for every gallon of liquid that passes through the meter. In any event, these pulses are sent to a device known as a predetermining counter. This counter accepts the electrical pulses and in turn keeps track of the number it has received.

Before starting the system it is necessary that the operator first determine how many gallons of this particular liquid are to be used in the batch. Having determined the number of gallons—or pounds or what-

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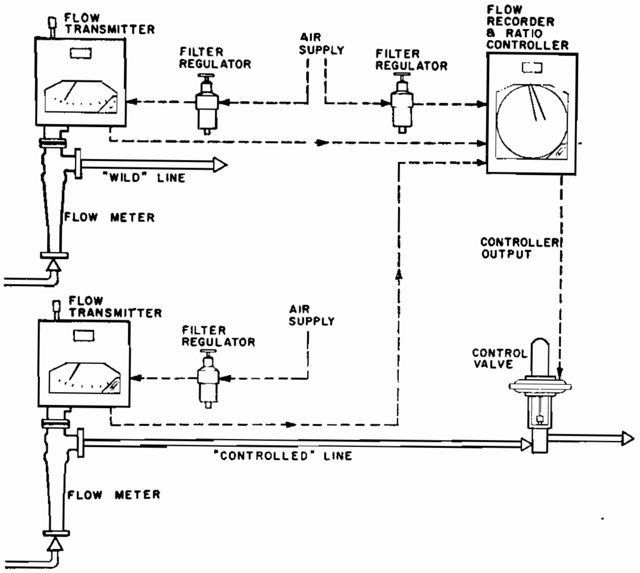
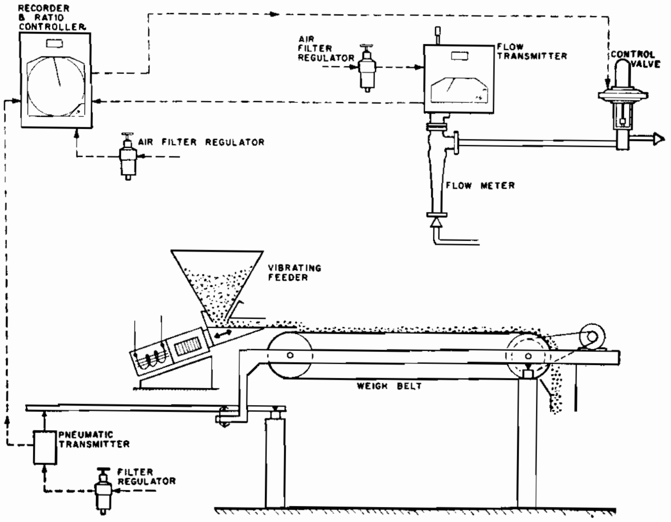
*Presented at the Fertilizer Industry Round Table, October 16, 1956, Washington, D. C.

The continuous ratio control system for two fluid streams, one of which is controlled so that its flow rate remains a fixed ratio of the other.



The ratio control system used on dry feed lines works with the same principles as the fluid stream system. The conveyor belt measures the dry material as it passes over.



ever the units may be, it is then necessary to determine, from the characteristic of the integrator, how many electrical pulses this represents. The predetermining counter is then set manually to a number on its face which corresponds to the number of electrical pulses that are to be expected from the flow integrator. It is a characteristic of the predetermining counter, that while it is receiving electrical pulses from the integrator it does nothing except count them until it reaches the number which corresponds to the predetermined amount that has been chosen as the required number of pulses for this particular batch.

Having received the correct number of pulses, the predetermining counter then operates a solenoid valve which may be used either directly to shut off the flow stream or may be used to operate a diaphragm motor valve, which in turn will shut off the flow stream. The important thing to note is that there has been a predetermined amount of the fluid passed through the pipe line before the valve shuts off the flow. An arrangement like this can be used for each ingredient required for the particular batch. This means that if we are mixing anhydrous ammonia, potash, sulfuric acid and water, there might be four separate systems, one for each of the fluids involved, and they could all be dumped simultaneously into the mixing container, or they might be dumped into it in a certain sequence, but regardless when all of these streams have been shut off we will have in the container a known amount of each of the materials, thus providing a controlled batch. This system has certain advantages over manual control, first, it provides precise proportioning of the ingredients, because measuring and counting are done automatically; second, it operates unattended after the push button is first used to start it, thus there is no need for a man to monitor the flowing streams, or to determine when sufficient fluid has been added; thirdly, any variation in flow rate within the pipe line does not affect the amount of the batch, since the integrator determines total quantity and does not concern itself with instantaneous flow rates.

The batch control system also has some disadvantages; one, it requires a man or some specific action to start the process; two, it requires mixing containers and storage capacities, since only a portion of the output is mixed at any one time; three, it sometimes takes considerable time to empty and refill the mixing containers.

These disadvantages can be overcome by going to a further refinement of the control system. It is completely possible to mix fluid streams in predetermined proportions on a continuous basis by using a device known as a ratio controller. Figure 2 shows a schematic diagram of a continuous ratio control system involving two fluid streams, one of which is controlled so that its flow rate remains a fixed ratio of the other. One stream is shown as a "controlled line" while the other, whose flow rate is not under control is known as the "wild line." The system operates as follows.

Again, we have a flow meter with a pneumatic signal output as described previously. In this case, the flow meter is measuring the rate of flow of a fluid through a particular line, and there is no need to control this flow in any special fashion except to be certain that it remains within range of the measuring device. The pneumatic signal from the flow meter this time goes to a ratio controller where it becomes one of the input signals. In this case, it is the wild signal, since it is not under control.

At the same time we have a second fluid stream whose flow rate is also being measured by a second flow meter. In the same manner a pneumatic signal is sent from this flow meter to the ratio controller. Here we are going to control the flow rate of the fluid, and hence this line is known as the controlled line. The ratio controller has had set into it manually a value which represents the desired proportion of these two fluids. For example, if it is desired to maintain the fluid in the controlled line at a flow rate which is one half that of the fluid in the wild line, the ratio controller would be set for 50%, or if it is desired to have the flow in the controlled line twice what it is in the wild line the ratio controller would be set for 200%.

This controller operates by comparing the ratio of the signals as received to the ratio as set into it by the operator. If the controlled line is not up to the required ratio, it will operate to change the setting of a valve in the controlled line to increase the flow rate. If the flow rate in the controlled line is too high, it will operate the valve in such a manner as to reduce the flow rate to the required proportion.

This system can even be used when handling dry material, and figure 3 shows such an arrangement with the dry material being the wild line. As can be seen, the dry material is handled through a hopper arrangement and out on to a conveyor belt which has a weighing arrangement supporting one end. This weighing arrangement incorporates a pneumatic transmitter, which provides a pneumatic signal proportional to the weight of the material on the belt. Since the belt is running and dumping off the material which is on it, it follows that the weight of the belt as measured by the pneumatic device, with proper zeroing and adjustments becomes proportional to the rate of feed of the dry materials.

This rate of feed can now be made the wild signal to one or more ratio controllers which will operate to maintain the flow rate of a liquid stream at a fixed proportion to the weight of the dry material being handled. A good application of this type of system occurs in the process of recycling fines. Here, of course, you have a typically wild situation, the amount of fines goes up and down during the course of a day and even from minute to minute. With this system you can effectively measure the rate at which fines are being recycled, and can control the addition of liquid materials so that maximum production can be achieved without causing a too dry mixture to provide a high percentage of fines, while at the same time preventing the mixture from getting too wet and thereby producing an unsatisfactory product.

All costs must be reduced and quality must be increased. These two requirements appear on the surface to be incompatible, but yet the manufacturer who achieves both of them and achieves them on a continuing basis will grow and prosper while his competitor who does not do these things will decline and disappear. I have shown you that instrumentation can help you to achieve this result.★★

CHARACTERISTICS OF NITROGEN MATERIALS

by J. Sharp

Spencer Chemical Co.

IMPORTANT characteristics of nitrogen materials, insofar as ammoniation and granulation are concerned, are shown in Table I and Figure I. They are listed as a convenient reference.

The examination of plant scale operating data, covering a wide variety of fertilizer grades indicated the granulation of mixed fertilizers, for most grades, was obtained at a range of from 33 to 35 per cent liquid phase. This concludes that our ability to determine liquid phase, based on the ideal solubility of the salts present, is sufficiently accurate to develop a theoretical method of study. For the purpose of this discussion, we define liquid phase as "The sum of the per cent salts in solution in the mix plus the per cent water at a given temperature, based on the ideal solubilities of each individual salt at that temperature." A figure thusly determined is not the accurate solution phase at all, but it does give us a tool with which to work and a means of calculating, or perhaps estimating conditions to shoot at in the ammoniator.

The relationships between raw materials, autogenous temperature, and required temperature for a 33 to 35 per cent liquid phase, graphically represented, allow determination of temperature-moisture requirements, in the TVA ammoniator, to produce the desired liquid phase.

Since recycle of fines is the commercial practice to granulate undersize particles and control the temperature and moisture, a step-wise procedure has been developed in order to calculate the recycle rate necessary to granulate a particular blend.

By plotting temperature vs. recycle, based on 12-12-12 operating data, the 35 per cent liquid phase falls along the top solid line and the 33 per cent liquid phase falls along the lower solid line. The actual operating point fell between the two solid lines at a temperature of 175° degrees Fahrenheit and a recycle rate of 2.5 to 1. Since the dryer temperature must be controlled to prevent the mix from becoming too hot and increasing the liquid phase in the dryer, reducing the moisture content to the proper level in the finished product has been a problem. To create more favorable drying conditions, a lower moisture content recycle was assumed. The dotted lines indicate the shift in 35 and 33 per cent liquid phase, based on a 1 per cent moisture recycle, as opposed to 2 per cent for the solid lines. The estimated operating point is at 215° degrees Fahrenheit, an increase of 40° degrees, whereas the recycle rate remains almost constant. This allows a 40° increase in dryer temperature also.

The obvious question then, is how can the moisture be lowered in the ammoniator so that higher temperatures may be employed. There are two ways that we know of and have used to some extent. One is to lower the moisture content in the raw materials. The other is to use a dryer recycle.

In commercial plant tests, it has been possible to reduce recycle about 50 per cent and to raise the ammoniator temperature about 25 degrees by using Spensol C-7 in place of Spensol C, or Spensol A-6 in place of Spensol A. Spensol C contains 16.6 per cent water, whereas Spensol C-7 contains only 7 per cent water. The ratio of free ammonia to ammonium

TABLE I

Comparative Tendency of Important Fertilizer Materials to Absorb Moisture*

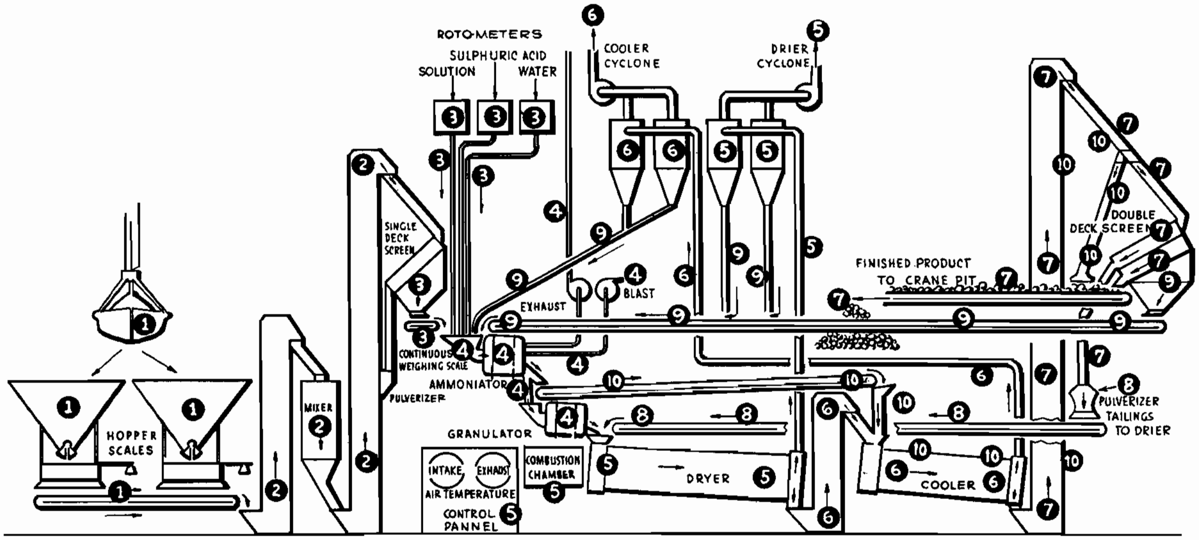
Material	Relative Humidity of air at 86°F (30°C) Above which material absorbs moisture (Critical Rel. Humidity)
Urea and ammonium nitrate	18.1
Ammonium nitrate and straight mono-calcium phosphate	38.6
Urea and nitrate of soda	45.6
Calcium nitrate, alone	46.7
Urea and common salt (as found in manure salts)	52.8
Urea and sulfate of ammonia	56.4
Ammonium nitrate, alone	59.4
Urea and muriate of potash	60.3
Ammonium nitrate and sulfate of ammonia	62.3
Urea and straight mono-calcium phosphate	65.1
Ammonium nitrate and muriate of potash	66.9
Nitrate of soda and common salt (as found in manure salts)	67.6
Manure salts, alone	72.2
Nitrate of soda, alone	72.4
Urea, alone	72.5
Sulfate of ammonia, alone	79.2
Muriate of potash, alone	84.0
Ammonium phosphate, alone	91.6
Mono-calcium phosphate (the pure compound, entirely free of acid)	93.7
Di-calcium phosphate, gypsum, limestone, sand and other insoluble materials	100.0

**Manual on Fertilizer Manufacture*, Second Edition, 1954, page 116, Vincent Sauchelli, Davison Chemical Company.

nitrate is held constant. Spensol A contains 12.8 per cent water and Spensol A-6 contains 6 per cent water with the same ratio of free ammonia to ammonium-nitrate.

The other way is to use hot recycle; provided that the hot recycle is also lower in moisture. Plants so equipped to return material exit the dryer directly to the ammoniator will need t ostart-up by putting material into the system and circulating it until it is hot and dry before raw material feed is begun. When this is done properly, the temperature in the ammoniator and dryer can be increased 30° to 40° degrees, which results in a much lower finished product moisture and better mechanical condition.

A continuing study on this approach is being made and supporting data on pilot plant and commercial scale production will be developed.★★



Flow Sheet for fertilizer granulation at FMCI

KEY TO FLOW DIAGRAM

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

(2) Mixing and Screening Unit—All of the dry materials are assembled by batch method and thoroughly blended through a Sackett Gravity 1 Ton Mixer. From the mixer each blended batch is dumped into a vertical bucket elevator and lifted to a single-deck Tyler-Niagara 4′ x 8′ screen. The screen (4½ mesh) screens out all of the foreign material, the oversize is put thru a Sackett #10 pulverizer for recycling. Screening conditions the dry batch for ammoniation.

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

(4) Continuous Ammoniator and Sackett Star Granulator—The rotary T. V. A. developed ammoniator is 7 feet in diameter and 7 feet long equipped with 15 H. P. variable speed motor. The rotary Sackett Star granulator is 6 feet in diameter and 8 feet long equipped with 10 H. P. variable speed motor. Vapors, principally moisture, generated by the chemical reaction in the ammoniator, are forced through a stack into the atmosphere by a suction fan equipped with 5 H. P. motor. Sometimes the mixture becomes too wet for rolling, and when this happens air is blown on the bed of material in the ammoniator to help in the drying. The fan used is equipped with 3 H. P. motor. A fair percentage of the granulation takes place in the ammoniator. The Sackett Star granulator helps considerably to give a greater proportion of on size material desired for the finished product, with a minimum of oversize and fines to be crushed and recicled.

(5) Drying Equipment — The damp granules flow from the Star Granulator by chute directly into the dryer. The rotary dryer is 6 feet in diameter and 50 feet long and is driven by a 50 H. P. motor. A co-current 15 million B.T.U. two burner natural gas-fired combustion chamber feeds the hot gases thru the dryer. The first several feet of the dryer has directional flights to move the material away from the intake of dryer, and from this point lifting flights move the material to the discharge end. Combustion air is furnished by a fan equipped with 20 H. P. motor. Moisture is removed from the dryer through a stack to the atmosphere. Dual cyclones recover the fine dust. A 75 H. P. motor is used to drive the cyclone fan.

(6) Cooling Equipment — Material from the dryer empties into a vertical bucket elevator, which feeds the cooler. The rotary cooler is 6 feet in diameter and 40 feet long, and is driven by a 40 H. P. motor. A counter-current air flow is used for cooling. The cooler flights are similar to the dryer flights. Additional moisture, during the cooling process is removed through the stack to the atmosphere. Dual cyclones recover the fine dust. A 60 H. P. motor is used to drive the cyclone fan.

(7) Product Classifying Equipment —The cooler discharges onto a conveyor, feeding the material into a vertical bucket elevator which lifts the product to a double deck 5′ x 15′ Tyler Hummer classifying screen. This screen gives 3 classifications: oversize, fines and finished product. The oversize passes thru a #15 Sacket double row hammer mill equipped with 15 H. P. motor. Crushed oversize can be returned to

elevator leg for reclassifying or if too damp can be returned to dryer for more drying. The fines are returned via belt conveyor to the ammoniator and the desired product flows on a belt conveyor to storage pit for crane to pick up to store to desired bin.

(8) Crushed Oversize—When necessary to redry the crushed material from the pulverizer, the material is dropped on belt conveyor feeding directly into the dryer.

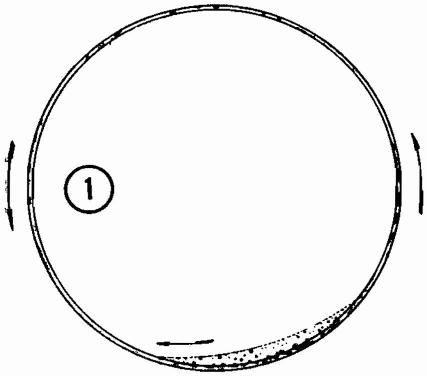
(9) Fines Returned for Granulation—Fines from dryer and cooler cyclones and from classifying screen flow on conveyor belt back to ammoniator.

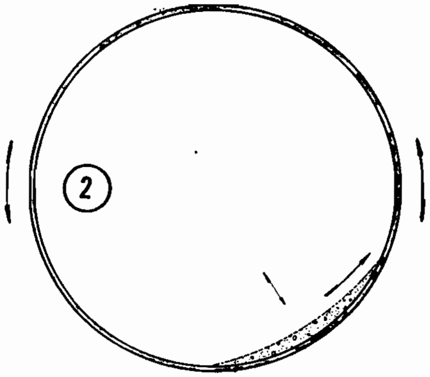
(10) Ammoniating and Cooling Powdered Mixed Goods—Conventional powdered mixed goods are batch-weighed and the dry materials required for the formula are assembled and blended similar to the granular dry materials. The T. V. A. ammoniator is used for blending the dry materials with the required nitrogen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

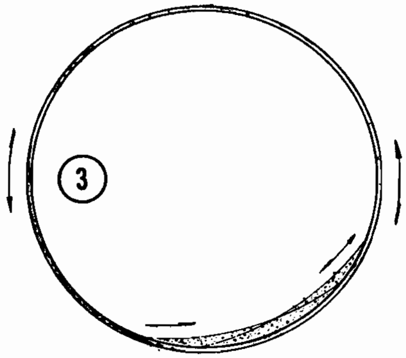
The Sackett Star Granulator

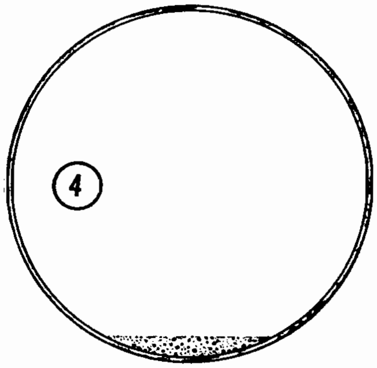
by Albert Spillman

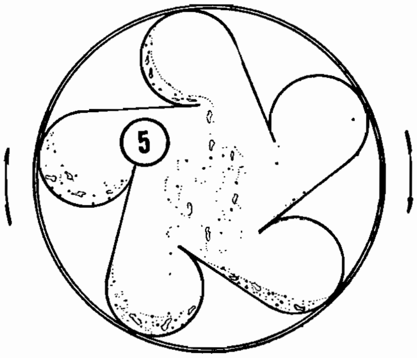
Fertilizer Engineering Mfg. Coop.
Baltimore, Md.

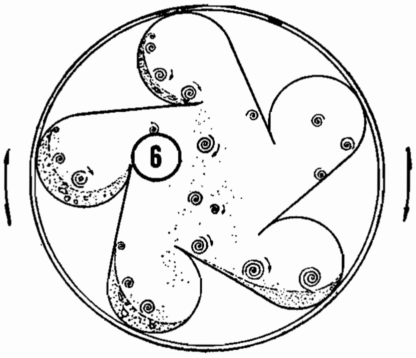


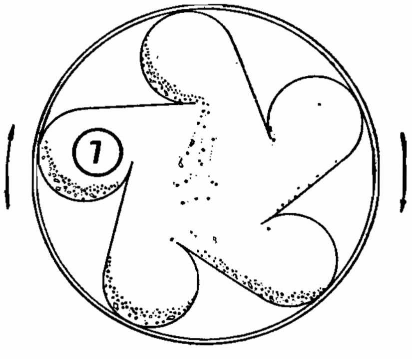


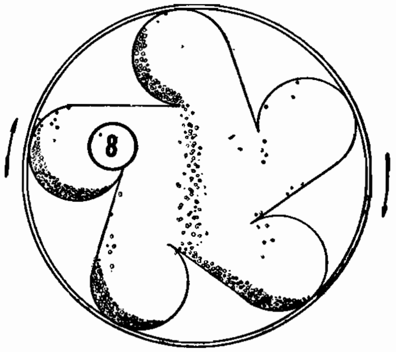












Conventional Granulator:
1. Material at bottom of slide.
2. Material at top of climb.
3. Composite view showing climbing and sliding action
4. Material at rest.

Sackett Star Granulator:

5. Initial action at feed end.
6. Intense rolling action.
7. Continued rolling action at mid-point showing snow balling into granules.
8. Completion of rolling action at discharge showing firmed-up granules ready for drying.

The Sackett Star* Granulator

*U.S. and Foreign Patents Pending

THE Sackett Star Granulator was installed at our (Fertilizer Manufacturing Co-op.) Baltimore plant, May 15, 1956. This report summarizes our actual working experience, and results obtained from this new machine.

All of us, manufacturing granular fertilizers, have at times taken hands full of the hot ammoniated damp material direct from the ammoniator to roll from palm to palm obtaining good conditioned granules. In principal, the working characteristics of the Star Granulator are identical. Frankly, we were somewhat concerned, before installing this new machine, fearing the damp ammoniated mass flowing from the ammoniator into the Star Granulator would gum up the rolling trays and cause a continuous cleaning problem. Our fears did not materialize. The 15 heavy steel balls, 3 for each rolling tray, inserted between the steel shell of the granulator and the steel shell of the rolling trays, tap sufficiently to keep the equipment self cleaning.

We have operated the Star Granulator at Fertilizer Manufacturing Co-op. five months and are very happy with the outstanding results obtained. Comparing our experience with the conventional granulator, we found that the Star Granulator produces a more uniform, closer sizing of granules, less fines to recycle, less oversize to be crushed, no plugging in chute feeding our dryer, less water added to aid granulation, more efficiency in the dryer and cooler, lower drying costs for fuel and, most important, higher product recovery.

Complete operating control records have been kept of our granulating operations since we started manufacturing 18 months ago, therefore, we are well qualified to give the results obtained from the Star Granulator versus the conventional granulator. The studies made are real and factual data. On each 8 hour shift of our granular operation, a detailed record is kept of temperatures at intake and exit of dryer, exit of cooler, finished product to storage, ammoniator and granulator product and air intake. Also recorded are formula used, water added, cubic feet of gas used, screen tests of the cooled granulator material before classifying to give us the record of product recovery, fines to be returned, oversize to be crushed, final product to storage, weather conditions and other miscellaneous data.

To be absolutely fair with our comparisons of recovery results obtained from the Star Granulator, careful consideration was given to the method used to establish the data— to be sure nothing was done to exaggerate a favorable report for the Star Granulator or an unfavorable report for the conventional granulator. We decided to make the study, using 10-10-10, 8-16-16, 6-18-18 and 6-12-12 grades. These four grades represent about 95% of the total tonnage of all grades of granular shipped by Fertilizer Manufacturing Cooperative. We picked at random 10 reports of our day-to-day operation of each grade, and averaged the results shown 12 thru 16.

I shall not get into any controversy as to whether granulation operations should have both the ammoniator and the granulator, because about 50% of the operators I have had contact with state definitely that even the conventional granulator is effective, and 50% state that they can see no benefits. I, personally, am satisfied the granulator offers real benefits and helps considerably to do a better job.

Before installing the Star Granulator, we were classifying our product with 4 mesh top and 35 mesh bottom standard opening screens. Soon after installing the Star Granulator, our recovery improved to permit changing our classifying recovery screens to 4 mesh top and 24 mesh bottom standard opening screens. We are now confident we can use closer sizing, and plan to change within a few weeks to 5 mesh top and 20 mesh bottom standard opening screens.

The Sackett Company, in my opinion, have made an outstanding revolutionary contribution in the development of the Sackett Star Granulator, and we were very happy to do the major experimental plant operating work.

In the past year, we have added a gas hot-water system for furnishing up to 4000 lbs. of 180 degree heated water for use in ammoniation, when water is required mostly in lower nitrogen granular formulas. Adding hot water versus cold water reduces water to be added, and saves fuel to drive off excess moisture.★★

10-10-10
GRANULAR

Screen Tests Represent Average of 10 Samples	Conventional 6' x 8' Rotary Granulator	6' x 8' Rotary Sackett Star* Granulator	Percentage Change with Sackett Star* Granulator
ON 4 Mesh	15.622	11.193	—28
ON 10 Mesh	34.110	42.637	+25
ON 20 Mesh	17.404	23.427	+35
ON 24 Mesh	3.704	4.335	+17
ON 35 Mesh	12.489	11.888	— 5
ON 100 Mesh	15.159	9.425	—38
THRU 100 Mesh	1.252	.095	—92
Recovery Before Classifying			
Minus 4 Mesh Plus 20 Mesh	51.514	66.164	+28
Minus 4 Mesh Plus 24 Mesh	55.218	69.868	+26
Minus 4 Mesh Plus 35 Mesh	67.707	81.756	+21

U. S. and Foreign Patents Pending.

8-16-16
GRANULAR

Screen Tests Represent Average of 10 Samples	Conventional 6' x 8' Rotary Granulator	6' x 8' Rotary Sackett Star* Granulator	Percentage Change with Sackett Star* Granulator
ON 4 Mesh	8.053	3.186	—60
ON 10 Mesh	29.021	34.581	+19
ON 20 Mesh	19.730	27.484	+39
ON 24 Mesh	5.093	7.064	+39
ON 35 Mesh	18.887	19.642	+ 4
ON 100 Mesh	18.283	8.043	—56
THRU 100 Mesh	1.032	....	—100
Recovery Before Classifying			
Minus 4 Mesh Plus 20 Mesh	48.751	62.065	+25
Minus 4 Mesh Plus 24 Mesh	53.844	69.129	+28
Minus 4 Mesh Plus 35 Mesh	72.731	88.781	+22

* U. S. and Foreign Patents Pending.

6-18-18
GRANULAR

Screen Tests Represent Average of 10 Samples	Conventional 6' x 8' Rotary Granulator	6' x 8' Rotary Sackett Star* Granulator	Percentage Change with Sackett Star* Granulator
ON 4 Mesh	5.626	4.364	—22
ON 10 Mesh	21.596	40.182	+86
ON 20 Mesh	36,287	40.545	+12
ON 24 Mesh	6.259	5.818	— 7
ON 35 Mesh	14.058	7.455	—47
ON 100 Mesh	15.331	1.636	—89
THRU 100 Mesh	.843	....	—100
Recovery Before Classifying			
Minus 4 Mesh Plus 20 Mesh	57.883	80.727	+40
Minus 4 Mesh Plus 24 Mesh	64.142	86.545	+35
Minus 4 Mesh Plus 35 Mesh	78.200	94.000	+20

* U. S. and Foreign Patents Pending.
** NOTE:—Coarse Potash Used.

6-12-12
GRANULAR

Screen Tests Represent Average of 10 Samples	Conventional 6' x 8' Rotary Granulator	6' x 8' Rotary Sackett Star* Granulator	Percentage Change with Sackett Star* Granulator
ON 4 Mesh	8.072	4.441	—45
ON 10 Mesh	29.528	42.980	+45
ON 20 Mesh	29.729	36.103	+21
ON 24 Mesh	13.982	4.369	—69
ON 35 Mesh	12.095	8.955	—26
ON 100 Mesh	7.670	3.152	—59
THRU 100 Mesh	.294	....	—100
Recovery Before Classifying			
Minus 4 Mesh Plus '20 Mesh	57.257	79.083	+38
Minus 4 Mesh Plus 24 Mesh	71.239	83.452	+17
Minus 4 Mesh Plus 35 Mesh	83.334	92.407	+11

* U. S. and Foreign Patents Pending.
** NOTE:—Coarse Potash Used.

Research on Paper

by K. A. Arnold
St. Regis Paper Co.

A continuation of comments on bags and bagging, see pages 24, 28, 29.

A further discussion of research activities in the pulp, paper and packaging industry was presented by K. A. Arnold, who advised that general objectives in research and development are:

1. To develop new processes to maintain the profit margin between rising manufacturing costs and stable selling price.
2. To develop new products to diversify and expand into more profitable fields, in order to increase company income and to increase our services to customers.

Mr. Arnold reported that recent research has produced frictionizing coatings which prevent stacks of bags from slipping on trucks and in warehouses. New types of end closure for sewn bags, which prevent transmission of moisture through the needle holes, have included heat sealed polyethylene coated paper closures and pressure sensitive tapes. Such closures, he said, may also prevent siftage of contents and infestation by insects. The development of the pasted moistureproof bag for fertilizer has resulted in greater economy, with equal or better moisture resistance. Recent developments in stepped end bags give possibility to a completely moistureproof bag at a reasonable cost. Specifications for bag constructions for over a thousand new products have been issued in recent years as the result of chemical tests made on the product to be packed.

Among newer developments, still in laboratory stage, are the plastic stitch to replace present moisture permeable sewn ends, a tear strip for the pasted bag, which enables easy opening of the filled bag; development of foam plastic, which will result in better insulating bags; and the eventual development of an all plastic bag.