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SCOTTISH AGRICULTURAL INDUSTRIES LIMITED

PRODUCTION OF GRANULAR COMPOUND FERTILISERS.

NOTES ON THE DEVELOPMENT AND OPERATION OF COMPOUND
FERTILISER GRANULATION IN S.A.I.'S WORKS IN SCOTLAND.

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NOTES ON THE DEVELOPMENT AND OPERATION OF COMPOUND
FERTILISER GRANULATION IN S.A.I.'S WORKS IN SCOTLAND.

1. The production and use of fertilisers in granular form is a subject of very general interest at the present time in the fertiliser industry, and not least in that part of the industry which is concerned with superphosphate and with complete compound fertilisers containing superphosphate as the main phosphatic ingredient. In this paper we present some particulars of our experience at a number of our works in Scotland in introducing and operating commercially plant for the manufacture in granular form of complete compound fertilisers based on superphosphate. Much has been said and written on the merits and demerits, agronomic and otherwise, of fertilisers in granular form, but little has been published - apart from American practice in the granulation and waterproofing of ammonium nitrate - in the way of works operating data and works scale production problems. It is hoped therefore that a simple and straightforward account of our experience in Scotland may prove of interest and of benefit to manufacturers of superphosphate and compounds, whether already operating granulation processes or not, and that this paper will serve to encourage other manufacturers to contribute in due course of their experience to the general fund of knowledge of this subject available to the members of the International Association and to the industry generally.

It is necessary at the outset to make clear that in what follows no attempt is made to present as-it-were a scientific treatise on the subject of the granulation of compound fertilisers based on superphosphate. Our own developments in this field, which were seriously delayed by the outbreak of the war, were based on a somewhat empirical approach to the subject. We do not think we were alone in this, and indeed it may be suggested that most of the fundamental scientific studies of the physical and chemical changes which occur in the process of granulation of materials containing superphosphate have still to be carried out. It is to be hoped that in the course of the next few years considerable progress will be made in such studies. Furthermore we would not claim that we have either greater knowledge or more experience than many other manufacturers of granular fertilisers. Nevertheless any practical experience is likely to be of interest, and we can at least claim the experience of operating commercially some five plants, each with slightly varying characteristics, and to have produced large tonnages of granular compounds.

2. In Great Britain the production and use of factory-made compound fertilisers containing nitrogen, phosphate and potash in proportions designed to suit particular soils and crops is wide-spread. Approximately three-quarters of the superphosphate used by British farmers is bought by them in such form. In the 1920's most compounds were made by a dry-mixing process, the superphosphate and other manurial and conditioning constituents being mixed together, stored in bulk to cure, and later recovered from bulk store, milled, bagged and despatched. The principal nitrogen constituent was sulphate of ammonia, and this forms ammonium phosphates and double salts - syngenites - with the calcium sulphate present in superphosphate. The formation of ammonium phosphates and double sulphate salts, etc., takes place during curing and leads to the mass setting hard. Furthermore, the mixture may set again after milling, and for this and other reasons it was the normal practice to defer milling and bagging as far as possible until despatch to farm was called for. Compound fertiliser set into a single concrete-like block in a bag is little use to a farmer!

These facts are well-known, but are recapitulated here so as to draw attention to one point about granulation which is important in the technique of manufacture of granular compounds. Over and above all the well-known/

well-known factors of drillability, cleanliness in handling, possible superior agronomic efficiency in respect of greater ease of placement and possibly also of greater phosphatic "availability" under certain conditions, etc., which make the production of fertilisers in granular form interesting and attractive, there exists the possibility of making use of the natural "set" of a compound to secure a hard granule which will stand rough handling. In making a powder compound, the aim is by use of conditioners to reduce the setting tendency of the mass. By adopting the technique of granulation it is possible to turn that setting tendency to advantage and consequently the aim is to create the conditions where setting will be at a maximum and to secure that setting within each granule.

Granular compounds first appeared on the British market in substantial tonnages about 1931/1932. These were based on ammonium phosphate, not superphosphate, and it so happened that these fertilisers could more easily be produced in granular than in powder form. The attractiveness of the granular form was soon recognised, and it was not very long before makers of compounds based on superphosphate were considering granulation. The first large scale production of granular compounds containing superphosphate was inaugurated in the middle 1930's, and now rather more than half the total British production of compound fertilisers is in granular form, and the proportion is increasing steadily.

3. There are, of course, many ways of producing a granular material. In the cases of superphosphate and compounds probably the simplest way is to wet, roll or tumble by appropriate means, and then to dry off surplus moisture, if any. The formation of the granules by rolling can be arranged to proceed as part and parcel of the wetting operation, or as part of the drying operation, or partly at each stage, or as an entirely separate operation. In the case of compounds a mixing process is also involved. This may be essentially the dry mixing of superphosphate and other manurial ingredients followed by wetting, rolling and drying, or the wet-mixing of phosphate rock, sulphuric acid, and other manurial ingredients, in which case a separate wetting stage is usually unnecessary, followed by rolling and - usually - drying. The former process is that described hereafter, and the latter process is exemplified by the Proctor Process. Variants of each and combinations of the two are of course also possible.

4. Our own preliminary experimental work was started some ten years ago, using a horizontal pan mixer 3'3" diameter, 1'6" depth and 150 lbs. capacity. The pan, which was fixed, was fitted with a central shaft carrying a spider on which were mounted two outer paddles rotating clockwise and feeding towards the centre. There was also a small star paddle rotating in a counter-clockwise direction. The pan was fitted with a Monarch spray above, for wetting the mixture. The ingredients were placed in the pan, and uniformly mixed by the rotation of the paddles, then the water was turned on and granulation proceeded simultaneously with wetting, the movement of the paddles providing the requisite rolling or tumbling action to cause the formation of granules.

Encouraging, but not satisfactory results, were obtained with this apparatus. For example, a granular compound, approximate analysis 4,7,4,7 was produced from sulphate of ammonia, superphosphate, ground phosphate rock and potash salts with a water addition of 10% and a mixing-wetting-granulating time of seven minutes, of which 65% consisted of granules between 1 and $3\frac{1}{2}$ mm. diameter. This figure of "granulation efficiency" (65%) did not seem high enough and the granules were neither smooth as to surface nor spherical in shape. It had however been established that granule size varied directly with paddle speed, with time of mixing, and also with amount of water added.

5. Experiments were continued with a larger horizontal pan-type mixer granulator. The apparatus chosen was the "Gunflow" concrete mixer made by the Limer Concrete Machinery Co. Ltd. This machine was similar to the German/

Gorman "Eirich" mixer which was known to be a successful mixer-granulator. The pan, 5'0" diameter and 1'3 $\frac{1}{2}$ " depth, itself revolved at 12 r.p.m., and was provided with a mixing star carrying three independently sprung blades which revolved at 70 r.p.m. in the same direction as the pan. (The mixing star in the Eirich mixer revolves in the reverse direction.)

It is worth noting that this type of mixer is very efficient as a mixer of dry materials. It was established that a completely homogeneous mixture was obtained in 15 to 30 seconds. Consequently the operations of mixing, wetting and granulating can be carried out simultaneously, and this is normal practice on granulation plants using horizontal pan type mixer-granulators.

A long series of experiments was carried out, with the mixing star rotating in the same direction as the pan and also with mixing star rotating in the opposite direction, and the experimental results may be tabulated as follows:-

1. Better granule appearance and lower water usage is secured by rotating the mixing star in the reverse direction to the pan.
2. For any particular mixture and speeds of rotation of pan and paddles, the size of granules produced varies directly with the amount of water added, and is independent of the speed at which the water is added, up to a critical point above which massive lumps are formed which do not break down on continued mixing.
3. Granulation may be effected either by slow addition of the requisite quantity of water and gradual building up of granules to the desired size, or by fast addition of water, formation of large masses of plastic material and subsequent gradual breaking down to the desired size. The latter system gives a higher proportion of oversize and a smoother rounder granule. Since the oversize can be broken down to give further quantities of granules within the desired size range, a greater ultimate yield can be secured by fast addition of water.
4. Variation of particle size of the ingredients of the mixture does not markedly affect the yield of granules of the desired size, but directly affects the water requirement - the finer the ingredients, the more water is required.
5. On the other hand addition of fine superphosphate or compound fertiliser dust to the granules in the pan, once these have been formed, has the effect of breaking down aggregates and increasing the yield of granules some 5/10%.
6. Other things being equal, granule size varies directly with the time of mixing and with the speed of rotation of the pan and paddles.
7. Mixtures of dry ingredients are more difficult to granulate than those which have some moisture present.
8. Other things being equal, the yield, appearance and hardness of the granule varies with the chemical composition and physical condition of the mixture to be granulated.
9. Generally the analysis of the granular product varies with the size of granule, the fine particles being higher than average in N and lower in P₂O₅ and the large particles lower than average in N and higher in P₂O₅.
10. An overall granulation efficiency of about 80% (after cracking the oversize) can be secured.

One other point of interest was noted. The reactions during the granulation process are exothermic, and the product warms up slightly. The granules are self-hardening and on standing some surface drying takes place. Artificial drying did not appear to be necessary to secure the requisite degree/

degree of hardness of granule, but the self-drying obtainable was not far enough to permit of efficient screening out of the granules of the desired size under practical conditions of working. It was concluded that an artificial drying stage could not be avoided for works scale operation unless a direct yield of 100% granules of desired size could be obtained.

We had thus verified for ourselves that a practicable process of granulation of compound fertilisers could be introduced using the horizontal pan type of mixer granulator, and probably having to dry the product artificially prior to selection by screening of the desired size range. It was clear however that the exact disposition and speed of the paddle gear was critically important in securing high granulation efficiency and that the design of the discharge arrangements was important in securing an economic output. Furthermore, that optimum spray water requirement varied with the chemical composition and physical condition of the mixture being granulated and that in practice good granulation would prove to be an art dependent on the skill and experience of the operator.

6.

At this stage in the work the war broke out, and the decision was taken to defer the installation of full scale plant based on the experimental results obtained so far, so as to concentrate all our energies on securing from existing plant a large and rapid expansion of fertiliser production, which was called for to meet the requirements of the war situation. Experimental work was not discontinued, but returned to the laboratory and was directed to further investigation of the wet process for production of granular compounds - work which had been started a little before the outbreak of the war. In this method which has already been referred to, one starts with sulphuric acid and ground phosphate rock instead of with superphosphate, and the hot damp mixture of these and the other manurial ingredients is granulated. This method appeared to have advantages in that the manufacture, storage and handling of superphosphate for compounding would be obviated, and in that the mixture would be chemically more homogeneous and physically in good condition for easy granulation.

We used Kalaa Djerda rock ground to pass 80% through 100 B.S.S., sulphate of ammonia, muriate of potash, and sulphuric acid of varying strengths from 60% to 72% H_2SO_4 and an SO_3 /phosphate ratio of 48/100. Mixing was carried out in beakers held in a water bath, granules formed by stirring, drying carried out in a hot air oven, and the sample tested and bottled for subsequent examinations. In general best results were obtained with 69% acid, and conversions to water soluble P_2O_5 were around 75% immediately after mixing. The conversion improved on storage and after one month had risen to 88/89%, while after six weeks some samples showed conversions of around 91/92%. The conversion to water solubility was thus somewhat less than in the manufacture of straight superphosphate, but this is not necessarily a defect in the case of compounds required to contain some slow-acting phosphate. The granules made by this process were most attractive in appearance and it was confirmed that they were reasonably homogeneous chemically.

Experimental work in the laboratory having shown that the process had possibilities we were anxious to erect semi-scale plant in one of our works to try it out, but unfortunately our Chief Engineer died just about this time and under war-time conditions of shortage of technical staff and high production commitments we had to abandon all ideas of any immediate works-scale development work.

7.

By 1943 the problems of building up fertiliser production to high levels had been overcome, and it was possible to consider the introduction of a full-scale granulation process. But the high level of factory production and the continued shortage of technical staff did not permit of a resumption of development and design work on a technical scale. It was therefore/

therefore decided to proceed immediately to the installation of a complete plant offered by a firm of chemical plant manufacturers, in which the granulation was effected by wetting and rolling in a rotary tube. This gave an opportunity of comparing the efficiency of a rotary tube granulator with the horizontal pan-type mixer-granulators for which we had experimental experience. The plant, of rated output capacity 5 tons per hour, was connected up to an existing dry-mixing plant from which the mixed dry batch of 1 ton was fed through a smoothing hopper. From this hopper the mixed ingredients passed through a four cage Carr's type mill to the granulation tube - this was 15' long by 4' in diameter inclined at an angle of about 3° and rotated at a speed of 12 r.p.m. It was provided with some six water sprays at the inlet end and a number of rapping hammers mounted on the outside of the shell at the discharge end. From the tube the granules fell into a co-current drier 38'6" long by 4'6" in diameter, the hot gases for which were supplied by a coke furnace. The drier discharged into an elevator feeding the top of a multi-tray cooler rather like a Herreschoff furnace in design. From the bottom of the cooler the granules were again elevated to a double decker screen mechanically vibrated by hammers operated by a series of cams, where oversize and fines were removed. The oversize was broken through a twin roll breaker (one roll being a smooth drum and the other built up of flat bars) and returned to the elevator feeding the screens. Fines were collected and returned to the batch. The gases from the drier and cooler, each 6,000 cubic feet per minute at N.T.P., were exhausted through 7' diameter asbestos cement cyclones and after passing through the fans were blown through asbestos cement wash towers to the atmosphere.

The tube type of granulator proved in working to be successful, the granules being attractively smooth and round. Granulation efficiency was less than we had hoped - about 65% to 75%, with about 5/7% oversize and the balance fines. (This is, however, as good as we have achieved since with pan mixer-granulator plants.) On the other hand many troubles were experienced with the plant as a whole.

- (a) The drier and cooler were both seriously under-capacity, as we learnt from the setting and bag rot troubles which we experienced as a result of bagging material which contained 6/8% moisture and was some 30/45°F above atmospheric temperature.
- (b) Spillage at the feed end of the drier, from the cooler trays and from conveyors and chutes was excessive.
- (c) The oversize breaker had too little clearance between the rolls and the casing, and a hard cake of fertiliser built up and jammed the rolls.
- (d) The double decker screen of necessity has the oversize screen on top. The fines screen below was not sufficiently accessible for cleaning and serious blinding was experienced.

These points are mentioned to illustrate the need for great care in detail design to secure satisfactory and steady working. The first point may usefully be discussed in greater detail:-

Drier. The average moisture content of the granules before drying was 14%. Allowing for the fines which are recycled and which dry out quickest, it is found by calculation that the water to be removed to give a net output of 5 tons per hour is as follows:-

| | |
|-------------------------|------------|
| Granules at 4% moisture | 0.70 tons |
| " " 5% " | 0.63 tons |
| " " 6% " | 0.57 tons. |

At best the plant would dry down to 6%. To give 5% required an increase in drier duty of about 10%, and to give 4% the increase required is 23%. This illustrates the need to have an adequate margin of drier capacity.

Cooler. The tray type of cooler proved in our experience to be unsuitable for granular compounds, and better results were later secured by replacing it with/

with a rotary tube cooler, equipped with lifting flights.

Apart from such mechanical and design lessons the most interesting point discovered in the operation of this plant was that a fine crystal form of sulphate of ammonia in the mix required much more spray water than was the case using large crystal material to give granules of the desired size. On changing from large to fine crystal size the spray water requirement increased by some 20%, and the drier duty correspondingly - the moisture content of the feed to the drier increasing from 14% to over 16%. The importance of this in terms of drier capacity and fuel consumption per ton will be clear.

To overcome the defects and difficulties described above substantial changes were made in the plant. The drier was replaced by one 50' long by 6'7" in diameter fired by a coal fired furnace equipped with an underfeed type automatic stoker thermostatically controlled by the gas temperature at the outlet of the drier, and draughted by a fan with a capacity of 12,000 cubic feet of air at N.T.P. The cooler was replaced with a horizontal tube type cooler 30' long by 6' in diameter fitted with internal lifting flights and draughted by a fan with a capacity of 6,000 cubic feet per minute. The increased draught on both drier and cooler required new cyclones and these were of the high duty Terlindan design. A new rotary oversize screen, a new mechanically vibrated inclined fines screen and a new oversize breaker were all necessary. The changes have improved working considerably and the plant has been in continuous steady working, 24 hours a day apart from routine stoppages, ever since. Performance data for a typical month's work are given in Appendix I, and the essential features are as follows:-

| | | | |
|--|--------------------|---------------|-------|
| <u>Product:-</u> | Size analysis - | + 3.35 mm. | 2.7% |
| | | 3.35/1.00 mm. | 85.0% |
| | | - 1.00 mm. | 12.3% |
| | Moisture content - | | 5.2% |
| | Temperature - | | 80°F. |
| <u>Output Rate:-</u> | tons per hour | | 5.1 |
| <u>Services Usages:-</u> (per ton of product) | Water - | tons | 0.071 |
| | Coal - | tons | 0.030 |
| | Electric Power - | KWH | 10.73 |

This plant - the first installed - is not yet fully satisfactory and further improvements are scheduled. The product size analysis should show 100% between 3.35 and 1.00 mm. and the figure of 85% just reported is an indication of screen inefficiency. Furthermore drying and cooling still leave something to be desired to minimise after-setting and bag rot - problems which will be discussed more fully later on.

One interesting point was noticed during the early working of this plant, before the original drier had been replaced. Some of the first granules produced were particularly smooth and round, but they had an exceptionally high moisture content. When examined under a microscope it was found that the outer skin of the granule was fused and this fused skin served as an impermeable membrane through which the moisture could not diffuse. At this time the gas inlet temperature to the drier was around 12/1300°F and the granules quickly fused on the outside and thereafter were almost impossible to dry. The secondary air to the furnace was increased, the inlet gas temperature was decreased to around 900°F and thereafter much better drying was obtained though the product was not so attractive in appearance.

8. For our second plant, also of 5 tons per hour capacity, it was decided to use a pan type mixer granulator. This was along the lines of the Eirich mixer but made by a British manufacturer. We had experience of the tube type granulator on the works scale, our own experience of working the pan type granulator was obtained on a semi-scale unit and we wished to compare the working of two full scale units. The use of a pan type granulator has the attraction that it serves to mix the dry ingredients simultaneously with granulation so saving one item of plant and probably one man/

man compared with the tube type unit.

The plant is now described in detail:-

The plant consists of a preliminary breaking arrangement designed to break down the ingredients to be granulated, comprising a receiving hopper 6' by 4' by 3' deep covered by a 3" mesh grid through which the batch is fed. A belt-feeding conveyor 24" wide 11'0" centres moves the material to the boot of an inclined bucket elevator 37'0" centres with a single strand of No. 503 chain running at 90 feet per minute and carrying 12" steel buckets fitted with skidder bars. This elevator discharges the ingredients into a four cage Carr type disintegrator. This machine has all-steel rotors each rotor consisting of two rows of $1\frac{1}{8}$ " diameter bars about $4\frac{1}{2}$ " pitch. The diameter over the bars of the outer casing is 4'7" and the width 16". The running clearance between cages is $1\frac{1}{2}$ ". The outer rotor sleeve and the inner rotor sleeve run at 950 revs. in self-aligning ball bearings of a type which take not only radial but thrust loads and each rotor is driven by Vee ropes from independent motors each of 14 H.P. The side casing is of $\frac{3}{16}$ th inch sheet steel and the top and ends (which are easily removed for cleaning) are of $\frac{3}{8}$ " plywood. There is an air balance pipe from the bottom to just above the centre on the inlet side of the casing.

From the disintegrator the material falls into a batch hopper which holds rather more than one ton. When the whole batch is collected in this hopper it is dropped into the skip of a skip hoist and elevated to the pan-mixer.

The skip has a capacity of 20 cwts. fertiliser and has a hoisting speed of about 45 feet per minute. It takes 2.2 minutes to fill, hoist, discharge and return empty. Operation is by push button from the mixer platform. The skip is fitted with flanged rollers running in channel section guides and is raised and lowered by steel ropes running over large diameter guide sheaves to a winch on the lower platform. The winch has machined barrels, and enclosed fan-cooled worm-reduction gear mounted with the 5 H.P. motor and electric brake on a cast iron bedplate. Additional wire ropes are led from the winch barrels to balance weights arranged to slide in steel guides and these weights are so arranged that when the skip is discharging into the mixer the weights are automatically reduced to compensate the varying load on the winch.

The skip discharges the batch into an Elrich type "Liner" Mixer. This has a 9'3" diameter pan 1'7" deep and running on adjustable ball bearing trunnions. The pan is fitted with renewable side and bottom wearing plates and rotates in a clockwise direction at 6 r.p.m. It is fitted with two mixing stars which rotate in a counter clockwise direction at 43 r.p.m. These stars each have three spring loaded arms, two carrying ploughs and one carrying a fork. The discharge from the pan is through a power-operated central door. This door is mounted on a carriage below the pan so that it can be moved out of the way of the material when it is being discharged. The drive to the Mixer is by Vee rope to countershaft and thence by machine cut gears to pan and stars. A spray-pipe with Monarch type sprays is used for the addition of water, a Centrifugal pump with 10 gallons per minute capacity is used to supply water at constant pressure and the water is measured through a Wayne gauge which shows gallons and half gallons and has an integrating counter.

After the batch has been granulated in the mixer it is dropped on to a horizontal feeder conveyor which moves it to the drier. This conveyor is of 24" wide rubber and canvas belting and is supported on close-pitched tubular idlers having steel spindles running in enclosed end eye bearings. Sheet steel decking protects the return side of the band. The terminal pulleys are of large diameter; steel shafts running in grease lubricated bearings with adjustable brasses are provided for the head end countershaft, whilst the tail shaft runs in bushed swivelling bearings with bronze screws for adjusting the tension in the belt. The belt does not run continuously but is moved forwards intermittently by a ratchet arrangement on the head pulley shaft. The whole conveyor is housed in with sheet steel.

From the conveyor the granules are dropped down a chute into the drier. This chute has a damper which can be inserted to prevent overheating the/

the belt should the plant be off while hot gases are still passing through the drier from the furnace.

The drier is 50' long by 5' internal diameter and is built of 5/16th inch thick steel plate, suitably insulated. The insulation is protected by sheet steel covers. The shell is supported by steel roller paths running on cast iron rollers with hard steel tyres. Thrust rollers prevent any lateral movement of the shell. The internal lifters, conveyors and baffles, of which there is quite a maze, are of 1/2" steel plate. A steel spur ring specially attached to allow for expansion is used to drive the drier. This spur ring mates with a steel pinion mounted on a counter: shaft coupled to a totally-enclosed-fan-cooled worm-reduction gear. Both the supporting rollers and the motor and gear are mounted on a combination bedplate. Looking at the feed end the drier rotates in a clockwise direction at 4.7 r.p.m. It is inclined at an angle of 3° and material takes 20/30 minutes to pass through.

The hot air intake to the drier is heated by a coko furnace with a grate area of 30 square feet. The draught for the furnace and drier is obtained from a paddle type radial-bladed fan of 42' diameter. The impeller shaft is carried in ball bearings and is driven by Vee rope from a 45 H.P. S.R. motor. The capacity of the fan is 12,000 cubic feet per minute and it is protected by a thermostatically controlled false air inlet. There is also a damper which helps to control the draught on the furnace and drier. From the fan the gases pass to two Buell dust cyclones which collect the dust picked up in the furnace and drier. Platinum resistance thermometers recording on a thermograph show the gas temperatures at the inlet and outlet of the drier.

From the drier the granular material falls to the boot of a vortical elevator. This elevator is of the dumper type with 12" buckets of 10 gauge sheet steel carried at intervals of 15" on two strands of No. 503 Gray pin type chain running at approximately 80 feet per minute. The sprockets are of special hard cast iron and in order to improve the delivery of the material the down-coming chain is diverted by means of guide sprockets. The head and guide shaft bearings are grease lubricated plain type with renewable brasses and the boot shaft runs in bushed slide blocks fitted with bronze adjusting screws. The drive is by enclosed roller chain from a totally-enclosed fan-cooled worm-reduction gear mounted on a cast iron bedplate extended to receive a direct coupled 3 H.P. motor.

From the elevator the granular material falls on to a plate and thence on to the oversize screen. This is of the mechanically vibrated type, rather like the Selectro. It has a strong transverse shaft with polished eccentrics running in heavy duty roller bearings with precise balanced fly wheels, the shaft being mounted in self-centering roller bearings. The screen area is 30" by 80" and the cloth of high carbon steel woven wire with 1/2" by 2" elongated apertures. The inclination of the screen is adjustable - the head being supported on spring hangers. The drive is by Vee rope from a 5 H.P. motor and a hood over the screen prevents any dust nuisance. The capacity is said to be 14 tons per hour with 70% passing through the cloth.

From the screen granules and fines pass to the cooler, and oversize passes through the Cracker Mill and back to the drier elevator.

To protect the Cracker Mill from tramp iron the upper portion of the chute feeding the mill is constructed of timber and is covered by an Electro Magnet suspended from an oversize trolley.

The Cracker Mill consists of two rotating beater drums and a central cracker bar. The beaters are of special hard steel attached to steel discs mounted on strong shafts running in self-aligning ball bearings at 300 r.p.m. in opposite directions. The position of the rotors and cracker bar are adjustable. The steel casing is provided with access doors on top and is lined at several points with 1/2" thick cast iron plates. Each rotor is separately driven by Vee ropes from a 5 H.P. motor. In order to prolong the life of the beater and cracker bars these parts can be turned/

turned about so that each edge can be used before replacement is necessary. The Cracker Mill is designed to break oversize without pulverizing it.

The product passing through the oversize screen goes to the cooler. This is designed on much the same lines as the drier and is 20' long by 5' in diameter inclined at an angle of 3° and rotating at 1.54 r.p.m. Material passes through the cooler counter current to air drawn by a 25" diameter fan with a capacity of 6,000 cubic feet per minute. From the fan the air passes to a steel Buell cyclone and thence to the wash tower.

The wash tower is built of brick 8' by 4' x 30' high and provided with six Monarch type water sprays.

The granules and fines leaving the cooler are elevated to the fines screen by an elevator of exactly the same design as the drier elevator.

The fines screen, made by Hoad Wrightson, is of the "Selectro" design and is of the general type already described. It has 40" by 96" cloth with 1/25th inch by 1 inch apertures and is designed to handle seven tons per hour with 25% passing through the cloth. A cover prevents any dust nuisance. From the fines screen the granules pass to the granules hopper and the fines to a band conveyor which takes them back to the batching point. This conveyor is of capacity sufficient to take the whole output of the plant. It is fitted with a 16" wide rubber and canvas belt and supported on 3-roll type plain bearing troughing idlers of 5" diameter at 4'6" pitch. The belt runs at about 100 feet per minute.

Whereas in our first plant we passed all the granules direct from the drier to the cooler and thence to the oversize and fines screens, it may be noted that in our second plant we passed the product from the drier over an oversize screen before going to the cooler. The first system has the advantage that the cracker mill or oversize breaker handles cooled material and this has less tendency to paste and of course no loss elevator is required, and the second system has the advantage - especially when cracked oversize is returned to the drier - of securing some cooling of the material before it reaches the cooler proper. As we have always found cooling difficult and as we do not find too much pasting in the cracker mill we rather prefer the second system.

When we started up this plant we had the usual teething troubles, but compared with the difficulties we had with the first plant these were comparatively mild. On one occasion a prong from one of the forks in the Eirich mixer became detached and travelled through the drier, up the elevator, across the oversize screen, and - in some way we have never found out - it passed the magnet and fell into the cracker mill. There was a sound like a shot from a gun and when we examined the mill we found the cracker bar bent and the housings for all four bearings smashed. We now use shear pins to support the cracker bar and as these suffer from fatigue we replace them every week. We also fixed chains to the cracker bar so that if it does fall we will not have to search for it in the elevator boot.

One of our problems when determining what we wanted in the way of preparation plant for the materials making up the mixture before they pass to the granulator was to decide whether all ingredients should pass over a screen and only the tailings from this screen should go through a Carr's type disintegrator, or whether to dispense with the screen and put everything through the disintegrator. In the latter case some large particles might pass the disintegrator but the average particle size of the product would be smaller than if a screen is used. This because the screen cloth has to have a fairly large aperture to prevent blinding. We decided to put everything through the disintegrator and not to use a screen. This gave us finely divided material passing to the granulator but unfortunately there was a great deal of build up between the cages of the mill and on the inside of the casing. We improved the draught on the mill and increased clearances all round, but we have never really overcome our difficulties and in subsequent plants we arranged to use a screen and pass only the tailings through the disintegrator.

On this plant the product was dried to around 4/5% H₂O and cooled to within 15°F of atmospheric temperature. It was most noticeable that, save for occasional spells, the product from this pan type granulator was not so attractive as that from the tube, being neither so uniform in size range nor so smooth or round.

Our third plant was similar in design and size to the second, save that we used a screen on the preparation section and passed only the tailings to the cage mill and this time we used a three bar instead of a four bar mill. We also increased the number of weir plates in the drier so as to increase the average time the granules took to pass through from 30 to 45 minutes.

When this plant was started up it gave us very little mechanical trouble, it dried granules down to 3/3.5% H₂O and save in the hottest weather it cooled them to within 15°F of atmospheric temperature, but it was a very dusty plant. There was a great deal of dust emission, particularly from the screens even though these were provided with dust hoods which we draughted. We have since improved conditions by drawing the air for the cooler down the fines elevator and by removing the air seal between the drier and the oversize elevator so that the drier fan draws a proportion of air from the oversize screens and oversize elevator. This is wasteful in power, but it does help to minimise the dust nuisance. It is particularly hard to keep a granulation plant free from dust as where you have elevators or chutes discharging dusty materials on to band conveyors, even though the discharge point is well shrouded, there is almost bound to be dust emission. When talking of this point there is a trick we found quite useful. We found that there is a tendency for granular material fed on to an inclined band conveyor to spill off, but if a piece of 1" diameter heavy rope is loosely fitted to the feed chute so that it lies on the band conveyor below the chute, it prevents this spillage.

10.

Our fourth plant was very similar in design and size to the third save that in this case, in order to improve drying and to try not only to lower the average moisture content of the granules, but to get the large particles down to as low a moisture content as possible, we provided means of redrying the cracked oversize, a return conveyor from the cracker mill to the drier inlet being installed. This proved to be of considerable advantage and we found the moisture content of our product could now be brought down to 2%. Cooling was reasonably efficient and the tonnage output of the plant was satisfactory. We ran into one interesting difficulty at this time. The first Carr's mill was built up of mild steel bars rivetted into the rings, but on the later mills the bars, of rail steel, were welded into the rings and after some months of service the bars broke away from the rings at the welds. The bars were welded to the rings again and the cages annealed, but this was still unsatisfactory and the only satisfactory construction to date is the first one tried.

At this plant special attention has been paid to the tension of the springs loading the ploughs in the pan mixer and it has been possible to keep a mirror finish on the pan, which helps materially to reduce cleaning time. (At another works, in an attempt to reduce wear on the ploughs and to minimise cleaning troubles, they use a chisel shaped plough.)

At all our pan mixer plants we have been bothered by mud formation on the outside of the pan. The spray water is in a very fine state of division and tends to blow out of the pan over the sides where dust settles on the moist surfaces and a mud is formed which works its way down to the bearings. To overcome this trouble, perhaps because we had just painted the pan during a three weeks summer maintenance period, we fitted only last week a perforated spray pipe - this does not give atomised water but rather a series of streams such as from a garden watering can. It is working very well to date; we are using rather less spray water than with the finely divided sprays, and we are having no trouble with mud.

Performance/

Performance data for a typical month's work with this fourth plant are given in Appendix I, and the essential features are as follows:-

| | | | |
|--------------------------|----------------------|---------------|-------|
| <u>Product:-</u> | Size analysis - | + 3.35 mm. | 11% |
| | | 3.35/1.00 mm. | 87% |
| | | - 1.00 mm. | 2% |
| | Moisture content | | 2.3% |
| | Temperature | | 78°F |
| <u>Output Rate:-</u> | tons per hour | | 5.5 |
| <u>Services Usagos:-</u> | (per ton of product) | | |
| | Water | - tons | 0.11 |
| | Coal | - tons | 0.041 |
| | Electric Power | - KWH | 13.1 |

As in the case of the first plant, for which operating data were earlier given, screening is not altogether efficient - in this case some oversize having appeared in the product. A very low moisture content (2.3% as against 5.2% in the other case quoted) has been achieved in spite of the fact that more water is being used on account of the greater proportion of fines recycled, and the coal consumption is some 35% higher. (Part of the increase is however due to using a lower grade coal.) Electric power consumption is also higher, by 22%, because of the greater complexity of the plant.

A fifth 5-tons/hour plant has recently started up, but need not be described as it is very similar to the third and fourth plants. A sixth plant, to complete our granulation equipment, is in the final stages of erection and will be working very soon. This last plant is of 10 tons/hour capacity and in this case we have gone back to the rotary tube system of granulation. The plant has twin granulators, and twin screen and cracker units so as to allow of cleaning and minor maintenance without interruption of working, each twin unit being capable of taking the full load for short periods.

It may be interesting now to discuss some practical points of operation as we have found them in our experience. Operation of granulation plant is an art, as we expected, and much depends on the skill and experience of the individual operator. As is usual in such cases it is highly desirable to secure steady conditions of working and freedom from interruptions. The plant should operate continuously day and night, both for the sake of thermal efficiency and to avoid disturbance of steady working. The mixture should be changed as infrequently as possible.

Cleaning is an important matter, and cleaning and routine maintenance should be planned on a definite routine. Our practice is to have a definite cleaning task lasting one half-hour assigned to each shift, with a more comprehensive cleaning, greasing and general maintenance programme covering the whole plant carried out at the week-end when the plant is shut down for an 8-hour cleaning shift.

Granulation in the pan mixer-granulator plants is a batch process, and maintenance of correct output is dependent on keeping the batch time cycle within the limits imposed by the capacity of the drying, cooling and screening plant to handle not merely the net output of granules of the selected size, but also the rejected fines which are recycled to the granulator and the cracked oversize which is recycled to the drier. As an example of the time cycle which can be achieved in practice on a 5-ton/hour mixer granulator pan, the following typical figures are cited, for a 15 cwt. batch of potato compound:-

| | <u>Time</u> |
|---|--------------|
| Skip ready to deliver charge to pan | 0 |
| Skip empty | 7 seconds |
| Mixing and granulation complete, pan discharge door opened | 167 seconds |
| Pan discharge complete | 257 seconds |
| Pan discharge door closed and pan ready to receive fresh charge | 269 seconds. |

Thus/

Thus the maximum theoretical rate of granulator throughput in this particular case is about 10 tons per hour. In practice the time cycle is bound to be slightly longer as an average but no difficulty is experienced in keeping the granulator throughput up to about $7\frac{1}{2}$ tons per hour which provides a full load for the drier.

It was earlier stated that in preliminary experiments it was found preferable to add water fast and to work down to correct granule size, rather than to add water slowly and work up to correct size. In works practice we have not found this possible. Few of the plant operatives have the courage to risk the possibility of adding just more than the critical amount of water, in which case the large lumps formed on fast water addition will fail to break down on continued stirring. It has therefore been found that slow addition of water is the safer course to adopt in normal practice. In the example just cited, 160 seconds elapsed from completing the charging of the pan to starting emptying, and the water addition was spread over 135 seconds of this period.

In the summarised operating data already given, reference was made to the fact that screening was not completely efficient and the final product contained some oversize and fines which should have been rejected. This inefficiency in respect of oversize arises from deliberate use of a screen mesh with elongated apertures. As regards fines, which are retained because of slight "blinding", we have still some doubt as to the effect of 100% complete rejection of fines for recycling. There is some evidence, not by any means conclusive, that recycled fines do not granulate well. This is quite likely since much of the fines consist of undersize granules of very low moisture content with a hard surface impervious to water. If so it is possible that fines would build up in the system and would require to be purged. The screen inefficiency we have experienced acts, of course, as an automatic purge. The alternative would be to treat the fines to improve their granulability before feeding them back into the system.

Incidentally our latest view is that the most efficient screening material for removal of fines is a wedge-wire cloth.

13. This question of fines also enters into the problem of variation of chemical composition of a granular compound with size of granule. This variation has been experienced, we believe, by all manufacturers using the type of process described in this paper.

Some of our early production showed very wide fluctuations in analysis with varying size of particle, so we have always been particularly careful to return the proper proportion of fines to be re-incorporated in the granules.

Even though we take such precautions as are practicable there is still far too wide a variation in analysis throughout the size range. This can be seen from the following table which records an examination of a typical average sample of the recent make at one of our works.

| Screen Size | Size | | N ₂ | P ₂ O ₅ | | P ₂ O ₅ Total | P ₂ O ₅ f/a | K ₂ O |
|-------------|--------|--------|----------------|-------------------------------|--------|--|--------------------------------------|------------------|
| | Ancl. | Moist. | | Sol. | Insol. | | | |
| Gen. Sample | 100.0% | 5.11% | 5.78% | 8.74% | 1.69% | 10.43% | 0.53% | 11.09% |
| +5 BSS | 17.7 | 4.39 | 5.29 | 9.51 | 1.53 | 11.04 | 0.53 | 10.07 |
| +6 BSS | 25.9 | 4.71 | 5.46 | 9.18 | 1.77 | 10.95 | 0.53 | 10.59 |
| +7 BSS | 19.5 | 4.59 | 5.85 | 8.69 | 1.69 | 10.38 | 0.53 | 11.36 |
| +8 BSS | 13.9 | 4.29 | 5.88 | 8.58 | 1.53 | 10.11 | 0.35 | 12.03 |
| +10 BSS | 12.0 | 4.17 | 6.02 | 8.42 | 1.58 | 10.00 | 0.53 | 12.37 |
| +12 BSS | 7.4 | 3.96 | 6.06 | 8.50 | 1.47 | 9.97 | 0.53 | 12.78 |
| +14 BSS | 2.1 | 3.64 | 5.99 | 8.42 | 1.55 | 9.97 | 0.35 | 12.76 |
| +16 BSS | 1.1 | 3.63 | 6.00 | 8.33 | 1.62 | 9.95 | 0.35 | 13.01 |
| +30 BSS | 0.4 | 3.74 | 6.05 | 8.42 | 1.51 | 9.93 | 0.35 | 13.11 |
| +52 BSS | negl. | 3.22 | 6.30 | 8.74 | 1.69 | 10.43 | 0.53 | 12.79 |
| -52 BSS | negl. | 3.63 | 6.51 | 9.40 | 1.89 | 11.29 | 0.53 | 12.73 |

It is obvious from these results that care must be taken in recycling the proper/

proper proportion of fines during the course of manufacture, and that the laboratory control of production must depend on very careful selection and preparation of the sample.

The variation in analysis is caused by differences in solubility, in wetability and in what we might call "granulability" of the different ingredients and by differences in particle size. It is obvious that a particle of superphosphate which is 3.5 mm in diameter is not likely to be incorporated with the proper proportion of salts in a compound containing perhaps 40% of salts and still yield a granule within the size range 1-3.5 mm.

Statistical methods can be employed to find the chance of getting a perfect mix of ingredients of different particle size, and it may be shown that in order to have a reasonable chance, a very fine particle size is required for all ingredients, particularly for those added in small proportions only. When the problem is further complicated by differences in solubility, wetability etc., it becomes apparent that in order to prevent variations in the analysis of granules with varying particle size it is necessary to grind all ingredients to a very fine powder. This is clearly impracticable on many counts.

Our early experimental work indicated that the wet mixing method yielded a product which was remarkably uniform in analysis with varying particle size, and our own experimental observations have been confirmed by examination of samples of granular compound made by the Proctor process. In this respect therefore the wet-mixing granulation process would appear to be definitely superior at the present time. It is possible however that practicable ways and means of overcoming this defect in the process here described can be found. So far no great progress has been made by us. Unsuccessful experiments have been carried out on the use of wetting agents. Something may be done by the pre-treatment of recycled fines as mentioned earlier. Some of the more soluble ingredients may be added in solution form as part of the wetting process, and the incorporation of sulphate of ammonia in this form appears to be particularly promising since the fines contain rather a high proportion of this material as whole and fragmented crystals.

14.

In this connection reference may be made to the publications of Hardesty and others from the U.S. Department of Agriculture, Bureau of Plant Industry, Beltsville, Maryland. These workers have considered many theoretical aspects of granulation of superphosphate and compounds and stressed the need for uniform moisture distribution. In U.S. Patent No. 2,287,759 Hardesty claims that for good granulation it is essential to make full use of the moisture in the ingredients, to use the minimum of spray water and to secure maximum uniformity of distribution of the water. He proposes to wet the mixture first, and then to hold it in a non-drying atmosphere for about 12 hours to secure the maximum amount, and most uniform dispersion, of solution phase. Thereafter granulation is to be carried out at a temperature of between 60°C and 100°C.

It is not proposed to enter into any detailed discussion of these and similar claims, but it may be repeated that much remains to be discovered as to the physical and chemical changes which occur during the granulation process described. It is reasonable to suppose that as fundamental knowledge accumulates the particular problem of variation in chemical composition with granule size will prove to be capable of effective solution. In particular it is to be hoped that the process can be improved to the point that there is substantially complete production of granules of the desired size in a single operation.

15.

The simplest method of calculating "granulation efficiency" under conditions of continuous works operation is by measurement of the quantities of fines recycled to the granulator and of granules of product size delivered from the plant. Unfortunately we cannot present exact figures of our average efficiency since we have not provided means for continuous measurement of the quantity of fines recycled. Spot tests under conditions of/

of continuous operation are not very reliable, and indeed the results of such tests show considerable variation. On the whole however we put our average granulation efficiency (selected granule size range being 3.35 mm. to 1 mm. diameter) at 75% to 80%. This is further away from the ideal of 95% to 100% than we had originally expected from the results of our preliminary experimental work. Furthermore this is the overall efficiency after cracking and returning oversize, whereas the ideal is to secure a much higher direct yield at first pass through the granulator.

16.

Bag rot is one of the major problems facing the manufacturer of granular compound fertilisers. We did not have any trouble with the rotting of bags containing granular compounds for several months after starting up our first plant, but after this brief respite we have had the problem with us ever since. Superphosphate makers are familiar with the rotting of hessian bags used for superphosphate, especially if the superphosphate is not fully matured, and if the material lies in the bags for a long time before it is used. The hessian fabric becomes reddish brown in colour and the fibres lose their tensile strength so that the bags rot and become useless as containers. We used to believe that this rotting was caused by the free phosphoric acid in the superphosphate and it was easy to assume that the cause was the same when we first experienced the rotting of hessian bags used as containers for granular compound fertilisers. On the other hand bag rot with granular compounds was not only greater than with powder compounds but more rapid and more intense than with straight superphosphate. Furthermore we soon discovered bag rot in compounds which had no free phosphoric acid whatever. We found we were faced with the expense of rebagging large tonnages of fertilisers held in our stores. Some of our friends overseas, where they have high atmospheric temperatures and where they had been bothered with the rotting of both hessian and paper bags both with superphosphate and powder compounds, had found the cause of the trouble to be the liberation of HCl, H_2SiF_6 and HF by the action of free phosphoric acid on chlorides and on unreacted phosphate rock. We made a number of tests in the laboratory and confirmed that the acids HCl, H_2SiF_6 and HF, in that order, are responsible for the attack on the cellulose in hessian and that phosphoric acid, in the concentrations present in either superphosphate or compound fertilisers, does not itself directly cause any damage. Unfortunately the problem cannot be solved by neutralising the small amount of free phosphoric acid present in ordinary superphosphate. Monocalcium phosphate, an acid salt, is present in large quantities and is liable to hydrolyse when the granules are heated in passing through the drier, phosphoric acid being released. This will react with chlorides giving HCl, and with unreacted phosphate rock giving H_2SiF_6 and HF. The amount released when drying granular fertilisers seems usually to be sufficient to liberate enough of these acids to cause serious bag rot.

The variations experienced are however noteworthy. We have had widely different and very contradictory results at different times and in different works. In some cases they have had serious rot with fertilisers containing chlorides and in other cases they have had none at all; in some cases rot has been serious in compounds free from chlorides and in another factory, using the same type of plant there has been no trouble at all. We have had so many conflicting results that we have the greatest difficulty in making any reliable deductions. At the present time we believe that the best ways of reducing trouble are to:-

- (a) Neutralise the free phosphoric acid in the compound, preferably leaving a fairly high proportion of insoluble P_2O_5 in the compound.
- (b) Dry to a moisture content of from 2 to $2\frac{1}{2}\%$ H_2O , carrying out the drying at the lowest economic temperature.
- (c) Cool to within at least $15^{\circ}F$ of atmospheric temperature.

To date however the only safe solution is to use a package which does not rot even when subjected to acid attack. A paper lined hessian bag with a bitumen interlayer has proved effective as also have multiply paper bags with/

with a waxed inner lining and a bitumen sandwich. These, however, are expensive solutions and the correct lines of progress are to minimise hydrolysis of monocalcium phosphate and to avoid bagging while evolution of acid gases is still in progress.

17. In conclusion a brief word may be said with regard to our experience as to after setting, i.e. caking of the granular compound during subsequent storage either in bulk or in bags. We have experienced such setting, clearly for the following reasons:-

1. The lack of complete chemical homogeneity in the compounds has meant that syngenite formation can still readily take place at the interface of adjacent granules.
2. The number of points of contact between adjacent granules in the mass has been relatively high owing to lack of complete uniformity of granule size.

At times this has been aggravated by the need deliberately to leave all fines in the product in the interests of maximum production.

3. Owing to temperature and moisture gradients in the mass, due partly to inadequate drying and cooling and partly to differential drying as between large and small granules, heat and water movements within the granules themselves and between adjacent granules in the mass arise and cause recrystallisations to take place.
4. Atmospheric humidity changes cause surface phenomena of solution and recrystallisation.

The ways and means of lessening the tendencies to after-setting will be clear from the above analysis of the causes. There is therefore no reason for pessimism as to our ability to reduce after-setting, and even at this early stage in our experience the amount of after-set is usually minor and agglomerations break down easily to a free flowing granular product.

18. This, then, is our story to date. We have no regrets at our decision to turn over to the granulation of compound fertilisers and our experience confirms our original belief that this form of product would be welcomed by our farmer friends. We find also that its cleanliness and general free-flowing nature made handling and storage in the works easier and more comfortable. The story has however only just begun and we feel sure that new chapters remain to be written hereafter. In the meantime may we hope that this account has been of interest and of some service.

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27th August, 1947.
HH.

APPENDIX I.

OPERATING DATA FOR TYPICAL 4-WEEKS PERIODS.

| | Works No. 1 | Works No. 4 |
|--|----------------|----------------|
| Type of granulator | Tube | Fan |
| System of granulation | Continuous | Batch |
| Rated capacity - tons/hour | 5 | 5 |
| Production for period - tons | 2958 | 3316 |
| Hours manned | 583 | 600 |
| Effective productive hours | 459 | 490 |
| Average output - per hour manned | 5.07 | 5.53 |
| per productive hour | 6.44 | 6.77 |
| Analysis of hours manned (percentages):- | | |
| Effective productive hours | 78.8% | 81.7% |
| Meal breaks | 6.2 | 6.0 |
| Cleaning | 6.8 | 8.0 |
| Mechanical breakdown | 6.8 | 1.6 |
| Electrical breakdown | 0.4 | 1.2 |
| Changes of mixture | 1.0 | 1.5 |
| | <u>100.0</u> | <u>100.0</u> |
| Average Temperatures:- °F | | |
| Granulos to drier | 55° F | 60° F |
| " from drier | 164 | 161 |
| " to cooler | 159 | 144 |
| " from cooler | 75 | 83 |
| " from plant | 70 | 78 |
| Hot gas to drier | 804 | 850 |
| " " from drier | 265 | 210 |
| Cold air to cooler | 43 | 36 |
| Services Usages:- per ton product | | |
| Wash-tower water | not available | |
| Granulation water - tons | 0.071 | 0.11 |
| Coal - tons | 0.030 | 0.041 |
| Electric Power - KWH. | 10.73 | 13.1 |
| Product:- | | |
| Size analysis + 3.35 mm | 2.7% | 11% |
| 3.35 to 1.00 mm | 85.0% | 87% |
| - 1.00 mm | 12.3% | 2% |
| Moisturo Content | 5.1% | 2.3% |

JA/EPH/HH.

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