PROCEEDINGS
OF THE
FERTILIZER INDUSTRY ROUND TABLE
1959

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Held at the
MAYFLOWER HOTEL
Washington, D. C.
November 4-6, 1959

Executive Committee

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Wednesday Morning Session, Nov. 4, 1959

The Fertilizer Industry Round Table convened at ten o'clock a. m., in the State Room, Mayflower Hotel, Washington, D. C., Dr. Vincent Sauchelli, as President, makes opening remarks.

ONCE again it is my privilege on behalf of your Executive Committee to welcome you to our 9th Annual Session of the Fertilizer Industry Round Table.

Here are assembled the country's most competent practitioners of the art and science of producing modern and conventional plant foods.

It is indeed a pleasure to greet you and to wish you a profitable attendance. We believe we have organized an agenda which will hold your interest and provoke your mental machinery into action.

With the cooperation of my fellow committeemen, I have the following comments preliminary to the start of the planned agenda.

Our Round Table Sessions have developed through experience along the novel, non-stereotyped pattern suited to the needs of our members. Fundamentally, the distinguishing feature of our conferences is the manner in which the program is drawn up and carried out.

We have stressed from the start that the sessions should reflect the thinking of this group. The members act as a committee of the whole in suggesting the agenda items and the persons to choose for leading the discussions.

The program is not something imposed from above; it is your program reflecting your problems and desires as to possible solutions.

Your Executive Committee serves as handmaiden to your needs; sometimes it may appear that this Committee has arrogated to itself powers and perpetuation of office. Let me here emphasize that such is not the case. Ours has been and is a convenient arrangement. All members of the Committee reside in Baltimore; can communicate with each other promptly by phone or in person and have always worked together as a team. Each has given generously of his time and labor, motivated solely by the desire to be of service to his colleagues. Anyone in this audience is always welcome to participate in making up the agenda through written or oral suggestions.

In fact, we earnestly urge such participation since we are always in need of new ideas for the succeeding year's program. You will have an opportunity to express yourselves on this matter during our brief business session tomorrow.

Please remember, we want to keep our organization as informal, as spontaneous, as possible. Discussion from the floor is one of the most valuable functions of these sessions. More time has been given this year for this purpose.

Again, we know that these get-togethers constitute a valuable opportunity to meet personally many fellow workers not otherwise possible. Visit with them during recess, lunch hours and evenings; such personal visits give you an unusual chance to exchange ideas on problems of mutual interest which might not be of general interest in the open forum of our meetings.

Registration Fee

The registration fee had to be increased this year. The money so collected is used exclusively to pay for the cost of printing and distributing our proceedings. Many free copies are sent to industry, university and public libraries both here at home and abroad.

There's a lot of interest in these proceedings among world fertilizer manufacturers — you'd be amazed at the number of requests we get from abroad.

Last year we had a tight squeeze, financially speaking. Our Treasurer, in desperation, conceived a new source of revenue, renting space to exhibitors. We trust this small increase in fee this year will remove all anxiety about the printer's invoice this coming year.

By the way, we have on hand a quantity of 1958 proceedings. These will be made available to members for distribution among their plant personnel responsible for manufacturing and among local libraries. You are welcome to them and if you can pay for them, your contributions will certainly be appreciated.

The fertilizer industry is expanding at a remarkable rate all over the world. Process developments abroad are well worth your study. We in this country, don't have a monopoly of technology in granulation, ammoniation and other phases of manufacture. We hope in the future we might get more information from foreign developments.

Reference to procedures

During the first four years of our existence the number of persons attending the Round Table Session was limited through a system of invitation. Spontaneous discussions of common problems was the procedure. Results were gratifyingly successful. There followed a strong request for broader participation by many colleagues who had heard about these meetings and the benefits to be derived from them.

In this early period we set the dates of the Round Table meetings to coincide with the Fall Meeting of the American Chemical Society. This practice was discontinued when our attendance became large enough to warrant complete independence and freedom from other distracting conferences.

The first week in November has proved popular and, may I say now, we have the opportunity of reconvening in this hotel next year the first week of November if you elect to do so at our business meeting.

How to organize these meetings

so as to retain as much as possible the spontaneity of the original gatherings has been a continuous challenge. Experience has shown that this could be accomplished by having you all as a Committee of the Whole. You have been asked each year to give us your suggestions as to the problems you wanted discussed and in most instances you have nominated those colleagues you considered most competent to lead the discussions on each major problem. This practice has been followed by your Executive Committee because it seemed to work very satisfactorily.

We need your suggestions for this coming year.

We have a large group here and sometimes it seems too unwieldy to follow the essential practice of a Round Table, that is, to stimulate discussions from the floor. We urge all not to be afraid to get up and comment and exchange ideas with your colleagues here on problems that may be aroused in the discussions.

I know the speakers expect questions from the floor. They are prepared to answer as intelligently as possible on the problems that are raised and in that way, only, can this Round Table be of the most benefit to all.

A. C. S. Monograph

If you will permit a brief personal reference at this point; about five years ago Dr. W. Hamor, Monograph Editor of the American Chemical Society, invited me to edit a monograph for the Society on the Chemistry and Technology of Fertilizers.

I told him then that the time was not right for such a book; that many of the new processes had not yet been fully worked out; that modern fertilizer technology was still in a flux. Developments, however, have been rapid and widespread. I am now happy to tell this group that by the time we meet again next Fall the American Chemical Society's monograph on the Chemistry and Technology of Fertilizers will have been published. You all will be as agreeably pleased as I am to see revealed for the first time in one volume the tremendous changes and engineering advances our industry has experienced.

The only other business I have at this moment is to appoint an Auditing Committee. I'd like to have Joe Bosman and "Slugger" Frank Nielsson act as an Auditing Committee.

We have arranged this year to have a couple of excellent speakers from our neighboring country to the north, and we're dividing the monitoring here. I'm going to turn the meeting over now to Albert Spillman, who will introduce the first two speakers.

Albert Spillman

CHAIRMAN SPILLMAN: It is indeed most gratifying to see this splendid large attendance and to have an opportunity to meet with my good friends in this audience.

The Round Table idea conceived by our Chairman, Vince Sauchelli, eight years ago, has grown into an impressive forum for airing ideas about all aspects of fertilizer technology.

It has been my pleasure and privilege to serve as a member of the Executive Committee with your Chairman, Joe Reynolds and Dr. Marshall. The time and effort spent helping to organize this program was interesting, educational and worthwhile.

The topics to be discussed during our two and a half-day meeting were carefully formulated from the questions sent to our committee by members of the Round Table. The leaders who very kindly accepted to lead the discussions of each subject assigned to them are experienced and capable men. The subjects to be discussed are timely and important to all of us.

Our industry in recent years has made great progress in all phases of fertilizer manufacturing technology; refinement of processes, formulation and equipment has been progressively improved.

We will undoubtedly have more rapid, improved changes in the near future. Fertilizer manufacturing is gradually changing from an art into a science and the latter will make techniques more and more efficient.

Raw material suppliers are doing an outstanding job in the field of research to improve their products, to supply the quality specification materials needed in our formulation program.

Grade ratios are changing. Demand for higher analysis mixtures is increasing. Granulation is increasing. New materials have come into the picture. Phosphoric acid is now used in large volume.

To keep abreast of the forward innovations in fertilizer technology all of us responsible for operations will have to be alert and continually on the job; otherwise we will get lost in the shuffle.

The Round Table discussions and exchange of ideas will help us considerably to reach our mark. With the combined cooperation of all agronomists, TVA research people, Beltsville research people, researchers in our industry, machinery suppliers, raw material suppliers, fertilizer production personnel as a group discussing all problems, we will gain our objective.

In my opinion the Round Table has opened the horizon for all of us responsible for fertilizer manufacturing. I don't believe any one segment of the industry can do this job by itself.

We have, as our Chairman has told you, a wonderful opportunity to gain progress in manufacturing technology due to Round Table discussions and by contacting one another through the year to exchange information.

The first discussion now on our program is "Plant Processes from Raw Materials to the Bag." This topic will be discussed by two outstanding fertilizer companies with many years of experience in all types of fertilizer manufacturing. The two companies are Canadian Industries of Canada and Wilson and Toomer of Florida.

We are very fortunate and thankful to have our good friends, the Canadian Industries, Ltd. of Canada to start this symposium. This fine organization operates seven fertilizer plants in Canada: Mr. Les Clegg, formerly Production Manager and recently appointed Purchasing Agent for the Canadian Industries, will handle this discussion with the assistance of his associate, Elgin Doidge, Production Supervisor.

Canadian Industries during the past year started operations on three new granular plants. Mr. Clegg has held responsible positions in a number of his company's agricultural chemical plants; since 1951 he has been Production Manager in Charge of fertilizer manufacturing at seven plants in Ontario, Quebec and the Maritimes.

We are very grateful to Mr. Clegg and his associates for taking on this chore and for giving our Round Table their valued experience on fertilizer technology and control of operations.

Mr. L. V. Clegg (Canadian Industries, Ltd.):

I've always felt privileged to be able to come here to your country to attend these meetings. The information that I have gained has been a great help to me personally and has been a great help to my company and I'm very pleased this morning to have the opportunity of trying to make some small contribution to your program. I thank you.

A Multi-Plant Operation

L. V. Clegg

Introduction

I HAVE been given the opportunity to tell you about the organization used by our company in operating its fertilizer plants. I know there are a lot of people here who are responsible for the operation of more than one plant. I have always been interested in the types of organizations which control production in the many companies represented here and I think we can all benefit from the exchange of ideas on this subject because there is a lot to be learned in this science of building and keeping a production team producing. I am not here to say one system or organization is better than another. I propose to tell you what we do and if there are any here who have also tried these systems either successfully or unsuccessfully I hope it will be brought out in the discussions.

In order that you will better understand what I have to say I would like to tell you something about the company with which I work. As the name implies, Canadian Industries Limited is a Canadian chemical company operating 25 plants across Canada. With the head office in Montreal, there are 8 operating divisions, Explosives, Ammunition, Textile Fibres, Plastics, Coated Fabrics, Paints, Chemicals and the Agricultural Chemicals Division. At the present time C-I-L is the largest company in the chemical and allied products industry in Canada in terms of dollar value of sales.

The Agricultural Chemicals Division operates seven fertilizer plants in Eastern Canada. At two of these plants single superphosphate is manufactured by the Ober Process. The Chemicals Division and Explosives Division operate the sulphuric acid plants, ammonia plant and nitrogen solution plant from which we obtain some of our raw material requirements.

The Agricultural Chemicals Division's staff is headed by a General Manager. Responding to him is a Sales Manager, responsible for the sale of the Division's products; a Control Manager, responsible for Divisional accounting; a Technical Manager, in charge of process development, market surveys and research; and a Production Manager, responsible for the operation of the plants.

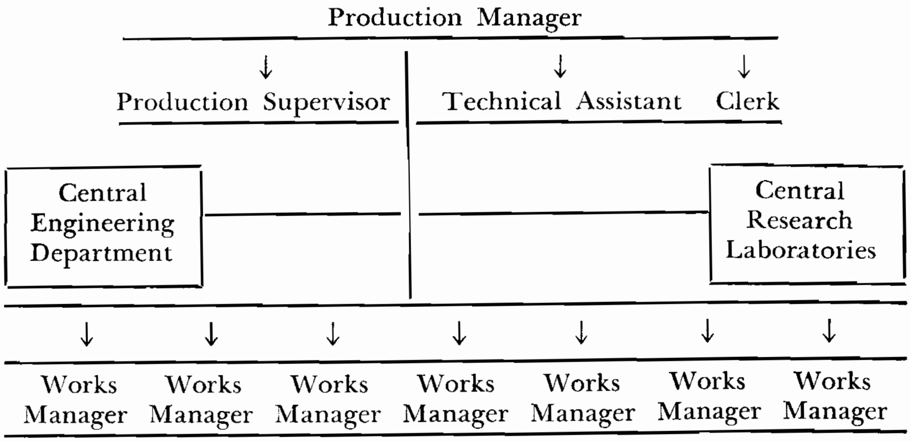
Production Department

The control of a multi-plant operation is usually the function of a Production Department and I am sure that those of you who are connected with a multi-plant company will realize the need for such a department, because unlike many chemical operations all fertilizer plants use the same kind of raw materials, the same processes, the same type of buildings, ship the same products and have the same problems.

In my opinion some of the functions of a Production Department are:

(1) Ordering and scheduling raw material deliveries.
(2) Control quality.
(3) Control formulations.
(4) Keep abreast of new ideas for processing; analytical methods; to process and interpret to higher management appropriations for capital expenditures.
(5) Select and train new technical staff and see that they get the opportunity to broaden their experience and move up in the organization.
(6) Help to keep an active safety programme.
(7) Last but not least, keep plants informed of their efficiencies and how they compare with the other plants in the organization.

Before discussing these functions in detail I should show you the organization of our Production Department, which is shown in the following chart:



It is common practice in fertilizer organizations to have a district manager who is in charge of both sales and operations.

In our organization there is a Works Manager in charge of each plant and when a Sales Office is located at the plant site the District Sales Manager is in charge of the district sales responding to the Divisional Sales Manager, while the Plant Manager responds to the Production Manager. You might think that this would result in disputes and a slow down in communications. We have over the years had both types of organizations and we favour our present type.

When you have a Works Manager on the site he has this position because he has been tried and proven as a man with good judgment, a good manager and one with the ability to get along with people. With a Works Manager and a Sales Manager on the site, the Sales Manager has the assistance of a strong man. He also has a man on the site who can make quick decisions. All our Production people and Works Managers have learned through training courses, attendance at sales meetings and the policy of the Division that the District Sales Manager is the man who knows what should be made and the rate it should be shipped. Our Works Managers work closely with the District Sales Managers and do their best to give sales the best quality and the best possible service. We think this makes a strong team. Actually it does not mean extra staff; it is replacing the plant foreman or superintendent with a stronger man.

As an argument against the other type of organization the District Sales Manager has come up through the sales organization and cannot be fully acquainted with all the new tools used in modern plant management, industrial engineering, about relations, and quality control.

Now to get back to our list of functions of a Production Department.

(1) *Order and Schedule Raw Materials*

As you all know most fertilizer raw materials are contracted for on an annual basis by the Purchasing Department. Once these contracts have been made the actual scheduling of the shipments is done by the Production Department.

The slide shows the raw material requirement report. Each month, rather than each plant ordering its raw materials, the Plant Managers make out this report telling us how much of each raw material they require and when they want it. All the purchase orders are prepared in the Production Department, a copy of the orders being sent to the plants. We have found that this system works well, the plants like it, we have complete control of the movement of raw materials and it requires less work for them. If there is a delay in shipment to one plant or a plant has incorrectly estimated its requirements for the month we can quickly find a car enroute to some other plant and have it diverted without delay. We have sufficient information in our daily and monthly reports to make changes in raw material movements without first contacting the plant. We need only tell them after the fact. The plant staff are responsible for seeing the shipment arrives on schedule and they have their own system to control it. With this system we are fully aware of our raw material position at all times and can provide the General Manager and the Purchasing Department with information quickly.

(2) *Control of Quality*

We have chemical laboratories located at four of our seven plants. These, between them, do the analytical work for all plants. Two of these laboratories are located at Works which are made up of fertilizer plants and plants of other divisions, and are operated by other divisions of the Company. The other two are located at fertilizer plants. There is no effort to exercise any control over the analytical methods used in these laboratories but the Production Department, with the help of the Central Research Laboratory, keeps abreast of new developments in fertilizer analysis and passes them on to the Works laboratories. Visits of personnel are arranged between laboratories. We have found that the quickest and easiest way to watch the analytical results is to have the laboratory make an extra copy of their report to the plant and send it directly to the Production Department. These are watched carefully in the Production Department and if any trends are developing the Works Manager concerned is asked for an explanation.

(3) *Formulation Control*

At one time we had an operating manual which listed all the formulae and other pertinent data and the Works Managers were required to follow these formulae. We soon found that due to the different characteristics at each plant some could formulate with a higher ammoniation rate than others. In order to allow each plant to use the optimum rate the manual was changed to outline the chemistry of ammoniation indicating what might happen if certain limits were exceeded. All plant staff knew, of course, that a low ammoniation rate could mean higher raw material costs and it was their responsibility to find the best ammoniation rate giving low raw material costs and avoiding high ammonia losses.

With the new granulation plants the Production Department Engineers, who had visited operating plants in the United States and who had worked on the plant design, were on hand to assist with the start-up at which time general formulations principles were developed at each plant for the grades made. We are as yet not certain if we should establish a formulation manual or allow each plant enough freedom to allow them to develop better formulations. Each plant has technical personnel in charge of the granulation operation, so they are capable of developing formulations which they think fit their requirements best. With all plants working on the problem and a complete exchange of information between plants the best formulations should be developed.

(4) *Process Improvement*

We encourage all supervisors and foremen to develop new ideas and improve existing processes. However, since the specialists are located at Head Office in engineering and research, the Production Department can discuss the ideas suggested by the plants with the specialists. Also, anything new in process techniques which might be found in research and engineering can be discussed with Production Department, who can pass on to the plants or arrange for a trial at one plant before accepting it for all plants.

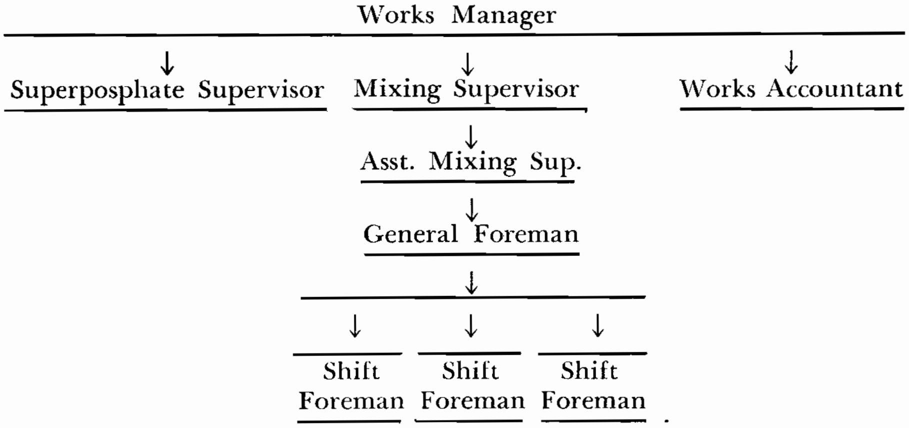
Capital expeditures over certain maximums require higher approvals. These are usually prepared and processed by the Production Department because we are able to interpret and explain the ideas to management who may not be familiar with all the operating details of a fertilizer plant. If the Sales Department require a new product, Production Department will study the requirement, do the initial planning and design of equipment. The Engineering Department then joins them to complete the design details and to prepare the appropriation estimate.

(5) *Select and Train New Technical Staff*

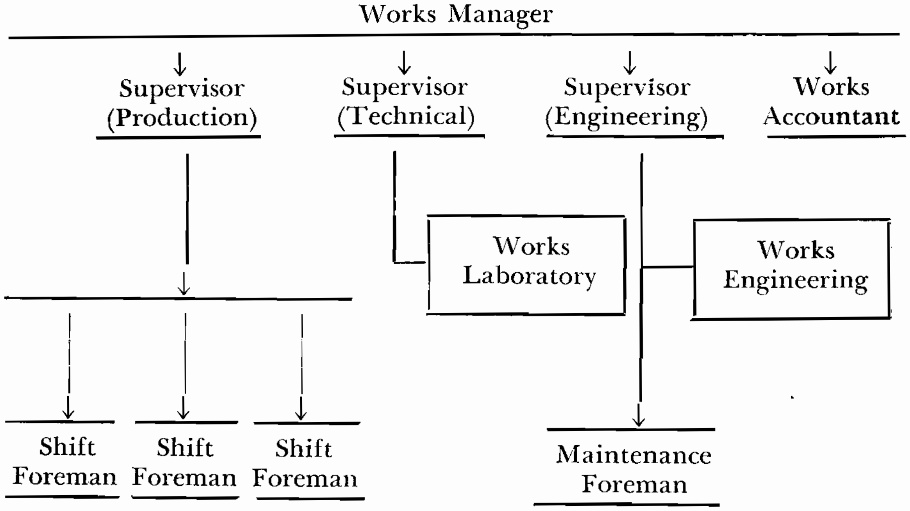
This is one of the most important functions of the Production Department. We all know that if a Company is to grow and expand or even just keep up, new staff has to be selected and watched as they move up to positions of increased responsibility. In order to get a broad training under different Works Managers who may have different methods of operating, it is necessary to transfer staff to other plants and sometimes other types of positions. C-I-L provides staff training courses; one series run by the Engineering Department, covers industrial engineering techniques; another course, using the Harvard Case Method techniques, covers the many functions of management.

One problem that has been with us and may be with some of you is how to work technical graduates into the operating staff and give them responsibilities which will give them experience and at the same time give you a chance to judge their performance. One of the difficulties is a long experienced foreman who blocks the way. It is realized with the recent trend to granulation come many technical problems. When the technical graduate joins the organization he is anxious to learn and he quickly masters the details of the process, and soon becomes an important member of the staff, but to keep him interested and keen he must get promotions, but how are you going to promote him with a foreman blocking the way? We had this problem and the practice was to move the technically trained supervisor up over the foreman to department supervisor, plant supervisor, production superintendent and to works manager. The foreman, like the Sergeant Major in the Army, remained foreman.

I came up this way and in the process I got to know foremen who although lacking in formal education, were very intelligent and were very important members of the staff. Many of these foremen had long experience and had been tried and proven on many an occasion. This past year we decided to do something about this problem: we set up an organization which allows full scope for the foreman. We use the technical supervisors in their own fields, but give them enough operating experience to develop them as operating supervisors. The previous organization is as follows:



The next diagram shows the new organization. This has been in operation for three months. The foremen are very pleased as are the technical supervisors.



(6) *Safety*

We all know that there are many factors which must be considered in a good safety program and like the experience of some here we have some plants with an outstanding safety record; others with a poor record. However, here I am only discussing the function of our Production Department in a safety program. Although the Production Department is well aware of the plants' safety activities each plant plans and carries out its own safety program. We have no one in the Head Office Production Department whose function is the central administration of the safety program although C-I-L as a Company does have a central accident prevention section. Copies of the minutes of the plant safety meetings are sent to all other plants in the Division. We make it a point to go over all these reports and write the plants when a serious situation appears to be developing or a new idea is suggested. This is brought to the attention of the other plants by a letter from the Production Manager.

All unusual occurrences whether or not they result in an injury are investigated by the plant safety committee and a full report issued, with copies sent to the Production Department. These are carefully reviewed and a copy is sent to each plant under the cover of a letter from the Production Manager. These letters, reports and minutes are used as subject matter at plant safety meetings.

There is one thing in our program which I think has helped considerably; The C-I-L No-Accident Record Plan. In this plan the plants are classified according to the number of employees. When a plant goes a specified number of days they win the General Manager's Prize. As they continue to operate without an accident they go on to win successively the President's Prize, Board of Director's Prize and the C-I-L Prize, each prize increasing in value. When a plant has won a prize each employee receives a gift which he chooses from an assortment chosen from the local stores. The Company gives a dinner at which we try to get as many dignitaries as possible to grace the head table. The mayor, the local member of parliament, the fire chief, the police chief, some company brass and always an employee representative of the safety committee is included. Speeches are made, pictures taken, the prizes are presented by the highest company official present. After the dinner the celebration continues in order to give employees a chance to meet the dignitaries at the head table and to let them get to know better the Company officials. The Public Relations get in on the act and it is usually possible to get a group picture of some employees in the local newspaper.

(7) *Communication*

In a multi-plant operation good communications are essential to a good productive team and I think we all agree that a system of reports is necessary to good communication. The question is how much information do we want in our reports.

We always want to know how busy the plants are so we want to know production figures and we want to have the figures frequently enough to be able to initiate corrective action if it is needed. We also want to know our plant costs and usually on a unit basis $/ton. We also want to know if we are doing any better or worse so for convenience we want figures to show how we did the same month last year, and in the seasonal fertilizer business it is very important to know how we are doing to-date this year compared to last year. If we are going to keep raw materials flowing to the plants we must have some idea of the raw material inventories at the plants. In order to know how the manufacturing program is progressing we should know inventories of compound fertilizers by grade so we can compare with the sales forecast for that plant.

We have found also that it is helpful to have some comparison of the performance of each plant. Cost figures do not always give a true comparison of one plant with another or any plant's performance over the years, because raw material costs can vary, labour rates are not uniform throughout the country and the value of the dollar has been dropping over the years. However, fertilizer plants use the same raw materials, essentially the same processes, use the same equipment and ship the same products, and a man hour of work is the same at any plant on a current basis and over the years. Therefore, if you know the number of man hours per ton of product for each plant the efficiencies of each plant can be compared.

In order to help the plant managers to know how they are doing, to help them to know how they are doing opposite other plants and to give the Production Department this information, we have daily reports, monthly reports and annual reports. It is our feeling that foreman, supervisors and the works managers are anxious to know each day what has been done. While they are summarizing their daily figures all we ask is that when they are making it out they insert a piece of carbon and give us a copy. We do not require accounting figures and the report is drawn up so that the information can be entered with the least possible effort.

Our monthly report is in two sections, the first section gives production information, lists repairs, safety data, labour requirements, control analysis, inventory of raw materials and finished goods, a manufacturing schedule for the remainder of the fertilizer year and a list of visitors to the plant.

The second section shows the production information, efficiency figures. If the current month shows a variation of more than 10% from the same month a year ago or the to-date figures an explanation is given. You will note that we have cost figures for mixing, sacking and shipping and handling-in. We feel that if you are going to control costs you should know the parts that make up the whole. These figures can indicate where labour saving equipment should be provided and where studies should be made to look for better methods. We feel that savings can be made if everyone of the operating staff knows the cost of each step in the process. In order to let each plant know how its performance compares with other plants each plant receives a copy of the other plants' reports. This is an easy method of keeping all plants informed of what is going on in the Division; it also sets up some competition.

No doubt some of you will be thinking that these reports are detailed and must take a lot of the supervisor's time. I know that most of us think that supervisors should be out in the plant where they can see what is going on but they cannot be fully aware of what is going on by spending all their time in the plant. They must also have time and a place to study their costs and performance figures to find areas for improvement.

In our case most of this cost

information is required by the Accounting Department so the Works Office enters the same information in a different form (we use tons, they use pounds) and makes some arithmetical calculations. The supervisor of Works Manager, in reviewing these costs, will be searching for the reasons for any changes or variations in the cost figures from last month and last year. All we ask him to do is to put his thoughts down on paper.

All plant managers and supervisors want a record of their repairs. We ask them to record this information in their monthly report. Then it is available to all plants and the Production Department.

You might think that this looks like a lot of paper moving around. Actually it cuts down on paper or distributes it more efficiently. The daily reports we receive from the plants often contain messages which can save considerable correspondence. It certainly provides good communications between the plants with little effort.

A copy of all approved appropriations is circulated to all plants so they will know how one plant corrected a problem and they have all the necessary information to consider the same problem at their plant, and we also have appropriation experience reports. When a project has been completed and in operation for a year an appropriation experience report is submitted to those who approved the original project. This outlines what the problem was, how it was corrected and lists the results of one year's operations. This lets us know if our estimates have been correct. It might indicate further treatment. These reports are also circulated to all plants so they can see how the project has turned out, and from the basis of the first year's experience can decide if a similar project would benefit their operations.

We also have an annual report. This reviews production and operating data over a five year period. As you well know when only the current and previous year are compared you can under some conditions get a wrong impression of the plant's performance. For example, if 1958 has been the worst year in certain items for several years, 1959 being slightly better might look good. However, when the figures for five years are compared you can see the general trend.

In an annual report there are other items which cannot be included in a monthly report, and these are:

A Summary of Complaints

The Division has a complaint report system which originates with the salesmen. Copies go to the plant concerned, Sales and Production in Head Office. The plant staff completes the lower part of the form explaining if they can, what has happened and what they are going to do about preventing a recurrence.

Each year each plant summarizes the complaints under the various categories. These are summarized in the Production Department. This summary which may include several years can indicate trends and show where action is needed.

Summary of Truck Waiting Time

Each plant has electric stamping clocks at the shipping clerk's desk and on the shipping platform. When the trucker or customer receives his order the time is stamped on the top. When the loading is completed the shipping foreman stamps the time. At any time the Works Manager can find out what kind of service the plant is giving by having one of the clerical staff take off the time intervals from the bill of lading. He is also able to reply factually to any complaints on service. At the end of the season these are all summarized showing for each day the percentage of the trucks waiting for less than one-half hour, one hour, two hours, etc. This lets Production Department know which plants are giving the best service and if money is needed for additional facilities these summaries are a great help in selling the idea to Management.

Summary of Tractor Lift Truck Expense

All tractors and lift trucks are equipped with hour meters. A record is kept of the fuel consumption, and repairs for each tractor or lift truck and shown as a per operating hour cost. This was started some time ago when we thought that by keeping a record we could see if one model or make of tractor was better than another. We do know what it costs us to operate tractors, we know how diesels compare with gasoline, but we were surprised to find that as a tractor got older it became cheaper to operate and the plants were sometimes wanting to trade-in the machine with the lowest cost. The reason was that new models were easier to operate and some times had more capacity so the older tractors were kept on light jobs, lowering their operating costs.

While we are talking of tractors, at one time we owned enough tractors to handle the peak days of the year but at such times had no spare. We found that when a tractor became an unreliable machine it was traded in on a new machine, the allowance for the old machine was about $1,000. However, if such machines were tuned up and kept as a spare they could often fill in for a day if one of the newer machines failed. We follow this policy at all plants.

Handling Loss Experience Report

As we all know you cannot take in 50,000 tons of raw materials into a plant and ship 50,000 tons. There is a handling loss which can go as high as 2%. This I am sure is a problem at all plants and I am pleased to know that the National Plant Food Institute is making a study of this problem. We attempt to get as many showdowns as possible on both raw materials and finished goods and once a year these are summarized and our comments attached. We know there are losses in moisture, dust losses, some of which might be in the plant on beams and ledges, but not accounted for. There is always the problem of accurately measuring any bulk inventories. It is the Production Department's function to explain these things to management and the Accounting Department. This is some times a difficult job as the handling loss on one particular material may vary from year to year. We have found that often in such cases a more accurate figure can be obtained by going back a few years if the figures are available. In getting showdowns where the measurement of bulk stocks is involved the measurement may be inaccurate and finally when a good clean showdown is obtained a large shortage is found. If this is considered over the entire tonnage handled since the last good clean cut-off the loss as a percentage of the quantity handled is often found to be reasonable. The Production Department's problem is explaining these abnormal shortages on single items. To re-assure ourselves we prepare with the annual summary a table showing for each plant the quantity in and the quantity out of the plant during the period under review. The quantity on hand at the start of the period plus the quantity handled, based on suppliers' invoices, represents the tonnage into the plant. From this we subtract the sum of the quantity shipped as shown on our invoices and the inventory at the end of the period. This of course is subject to some errors since some inventory figures may not have been verified. However, it gives an approximate value to handling losses and from our experience the overall handling losses approximated in this way are much lower than some of the individual shortages and can have a reassuring effect.

We all realize that one source of losses could be over-weight bags and we have a variety of systems to try and control bag weights. At some plants we have all bags passing over a check weight scale controlled by an electric eye; at other plants bag weights are sampled by weighing groups of bags at intervals during the shift and recording the weights on a control chart. However, to have some additional assurance we arrange, at plants having truck scales, to weigh loads picked at random during the shipping season. These weights not only indicate average bag weights but also indicate incorrect bag counts which can also contribute to a shortage.

In the foregoing I have pointed out the many uses of these reports but I did not mention one important point. By making it necessary for the works staff to submit these reports, daily, monthly, annually, you are assured that they are aware of what is going on in their plants and you know that they have searched for the reason for the changes. It also gives you an opportunity to judge the ability of your Works Manager or Staff to analyse the plant operations from figures given them by the works office.

Conclusions

I have given you some idea of the type of organization and the systems we employ in operating our fertilizer plants and as I stated at the start of this paper I hope it will stimulate discussion on this subject.

CHAIRMAN SPILLMAN: Thank you, Mr. Clegg, for giving us this fine report. We appreciate the time and effort you spent in getting this report up. I am satisfied a lot of the items that you mentioned can be used very nicely in our own operations. The next speaker is Mr Elgin Doidge, also of the Canadian Industries Ltd.

Mr. Doidge is Processing Engineer as I understand it; he's in charge of all technical problems in the seven fertilizer plants at Canadian Industries. Mr. Doidge has had many years of experience in fertilizer technology and other chemicals handled by the Canadian Industries and Mr. Doidge will take up some of the discussion at this time.

E. C. Doidge

IN May, 1957, the construction of three continuous granulation plants was authorized by the Board of Directors of Canadian Industries Limited. The first of these plants at Chatham, Ontario, came into production in June, 1958, and the remaining two in November of the same year at Hamilton and at Ingersoll, Ontario.

I will try to outline to you how we arrived at decisions influencing the basic design of these plants and of the multiple problems faced in building three plants simultaneously wherein lay the danger of making three mistakes for each error rather than just one.

C-I-L, as Mr. Clegg has mentioned, is a composite company of several operating divisions and service departments; among the latter are an Engineering Department and a Central Research Department.

In this period of rapid technological change it may be of interest to relate just how C-I-L got into the fertilizer business in the first place. A little over thirty years ago the ammonia oxidation process for the production of nitric acid became well established. For some 60 years prior to this, C-I-L and its predecessors had operated explosives manufacturing plants—in fact they were the basis of our corporate beginnings—and of course considerable quantities of nitric acid are required in explosives manufacture for the nitration of cellulose and toluene or glycerine.

In the production of concentrated nitric acid by the ammonia oxidation process, substantial quantities of dilute—68-70% sulphuric acid are a by-product, resulting from the concentration of the dilute nitric acid. Prior to 1928 when nitric acid was made by the Soda Process, this waste acid was used again to react with sodium nitrate to make more nitric acid. However, with the introduction of the ammonia oxidation process there was no such use for the dilute sulphuric acid. The company surveyed potential markets for this dilute sulphuric acid and decided that the manufacture of superphosphate was the best potential market. A survey of manufacturing processes available at that time resulted in a decision to buy the "OberPhos" Process which had been developed in 1926 by Gustaf Ober and Company in Baltimore. This company was later incorporated into the Davison Chemical Company which is now a part of the W. R. Grace & Company.

One plant using this Ober Process was built at Beloeil, Quebec, where an explosives plant has been operating since 1878, to serve the Quebec and Maritime provinces and in the following year a second plant was built at Hamilton, Ontario, where C-I-L already had a sulphuric acid plant in operation.

Of course, having acquired the Ober Process, the first thing our Research Department started out to do was to improve its efficiency and over the years there has been a steady improvement in operating efficiencies. Originally designed for 50,000 tons per year, each plant can now make 150,000 tons annually. It is interesting to note that the Ober Process has incorporated for over thirty years the basic flow mechanisms of the so-called TVA "Cone Mixer," the only difference being that in the Ober Process, the mixing action is carried out at elevated pressure and much higher flow-rates in the order of 300 tons per hour are experienced during the charging cycle.

From being in the superphosphate manufacturing business, it was only a short step over into the manufacture of mixed fertilizers which began in 1930.

Through our former joint ownership by and association with the DuPont Company in the United States the use of ammoniating solutions was tried by C-I-L as far back as 1933 with full plant trials being made under the direction of Dr. Keenen of the Du Pont Company, who was a pioneer in this field.

Through the work of our Research Department in studying the "Ober" Process and in the investigation of the many problems in trying to make non-caking mixed fertilizers, we were gradually led into a study of the mechanics of granule formation including the direct use of sulphuric acid, and in 1953 constructed a pilot plant for the continuous production of granular fertilizer which operated throughout 1954 on a full program of formula investigation and many other problems. This plant built in 1y53 had a rotary ammoniator 4′ x ′ in size and a cooler, but no dryer.

At the same time our Central Research Department was doing this work, our Agricultural Chemicals Division staff visited from 1952 onwards most of the fertilizer granulation plants being built in the mid-western States, as well as in Europe and in Great Britain.

It was therefore with this background that we faced the problems of incorporating and translating all the information gathered into the design and construction of the three granulation plants. I would like to say that we appreciate very much the opportunities of visiting many plants in the United States and of exchanging ideas with the operating personnel, many of whom are here today.

Product Specification

Arising out of many discussions and investigations into the caking properties of fertilizers, both granular and powdered, we had arrived at the conclusion that to ensure non-caking properties in the granulated product, the dried granular fertilizers should have a moisture content of from 0.5% to 1% free moisture.

We also had decided that the product should be sized between 6 and 14 mesh, i.e. about 1 to 3 millimeters with not more than 10% of the bagged product finer than 14 mesh. Since the granulation process was adopted to meet a higher competitive market situation, it was necessary that the product should be of the optimum quality.

Since the entire production of the three plants was to be converted to granular fertilizers we were faced with the problem of granulating substantial tonnages of 0, 2% and 3% nitrogen grades.

Selection of Process Equipment

Ammoniation

After visits to U. S. plants which had been in operation for two years or more by this time (1956) we decided in favour of the TVA type rotary ammoniator rather than the double or single shaft pug mill, the basis for our decision being the high repair costs and high power input required for the pug mill, without, so far as we could determine, any corresponding increase in efficiency of granulation. We were influenced in our choice of separate ammoniator and granulator units by the operating difficulties seen at plants operating a combined unit.

Dryers

At many of the plants visited, the opinion was expressed that the dryer capacity was the limiting factor in the plant out-put. Therefore, and especially because we were expected to granulate 0, 2% and 3% nitrogen grades, which would require a high moisture content to produce granulating conditions, we made sure that we had ample dryer capacity both in heat in-put and retention time. The dryer size first proposed by the equipment supplier was 7′ diameter and 50′ long, which we felt to be too small. Our dryers are 8′ diameter and 60′ long with a slope of ⅜″ to the foot and a retention time of 28 minutes. The maximum heat in-put is 25,000,000 BTU per hour. To-date our maximum actual in-put has been about 18,000,000 BTU when granulating at 30 tons per hour.

Since the fuel required is an important element of the cost of granulating fertilizers our choice of fuels was carefully considered. At our Chatham plant, natural gas was the most economical fuel but it was not available at an economical price at either Ingersoll or Hamilton. The differential in price of Bunker "C" fuel compared to lighter grade fuels of some 4¢ per gallon led us to select Bunker of 60,000 tons per year with an average fuel consumption of 4 gallons per ton, annual fuel savings are about 10,000 dollars. The burning of Bunker "C" oil does present some problems not all of which have been solved to-date. We are using North American Model 212-8B burners with two burners on each dryer. The use of Bunker "C" oil of course requires the installation of preheaters to ensure that the oil is at the correct temperature and viscosity to be pumped and handled through the burner.

Air flow through the dryer is rated at 40,000 c.f.m.

Cooler

The cooler shell was supplied in the same size as the dryer i.e. 8′ diameter and 60′ long with the same air flow rate of 40,000 c.f.m. In actual experience, we have found this air flow is greater than that required to cool the fertilizer down to 100°F and we have now reduced the air flow through the cooler to a maximum flow of 20,000 c.f.m. during hot weather operations and down to a minimum of 5,000 c.f.m. when the air temperatures get down below zero, as they frequently do in our Canadian winters.

The drive units on the dryer and cooler shells are all identical 60 H.P. gearmotor units. One spare gearmotor provides protection for the 6 units at our three plants. A problem arose during construction. There is a 6″ air gap between the pinion and ring gear, although the gear reducer frame is up against the dryer shell. This was caused by relocating the drive from one side of the shell to the other without turning the gearmotor through 180° on the drawing boards. We solved this dilemna by turning the gearmotor through 180° and then relocating the ring gear on the shell to match the position of the pinion.

The double deck Tyler Hummer Screen 4′ x 15′ equipped with 3 V-50 vibrators was seen in service in many of the plants visited and was given good recommendations. In our design, we provided for ventilation of the screen housing as a dust control measure. We did not include a movable separator plate under the lower deck of the screen because we did not want to leave in the plant operator's control, a variable factor affecting the product size specification.

Fume Control

The problem of dust and fume control were two points on which a considerable amount of investigation was done. During our visits to U. S. plants, at practically every plant visited in 1956 and 1957, conditions were observed in stack exhausts which in our opinion would have caused us problems. Plant location is however a critical factor in establishing the amount of fume or dust emission which can be tolerated from any plant and what is satisfactory in one area may not be in another.

All three sites at which our granulation plants are located are within or on the edges of built-up municipalities.

Besides this, the Province of Ontario passed in 1957 an Air Pollution Control Act with enabling legislation designed to permit towns and cities or townships to establish local by-laws on air pollution within certain designated limits.

A careful search of the literature at this time—July, 1957—revealed only two published papers dealing with the level of fume and dust emission from fertilizer plants and the problems resulting therefrom.

Two separate problems were felt to exist:

1) The Recovery of fume—defined as submicron particles which were thought to be largely ammonium chloride formed in the ammoniator and granulator and to a lesser extent in the dryer, and

2) Entrained dust particles either of ungranulated materials or particles formed in the dryer and cooler by abrasion of the falling particles in the normal dryer action.

High efficiency cyclones can be expected to remove substantially all particles larger than 5 microns. For smaller sized particles, the problem of removal becomes more difficult and there is a choice of Venturi Scrubbers, Electrostatic precipitators, wet fan scrubbers of various types and bag filters.

From our association with Scottish Agricultural Industries, we learned that a Venturi Scrubber would remove fume particles from the gas stream since S.A.I. had had two years' operating experience with a Venturi Scrubber on a fertilizer unit. Through the cooperation of a U. S. chemical company we were also able to witness a test of a Venturi Scrubber handling fume from a dryer, after the gas had already passed through a wet scrubber fan. We were thus assured that a Venturi Scrubber would quite effectively remove fume from a gas stream. After assessing the cost differentials of various equipment we decided on Scrubbers with a 10,000 c.f.m. air capacity to scrub the gas stream from the ammoniator and granulator exhausts.

This air capacity of 10,000 c.f.m. was designed on the basis of using an air blast of 5,000 c.f.m. on the ammoniator bed when necessary which our Central Research Laboratory had recommended from their pilot plant experience. Such an air blast system was installed at Chatham but full scale plant trials did not indicate any benefit from its use and it was abandoned after several months' trials.

As a result, the Venturi Scrubber systems are considerably overdesigned in capacity. In order to maintain their efficiency however, it is necessary to maintain air flow at the rated capacity. Air is drawn into the in-let through a T connection and the damper is adjusted to prevent fumes from coming out of the ammoniator or granulator fume hoods.

The danger in having efficient fume removal equipment, however, lies in the possibility that plant operators may become careless in sparger maintenance and cleaning and this is a point which is continually being emphasized to the operators.

Water in-put to the Venturi is about 60 gallons per minute at 5 lbs. pressure and the fan is powered by a 75 H.P. motor. Air velocity through the throat of the Venturi is about 180 m.p.h. The Venturi itself is fabricated out of Type 316 stainless steel; the water separator tank is of mild steel with a PVC lining. The fan and exhaust ducts are also mild steel lined with a PVC coating.

Performance of these units has been quite satisfactory.

From a calculation of the dust loading in the exhaust gases from the dryer operation reported in the two papers published up to that time it was estimated that an annual stack loss of 700 tons of fertilizer per year could be expected on the basis of 3,000 hours operation based on a dust loading of 1.35 grains per cubic foot of exhaust gas. This loss would be expected after the exhaust gases had passed through cyclones which would remove most of the dust particles in excess of 5 microns in diameter which is about as effective a performance as could be expected with a cyclone.

We reviewed the cost of a Venturi system for the dryer but, whereas there was very little difference in the cost estimates for a 10,000 c.f.m. Venturi compared to a wet fan scrubber of the same size, at 40,000 c.f.m. capacity the capital cost of the Venturi unit was about twice that of the Rotoclone, as was the power consumption. The best economic method of recovering a part of this loss seemed to be some type of water scrubber and after reviewing the different types available on the market, a type W 45 Rotoclone was specified for our Chatham plant with a settling tank and recirculation pump installed to reduce the water consumption. A sludge pump was provided to pump the thickened solids back to the ammoniator. All water required in the ammoniator is taken from this tank. It has been found necessary to supply supplementary heating to this water and this is done by injecting low pressure steam through a steam-water blender to avoid scaling problems which would have been encountered in a heat exchanger.

When granulating "0" nitrogen grades a high corrosion rate is experienced on the spray pipes in the ammoniator.

However, despite the selection of this wet scrubber fan, some reservations were harboured about its efficiency in fume removal and also the maintenance costs. The plant at Chatham was under construction by this time and detailed plans were being prepared for the Hamilton and Ingersoll plants by our own Engineering Department.

Along about mid December, 1957, it was learned that a bag filter installation had been in operation on the dryer gases in a fertilizer plant in the midwestern United States and another fertilizer company was planning a similar installation. Arrangements were made at once to visit these plants in January, 1958. At both plants, the bag filters were operating satisfactorily after experiencing some start-up difficulties but actual bag life was in some doubt. However, despite the possibility of having to replace all the bags every 18 months, our assessment of the advantages to be gained by the recovery of all entrained solids, including ammonium chloride fume, at a value of $30,000 per year showed that a substantial return on the required investment should result. We also carried out plant trials on a small bag filter installation on a phosphate rock grinding unit at one of our own plants. We generated ammonium chloride fume by reacting anhydrous ammonia and anhydrous KCl. This test revealed that a bag filter would remove ammonium chloride fume from an air stream.

Our Engineering Department made a careful analysis of the capital cost and annual operating cost of the four alternative dust collection systems studied.

Scheme A was a dry fan and three cyclones. Scheme B for a wet scrubber fan and three cyclones. Scheme C for bag filter equipment and Scheme D for a Venturi Scrubber.

Scheme A was ruled out because it would not do the job which we felt had to be done at our plant locations. Of the remaining methods, Scheme C offered annual savings in operating costs of almost 13,000 dollars over Scheme B at an additional operating investment of 47,000 dollars or an annual return of 36 percent before taxes on the additional investment.

On this basis, we obtained management's approval for the additional expenditure required for the installation of bag filters, amounting to about an extra 47,000 dollars per plant and our Engineering Department proceeded to complete the design incorporating thees units into the plants.

At each of the three plants complete batch dry mixing facilities were already provided.

The problem of where to put the granulation equipment into the existing plant or whether to put in a separate building is always, I think, a very difficult one. At least we found it so. At our Chatham plant, it was somewhat easier since there was no available space in the existing buildings and it was felt that no storage space could be sacrificed.

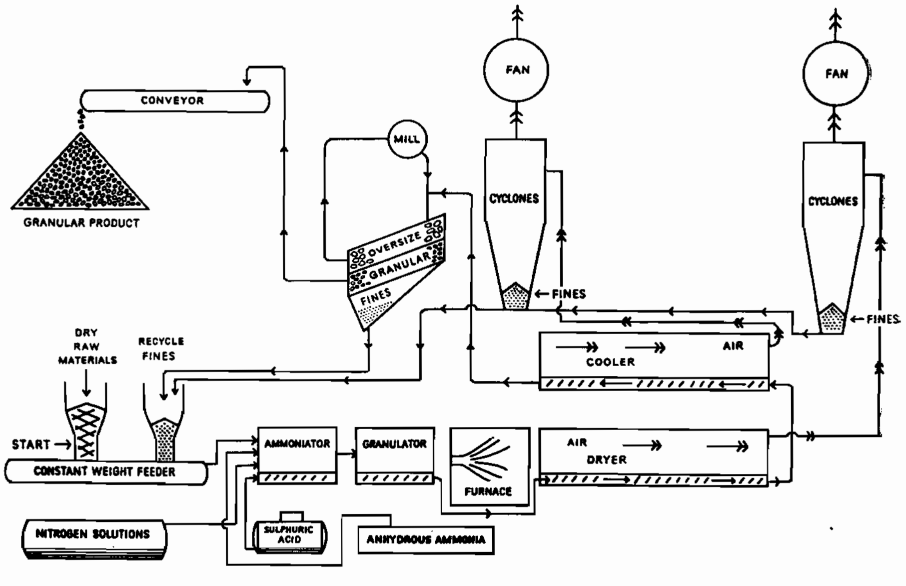
The only available space was an area at the end of the existing storage building which was about 265 feet from the dry mixing plant. However, adequate space was available in this area and there was also room for acid storage tanks and an anhydrous ammonia storage tank. A transformer station was also installed quite close to the granulation plant to minimize line transmission losses at the stepped-down voltage of 550 volts which is the usual operating voltage for industrial equipment in Ontario. Power is purchased at primary voltage of 27,000 volts.

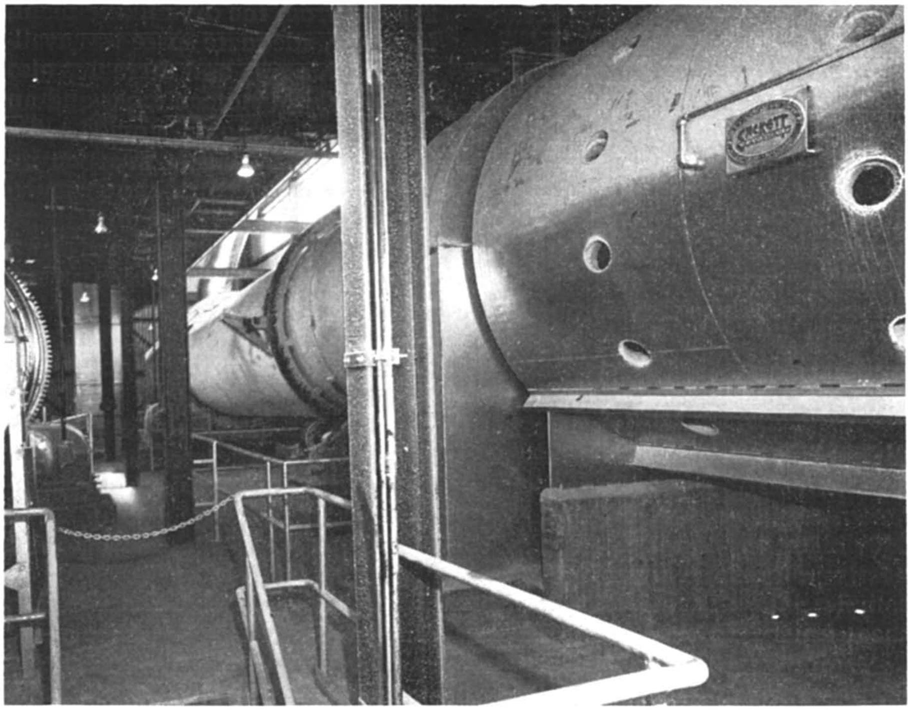
The equipment lay-out in the buildings was arranged by our own Engineering Department in consultation with the major equipment supplier— A. J. Sackett and Sons of Baltimore, Md.

The building at Catham is a four storey building 100 feet long and 32 feet wide of steel frame construction with suspended re-inforced concrete floors in the main operating area and steel open grid type floor in other areas. Siding material is corrugated asbestos cement with ventilating louvres at the top and fibre glass illumination panels in the side walls.

Since a separate ammoniator and granulator are used, an additional floor may be claimed by some to result. However, the critical height was established not by the ammoniator granulator floor level sequence but by the height of the cyclones above the second

CANADIAN INDUSTRIES LIMITED
FLOW SHEET-GRANULATION PLANT-CHATHAM





Huge drier 60 feet long and eight feet in diameter at the new granular fertilizer plant of Canadian Industries Limited at Chatham, Ont. Forepart is the gas-fired furnace which generates the heat for the drying process.

floor level where the dust discharge of the cyclones was returned via a screw conveyor to the fines recycle hopper suspended from the second floor.

Recycle fines consist of dust recovered in 3-24 feet high by 6 feet diameter cyclones on each of the dryer and cooler units and of the −14 mesh material passing through the lower screen.

Oversize material is recycled through a chain mill and returned to the elevator handling the cooler discharge for re-screening.

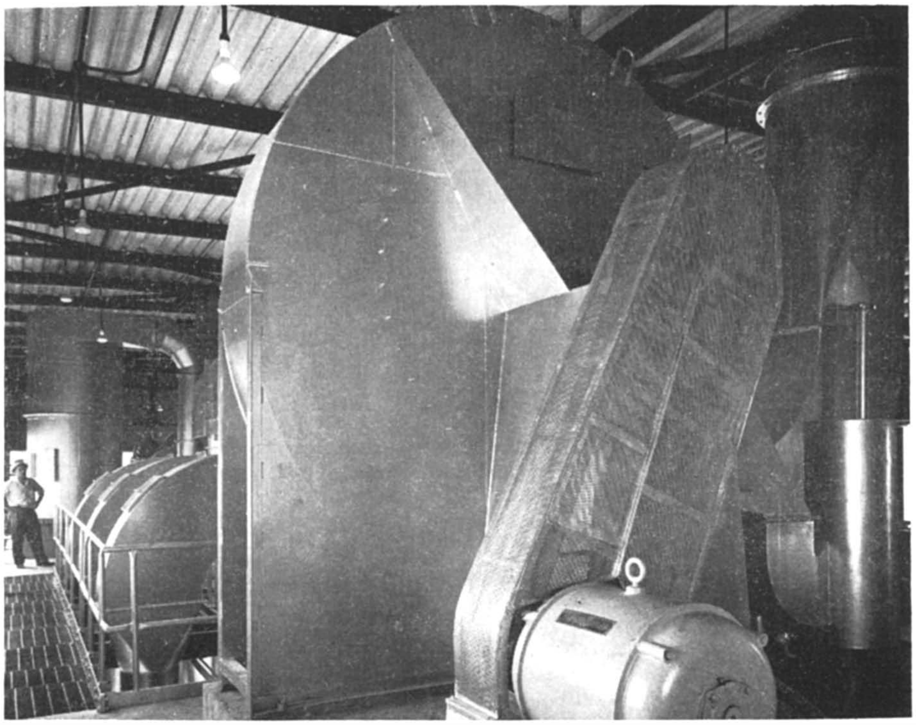
Separate weigh feeders are installed to feed the raw materials and the recycle fines at predetermined rates. High and low level indicators are installed to warn the operator when the hoppers are in danger of becoming empty on the one hand or too full on the other.

Construction of this plant was handled by the small plant maintenance forces with a resident engineer from our Central Engineering Department aided by an erection supervisor from our equipment supplier. Major trades were subcontracted. Ground was broken in October and the plant commenced production in June of the following year.

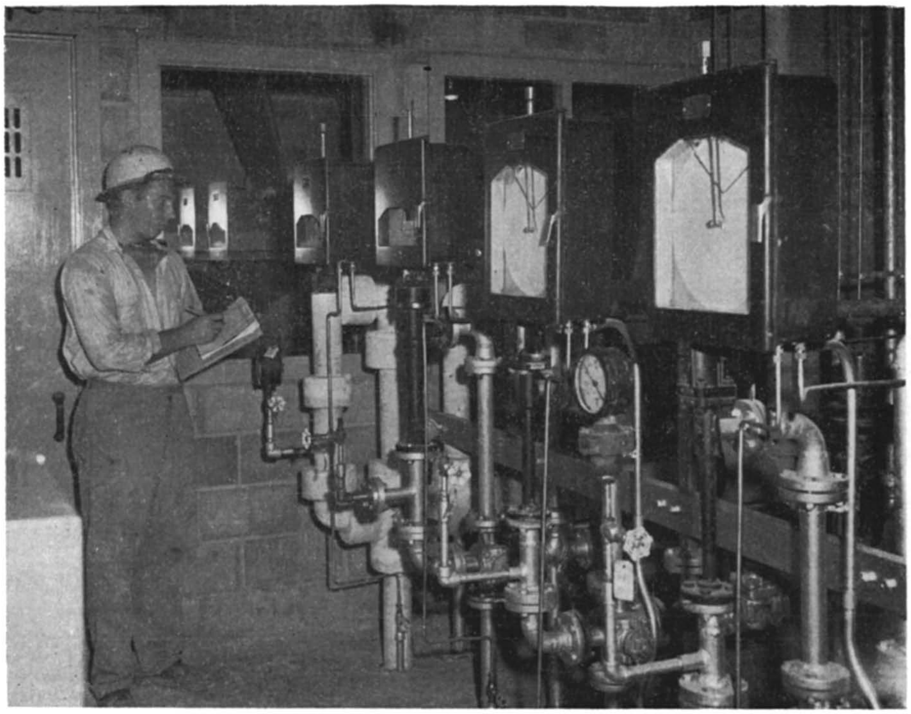
After erection got underway at Chatham, the design of the Hamilton and Ingersoll plants, which had been delayed pending our final selection of dust collection equipment, was being completed by the Engineering Department.

At Hamilton the plant location was decided on fairly readily and a good location was possible close to the existing dry batching system and it was quite easy to receive the dry premix and return the finished product to existing conveyor systems. At Hamilton again we elected to handle the construction using our own maintenance staff which was much larger than at Chatham. Construction started in May, 1958, and production began on 20th November, 1958.

The last plant design to be completed was the Ingersoll plant. At this location, we felt that from an operating view point we could install the equipment within the existing building with some changes in roof elevation and in fact had almost completed a design after many different tentative arrangements when the change was made in dust collection equipment. In order to fit in this larger sized equipment, another series of layouts were prepared. In fact, all told about 15 different layouts were studied in some detail before being rejected for various reasons. This study took considerable time and a model of the existing plant was finally built to study quickly possible equipment arrangements. Now a model can be as complicated or as simple as necessary to do the job required of it. This model was built in two days time for a total labour in-put of about 20 hours and a material cost of



This huge 12-foot wet-scrubber fan, driven by a 150 horsepower motor and revolving at a speed of 560 r. p. m. pulls dust laden air through the drier and a battery of cyclones before discharging the cleaned air to atmosphere.



Operator checks 24-hour recording charts showing flow of water, sulphuric acid, nitrogen solution and anhydrous ammonia into ammoniator. Control room is fully automatic.

$15.00. All existing steelwork, conveyor belts, process equipment were located to scale.

After a few days of making tentative equipment arrangements with this model and with engineering estimates of the cost of installing new steelwork to carry the new loads, since the existing building steel was too light, we came to the conclusion that it would cost no more to build the granulation plant as a separate building than to "stuff" it into the existing building.

However, this entire plant site had been acquired as a going business the previous year, but very little was known of soil conditions in the immediate area. The plant is located in a valley of recent geological origin and some changes had occurred in the river bed within living memory. Therefore we had some soil test holes drilled and in one of the holes we encountered no resistance for a distance of 3 feet, between the 6 foot and 9 foot level. Over the entire area we found bed rock at 16 to 18 feet. Therefore our Engineering Department decided that piling was necessary and a total of 86 piles were driven. These were "Franki" piles—an 8″ diameter steel pipe is driven into the ground by dropping a ram inside the pipe onto a plug of dry mixed sand and cement at the bottom of the pipe. The friction between the pipewall and the sand cement mixture transfers the energy from the falling ram to the pipe. Since the entire pipe length merely follows the lead, old or salvaged pipe can be used. The pipe is then cut off at the proper length after refusal and filled with reinforced concrete. In our design, piles were driven under each main column and the pile tops were rigidly held by a concrete cap over the group of three or four piles.

Because of all these delays, construction did not start until June 10th, 1958, at Ingersoll, and here we chose to use the services of a general contractor who had done considerable work for C-I-L in order to speed up the construction phase. This plant started into production on 15th November, five months after the first pile was driven.

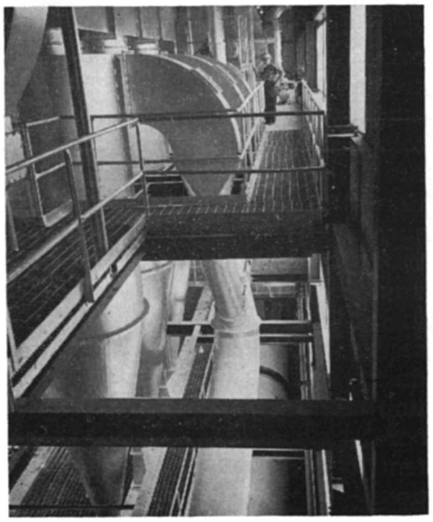
A few start-up difficulties may be of interest.

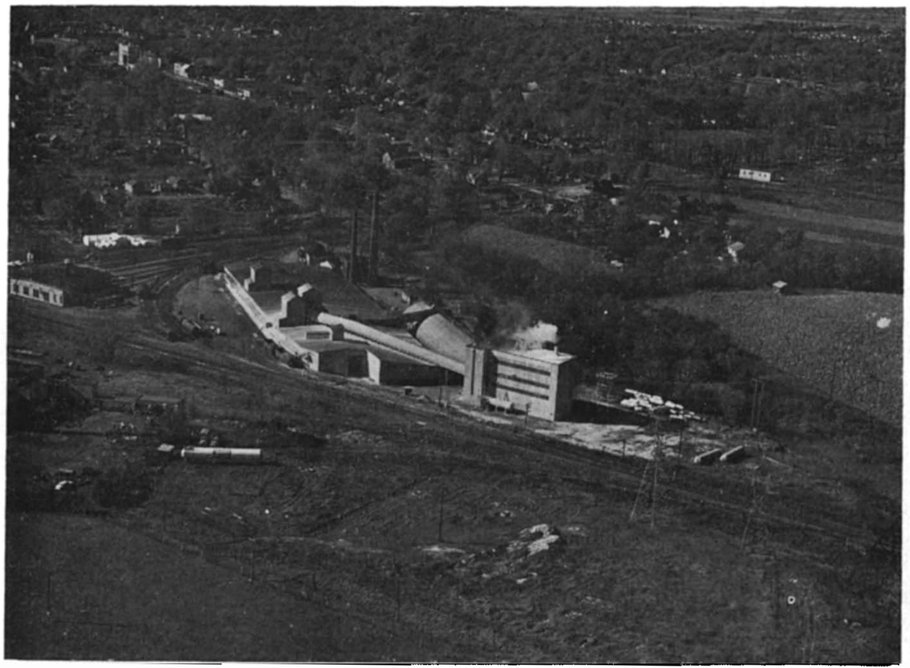
To illustrate one of the problems encountered in starting up these plants, a low volume high pressure pump had been installed at Ingersoll to supply process water from an adjacent river for the Venturi Scrubber and for addition to the ammoniator when required. A large volume low pressure pump and main were already installed which had previously served a rendering operation at this plant now discontinued.

When we started up for the first time, everything went pretty well at first. Then we noticed a heavy fume coming out of the Venturi Scrubber. What is wrong here? We checked the ammoniator first—nothing too bad there for a start-up operation. Then we checked the Venturi. Ah, ah—no water going through; guess we forgot to turn on the valve. Open the valve—no pressure. Check the lines back all the way to the ground—everything o.k.—then back out to the pump well by the river bank. Pump running o..k—what, no water in sump. That's right—no water in the sump! An abnormally dry fall had just lowered the water level in the river to an all time low and this had not been noticed, although a week earlier the pump had been checked out o.k. in a test run. At once, we built a weir out into the river and soon we had all the water we needed. However, more than the river was "damned" that day, as it was at other locations on other days.

We had of course checked over all mechanical equipment and process liquid lines had all been pressure tested and flow meters zeroed and checked over. Despite all this at one plant during start-up we could not get any nitrogen solu-

An elaborate dust control system keeps the new C-I-L granular fertilizer plant at Chatham, Ont. as clean as a tidy kitchen. Here, we have a view of three floors of the plant showing cyclones on left with inlet ducts leading to them.





Aerial view of the fertilizer plant of Canadian Industries Limited at Chatham, Ont. The granular fertilizer building, at right, was completed last year and is one of three granular units built by C-I-L in Ontario. The other two are located at Hamilton and Ingersoll.

tions to flow. We checked the sparger and checked every valve back to the tank car but still no solutions. We dismantled the flow control valve to ensure that it was not working backwards since we had already experienced this at another location.

Finally, we shut off the tank car, released the line pressure and started to dismantle the line. Fortunately we started at the top to work down (always nice work if you can get it) and at the first flange we opened we found an old rag wadded into the solution line just outside the ammoniator. Apparently a pipe fitter had tucked it in there to keep any dirt from entering the line and causing any trouble, while the rest of the line was being pressure tested.

At two plants, we experienced considerable trouble due to dust accumulation from the cooler stack settling on the 27,000 volt insulators on the primary power lines supplying the transformer substation and also at one location, on a nearby public utility 110,000 volt transmission line. When humid weather followed a prolonged dry spell without rain to wash off the accumulated dust, the accumulated dust would pick up enough moisture to permit severe arcing to occur on the insulators to the extent of causing power interruptions.

We learned of a silicone paste sold by Dow-Corning under the trade name of DC-5 which was advertised as overcoming this problem so we secured a supply and applied it to all the insulators where trouble was experienced. Since that time we have not had a single power interruption due to insulator arcing. The DC-5 paste is applied in a thick film and it literally swallows up the dust particles—encapsulates them—so that the dust particles cannot pick up moisture and form an ionized solution which formerly provided the current path producing the insulator flash over.

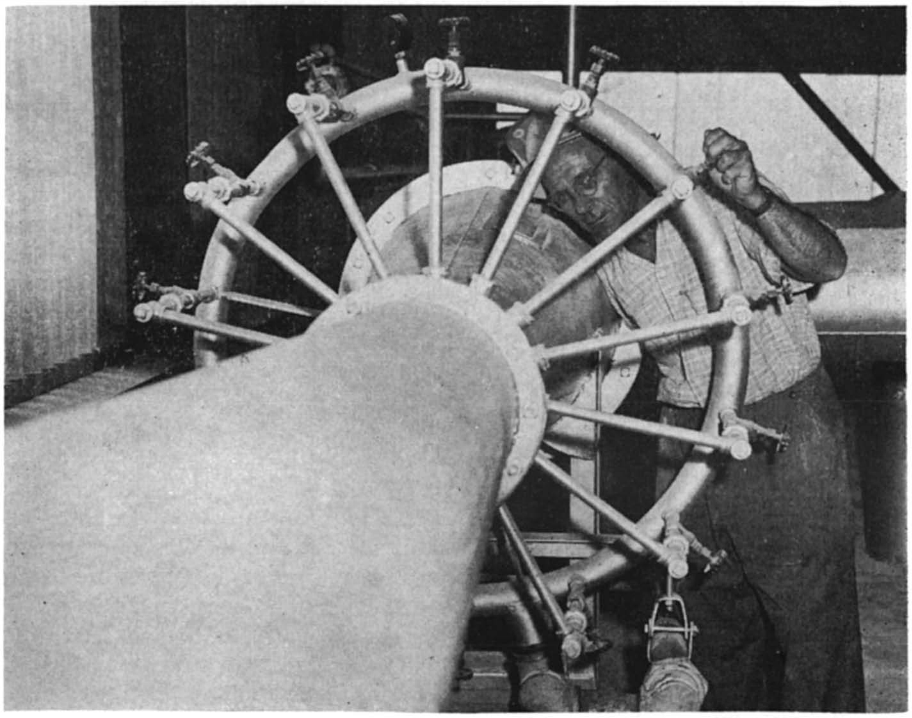
We are currently investigating dust losses from the cooler stacks at our plants. In our visits to U. S. plants, the dryer and ammoniator stacks appeared to be the worst offenders and relatively light stacks were observed on the coolers.

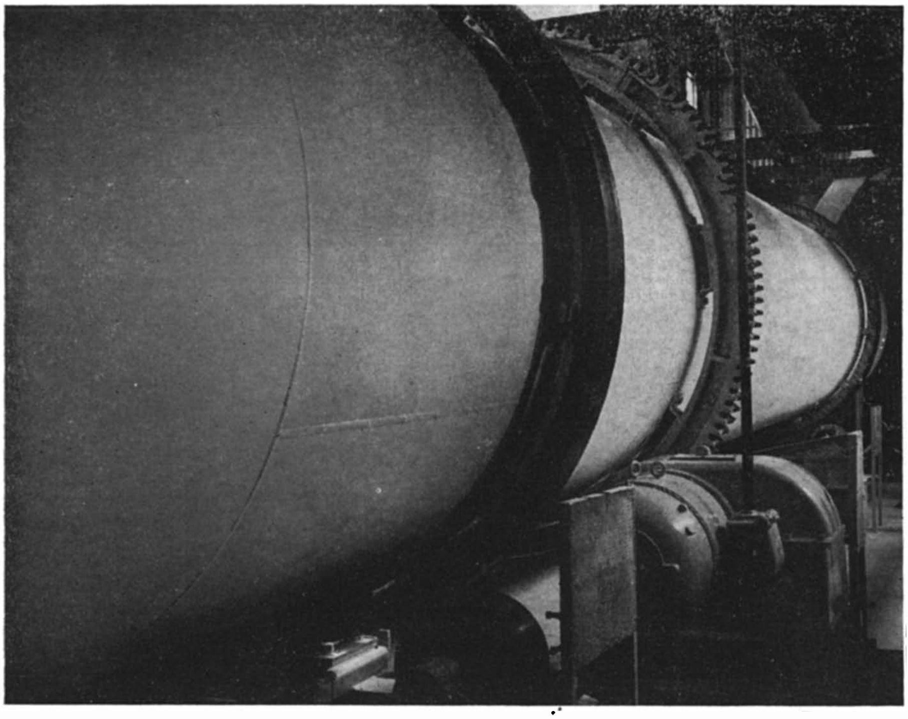
However following our troubles with the insulators, we are presently investigating dust losses from the cooler stack and finding some surprising results.

In our thinking at the time we were designing the plants, we felt that all the fine dusts would be removed in the dryer air stream. However, while this may still be true, there appears to be a considerable amount of very fine dust produced in the cooler by the cascading action of the granules.

In a sampling of the cooler stack on a run of 10-10-10 which gives quite hard granules, we have found dust loadings in the exit gas after passing through the cyclones of 1.25 grains per cu. ft. At the reduced air flow of only 20,000 cubic feet per minute, this produces a dust loss to atmosphere of almost one ton of fertilizer on every 8 hour shift. I am sure that

Jets of water in this Venturi scrubber remove undesirable fumes from off-gasses. C-I-L's granular fertilizer plant at Chatham, Ontario.





Great volumes of air are forced through this revolving cooler which brings the hot fertilizer from the drier down to room temperature.

if someone was stealing one ton of fertilizer from your plant every shift, you would soon do something about it. At the plants equipped with bag filters, we are planning on recycling the cooler exhaust air to the dryer since we know that the bag filter units will remove any fume produced by introducing fine dust into the combination chamber.

Another most unusual and totally unexpected fault was finally located in the internal wiring of one of the oil burner safety control units. We spent about two days tracing that one down and finally our instrument engineer, who had also done a good part of the instrument drawings and so was intimately familiar with the wiring arrangements, came to the conclusion that the "Protecto-Glo" unit had been incorrectly wired by the manufacturer. If any one of you have ever had to replace one of these W-124 A relays, you will know that the enclosing box must have been designed for the unit to be installed by trained mice. By springing two sides of the box apart as far as possible with vice grip pliers, we were finally able to remove the relay and our inspection soon confirmed our instrument engineer's diagnosis. I suppose there always has to be a first time for everything and we were assured by Minneapolis-Honeywell that this was it. But why, oh why, don't they make the enclosing box just a little bit bigger. One would almost think the unit was designed to go into the limited confines of a space vehicle.

One feature of our plants which I would like to mention briefly is the instrumentation. In our visits to U. S. plants we found a wide range in the level of instrumentation from practically none to fully automatic recording controlling and intergrating types of meters. At some plants we observed the operators making very frequent manual adjustments to the acid and solution valves as the bed appeared a little too wet or too dry. In short the process was still very much at the mercy of the operator. In the hope that fertilizer manufacture may some day emerge from an art to a science, we installed automatic indicating, recording and controlling liquid metering equipment at all three plants on all the liquid lines.

Only rarely, however, in our trips did we find either these instruments or electrical control equipment protected from the dusty atmospheres present in most fertilizer plants. At its worst, this condition was almost but not quite as bad as the condition reported here a few years ago by a sales engineer of a company making weigh feeders who reported on being called to a customer's plant on a complaint that a constant weight feeder was not working properly. After twenty minutes digging with a shovel, he said he finally uncovered or was it "unfertilized" the weigh feeder?

Now it seems to us that if we were going to spend $10,000-$12,000 on process flow instruments and on furnace control instruments, and $13,000 for the complete electrical control center, we should be prepared to spend some money to safeguard this investment and so we built two separate fully enclosed rooms—one to house the electrical control equipment and one to house the process control equipment. Both these rooms are maintained at a slight positive air pressure by outside filtered air to minimize the entrance of dust and dirt into these rooms. Thus far, I think I can say that we have had no maintenance expenses on any of this equipment which could be laid to dust or dirty conditions. I think the additional expense of providing adequate protection for these rather delicate instruments has been fully justified and I would recommend it to anyone.

Corrosion is always a big problem in fertilizer plants and we spent considerable time in our design phases in trying to minimize it. We had settled on a steel frame construction for these granulation plants and considerable portions of the steel horizontal members were protected by suspended concrete floors.

We examined the use of a protective tape to be applied to all structural steelwork, piping and conduit to prevent corrosion and this material looked very good. It was specified for our Chatham plant but fortunately for us, we learned of the experience of Imperial Chemical Industries Limited who had used this material for protecting piping in a plant. A relatively minor fire in an upper floor level was started by a welder's torch. The protective paste in the tape melted, caught fire and dripped down to the lower banks of piping where it ignited the tape there and the resulting fire completely destroyed the plant equipment and building. An ideal tape would provide protection against corrosion and be fire resistant too. So far as we know, there is no such tape on the market. When we learned of the fire hazard of this tape we stopped its use at our Chatham plant.

The exposed steel has been protected by a primer coat of red lead followed by two coats of an asphalt chromate emulsion paint. This gives a relatively soft finish which is its only disadvantage but so far it is standing up very well. For siding material, a corrugated asbestos cement sheet was specified and day time illumination was provided by a row of corrugated translucent fibre-glass reinforced plastic panels. Architecturally this gives a satisfactory appearance to the building and the absence of any window frames to paint or rot over the years should give a very low maintenance cost to the building.

We were prevented by the Ontario Electrical Code from using cab tire cable for electrical distribution lines which we had seen in some U. S. plants. A rigid or semi rigid type of conduit is required in industrial plants in Ontario. Our experience with BX cable in fertilizer plants had not been good —5 to 6 years maximum service and rigid conduit lasted about 10 years unless painted every 2 or 3 years. The Electrical Engineering Section of our Engineering Department strongly recommended that we use a polyvinyl chloride coated conduit and this recommendation was adopted since the additional cost of the PVC coatings was less than 3 paint jobs and the life of it was considered to be much longer. This material requires careful handling during installing to ensure that the coating remains unbroken and particular care is necessary in cutting and threading the rigid conduit. If a cut is accidentally made, however, it can be covered by taping with plastic tape. All outlet boxes, switch boxes, junction boxes, etc., were coated with the asphalt chromate emulsion paint or with Glyptol enamel. Since our Company is also in the paint business manufacturing a full range of industrial paints, we naturally used our own products wherever possible.

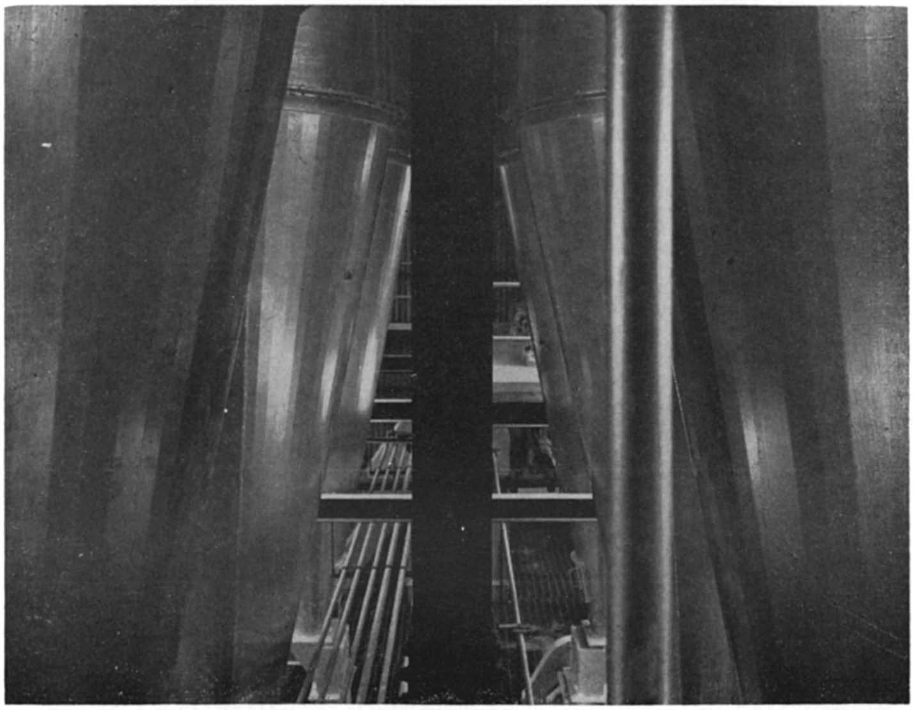
Safety is another factor on which a great deal of attention was placed at the design stage of these plants. This is the time at which safety features should be considered rather than after the plant is built.

In addition to the Engineering Department which had the job of preparing the detailed designs, C-I-L has a Safety and Fire Protection Section in our Industrial Relations Department and the detailed plans were carefully scrutinized by this group to ensure that such precautions as handrails around all openings as well as toeboards at floor level were specified; that all stairwells were provided with handrails, that doors opened outwards, that adequate clearances were provided, that elevator pits had chains across ladder openings, that adequate levels of illumination were provided, etc.

We believe that attention to such details is a pre-requisite to achieving a good safety record but of course by themselves do not assure a good record. The most important link is the employee and his attitude to safety.

Despite all these checks, however, one or two things did slip through. I keep a photo as a reminder of how not to install a double deck screen. It is rather difficult to adjust the screen cloth tension. We have since enlarged the opening around the screen and installed a stairway up alongside the screen to provide easy access.

Careful attention was given to escape routes to ensure that if a major accident such as an ammonia line failure occurred, no one would be trapped inside the building. With a cubic content of 150,000 cubic feet inside the building only 100 gallons of liquid ammonia are required to reach a concentration of 1% NH_3 in the air which is lethal level. With a centrallized push-button electrical panel in the operating control room, which is supplied with pressurized ventilation from outside the building, an operator can shut off all equipment in 10 seconds. An ammonia mask is available to him to put on if necessary to get to the nearest exit. Inside stairwells are fully enclosed to ensure that fumes would not readily penetrate the escape route. In the event of an emergency however, the operators are instructed to leave the dryer and cooler and Venturi fans running since their combined capacity will change the air in the granulation building in about three minutes. At one plant an outside fire escape is not enclosed except by a wire mesh enclosure to ensure nobody falls over the stair railing. However, at the other two plants when the plans were sent to the Ontario Department of Labour for approv-



This battery of cyclones collects dust from drier and cooler and returns it to the granulating system. C-I-L's granular fertilizer plant at Chatham, Ont.

al we were advised that these escape stairs would have to be enclosed because the building was over three storeys high and was an industrial plant and that is what the building code called for. We argued in vain that an open air stairway would be safer in the more probable emergency to ensure adequate fresh air but all in vain. Rather than jeopardize our future relations with the Department, we put on asbestos sheeting on the stairwell satisfied with the knowledge that if the necessary arose, a good kick of a panel would give a man an opening to breathe fresh air.

In the overall design of the plant we were aiming primarily for a quality product which could be produced with a lower labour content in a plant in which minimum maintenance costs would be experienced and from which no air pollution would result. How well we have done only time will tell. However, we have provided sufficient room in these plants that we think modifications can be incorporated very readily.

While technological advances may come along it still appears that people are going to continue to want to eat for some time to come and so long as we do, fertilizer will be needed to grow the crops.

I still recall the remark made to me on the morning we were about to start up the first plant. "Just remember," this experienced operator said, "You'll never know as much about granulation as you do right now." There are many times since when I have almost been ready to agree with him.

(Chairman Sauchelli resumed the Chair.)

CHAIRMAN SAUCHELLI: Thank you very much, Mr. Doidge. We know that a great deal of time and effort was put in that paper.

We'll have discussion right after lunch and hope that you'll have many questions to ask the speakers.

(The meeting adjourned at 12:05 o'clock, p.m.)

Wednesday Afternoon Session, November 4, 1959

The Round Table reconvened at one forty-five o'clock p.m., Mr. J. E. Reynolds, Jr. presiding.

CHAIRMAN REYNOLDS: We're most appreciative of the fine discussion and papers, slides, et cetera, that were given this morning; we'll try to proceed now with part two of that discussion.

We're indeed fortunate to have as the second part of this discussion a review of fertilizer manufacturing processing techniques in the Southeastern section of the United States. Particularly of interest is the approach to granulation in the State of Florida.

Most of us have definite reservations concerning the combining of granulation formulas with complicated multi-ingredient mixture formulas. Wilson and Toomer of Jacksonville, Florida is a company with many plants and believed that Florida was ready for granulation.

One of the persons largely responsible for establishing the process to meet the difficult requirements is Mr. Albert Henderson, Chief Chemist and Technologist for Wilson and Toomer. It is my privilege and pleasure to introduce Mr. Albert Henderson who will discuss mixed fertilizer processing and handling as related to Wilson and Toomer's Jacksonville operation.

MR. AL HENDERSON (Wilson and Toomer):

I feel sort of unnecessary after that fine report that we had this morning and I wonder if I can add anything. My presentation consists of two parts; the first is a brief description including a flow sheet primarily to quickly acquaint you with the process and for record purposes. The second part is a more detailed description of what we do and how we do it. This phase depends largely on you. I'll try to answer questions that you may wish to ask so please fire away.

We ask only that you place what I have to say in its proper context, mainly that our plant is a deviation from the usual and our specifications are much more rigid. I passed out samples of some grades that we produce and I think that after you look at the closely-sized products you'll understand what our problems are.

In the course of touring this installation, please interrupt at any time. I'll be assisted from time to time by Mr. Allen Jackson and Ricky Rifé; Ricky is from Cuba, Productora is his Company. I think he has some interesting information. See Figure 1.

The process begins at a ten foot by fifteen foot six compartment cluster hoppers and a ten ton superphosphate hopper. You'll notice the superphosphate hopper is not shown here. Solid raw materials are weighed from these hoppers by means of a 4,000 pound dial scale and an automatic beam scale; the automatic beam scale is used for superphosphate.

The weighed raw materials proceed via belt conveyor to a bucket elevator which delivers to Tyler Niagara screen fitted with three and one half to four mesh Ty-rod wire.

The screened raw materials are mixed in a two-ton Ransom mixer and discharged to a 24-inch belt conveyor which delivers to an automatic storage hopper.

This hopper is part of the continuous solid feed system and Allen Jackson will now describe the solid feed system.

MR. A. S. JACKSON (Fertilizer Equipment Sales): In this solid feed system the solids are accepted from the batch mixer in a hopper that holds seven to ten tons.

The materials are then weighed out batch-wise in a timed cycle; one minute intervals. If, say you wanted 1,000 pounds per minute rate of flow, the material is fed by

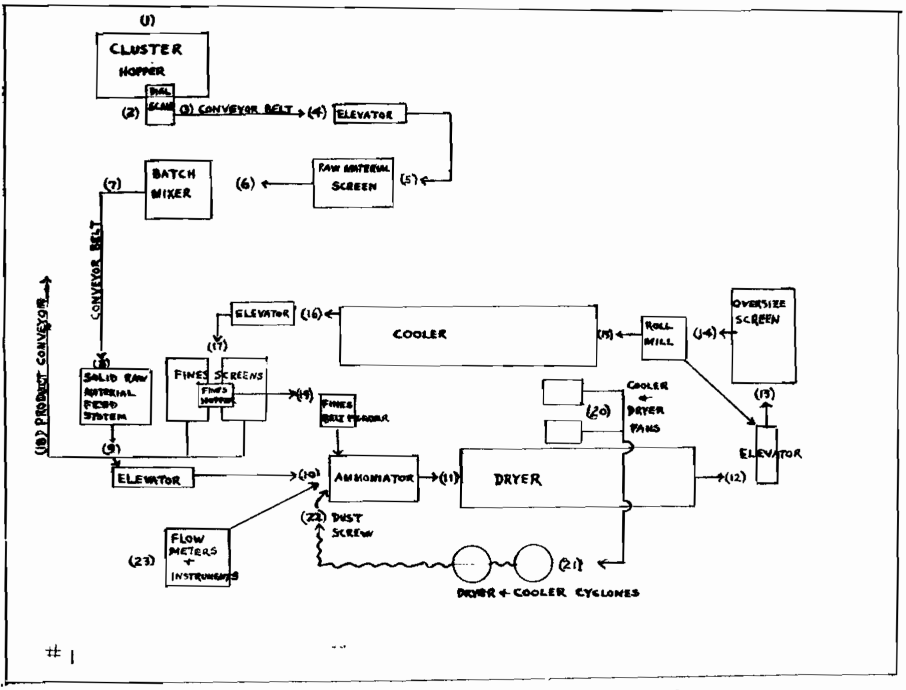


Figure 1. Flowsheet used to illustrate talk.

a belt feeder into a weight hopper, batch-wise, weighing out a thousand pounds.

At the end of the minute cycle the weight hopper gate opens, the material is dropped into a surge hopper. At this point then we've changed an irregular batch flow from the batch mixer to a weighed amount, correct in the amount, per minute.

The next feature is a surge hopper that smooths out the flow as it is dropped from the batch hopper so that it takes the 1,000 pound, batch and feeds it evenly on to the ammoniator.

The advantages of a system like this is that as far as the scales go there is nothing more complicated than a beam scale. The automatic controls consist of a timer, it starts the feeder on the surge hopper, when the required material is up to weight the scale hopper will shut the feeder off and the unit just remains stagnant until the end of the minute when the weighed batch is dropped to the surge hopper.

The bottom hopper is emptied by a belt feeder at a rate that will cause it to empty just a few seconds before the next batch would drop: There's nothing more complicated than a beam scale and a belt feeder. The unit, however, has some disadvantages; it takes a lot of head room. You can see by the time you get a belt feeder, a weight hopper and a surge hopper, you've taken up fifteen feet or so.

If the mill was not designed for this system it would probably require another elevator. This one, however, did not. A gravimetric feeder, a pure gravimetric, would possibly take maybe three or four feet of head room plus the surge hopper.

The advantage is an almost negligible amount of maintenance; it can be calibrated with dead weights, and it is relatively trouble free. The most trouble, I believe, that you have is the counter that is used to count the number of batches. This has been our biggest source of trouble. It's easily maintained with average mill maintenance without any special tools.

Mr. Henderson: We left off at position number nine on the flow sheet. Solid raw materials are delivered from a continuous feeder to TVA type continuous ammoniator by means of a bucket elevator. As we shall see later this elevator is used to deliver recycles simultaneously with the solid raw materials.

Liquid raw materials are metered by means of flowraters which are located in a central control room. All liquid materials except sulphuric acid and water are taken directly from tank cars which are located a few feet away and parallel to the mill. Sulphuric acid comes from a lead storage tank and anhydrous ammonia is cooled prior to delivery to the flowraters in order to prevent flashing which causes faulty flow rates.

This is accomplished by use of a forty foot pipe-in-pipe type heat exchanger and I'm sure most of you are familiar with that.

At this point we have both solid and liquid raw materials being discharged into the ammoniator. This machine has a total length to diameter ratio of 2.3 to 1 with an ammoniation section ratio of 1.6 to 1.

The discharge end or granulation section contains retaining rings which allow granules to overflow ahead of small particles and therefore offers greater area for fine materials to agglomerate.

The drum is turned by means of a chain drive consisting of RC-200 chain and matching sprockets. It is powered by a 30-horsepower motor with fluid drive and is equipped with reversing switches.

After ammoniation and varying degrees of granulation the material is discharged directly to a rotory drier. This machine has a length to diameter ratio of 6.25 to 1. It's turned by means of a chain drive consisting of RC-240 chain and matching sprockets. It's powered by a 50-horsepower motor with fluid drive.

Material is conveyed from the drier to a Tyler hummer screen by means of a bucket elevator. This screen is single decked and removes oversize only. The screen cloth consists of Tyler No. 9457 wire with a .113 width of opening and a .054 diameter wire. It's mounted at a slope of 36 degrees. Oversize material from the screen is discharged directly to a roll mill which Mr. Jackson will describe.

Mr. Jackson: I'll describe this particular one—the two rolls are of solid cast iron; they are chill surfaced to give a surface hardness of about 64 Rockwell C which is roughly that of a file. The rolls are 16 inches in diameter and four feet long.

Each roll weighs roughly a ton. The necessity of the hard surface rolls, is in doubt. We all believe that soft cast iron rolls would be just as adequate. Up to this point the rolls are no different from any other commercial type of horizontal rolls that you might see.

The similarity ends there in the use for fertilizer and say for rolling soy beans or some vegetable products. The rolls are held together by air cylinders. Now many commercial rolls for soy beans would be held by springs.

We picked air cylinders for one reason particularly; so we would have control on opening our rolls. The forces holding the rolls together are about 1,000 pounds per foot of roll length.

This is adequate to give plenty of breaking; it also allows the rolls to bounce back if any solids or hard obstruction should go through.

By having the air cylinder double acting, any fertilizer that falls in the crack between the rolls when they are not operating can be emptied. This is accomplished simply by opening the rolls 2 or 3 inches and then close them.

In the event that you should get a charge of wet oversize, which is the hardest thing for the roll to handle, the power may go up above what your motors will handle, the rolls can be opened by the operator while they're running to drop the charge on through and then closed and continue running. This part of the operation could be made automatic so that an excess load on the motor would throw a solenoid valve and open it.

The scrapers for the rolls are tool steel; they are one piece, full length and when they are new they are set up relatively close to the roll. I have a feeling that after the rolls are operated for a while the scrapers don't do much of anything. I don't believe they're really necessary.

The maintenance and operational problems are almost nil. We had some early problems in choking it down with wet material; after we bridged that gap, I don't believe Albert's had a whole lot of operating problems with it.

The cushioning of the rolls by the air cylinders is cushioned in two directions. Other roll mills with air cylinder cushions on one side had a fixed roll on the other side. When a good healthy solid lump would go through and kick a roll back, the building moved the other way, so by air loading both cylinders and having no fixed stop in any direction, that problem was eliminated.

Mr. Henderson: We had finished position fourteen on the flow sheet. All materials, fines plus product, which pass the oversize screen are discharged directly to a rotary cooler which is for all practical purposes an exact duplicate of the rotary drier.

A bucket elevator takes the material from the cooler to two single deck Tyler hummer screens mounted a 33 degree slope. The screen cloth consists of Tyler 9040 Ty-Rod stainless steel wire with an .070 width of opening and .041 wire diameter. The material remaining on the screen which is the product is chuted to a belt conveyor which is then taken to storage.

I gave wire sizes and numbers of our screens because I think this is, at least to me, one of the most confusing points that we're confronted with. I hear people talk about six mesh product or five and sixteen mesh product and then after you look at the product it doesn't stack up with laboratory screened samples.

Material which passes the screen is collected in hoppers and discharged from these hoppers by a continuous belt feeder. The feeder belt discharges into the raw material elevator by means of a chute. The belt feeder is manually operated to give desired recycle rates.

The air system consists of two large fans which are located directly on top of the drier and cooler. Air is pushed from this point overhead to two long cone cyclones which discharge dust into a screw conveyor. This conveyor delivers dust directly to the ammoniator or on to the product storage belt.

The reason for this is that when we're making pulverized mixtures we didn't want to put fines back through the ammoniator, accordingly we sent it directly to the product storage belt. No valves or gates are necessary at the cyclone discharge because the air system is a push through type. It is only necessary to adjust air outlet discharge by means of a cone top so that a slight blow back occurs at the cyclone dust discharge.

Since this adjustment is not very critical, adjustments are seldom made—as a matter of fact we probably don't adjust more than once every two or three months.

Drier air is heated by means of an air cooled refractoryless furnace which Mr. Jackson will describe now. Our fuel is propane and the furnace furnishes about 15 million BTU's.

Mr. Jackson: There's nothing particularly unconventional about this furnace; it's a simple air-cooled jacket with a fire box steel liner, without refractory. It was designed primarily to operate at less than 1,000 degree F discharge air temperatures.

If the discharge air temperatures are cool enough the refractoryless units are very satisfactory. When you start getting up much over 1,000 degrees the inner chamber begin to get quite red; some sag can be expected; you could expect a little bit of trouble from them. It is important in a refractoryless unit to use a burner that has a very, very small amount of flare. A lot of radiant heat will heat the jackets or the inner liner quite hot—hotter than it should be.

Outside of that, it's a perfectly conventional propane fired furnace.

Mr. Henderson:

I've got some, somewhat short, quick, to the point, and blunt statements to make.

I've done this to save time and later we can come back to the individual points and discuss them more fully. If you will please ask questions you'll help a lot. This meeting is supposed to be informal and spontaneous and that's the phase of the report that we're now trying to get across.

Actually this is a to-scale drawing of the plant, at least most items.—As you can see, there are two or three free hand items, but not many.

We pre-mix solid raw materials because we think it aids analysis, aids granulation and provides a uniform mix to the ammoniator. It really doesn't cost very much particularly if you already have a batch mixer. Most plants have this kind of equipment.

Solid Feed System

The solid feed system — Mr. Jackson has already described it. Now I'll make some comments about it. It's easy to repair; has excellent accuracy, plus or minus one per cent or actually better than that; can be checked continuously visually as well as by scale.

This is the solid feeder that we've been talking about. If you are on this side you can see that the weigh hopper is located on the ammoniator floor.

This is the control room which is roughly 25 feet from this point to this point and you can walk over from the control room to the ammoniator discharge where our operator can stand, and look into the weigh hopper; if the hopper dumps and all the material is not out you know you're in trouble.

You can look down into the lower continuous feed hopper. If the material has not all been discharged in 58 or 59 seconds something is wrong—If the material has discharged sooner than this, you are also in trouble.

We also have an over-under indicator here and it's mounted up in the air about in this position so that you can look out of the control room and see the over-under indicator any time you want to; simply look over here and see if it's coming up to zero, below or above zero.

The lower hopper, here, is also equipped with a check scale so that we can check the previous weight batch by stopping the system just after the scale hopper dumps and just as the continuous hopper runs empty.

We very seldom find this weigh system out when we're running at rates of 1,000 pounds per minute. We frequently get within two to three pounds of preset weight.

As Mr. Jackson has already mentioned, this system requires considerable height and sometimes an elevator. I think the chief disadvantage of this type system is that it produces an awful lot of dust. This depends on what type grade you're running. Of course you know some grades don't have dusty materials and you wouldn't have a problem, but, as you can see, materials falling through the air at three or four different places can produce considerable dust.

Now as to liquid feeders—we find that our flowraters reproduce themselves well provided you have only a few grades and they are calibrated for those particular rates. When you operate the way we do, many grades and various tonnages and over a wide range on the flow meters running anywhere from 20 to 80 per cent, then we have trouble and because of this we have been trying a magnetic meter for about a year now. The results have been excellent; I don't think there's any comparison between a flowrater and a magnetic flow meter and as soon as practical we hope to replace all of our flowraters with magnetic flow meters.

Some people seem to think that they cost a lot and of course the initial cost is fairly high, but when you consider the fact that the magnetic meter has a range equivalent to about three flowraters then it's not as costly as it might seem.

Ammoniator

The next step is the ammoniator. I think the factors to consider regarding the ammoniator are, slope and speed, ratio of length to diameter. If you use a pipe distributor use 1/16″ or 3/32″ holes in a spiral pattern for acid and solution. We haven't tried this for anhydrous.

A Voice: What do you mean by spiral?

Mr. Henderson: Holes in a pattern similar to red on a peppermint candy stick.

The reason for the spiral system is that it represents a minimum of hot spots because you distribute the heat around the pipe; you don't have localized hot spots and you have a minimum of puddling.

You do get a little buildup on this particular type of sparger, but it is not enough to hurt the operation. You don't have to stop and ammonia loss is not a problem.

It takes about five to seven minutes to change all of our spray pipes which consist of anhydrous, solution, and acid. We change them as frequently as possible, sometimes several times a day.

We also use varying lengths of spray pipe. Three arms support the spray pipes and our spray system is about eleven feet long.

The center arm of the spray pipe runs down to a "T" in this manner.

Now the discharge—the ratio of length here—the distant end as opposed to the inlet end is about 60/40.

You have a total of six pipes; two anhydrous and two solution and two acid. I mentioned earlier that we have a small water spray pipe in the discharge end. We use a separate flow meter for it. eW can add water separately at the discharge end of the ammoniator.

The flow meter for this purpose is mounted on the ammoniator frame right here and the operator can stand here with his hand on the valve and see what he's doing. Ricky Rifé has some excellent data on a situation similar to this; actually he's had more experience with it than we've had. Ricky, would you give us that information?

Mr. Enrique Rifé (Productora):

Our ammoniator is 11.5 ft. long by 6 ft. diameter. The granulating section is 2½ ft. long. It is limited by two retaining rings: one 18″ high at the end of the ammoniating section; and the other, 9″ high, at the end of the granulating section. A third ring, 13″ high, divides the granulating section into two equal parts.

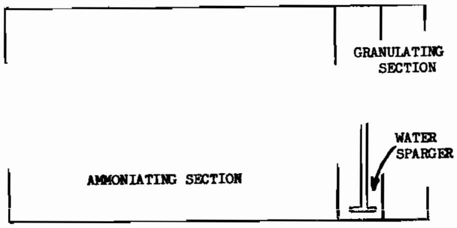


Figure 2.

With the addition of 30 #/ton of water in the granulating section, the temperature of the material decreases 6° F., which agrees with the heat balance, considering no water evaporation, at least at the usual temperature of 200° F. of the material coming out of the ammoniating section.

In order to determine the rate of response of plasticity with a change in the amount of water added, several tests were made increasing the quantity of water and making screen and water content analysis of samples from the ammoniator output at definite periods of time.

A few months ago we were adding the water in the ammoniating section through the solution lines, but we found that the rate of response of the plasticity of the material in the ammoniator was very wrong. I mean, it takes too much time to change the condition inside the ammoniator.

We tried water only in the granulating section. We tried water with a sparger spraying over material, but we found that those spargers didn't work right. So we changed and now we're adding the water through a pipe eight inches long with one-eighth spiral holes under the material bed in the granulator section. Our ammoniator is eleven feet long by six feet diameter; the granulating section is two and a half feet long. It is limited by two retaining rings, one 18 inches in diameter at the end of the ammoniating section and the other 9 inches at the end of the granulating section. (Fig. 2)

A third ring, 13 inches in height, divides the granulating section into two equal parts. Easier operation is obtained by adding the water in the first half of the granulating section. Usually our grades require a small amount of water, 50 pounds per ton or less. All the water is added at the granulating section.

Comparing our system with the usual addition of the water in the ammoniating section, the advantages obtained are: The ammoniating section runs with less plasticity and a better distribution of the liquid phase is obtained, avoiding lumps and fumes.

Less power is needed to run the ammoniator and the plasticity of the mass in the granulating section is easier to control; the mass that must be adjusted to proper plasticity at each moment is extremely small compared with the whole mass in the ammoniator.

In order to determine the rate of response of plasticity to the change in the amount of water added several attempts were made increasing the quantity of water and making an analysis of screen sizes and water content of samples from the ammoniator output at definite periods of time.

The resuls are shown in Fig. 3. The screen analysis of the material has been arbitrary divided in two ranges, plus and minus mesh 6.

The increase of water added was 10#/ton, and after 2 minutes the water content was the theoritical expected, showing that the system was stabilized and no evaporation took place.

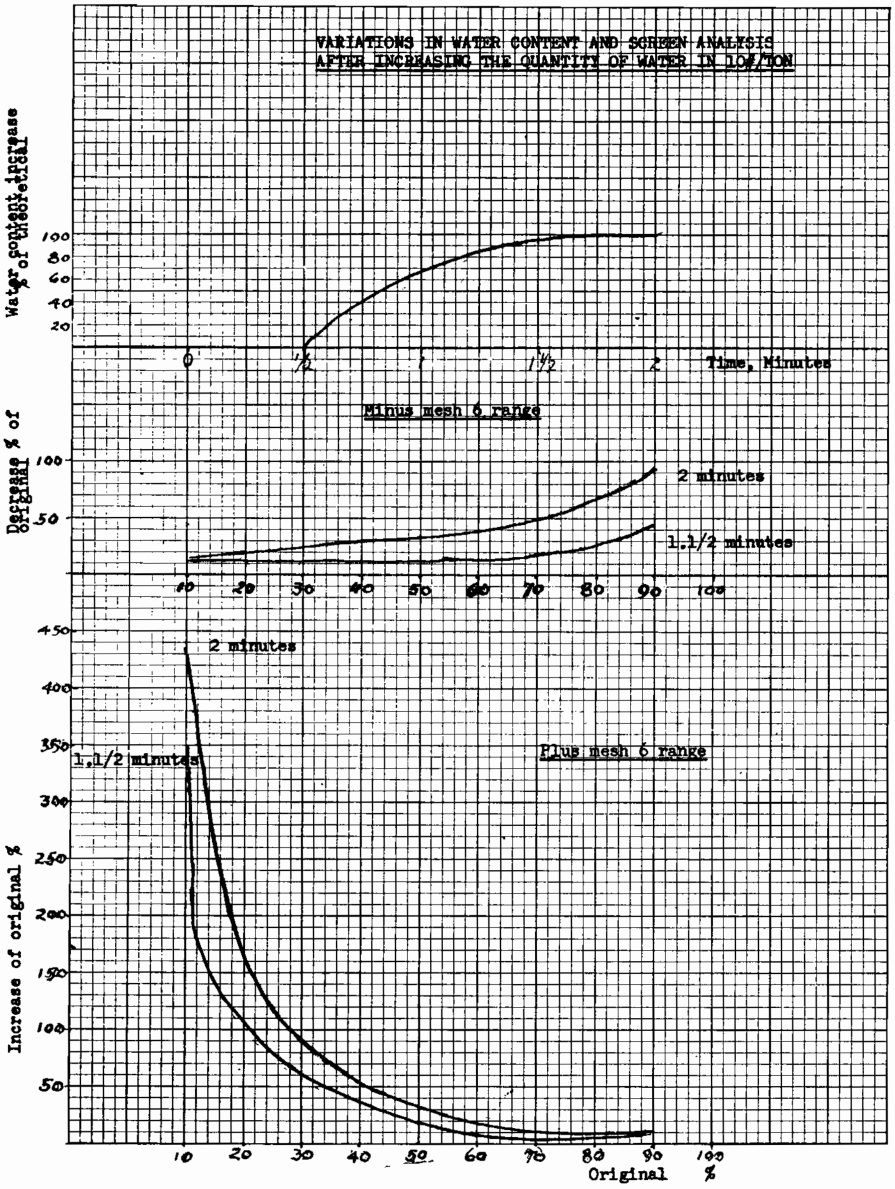


Figure 3.

In the Figure, the decrease in the quantity of material in the minus 6 range is plotted against the original percentage in this range and the increase in the quantity in the plus 6 range is plotted against the original percentage in this second range.

A direct correlation is found in the minus mesh 6 range, and an inverse correlation in the plus mesh 6 range, with the decrease or increase in reference to the original percentage.

Mr. Henderson: Thank you, Ricky.

The question was a comparative one. Allen Jackson wanted to know the difference in time before and after he made this addition. About five minutes when he added the water in the back end of the ammoniator and after he made this change it took about two minutes to reach equilibrium.

In our somewhat longer and larger ammoniator the difference is much greater than that. About a minute and a half to two minutes to make a change compared with eight to ten minutes. Of course the tonnage determines your retention time in the ammoniator and that has a lot to do with it.

Before we get away from the ammoniator, I think that we all know that granulation is a system of stabilized equilibrium and that anything we can do to maintain equililbrium without making big changes is a definite help to us. I think if we would constantly remember this we'd probably stay out of trouble a great deal more than we do. Of course, stabilized equilibrium is an unscientific term just as granulation, as practiced, is an unscientific process.

Our greatest efforts are concentrated toward operating the plant at the ammoniator. Recognition of product condition at this point is the key to good operation.

We also spread our spray pipes apart as much as possible.

The reason for spreading the pipes as far as possible is, I think, fairly obvious. You want to get solution and anhydrous away from your distribution system. When you do that you don't tend to puddle. The closer you stick those pipes together and the smaller area you use to add liquid the more puddling you're going to get and the more oversize you're going to make.

To me the thing to do with any ammoniator is to stop making oversize. We make enough of that in the drier.

Driers

Driers; many people are trying to do away with driers and we'd like to have one about twice the size of the one that we've got, then, we think, we could run some real tonnage.

We vary our tonnages upward of 25 tons per hour. Production of a 1-1-1 ratio at 40 tons per hour is not difficult when product quality is based on the so-so standard 6-16 product frequently referred to in the literature. In Florida production of granular produces added problems because of possible contamination from so many different materials (40 or more) used in complicated multiple mixtures.

While we're manufacturing we tolerate no fines. If fines happen to get on the product belt then we would stop the plant and run the fines off or do something else with them. When we first started granulating we had problems with fines.

As you can see from the drawing up here we have two fines hoppers and two fines feeders. We have what I call a coarse adjustment and a fine adjustment. Now your fines hopper located directely below your two fines screens is where we make our major or coarse adjustment. At the top of the fines hopper we have an opening so that when the fines hopper fills up material will run out into a chute and down to another fines hopper and this is fed by means of a belt feeder back to the same elevator, with raw materials discharged to the ammoniator.

When we initially made this change I think we ran for several weeks before we used the second fines hopper, so I don't know whether it was psychological or what; we had trouble with fines, we put in a situation to correct it and we didn't have trouble anymore. I dare say that 95 per cent of the time no material gets in the second fines hopper.

So if for no other reason than psychological factor it's a good idea to have a second system.

I also feel that a drier is the chief factor for upsetting the equilibrium that we were talking about. I know if you're running a limited number of grades you know what you're doing all the time because you run those grades every day and you know what the conditions are.

But where you're running many grades, non-nitrogen grades, X-o-X grades, low nitrogen grades, adding secondary elements such as iron and magnesium, manganese and so on, at times, some strange things happen.

I've been told that magnesium is not a problam with some granular fertilizers. It certainly is with others because sometimes the product goes to pieces in the dryer.. SPM as a source of MgO helps reduce this factor.

I've heard a lot of discussion about ammoniators and pug mills and the type of reaction vessels that people use and I used to fuss about that a lot myself; of course, I'm an ammoniator man, but really when you look at it, the limiting factor on a granular mill is not an ammoniator, it's not a pug mill, it's not a reaction vessel, it's a drier.

We can run material so wet it comes out of the ammoniator as mud so we don't have a limitation on how wet we can run material in an ammoniator; our limitation is what we can put through a rotary drier and what we can screen and crush.

Screening

The next subject is screening and that's the one that has received a lot of attention. You can see we have three, four by fifteen, hummers, which is a lot of screen. Two fines and one for oversize. We have sufficient screen area for fines; we need a little bit more for oversize.

A hummer is an excellent screen for fines. I think possibly a mechanical screen would be better for oversize—now I'm talking about removing oversize at the drier discharge and there's an awful lot of difference. When you dump a lot of romantic material on an oversize screen—and I call it "romantic" because if you get wet, hot, sticky material on a hummer it hugs that screen like a baby monkey hugs mama and that's pretty tight.

It takes the hum out of the hummer and it takes the bounce out of a mechanical screen, but I think it takes longer to take the bounce out of the mechanical screen than it does the hum out of the hummer, if that makes sense.

As we get to the roll mill that Allen Jackson has already described and for some reason or other our roll mill, like some football teams, hasn't read all the dirty remarks about it because it works real well. We clean it about once every two months, just on general purposes, because we like to do something to it.

We had a large cage mill in series with the roll mill just in case, at high production rates the cage mill would operate perhaps 30 minutes. The roll mill almost indefinitely.

This roll mill will not handle romantic materials nor will any other piece of crushing machinery. When we have trouble with wet material being discharged to the screen a simple correction can be made.

We struggled for about 12 months and we stopped and cleaned screens and had all kinds of trouble. We finally put a little chute down at our drier elevator discharge in this position.

When the romantic material first hits the screen then you bypass the screen for perhaps one minute, maybe two, seldom longer than that, and run this material on the floor. Some time later, maybe 30 minutes or an hour, at various intervals we come back and shovel the material, after it's had a chance to cool, back into the system.

This saves an awful lot of trouble (one little simple chute)

Screens—When you change an oversize screen from five mesh to six mesh you've made a considerable change. When you change it from six mesh to seven mesh you're in an altogether different world. If you will think about it—and I'm sure—of course you can talk to anybody that screens at the dryer discharge as soon as you start screening you get a build-up on your screen. Now if you start with a six mesh and in 30 minutes you've got a seven mesh, after this for some reason or other it doesn't continue to build up. So after the initial build-up in perhaps 30 minutes and after you reduce the size of the screen from a six to a seven or maybe an eight, you don't have more build-up and you can run the same screen for several weeks befor you have to clean it, that is, providing there are no accidents.

We find that by changing our fines screens from ten to twelve mesh or from ten to fourteen mesh makes some difference in the operating characteristics, but not much; no where near as much as changing the over-size screen. If you're wondering what kind of recycle rates we have, we start off at perhaps 75 per cent and go to 300 per cent. So we have a tremendous amount of recycle and that's part of the reason why we need more drying capacity. If you look at this product [shows sample] you'll notice a small size; it's not easy to make and it certainly does present some problems.

One advantage that this system has, I believe, over other systems is that by screening at the drier discharge and crushing at the drier discharge you produce a product which will stand up in storage with somewhere between one and two per cent more moisture than a product from a system where screening is done, at the cooler discharge.

Another thing that you can do with this type system is that by adjusting your roll mill you can make a spherical product. If you adjust the rolls very close together you don't have any crushed oversize in your final product and you'll get a spherical product. A closely-sized spherical product apparently is a superior type product as far as standing up in storage is concerned. It's proven that way with us.

Let me add one thing. We try to hit a happy medium here; we don't make all fines out of our oversize and we do get some irregular shaped particles in our product, as you probably noticed. I'm just saying that we can, by closing down on the roll mills, make a spherical product and we've done this, but then our recycle is not 75 per cent to 300 per cent; it may be double that figure and our tonnage begins to come down.

Cooling

About cooling, we feel about the same way about cooling that we do about drying. We would like to have a bigger cooler. In pulverized grades we have no trouble at all cooling down within five to ten degrees of ambience; on granular grades apparently the determining factor here is the moisture content of the final product and even at 25,000 to 30,000 CFM on a product which has less than one per cent moisture we just don't get the temperature down. It's probably 135 to 130 degrees F. Of course, extremely cold weather helps but we don't have cold weather in Florida.

If the moisture content of our product is say three per cent we can get down to 110 to 115 degrees. If it's higher than this we can bring the product temperature down within 5 to 10 degrees of ambience. It was interesting this morning to hear that somebody has an efficient cooler. I understand that the Canadian folks bring their product temperature to almost ambient; that's apparently an excellent job. We haven't found this to be true. Just the same we wouldn't trade weather men with them.

Our biggest mistake initially with the mill was in our air system. Our air stystem gave us holy hell for 12 months and I mean holy hell. We started off with our fans about half way between the drier and cooler and the cyclones about in this position.

The fans were just in front of the control room. Of course we had duct work from this position to the fan and from the fan to the cyclone. The duct was 38″ and the fans 52″ impellors. We were getting somewhere in the neighborhood of 25,000 to 30,000 CFM and I don't know how many times we cleaned the duct system but it was many, many times.

One of the weaknesses of a push through air system is build-up on fan blades and that was our greatest single problem. We have made some changes in the air system; we've gone to a smaller duct; we've moved the fans back directly on top of the drier and cooler and changed to new type fans. The fan system is no longer a problem.

We apparently had very little dust loss from our cyclones, at least it's not visible and I'd say almost none.

We're real satisfied with the air handling system now and our push system does have some advantages; the location—it requires less duct work and the fans are located down close to the floor where they can be watched and if you have to work on them they are readily accessible. We do have to clean them now, but seldom. We clean at planned times while we're down. Before this, sometimes we would stop, in a 20-hour period, perhaps three or four times for cleaning purposes—so we had some real problems.

We initially started this plant with our oversize screen at 33 degrees and believe me, this doesn't work. Not only romantic materials were troublesome but normal materials as well.

Switching to 36 degrees made a tremendous improvement in the operations of the screen.

Of course I mentioned the fact that we've had our share of problems with flow meters and I think switching to the magnetic meter has helped this considerably.

One other thing. I talked a little bit about this before and I don't have specific figures because this was done under operating conditions, but I'd say that we can tolerate more moisture than perhaps with a conventional type mill. We sometimes allow certain grades to contain several percent more moisture than many other producers. Though considerable tonnages have been produced on this basis I haven't seen a single complaint.

Any questions?

A VOICE: You changed the duct size. What did you change to?

MR. HENDERSON: From 38 to 30.

We'll have the questions a little later, if you don't mind.

CHAIRMAN REYNOLDS: Thank you, Al, Allen and Ricky. I think that was a real fine presentation.

I know we all have a lot of questions so I think we will move on to this question and answer session. For that Mr. Al Spillman will take over and moderate the CIL and also the Wilson and Toomer discussion.

(Mr. Spillman assumed the Chair.)

CHAIRMAN SPILLMAN: We have had some excellent papers on organization and control by Les Clegg and on all types of processing by Elgin Doidge, Al Henderson, Al Jackson and Ricky Rifé.

At this time we'll open our discussion to questions from the floor and we'd appreciate those of you asking questions to give us your name, whom you represent and direct your question to any of the speakers that have been here this morning.

Now we'll open it for questions. We have two microphones, one in the front of the room and one in the back of the room.

VOICE: I'd like to ask the first question to Mr. Doidge.

Do you have your figures or approximate figures on costs for manufacturing granular versus conventional goods; the additional cost of manufacturing granular versus conventional goods, taking into consideration the fixed assets, depreciation, additional maintenance and what not. Approximately. Operating costs.

MR. DOIDGE: I would say, speaking roughly, it would be about a dollar and a half per ton, probably.

CHAIRMAN SPILLMAN: About a dollar and a half per ton.

MR. DOIDGE: It all depends. The difficulty in the question is it all depends on what you take for depreciation and how you take it and so forth.

CHAIRMAN SPILLMAN: Ten per cent depreciation on equipment and five per cent on building.

MR. DOIDGE: Well, I mean I don't know what we take.

CHAIRMAN SPILLMAN: Oh, I see.

CHAIRMAN SPILLMAN: Approximately a dollar and a half.

MR. DOIDGE: About that.

CHAIRMAN SPILLMAN: We're open for questions.

A VOICE: We can't hear you in the back of the room.

CHAIRMAN SPILLMAN: Okay. Mr. Doidge said it costs approximately—he hasn't got the exact figures—it costs approximately $1.50 a ton more to manufacture granular fertilizers than conventional fertilizers. That additional cost takes into consideration fixed assets, depreciation, additional labor costs, maintenance and all the other factors.

A VOICE: This last speaker was talking about moving the fans back on dust collectors—

CHAIRMAN SPILLMAN: You're addressing your question to Al Henderson?

VOICE: Yes, that's right.

SAME VOICE: Well, what do you do? Move the fan back to the source of collecting the dust?

MR. HENDERSON: No, He asked if we moved the fan back—I'm not quite sure I understand.

SAME VOICE: Maybe I misunderstood you, but I thought you moved the fan back to the source of dust.

MR. HENDERSON: Actually our fans are now mounted directly on top of our drier and our cooler whereas previously they were mounted about half way between our drier or cooler and the cyclones.

SAME VOICE: Well, do they still blow into your dust collector?

MR. HENDERSON: We still use a push-through system. We still have our fans before the cyclone and not after the cyclone. We haven't changed this system; we've only changed the size of the duct and fans.

CHAIRMAN SPILLMAN: We've had some very interesting topics and I'm sure a good many of you have some questions in mind. We'd appreciate having them.

A VOICE: I want to ask Al, have you some idea of your loss on granular fill?

CHAIRMAN SPILLMAN: As far as we can find out from the records that we keep (and we keep pretty accurate records,) our losses show anywhere from two to five per cent, depending on the grade that we make.

SAME VOICE: It varies with the grade?

CHAIRMAN SPILLMAN: Yes, it does. Actually, I don't think we have losses. We have paper losses.

What I'm driving at, I think we have a legitimate shrink that we could figure on at around two per cent, but I think some of the excessive shrink is taking place in our calculations.

One of the reasons for having the subject this afternoon on the mechanics of formulation is there may be some factors in the mechanics of formulation; but, answering your questions, we have a shrink anywhere from two to five per cent on different types of grades.

SAME VOICE: The figures given this morning now—if I read from those figures right,—the Canadian Company had the loss of — which went up 500 per cent. In other words, they went from a three-tenths loss to one and one-half per cent. Is that right?

CHAIRMAN SPILLMAN: Mr. Doidge, would you mind?

MR. CLEGG: What I was trying to point out in this is that most production people, and we do anyhow, have trouble explaining these shortages to accountants and sometimes to management and this table that I have drawn up indicated, to the best of our ability, the amount of material in the plant and the amount of materials out and the difference we called shrinkage.

There may be some inaccuracies in measuring the materials in inventory and so forth, but the interesting thing about this was, as you said it, it had gone up.

We think that some of this is perhaps physical loss but as Al Spillman says, it might be the other type and I'm sorry, but I can't tell you which it is, but the only difference between the last year and the three previous years was that we were into granulation.

We must remember, as Mr. Doidge pointed out, that we started three plants up at the same time;

we had the coldest winter in 40 years, so the natives told us, and the river ran dry and so forth, so we hope we'll better that next year.

Sorry I can't be more accurate in answering your question.

CHAIRMAN SPILLMAN: Do we have any other questions?

MR. ROGER SMITH (Eastern States): Two questions for Al Henderson.

If I understood him correctly, he mentioned that with their flow-rate feeder they let it run dry for a second batch between the batch hopper and the pre-mixer. I assume this means that the surge hopper up above the weigh feeder is empty at that time.

The question is, is that accurate?

MR. JACKSON: Roger, the surge hopper on top of the batch weighing device never runs empty. It stays somewhere between three and seven tons all the time. The hopper that Albert is speaking of is the bottom hopper that is used to smooth out the surges underneath the weigh hopper. By letting it run empty one or two seconds, you never end up with that hopper gradually increasing its quantity during the day. It stays empty. In the event a lump or something should fall down and bridge the gate momentarily you have a margin for catching up in case it has choked momentarily, which is not very likely.

The elevator, the conveyance of material from this bottom surge hopper to the ammoniator, up an elevator, the elevator discharging its final dribble slowly, you can not see that interruption at all by the time you get to the ammoniator.

MR. SMITH: Thank you. One further question for either of you.

With your roll mill, do you have any figures on the percentage of fines which are produced with the roll mill. In other words, the material passing the roll mill, what percentage would pass through your fine screen?

MR. HENDERSON: Roger, that depends entirely on how we adjust our roll mill.

If we spread your rolls apart, say, just a little bit more than the product size would be, then you're going to fracture a great deal of oversize and convert some of it to product.

Now I don't have any actual percentages because that sort of thing would be very difficult to measure, but it's considerable.

Roger, Ricky Rifé had some information. It varied from a maximum of 27 to a minimum of 10. That's a difficult thing to measure and just by watching our operation I would say that you could go up on the maximum and come down on the minimum. I know that you can break practically all of your oversize; you can convert it to dust if you wish.

MR. WILLIAM HENDERSON (Indiana Farm Fureau): What would be the speed of that roll mill?

MR. HENDERSON: Allen says 200, and actually both rolls run at the same speed and it's 198.

CHAIRMAN SPILLMAN: Al, what is your average moisture on your finished product?

MR. HENDERSON: That's a $64 question. We look at the grade. On a 10-10-10 we say we have a maximum of one and a half per cent. We like to see a 10-10-10 run from one to one and a half per cent. A 4-12-12 we prefer to have about a three and a half per cent moisture content, but we will allow it to go several percent above this at times. I think it's entirely unnecessary on all anhydrous 4-12-12 to go below two and a half per cent.

CHAIRMAN SPILLMAN: In other words you get by on those mixtures with a higher moisture content than we fellows are doing, trying to get it down below two per cent.

MR. HENDERSON: That's right.

Now, Al, to further answer that question, our 15-0-15 runs around .75; that's an average figure. Anything from 0.5 to 1.0% is OK. A non-nitrogen grade (we have limited experience) may be all right at 3 or 4%.

Now one problem that I should have pointed out while I was on the stand is that when you make a non-nitrogen grade you—of course it depends on the formulation,—we use hot water and sometimes we make a little superphosphate in the ammoniator.

You come out of the ammoniator at anywhere from 120 to 165 or so degrees F, depending on the type of formulation. When you get into the drier you actually increase the temperature of the product and in many cases you do most of your granulation in the drier This is the sort of thing that we would like to avoid, having two machines doing our granulating for us, because it tends to cut down your equilibrium conditions and keep you out of phase.

CHAIRMAN SPILLMAN: Thank you.

We've had some real subjects greet us here and I'm sure a good many of you have questions. This is the place to get them answered.

VOICE: I'd like to ask a question of Al Henderson, about ammonia losses.

Do you find any ammonia losses in your system?

MR. HENDERSON: In 15-0-15? Is that the grade?

VOICE: Yes.

MR. HENDERSON: Absolutely none.

We don't have any ammonia losses on any grade at any tonnage at any time. Now I know that's an odd statement to make here in front of so many people, but I swear it's the truth.

VOICE: Albert, what is the temperature out of the drier?

MR. HENDERSON: The temperature of the 15-0-15 out of the drier?

QUESTION: I mean the maximum temperature at the drier—the exit?

MR. HENDERSON: The drier exit temperature on a 15-0-15?

VOICE: Yes.

MR. HENDERSON: About 190 degrees F.

VOICE: That is the maximum you use?

MR. HENDERSON: That's the maximum drier outlet air temperature.

VOICE: Is it the product temperature or the air temperature?

MR. HENDERSON: Air temperature. I haven't actually checked the product temperature and 15-0-15 is one of those grades that we run dry at the ammoniator and allow it to granulate in the drier.

VOICE: Thank you.

MR. J. E. REYNOLDS: I have a question for either Allen or Al.

Your discussion concerning retention time in the drier. I think it was mentioned this morning it was somewhere around 20 to 28 minutes. You also mentioned the need for a large drier. Do you have figures on retention time in your present drier?

I have another question to go along with this.

It was mentioned earlier that the furnace was a typical air cooled system. I think there is more to it than that. I'd like for either of the Als here to elaborate on that as far as the type of burner, the offset features they have and relation to fume formation.

MR. JACKSON: Joe, in answer to your first question, the retention time in the drier is about 15 minutes. On the combustion chamber the burners are the simple, high pressure, inspirator type of burners as manufactured by many of the burner people.

They operate up to 20, 25 pounds per square inch pressure of propane; there are seven burners, each capable of a little better than two million btu's per hour.

The unit is arranged so that radiant heat does not project into the drier at any time. There are two 90 degree turns between the combustion chamber and the drier by the duct work.

The flames from the burners probably extend down the combustion chamber under high gas pressures maybe three feet or two feet; probably two feet, and the low gas pressures with a little more flares, maybe three feet.

In other combustion chambers that have been made, that we have made with bigger burners that have a longer flare, we have run into trouble on the radiant heat of the flame heating the inner liner of the refractoryless combustion chamber a little too much or having some structural defamation of the same.

It requires a very small flame to make one work.

MR. HENDERSON: Joe, I'd like to add a little bit to that.

We had trouble initially with our furnace because—well, not initially, but after we'd run several months, because we had an awful lot of trouble with our air system. When you almost stop an air duct up—you simply can't get 25 to 30,000 cfm thru a grapefruit sized hole. We just simply didn't have the air and that's the reason our furnace would overheat at times. The reason then that we could go to a higher initial inlet drier temperature is because we corrected the air system and we are now getting our 30,000 CFM.

I agree with what Allen said here; another thing is I think that we're going to add about three inches of a plastic refractory material so that we can go on up to 20-odd million BTU's, but when we make that move we're also going to increase our quantity of air. This is real significant, we do not believe in high drier inlet temperatures. That's where you have trouble with equilibrium and when you have a multiple system like ours where you make a lot of different grades you've got to maintain control. High drier temperature is one item which certainly hurts control.

So when we go to a higher BTU output we're also going to more air.

MR. G. C. MARBURGER (Spencer Chemical): Al Henderson, there are many of us more concerned about the capacity of an ammoniator. You slipped through this pretty fast. Specifically, do you have any indication that there would be an upper tonnage capacity in your ammoniator?

MR. HENDERSON: The highest tonnage that we have tried is 50 tons. This was what I call the 6-16 product and the ammoniator handled that very easily, but if you want to talk about ammonia capacity in an ammoniator, let's talk about something that has some significance like a 6-12-12 with all six units from anhydrous at 40 tons an hour. This is a pulverized grade and ammoniator handles 6000 pounds of ammonia per hour.

We just simply do not understand this problem about nitrogen loss. I think that the reason we've been able to avoid it is because our ammoniation section is longer than most. We have an awful lot of small holes in our spray pipe. One spray pipe will contain approximately 120 small holes.

If you locate spray pipes in the proper position — and that's somewhere between seven and eight o'clock and if you get it the right distance from the shell and if you have the proper slope and you turn the ammoniator at the proper number of RPM's, then you just simply will not have nitrogen loss.

As far as slope and speed is concerned we followed the TVA data. As a matter of fact, we followed an awful lot of TVA data and contrary to a lot of popular belief, we've gotten a lot of use from this and other TVA data. Blowing air on the bed, I believe, is an effective means of granulation and we've tried it some.

We don't use it on a routine basis now, but we plan to look into it.

Also, I initially wondered why in the world TVA used a granulator after the ammoniator. I understand that now. I think that we'll all come to a granulator someday when we try to improve our product quality. We think that a granulator would help us considerably in our operation. We believe that we should do our ammoniation in our ammoniator and granulation in a granulator. It would be a machine built to granulate—nothing else.

When you take one machine and try to make it do three or four jobs you certainly have limitations.

CHAIRMAN SPILLMAN: Al, what was the ammoniation rate in this 6-12-12?

MR. HENDERSON: Our ammoniation rates are based on six pounds for single super and four pounds for triple super and we have gone to 4.7 for triple and 7.2 for single with no detectable loss. We don't do it on a routine basis because we just haven't gotten around to this. Some day when we have enough time we're going to investigate this a little more closely.

When you're talking about going from a six pound ammoniation rate to a seven pound ammoniation rate, and a lot of times it's really not significant, financially.

A VOICE: When you're talking about 6-12-12 are you talking about formulating that with 120 pounds of anhydrous ammonia and no losses?

CHAIRMAN SPILLMAN: At a six pound ammoniation rate he said.

SAME VOICE: He talked to me. It was up from 4.2 or 4.7

MR. HENDERSON: We have. It

it 7.2 ammoniation rate. We have tried some grades at a seven pound ammoniation rate. We didn't do it on purpose, we had to do it. We expected to lose some nitrogen, but didn't.

A VOICE: I think the question is: do you over formulate?

MR. HENDERSON: Let's put it this way. In all of our formulations and calculations we use six pounds for single super as a rate and four pounds for triple super and if we have any additional ammonia we use sulfuric acid, 60 Baume' acid, at about 3.7 pounds per pound of ammonia.

Does that clear it for you?

SAME VOICE: May I state it more simply. Do you formulate and count a certain amount of losses and allow for no losses after that?

MR. HENDERSON: No, we don't over-formulate. Sorry I misunderstood your question.

CHAIRMAN SPILLMAN: We have time for a few more questions before we go into the next subject.

MR. KENNEDY (National Engineering): For Mr. Henderson. When the duct size was reduced from 38 inches to 30 inches were the original fans able to carry the same volume by merely speeding them up?

MR. HENDERSON: When we changed our duct size we also changed our fans. It so happens that the new fan, although of much improved design for our purposes, has about the same capacity—has about the same area and uses roughly the same horsepower.

Does that answer the question?

MR. KENNEDY: Yes.

MR. JOHN M. DANIEL (Virginia-Carolina Chemical): I have two questions for either one of the gentlemen from Canada.

One is: How many men a shift do they use to run their granular operation? What material are the bags made of in the dust collector?

MR DOIDGE: In the granulation plant we have a shift foreman who supervises both the granulation operation and also the dry batch mixing plant which is located some distance away.

We then have usually three operators or three men in the plant; one ammoniator operator who is in front of the ammoniator who also controls all the process, the liquid flows to the system; we have another man on the floor below at the granulator and then we have what we call a patrolman who is going about the plant watching conveyor belts and that type of thing.

Usually in the dry mixing operation there are two men; one on a tractor feeding raw matrials to the hopper system; another batch weigh operator.

In the bag filters we are using both orlon and dacron bags at the present time.

MR. HENDERSON: That spray pipe—that top figure I have there with the vertical pipe and then a "T" and then the horizontal pipe —this is the way the spray pipe looks.

Now that's just a "T" and of course a pipe coming down to the "T" and then you have two pipes, one that screws in the "U" end of your "T" and that acts a distributor.

The position in the bed would be indicated by those three dots. I've tried to draw a rounded surface there that represents the ammoniator shell, but the last pipe would be the acid pipe and then the solution and then the anyhdrous.

Those pipes are about eight inches apart except—I mean, from the solution to the acid is about eight inches. Eight inches on this basis.

A VOICE: Do you have perforations in that horizontal pipe?

MR. HENDERSON: The holes as I said earlier (drawing on blackboard) the holes in these pipes are in a spiral pattern and they are one-sixteenth and three-thirty seconds size holes and it takes about 120 of them to make a complete pipe; that's the two pipes together.

A VOICE: How far apart does that put the holes?

MR. HENDERSON: Well we've changed from one inch to one and a quarter inch pipes. It's somewhere in the neighborhood of an inch. We have simple holes and just a steel plate and you just stick this pipe in, like this, and push it back up against the "T" and just screw it in.

MR. JOHN B. STURGESS (Collier Carbon & Chemical): Mr. Henderson, when you speak of tonnage at 40 tons per hour in the case of 6-12-12, do you mean net production or does that include the run?

MR. HENDERSON: No, sir, When I talk about tonnage I'm talking about net production, not recycle.

(Laughter.)

MR. REYNOLDS: I have a question concerning how long it takes to clean out a system to change grades?

MR. HENDERSON: I'm afraid we're not very proficient on this. It takes us 30 minutes just to run our system clear. If it happens to be a somewhat similar grade that follows, and we can get our cluster hoppers ready, then we can follow up fairly fast, within ten or fifteen minutes, but it takes us at least 30 minutes to run our system clear.

Incidentally, when we run it clear—say we've been running 4-12-12 for 20 hours or so, we run the system clear and we'll wind up with anywhere from five to eight tons of fine and this is our total loss as far as fines are concerned.

A VOICE: What do you do with your fines?

MR. HENDERSON: We reformulate them or in case of 4-12-12 we just put them in pulverized 4-12-12 which we also stock.

A VOICE: Did you say you choke that elevator down and put this thing through your ammoniator, about how much water are you using there? How many horses have you got pulling that ammonia in?

MR. HENDERSON: We had a great deal of trouble initially with overpowering the ammoniator until we found out what our trouble was. Right now our ammoniator looks like a porcupine. It has 21 hammers that weigh anywhere from 15 to 35 pounds and we're mistreating the shell — a better system should be used.

I thought initially that the reason for horsepower problems was because of our scraper and that certainly does have something to do with it. There are, however, other factors which have as much or greater effects on H.P.

Really our problem was that we were blocking the discharge; the retaining ring where the material discharges into the granulation section. We were blocking that with our support arm for our spray pipe and that will raise cain with your horsepower. Now we don't seem to have any horsepower problems, although it did take us probably eight, nine months to finally figure out what in the world our troubles were.

CHAIRMAN SPILLMAN: Time is going on and we want to get on with the next subject and we wish to thank Mr. Clegg, Mr. Doidge, Mr. Henderson, Mr. Jackson, Mr. Rifé, for giving us excellent papers on processing.

I'll now turn the meeting over to Joe Reynolds, who will monitor the next subject, Mechanics on Formulation and Calculation.

(Mr. J. E. Reynolds assumed the Chair.)

CHAIRMAN REYNOLDS: This next subject, "Mechanics of Formulation" has been included in the 1959 Round Table program to permit a review of methods and techniques used in preparing granulation formulas.

Granulation formulas were initially an adaptation of pulverized formulas. Many changes have evolved through trial and error, an overall better understanding of processing variables and influences.

The pulverized shrinkage factors of two to three percent as applied to granulation have long since passed. Experience has pointet out formulation weaknesses and adjustments have been made during the past three to four years.

We now hope to consolidate the comparative thinking of the Round Table to determine if we are formulating in an efficient and accurate manner.

We have assembled a panel of men experienced in mechanims of formulation; I'm sure their discussion will be most enlightening.

The panel consists of Mr. Bill Tucker of G.L.F.; Mr. Ted Schmalz, F. S. Royster Guano; and Mr. Bert Tucker of Sohio Chemical Co.

Our first speaker is Mr. Bill Tucker of G.L.F.

MR. W. J. TUCKER: I was kind of intrigued by this "romantic" material. I was talking with Elmer Perrine just a couple of days ago and he was talking forever about "exotic" nitrogen materials like 16-8-8 and things like that and so I thought maybe I'd talk about "endearing" formulation practices for a few minutes here.

Mechanics of Formulation Calculations

W. J. Tucker

FORMULATION practices have been discussed at nearly all of our previous Round Table meetings and several fine papers have been presented on the subject.

The fact that questions continue to be asked is an indication of the intense interest in formulation; particularly as it relates to granulation. For this reason it appears again this year on our program.

My job is to review the calculations which are required in order to come up with a starting formula for production. In a sense, we will be quite specific because it is of little consequence to an operator that a particular nitrogen solution, acid, or superphosphate is best to use if he does not have on hand these particular materials. What I mean is that oftentimes and for various reasons the operator will have to proceed with the existing materials although they may not necessarily be the best ones which might be selected.

With these preliminary remarks then, let us consider the specific situation in which an operator is called on to formulate an 8-16-16 granular fertilizer.

The following three step approach is used.

STEP I

Assess the materials on hand listing their nutrient analysis and moisture content as indicated by shipper's analysis or by company lab analysis.

Decide upon the ammoniation rates which will be used.

Decide on the overages of plant food to allow.

Decide on the end product moisture content which is desired.

STEP II

Set up and solve the necessary equations which reflect the known facts, assumptions, and end product requirements stated in Step I.

STEP III

Complete, extend and check the formula against the original requirements.

Other facts, assumptions or requirements

(1) All 8 units of nitrogen will come from solution.

(2) At ammoniation rates of 5.8# NH_3 per unit of P_2O_5 for super, 3.5# NH_3 per unit of P_2O_5 for triple, and 7.2# NH_3 per unit P_2O_5 for phos acid, the ammonia input to the formula shall balance the ammonia capacity of the formula.

(3) Overages of 1% (.16 units) shall be included for P_2O_5 and K_2O, and 6% (4.8 units) shall be included for nitrogen.

(4) End product moisture should be 1%.

STEP II

Our potash needs of 323.2 lbs. K_2O are taken care of by 530 lbs. of muriate at 61% K_2O. We shall consider the potash to be essentially water free.

The 170 lbs. of nitrogen (including overage) is derived from 386 lbs. of nitrogen solution. This amount of solution fixes the ammonia input to the formula at 386 x 0.22 or 84.9 lbs. NH_3

The final question then is: how much of each of the phosphatic materials will be needed to absorb the 84.9 lbs. of ammonia

Proceeding with our 8-16-16 problem:

STEP I

Material	Nutrient Analysis	Water Content
Nitrogen Solution	44% N (22-66-6)	6.0%
Muriate of Potash	61% K_2O	———
Ordinary Super	19.5% APA	6.0%
Triple Super	46.0% APA	4.0%
Wet Process Phos Acid	54.5% P_2O_5	16.0%

added, supply the P_2O_5 required, and enable the operator to end up with 2000 lbs. of end product of the desired N, P_2O_5 and K_2O at 1% moisture?

It should be noted that if any of the factors of ammoniation, moisture, or nutrient content are neglected, or considered independently without due regard for each other, our operator will soon be in serious trouble with loss of ammonia, off analysis, false inventory pictures on materials or mixed goods, lack of process control, excessive down time, or other difficulties which will be reflected in plant costs and product quality.

Since one of our initial aims was a 1% moisture end product, each ton of end product can be thought of in terms of 1980 lbs. of dry material and 20 lbs. of water. In other words our formula should contain a total of 1980 lbs. of raw materials on a dry basis. We already know that 530 lbs. of muriate (dry basis) and 363 lbs. of nitrogen solution (dry basis 386x.94) are needed. Consequently, 1980 — (530 + 363) = 1087 which is the space available to us for the three phosphatic materials on a dry basis.

Let us consider the three phosphates as:

A = lbs. of super to be used.
B = lbs. of triple to be used.
C = lbs. of phosphoric acid to be used.

We can now write an equation representing the three phosphates on a dry basis to our 1087 lbs. of space. (Equation 1.)

#Super dry basis		#Triple dry basis		#Acid dry basis		#Space available dry basis	
.94A	+	.96B	+	.84C	=	1087	(Equation 1)

We also know that the ammonia added from solution is 84.9 lbs. and this amount is to be absorbed by the phosphates according to their respective capacities. This can be written in equation form also. (Equation 2)

NH_3 absorbed/ unit of P_2O_5 Super	NH_3 absorbed/ unit of P_2O_5 Triple	NH_3 absorbed/ unit of P_2O_5 Acid

$$\left(\frac{.195A}{20} \times 5.8\right) + \left(\frac{.46B}{20} \times 3.5\right) + \left(\frac{.545C}{20} \times 7.2\right) = 84.9\ NH_3 \text{ added from solution} \qquad \text{(Equation 2)}$$

Finally, all of our P_2O_5 is supplied by these same three phosphates and this fact can be expressed as our final equation. That is:

# P_2O_5 Super		# P_2O_5 Triple		# P_2O_5 Acid		# P_2O_5 required
.195A	+	.46B	+	.545C	=	323.2

(Equation 3)

Summarizing our three equations with the three unknowns:

$$.94A + .96B + .84C = 1087 \qquad \text{(Equation 1)}$$
$$.05655A + .0805B + .1962C = 84.9 \qquad \text{(Equation 2)}$$
$$.195A + .46B + .545C = 323.2 \qquad \text{Equation 3)}$$

Any one of the three equations can now be solved. I shall choose Equation 1.

$$.94A + .96B + .84C = 1987$$
$$.94A = 1087 - .96B - .84C$$
$$A = \frac{1087 - .96B - .84C}{.94}$$

$$\boxed{A = 1156.4 - 1.0213B - .8936C}$$

(Equation 4)

Having solved for A, we can substitute the A value in our other two equations #2 and #3.

$$.05655\,(1156.4 - 1.0213B - .8936C) + .0805B + .1962C = 84.9$$
$$.195\,(1156.4 - 1.0213B - .8936C) + .46B + .545C = 323.2$$

Simplifying the above, we get:

$$.0227B + .1457C = 19.5 \qquad \text{Equation 5)}$$
$$.2608B + .3707C = 97.7 \qquad \text{(Equation 6)}$$

Multiplying Equation 5 by 11.489, changing the signs of each term and adding 5 and 6 we end up with:

$$\begin{array}{r} -.2608B - 1.6739C = -224 \\ +.2608B + 3.707C = 97.7 \\ \hline -\ 1.3032C = -126.3 \end{array}$$

$\boxed{C = 97 \text{ lbs.}}$ Phos. acid

If we now substitute the phos. acid value of C = 97 into either Equation 5 or 6 we can get the B unknown which is the amount of Triple.

$$.2608B + .3707\,(97) = 97.7$$
$$.2608B = 61.7$$

$\boxed{B = 237}$ Triple Super

Finally C and B can be substituted into any of our original three equations 1, 2 or 3 to get A.

$$.195A + .46\,(237) + .545\,(97) = 323.2$$
$$.195A = 161.3$$

$\boxed{A = 827}$ Ordinary Super

STEP III

Completing our formula we now have:

Lbs.	Material
Nitrogen Solution 44% N (22-66-6)	386
Muriate of Potash 61% K_2O	530
R. O. P. Super 19.5% APA	827
Triple Super 46% APA	237
Phos. Acid W. P. 75% 54.5% APA	97
	2077

The moisture, ammoniation and plant food balance are shown in the following expanded formula

Chairman Reynolds: We're going to hold off on any questions until all the speakers have finished and then we will come along with questions. I'm sure we all will have some at that time.

The next speaker is Ted Schmalz of F. S. Royster Guano.

Mr. T. R. Schmalz: The scratching on the blackboard is mine over here and as I told Joe at lunchtime I was going to have somewhat of a problem using a hand mike and the paper and the chalk all at the same time, so I put it up here at lunchtime.

Material	Lbs.	N	P_2O_5	K_2O	H_2O	NH_3 added	NH_3 absorbed
Nitrogen Solution 44% N (22-66-6)	386	169.8			23.2	84.9	
Muriate of Potash 61% K_2O	530			323.3			
Ordinary Super 19.5% APA 6% H_2O	827		161.3		49.6		46.8
Triple Super 46% APA 4% H_2O	237		109.0		9.5		19.1
Phosphoric Acid W.P. 75% 54.5% APA 16% H_2O	97		52.9		15.5		19.1
	2077	169.8	323.2	323.3	97.8	84.9	85.0
Water lost in drying	77				77.0		
	2000				20.8		
Analysis at 1% end product moisture and no ammonia loss		8.49	16.16	16.17			

T. R. Schmalz

THE methods employed by the F. S. Royster Guano Company in formulating granular fertilizer is in my opinion one of the best. The advantages to the Royster method are that it eliminates the necessity of trial and error calculations and it precisely accounts for moisture losses and for material losses due to processing inefficiency, that is in ammonia absorption and dust collection efficiencies.

The method has been in use for several years. It has been revised somewhat since its inception but has basically remained the same.

The underlying principle of the method is formulation on the basis of the product rather than on the basis of the raw materials used. This concept requires that the raw materials used in manufacture be handled as they appear in final product. To do this moisture content of final product is controlled to close tolerance. Our moisture in product is a quality control item and is handled the same way that the plant food content analysis is. The moisture in all granular goods is maintained as close to 1% as can be obtained. It is a simple matter then to put all the raw materials used on a 1% moisture content basis. This is easily done by calculating the analysis of the raw materials to a 1% moisture basis and formulating the goods to a 1% moisture content 2000 pound ton. Tables of analysis for various moisture contents of super, triple, and phosphoric acid as well as the solutions which we use have been prepared for quick reference by those doing the day to day formulating.

An example of what is involved for instance would be calculation of the analysis and what we have termed the moisture factor, which will be used later for calculation of actual material usage, of superphosphate. In this case let us say we have super at 19.83 APA and 7.25% moisture. To reduce this material to a 1% moisture would result in increasing the APA by an amount equivalent to 19.83 x 1.067 or 21.16% APA. The figure 1.067 is the moisture factor for the material containing 7.25% moisture and is calculated by the expression:

$$\text{Moisture Factor} = \frac{99}{100\text{—material moisture}}$$

or in this case:

$$\frac{99}{100 - 7.25} = 1.067$$

Let's assume we are using this super to manufacture 5-20-20. For our requirements a 20.2 APA is formulated since we use a 0.2% overage in APA for this grade. For this particular run we have triple which runs 45.25% APA and 6.80% moisture. The moisture factor in this case is

$$\frac{99}{100 - 6.80} = 1.062$$

and the dry basis analysis is 45.25 x 1.062 or 48.06. Let us say that the other materials, on the dry basis in this formula, are as follows:

Anhydrous Ammonia	73
410 (26.3-55.5-0)	81
Muriate of Potash	662
Sulfuric Acid (66° Be)	75
	891

There are then 2000 — 891 or 1109 pounds of room left for APA addition. The standard method of obtaining the super and triple requirements is used with the dry basis analyses:

$$\text{Super requirement} = \frac{1109 \times .4806 - 404.0}{0.4806 - 0.2116} = 479 \text{ pounds}$$

$$\text{Triple requirement} = 1109 - 479 = 630 \text{ pounds}$$

Our 1% moisture basis ton is now complete and must be converted to the wet basis or the requirements of materials as they actually occur.

This done by application of the moisture factor to the dry basis weights in the following manner:

Material	Dry Basis	Moisture Factor	Wet Basis
Anhydrous Ammonia	73	1.000	73
41% Solution	81	1.210	98
Super	479	1.067	511
Triple	630	1.062	669
Muriate	662	1.000	662
66°Be H_2SO_4	75	1.062	80
	2000		2093

We see immediately that there will be a 93 pound moisture shrinkage for this formula and there is no necessity for trial and error calculations to obtain the super and triple requirements.

We have added one other feature to this method for our purposes of material accounting. The final formula which we will use in the plant will have another factor applied to it which we have termed the process loss factor. This merely is an empirical value established by experience in the various grades and factories to express the handling losses which occur in the operation. For the sake of example let us say that we have determined that a 2% handling loss is characteristic for this case and that for Free Amonia containing materials in this formula a 5% loss has been experienced. The final formula for plant use will then be as follows:

Material	Wet Basis	Process Loss Factor	Final Formula
Anhydrous Ammonia	73	1.05	77
41% Solution	98	1.05	103
Super	511	1.02	521
Triple	669	1.02	682
Muriate	662	1.02	675
66°Be H_2SO_4	80	1.02	82
	2093		2140

At first glance this method may seem cumbersome, but I assure you that it has worked wonderfully well for us in all respects. With this method of approach we have been able to not only simplify the mechanics of formulation, but have been able to keep close surveillance of operations from efficiency and inventory respects.

The individual grade formulating instructions are sent to each factory with the following information:

Nitrogenous materials requirement in units of Nitrogen.

Sulfuric Acid requirement in pounds per ton.

Phosphoric Acid requirement in pounds per ton.

Process loss factors for Ammonia and Non-Ammonia containing materials.

Rigid control of plant practices is maintained and the technique of improvising during operation is frowned upon. Material accounting is one of the highly important phases of operation, since profit margins are narrow and must be maintained accurately.

Chairman Reynolds: Our next speaker is Mr. Bert Tucker of Sohio.

H. H. Tucker

THIS section of the Round Table is to discuss calculating fertilizer formulations and methods or short-cuts which are being used when adjustments in formulations are necessary. If you are not already using triangular diagrams as a tool, I suggest their use to help in original formulations and calculations and in adjusting formulations. At first the use of these triangular diagrams may seem very complicated but with use they have proven to be helpful, at least to me.

Before we discuss the actual formulation calculations we should consider a few of the principles of fertilizer manufacture. Formulation of dry fertilizers to obtain a desired product varies widely depending on a number of factors. Some of these factors are:

1. Grade of fertilizer.
2. Type of fertilizer to be manufactured (granular, semi-granular, or pulverant).
3. Desired rate of production.
4. Raw materials available.
5. Costs of raw materials.
6. Variation in qualities of raw materials.
7. Equipment of production.
8. Plant location.
9. Length of storage of product.
10. Use as bagged or bulk fertilizer.
11. Atmospheric or weather conditions.

These factors certainly could not be listed in the exact order of their importance. In fact, their relative importance will vary greatly depending on these and a number of other conditions and factors.

Nitrogen Materials:

In this discussion of formulation calculations I propose insofar as possible to deal only with nitrogen raw materials. Nitrogen calculations may prove to be more complicated than for phosphate materials since as many as three raw materials may supply the plant food nitrogen. In addition the amounts of free ammonia nitrogen and nitrogen from salts must be considered separately. Furthermore, various nitrogen salts differ in their effect on the mixed fertilizer in which they are used. The amount of the so-called soluble salts (ammonium nitrate and/or urea) will affect the moisture content which can be tolerated in a fertilizer. Still further the proportion of urea to ammonium nitrate is extremely important as this proportion affects the total solubility of the two salts.

A few rules of thumb may be listed as guides to formulation. Some of these are:

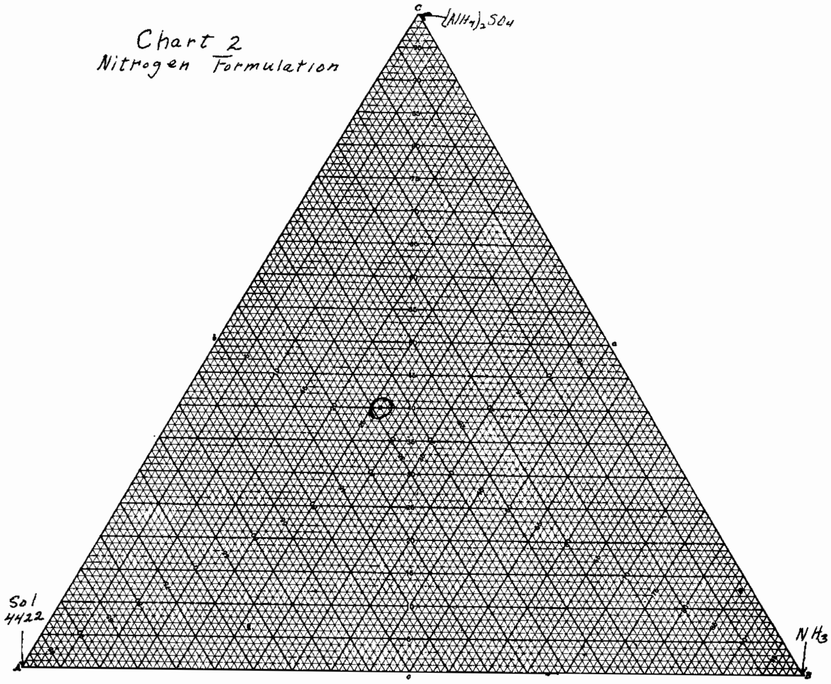
1. Ammoniate to the highest possible rate consistent with phosphates used, phosphate reversion, nitrogen losses, and manufacturing facilities.
2. Adjust total soluble salts to manufacturing and drying facilities, storage conditions, and use of product.
3. Limit amount of urea when used in conjunction with ammonium nitrate.

Ammoniation Rates:

Phosphate ammoniation rates were discussed very thoroughly at the 1958 Round Table by Mr. C. P. Schumacher. It was pointed out that ammoniation rates will be affected by a number of factors. The following ammoniation rates per unit of phosphate were used as rates from which to "tee off":

4.0# for triple super.
6.0# for normal super.
7.2# for phosphoric acid.
7.6# for phosphoric acid and normal super.

The 7.6# ammoniation rate was pointed out as giving a possible .4# bonus factor over phosphoric acid alone when phosphoric acid and normal super were used together. Other cases of enhancement due to a combination of other materials are well known to the fertilizer industry. In this dis-



cussion I will use these ammoniation rates.

Soluble Salts:

The upper amount of soluble salts which may be used will depend largely upon the kind and grade of fertilizer, and upon the ability to dry the end product. Adjustments in nitrogen materials as well as in the amount of acid used will affect agglomeration, stability of the product, and amount of drying required.

Use of Urea:

A general rule of thumb on the amount of urea which can be used in conjunction with ammonium nitrate is not over 50# of urea per ton of end product, and perhaps preferably in the 20 to 40 pound range.

Calculations:

The foregoing discussion presents some of the principles to be considered in calculating formulations. The complexity of considering all of these principles together shows why formulations often need to be adjusted and why the first formulations used may not be satisfactory.

Now we get to one short-cut which can be used in calculating original formulations and in calculating adjustments in formulations. As previously stated I will discuss the use of triangular diagrams as a tool in calculations. Here are some of the things which can be portrayed on such triangular diagrams when calculating nitrogen formulations:

1. Sources of nitrogen material.
2. Amounts of nitrogen from various sources.
3. Ammoniation rates.
4. Amounts of various nitrogen salt . . . ammonium sulphate, ammonium nitrate, urea, or any combination of these salts.
5. Total water content of nitrogen materials.
6. The effect of acids on ammoniation rates.

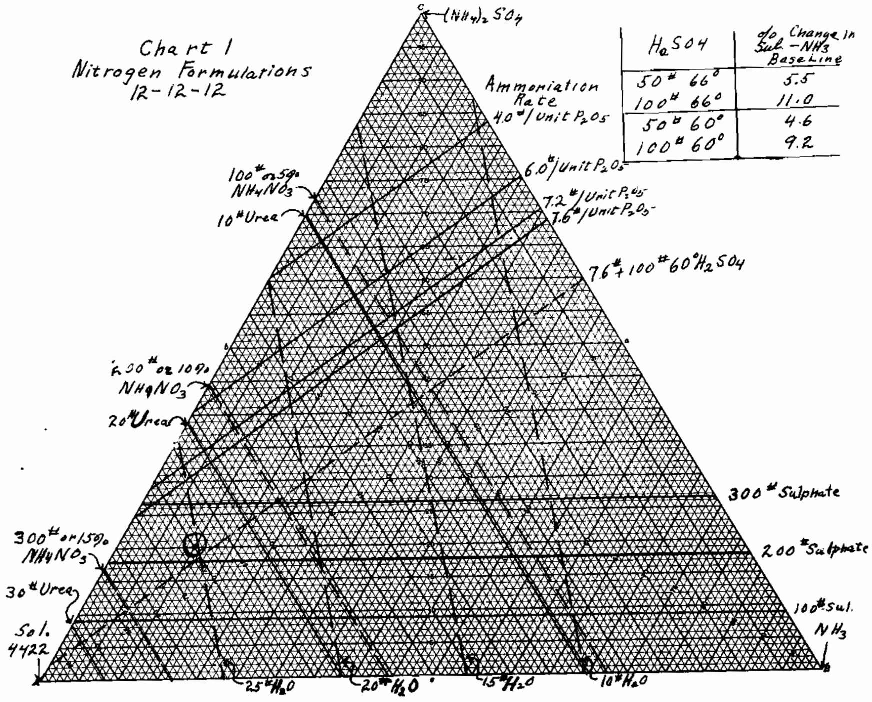
These factors are all presented in Chart 1 for a 12-12-12 fertilizer grade.

At first glance this chart looks very complicated, which it is and it certainly will require study. To simplify and better understand this chart we will break it down and study it by steps. The first step is to understand how a triangular diagram is constructed. Chart 2 is used for this purpose.

Chart 2.

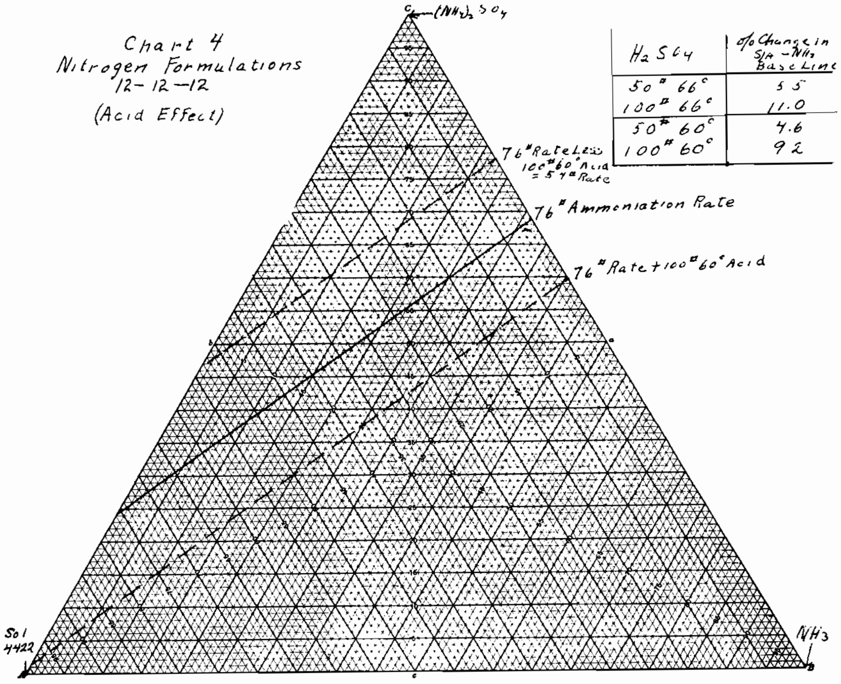
Triangular diagrams with intermediate lines parallel to each base line are used to read respective portions or percentage of each of three materials. Chart 2 like Chart 1 represents nitrogen formulations where certain portions or percents of the total nitrogen are derived respectively from ammonium sulphate, anhydrous ammonia and nitrogen solution 4422. This 4422 abbreviation refers to solution 440 (22-66-6) as adopted by the National Plant Food Institute. Abbreviation 440 was not used since other solutions also contain 44.0 percent nitrogen.

Each labeled apex represents a point where 100% of the nitrogen comes from the respective material as labeled. The base line opposite each apex represents formulations where none of the nitrogen is derived from the apex material but would be derived entirely from one or both of the other materials. Points within the diagram represent formulations



where each of the three materials supply a part of the nitrogen. The point shown in this diagram illustrates a given formulation. In this formulation 40% of the nitrogen is derived from ammonium sulphate. This 40% is determined by reading from base line "c" toward apex "C" representing ammonium sulphate. Likewise, 26% of the nitrogen in this formulation is derived from anhydrous ammonia as can be read on the lines parallel to base "b" and extending to apex "B". Also reading from base "a" toward apex "A" we determine that 34% of the nitrogen is derived from solution 4422. The accuracy of these readings is proven by the fact that 40, 26, and 34% total 100%.

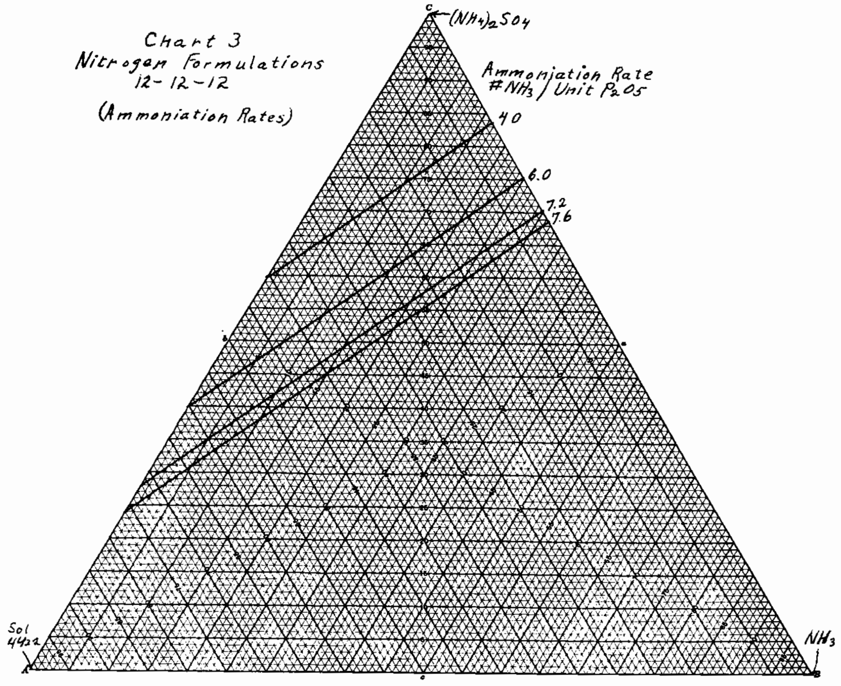
The amounts or percents read directly from the diagram are percents of the total nitrogen in the fertilizer. Since a 12-12-12 contains 240# nitrogen per ton, the percents read from the diagram would be percents of 240#. 40% of the nitrogen which is from ammonium sulphate would be 96# (40% of 240). This 96# divided by .21, the nitrogen content of ammonium sulphate, equals 458# of ammonium sulphate per ton of end product. Amounts of anhydrous ammonia and solution 4422 are dedermined in the same way. Anhydrous ammonia will furnish 26% of the total 240# of nitrogen per ton. 26% of 240# equals 62.4.



H_2SO_4	% Change in S/A - NH3 Base Line
50# 66°	5.5
100# 66°	11.0
50# 60°	4.6
100# 60°	9.2

62.4 divided by .82, the nitrogen content of anhydrous ammonia, equals 76.1# of anhydrous ammonia per ton of 12-12-12. In the same way, 34% of 240# equals 81.6# nitrogen from solution 4422. 81.6 divided by .44, the nitrogen content of solution 4422, equals 185.5# of this solution per ton of 12-12-12.

Next we look at the ammoniation lines from the original formulation chart. These lines are shown in Chart 3 and represent the various ammoniation rates as discussed earlier.



The 4.0% ammoniation rate was used by Schumacher as being typical for triple super, the 6.0 rate for normal super, the 7.2 rate for phosphoric acid, and the 7.6 ammoniation rate as typical for a combination of phosphoric acid and normal super.

With this chart it is possible to calculate the amounts of nitrogen to be derived from any of the three nitrogen sources in any possible nitrogen formulation of a 12-12-12 with either of the four ammoniation rates. All possible formulations with a 4.0 ammoniation rate will fall on the 4.0 ammoniation line, and likewise all formulations with a 6.0 ammoniation rate will fall on the 6.0 ammoniation line.

Other ammoniation lines may be added as desired. This can be done by determining the two formulation points on the base lines having this ammoniation rate and connecting these points with a straight line. All possible formulations with the calculated ammoniation rate will then fall on the established line.

Acids Effect Ammonia Usage:

We next look at the effect that added acid will have on the amount of ammonia which may be used. If additional ammonia is not added with additions of acid, the acid will decrease the ammoniation rate. Chart 4 features one ammoniation rate shown on the previous chart, that is a 7.6# ammoniation rate, and the effect 100 pounds of 60° Be′ H_2SO_4 may have on increasing the amount of ammonia which may be used, or on reducing the ammoniation rate. Chart 4 on preceding page.

The dotted lines on either side of this ammoniation line shows the effect of adding 100 pounds of 60° Be′ sulphuric acid. The lower dotted line shows the additional ammonia which can be added as anhydrous ammonia, and/or solutions with this amount of acid and still maintain the 7.6# ammoniation rate. The upper dotted line shows how much the ammoniation rate would be reduced were this amount of acid to be added merely to generate heat and improve agglomeration without using additional ammonia to neutralize the acid.

As can be seen by the table in the upper right-hand corner of Chart 4, the use of 100 pounds of 60° Be′ sulphuric acid would make it possible to obtain 9.2% more of the total nitrogen from anhydrous ammonia and a corresponding less amount of the nitrogen from amonium sulphate. Were all of the nitrogen to be derived from ammonium sulphate and solution 4422, which contains both ammonia and nitrogen salts, the percentage change would be much greater. Formulations where only these two materials would be used will fall on the "b" base line. Here, 100 pounds of 60° Be′ acid would make it possible to use 22% more of the total nitrogen from solution 4422 with a correspondingly smaller amount of the nitrogen from ammonium sulphate. When all three nitrogen materials are used at this ammoniation rate and with this amount of acid, all formulations will fall on the lower dotted line within the diagram.

The use of 100 pounds of 60° Be′ acid without adding additional ammonia would have the same effect as using 9.2% more of the nitrogen from ammonium sulphate and a correspondingly smaller amount from ammonia. This would reduce the ammoniation rate of the phosphate from 7.6 to approximately 5.4 pounds of ammonia per unit of phosphate as shown on the upper dotted line.

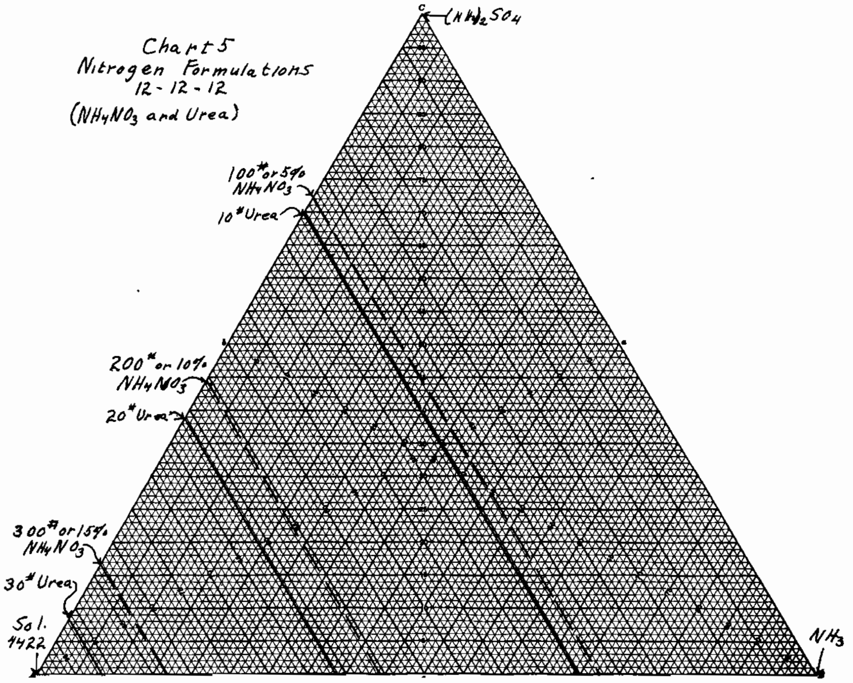
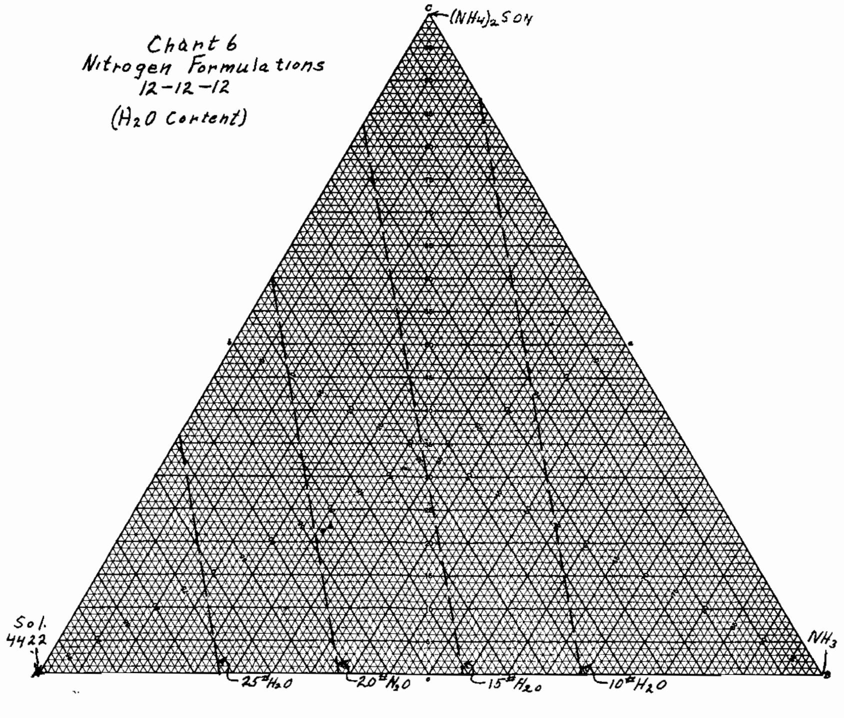


Chart 5 shows the effect that increments of solution 4422 will have on the amount of ammonium nitrate and urea per ton of 12-12-12.

With adequate drying facilities all of the nitrogen in a 12-12-12 could be obtained from solution 4422 without having an excess of ammonium nitrate or urea. Should experience have shown that the upper limit of ammonium nitrate is less than 300 pounds or 15% of the weight of the fertilizer, it would be necessary to limit the use of solution 4422 to 450 pounds or less.

Were all of the nitrogen in a 12-12-12 obtained from solution 4422 the 6% of urea it contains would not be in excess of the 40—50 pound upper limit of urea, previously discussed. Urea content would therefore not be a controlling factor on the amount of solution 4422 which could be used in a 12-12-12. The use of other solutions containing a higher percentage of urea may be limited however because of the urea they contain.

The amount of water added to the fertilizer mixture by these nitrogen formulations is shown in Chart 6.



Since other fertilizer materials contribute water to the mixture also, the inclusion of water lines on this formulation diagram is probably not as meaningful as are other factors shown on Charts 3, 4 and 5.

Use of Triangular Diagrams for Formulation:

We now return to Chart 1 which combines the ammoniation lines, the 100 pounds of additional acid line, the urea and ammonium nitrate lines and the water content lines. Ammonium sulphate lines at 100 pound increments have been added for quick reference.

We now set up a hypothetical formulation based on various assumptions. These are:

1. Nitrogen materials available are sulphate of ammonia, anhydrous ammonia and solution 4422.
2. With the phosphates we will be using we believe we should obtain a 7.2 to 7.6 ammoniation rate.
3. Experience has led us to believe we will need approximately 100 pounds of sulphuric acid to obtain a satisfactory granulation.
4. Experience has also indicated that under our manufacturing conditions we will want less than 15% or 300 pounds of ammonium nitrate per ton of end product.
5. That urea should be limited to not over 40# per ton of 12-12-12 is also indicated by experience.
6. A minimum of ammonium sulphate will be used, preferably not over 300 pounds per ton.
7. Nitrogen formulations will be calculated without overages or at 240 pounds of nitrogen per ton of 12-12-12.

With these facts in mind we select the formulation represented by the point marked on Chart 1 as a starting formulation. This formulation would derive the indicated 20% of its nitrogen from ammonium sulphate, 10% from anhydrous ammonia and 70% from solution 4422. These percentages are applied to 240 pounds, the nitrogen content of a ton of 12-12-12. We find that 48# of nitrogen will be derived from ammonium sulphate which will require 299# of this material. 24# of nitrogen will come from anhydrous ammonia or 29.3# of anhydrous ammonia will be used, 168# of nitrogen will come from solution 4422, or 382# of solution 4422 will be used.

From the chart we see several important things. Even with a 7.6# ammonium rate, about 75# of sulphuric acid would be required to neutralize the excess ammonia in this formulation. Should 100 pounds of acid be used, the extra 25 pounds would reduce the ammoniation rate to approximately 7.2# ammonia per unit of P_2O_5.

We can also see that this formulation would contain about 23# urea per ton of 12-12-12 which is not considered excessive. This nitrogen formulation would contain about 250# or 12.5% of ammonium nitrate per ton. This amount of nitrogen material would not contribute much water since the highest water content nitrogen material is solution 4422 which contains only 6% of water. The water content of the combined nitrogen materials would be approximately 25 pounds.

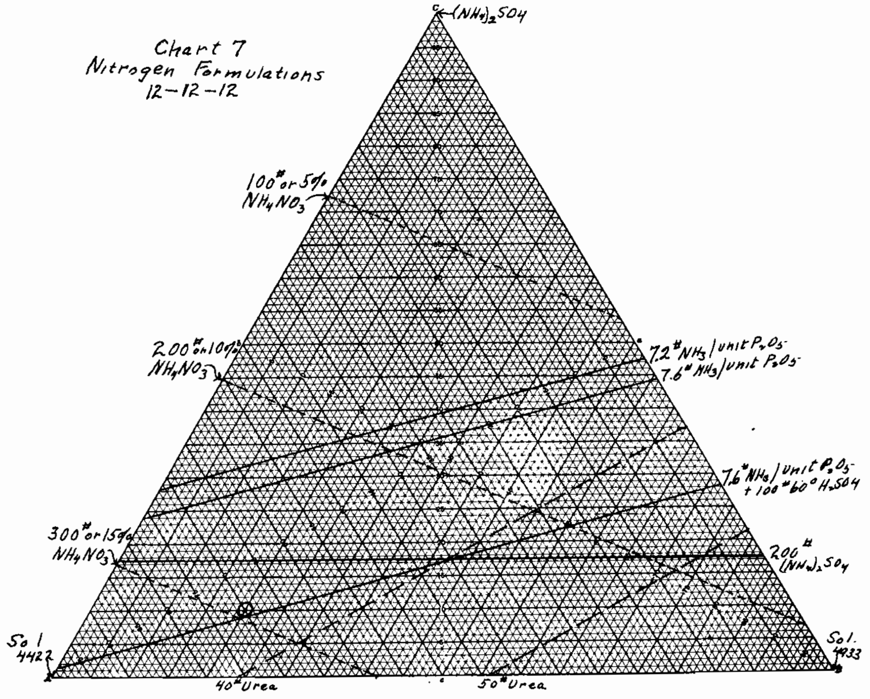
Adjusting Formulations:

From the formulations represented by the point on the diagram we can adjust formulations very much on the "cut and try" plan normally used. These adjustments will be made using known principles and experiences in manufacture. Some of the principles stated positively would be:

1. Addition of H_2SO_4 generates heat, improves agglomeration, but at the same time reduces the ammoniation rate.
2. Addition of free ammonia also generates heat and dries the product but may at extremely high rates of ammoniation cause ammonia losses or even reduce availability of phosphates.
3. Agglomeration is increased by:
   a) Increasing the so-called soluble salts (ammonium nitrate and/or urea).
   b) Increasing the proportion of urea to ammonium nitrate.
   c) Increasing water content.
   d) Increasing heat of product.

Were the product produced from this formulation not satisfactory, we would reason from known facts and experience and would then reset the formulation point a percent or two in the direction indicated and try the new formulation. Moving in any direction from a given formulation on the diagram will decrease or increase the amounts of ammonia, soluble salts, urea, water and heat of reaction.

Using the calculation method previously described we quickly re-



formulate the nitrogen materials, cutting back on one nitrogen source and compensating for this nitrogen cut-back with increases in one or both of the other nitrogen materials.

Chart 7 is shown to point out how all of these principles might be put together with a minimum of confusing lines. (See page 37).

In this formulation, solution 4933 or 490 (33-43-13) is used instead of anhydrous ammonia. Other nitrogen materials are ammonium sulphate and solution 4422. Solution 4933 with its low fixed-to-free nitrogen ratio, its relatively low ammonium nitrate content and high percent of urea when used with solution 4422 also containing ammonia, ammonium nitrate and urea, but in different proportions, brings about all of the possible variables into play. On this diagram are shown:

1. A 7.6# ammoniation line.
2. A 7.6# ammoniation line plus 100# of 60° Be′ H_2SO_4.
3. The 40 and 50# urea lines.
4. Ammonium nitrate lines in increments of 5% or 100#.
5. The 200# ammonium sulphate line.

Assuming that we consider 40# of urea and 15% of ammonium nitrate as possible maximums, that we probably will not want to use more than 100# of 60° Be′ acid, that we hope for an ammoniation rate in the range of 7.2 to 7.6, and that we would be willing to use 200# more or less of ammonium sulphate, we would then probably select the formulation indicated by the point on this diagram as the "tee off" point. At this point, 10% of the nitrogen would be derived from ammonium sulphate, 20% from solution 4933 and 70% from solution 4422. Formulation adjustments could then be made as needed around this point. The lines shown on this chart prescribe a set of limitations which we woud like to approach but would not want to exceed. We can then quickly evaluate the many factors involved and calculate or adjust nitrogen formulations to give a desired product. It is the evaluation and successful application of the many factors involved in all phases of production to obtain the desired result that is the science and art of fertilizer manufacture.

Chairman Reynolds: I have some comments here according to the program that I'm supposed to add at this time, but because of time I think I will withhold those and they will be in the proceedings.

I think one of the things that we do want is some discussion here. We've discussed many avenues here of formulation; I'd like to add something here as food for thought; maybe we can toss these around a little later.

This is a portion of what I had originally planned to say here; this is toward the end.

The foregoing discussions outlined in a general way the mechanics of formulation. Many of the features are very specific. Some controversies and indecisions will exist and it is hoped that more exact approaches can be developed rather than continue in the use of empirical factors.

On the other hand, maybe the use of empirical factors is the best method, but all possibilities should be investigated. The following discussion reviews several of these problems, but does not necessarily provide the answers. It is hoped that enlightenment will be prompted from this group.

One of the problems facing a superintendent is to establish realistic available P_2O_5 and moisture values for phosphate materials. How many times have known P_2O_5 values been used in calculating formulas only to find the product P_2O_5 value has little resemblance to that formulated.

Correlating the raw material P_2O_5 value with the finished product P_2O_5 value is one of the immediate problems of our industry.

Another consideration is the effect of the dilution of analysis by moisture pickup. In the 8-16-16 example that was used by Bill Tucker, standard moisture value of 20 pounds of water per ton of product was established as his quality goal.

Experience has shown that a product moisture content of one per cent for 8-16-16 can be maintained within two-tenths of a per cent between the time of production and the time of shipment.

However, when you get into more hygroscopic grades and grades of higher concentration these grades can pick up as much as one per cent moisture and if this dilution can be predicted, should compensations be made in the formula?

In the production of high analysis mixed fertilizers formulated from large amounts of triple superphosphate and phosphoric acid the problem exists of what to do when the granules are dry, hard and gritty; the product temperature leaving the drier is 230 to 240 degrees Fahrenheit, but the analytical laboratory reports a five to six per cent moisture which is two to three per cent above formulated quality standards. As we know, this is often high enough to cause caking.

If two to three per cent or 40 to 60 pounds of additional water are actually present in the ton of product, the analysis will also be reduced by the same amount as if 40 to 60 pounds of filler were added. Should weight compensation be made for these conditions? Is additional drying necessary?

An investigation of the relationship between free water and water of crystallization in high analysis grades and some system of differentation between the two is needed.

These thoughts that we have mentioned here, I think together with the other papers, should bring some questions from the floor, so let's turn the meeting back to you in the audience and see if we can have some discussion.

Would you come up this way and get on the mike, please?

Mr. A. S. Jackson: Joe, I want to ask about formulation, they are being made mechanical but the end product of all our labors is still dollars. For instance, in the first formulation that Bill Tucker gave he gave an algerbraic solution of normal, triple and phos-acid. Over here in the Southeast of eastern states, I'm sure that that would be the cheapest as the cost of P_2O_5 goes up as concentration increases.

But if you go out to the West Coast wet phos acid is cheaper than triple. Don't mechanical formulations break down when you begin to try to apply them to dollars which are what we are trying to make?

The next thing we've got to worry more about, is the capacity of an ammoniator, for instance, is more limited by heats of reaction than by finite material going through it. Shouldn't heat be a function of formulation or chemical activity? Shouldn't there be some way to at least estimate liquid phase?

I don't necessarily mean that you have to hit it on the head but to approximate, to give you some idea of what you're going to run into when you start a grade that you're not familiar with.

Doesn't mechanical formulation by formula soon break down and come right back to an art in which the final end part gets back to trial and error by the formula? I would like some way of doing it mechanically if it were possible to accommodate dollars, operating characteristics that we have to work with?

Does anybody have that in their method of formulation?

Chairman Reynolds: I think that's a very deep subject, but I think it is very timely.

I think there have been attempts through the use of IBM machines and conputers to include all those factors as far as raw material costs, liquid phase and so on. I think the Monsanto people have done a tremendous amount of work along that line.

Maybe they've been able to relate that so that the superintendent can put a formula together.

Does anyone have any comments?

When we were considering grades like 5-10-5 or 3-12-12 we had one type of problem; now as we go to higher analyses, and our states become a little more strict and rigid, we have to figure out, are we doing this right or should we be doing something else?

Any one have any other methods of formulation?

Mr. Larry Lortscher: In answer to what Allen asked about, without a very expensive computer, if you put it in, even then I question if we know enough about the basic relationships on which a formulating equation would stand. On the whole range of formulas we can put it on and be sure of, but without that all we can use that I can see from the work in the trade is the text service work that I've done. You take certain guide rules.

Various people have different ways of doing this. To take a formula that has'n been run before in a plant, the formulation that we were talking about and take the 8-16-16 example, when they run that they have ideas of whether it will work or whether it won't.

Their problem is to make sure they have analysis. That was the critical point in that formulation for formulating whether it would granulate or not.

I've got, for instance, my own touchstones for liquid phase and heat. You can't plug in the heat factor on phosphoric acid and on your anhydrous ammonia, it will react with the sulphuric and react with P_2O_5, and the effect is super triple, then add them up and they come up to a figure that you might use as a general criterion.

To bring it down to myself, I use satisfactorily, generally, the open system. I'm talking about 200,000 btu's. Using the figures in very many handbooks along the liquid phase, you add up your total liquids dissolved as well as the water—in my own case it runs around 20 per cent, which is something to try to hang your hat on, but as far as trying to pin it down using triangular charts and try to pin it down, not just using it as a guide, I don't think we have the knowledge for all the complex materials that we're actually using to pin it down any tighter.

Chairman Reynolds: Al Henderson.

Mr. Henderson: I think as long as we use variable materials we're going to have variable formulations and also we can't control the weather. Sometimes we use superphosphate that's six or eight months old; maybe three or four days later the superphosphate's nine months old. We might have seven or eight thousand tons of superphosphate in storage and we ran out of room so we piled green super on top of old super; one day we use super two days old and a week later we've got down to the bottom of that pile and it's nine months old. It has a moisture content of five and a half to six per cent for the green, but we had nine per cent.

There's a lot of difference between old and green superphosphate besides just moisture content. I don't know what it is, but there's a significant difference. I mean besides the free acid and insolubility and that might be responsible for it, but green super is peculiar stuff.

Also the weather even in Florida where we have fairly constant and warm temperatures — the weather affects our formulations when the weather changes we change something else. We change the amount of recycle or the amount of "wetter" that we add to the system.

But those are two influences that are variable and I think will be variable for a long time and I think it deserves some consideration.

Chairman Reynolds: Ted Schmalz.

Mr. Schmalz: I'd like to answer Allen's philosophical plea with a philosophical answer. By way of comparison, let's say of the fertilizer industry, with medicine—you can send a boy to school for eight years and after a review he's still got to go out and practice to get the art. I don't think we're ever getting away from the art proposition on this deal. You have to work in a plant; you've got to feel your way along. I just don't see any other way of going about it.

Chairman Reynolds: You sound a whole lot like our management. It just seems to me that we ought to be able to do it without all this trouble.

Mr. Jackson: What I was trying to establish is that formulation, no matter what mechanics we use to get specific numbers, is still an art; is going to be an art for a long time. The formulator is going to gain through the experience. I'm sure most of you can make a formula, look at it, run it, half the time, or better than half the time, you'll hit it the first time.

Certainly you'll get it the second time. Probably the first time you formulated, it took three or four trips. You do things in your head that you don't put on paper. If people would write them down we would get a little more uniform, cut some of the trial and error for people not quite as experienced.

More than anything I think the dollar sign should be put in every formula sheet. I see as I go around to different mills, people formulate in many mills, the operating personnel don't know what raw materials cost.

I think they are hurting themselves terrifically be formulating purely for something that runs easy rather than formulating to get a minimum cost fertilizer.

I'm not wanting a cure-all; I'm simply stating that a condition exists and it is an art.

CHAIRMAN REYNOLDS: Thank you, Allen.

Any other questions? Comments?

Are there any other approaches to actual formulation? I mean we've talked primarily about this wet-dry basis; we've talked about the zero moisture; we've talked about a one per cent moisture; it there any other system?

Seems like we're doing it all the same way.

MR. E. MORTENSON (Swift and Company): I thought that a few years ago there was one approach that was quite of value to all of us and I haven't heard it mentioned at all today, the pilot plant approach. I'm wondering if that fits in the picture at all. We've just one unit with a 100 tons. We make a mistake and throw it in a pile and then start over again and try another formula or do we go back to the laboratory and the pilot project? I'm just asking the question, is the pilot plant still in the picture or is it out?

CHAIRMAN REYNOLDS: That's a very good question. Anyone have any answers? Comments?

MR. MORTENSON: My question is this: Should we fall back on the pilot plant as the place to solve some of these problems or has there been experience that that doesn't answer our question?

CHAIRMAN REYNOLDS: Can we hear from two or three companies that have pilot plants where they have taken data from or made them back through pilot plants or vice versa?

Someone from Spencer or Monsanto?

MR. MARBURGER (Spencer): I don't think I can answer very much of the pilot plant experience except to go back to a first answer—we'd have to define a specific problem we can determine whether it can be an advantage or not.

All pilot planning to this industry or elsewhere—there are some aspects of the type problem that a pilot plant can give a complete answer to and in others they can just give some marks that should be settled up.

Over all though, I think, in many of the problems facing us in granulation today we are beginning to find that basic data from a pilot plant, even in the small scale that we operate, are extremely useful in taking it to commercial size. I think our biggest fault is in not trying to add the scaled up factor but saying that, well, the pilot plant operates this, therefore this is what it should be and I think I'm probably straying from your question quite far, but I'm not sure I can answer it any better.

CHAIRMAN REYNOLDS: Do you have something pertaining to your pilot plant experience?

MR. C. FRANKLIN (Phillips): The one thing I've got is, there are so many variables going from plant to plant that don't behave the same as they do in our one particular pilot plant. If you had one particular pilot plant or one commercial plant probably you could solve a lot more of those problems than you can when you're trying to use one pilot plant for several different commercial plants.

CHAIRMAN REYNOLDS: Thank you.

Any other discussion?

We have time for about one more question or one more comment.

MR. T. STEWART (Deere and Company): I think a pilot plant is very useful if you don't have any experience in fertilizer manufacture, it's a good way to get it. I mean, if you haven't been around a fertilizer plant much and you want to get fast experience the cheapest way, the pilot plant is the best way to do it.

But once you've got your experience then your pilot plant is of limited use to you. You're going to have to work out the fine points in a larger plant.

MR. T. J. BOSMAN (Federal Chemical): In a lot of cases where they do pilot plant work and it's very good and all, but oftentimes they have to step that up to the plant scale on a semi-work scale first and they get more and make a lot of changes in between pilot plant and the final operations from the semi-work results.

CHAIRMAN REYNOLDS: Thank you very much.

I think we're going to have to conclude this particular session. Thank you very much. We have still one more feature on the program. I'm going to turn the meeting back to Dr. Sauchelli, but thank you very much for your cooperation and your patience.

(Dr. Sauchelli resumed the Chair.)

CHAIRMAN SAUCHELLI: We did have one more item on the program, but I think we'll transfer that to tomorrow morning if we have the opportunity. It was a discussion of modeling by a representative of the Dorr-Oliver Company.

Let's assemble tomorrow morning at nine o'clock sharp because we have a big day.

(The meeting adjourned at four fifty o'clock, p.m.

Thursday Morning Session, Nov. 5, 1959

The Round Table reconvened at nine fifteen o'clock a.m.,

Dr. Vincent Sauchelli presiding.

CHAIRMAN SAUCHELLI: It's a fact that in many sections of the country the manufacturer of conventional type fertilizers continues to represent the largest percentage of the total manufactured. Granulation has in the last few years made enormous gains and it is freely predicted that before long, even in the present predominantly conventional type areas, granulation will have succeeded in making many converts. However that may be, we believe that we should face realities and recognize that our colleagues who produce the conventional fertilizers also have problems worthy of frank discussion in our conferences.

Accordingly, our agenda for today's session will be devoted largely to such problems. In the past we have discussed some of the problems of the conventional type, but we've heard from many of you who attend regularly that perhaps we are neglecting conventional type fertilizers. We never intended to do that. Today's program reflects our thinking.

One change in the agenda is to be noted. It was not possible for Mr. Vance Ward to be with us in time to appear as scheduled, consequently Dr. McCall will give his talk on Statistical Quality Control as scheduled, but Mr. Ward will give us his lecture the first thing tomorrow morning. Don't miss these two talks. They have been integrated and will be very helpful to everyone who is interested in knowing more about this fascinating science.

We made a special effort to get statistical quality control on our program this year. Last year we tried to get Mr. Vance Ward to address the Conference on Chemical Control Problems. He made a special trip from Great Britain and got as far as Boston, but the plane was held up in Boston and he was just disappointed that he couldn't make Washington that day. So the Chemical Control Conference was unable to hear him. But we are able to get him to come this year and I promise you all it's going to be a most interesting and valuable lecture, and with the talk preliminary to his by Dr. McCall this afternoon you will have, I believe, enough on statistical quality control to interest you and make you want to know more about it, those of you who are not completely familiar with it. I think it's becoming one of those indispensable tools in modern industry that everyone here will want to know more and more about, and how to use it in the works.

We start this morning's session with a discussion of the Whys and Wherefores of Mechanical Condition. The speaker needs no introduction to this audience. He is recognized by us all as a most helpful investigator of fertilizer problems, has appeared frequently on our programs and has always been ready and generous with help and advice. It's a real pleasure to call on our friend John Hardesty of the United States Department of Agriculture, Agricultural Research Service.

Physical Condition of Mixed Fertilizers—Influence of Ammoniating Solutions

John O. Hardesty

THE purpose of the present discussion is to review briefly some significant data pertaining to the causes and alleviation of poor physical condition in mixed fertilizers and to consider certain effects of present-day ammoniating solutions on the physical condition of mixtures.

Hygroscopicity

There are two avenues which lead to poor physical condition in mixed fertilizers. One allows entry of sufficient moisture from the atmosphere to make mixtures damp and sticky, and the other allows caking of the bagged product. Moisture absorption is caused by the presence of excessive proportions of hygroscopic salts, or mixtures of such salts, in the fertilizer. Remedies for this type of poor physical condition include (a) formulation with tolerant proportions of hygroscopic salts, (b) storage in air-conditioned buildings, and (c) packaging the dried product in high-grade, moisture-resistant bags. The subject of hygroscopicity of fertilizers is well-covered in the technical literature (*2, 3, 7, 8, 9*), and appears to need no further elaboration here.

Caking

Caking of mixed fertilizers is of great concern to the manufacturer and the consumer because of its adverse effect on drillability of the product. Caking is the result of crystalllization of salts from solution at the surface of particles (*8, 10, 11*) which tends to cement, or knit, the particles of the mixture together. The degree of crystallization within the mixture is affected by moisture content (*6*), proportion of soluble salts in solution (*7, 10*), chemical reaction (*11, 13*), temperature (*13*), the number of contacts between particles per unit mass of the mixture as related to particle size and shape, and the mechanical pressure on the product such as that produced in the bottom bags of a high bag-storage pile.

Conditioning Agents

Conditioning agents are of practical value only when they eliminate caking (*6*). Consumer demand is for completely free-flowing products. Mixtures with a moderate caking tendency yield to conditioning agents (*4, 6*); those with a severe caking tendency do not. Control of the above-mentioned factors which cause caking (*13*) usually eliminates the need for conditioning agents.

Liquid Phase

Interactions and mutual solubilities of salts in a mixed fertilizer sometimes make practical methods of control a complex technical problem. Let us consider an example of the effect of mutual solubilities of ammonium nitrate and urea on the liquid phase imparted to mixed fertilizers by ammoniating solutions. Table I shows the relative volumes of aqueous solutions saturated with ammonium nitrate and urea in varying proportions at 86° F. (*5*). Total salt in solution and the relative volume of solution per unit volume of water increase with increase in the proportion of urea present up to the eutectic point of about 46% urea in this salt mixture, and then decrease on further increase of the proportion of urea present. At the eutectic point one gallon of water dissolves 131 pounds of salt mixture (60 lbs. urea and 71 lbs. NH_4NO_3) at 86° F. to produce 12 gallons of liquid phase. Data from this table are used in Table II to show the proportion of liquid phase imparted to a mixed fertilizer per unit of nitrogen supplied by four different amoniating solutions. Two of the ammoniating solutions are ammonia-ammonium nitrate solutions, 414 (17-74-0) and 530 (49-36-0), selected on the basis of the maximum and minimum contents of ammonium nitrate usually present in this type of solution. The other two are ammonia-ammonium nitrate-urea solutions, 430 (20-68-6) and 440 (28-40-15), representing the maximum and minimum contents of urea, usually present in this type of solution (*1, 12*).

Table I. Relative Volumes of Aqueous Solutions Saturated with Urea and Ammonium Nitrate in Varying Proportions at 30° C. (86° F.)

Component	Proportion of Dissolved Salt										
Urea	0	10	20	30	40	46	50	60	70	80	100
Ammonium Nitrate	100	90	80	70	60	54	50	40	30	20	0
	Pounds per 100 pounds of water										
$CO(NH_2)_2$	0	29	74	164	375	719	482	266	204	172	133
NH_4NO_3	242	259	294	381	562	845	482	178	87	43	0
Total salt	242	288	368	545	937	1564	964	444	291	215	133
	Relative vol. of solution per unit vol. of water										
Solution volume	2.5	2.9	3.5	4.7	7.6	12.1	7.9	4.2	3.1	2.6	2.0

The first five items in Table II may be found in, or easily calculated from, the data usually supplied by manufacturers of ammoniating solutions. In Item 6, use is made of the data given in Table I showing the relative volumes of aqueous solutions saturated with urea and (or) ammonium nitrate in varying proportions at 30° C. (86° F.). For example, from the data in Table I it may be observed that 242 pounds of ammonium nitrate will be dissolved by 100 pounds of water to form a saturated solution at 86° F., thus 36 pounds of ammonium nitrate (Item 5) incorporated in a fertilizer mixture by introducing one unit of nitrogen from Solution 414 (17-74-0) will dissolve in 15 pounds of water (Item 6) or 1.78 gallons (Item 7) to form 51 pounds of a saturated liquid phase (Item 8) weighing 11.3 pounds per gallon, or 4.5 gallons of liquid phase (Item 9) in the fertilizer. Likewise the solubility data in Table I show by interpolation that 492 pounds of a salt mixture having a urea/ammonium nitrate ratio of 27/73, such as that in Solution 440 (28-40-15), will dissolve in 100 pounds of water. Thus, 25 pounds of this salt mixture incorporated in the fertilizer by introducing one unit of nitrogen from Solution 440 (28-40-15) (Item 5) will dissolve in 5 pounds of water (Item 6) or 0.61 gallons (Item 7) to form 30 pounds of a saturated liquid phase (Item 8) weighing 11.3 pounds per gallon, or 2.7 gallons of liquid phase (Item 9). Dividing Item 9 by Item 7 gives the relative volume of liquid phase present in the mixture per unit volume of water present.

Table II shows that ammonia-ammonium nitrate-urea solutions

Table II. Liquid Phase Imparted to Mixed Fertilizer by Ammoniating Solutions

Item		Ammonia-ammonium Nitrate Solution No.		Nitrate-Urea Solution No.	
No.	Description	414	530	430	440
1	NH_3-NH_4NO_3-Urea, %	19-74-0	49-36-0	20-68-6	28-40-15
2	Nitrogen content, %	41.4	53.0	43.0	44.0
3	Ratio urea/NH_4NO_3, %	0/100	0/00	8/92	27/73
4	Total salt content, %	74	36	74	55
5	Salt content/unit N, lbs.	36	14	34	25
6	Water content of mixture required to form a saturated soln. at 86° F., lbs./unit N	15	6	12	5
7	gal./unit N[1]	1.78	0.67	1.48	0.61
8	Total wt .of liquid phase, lbs./unit N.	51	20	46	30
9	Volume of liquid phase, gal.[2]/unit N[1]	4.5	1.7	4.1	2.7
10	Gal. liquid phase/gal. water[3]	2.5	2.5	2.8	4.5

[1] Water, 8.35 lbs./gallon.
[2] Liquid phase, 11.3 lbs./gallon.
[3] 1 gallon water equiv. to 0.417% in mixed fertilizer.

provide from 12 to 80% more liquid phase for equal amounts of nitrogen than the ammonia-ammonium nitrate solutions. It is not surprising that some products formulated with Solution 440 (28-40-15) have undersirable properties as compared with those formulated with the other three solutions shown in Table II. Such products may be wet and sticky on emergence from processing. They may be difficult to dry artificially and they may not become dry and friable during the curing stage. Data on the relative volume of liquid phase imparted to mixed fertilizer by the salt contact of ammoniating solutions would seem to be a useful addition to the instructions to plant operators on formulating with solutions.

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CHAIRMAN SAUCHELLI: Thank you, John. That certainly was a most interesting analysis of the situation. We have a few minutes for some discussion. Now, remember the essential part of these sessions is the discussion period and the free, frank commenting from the audience. Let's have some questions now for Mr. Hardesty.

MR. PHILIP STONE (Virginia-Carolina Chemical): I'd like to ask Mr. Hardesty about this particular point with regard to the urea in solutions. It seems to me that I have noticed that when urea is in a solution and you granulate, that the granules formed are much rounder and more spherical than products made with solutions containing ammonia and ammonium nitrate. Could you comment on this and possibly give us some reasons as to why this would be?

MR. HARDESTY: We've noted that, too, and we've heard others say that urea mixtures have less oversize than equivalent mixtures with ammonium nitrate, the particles are rounder. We haven't been able to see why that would be, except we could make some guesses as to why it would be. Crystallization, of course. If you get rapid crystallization of the solution as you do with ammonium nitrate, more material in solution, it comes out faster and you have a tendency to get popcorn type of granules when you have material like that.

Urea coming more slowly out of solution, with perhaps a finer crystal, I don't know, you would be apt to get a round, more rounded, spherical granule.

Now as far as the particle size business is concerned I think that's probably just a matter of how much moisture you have present. According to all the fundamental principles of granulation, it's merely a coming together of small particles by surface tension in the liquid pulling the particles together and if you have small particles you'd get a more rounded granule than if you have very large particles that don't mesh into each other. That would be the only suggestion that I would have and that isn't an accurate answer, I know.

CHAIRMAN SAUCHELLI: That was a starter. Who else has something they would like to have discussed? Or maybe somebody else has some ideas about the roundness of these particles due to the influence of urea. .

MR. ELGIN DOIDGE (Canadian Industries): I'd like to ask Mr. Hardesty whether this data applies to the solutions themselves or does it apply to the mixture after you have added the solutions in the ammoniator? In other words, how do you determine the volume of liquid phase that you have in a rolling bed in an ammoniator, for example?

MR. HARDESTY: I'm sorry; I should have said that and the table does show that this moisture comes from the mixture. It has nothing to do with the moisture in the solution phase. Now you can tackle this thing from another direction: How much moisture do you put into the mixture from what you have in the ammoniating solution itself? Some ammoniating solutions have far less water than others, but this moisture that we're talking about comes from the fertilizer, whether it's from the original moisture content of the superphosphate or whether it's from added moisture, or whether it comes from solution doesn't matter. If it's there, and we're talking about conditions in which you get a saturated solution of ammonium nitrate and/or urea, I should say, in a mixture, that is, the solution is saturated when you have a certain amount of water present, and you get that amount of water present when you start to dry the mixture or when you add enough salt to the amount of water which is there to make a saturated solution.

We're always dealing with a a saturated solution, and that's all we're doing. We're not trying to put this over into practical operation in this paper we gave this morning. That is something I think will come later. For every mixture, you see, it would be different, for every solution that you use.

Chairman Sauchelli: That was a good question. We have time for one or two more, now. Who has the next comment?

A Question: Mr. Hardesty, is there actually much ammonium nitrate in there? Doesn't it react largely with the urea present and remain in the form of potassium nitrate?

Mr. Hardesty: Was the question: does ammonium nitrate react with other materials in the mixture?

Same Questioner: Yes, I've been told that it does react with muriatic potash to give ammonium nitrate and ammonium chloride to get this material, therefore would there be much ammonium nitrate present in the liquid phase of fertilizers?

Mr. Hardesty: That's something else to cogitate on. I haven't seen a good measurement of how much ammonium nitrate is taken out of the mixture by the metathesis reaction that you get with potassium chloride, but we know that it happens. It must happen in solution. You get chemical reaction. Now, when you put these solutions in a mixture, supposing that you have this 4-40 solution with the 15 of urea and 40 of ammonium nitrate, you use any amount of that solution and you get one that is ammoniating up to 2, 3, or 4 pounds of ammonia per unit of P_2O_5, you get ammonium nitrates far in excess of the urea.

Now, supposing that some of these metathesis reactions contain ammonium nitrate and urea and start to occur and they take the ammonium nitrate out, the ammonium nitrate keeps coming down, there are other things that influence the amount of ammonium nitrate in the solution, too, the other salts that are present would cause it to be less soluble. You start coming down with your ammonium nitrate and your urea remains about the same, because it doesn't react with other materials. You hit this eutectic point, because you're taking ammonium nitrate out and leaving so much in solution; this explains why we look at a pile one evening and it looks all right, and come back the next morning and it looks wet all the way through. How many have noticed that? I don't know whether that's just a peculiarity of mine. How many have noticed that?

(A showing of a large number of hands.)

A good many hands. It isn't due to hygroscopicity. It's due to the fact that you hit a point there at which more salts come into solution. Does that help to explain?

Chairman Sauchelli: You see what I mean? You get a few questions and comments from the floor and you get a lot of supplementary information there that helps everyone. We have time for one more. Who has the next question or would like to comment.

Mr. Dudley George (Richmond Guano): When we calculate moisture on pulverized formulas from ingredients and then get the lab analysis the moisture always seems to run right much more than what we figured. Could Mr. Hardesty comment on that?

Mr. Hardesty: This is on ammoniated goods?

Mr. George: Yes.

Mr. Hardesty: In superphosphate you have monocalcium phosphate which has what we call one molecule of hydrated water and as soon as you ammoniate that water comes off, it becomes vapor. If you determine the water on the superphosphate, it doesn't show up in your free water content; when you determine it on the mixture, that free water will show up. We notice that right along. That's the only thing that I could point to that would cause that and give you a higher moisture content in your final product, other than some moisture absorption, of course.

Chairman Sauchelli: Thank you, John. We certainly appreciate your contribution, and the fine response from our audience.

We'll have to move on, but please remember that with the next speakers they are ready, willing and anxious to answer questions. They can't possibly take in all phases when they prepare their talks and the advantage of having questions from the floor is that it enlarges the discussion and makes it a more comprehensive contribution.

I now turn the meeting over to Mr. Reynolds who will take care of the next item.

(Mr. J. E. Reynolds, Jr. assumed the Chair.)

Chairman Reynolds: Urea ammonia and urea ammonium nitrate solutions have been discussed in some detail, particularly as related to granulation formulas. The whys of granule formation, particle softness, influence on liquid phase and relative hygroscopicity of the products still exist indicating the need for defining urea limitations. This panel discussion which is coming up will review the behavior of urea in pulverized mixed fertilizers as an aid to partially explain the behavior in more complicated mixed fertilizer formulas.

We have just heard an excellent presentation of the overall data relating to the causes and alleviation of poor physical conditions in mixed fertilizers. We now wish to proceed to further specifics relating to the presence of the urea in pulverized mixed fertilizers. We are indeed fortunate and pleased to have as our speakers Mr. Bert Tucker, Sohio Chemical, Mr. George Gilliam, Nitrogen Division, to discuss the use of urea-ammonia-ammonium nitrate solutions, and Mr. Bill Lewis of duPont to discuss the use of urea-ammonia solutions.

Our first speaker is Mr. Bert Tucker. We will ask each speaker to give his review and then we will hold questions for the end.

Urea-ammonium Nitrate Solutions May Improve Mechanical Conditions of Conventional Fertilizers

H. H. Tucker

Mr. Hardesty has given a very thorough and scholarly presentation on the subject of solutions containing both urea and ammonium nitrate and the mutual solubility of these two products. He has further analyzed how the mutual solubility of these two salts will affect the solution phase of fertilizer mixtures.

In fact almost anything which I could add would be to quite an extent repetitious. Repetition, however, may be good in that it can add emphasis. This may justify my proceeding with my few remarks.

First let me say that the use of four component, ammonia-ammonium nitrate-urea-water solutions has increased very rapidly during the past few years. This group of nitrogen solutions now makes up a sizeable part of all ammoniating solutions used in fertilizer manufacture. Some of the reasons for their acceptance and use of urea in ammonia-ammonium nitrate solutions are:

1 Lowers saturation temperature, salt out temperature and vapor pressure of the solution.
2 Makes practical the transportation, storage and use of higher fixed-to-free ratio nitrogen solutions . . . with a higher ratio of salts to ammonia.
3 Increases the amount of nitrogen which can be obtained from solutions.
4 Reduces the amount of acid required to neutralize reduced amounts of free ammonia and consequently prevents excessive heat.
5 Increases the volume of the solution phase, and, therefore, the agglomeration of fertilizers.
6 Alters the crystal structure of ammonium chloride.
7 Softens the end product or decreases the hardness of bin or bag set.

Some of these points might be enlarged upon to good advantage. First is the change in the crystal structure of ammonium chloride. Ammonium chloride though not added as a material, results from the reaction of ammonium nitrate and potassium chloride.

Ammonium chloride may form as either a branched fern-shaped crystal or as a cube crystal. The use of urea in fertilizers containing ammonium chloride favors the formation of the cube type crystals. It is easy to see how this change could be most beneficial in reducing the knitting together of fertilizer particles, particularly in the non-granular or conventional types. How much urea is required to bring about this change in crystal structure?

Research has indicated that at least 20 pounds of urea per ton of end product is required to thoroughly permeate the mixture and accomplish the desired result. Larger amounts may be used. However, other factors may set an upper limit on the amount of urea to use.

The tendency to soften the end product of course may be over done. This softening effect is at least partially brought about by the increase in the solution phase due to mutual solubility effect of urea and other fertilizer salts.

What then is the upper limit on the amount of urea which can be used with ammonium nitrate? This will depend on a number of factors including grade of fertilizer, moisture content, type and length of storage and atmospheric conditions, particularly temperature and humidity. A general rule of thumb is that 50 pounds of urea per ton of end product is the upper limit when used in conjunction with ammonium nitrate. The amount of solution to be used should then supply between 20 to 50 pounds of urea. An even narrower range of 30 to 40 pounds may be preferable. The common four component ammoniating solutions contain from 6 to 15 percent urea with 68 to 40 percent ammonium nitrate and also have a rather wide range of ammonia to salts. This permits the selection of a solution to fit specific requirements of fertilizer grade and desired formulation.

Solutions High in Urea and Low in Ammonium Nitrate

Fertilizer manufacturers looking for better materials and processes sometimes ask "will a small amount of ammonium nitrate increase the fixed-to-free ratio and lower the salt-out temperature of urea ammonia solutions?"

Many asking this question have used urea ammonia solutions successfully for the lower nitrogen grades of fertilizer. They now wish to be competitive in their sales and produce the higher nitrogen grades such as a 1-1-1, a 2-1-1 or perhaps even a 3-1-1 ratio. Of course they want to be competitive cost-wise and to use large amounts of solution and low amounts of acid. High fixed-to-free ratio solutions of this type can be made but not with the low water content and high total nitrogen content which they would also like to have in their ideal "nitrogen solution."

This is of course due to the fact that urea is not as soluble in aqua ammonia (ammonia and water) as is ammonium nitrate. This can be readily seen by study of almost any solution manufacturers' product card. In other words, the ammonium nitrate ammonia

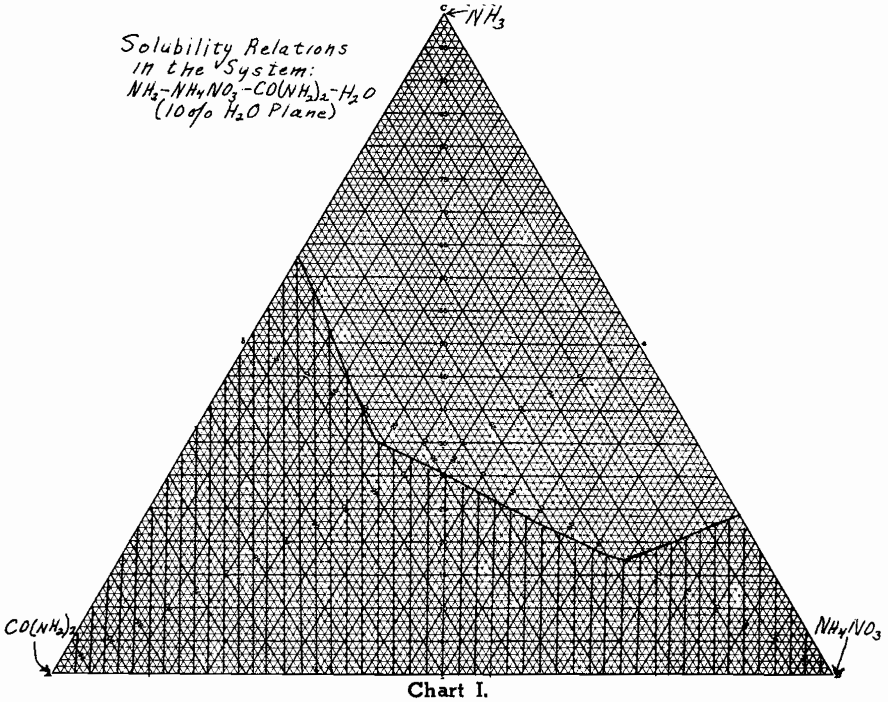

Chart I.

solutions already have a "head start" over the urea ammonia solutions as far as salt concentration is concerned. To expect a small amount of ammonium nitrate to overcome this solubility handicap and more too is a rather large order. Chart 1. showing at 32° F. solubility isotherm for a urea-ammonium nitrate-ammonia-water solution at the 10 percent water level will illustrate this point. The shaded area shows the region which would be supersaturated or contain salts while formulations in the unshaded area would be in solution at 32°F. This general pattern holds true with other water content solutions down to 6 percent or even to an anhydrous solution. We note here that the area of insolubility is much greater in the urea corner than in the ammonium nitrate corner and remains so even at higher contents.

The question of anhydrous solutions has been brought up. Such solutions are possible, but let me remind you that vapor pressure of the ordinary solutions is largely a function of the aqua ammonia component, that is, the ammonia-water fraction of the solution. Anhydrous solutions containing no water then would, like anhydrous ammonia, have high vapor pressures. Anhydrous solutions would be very vulnerable to even slight changes in ammonia content of the solution. Flashing of ammonia due to changes in pressure differentials could easily cause salt out problems of anhydrous solutions.

The nitrogen industry working with the mixed fertilizer industry has carried on extensive research on nitrogen solutions and has made great progress. In some cases even special nitrogen solutions have been formulated to fit individual needs. Any ideas you may have for further improvements are always welcome.

CHAIRMAN REYNOLDS: Thank you very much, Bert. I know there will be some questions, but we're going to hold those until the last speaker has finished.

The next panelist to discuss the urea-ammonia-ammonium nitrate solutions, is George Gilliam, Nitrogen Division.

Urea-Ammonium Nitrate-Ammonia-Water Solutions in Pulverized Fertilizers

George R. Gilliam

TWO questions have been submitted on use of urea-ammonium nitrate-ammonia-water solutions in pulverized fertilizers.

First — "Why use this solution"? and second — "What is the relative hygroscopicity of mixed fertilizers made from this solution as compared to the same mixed fertilizer produced from ammonium nitrate-ammonia-water solution?"

For purposes of this discussion the term pulverized fertilizers will be confined to those fertilizers which are proceesed on a once through basis with no mechanical drying under conditions of moisture and temperature that result in little or no agglomeration. In general little or no acid is used in formulating the materials. Nitrogen solution is used to the extent of the capacity of the superphosphates to react with the free ammonia, and ammonium sulphate is usually used to provide any additional desired nitrogen.

As to the first question, there are a number of reasons which favor use of this solution by the fertilizer mixer. First are the physical characteristics of the urea-ammonium nitrate-ammonia solutions which allow the mixer more flexibility in formulation. For example, lower free ammonia for a given total nitrogen is possible without increasing the salt-out temperature. Also, for the same free ammonia and total nitrogen, a solution of much lower salting out temperature can be obtained when urea is included. This can be a major advantage to mixers faced with winter temperatures and no heat.

Experience in the industry has shown in many cases that use of a limited amount of urea in some pulverized grades resulted in improved condition of the product. The urea-ammonium nitrate-ammonia-water solution is a means available to the mixer for including urea in his formulation without the increased burden of storing and processing an additional raw material.

It has already been stated that greater soubility of total nitrogen is possible in the urea-ammonium nitrate-water solution as compared to the ammoniating solution without urea but having the same free ammonia content. It is also true that after the free ammonia in comparable solutions has been reacted in the mixer, the remaining solution has an appreciably lower salting out temperature when it consists of urea-ammonium nitrate-water as compared to ammonium nitrate-water. To illustrate, a solution containing 34% free ammonia, 60% ammonium nitrate and 49% total nitrogen would salt out at 212 °F after the free ammonia is removed. A solution of the same total nitrogen but composed of 33% free ammonia, 45.1% ammonium nitrate and 13% urea would salt out at 117 °F after removal of the free ammonia. This property of increased solubility offers the possible advantage of more uniform distribution of the nitrogen on the other fertilizer solids, particularly where mixer temperatures are low.

It was mentioned earlier that field experience indicated more favorable product condition for some grades when urea was included in the formulation. Several theories have been advanced to account for this observed improvement. One of these is the effect of urea on the crystallization of ammonium chloride. The function of the urea is to suppress the tendency of ammonium chloride to form a network of interlocking needle-like crystals which are thought to promote caking. Another theory which also has merit is that the decreased vapor pressure of the goods made containing urea-ammonium nitrate reduces the degree of drying out in storage. This results in fewer crystal bridges being formed between particles thus making a softer material.

In regard to the second question: J. Richard Adams and Albert R. Merz published an article in Industrial and Engineering Chemistry, April 1929, entitled "Hygroscopicity of Fertilizer Materials and Mixtures." The data in this article show mixtures of fertilizer salts with urea to be more hygroscopic than the salts alone. Our experience indicates in general that fertilizers made with the urea-ammonium nitrate-ammonia-water solution are more hygroscopic than the same fertilizer made with ammonia-ammonium nitrate-water solution. However, this is not necessarily bad since, in many cases of pulverized grades, increased hygroscopicity has shown definite advantages.

CHAIRMAN REYNOLDS: Thank you very much, George.

The next speaker, Mr. Bill Lewis of duPont, will discuss the influence of urea in urea-ammonia solutions.

The Use of Uramon Ammonia Liquors in Conventional Fertilizers

James W. Lewis

IN order to avoid any possibility of confusion, we should define at the outset what is meant by the term "urea solutions" as used in this presentation. When used here, the term refers to a fertilizer ammoniating solution containing urea, ammonia, water and ammonium carbamate; there is no ammonium nitrate present.

"Uramon" ammonia liquors, hereafter called UAL solutions, are available in several different compositions designed to meet a wide range of requirements in the fertilizer industry. The history of their use in the manufactur of conventional fertilizers dates back to the early 1930's, and much of the first research on ammoniation of superphosphates was done with the use of these solutions.

The general topic for this portion of the Round Table is "Problems of Conventional Fertilizers," particularly in regard to mechanical condition. However, it is felt that a brief review of the basic principles of ammoniation should offer a sound starting point. These principles, in general, will apply regardless of the type of solution being used. In all probability, this will be "old hat" to everyone present; but it seems obvious that in the desire to raise nutrient ratios, reduce formula costs, and meet production schedules, these principles are frequently ignored. This often results in reverted phosphates and poor condition.

Superphosphates and triple superphosphate are essentially acidic salts of calcium phosphate with small amounts of free acids, moisture, and in the case of run-of-pile superphosphate, about half of the material is calcium sulfate (gypsum). A series of progressive chemical reactions is started when ammonia, either as anhydrous or free ammonia in ammoniating solutions, is added to superphosphates. The extent and nature of these reactions depends primarily upon:

1) the amount of free ammonia added,
2) temperature and storage time of the mixture, and
3) the moisture content of the mixture.

Since these chemical reactions significantly improve the physical condition of the superphosphates and mixed goods, it is therefore desirable to add the maximum amount of ammonia insofar as condition is concerned. However, beyond certain limits the ammoniation reactions produce basic calcium phosphates that are unavailable, i.e., P_2O_5 reversion occurs, even under the best storage conditions.

The combination of ammonia with acids or with calcium phosphates is a strongly exothermic reaction, i.e., heat is generated. Although high temperatures are helpful during the mixing cycle, removal of this heat is necessary before storage, if phosphate reversion and caking are to be minimized. It should also be recognized that aside from the ammoniation rate and temperature, reversion is very much dependent upon the P_2O_5 content of the mixed goods. As the P_2O_5 content is moved higher and as an effort is made to obtain all the nitrogen from solutions, this point may have been overlooked. The following table illustrates this point. These values presuppose that the mixture will be cooled before going into the pile.

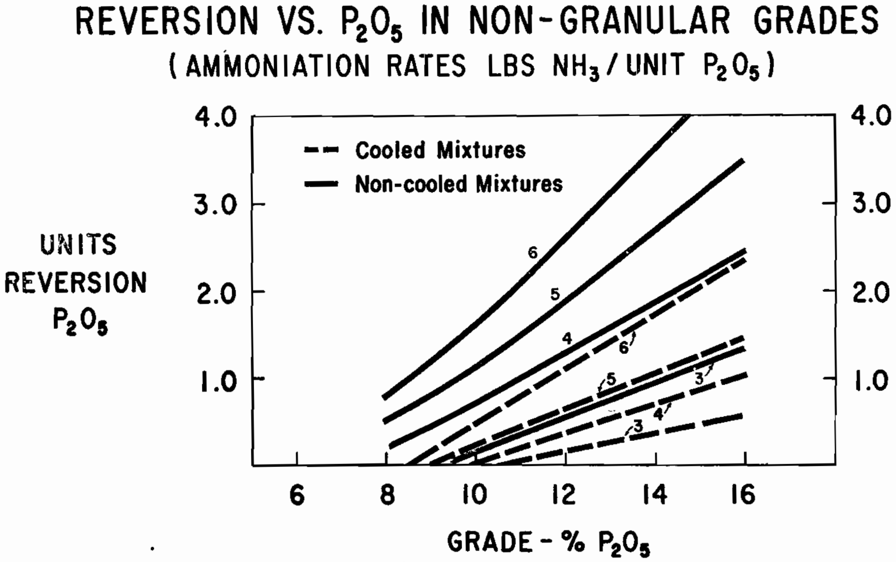

Figure I

This situation results entirely from the fact that in a constant size sample (1 gm.) used for the citrate solubility test during the analysis, the ratio of the actual amount of P_2O_5 present to citrate solvent increases as the grade goes up. Obviously, there is twice as much P_2O_5 per unit volume of citrate in a 16 per cent goods as in an 8 per cent mixture. This means that less of the lesser soluble P_2O_5 is dissolved and the insoluble comes out higher.

The graphs (Fig. 1) are "road maps" indicating the best and worst that can be called typical for ammoniation of normal superphosphate, and the results are independent of the free ammonia source.. These data are not applicable to triple superphosphate. The graph labeled "not cooled" means long storage (several months) of goods going into the pile around 130 degrees Fahrenheit or higher and gradually cooling down so that most of the storage life averages 110 degrees Fahrenheit or above. The graph labeled "cooled" means cooling to 110 degrees Fahrenheit or below within 24 to 48 hours after ammoniation. Such cooled goods will not remain at low reversion levels forever, but will approximate the graph lines up to four or five months. Old goods carried over a full season can show reversion even if cooled immediately after manufacture.

Consequently, one of the most important processing steps is the removal of the heat generated by the ammoniation reaction. By removing the heat, the amount of free ammonia added per unit of P_2O_5 can be increased significantly before reversion is encountered. The importance of this temperature effect on conventional goods cannot be over-emphasized. For example, a 10 per cent P_2O_5 grade, ammoniated at the rate of 4.5 lbs. free ammonia per unit P_2O_5 and stored for several weeks without cooling, might show as much as 1.0 unit P_2O_5 reversion, whereas the same mixture cooled below 100 degrees Fahrenheit soon after ammoniation would show little, if any, loss of available P_2O_5. In general, for normally ammoniated conventional mixtures, 110 degrees Fahrenheit seems to be a dividing point, below which reversion reactions are greatly retarded and above which they increase in speed and extent in proportion to the storage temperature.

Up to this time, we have mentioned the moisture factor only in passing. Much more time than is available here could be spent in discussing this single point, for moisture is the "highway" over which most of these complex chemical reactions travel. The soluble salts which are largely responsible for the condition of the goods also move back and forth over this road. This shifting of the conditioning reactions in the free water present will determine the condition of the fertilizer at any given time.

This leads to another point concerning the analytical methods for moisture content. We believe that the majority of the industry uses the AOAC "Total Water Method" for moisture analysis. This method prescribes placing the sample in a constant temperature oven at 100 degrees Centigrade for five hours. When this method is used for goods containing urea, the result will invariably be erroneously high. Under such strenuous conditions, part of the urea will hydrolyze and this loss will be measured as moisture. For those using one of the portable devices with a heat lamp, the same situation exists, but usually to a lesser extent. The preferred method of moisture analysis for goods containing urea is the AOAC "Free Water Method"; or, if a moisture balance is being used, voltage on the lamp should be controlled by a rheostat set at 70V and the sample heated for 20 minutes. The result will closely approximate the free water method.

Now, let's look more specifically at UAL solutions in conventional goods. When these solutions are used for ammoniation, only the free ammonia and ammonium carbamate enter into the reactions. The free ammonia behaves in the usual manner while the ammonium carbamate immediately decomposes into NH_3 and CO_2 with the ammonia being absorbed by the phosphates and the CO_2 liberated to the atmosphere. The carbamate decomposition absorbs heat from the mixer contents, reducing its temperature and the CO_2 liberated tends to aerate the goods and sweep out moisture. Experience has shown the exit mixer temperature for a 5-10-10 grade to be 10 degrees Fahrenheit to 15 degrees Fahrenheit lower with UAL than with other solutions when used at equivalent ammoniation rates. The temperature obtained in the mixer is high enough to promote the desirable reactions without becoming excessive.

The urea in UAL remains unreactive and simply becomes evenly dispersed throughout the fertilizer mixture. Since urea is an organic compound, it does not undergo the double decomposition reactions associated with inorganic salts. However, under periods of long storage (several weeks) at elevated temperatures (around 140 degrees-150 degrees F.) and in the presence of moisture urea slowly breaks down (hydrolyzes) into ammonia and carbon dioxide. It is appropriate to note here that hydrolysis does not occur to any significant extent during the granulation process even though temperatures are usually high during the granulating and drying steps. In this process, the time of exposure to these conditions before cooling is not long enough to cause more than three to four per cent hydrolysis.

In conventional goods, temperatures below 120 degrees Fahrenheit are recommended for storage periods longer than two to three weeks. If urea hydrolysis does occur, there is no loss of nitrogen from the goods. Any ammonia so released is absorbed by the super phosphate just as if it were free ammonia in the first place. The only real problem arising from urea hydrolysis would be an increased tendency toward reversion if the super had been heavily ammoniated previously. It has also been established that no biuret is formed from urea hydrolysis in mixed fertilizers, even under the severest granulating operations.

The following table shows the recommended rates of usage for three typical UAL solutions at various P_2O_5 levels. These values presuppose that the mixture will be cooled before going into the pile.

Typical formulas for 5-10-10 and 4-12-12 grades which are to be given several weeks of curing time follow.

At times, it may be necessary to deviate from the principles previously discussed in order to meet an unusual set of circumstances. For example, you might find it expedient to manufacture, bag and ship goods before it has undergone the usual curing period.

During the past summer, we had occasion to encounter such a problem. The grade involved was conventional 5-10-10 made from

P_2O_5 Grade	Lbs. NH_3/Unit P_2O_5	Approximate Pounds of Solution		
		UAL-A	UAL-B	UAL-S
8	6.0	130	160	180
10	5.0	135	165	185
12	4.5	145	175	200
16	3.5	150	180	205

(Note: If mixtures cannot be cooled for extended storage, ammoniation rates should be decreased by 25 per cent from those above to minimize P_2O_5 reversion.)

	5-10-10	4-12-12
UAL-S	4 units N	4 units N
Ammonium Sulfate	1 unit N	----
Super	10 units	12 units
Potash	10 units	12 units
Filler	To balance	To balance

UAL-S. The material was bagged from the bin three days after manufacture and stored 15 bags high for another three days. The stacks were then torn down and the bottom bags examined. These bags had a very soft form set which disintegrated completely with one drop. Other bags from the bottom were simply opened and dumped into a fertilizer spreader in a normal manner. Normal handling was sufficient to completely break up the form set.

The spreader was then operated at 380 pounds and 190 pounds per acre with no stoppage or bridging.

In summary, the starting point in formulating conventional complete fertilizer mixtures is the selection of the amount of free ammonia to be used per unit P_2O_5. This depends upon:

(1) the grade,
(2) past experience with the particular superphosphates,
(3) use of a cooler,
(4) expected storage time, and
(5) what other formula ingredients are involved, especially those of a strongly alkaline nature (cyanamid, active limestone).

The rates of ammoniation recommended are three pounds free ammonia per unit P_2O_5 from triple and five pounds ammonia per unit for ordinary super. The lower limit of ammoniation is largely a question of physical condition while the upper limit is much less definable. As the rate of ammoniation is increased beyond four pounds NH_3/unit P_2O_5, consideration must be given to phosphate reversion, absorption capacity of the particular superphosphate, mechanical equipment, grade of mixture and other factors. Rates beyond six pounds/unit for ordinary super should be considered as specific plant developments. Each manufacturer should determine the feasibility of such operations and he should plan on using a cooler following ammoniation.

Chairman Reynolds: Thank you very much, Bill. You've heard four excellent presentations here relating to urea in various combinations, solution in relation to pulverized mixed fertilizers as well as granulated mixed fertilizers. We would like to turn the meeting over to the floor, because I am sure there are many questions out there that these people can help us with. Who has the first question?

Voice: Is there any standard rule of thumb on the maximum soluble salts content when making powder mixes with the four component solutions. I believe in the old days you'd say, well, not over, say, three units of ammonium nitrate. Is there sort of a comparable limit with the four components solution?

Chairman Reynolds: Are you directing this toward anyone in particular?

Voice: No.

Chairman Reynolds: George? Bert?

Mr. Gilliam: I can only comment.—I don't know that I can answer your question directly. I think in the case of pulverized grades your limit of maximum soluble salts is going to be the amount that you can tolerate in the form of liquid phase and not have stickiness. Do I make my point there?

In pulverized grades you can stand a certain amount of liquid phase without having the goods sticky. If you increase that liquid phase either by adding more urea or more soluble salts then you have exceeded the limit to which you can add it because of the poor condition of your goods.

Normally, I would say the liquid phase that you can tolerate and have satisfactory condition would be on the order of 15 per cent. That would be moisture plus soluble salts in solution.

Chairman Reynolds: Bert, would you care to add anything to that?

Mr. Tucker: No.

Chairman Reynolds: Mr. Hardesty, would you care to add anything to that?

Mr. Hardesty: No.

A Question: Mr. Tucker has been talking about 20, 30 and 40 pounds of urea per ton of fertilizer. I think that if you use a six per cent urea solution you wouldn't want to go over—let's see, six goes into fifty eight times—you wouldn't want to go over eight hundred pounds on a six per cent combination solution.

From that angle, that is if you use the basic theory that you don't want over 20, or 30, or 40 pounds of urea when you have ammonium nitrate, so that you normally would never hit the 20 or 30 pound range because you won't be able to put that much in solution period.

Chairman Reynolds: Bert, any comment?

Mr. Tucker: The only comment I would have is that with some of the urea-ammonium nitrate solutions that contain 11 to 15 per cent urea you might not be able to get all of the solution nitrogen that you wanted because you could run over this 40 to 50 pound limit. I believe some people have — maybe you've experienced that, Joe.

Chairman Reynolds: Yes.

Mr. Tucker: For instance, if you're using 750 pounds of a 40 per cent nitrogen material to give 15 units of nitrogen and you were using a solution with 11 per cent of urea you would then have better than 75 or 80 pounds of urea which would be too much and that is the reason why these solutions with only 6 per cent of urea in the high salt, high fixed-to-free ratio solutions, have been developed.

Chairman Reynolds: I think we discussed some of those features last year and followed through from the no urea solutions, urea-ammonium nitrate-ammonia up to the 11 per cent. I don't think anyone was brave enough to try the 15 per cent urea solution that Mr. Hardesty mentioned and then following back down to the 6 per cent urea which seems to be the one that is widely accepted.

You are right as far as the adverse difficulties with the 11 per cent urea when you try to go above the 50 pounds which you could get into with a 10 per cent grade, or 12 per cent nitrogen grade.

Let's have some more questions or comments.

A Question: I'd like to direct this question to Mr. Tucker. Some of us old fashioned boys don't put a solution in a mix. Now to get that 20 pounds of urea do you advocate adding solid urea into your mixture to give you the 40 pound unit per ton and give you a better condition or not?

Mr. Tucker: No, I would not be advocating the addition of urea to a mixture to go up to that 40 pound or 50 pound limit; that is the ceiling, so the range—if you are trying to get the advantage of the change in crystal structure of ammonium chloride, it will take the 20 pound minimum, so you'll probably want to select a solution that will give you at least 20 pounds, but not over 50 pounds. I wouldn't advocate using additional urea to go up always to the ceiling.

Do you have any comments on that, George?

Mr. Gilliam: Very good.

Chairman Reynold: Would anyone from the floor care to comment on the addition of solid urea to the mixed fertilizer formulation, particularly on granulation? (No response.)

I know it has been tried out there. There are some people who are just holding back.

Al, are you holding your hand up back there?

Mr. Al Henderson (Wilson and Toomer): Joe, we've used a little bit of the solid urea in a two unit and it was the most peculiar grade. It was 6-13-17 sulphate potash and that's a hard one to granulate; you have to use a lot of water, two units of anhydrous, two units of ammonium nitrate and two units of solid urea, and if you want to talk about eutectics, I think you get them there.

Chairman Reynolds: We've had exotic mixtures and romantic mixtures and now we have peculiar mixtures.

Mr. Richard Powell (I.M.C.C.): Back before they developed the urea solutions or at least put them out as a popular grade in Iowa, particularly, very frequently we used to put in four and a half units of solution for solution D and then call it 4-9-D to help the urea dry considerably and reducing the amount of caking we had in the bag. You have to cool it, though, one way or the other.

Chairman Reynolds: Any other comments or questions?

Mr. Charles Lubow (Star Fertilizer Company): I'd like to direct my question to Mr. Lewis. Do you think the weather is a factor in deciding your formulation?

Mr. Lewis: Yes, I do. As a matter of fact, that was taken into consideration in arriving at the formula which we used to make this 5-10-10 which we showed you in the pictures a while ago and the reason I didn't go into any further detail as to how we did that was simply because we had violated virtually all principles that I had previously been talking about.

That may not be of real help to you, but that's probably as good as I can do at the present time.

Chairman Reynolds: Another question in the back.

Mr. Al Henderson: I'd like to ask George Gilliam a question. I probably misunderstood but I thought that his closing statement was that it was desirable in some cases to have mixtures that were hygroscopic; am I wrong about that?

Mr. Gilliam: That's what I said, Al.

That's the reason we use this solution of urea-ammonium nitrate-ammonia-water, to make our goods more hygroscopic. All the data that I have seen indicate that that's what you do.

It means that you cannot improve condition for every formulation by using this solution. For example, I think in a 1-1-1 ratio you might get into right serious difficulty by using it, but possibly in a 6-12-12 with this solution and either all or part of your nitrogen from it as compared to an ammonia nitrate—ammonia water solution you can improve your condition in storage in that you have less salting out and you have a material with a lower vapor pressure which does not dry out as much, and as Mr. Hardesty said, caking is a function of interlocking of crystals or, you might say, salting out of your salts.

If you put in an "anti-freeze," so to speak, and avoid this salting out then you do not have solids forming in storage, and thus you have better condition.

Now, if you get too much anti-freeze in and get on the wet side, you've got a muddy material which is just as bad as having a caked material. It's in between where you get your advantage.

Chairman Reynolds: Any more questions or comments?

I think George's illustration there of what happens to the salts in the ammoniator once the ammonia, the free ammonia, has been taken out was very graphic. We are also talking about this moisture problem. In yesterday's discussion on mechanics of formulation we were all around this problem of moisture and free water, total water, water of crystallization. We'll just also mention this again as some of the problems that determine a realistic moisture value.

Mr. Hardesty mentioned here the influence of moisture on caking. How can we determine where we're going if we can't determine the realistic moisture value?

Mr. Gilliam: I want to clear up one point. All my remarks have been concerned with pulverized grades, which I think of as having moisture in the range of not less than two per cent and probably not to exceed seven per cent; that's built into your pulverized grade in manufacture.

Now, if we turn around and begin to think in granular grades where we have mechanical drying and cooling, it means changes in liquid phase other than just from the solubilities and the moisture in formulation; that's another story.

CHAIRMAN REYNOLDS: Thank you. Our original intention with regard to the panel was to develop it more in the direction of granulation, granulated products, but we soon found that there was too much information lacking to really put together a discussion, so we do have questions. Many of you were very cooperative in sending questions in on this subject. This group before us here has those questions and we're going to try to do something about them. Maybe we can include the subject on next year's program, because the whys of the behavior of these various solutions is still unanswered. We're going by empirical reasoning and by feel; it would sure be helpful if we had some more definite approaches to this.

Let's have some more questions or comments.

MR. ROBERT HENNESSY (New York): Comment. We've had considerable experience with urea-ammonium nitrate and have used it very successfully and made several thousand tons of 5-10-10 with urea-ammonium nitrate solution and it gave us a very sharp, free-flowing material. We don't know why; it just happened. We used Crossroads' philosophy. You know, some of you boys have. But at one point we were forced to use some urea in the dry form to supplement the solution and our experience with that was bad, very bad. It didn't work.

CHAIRMAN REYNOLDS: Is there further discussion? I think we can have one more question, comment, or what-have-you here and then we're going to turn this program over to the next subject.

A QUESTION: I'd like to ask Mr. Lewis one question. You do not aerate, do you have any close rule of thumb for the amount of ammonia for P_2O_5? Say, you're making 12 per cent goods, do you have any rule of thumb, or what is your range?

MR. LEWIS: The chart that I showed a moment ago would give the recommended rate. In that particular case, if you were going to cool the material along the lines that I have indicated earlier, a four and a half pound rate would be about the maximum that you could expect to get with normal super, with the average normal superphosphate. Your own experience would actually determine whether or not you could go higher than that, but at that point you are at the point at which you start, or can get into trouble in the case of a 12 per cent goods.

The figures that I showed on the slides will be in the proceedings —I presume—so that you will have a copy of them eventually.

CHAIRMAN REYNOLDS: Thank you very much. We are indeed indebted to this panel for bringing these points to us, and this very lively discussion. We appreciate the cooperation of our audience. I turn the program back to Dr. Sauchelli who will introduce the next topic and speakers. Thank you.

(Dr. Sauchelli resumed the Chair.)

CHAIRMAN SAUCHELLI: I have one comment to make before I get into the regular program. We place great emphasis on these discussions, and when it comes to preparing the proceedings I know it's difficult if we don't have the names of persons. We'd like to name the person who asks the question and therefore we like to have you give it. We like to have a good record of the discussion. We would appreciate it if the speakers will kindly step to the microphone and identify themselves when they ask a question. I know it's a little bother but we would appreciate the cooperation.

Now the next subject is segregation. As far as I can go back in my connection with the industry, segregation has been a — (Laughter.)

— we're talking fertilizers — segregation has been always a lively subject. We thought that we had overcome that or had solved the problem with granulation, but we seem to go in cycles. Segregation is still a problem in the fertilizer industry, as well as elsewhere, but in the fertilizer industry we still have that problem to contend with.

The next speaker is one of the sharp minds in our research group and he is of a philosophic trend. He has always liked to get at the bottom of things and we are privileged to have his thinking on the subject. He has had a wide and long experience on this and related subjects. As was brought out in some of the previous talks, we still need a great deal of fundamental research in fertilizer problems, and so it gives me great pleasure to call on the next speaker, Mr. W. L. Hill, of the United State Department of Agriculture who has addressed these meetings in the past and he doesn't need much introduction to this group.

Mr. Hill.

Segregation of Fertilizers

W. L. Hill

SEGREGATION is the opposite of interspersion. In descriptive use the term implies an unmixing of something once mixed. The U. S. Department of Agriculture gave prominent attention to segregation of fertilizers, particularly in its relation to their drillability, some 25 years ago *(1, 4, 5, 7, 8)*, and then the popularity of the subject gave way to an intense interest in granulation of fertilizers, which was looked upon as a panacea for all segregation problems. In those days emphasis lay on the granulation of finely-divided blends. Recent movement towards the blending of granulates restores the interest in segregation—a circumstance witnessed by the topic assigned at this time.

Homogeneity of Fertilizer Mixtures

A well-mixed lot of close-sized, but chemically unlike, fertilizer granules possesses a built-in heterogeneity, which increases with granule size, so that the smallest sample typifying the lot will be the larger the greater the granule size. This principle, in reverse, is utilized in the analytical laboratory when coarse samples are ground for subsampling. With only modest generalization it follows that the size of the smallest typical sample is a measure of the inhomogeneity of the lot.

Usually fertilizer homogeneity is judged on the basis of chemical analyses, because this criterion is implied in the guarantee. In the study of segregation problems, however, physical homogeneity possesses prime interest, and properly so, for physical homogeneity implies chemical homeogeneity, whereas the converse is not necessarily true.

Kinds of Segregation

One kind of segregation occurs during pouring operations. Sorting of particles of different sizes and densities in mutually hindering fall through eddying air takes place in conformance with known rules. A different set of rules applies when the granules strike a conical pile. Plant operators know how to minimize this type of segregation. Another kind of segregation occurs as a consequence of vibration. Settling in the pile or bag is an instance of mild vibration or jostling.

Segregation may be localized and randomized, and thus not be a cause of serious concern, so long as the typical sample is not thereby made too large. In fact, this form of segregation occurs normally in mixtures described in trade as nonsegregating ones. When, however, size sorting proceeds to the point of general stratification in the lot, segregation has reached its worst by any standards. Stratification occurs when a mixture of large granules contains some granules that are small enough to pass through the inter-granule channelways. Moderate vibration causes the small ones to fall to the bottom. This is the condition to be discussed at this time.

Structure of Fertilizer in Bulk

The structure of a fertilizer in a pile or bag refers to the mutual disposition of the granules in the assemblage. It is described in terms of spacing and packing factors. Structure constants for hard smooth spheres of diameter *1* in bulk *(2, 3)* are:

Type of Packing	Size of Spheres	Diameter of Critical Sphere for Occupancy of Void Space	Diameter of Critical Sphere for Throat Entrance	Inter-Sphere Porosity, % of Total Volume
Closest (rhombic) (array)	Uniform, *1*	0.414 *1*	0.154 *1*	25.95
Open (cubic) (array)	Uniform, *1*	0.732 *1*	0.414 *1*	47.64

Thus, an assemblage of spheres of diameter *1* in open packing exhibits an inter-granule porosity of nearly 48% and presents throat openings in the channelways that permit a sphere as large as 0.414 *1* to pass. In close packing the figures are proportionally smaller.

Non-smooth and generally nonspherical fertilizer granules cannot be expected to behave in the regular fashion found in the structure of smooth spheres in bulk. Nevertheless, spheres present an ideal case, which can be used as a point of departure for serious consideration of the structure of the fertilizer pile, insofar as it is disclosed by the magnitude of the intergranule porosity.

Porosity of Fertilizers. Inter-granule porosity of fertilizer in bulk is determined by the level of fineness, granule size distribution, granule shape and surface character, thoroughness of interspersion of sizes and shapes and granule packing. The packing is influenced by roughness or stickiness of surfaces. which prevents the granules from sliding into close packing, granule hardness, age of pile and pile pressure, among other things. Consequently, the packing, and therewith the inter-granule porosity of fertilizer *in situ,* depends generally on the history of the lot—fresh or aged pile, fresh-packed bag or one in storage, as initially charged to distributor hopper or after jostling the length of the crop row, and the like.

Special interest attaches to chance packing of fertilizer during handling operations, where settling stresses are moderate and sustained for only short periods, such as conveying, bagging and manipulations incident to field distribution. Porosities typical of fertilizers under such conditions are illustrated in Table I by results obtained on fer-

Table I. Inter-Granule Porosity of Some Fertilizer Materials

Character of Granules and/or Grade of Product	Fineness	Specific Volume of — Granules in Bulk[a]	Granules[b]	Inter-Granule Voids	Inter-Granule Porosity
	U. S. Sieve No.	cc/g	cc/g	cc/g	%
		Ammonium Sulfate[c]			
Regular, byproduct	—	0.967	0.549	0.418	43.2
Regular, byproduct	—	0.923	0.574	0.349	37.8
Regular, byproduct	—	0.903	0.572	0.331	36.7
		Ammonium Nitrate[d]			
Sprayed	—	1.164	0.641	0.523	45.0
Monocrystalline	—	1.028	0.592	0.436	42.4
Grained	—	1.013	0.606	0.407	40.2
		Superphosphate[c]			
Angular, 46%	-12[e]	1.366	0.474	0.892	65.4
Angular, 46%	-12[e]	1.454	0.523	0.930	64.0
Angular, 20%	6-20[f]	0.938	0.490	0.448	47.8
Rounded, 46%	6-20[g]	0.814	0.478	0.336	41.3

[a] Settled by gentle tapping of container on bench.
[b] Determined by displacement in glycerol.
[c] Determinations by J. H. Caro.
[d] Results adapted from published results for densities *(6)*.
[e] Angular granules synthesized, size distribution: U. S. No. 12-20, 15% ; 20-40, 30% ; 40-70, 35% ; 70-140, 15% ; -140, 5%
[f] Angular granules, size distribution: U. S. No. 6-12, 64% ; 12-16, 22% ; 16-20, 14%.
[g] Rounded granules, size distribution: U. S. No. 6-12, 53% ; 12-16, 30% ; 16-20, 17%.

Table II. Classification of Granule Finenesses

Sextave of Fineness	U. S. No. of Sieve[a] (Theoretical granule size in mm.)					
A	— (9.66)	2½ (8.12)	3 (6.84)	3½ (5.75)	4 (4.84)	5 (4.00)
B	5 (4.00)	6 (3.36)	7 (2.83)	8 (2.38)	10 (2.00)	12 (1.66)
C	12 (1.66)	14 (1.39)	16 (1.17)	18 (0.99)	20 (0.83)	25 (0.68)
D	25 (0.68)	30 (0.58)	35 (0.48)	40 (0.41)	45 (0.34)	50 (0.28)
E	50 (0.28)	60 (0.24)	70 (0.20)	80 (0.17)	100 (0.14)	120 (0.12)
F	120 (0.12)	140 (0.10)	170 (0.08)	200 (0.07)	230 (0.06)	270 (0.05)

[a] Sizes given in the B sextave represent openings of standard series. Sizes in other sextaves are obtained by multiplication of these figures by 1/0.414 for the A sextave, by 0.414 for the C sextave, by $(0.414)^2$ for the D sextave and so on. For these the tabulated sieve numbers are ones with openings closest to calculated sizes.

tilizer materials with the use of moderate experimental settling. The observed porosities range from 37% upward to 65%. Thus, it appears that chance packing of fertilizers under moderate settling stresses approximates open packing after the pattern of uniform spheres in cubic array. In the case of smooth spheres this packing is unstable, but in fertilizers the influence of granule shape and surface roughness seems to impart marked stability to it.

Critical Size of Channelways. Reasoning by analogy with the structure of uniform spheres in the light of observed porosities of fertilizers, and the type of packing thereby indicated, it is concluded that granules smaller than 0.414 times the diameter of the structure-determining granules may fall through the channelways of interconnecting void spaces. Thus, the structure determined by 4-mm. granules would not permit a granule larger than 0.414 x 4, or 1.66 mm., to fall through the channelways. This is the tentative factor for finding the smallest permissible granule for a non-stratifying assemblage. Deduced by devious reasoning, the tentative factor may be larger (it can hardly be smaller) than is necessary to provide a reasonable margin of safety in application to the variety of granules usually encountered in fertilizer making. The best value must be sought by experimentation.

Mixture of Granule Sizes. Since the size of the channelways is determined by granule size, shape and packing, meaningful discussion must be directed to particular kinds of granule assemblages under specified conditions. A frequently-encountered assemblage is one composed mainly (60% or more by weight) of granules in a fairly narrow size range with lesser amounts of smaller granules. In this case the structure is determined by the dominant size class, and, if the limits of the latter range be known, the critical diameter of the channelway can be found with the use of the tentative factor noted above. Assemblages with no dominant size class are less tractable and perhaps can be generally avoided in fertilizer practice.

Size Distributions Classified

Application of the notions about the channelway requires a procedure for dealing with size ranges in a functional manner. To this end, the screen scale is divided into intervals, or sextaves, of fineness as shown in Table II. Reading down the columns, each sieve opening after the first is 0.414 times the one immediately above it. The rows in the table, designated A to F, respectively, show the sequences of sieves in the major sextaves appear consecutively in both the second and last columns of the table. Similarly, the limits of other sextaves appear consecutively in the other columns. Thus, for an assemblage of granules in which the dominant size class is in the B sextave (U. S. No. 5-12), for example, the structure determined by granules in this size range would not permit granules in the C sextave (U. S. No. 12-25) to fall through the throat openings. Granules smaller than the C sextave would, however, be expected to pass through the structure. Then, a fertilizer in which the structure-determining granules lie in the range No. 5 to 12 should not, according to the tentative rule, contain any material finer than the No. 25 sieve. This sequence of sieves appears as consecutive numbers in the 2nd column of the table. If the dominant size range were No. 12 to 25, the next triad in this column shows that material finer than No. 50 should be absent. This rule applies, columnwise, to each triad of consecutive sieves.

A simple example will illustrate the potential value of the rule

Table III. Size Classifications of Specimens of Marketed Ammonium Sulfate and Nitrate and of Sodium Nitrate

Item No.	Character of Granules	Principal Sextaves, U. S. No.	Percentages of Lot in Principal Sextaves[a]	Coefficient of Self-Stratification
		Ammonium Sulfate		
1	Rice shape	8-18, 18-40	(4) 80, 15	1
2	Rice shape, small	12-25 ,25-50	(1) 60, 27	12
3	Regular	do	30, 45	<24[b]
4	Regular	14-30, 30-60	(3) 54, 35	8
5	Regular	18-40, 40-80	(8) 55, 29	8
6	Regular	do	(6) 52, 35	8
		Ammonium Nitrate[c]		
7	Sprayed	10-20, 20-45	96, 4	0
8	Crystallized	do	(?) 93, 6	1
9	Grained	do	(?) 88, 12	0
10	Sprayed	do	78, 17	5
11	Grained	16-35, 35-70	(5) 62, 33	<1
		Sodium Nitrate[c]		
12	———	8-18, 18-40	(7) 66, 20	7
13	———	12-25, 25-50	(4) 56, 40	<1

[a] Number in parenthesis is percentage retained on coarser sieve in 1st principal sextave.
[b] No dominant sextave.
[c] Results adapted from published screen analyses *(6)*.

Table V. Size Classifications of Specimens of Marketed Potassium Chloride

Item No.	Principal Sextaves, U. S. No.	Percentages of Lot in Principal Sextaves[a]	Coefficient of Stratification
		Granular Grades	
1	6-14, 14-30	97, 3	0
2	do	(15) 82, 3	0
3	8-18, 18-40	(4) 65, 29	2
		Coarse Grades	
4	8-18, 18-40	(4) 66, 29	1
6	12-25, 25-50	(9) 80, 11	0
7	do	(8) 80, 12	0
5	do	(10) 71, 19	<1
		Standard Grades	
8	16-35, 35-70	(12) 53, 31	4
9	20-45, 45-100	(3) 69, 26	2
10	do	(14) 66, 20	0
12	do	(2) 57, 37	4
11	do	(6) 51, 36	7
13	50-120, 120-270	(3) 77, 20	<1

[a] Number in parentheses is percentage retained on coarser sieve in 1st principal sextave.

in practical applications. A commercial granular superphosphate characterized by well-rounded granules showed sextave size ranges as follows; No. 5 to 12, 68%; No. 12 to 25, 29%; and No. 25 to 60, 3%. After tapping the container a few times the material finer than No. 25 was practically all in the bottom layer. The extent of stratification is indicated by the amount of material finer than the lower extreme of the second sextave of this 2-sextave size distribution. This percentage (3%) is the coefficient of stratification of the superphosphate.

Stratification of Single Materials

The problems presented by segregation of mixed fertilizers begin with the ingredients in the storage bins. Stratification at this point means increased variability in size classification among portions removed for successive blends, and consequently, some batches segregate rather seriously, whereas others prepared from the "same lots" of ingredients do not. The expected tendency to self-stratification in some marketed grades of nitrogen-bearing salts, phosphorus carriers and potassium chloride is indicated by the figures in the last columns of Table III to V, respectively. The coefficients of self-stratification range from 0 to 12%, or more, for the ammonium salts and from 0 to 7% for potassium chloride, which pose the question as to a reasonable tolerance. It would seem that 1% is a suitably fair figure. Movement of this proportion of a single-nutrient ingredient from the top layer to the bottom layer of a blend would alter the nutrient content of a sample from the top by no more than 1% of the grade, or 0.1% for a 10% guarantee.

Stratification of Dry Blends

Sextave Classification of Size Distributions. In a very real sense blending is a process for combining granule size distributions according to specified proportions. In dry blending, the ingredients being relatively dry, cementation between neighboring granules is minimized and the smaller granules can move through any openings that are large enough. Size distributions can, as was noted earlier, be simply classified according to the number of principal sextaves. In general, when there are more than two principal sextaves in the size range, no one sextave dominates the structure of the lot of material. In distributions two sextaves wide either the coarser or the finer sextave may be dominant, but dominance of the coarser appears to be the normal situation.

Compatibility of Size Distributions. Obviously, two size distribu-

Table IV. Size Classifications of Superphosphate and Ammonium Phosphate

Item No.	Character and Grade		Principal Sextaves U. S. No.	Percentage of Lot in Principal Sextaves[a]	Coefficient of Self-Stratification
			Superphosphate		
1	Run of pile,	20%	4-10, 10-20, 20-45, etc.	(1) 12, 12, 20, 25, 30	[b]
2	“ “ “	20%	12-25, 25-50, 50-120, -120	(4) 26, 25, >14, <35	[b]
3	Granules	20%	4-10, 10-20, 20-45, -45	(4) 35, 35, 22, 8	[b]
4	“	20%	5-12, 12-25, -25	(7) 48, 35, 10	[b]
5	Granules,	46%	6-14, 14-30	78, 20	2
6	Granules,	46%	12-25, 25-50, -50	(9) 44, 27, 20	[b]
7	Run of pile	46%	8-18, 18-40, 40-80, -80	(3) 26, 28, 27, 16	[b]
			Ammonium phosphate		
8	Ammophos[c]		8-18, 18-40, -40	(14) 42, 20, 26	[b]

[a] Number in parentheses is percentage retained on coarser sieve of 1st principal sextave.
[b] No dominant sextave.
[c] Results adapted from published screen analysis *(6)*.

Table VI. Combination of Size Distributions in the Synthesis of Dry Blends

	Size Distribution of Ingredient			Proportion in Blend			
Ingredient and Grade	1st Principal Sextave	Fraction in 1st and Succeeding Sextaves	Coef. of Stratification	Amount	1st Principal Sextave	Fraction in 1st and Succeeding Sextaves	Coef. of Stratification
	U. S. No.	%	%	Lb./Cwt.	U. S. No.	%	
			8-16-16 Fertilizer				
Ammonium sulfate, 21%	8-18	(4) 80, 15	1	38	8-18	(1.5) 30, 5.7	0.4
Superphosphate, 46%	do	(3) 26, 28, 27	16[a]	35	12-25	(1) 9, 9.8, 9.5, 9.6	15.1
Potassium chloride, 60%	do	80, 18	2	27	do	22, 5	0.5
Size distribution of blend				100	8-18	(2.5) 61, 20	16
			10-10-10 Fertilizer				
Ammonium nitrate, 35%	10-20	78, 17	5	29	12-25[c]	(4) 20, 5	0.3
Superphosphate, 18%	12-25	(4) 26, 25, >14, <35	[a]	55	12-25	(2) 14, 14	25.0
Potassium chloride, 60%	8-18	(4) 66, 29	1	16	12-25[c]	(3) 12, 1	0.2
Size distribution of blend				100	12-25	(9) 46, 20	25[d]

[a] No dominant sextave.
[b] In the combination 8-18 sextave is dominant; hence material finer than U. S. 40 contributes to the coefficient.
[c] Size distribution reclassificatio in terms of the sextaves 12-25, 25-50.
[d] No dominant sextave; this figure is merely material finer than U. S. 50 sieve.

tions not subject to self-stratification can in combination yield blends that do stratify. Such distributions are incompatible. Specifications for particular combinations of certain compatible distributions can be readily deduced. Three cases in increasing order of complexity will suffice to illustrate the rules operating in the synthesis of size distributions.

CASE I. Two or more 2-sextave distributions, congruent in the sense that all lie in the same two sextaves.

a. *The coarser sextave is dominant in all distributions.* Combinations in any proportions yield a blend in the same sextave class. The coefficient of stratification of the blend lies between the highest and lowest coefficient of the individual distributions.

b. *The finer sextave of one is dominant.* The dominant sextave of the blend, also the coefficient of stratification, depends on the proportions of the individual distributions.

CASE II. One of the two or more distributions extends over more than two principal sextaves with no dominant one. The dominant sextave of the blend will ordinarily be the coarsest principal sextave and the coarse structure thus developed will make the coefficient of stratification rather high. This situation is illustrated by the 8-16-16 blend in Table VI.

CASE III. Two or more two-sextave distributions overlap without being quite congruent, for example, three distributions whose first principal sextaves are 8-18, 10-20 and 12-25—a situation illustrated by the 10-10-10 blend in Table VI. The blend has no dominant sextave and carries an appreciable quantity of granules coarser than the first principal sextave and a larger quantity of granules finer than the second principal sextave. Nothing can be concluded as to the coefficient of stratification, because guide rules have not been developed for this class of size distributions.

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CHAIRMAN SAUCHELLI: Thank you very much, Mr. Hill, for making a difficult subject interesting and making it understandable.

The trend all through agriculture and in our industry is toward more and more precision. These considerations, these attempts to find out more and more the whys and wherefores are very important and that's why I think Mr. Hill's contribution to our thinking is worth while. We have time for about one, not more than two questions on this or comments.

We're going to adjourn promptly at twelve o'clock.

If there are no questions, I think we'll turn the meeting over to Mr. Spillman who will monitor and introduce the next speakers.

(Mr. Spillman assumed the Chair.)

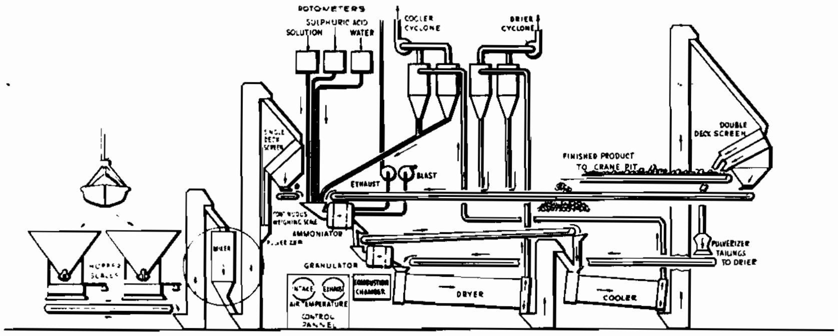
CHAIRMAN SPILLMAN: The next subject on the program covers mixing. It is obvious that proper control of mixing and proper mixing equipment play an important part in our operations for mixing either conventional, semi-granular, or granular mixes. Definitely both the rotary and gravity mixers play important roles in our manufacturing setup for a thorough mixing program.

We have on the panel for this subject Mr. Walter Sackett of A. J. Sackett & Sons Company, Mr. Kreuger of Stedman Machine Company, Mr. Robinson of the Atlanta Utility Company, and we're very sorry to report that Mr. Hefler could not make this meeting, who is with the Ranson Machine Company. However, we hope to get his paper and publish it in the proceedings.

The first speaker on this subject will be Mr. Walter Sackett, who will discuss the gravity mixer.

The Gravity Mixer

Walter Sackett



Floor diagram showing the location of the gravity mixer in the overall train of production operations. It is seen in the circle on the left side of this layout.

Figure 7

THANK you, Al. Mr. Chairman, members of the Executive Committee, fellow members of the Round Table, with the aid of some slides I've brought along, I want to present to you a basic function of the Sackett Gravity Mixer. This first slide (Fig. 1) shows an interior or phantom view of a Sackett patent Gravity Mixer.

This machine does a thorough mixing job of two or more solid ingredients without the use of any power except the air required to operate the mixing grates. Its outside shell is essentially a vertical cylinder with a square intake spout and a transitional type discharge hopper.

Its interior is divided into two chambers formed by two sets of semi-circular mixing gates. Located immediately above these gates are two vertically suspended rings or nests which create three compartments the bottoms of which are formed by the mixing gates. These nests in effect divide each batch into three portions.

In the next slide (Fig. 2) to aid in the clarity of this presentation we will in each case repeat the earlier illustration with each progressive slide. Slide two, again reveals therefore the phantom view of the Sackett Mixer as well as the position of both upper and lower mixing gates preparatory to receiving the first batch of ingredients to be mixed.

Now in the next slide (Fig. 3) this shows the ingredients as they enter and are accumulated in the upper chamber of the Sackett Mixer. While any number of ingredients may be compounded into the batch, it is very important to notice that the first third as well as the second and final third portions all come to rest directly on the mixing gates. The colors used portend to show only the manner in which the various ingredients may come to rest within the mixing chambers, depending entirely upon the numbers and proportions of ingredients to be compounded into the batch.

The next slide (Fig. 4) — as soon as all of the materials making up the batch have been accumulated in the top compartment the mixing gates are immediately lowered. Upon lowering of the mixing gates one can readily understand that the various ingredients contained in all three thirds of the batch are discharged simultaneously and are blended as they drop from the upper compartment in to the lower mixing compartment of the machine.

The next slide (Fig. 5) shows the batch as it has been accumulated in the lower compartment of the machine. The mixing gates of the upper compartment are closed to immediately receive the next batch.

The next slide (Fig. 6) show the mixing gates of the lower compartment in the discharge position and with a second blending-in action taking place as the batch leaves the mixer. Note the next batch being accumulated in the upper compartment while the first

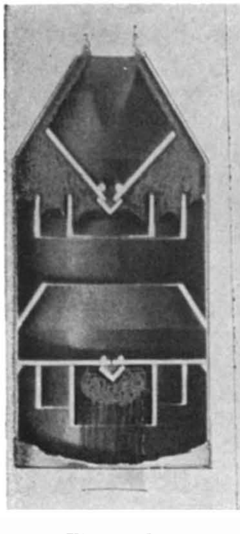


Figure 1

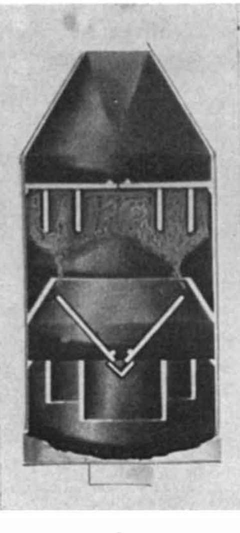


Figure 2

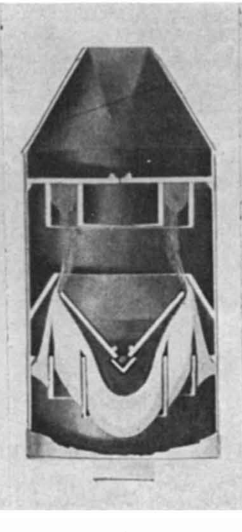


Figure 3

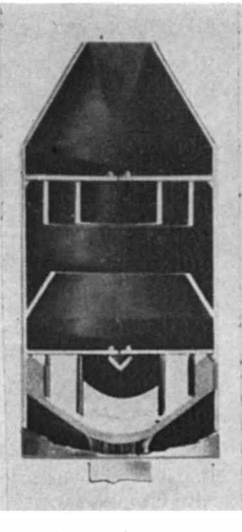


Figure 4

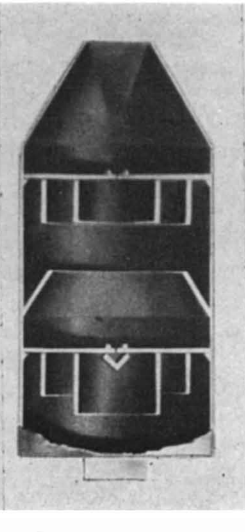


Figure 5

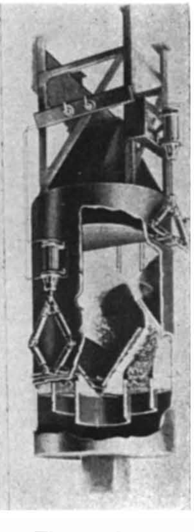


Figure 6

is being discharged. The Sackett Mixer's ability to handle two batches simultaneously in this manner greatly speeds up the mixing cycle.

The next slide (Fig. 7) shows the location of this particular case a granular operation, a granulating unit. It may be seen, the mixer may be seen in the circle on the left side of this typical layout.

In conclusion, gravity mixing as it is accomplished by the Sackett method offers the fertilizer manufacturer an effective and low cost means of thoroughly blending his powdered raw materials at high production rates. Its operation may be controlled from any remote point. Long machine life, quietness of operation and minimum operating and maintenance expense are assured by the almost complete absence of wearing parts and mechanical driving force. Thank you.

(Applause.)

CHAIRMAN SPILLMAN: Thank you, Walter. Mr. Harry B. Kreuger, Stedman Machine Company will discuss the Stedman Rotary Mixer.

Operating Procedures for Rotary Batch Mixer to Accomplish

(a) Dry Mixing (No ammoniation)

(b) Mixing and Ammoniation

(c) Mixing, Ammoniation and Granulation

H. R. Krueger

Questions:

1. What is the time required for dry materials to enter Mixer?

Charging time will depend on two things, first, location and design of holding hopper above mixer intake valve; second, the size of batch. If the holding hopper is located immediately above mixer intake, and the hopper has no angle flatter than 60°, the mixer should charge as follows:

½ ton batch — 8 seconds
1 ” ” — 12 ”
1½ ” ” — 16 ”
2 ” ” — 20 ”

There could be other small variables, such as:

Accessibility and ease of operation of intake valve.

Whether intake valve is manually or pneumatically operated.

Smoothness of lining of surge hopper.

Size of mixer inlet opening.

(The Stedman Batch Mixers have the following sizes of inlet openings)

½ ton	16″ x 16″
1 ”	18″ x 20″
1½ ”	24″ x 28″
2 ”	24″ x 28″

2. What is the time required to leave the material in the Mixer for satisfactory mixing?

The answer to this question will have to be in the three categories of dry mixing, mixing and ammoniation — mixing, ammoniation and granulation.

Dry Mixing: A maximum of one minute should be ample for ½ to 2 ton batches. The size and design of the shell and flights vary with the mixer size, so mixing time is about the same for all sizes.

Mixing and Ammoniation: Mixing time will vary, largely dependent on design of sparger pipes and pressure used on solution measuring boot. From 1½ to 2 minutes should be ample, and this should include time required to inject solution. We normally recommend 20 to 30 seconds mixing time after all of the solution has entered the batch.

Mixing, Ammoniation and Granulation: We assume a mineral acid is being used and that the design of spargers for both acid and solutions is adequate for maximum amounts of these ingredients used per batch. Holding time will vary with analysis, formulation and granulation properties of ingredients. As an average, we would recommend the following, after *all* materials have been introduced:

½ ton — 1 minute
1 ” — 1½ ”
1½ ” — 2 ”
2 ” — 2 ”

Frankly, there can be so many variables when granulation is required, the length of time required to accomplish the best granulation becomes a matter of "know how".

3. What is the time required to discharge the Mixer?

Again, this must be divided into three answers:

Dry Mixing — 20 seconds
Mixing and Ammoniation — 30-40 seconds
Mixing, Ammoniation and Granulation — 46-60 seconds

There are two things that can greatly aid in reducing time required for mixer discharge. First is the type of discharge chute. Our company makes two types of discharge doors on all sizes of mixers. For dry mixing the discharge scoop is inside the mixed during the mixing cycle and swings out when the door is opened. For ammoniation or granulation we recommend the exterior type of discharge scoop, which is outside of the mixer during the mixing cycle and swings in with the door to discharge. This type of discharge is highly preferable when the mixture is hot, damp or sticky, and is readily accessible for cleaning. The other aid to rapid discharge is a substantial bumper or knocker arrangement to prevent interior build-up of the shell.

4. How long should the operator wait before starting the ammonia and nitrogen solution?

As quickly as possible after the dry ingredients have entered and the intake valve closed.

5. Describe the spray system recommended.

There are many pet theories and designs of sparger pipe arrangements. In our humble opinion, the following requirements are basic:

a. The pipes should be as low in the mixer as flight design will accommodate.

b. They should be on the "Up-hill" side, about 20° from vertical.

c. They should be strong and easily removed.

d. Stainless is a waste of money, except perhaps for phosphoric acid spargers.

e. Frequent inspection for orifice erosion.

6. Can pulverized materials be fed separately into the mixer with batch hopper door open and the discharge door closed, and obtain satisfactory mixing?

Our answer is a qualified "yes," the qualification being to allow another 30 to 45 seconds mixing time. Fed individually, you lose the mixing action derived in the "coning-out" from an assembly or surge hopper.

7. When using the mixer for ammoniation and granulation, what is the recommended procedure for keeping the mixer tight?

a. Clean and check fits of intake valve or gate daily.

b. Provide good gasket of felt or soft rubber between intake valve and mixer intake chute. (Intake valve should not be bolted to intake chute)

c. Provide some type of gaskets where sparger pipes enter mixer.

d. If mixer has grease-type seals on intake or discharge rings, grease generously at least twice per shift.

e. Keep discharge door in proper alignment in relation to seal ring. Keep it clean.

f. Provide generous steam and fume duct on discharge hood.

g. Periodic check of shell alignments in relation to base and trunnions.

8. What is the recommended rate of introduction of the nitrogen solution and anhydrous ammonia, in pounds or units of nitrogen?

This is a difficult question to answer without knowing the balance of the formula. For economic and granulation purposes, free ammonia should certainly not be fed so fast that it does not have time to combine, thus escaping as a gas, which costs money and promotes bad working conditions. Since the heat of reaction and the humidity conditions inside the shell are prime promoters of agglomeration, it seems logical that free ammonia should be introduced as rapidly as possible, without incurring losses.

If all the granulation is being accomplished in the mixer, there being no granulator or dryer following it, retention time is inevitably longer, so it is possible to slightly lengthen the injection period without lengthening the cycle beyond economic time limits.

A cross-section check of plant practices indicates that free ammonia can be introduced at a rate of one unit (20 pounds) in 12 to 13 seconds.

CHAIRMAN SPILLMAN: Thank you, Mr. Krueger. We will now adjourn our meeting until one thirty this afternoon and continue our symposium on mixers when we return from lunch.

Thursday Afternoon Session

November 5, 1959

The Round Table reconvened at one fifty o'clock p.m.

Mr. Albert Spillman presiding

CHAIRMAN SPILLMAN: Our Round Table will now come to order. On behalf of the entire membership of this Round Table, many thanks to the Potash Companies for being our host to a wonderful lunch.

We will now continue with the symposium on mixers, and our next speaker is Mr. R. E. Robinson of the Atlanta Utility Works who will speak on the Atlanta Utility Rotary Mixers.

Batch Mixing of Fertilizers — Processing Problems

Robert E. Robinson

Abstract

THE underlying theory of mixing dry solids is based on certain natural properties of these solids. There are many ways of accomplishing mixing, and choice of method and equipment is dependent on these properties and other requirements. In the fertilizer industry the rotary drum batch mixer emerged as the primary equipment for dry mixing of solid fertilizers.

With the advent of synthetic nitrogen solutions, mixing of fertilizers became a chemical operation involving chemical reactions in addition to physical processing. Early ammoniation was accomplished in existing type equipment with minor additions and techniques were relatively simple and trouble-free. Proper utilization of a batch mixer for ammoniation requires an understanding of the mass flow of material in the mixer past the solution distributor and consideration of the time factor in introducing solutions into that mass. Distributors have been developed which match the concentration of solution flow to the mass flow to give uniform ammoniation in the minimum time.

Requirements for mixing, ammoniation, and granulation impose added problems on the batch mixer, regardless of whether granulation is accomplished in the mixer or in a rotary drum following the mixer. Controls must be used to maintain physical conditions suitable for accomplishment of the granulation process as well as for accomplishment of mixing and ammoniation reactions, and in addition conditions must be maintained within the operating capabilities of the batch mixer.

Batch mixers have always had many features affording good service to the fertilizer manufacturer. Recent developments in mixer construction have made possible operations once thought impossible for batch mixers, and mixer design has progressed with changes in fertilizer manufacturing practice.

Operating procedures must cover a very wide range of conditions and problems, including different types of fertilizer such as powdered, semi-granular, or sized granular, production rates required, methods of control available, and many others. Obviously, we cannot set down a few rules that will cover all situations.

Installation and maintenance of rotary drum batch mixers has become increasingly more exacting as the mixers have become more complex and operating conditions more severe. Suggestions for installation, inspection, routine service, and maintenance are given.

A chosen set of references are given. These contain much information on particular problems appropriate to processing of fertilizers not only in batch mixers but also in continuous ammoniator units.

Dry Mixing of Fertilizers

The theory of mixing dry solids rests on certain natural properties of the solids being mixed. First, there is no tendency towards diffusion as in some liquids and solutions. Solid particles do not have mobility of their own and a solid mixture will tend to retain its state of equilibrium when at rest. To mix we must disturb the natural equilibrium of the segregated unmixed solids to achieve a nearly uniform condition.

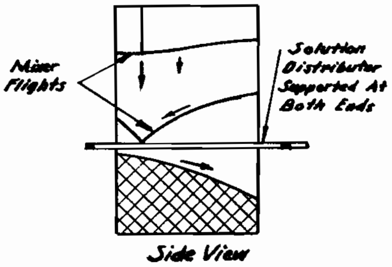
This mixing of dry fertilizers can be accomplished by using gravity to move the material through a series of fixed cone blenders, by using rotary or tumbling equipment such as tumbling conical blenders or flighted rotary drum mixers, or by power mixing with moving flight mixers such as revolving flight mixers, cut-flight mixing screw conveyors, paddle-type pug mixers, and ribbon flight mixing screw conveyors.

Mixing may be done in batches or continuously. Some types of mixing equipment are better for continuous operations and others are better for batch operations. Characteristics of the materials such as flow properties, specific gravities, particle sizes, particle shapes, abrasiveness, tendencies to cake, agglomerate, and aerate also influence choice of the mixing equipment.

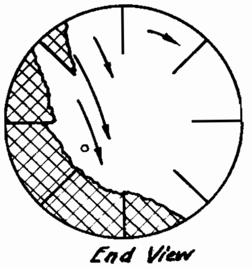
In the fertilizer industry the flighted rotary drum batch mixer emerged as the predominant piece of mixing equipment for a number of reasons: Relatively large batches and high tonnage were required and could be handled, capital cost was relatively low, power required was relatively low, life of equipment was long, and maintenance and operating costs were quite low. The rotary drum batch mixer gives good uniformity of mixing even for large batches in a minimum of mixing time, it can be charged and discharged easily and quickly, and in dry fertilizer mixing practice there are few problems of operation. (1), (2).

Mixing and Ammoniation of Fertilizers

With the advent of synthetic nitrogen solutions the manufacture of fertilizers became an operation requiring chemical recations in addition to dry mixing. Initially the rates of ammoniation were low and the mixers then in use were simply fitted with distributor pipes entering the mixers through the intake chute. Nitrogen solution



Longitudinal Flow Pattern Of Material
Fig. 1



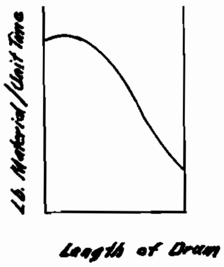
Radial Flow Pattern Of Material
Fig. 2

was sprayed onto the batch as it was being mixed. Very good results could be obtained in this manner at the ammoniation rates then being used without serious loss in production rate.

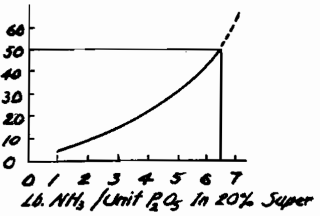
To properly consider more recent developments, we might consider that a mixture of dry solid particles will always have what we call random distribution.

A mixture is never perfect but on the average the particles are uniformly mixed. In a theoretically ideal mixer, each particle will eventually pass by any given point and all particles will pass that point before any particle passes that point again. Because of random distribution, this does not happen but we can come close, the probability following the familiar Gaussian distribution curve. Thus, we could theoretically ammoniate the batch with a distributor having only one hole if we introduced the solution slowly enough so that all partciles would pass the ammoniating area by the time all of the solution was introduced. This would be very inefficient use of the mixer insofar as time is concerned, and production would be very low. Some existing distributors are approaching this same inefficiency however by putting solution into only part of the mixer and into a poor part at that.

If we determine the mass flow of dry material past each section of the distributor we find that this mass flow is much higher towards the discharge end in our mixers because of the shape of flights. If we install a solution distributor extending the full length of the mixer drum with holes spaced to introduce solution into the mixer in varying quantities along the length of the distributor to match the mass flow, then each particle will pass an ammoniation point much more quickly and we can introduce the solution faster and more uniformly. Present mixer designs provide this, supporting the distributors at both ends. See figures 1, 2, 3, and 4.



MASS FLOW OF MATERIAL
FIG. 3



CUMULATIVE TIME TO AMMONIATE
(Courtesy E.C. Perrin, Nitrogen Division)
FIG. 4

Mixing, Ammoniation, and Granulation of Fertilizers

When proper conditions of temperature, moisture, liquid phase, and motion of a fertilizer mixture are achieved, a physical occurrence known as agglomeration or granulation occurs naturally. In some cases this is done entirely in a flighted rotary drum batch mixer, but sometimes the mixing and ammoniation are done in the mixer and granulation is completed in a rotary drum following the mixer, with or without additional heat. There are factors for and against use of the batch mixer for this purpose, but at the present time a number of manufacturers are carrying out this type of operation to produce either semi-granulated or sized granular fertilizers. The reactions provide the required heat. Liquid phase properties and moisture content are governed by formulation. Solid raw materials may be granular, and in some cases recycled fines are used as a control. Unfortunately, we frequently find that we are leaving the area of ideal rotary drum batch mixer operation. When we operate with a mix that is too wet we have sticking and material build up, power consumption goes up rapidly, the ammoniation efficiency drops sharply, and material is much harder to discharge from the mixer. Abrasion and corrosion become worse, fuming can become a bad problem, and maintenance costs can increase. Generally the machine required is heavier and will have more accessories such as knockers, extra doors, and other special features. Successful operation of batch mixers for this purpose is dependent on maintaining physical conditions suitable for accomplishment of the granulation process as well as for accomplishment of mixing and ammoniation reactions, and in addition conditions must be maintained within the operating capabilities of the batch mixer. Weather conditions have considerable effect. (4), (5), (6), (7), (8), (9), (10), (11), (12).

Description of Rotary Drum Fertilizer Batch Mixers

Our batch mixers have always been of heavy rugged construction. They have many mechanical features which afford good service to the fertilizer manufacturer. Starting with a rugged machined base, four independently adjustable trunnion wheels are provided to support the rotating drum on two trunnion tracks. Drive is by means of a ring gear and pinion. Flights inside the drum lift and tumble the batch, pitching it towards the discharge end. The design capacity is based on the drum being ⅓ filled, which means a one ton mixer is intended to mix one ton of fertilizer at a time.

With the drum correctly loaded, the material tends to roll back towards the intake and as it is deposited near the discharge end. There is a greater concentration of material near the discharge end at all times and the mass flow of material is greater at that end. During mixing, the drum is closed by a door, and at time of discharge the door and a discharge chute are moved into the drum. The discharge chute then occupies a zone in which material is deposited by the flights, and this material is diverted out of the drum. The rate of discharge is rapid at first and slows as the drum empties. Usual practice is to cut off the discharge at some arbitrary point, leaving a small fraction of the previous batch in the drum. The discharge mechanism is contained in a steel housing which may be vented if desired. Material is admitted to the mixer through a batch valve and an intake chute at the intake end of the drum. Intake and discharge controls are both located at the intake end, and may be either manual or air operated. Clean-out doors are provided, and mechanical features are designed into the machines to permit adjustments so that operation can continue over a long period with simple repairs. See figures 1, 2, and 3.

Originally, seals were loose running labyrinth or diverter type. With the finer dryer materials we now use packed seals.

Improvements in mixer design to handle higher ammoniation rates are centered around the nitrogen solution distributor. Fully submerged spargers are used in many cases, but there are disadvantages to cutting the mixer flights. Partially submerged distributors extending the full length of the mixer drum are available with support on both ends, which greatly relieves the strength problem. More recent developments have called for additional distributors for sulphuric acid and in some cases anhydrous ammonia. It should be recognized that distributors must be kept up if good results are to be obtained. Further, in some cases, several different distributors might be required for widely differing formulas. Distributors are easily removed for replacement.

Other improvements in mixers include larger steeper intake chutes with no obstructions from solution distributors, steeper discharge chutes, larger doors for clean-out, and a simplified discharge mechanism.

Necessarily, the batch mixer has become more complicated and maintenance procedures have become more exacting. For this reason, suggestions for installation, inspection, routine service, and maintenance are included below in this paper. Many of these suggestions will apply equally well to TVA Type Continuous Ammoniators and other equipment.

Discussion of Operating Procedures

Because of the wide range of conditions and problems, such as grades being manufactured, types of fertilizer such as powdered, semi-granular, or sized granular, production rates required, plant facilities, methods of control available, and economic considerations, we obviously cannot set down specific answers to any questions that will apply in all cases. The following information is intended as a guide only:

How long should the operator wait before starting the anhydrous ammonia, nitrogen solution, or sulphuric acid?

Many operators charge the batch so that superphosphate goes in first, and begin introducing solution before all of the batch is in the drum. Indications are that the presence of acid does not hinder the ammoniation of superphosphate as such, and most operators start introducing them together. The entire dry batch should be introduced promptly and solution should not be sprayed into an empty shell.

Describe the spray system recomended.

Fully submerged spargers are probably best for using anhydrous ammonia. Full length partially submerged distributors using the Perrin developed distribution pattern appear quite satisfactory for most ammoniation and used in conjunction with sulphuric acid distributors have given good results in ammoniation and granulation. Different distributors may be needed to cover the full range of grades. Stainless steel recommended for solution distributors and after a good design is determined, Hastelloy "C" is recommended for acid.

Can pulverized materials be fed separately into the mixer with the batch valve open and the discharge door closed to obtain satisfactory mixing?

Yes, if the completed batch is mixed for the required period after the last ingredient has been added. In the case of a very small amount being the last ingredient, the probability of a good mix is lessened, and the danger in many installations is that such a small ingredient might be largely lost in the elevator if charged to the unit separately. Good practice in batching is to "cushion" each batch with one of the larger ingredients at each end and hold possible losses of the lesser materials to a minimum by partially mixing them as soon as possible, as in the elevator. Then any losses will be of an adulterated mixture and not a pure ingredient.

When using the mixer for ammoniation and granulation, what is the recommended procedure for keeping the mixer tight?

On some grades, venting is advantageous. Vents have been placed into the intake chute, with valves so that they can be closed when not wanted. The discharge housing can be vented if desired. We have heard of instances where openings were cut in the discharge door, with baffles to cover the openings for venting. The discharge door can be kept open about ½″ to 1″ for venting.

Seals are provided at both ends of the drum and when the mixer is not vented it is reasonably tight. This is advantageous when scrubbing gaseous ammonia in the latter part of a high ammoniation reaction.

What is the recommended rate of introduction of the nitrogen solution and annhydrous ammonia?

The rate of ammoniation is commonly expressed as pounds of free ammonia reacted per unit of P_2O_5 in the solid phosphates. Additional ammonia being reacted with acid does not enter into the time requirement or ammoniation rate, so long as local overheating and over agglomeration do not occur to interfere with ammoniation of the superphosphate.

The first 4# of ammonia per unit of 20% superphosphate can be taken up at the average rate of 1# in 5 seconds. (3) See figure 4.

It is doubtful if the mass flow in the mixer past the solution distributor can match this rate however, and most operators allow at least 10 seconds and some 15 seconds, up to 4# rate. Additional ammonia is added more slowly.

Acid should be well dispersed in as finely divided spray as possible under considerable pressure to prevent local overheating and over agglomeration with attendant fuming and loss of ammoniation efficiency. Rate of introduction of acid might be 30 to 40 seconds for 100# of acid. Acid is generally started simultaneously with solution.

Installation and Maintenance Suggestions for Rotary Drum Batch Mixers

1. Installation of New Mixers:

Mixers should be adequately supported and bolted down with base level.

Drive should be checked for accurate alignment.

All bolts and fittings should be checked for proper tightness.

Mixer should turn over without binding or excess friction. Packing friction will cause appreciable drag, which is normal. If seal parts are rubbing steel to steel, heat will be apparent. Moderate heat from packing is normal.

Mixer should be checked for lubrication. Reducers checked for oil supply.

Pinion and Ring Gear should be checked for proper mesh throughout drum rotation.

"V" Bolt or roller chain drive checked for proper tightness.

Batch valve checked for proper installation and operation. Note that opening in batch hopper is recommended to be size such that valve will not leak, working on "bookshelf bulkhead" principle. Weight of hopper and material above should not bear down on mixer intake chute as this will distort position of seal parts. Telescoping batch valve is intended to prevent this.

Mixer discharge mechanism should operate freely and through full travel of discharge door.

If air operated discharge of batch valve is used, air circuit should be properly connected. Pressure should be reduced to conserve air, and cushioning in each cylinder and flow control valves should be adjusted to obtain proper speed of cylinder travel without shock to mixer or valve parts.

During start-up, mixer should be run empty long enough to check for heating or binding before actual mixing.

Note: Manufacturer's instructions should be followed for reducers, couplings, motors, anti-friction bearing units etc. Reducers are designed to operate with widely differing tooth pressures and it is important that the proper gear lubricant as recommended by the manufacturer of the reducer be used. Some reducers should be drained and refilled after a short break-in period. Flushing is usually not recommended. If an Extreme Pressure Gear Lubricant is specified it should be used.

Electrical wiring and installation should comply with the National Electrical Code and with Local Codes. Structural support should comply with American Standard Building Code Requirements for Rotating Machinery Live Loading, and with local building codes. Machine should not be operated without having all guards in proper place.

2. Daily Inspection, Lubrication and Preventative Maintenance:

It is recommended that mixer be greased once daily, immediately after shutdown when still warm.

Anti-friction bearings should be lubricated with a high grade medium consistency grease having a melting point well over anticipated operating temperatures, intended for ball or roller bearings.

Plain bearings should be lubricated with cup grease. All plain bearings are fitted with Alemite Fittings for this purpose.

Handwheel operated discharge mechanism should be lightly greased at worm wheel and worm wheel quadrant.

Pinion Shaft Pillow Blocks may be greased less often if desired, in accordance with the manufacturer' instructions.

Air circuit lubricator should be checked for oil, and filter drained.

Visual inspection should be made for evidence of any part working loose, or unusual wear. The cleaner the area around the mixer is kept, the better maintenance results to be expected.

Packing may require occasional light oiling.

3. Seasonal Maintenance:

Packing is a wearing part. Periodic replacement should be made as necessary.

Seal alignment should be checked when packing is replaced, as follows:

a) Loosen and back out one trunnion bracket at a time and check each trunnion wheel for looseness or play. If necessary, remove wheel for service as noted below. When replaced the wheels should be set as follows:

b) Radial adjustment is obtained to provide a seal groove the same dimension all the way around of ¾" for the packing. If the top of the groove is less than the bottom, the wheels should be moved in slightly. If the left side is more than the right, both wheels should be moved slightly to the right. Note that a new wheel can be used with an older wheel, since each wheel is separately adjustable and turns on its own shaft.

c) Axial adjustment: all wheels should be set so that they ride against the inside flange of the trunnion track (toward the drum) to hold the drum axially and prevent it from moving forward and back. This must not be so tight that the wheels can bind, however. After the wheels are set, the seal plates are adjusted with the tram screws to provide a close gap between the stationary plate and the rotating seal ring.

d) Packing should be installed so that the wearing surface if any faces the stationary seal plate, since the packing rotates with the drum. Packing lubricated with graphite is recommended. Oil may be used if required. Packing clamp support parts may be replaced and the set screws tightened only enough to prevent leaking of the seal.

Trunnion Wheel Service:

a) Check all trunnion wheels as above at least once each season.

b) It is recommended that one wheel assembly be kept on hand as a spare, since this wheel can be used to replace any one of the four wheels while necessary parts or repair to the other wheel is being made, after which the repaired wheel becomes the spare.

c) If ball bearings are replaced before complete failure the other wheel assembly parts can be frequently reused. If the bearings actually disintegrate during operation the wheel will let down and the bearing caps and trunnion pin and bushing will be worn from metal-to-metal contact. In addition, the inside bore of the wheel can be ruined. Normal life of wheel bearings is very good, but failure is most often due to lack of lubrication and presence of fertilizer material. Normal greasing will tend to keep foreign material flushed away from ball bearings. If mixer is operated with a wheel that has let down, additional damage will be caused to seal parts, both at intake and discharge ends of drum.

Post Season Shutdown:

a) At the end of a season, the mixer should be cleaned up and carefully inspected. Any deterioration should be noted and needed repair parts ordered so that they will be on hand when needed. Condition of drum, mixer flights, intanke chute, discharge door and chute and discharge mechanism, discharge housing, seal parts, trunnon wheels, gear and pinion, and drive should all be noted. Solultion Distributor should be cleaned and inspected. If roller chain is used in drive, it should be cleaned and relubricated. Mixer should be lubricated, and if possible operated a few minutes at regular intervals during a long shutdown period. Air cylinders should be kept in the retracted position for maximum protection to piston rods.

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CHAIRMAN SPILLMAN: Thank you very much, Mr. Robinson.

Good mixing and the proper size and type of mixers used are very important to produce the quality of the final product desired. Mr. Sackett, Mr. Krueger and Mr. Robinson have explained thoroughly the functions of their respective mixers. We will now open the discussion from the floor. I know it's important to most of you. A good many of you have written in to our committee asking various questions on mixers. We'd like to have a good discussion on this subject.

Are there any questions on mixing equipment?

MR. CHARLES LUBOW (Star Fertilizer Company): What is the most desirable speed for a rotary mixer and would this same speed apply when you are making pulverized and semi-granulated?

MR. ROBINSON: I think we'd both give you exactly the same answer. We probably have slowed our mixers down a little bit on the higher ammoniation rates and on granular mixes. We've slowed them down some. Our two-ton mixers we operate at 11 rpm, and the ton and a half mixer we operate at about 12½ or 12.7, and the one ton mixer at approximately 14 or 14½, and our half ton mixer at about 16 rpm. Those are drum speeds.

CHAIRMAN SPILLMAN: Any further questions?

MR. ROBINSON: Mr. Krueger uses a linear speed of about 250 peripheral feet per minute on his shell. I'm rather sure that those speeds will correspond with ours very closely.

MR. LUBOW: Does that apply to both pulverized and semi-granulated?

MR. ROBINSON: We would run a little slower. Actually, we use a range, sort of a range. I'd say perhaps ten per cent slower on your wetter mixes.

MR. LUBOW: On your semi-granulated?

MR. ROBINSON: Yes, sir.

CHAIRMAN SPILLMAN: Can we have any comments, or further questions? (No response.)

Apparently you fellows did a pretty good job of explaining mixers in that there seems to be no further questions. If there are none, I'd like to turn this meeting back to Dr. Vince Sauchelli.

(Dr. Sauchelli assumed the Chair.)

CHAIRMAN SAUCHELLI: We have a request from several members for some brief remarks on proper drilling of Hastelloy steel and Mr. Campbell of the Haynes Stellite Company has agreed to answer or to give us some information within five minutes time, so I'm going to call on Mr. Campbell and hold him to his promise.

MR. W. F. CAMPBELL (Haynes Stellite Company): Until the conversation of some of your members during the past lunch hour I was under the mistaken assumption that there were no problems with Hastelloy C. Basically our suggestions for drilling of Hastelloy would be to use the most rigid setup that is available in your shop. By that I mean, even if you have a real small piece instead of going to a small drill press, go to the most substantial one that you have. We would suggest a speed of 10 to 20 surface feet a minute. We would suggest a feed of 2 to 8 thousandths. Before you start that, we have some suggestions on the drill bit itself. We recommend the use of high speed steel drills but not high speed drills, as some of the fellows indicated they were trying to get for this job. Most of the fellows seemed to buy new drills, it's a good idea to cut it down to just a stub length, instead of using the full length of the drill, cut it down to a stub long enough to give you the hole that you want and the clearance that you need and cut the rest of it off.

Another suggestion on the preparation of your drill bit itself would be to wind the web thin at the tip. By that I mean this, when you look at the end of the drill instead of having the flight configuration at the tip, wind the web so it will look more like looking at the end of a screwdriver, just for the length of the tip. With those suggestions passed on to your shop people, I don't think you will have a problem in drilling the Hastelloy C.

You can use a coolant, a soluble oil, or one part cutting oil to one part kerosene, as a mixture.

I propose to have our company prepare a flyer containing the information on machining and also on welding to pass on to everyone on the mailing list.

As I recall, last year the mailing list took a little time to receive them. If anyone has a more urgent request, if you will jot down a name which I will give you now, yours will be sent out immediately before we get the mailing list. It should be directed to the attention of Mr. Bowley, Haynes Stellite Company, 30 East 42nd Street, New York 17, New York and he will send you out immediately a brochure on the fabrication of these alloys.

How much time has that taken? I have two other suggestions.

CHAIRMAN SAUCHELLI: Go ahead.

MR. CAMPBELL: Also, during this past lunch time's discussion I was horrified to learn that one gentlemen proposed to anneal a piece of pipe with an oxyacetylene torch, or propane it could have been. To put this thing in the right perspective, the possibility of annealing it came up only in the event that plug welding an old piece of pipe or any welding operation—say a piece of pipe was completely broken,—in some corrosive media, as welded materials will suffer degradation where the parent metal in its original condition will not, our suggestion would be this: if you decide to change the location of holes, it is perfectly all right to get a piece of hastelloy of the same grade. In other words, if you're using Hastelloy C pipe, get a piece of Hastelloy C wire and with either heliarc or metallic arc plug-weld the holes. Do not use oxyacetylene. When using oxyacetylene you are putting heat into this pipe, the heat as such will not materially hurt the alloy, in one sense, however the gradient of heat, the length of time you have the heat on one portion why somewhere else along this piece of pipe you're going to have a poorer grain structure, poorer corrosion resistance. It may or may not have any effect in your media. I'm thinking that as welded material it will be all right, but do not use heat in the form of a torch.

Now, if you have plug welded the holes in a piece of pipe and in putting it back into service you find rapid corrosion at the weld, that can be remedied by full- annealing or solution-annealing the piece of pipe. To do that, it should be done in a furnace with very close controls; it should not be done with a torch or anything of that type and the correct range, temperature range, for Hastelloy C would be 2200 degree Fahrenheit to 2260 degrees Fahrenheit. If you are located out in a section that does not have commercial heat treating firms available, contact our nearest district office or somebody in our company and they can suggest to you who can do this job and do it correctly.

The only other thought I'd

Machining, Welding, and Heat Treating of HASTELLOY alloy C for Acid Spargers

Machining		Roughing		Finishing			
Operation	Cutting Speed, SFPM	Feed per rev., inches	Depth of cut, inches	Feed per rev., inches	Depth of cut, inches	Tool	Coolant
Turning	35 - 55	0.012	0.050	0.006	0.012	Tungsten Carbide or HAYNES STELLITE 98M2 alloy	Vantrol 523* 1 Part Oil 15 Parts Water
Facing	35 - 50	0.012	—	0.006	—	Tungsten Carbide or HAYNES STELLITE 98M2 alloy	Vantrol 523* 1 Part Oil 15 Parts Water
Drilling**	10 - 20	—	—	0.005	—	H.S.S. or Core Drill	Vantrol 523* 1 Part Oil 15 Parts Water

* or Windsor K5

** Use stub drill for best results. Keep web as thin as possible.

like to pass on would be the question of corrosion distance itself. I talked with a gentleman last night who very definitely does not have conditions that would be usual in most of your plants. In his case before going out and buying a piece of Hastelloy C or B or any other grade, we would recommend a discussion on his mixture, the elements and percentages and temperatures and so on, and we would recommend corrosion testing of samples, which we will freely give.

We'll put it this way: if you do not know that your conditions are similar to an application where it is positively known that Hastelloy C alloy is the proper grade, contact us rather than go out and buy a piece of pipe which may not be the right grade.

I hope I haven't confused you too much. We've covered quite a bit here.

General Remarks: For turning, facing, and boring cutting edges should have an end relief of 5°, a side relief of 10°, and a side cutting edge of 30°. The use of rigid fixtures with good support is recommended for all operations.

Welding

Welding or HASTELLOY C can be accomplished by using coated electrodes applied by the metallic arc, or bare wire applied by the Heliarc process. This pertains to the repair of enlarged sparging holes as well as other weldments. The oxy-acetylene process should not be employed under any circumstances.

Heat Treating

The alloy is supplied in the solution heat treated condition to provide optimum corrosion resistance and it is suggested that it be used in this condition. If the alloy is welded either initially or in subsequent repair, it should be solution heat treated after welding. The solutioning treatment should consist of heating at 2250°F. for one hour followed by rapid air cooling or water quenching. In order to prevent overheating, which impairs corrosion resistance, good furnace control is essential. Pickling or descaling operations should be performed before heat treatment.

General Information

It is possible to straighten HASTELLOY alloy C pipe. Bending should be done cold.

If plant conditions differ from those in which the HASTELLOY alloy C pipe is known to give good service, consultation service and corrosion samples for testing are available through the Haynes Stellite Company. Also, more detailed information concerning the fabrication and heat-treatment is also available.

CHAIRMAN SAUCHELLI: Thank you, Mr. Campbell. I'm sure those are helpful suggestions to many.

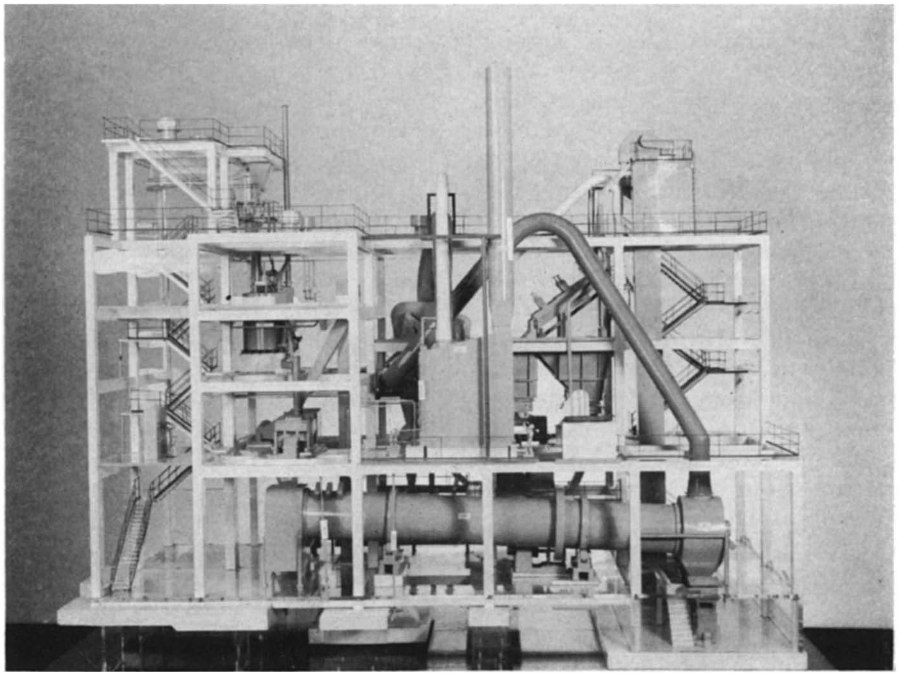
We have to go on with our program. The time is pressing, and at the end of the session, if there's some more time left, why, we might resume our discussions from the floor.

We have on our agenda an item: Models Replace Blueprints. All of you have had a chance to see the beautiful models here. Models today apparently are becoming very popular and as we learned yesterday, do-it-yourself is also a part of this modeling business and you can make models anywhere from twenty dollars up to ten thousand dollars. We have Mr. Weber of the Dorr-Oliver Incorporated to introduce the subject of models versus blueprints and he has an associate from the company, Mr. Kulp, who will devote about ten minutes time on telling us something about the subject. Mr. Weber.

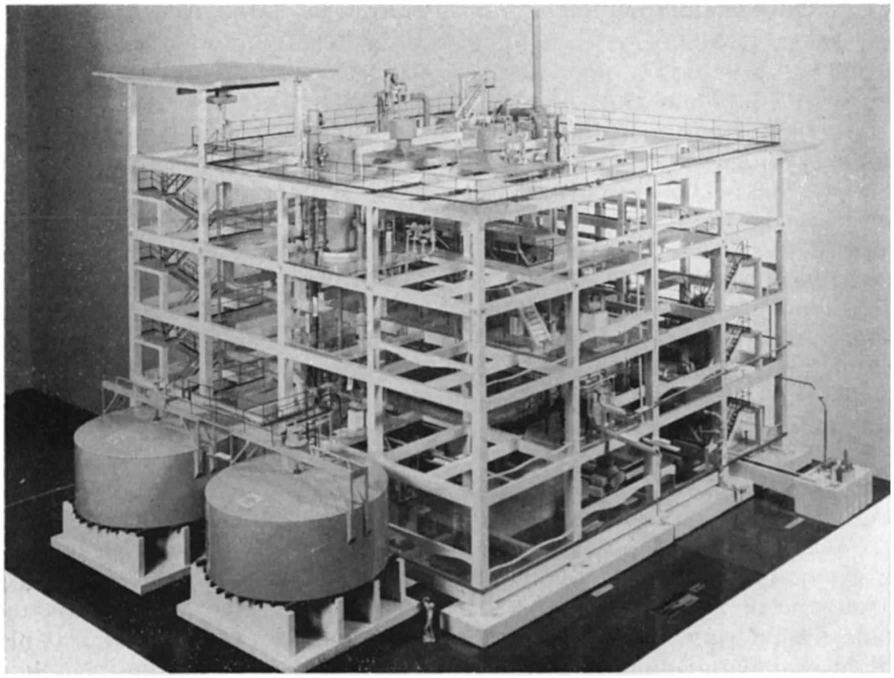
MR. WM. C. WEBER (Dorr-Oliver): I though I'd just tell you a little bit about these models before Mr. Kulp goes into their usefulness. We have here two models which we thought might be of interest to you when we brought them down here.

The one is a model of a phosphoric acid plant including evaporation, and the other is a granulating plant of the Door-Oliver type. This particular one is for making granular triple superphosphate, but the same system with relatively minor alterations is used for making ammonium phosphates and ammonium phosphate or triple superphosphate based, complete fertilizers. I think that as time goes on there is going to be a very definite trend to plants of this type. I think many of you gentlemen in this room will in the not very distant future be thinking of going into phosphoric acid and water-soluble fertilizers base don ammonium phosphates entirely.

They have many advantages economically and from what I have been able to hear about the problems in granulation, granulating problems are much simpler, because your processing and your formulations are quite independ-



Granulation Section



Reaction and Filtration Sections

ent of your granulating problems. Your granulation is not at all complicated by the processing of the formulation.

I think you will be interested in looking at these models. I'm not going to try to describe them or describe the processes. We have some reprints up here of a paper which I delivered in Stockholm to the International Superphosphate Association which describes our granulating process and technique and its rather broad usability and I would commend that paper to you to get an idea of this type of technique.

Our primary idea in coming here today was to possibly explain how we are using these models. We have made models of many of our plants in the past just for the purpose of illustrating what the plant is going to look like to our clients and also using them for training operating personnel. Many times in the case of plants for foreign territories they require models to show the VIPs. This one is somewhat in that category. These two units here of a very complete plant that we are building are going to Mexico, a triple superphosphate plant.

Mr. Kulp of our company will explain to you how we have found the models are a great aid and a great saving in the design of the plants and in the ultimate initial operation of the plant.

I'll turn this over now to Mr. Kulp.

MR. R. L. KULP (Dorr-Oliver): Over the past several years Dorr-Oliver has been investigating the use of three dimensional models to design and engineer chemical process plants. As we have come to understand model technique, there are several distinct types to be considered. First there is the preliminary study model which can be built after only a basic flow sheet and approxmiate equipment sizes have been developed.

We regard this type of model as being particularly valuable to those members of the design crew who have rather limited experience with the particular type of plant under consideration.

The model is built from a do-it-yourself kit. This model should be simple in construction, flexible in three dimensions and capable of quick alteration. Proper components will accommodate rapid changes in base sizes, floor elevations, location of ducts and so forth. Scales are optional but one quarter of an inch is quite usual.

In this phase of the work the various members of the design team, process, structural, mechanical and electrical engineers, have a visual aid to help them develop an optimum design, and before it is dismantled the major layout decisions should have been made.

Then there is the piping design model which is normally on a larger scale, perhaps 3/8 of an inch or 1/2 inch. This involves buiding a basic model from the structural and equipment drawings and making revisions as necessary on the model.

The piping designers then lay out the piping on the models and show all the important parts, values and instruments. The next phase is to have the piping designers make isometric sketches for each pipeline shown in the model. These sketches would then be turned over to draftsmen who would provide an isometric spool sheet for each pipe shown in the model, and pipe drawings as such would not be made. The spool sheet would contain the following information: configuration of the pipe, lineal dimension of each component leg of the pipe, location of all fittings, instruments and tie-ins, erection dimensions such as to permit the erection of the pipe without additional drawings, and builder materials.

Among the principal advantages that we see for this type of model are as follows: piping interferences — the possibilities of piping interferences are greatly lessened, the chance for locating inaccessible valves, manholes and instruments is greatly reduced, piping and fittings can be saved, which is not only somewhat of a saving perhaps in the piping itself, but it can reflect savings in maintenance as well as savings in power, because of a reduction in the number of fittings and less pipeline friction.

Then there is another advantage: location—several other advantages—there's the location of lifting devices which can be more suitably located, lighting can be accurately studied, better use can be made of the space available, safety hazards can be avoided, training of the operators is expedited, and models make it possible to understand the piping arrangement for those engineers who have difficulty in visualizing piping from conventional type drawings.

Then there is a third type of model, which is what they call the training model, built from check drawings. The primary purpose of this model would be for operator training, and it has no function in the design stage of the plant. Unless a poor checking job has been done in the drawings, this model should reveal few serious errors.

Of course, as with anything else, there are some disadvantages to using the model technique. One thing, they require space allocation in the drafting area and they do not file quite as easily as drawings do and they depart from tradition in the sense that they require thinking on the model rather than on paper.

Thank you.

CHAIRMAN SAUCHELLI: Thank you, Mr. Kulp, and Mr. Weber. They went to considerable expense to bring these models down here for our benefit and we certainly appreciate their efforts, and I want to say this: we certainly appreciate the cooperation of all the speakers in respecting their time allotment; that makes it possible for us to carry through with our program.

The next item is a very nice panel on semi-granular mixtures and it's going to be monitored by both Mr. Spillman and Mr. Reynolds. I turn this meeting over to them.

(Mr. Spillman assumed the Chair.)

CHAIRMAN SPILLMAN: Your committee has had a good many letters, telephone calls and questions on semi-granulation. Tnere seems to be considerable interest in semi-granulation. The summary of some of the questions sent to us by members of the Round Table ask for information on mechanical condition, how long can we keep semi-granular materials in bags in storage, something about formulation material costs compared with good granulation formulation costs, something about smog problems from ammoniators, equipment cleaning problems, cooling techniques necessary, approximate screen fractions of finished product, recommendation as for good formulation, and something about the good and bad features of manufacturing semi-granular mixtures.

I don't know whether we can answer all their questions today, however we have a good committee here to answer these questions and they consist of Mr. T. R. Schmalz of the F. S. Royster Guano Company, Mr. George G. Walton of the Tennessee Corporation, Mr. Joe Sharp of Spencer Chemical Company and Elmer Perrine of the Nitrogen Division. Our first speaker is Mr. Elmer Perrine.

Semi-Granulation of Fertilizer

Elmer C. Perrine

IT is rare that a great advance in industry quickly obliterates the thing that has been surpassed. Often the public gains much through improvements of the old product which raise it to a respectable level, perhaps to carve its own niche in the new order.

This is observed in the fertilizer industry where the great development of granulation has been followed closely by considerable advancement in the physical condition of fertilizers by means other than through granulation. Some of the things which became fundamental in granulation, such as reducing moisture and temperature in the storage pile, have been adopted with equal success in the storage of ordinary pulverized fertilizer. Even liquid phase, which has been the private domain of granulation, has become a controlling factor in a process which seeks to approximate the quality found in granulation at very low initial outlay for equipment. Semi-granulation is one of the developments at a respectable intermediate level between good pulverized and good granulated fertilizer.

If anything is unique in semi-granulation, it is the realization that perfection of product in this field may be too costly for some operators, combined with the realization that some popular grades are fairly easy to granulate at an acceptable level below perfection.

When semi-granulation first attracted much attention, there was some concern by those who had committed large sums for all-scale granulation, that their plants were being rendered obsolete before their time. Although the line is not sharply drawn, some of the limitations of semi-granulation accumulate against it as tonnage increases to render semi-granulation finally more costly or otherwise less attractive than full-scale granulation at about the tonnage level where full-scale granulation itself starts to become attractive for its several special merits. In fact, there may be a gap in the fields rather than an overlap of dominion.

The liquid phase that establishes a desired physical condition for granulation is attained by a balance among favorable ingredients, heat, particularly in the mixer, and moisture; it being possible to vary any of these factors to meet changes in the others within limits. Thus far these are the same conditions that are used in semi-granulation and the similarities start to end there. Beyond the facts of liquid phase, the flexibility of full-scale granulation comes into play and some of the limitations of semi-granulation make themselves known.

In the 1957 Fertilizer Industry Round Table, there was general agreement that the temperature of the product in the mixer should be at least 200°F, and many operators preferred temperatures of 230°F and a few 240°F, when it could be reached. Where high costs of ingredients and operating difficulties exist in semi-granulation, they often stem from the fact that high heats must be obtained in the mixer with low moisture levels, since all of the granulation must take place in that one machine on a one-try basis. Few of these operations are geared to recycle, although some operators are discovering the control that is made possible through some recycle. Recycle would further compound the problem of providing adequate heat in the mixer.

Semi-granulation could as well be termed "Minimum equipment granulation." It requires merely that sulfuric acid be added to the mix usually in conventional rotary batch mixers through a separate distributor pipe about duplicating the design of a good ammoniating distributor pipe. The flights of the mixer should preferably, though not necessarily be reduced in depth to about 12 inches in the one-ton and to 14 inches in the two-ton mixers. Refinements of worth are the use of meters replacing measuring tanks, particularly if one man is to manipulate the mixer as well as supply the liquids. Means should be provided for channeling off the considerable fumes sometimes released. One way to promote this is to have the discharge door remain open at least one-half inch during the mixing.

Heat and moisture are twin plagues in stored semi-granulated products as surely as in fully granulated ones. The cost of the minimum equipment for semi-granulation is very low, chiefly because no equipment is provided for drying or cooling. As a result, a very important difference in the products is the fact that much semi-granulated fertilizer is stored at and sometimes above the maximum water and heat content acceptable in full-scale granulating plants.

Due to the very high temperature of the product when made, there is considerable heat loss enroute to storage from the evaporation of some moisture and from radiation. When there is much drop through the air, this cooling may be considerable. Most operators find it necessary to recirculate the product through the system at least once, sometimes more, within one or two days, to reach a reasonable level of water and temperature content for storage. Some are finding it advisable to avoid semi-granulating in hot weather, as these means of cooling and drying are then inadequate. To cope with these problems, some operators are adding one rotary kiln without artificial heat which dries from the contained heat in the product as well as doing some cooling. However, one pronounced merit of semi-granulation is its freedom from any costly equipment being added to the normal ammoniating plant. On the other hand, the semi-granulating system may also be the nucleus around which a full-scale granulating plant may later be asssembled, without discarding much costly equipment.

One system that used a rotary dryer-cooler kiln without heat but with forced cooling air was semi-granulating a 12-12-12, with the product leaving the mixer at 200 to 210°F and leaving the dryer-cooler at 115°F and 2.5 percent moisture, both being about the maximum for lengthy storage. About the same temperature and moisture relations existed in another plant producing an 8-16-16, which obtained its cooling merely by rehandling, there being no cooler or dryer.

Economic considerations in any system justify crowding it to the maximum of its several features; moisture and temperature in storage are the first to suffer, unless getting granulation at all be the major problem in overcrowding.

What are the features of semi-granulation equipment and products that permit a system almost devoid of supporting equipment to compete with the highly-developed granulating systems with their necessary array of costly and effective equipment? Although the products may compete in the same market, the two processes by their very nature are applicable to such tonnage differences for efficient operations as to place the processes themselves out of competition with each other.

Semi-granulation must select a few favorable formulae. In the absence of externally applied, low-cost heat, it must obtain some heat through a means that often becomes costly. Those B.T.U.'s of heat generated by the action of acid on ammonia could, under some likely circumstances, cost from 3 to 8 times as much as the same B. T. U.'s of effective heat derived from fuel oil or gas used in rotary dryers.

With rare exceptions there is no recycle, so the product is "made to grade" in one trip through the limited equipment. Although recycle sometimes becomes the curse in full-scale granulation, its use may make possible the granulation of difficult formulae.

The grades of fertilizer that are easily and economically granulated, or semi-granulated, often have the disturbing quality of being hygroscopic by reason of the large amounts of contained salts. A development by the semi-granulating cousins could well be studied for possible adoption by the full-scale granulating fraternity.

This development results in a coating or lamination over the pellets of a material which has limited moisture pick-up potential. Finely divided superphosphate ammoniated from inside the pellet after the coating is applied is the method employed. A person should not be banned as a cynic if he promptly remarks "Nice work if you can get it." The process is simplicity itself where the rotary batch mixer is used.

Up to about 200 pounds of the superphosphate is withheld from the batch while all of the other activities of semi-granulation are consumated in the mixer. Formulation is planned to have some of the ammonia escaping from the pellets as the withheld superphosphate is admitted to the batch. To the extent that pelletizing is essentially completed before the withheld superphosphate is added, this maneuver provides a coating, or lamination that has improved the storage qualities of the fertilizer. This coating, tenuous as it may be, is still stable enough to withstand the subsequent handling in most semi-granulating systems. This may not be true for the considerable mechanical handling through the dryers, coolers, screens, and elevators of the full-scale granulating systems.

Some of the things which granulating plants perform for themselves are performed for the semi-granulating plants by the suppliers of materials, for the basic requirements for any of the systems must be served somewhere along the line. As the ability of semi-granulating plants to remove water is severely limited and is further subject to the whims of weather, it is almost mandatory that they use superphosphates, nitrogen solutions, acid and even any filler of the lowest water content, sometimes at increased costs.

Since all of the forming of particles is usually to be done in the mixer batch-wise, with only one try within a narrow period of time, the skill of the operator largely supplants any influence that continuous, automatic operations may have, such as continuous ammoniating, drying, cooling, screening and recycle.

Most operators quite readily acquire the needed skills to augment the equipment and controls. Good results in nitrogen retention and product quality were obtained through the following procedures in semi-granulating an 8-16-16. All of the nitrogen was supplied by a solution which contains 29.7% NH_3, 64.5% ammonium nitrate, and only 5.8% water, one of the driest solutions. This supplied 100 pounds of ammonia which, when reacted, generates about the minimum amount of heat which many operators believe is required for good semi-granulation. Enough 66°Be sulfuric acid was used (90 pounds) to produce at least 200°F temperature exit the mixer. This, fortuitously, was only the amount of acid required to neutralize the ammonia, thereby holding the costs of ingredients to a low level, a thing not always pratcical in every system. About one-quarter of the nitrogen solution was applied to the mixture of all dry materials through its separate distributor pipe, followed by the acid and then by the balance of the nitrogen solution. Variations of this procedure are also used effectivly, but they show that some ability of personnel is required.

Some operators in all types of granulation will diverge from reasonable practices to such an extent that all of the ammonia could be neutralized by the very excessive amounts of sulfuric acid being applied without asking the superphosphates to perform any of this service. This carelessness is wasteful and may become dangerous in any system, and the difficulties that led to such abuses can usually be identified and overcome by simple means.

Economy of materials and nitrogen retention are factors to be considered as well as cost of equipment and tonnage involved—when appraising granulating systems. Fully-equipped granulating plants should be able to use sulfuric acid effectively without wasteful excesses and many of them do. They should also be able to hold nitrogen losses lower than would be expected of semi-granulating units and some do. Where they fail to excel the semi-granulating systems in these respects, and many fail to do so, the great outlay of capital may be of questionable value, unless possibly the greater tonnage potential per day and the generally better storage quality are worth the outlay.

In 10-10-10 and 12-12-12 and *even* 5-1010 on occasions, there is a loss of .5 unit of nitrogen and sometimes .75 unit in conventional granulating equipment. There have been times when premature granulation, probably developed through excess liquid phase for some reason, resulted in nitrogen loss that reached more than a whole unit from seemingly safe formulae. This loss is serious where it develops in formulae that derive relatively small amounts of nitrogen from solutions and where all of this loss is presumed to be from the solution nitrogen. Good nitrogen economy can be achieved in granulation systems. The table shows formula and wide screen sizes for one grade made in a semi-granulation system. The screen sizes were determined after on a months pile storage.

Semi-Granulated Fertilizer 10-10-10
Nitrogen Formulated 10.5%; Analyzed 10.1%
Sample from 400 Tons production
After Rehandling Once and One Month Storage

Tyler Screen Through	Tyler Screen On	%	Formula	Lbs.
	4	2	66°Be H_2SO_4	120
4	8	10	Nit. sol. (8.2 units N)	350
8	12	13	47% N, 29.7 % NH_3	
12	20	18	64.5% NH_4NO_3, 5.8% H_2O	
20	32	15	Superphosphate 20%	1,000
32	42	16	Sulfate of ammon.	220
42		26	Potash (Fine) 60%	335
		100	Total	2,025

Water in formula 4.4%; in storage 2.1%
2 ton batches in 2 ton rotary mixer
12 batches (24 tons) per hour
Temperature; Exit mixer 240°F
In pile when made 180
After rehandling once 110
Cooler; countercurrent rotary

Influence of Rehandling on Particle Size of Semi-Granulated Fertilizer 8-8-8

Temperature: Exit mixer 236°F
" In first storage pile 186°F
" Rehandled after 8 hours storage 142°F
No mechanical cooler. Average atmospheric temp. 55°F

Tyler Screen Through	Tyler Screen On	Pile Exit Mixer	Rehandled Storage Pile	% Changes Decrease Increase
	4	.75%	1.00%	—
4	8	11.00	6.25	— 43.2%
8	12	32.50	14.50	— 55.4
12	20	32.50	23.50	— 30.7
20	32	* 12.00	16.50	+ 37.5
32	42	(7.00	* (17.00	+142.8
42		(4.00	(22.00	+450.0
		— 99.75	100.75	

* increase in "Through 32 and 42 mesh totals 354.5%

Semi granulation has found its place among operators who wish to produce limited tonnage of grades that are easily granulated with better storage and handling qualities than found in much of the ordinary pulverized fertilizer and who are willing to take some calculated risks since moisture and temperature are not under complete control. These operators must be willing if necessary to cease operations of this system in hot weather. Semi-granulation is a favored method for those who do not see sufficient tonnage soon to justify going all-out with full scale granulation to very nearly overcome all of the condition troubles. About 40 plants distributed widely over the fertilizer producing areas are now engaged in this practice, and within the limitations discussed and often beyond they are giving satisfaction.

The more sure way of solving the problems of physical conditions in fertilizer through full-scale granulation imposes burdens that their tonnage may not justify.

CHAIRMAN SPILLMAN: Thank you, Elmer, for that very fine discussion.

Our next speaker is Mr. Schmalz who will discuss semi-granulation using the rotary mixer.

Semi-Granular Fertilizer Production In A Conventional Rotary Mixer

T. R. Schmalz

IT is certain that there are others here who are more qualified to air this subject than I. Nevertheless, some of the more basic concepts of this subject can be covered without extensive personal experience in this type operation.

We have had limited experience where semi-granulation is the object. The grades involved in these operations run from 4-10-7 and 4-12-12 to 10-10-10 and includes grades such as 5-10-10 and 8-16-16.

Fairly close particle size production must be maintained, since no recycle is used in this method of operation. The requisites for this uniformity of particle size are few but critical and are primarily controlled by the raw materials. The grade to be manufactured naturally has a great deal to do with the characteristic of operation but some basic considerations can be discussed without considering the specific analysis. For instance, where potash is concerned it is safe to say that best results occur when coarse potash is used rather than fine and the product appearance improves with increasing potash requirement. In the same respect superphosphate does a much better job of covering and binding the particles of potash than does triple. Sulfuric acid plays a leading role in this operation and is used to react with ammonia from solutions and to elevate temperature in the mixer to promote plasticity and drying.

Varying amounts of acid are used, ranging from 80# of 66° Be acid per ton, all of which is in excess of that required for ammonia absorption, to 130# of 66° Be acid all of which is needed for ammonia reaction.

I think it can be said that if less than 80# is used too many fines will result and probably not enough drying will take place, and if more than 130# is needed, too much free ammonia will escape the mixer and oversize particles will become unreasonable.

Solutions recommended so far have been of two types; one an ammonia-ammonium nitrate solution, the other an ammonia-ammoniu mnitrate-urea solution, both of low water content. It appears that for grades with nitrogen requirements of 4% to 6% the urea containing solution with its greater solubility is best, for higher nitrogen requirements the ammonium nitrate solution is probably better.

Thus far we have not used coating agents for conditioning, but this does not preclude the possibility that they could be beneficial for stored goods.

Product moistures have been about 3% ± 0.5% in all grades made by this method.

Mixer arrangement is by no means radically different than normal. A good exhaust hood and stack at the mixer discharge and a good tight mixer are desirable since considerable volume of vapor is generated by the high temperature in the mixer. Distributor pipes should be as long as possible and with good distribution patterns for optimum results. Poor nitrogen recovery, non-uniformity of particle size, accelerated corrosion and flash fires all can be attributed to poor distribution. Extensive articles have been written on this subject relating to other production methods and the basic principles still hold true.

Cooling of the product is probably as important a consideration in this operation as the rest of the conditions stipulated. In our operations we have not experienced Phosphate reversion, even with pile temperatures which would cause reversion in conventionally made goods. This is probably attributable to the lower moisture content of the goods. However, for best results in minimizing particle breakdown, immediate cooling from the mixer temperatures of above 200° F down to around 120°-130° F, or lower if possible, are desirable. Cooling can be accomplished in several ways; the most desirable but most expensive is with a rotary cooler with an air handling system. Other methods which we employ are cascading over a baffle plate cascade and rehandling through the equipment after a short storage period of from 12 to 48 hours.

We have had relatively good results from this operation from the analytical and condition aspects as well as from appearance considerations and hope to improve with experience.

CHAIRMAN SPILLMAN: Thank you, Mr. Schmalz. I shall now turn the meeting over to Joe Reynolds.

(Mr. Reynolds assumed the Chair.)

CHAIRMAN REYNOLDS: We will proceed along here to the other members of this panel.

During the transition from pulverized to granulated mixed fertilizers many attempts have been made to adapt existing equipment to fulfill this need. A process of considerable merit has been developed which approaches granulation with minimum changes to equipment. The Eyman process has combined a revision of raw material specifications with a slight modification of equipment to achieve granulation. We are indeed fortunate to have Mr. George Walton, General Manager of Midwest Division, Tennessee Corporation as our speaker today to discuss the features of the Eyman process.

The Eyman Process

George Walton

I want to express my appreciation to the members of the hard-working and efficient committee for inviting me to talk about this process which we have installed at our Lockland, Ohio plant. Instead of calling our product semi-granular, we have chosen the name grained as being more descriptive. I am quite aware that Webster would find the word "grain" and granular pretty much synonymous, but for some reason the word grained has not been used among all the pelletized, pebblized, granulated, granulets and so on, and I'm hoping it will convey a somewhat different product.

The reason we chose a name that was not that of semi-granular is that in too many instances semi-granular simply means that you have the old powdered fertilizer with enough granules to take it out of the class of the conventional fertilizer as we knew it, but that semi-granular fertilizer still retained all of the disadvantages of poor storage, poor graining qualities, plus the added disadvantage of — and here we get that word again, "segregation". Anybody who tries to handle a semi-granular fertilizer over any kind of a conveyor system will find, as pointed out this morning by one of the speakers, that when you drop it on a conical pile the outside of your pile is a beautiful granular fertilizer, the center of the cone gets all the fines. The farmer gets one bag of granules and one bag of fines which doesn't make him any too happy.

That semi-granular product is usually made by introducing solutions and acids in a whirling mass in a conventional mixer. Now it takes a brave man to get up here and appear to contradict some of the statements made by such an expert as Elmer Perrine and if I appear to contradict some of those statements I hope I will be able to point out that the system we are talking about varies somewhat from the processes he was referring to.

Our grain product I can absolutely state is dust-free. It has a much wider size range than any of your fully granulated products and it is comparatively free of the objections that I mentioned about the semi-granular products; that is, poor storage qualities, poor drilling qualities and segregation to a large extent.

I must say that I'm indebted to Mr. Louis Eyman of the North Ag Chemical Company, Forest City, Iowa, for permission to describe this process to you since he has a patent-applied-for on it.

The initial installation as we have it—as we had it at Lockland, followed in detail the layout of his mixing unit. We've since made some changes which we feel have given us an improved product and increased production.

The mixing and graining is accomplished by batch system in a one and a half ton Atlanta Utility mixer — and I'm not being paid by Atlanta Utility for advertising — you could do the same thing in any other conventional mixer, in which all of the flights have been cut down to within five inches of the shell excepting in the last 12 inches at the exit end.

A sparger of somewhat similar design to that in use in a TVA amoniator was installed to permit introduction of solutions, acid, anhydrous ammonia and water below the rotating bed of solids. In other words, the solids are not being whirled as they are in a mixer as you buy the mixer; you cut some of the flights, you see, and you get a conical rotating bed such as you get in the TVA ammoniator.

A mixing cycle of three and a half to four and a half minutes is maintained depending upon the grade and the formula used. The mixer discharges to a cooler. In our case the discharge is to two coolers approximately six feet by twenty feet each, not because we think that's any better than a six by forty, but we just happened to have them on hand. I think that's as good a reason as any other.

Originally the cooled product was sent directly to storage, but we found that there was enough oversize—in other words, plus four mesh standard screen size—to create dust when reduced to size in the shipping mill. We therefore introduced a scalping screen to take off the oversize material and return it to the mixer, as is done with both oversize and fines in your conventional granulating systems.

We found that this recycled material being virtually inert on the second trip through the mixer helped in the graining process and increased our through-put of satisfactory size product. In other words, though we took part of it and recycled it we actualy got better production, particularly in the high nitrogen grades like 12-12-12 or 10-10-10 where over-agglomeration is frequently a problem.

No attempt is made to eliminate the minus 20 mesh material in our process with the result that our product contains a much larger percentage of the minus 20 mesh fraction than is found in the granulated pelleted or pebbled or whatever you want to call them fertilizers that are now on the market.

However, when sufficient care is exercised in shipping to avoid good product sized material being sent to the mill when it should have passed through the screens into the bagging hopper the grain fertilizer will contain less of the minus sixty fraction than the product of your conventional granulating systems.

In other words you will have very little fine dust; practically none.

The granulating processes using driers produce pellets with an anhydrous coating which by abrasion between particles is rubbed off—I had the word "scuffed off" and my secretary said she couldn't find the word "scuffed"—almost fume-like irritating dust is the result.

In our graining process the drying is accomplished by heat of reaction so that the surface of each of the granules contains a certain amount of the liquid phase or moisture, which acts as a binder.

The question, naturally arises, without artificial drying can we use as much solution in our formula as those who do use artificial drying. I've heard statements, and Mr. Perrine just made it previously, that some of the operators are getting ten uits of N from solutions in 10-10-10 and 12-12-12.

I've seen a great many more who don't attempt to go above 8 units in either of those grades. As a result, I don't feel too disappointed that we regularly use 7 and a half units from solution; that's solution 490 we are using.

Bear in mind we don't have the cost of fuel for drying and a lot of the other expenses that are incurred in full granulation such as power, maintenance and so on.

Also I firmly believe that if and when we get to the point where we can do some experimenting with some of the more concentrated solutions that we can probably introduce higher amounts of solution. In other words, I firmly believe our product will stand more ammonium nitrate than we are now putting in.

The advent to the market of diammonium phosphate has further narrowed the margin there of saving between solution and solid forms of nitrogen in the higher analysis grades, that is, in addition to its being a decided aid to graining, the slight amount of savings in formula costs between the conventional granulating systems and our process is therefore much less than the difference between the operating and maintenance costs of the two systems, to say nothing of the difference in the original investments, which is very considerable.

I might tell you that I figured originally on the full granulation system and got the okay to go ahead with it and then we investigated the Eyman process and figured that that would take the place of it.

This graining process is most successful when a preponderance of the solids fed to the mixer is in granular form although some fines, as Mr. Schmalz pointed out, in either the phosphates or potash appear to be beneficial to act as binders and coaters.

In the absence of granulated normal super those formulas which are high in triple super and potash, both in granular form, yield a bigger percentage of coarse grains than those of low potash content made with non-granulated normal super. Depending upon the formula used, from 80 to 90 per cent of our product as made will pass a standard four mesh and be retained on a 20 mesh screen; excepting in the cases of 3-12-12 and 10-10-10, virtually 100 per cent will be retained on a 40 mesh screen. That's far from being dust.

For some reason, which we haven't yet determined, we have difficulty in agglomerating 10-10-10 and usually wind up with 5 per cent to 8 per cent passing the 40 mesh screen. That formula has given us some trouble.

Now I won't elaborate on the 3-12-12. To be honest we don't give a darn about graining 3-12-12. It's been put on the market at such a price that we try to discourage the use of it, as a matter of fact. (Laughter.)

That's the economics of it.

The absence of dust, that is, of minus sixty mesh fraction is to my mind due to the agglomerating effect of the high heat and the steam present in the mixer. You see the steam is held under pressure in our mixer. Of course we do have a release valve so we don't build up too much pressure. We haven't had an explosion yet.

One noticeable advantage of the system is the greatly reduced amount of fume and dust as compared with the TVA ammoniator and drier. Again, this is probably due in part at least to the fact that mixing is carried on under steam pressure of in an airswept vessel as in your TVA ammoniator.

The other obvious advantages are the exceedingly small capital outlay. Anybody with a mixer and conveyor and a cooler can go into it, and you have savings in maintenance and operating costs.

Your conventional granulation system requires a tremendous amount of power.

I believe that's about all I can think of now that might be of interest to you. To my mind it is like all granulating systems, more of an art than an exact science, and as such is successful in direct ratio to the expertness of the operator.

Even your conventional granulating system is far from being a scientific method. I think if you will recall, I believe it was Mr. Hill, pointed out that your raw materials have stratification, which means that they are not in the same physical condition every time you feed—all the time while you're feeding to the mixer and actually even in the TVA ammoniation system your operator is the key to the whole thing, in my opinion.

Some of my old friends in the fertilizer industry and opponents at the poker table have said they are going to bombard me with some questions. I'm going to fool you. This was an idea of mine, but the actual work was carried out by our chemical engineer, Mr. Lanier, so if you get me out of my depth, he's here to answer those questions and I'll call on him.

Chairman Reynolds: Thanks very much.

We're going to hold these questions till the end and then the four speakers will have a chance to get this bombardment.

The design features of a liquid distribution system are extremely important in the efficiency of ammoniation and granulation. Improperly designed distribution systems promote localized, over-ammoniation; P_2O_5 reversion; and ammonia loss.

In the case of granulation, over-agglomeration of granules and mixer build-up go hand in hand with improper liquid distribution.

Spencer Chemical Company has developed a distribution system termed a "Block Sparger" which was greatly assured an even, steady flow of liquids into the raw materials. We are very fortunate and pleased to have Mr. Joe Sharp, Director of Technical Service of Spencer Chemical Company, to discuss the features of the "Block Sparger."

Spencer Block Sparger

J. C. Sharp

My part on the program as I understand it was on semi-granulation, too, so I'm not going to leave out anything I've already written down. I'll go ahead and talk about it some and then at a later time discuss the Block Sparger.

Under semi-granulation, in order for any material to agglomerate it won't unless there is a wetting medium or liquid phase present. Water or moisture is a basic ingredient of liquid phase; although other liquids are used in formulas they are converted to solids through chemical reaction and the materials are brought together. The water present plus the desired salts constitute the liquid phase.

For example, if there's a hundred pounds of free water in a ton of fertilizer and it has dissolved in it 200 pounds of compounds, soluble salts, then you have 300 pounds of liquid phase present.

Temperature has a great deal to do with liquid phase because most fertilizer salts are more soluble in hot water than they are in cold. There's more to it than just getting liquid phase; you must have some materials present that have some plasticity or else it won't agglomerate. You can't agglomerate potash by itself, dolomite by itself or sand. By having some of the plastic materials you can bind those materials along with it.

Probably semi-granulation in fertilizer first occurred in the manufacture of superphosphate. This was caused by the handling of wet superphosphate from the reaction and this was not a desirable result. It made the product of ammoniation difficult and usually also because it was rather hard and difficult to grind, and since we waste P_2O_5, purging was necessary quite often.

A well known nitrogen producer pointed out the advantage of granular properties of ammoniated goods in a company publication copyrighted in 1945. He may have pointed it out earlier than that, but this is the oldest one I could find. He had this to say:

> "A discharge from the mixer fertilizer ammoniator with nitrogen solution is warm and somewhat moist to the touch. When cooled it becomes dry and friable, the desirable granular properties.
>
> "The ammonia nitrate contained in nitrogen solution is an excellent agent for promoting granulation, tending to agglomerate the objectionable fine particles and thus reduce dusting and sifting of materials through the bag.

—The bags have changed, too.—

> "Under some conditions fertilizer made with nitrogen solution have pronounced granular characteristics."

When this publication was issued the author could recommend three per cent ammoniation. It seems pretty tame after yesterday's discussion, doesn't it?

The greater advancement into semi-granulation came as a result of new fertilizers in the fertilizer industry in the late 1940's. Prior to this time the farmers in the Midwest got their plant foods mainly from triple superphosphate and ammonium nitrate. Potash was not used at that particular location to any significant extent.

Accustomed to these concentrated materials when the fertilizer plants did start to appear out there, they met quite a bit of resistance in selling low analysis grades to the industry. In producing high analysis fertilizer to satisfy the demand, using nitrogen solutions with salts of nitrogen, the solution phase just happened to be almost ideal for the agglomeration.

The original work on these grades, namely 10-20-0 and 9-36-0 was carried on in a small plant with a one and a half ton batch mixer. Because this slow method of production was used, quick cooling was not hard to accomplish which resulted in forming fine granules with relatively low moisture content. Many of these grades were made in the two ton batch mixer with almost no means of aerating or cooling. The pellets were soft and soon turned into mud. Reversion was terrific although the ammoniation rate was only slightly above three per cent.

After necessary steps were taken to cool the product quickly, as soon as it left the mixer, this plant was also able to make a product with good qualities, almost satisfactory quality, we might say.

Many mixers started producing these grades but were not well pleased with the storing qualities of the product when bagged. This led to the installation of experimental type driers to remove moisture so that these products could be stored satisfactorily.

The first drier, I believe, was a used alfalfa drier which lasted one season. It wasn't too long after this experience before continuous ammoniators came into being along with screens for classifying. This, of course, led to complete permeation.

In order to produce semi-granulation to an acceptable extent in a low analysis grade, if we don't just normally have the right amount of liquid phase, it is necessary to add acid, and generally sulfuric acid is the one that's used.

Proper aeration greatly enhanced drying and the properties of the fertilizer were better when sulfuric acid was added to the mixture, but there were other problems that came up. The reaction between sulfuric acid, potassium chloride and nitrogen solution can produce quite a smog. This smog settles to the ground and harasses neighbors, and then they start a little harassing all their own.

The method of introducing the acid in the nitrogen solution, as well as the amount of the liquid used, generally influence the volume of the smog forming.

Introducing these liquids as evenly as possible and in a manner in which they are diluted with other material before coming in contact with each other is the best way to reduce this smog. What this is really saying is eliminate local hot spots with even distribution and allow some time for mixing of the liquids with the solids before the liquids come in contact with each other.

Now, regarding cost, the grades that require sulfuric acid to neutralize excess ammonia from the nitrogen solutions, the formulae are generally lower when making the same grades without the use of the acid. If there is more excess free ammonia when all the nitrogen is derived from the solution, the formula cost is generally increased by the cost of the acid required to bring about semi-granulation.

Now the Block Sparger was developed by the technical department of the Spencer Chemical Company and it has seemed to do a very good job of meeting the con-

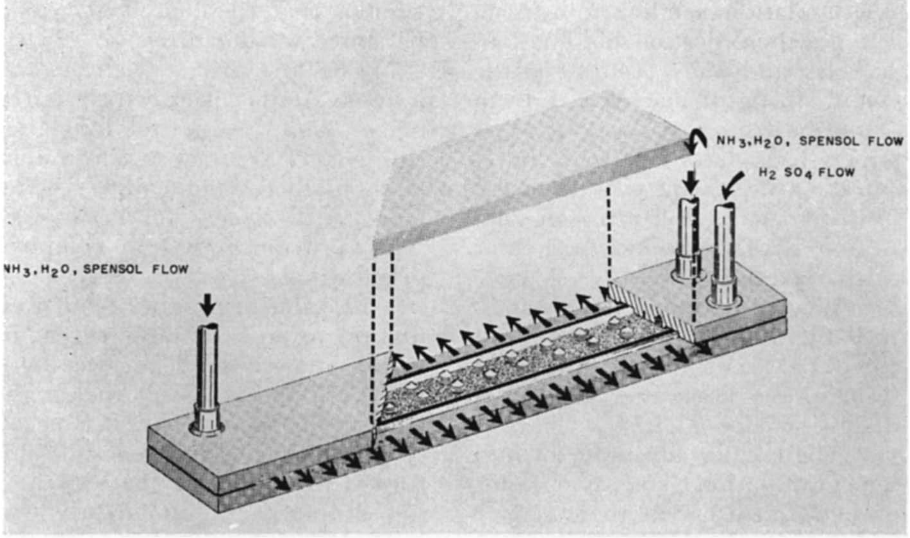


Figure XII - Continuous Ammoniation Block Sparger

ditions of minimum smog formation. Although the block sparger is designed for continuous ammoniation, it has been used successfully in a few batch mixers. Some day its use in a batch mixer may be dominated by the Eyman patent.

This shows an isometric cutaway of the block sparger. One side is for the acid and the other side is for the solution. This is the sulfuric acid side here and this is the solution side. There are grooves in these blocks; that's where it gets its name. They are three quarters inch from here to here by six inches and the length, of course, depends upon the length that you want.

There is a gasket that separates the acid from the solution, and since you are generally feeding more solution than you do acid, this side over here is machined down so that you get a separation greater than that the gasket itself gives you.

Question: What is your block made of, Joe?

Mr. Sharp: It's made of carbon steel, it can be made from any metal you want to make it from, depending on how much money you've got to spend for it.

I might say in this regard, since I've been to this meeting I've gotten one unsolicited testimonial, and this was used in a continuous ammoniator, too. It wasn't a batch mixer. They have been able to get 17,000 tons of production with the use of one block sparger made out of carbon steel. I asked this gentleman how much he had been getting out of the carbon steel spargers he had been using, and he said 1,500 tons, so that's quite a spread.

Chairman Reynolds: Thank you very much, Joe.

Without further comment from myself here, I'd like to turn this over to the discussion period. We have the four previous speakers at the Round Table just waiting for these questions.

Mr. Larry Lortscher (Spencer Chemical Company): I have a question for Ted Schmalz.

Ted, your talk yesterday indicates you've done quite a bit of work in determining what your nitrogen losses are in your various plants for various grades. Do you have figures that you can give us to compare in your rotary semi-granulation nitrogen losses versus your full complete granulation plants?

Mr. Schmalz: In that respect, I'd say empirical relations between the granular and the semi-granular operations are comparable. You would probably lose similar amounts of ammonia at the same ammonia levels. That's about all I can tell you on that.

Mr. Lortscher: In other words, no higher?

Mr. Schmalz: No, I don't think it's any higher.

Chairman Reynolds: Anyone else care to comment on that question?

Mr. Perrine: I want to take a little bit of exception to that because I think in the semi-granulation insofar as you are deriving quite a lot of your heat for the granulation from acid you are quite apt to have quite a loss of nitrogen. Where you have that tremendous amount of sulphuric acid you'll get sometimes premature granulation that closes off the superphosphate particles from further ammoniation. As uniform as we'd like to have these things, we know there's not 100 per cent uniformity. You'll have the possibility of some of the acid reacting on the ammonium nitrate that perhaps is a worse condition under semi-granulation than you'll have in the better regulated full granulation system.

The figure we showed here—I think that chart we had up there did not show the nitrogen—that's just one; but we formulated one at 10.5 nitrogen and it showed a 10.1 at a four tenths unit, but that was on about 8.2 units nitrogen from solution and that should be compared with a loss of perhaps the same amount of nitrogen from say ten units of nitrogen from solution in the full granulating which would represent maybe, oh, roughly, 20, 25 per cent more loss of nitrogen on a comparable basis. Now, maybe Mr. Schmalz was not taking that into consideration, when he made that statement.

Mr. Schmalz: I'm not going to take exception to it.

Chairman Reynolds: Any other experiences on semi-granulation that might tie into that question?

Mr. Charles Lubow (Star Fertilizer):

Mr. Walton, I am not a member of your poker table. You can accept that as a challenge, if you wish. (Laughter.) But I am in the fertilizer business and several years ago we were invited to Mr. Dudley George's plant up in Richmond, Virginia to witness the Eyman process. I think what they were doing there was using a type of superphosphate that was a little coarser than the balance of the pile. Now what superphosphate do you use?

Mr. Walton: In the Middle West, of course, most of our popular analyses are much higher than you'll find in the East or South. Consequently, the majority of our grades contain large amounts of triple superphosphate and we use either the granulated or coarse pile run triple superphosphate in our grades.

Does that answer your question?

Mr. Lubow: Yes, I think that answers the question, but I think here in the Eastern seaboard we have difficulty in finding that type of merchandise. To get a coarse grade from the pile, that's almost impossible.

Mr. Walton: Well, I could tell you where you could get it, but advertising is not allowed. (Laughter.)

Chairman Reynolds: I saw six hands go up in the back.

Any other discussion? I'm sure there are more questions.

Mr. J. Howard McNeill (Monsanto Chemical): I'd like to ask Mr. Perrine if he has any range of pH in this semi-granulation with batch formulas?

Mr. Perrine: No, I do not. Sorry to say. That's probably one of these refinements we're going to have to get into. We're kind of new in this. I don't believe we have it too well on the granulated grades which have been going now rather full blast for seven or eight years. Does anybody else have something to offer on that?

Chairman Reynolds: Any discussion on that question?

Any other questions? Discussion?

Mr. Paul O. Schallert (Tennessee Corporation): I do not understand how the block sparger is mounted in the mixer? Is it mounted by sparger arms inside the twins of the mixer?

Mr. Sharp: Well it's mounted just almost identically as a TVA sparger is mounted; supported the same way and I imagine there are probably a couple of dozen different ways to support them, but the pipes that you saw sticking up top connect with pipes that run down from the central support and of course that has to be well anchored with sufficient flat iron metal—some way to give it the strength to stay in there.

I don't think that it's any harder to mount than the other type sparger. I think it's about the same.

Mr. Schallert: One thing more on that. I wonder if you ever contemplate or tried making that out of carbon or graphite? Being as massive as it is you might be able to use those and still have enough structural strength for it to stand up and still withstand the corrosion instead of going to the Hastelloy C spargers.

Mr. Sharp: Well, we haven't done anything on that. It might be a possibility. We've never contemplated going any further than just having Hastelloy C lips for the place where the solution, or rather the acid—on your acid side would come in contact with it.

This thing hasn't had too wide a use yet. We've had quite a few throughout the country and they don't cost too much money actually, but one thing that I failed to point out when I was up there, is machining the face of this block to get an good even flow is most important. If you don't—it's a pretty much of a precision piece of equipment—and if you don't have it even then it just doesn't give you the proper distribution and the main advantage, I think—I believe you can say the main advantage, it gives a good pattern and I think that's a good advantage, — but I think the main advantage is that you have so much more space that can corrode away before it destroys the pattern of your sparger.

You have that metal there, of course, three-quarters of an inch thick, each block; then you have—I don't recall the dimension right now, but it's an inch and a half, maybe, between the groove portion of the block sparger and the outside lips, so you do have quite a bit of metal there that will have to corrode away before you lose your pattern—completely lose your pattern.

Of course, any time you begin to lose it, I'm sure that you begin to lose efficiency somehow. There's no question about that, but it's not completely destroyed.

Chairman Reynolds: Any other questions?

Mr. Warren A. Button (Freeport Sulphur Company): My question is directed to each of the gentlemen at the table.

Mr. Walton has indicated he prefers granular raw materials in the Eyman process. Is this true of the other semi-granular processes?

Mr. Sharp: Since it's close by, semi-granular is such a broad term, it just depends on what it takes to satisfy the trade. No one is anxious to do too much more than he has to to sell his product and go to too much extra expense. Certainly if you want to approach complete granulation as near as you can, then you need to start off with some granular products to begin with because you just don't have enough of plastic materials, you don't have enough control over liquid phase to bring about the proper conditions without starting off with a little help.

Chairman Reynolds: Do you have anything to add to that?

Mr. Schmalz: I've just got one small comment. We made a grade in the South, top dresser grade 20-0-20 which is a completely granular dry mixed material so you can get anything you want, as Mr. Sharp says, you can get full granular materials and not do anything, let somebody else do it for you, or you can start out fine and go through the grief and misery that you have to go through to get granulation. Suit yourself.

Mr. Perrine: If you're speaking of semi-granulation as the process rather than the product a method by which you change the physical shape and size of the products, I would say that you would want finely divided superphosphate in order to get the maximum amount of ammoniation there as we've observed about, I think, 30 mesh particle on superphosphate. Larger than that you would drop off very rapidly on granulation.

Then if you have semi-granulated particles there you will do one of two things; either build up tremendous large particles or failing that you will get—I'm not going to use the word "segregation" — we'll have a separation of particles there which detracts from the quality of product.

You can't ammoniate superphosphate very high. You're going to be running up the total cost of your product by the use of sulphuric acid usually.

Chairman Reynolds: I think all the speakers have mentioned modifying the flights in the mixers even to the point that a new mixer out on the siding is modified before it gets into the mill.

Do we have any comments from some of the mixer people? In other words, are we faced with having two types of mixers?

A Voice: I could take a crack at that if you want me to.

Unfortunately, quite often as you know, you gentlemen and our good customers and those of the other mixer manufacturers here expect these mixers to do quite a few things. No one here has brought up the ugly word of "dry bulk blending" and the numerous other things you expect this mixer to do.

We would just love to be able to design a flight which you could pull in and out like a Shick Injector Razor—but if we make them for the semi-granular process such as my friend, Mr. Walton—I say, friend, advisedly— if we make it that way and it discharges slower, which it will when we cut the flights off, then somebody says, "I can't get the doggone thing to unload," and if we bring them up then the flights are too high.

So all I can say is if you people can help us design a multiplicity of flights, we'll be happy to cooperate.

Chairman Reynolds: Robie, you were up also a minute ago.

Mr. R. E. Robinson (Atlanta Utility Works): I really don't have much to add. Possibly one thought —Mr. Walton did bring out very clearly that he trimmed down only part of the length of the drier cut off toward the intake end of the mixer, but he did leave his full flight, or essentially his full flight, at the discharge end and there are indications there, though, that that does revamp this mass flow pattern.

Now you can still adjust for that and still get good results, but we would just say this, be careful.

Chairman Reynolds: Another thing that was mentioned was this build up of steam and pressure in these mixers and I think there was some mention as far as relief valves and escape hatches and a few other things.

Does anyone care to elaborate on how that's accomplished?

Mr. Clarence B. Kirby (Allied Chemical Corporation): I'd like to say something about these flights we can get away from. I've had a little bit of semi-granulation experience—the best semi-granulation that I've done had a 16 inch flight; just room to get the pipes in there and I think that when we cut off the flights too much we have trouble in discharging and leaving material in the mixer that builds up oversize before the next batch.

Chairman Reynold: Thank you.

We've really started something it looks like. That's what we want.

Mr. John A. Stark (Cooperative GLF Exchange): I'd like to know if anyone has thought of screening the finer particles out of the semi-granulation and re-introduce them? Is this a good idea or is there too much segregation in small particles if you did it and if they haven't tried it, why haven't they?

Mr. Powell:
We have done quite a bit of work —we put a Tyler hummer after the three-quarter ton batch and screened out all the fines and reversed the oversize and came back over the screen and recycled that also. Surprisingly enough the analysis has held up extremely well. I didn't believe it would do it, but it did. They have an extra Tyler hummer—it's an expensive proposition, but it will work all right. Recycle roughly 250, 300 pounds.

Chairman Reynolds: Thank you.

Mr. Perrine: You can recycle as much as 400 pounds in products —I'm thinking of an 8-16-16, I believe it is. Bear in mind you're putting back into your process there, well in that case there, a fifth of your ton, cooling the product and you've paid out good money to get that product back. It's a choice of whether you want to tolerate the troubles that fines will give you as we've shown on the table up here by Mr. Hill, I believe it was—and you have that problem then of paying to get the heat back that you took out.

There is recycling being done. It's not set up for it very well; they have to weigh the stuff back in and in the batch operations there's usually not an automatic way for getting it back in there as you have in continuous operation.

By the way there's very little continuous operation going on in this because in a continuous ammoniator — I can't recall offhand any place where they're doing that and not granulating. It's inherently a product for the continuous system of granulating.

Mr. Larry Lortscher: One other answer on that last question. One extensive job I worked on was a semi-granulation. We ran into the problem the mixer boys cautioned us on. If you start putting in more in that mixer than it's designed for, especially when you get in these wet plastic materials, we ran into a heck of a lot of trouble in trying to maintain tonnage rate by going 25 per cent more in that mixer.

Now the man finally settled on about 10 per cent more and even that causes some problems, but that would indicate when you start putting recycle back you can't hold your fold—you've got to take a part of your charge, so you will reduce your through-put rate even more so than you can do by using higher recycle in the continuous granulation system.

Chairman Reynolds: Are there any other comments. We've mentioned several different types of distributions systems here. Are there any others that you're particularly proud of that you might want to bring before the group?

A Voice: This doesn't have to do with distribution. I'd like to have someone comment on the problems they've had with cleaning and so on.

Chairman Reynolds: George Walton.

Mr. Walton: Nobody was more amazed than myself when we started on this process we're using—I'd been through all these pains of trying to make a semi-granular material only to find the mixer coated up after an hour's work had to be dug out. Apparently this rotary movement with the sparger imbedded in the solids tends to keep the mixer very, very clean and we've had, other than a little build-up on the top of the sparger itself, we've had very, very little trouble with build-up in the mixer.

I'd say we seldom, if ever, change—stop to clean out more than at the change of shifts.

A Voice: I wonder if anybody else has had any experience with the mixer in any other process, would care to comment on that?

Mr. Schmalz: If you choose to call it build-up that requires periodic rapping with a maul or something of that nature, a real difficulty, why you do have some of that, we've found it in our operations. It requires fairly constant attention; normally you'd put on a set of knockers which stand you in pretty good stead; but your discharge chutes and your elevator buckets and one thing and another, do have some build-up in our operation. You do have to take care of it; there's no two ways about it. It depends on how much trouble you consider that.

DR. FRANK T. NIELSSON (International Minerals and Chemical Corporation): I think that the basic thing that some people have forgotten in all this discussion is that to them the word "Eyman" process is a magic word, and "semi-granulation" seems to be a not magic word.

Now we have some plants that ammoniate bad tries and turn out a variety of products. I'm not talking about conventional—you might call them your Eyman process or you might call them semi-granulation. To us the difference is this, that if you take all granular raw materials and you sprinkle them with a little bit of solution, a little bit of acid, you end up with a good looking product and it hasn't gotten sticky and it doesn't get very hot and it comes out looking good and you don't have any problems and it'll cost you a little bit more.

Then if you want to do it a little bit cheaper and you don't have the good raw materials, take the powders and put in a lot of solution, you put in a lot of acid, you find the stuff is sticky, it lumps up and you've got some problems and your stuff looks so, and you can't call Eyman semi-granulation. What most people, to my mind, I may be wrong, Mr. Walton, but I think I'm right in this because without the word "Eyman" —I don't know what the word "Eyman" means actually,—but we have some plants that turn out as good a product with nothing but a batch mixer without a cooler using some half baked recycling deal with an elevator. I think Mr. Stockman was telling me he doesn't know if Eyman can make as good a product as he's doing—it looks as good as the stuff from one of our high investment plants, but the formulation costs are completely out of line.

Now you go down on the East Coast where the boys are trying to make 5-10-10 with fine muriate and fine super and trying to make that stuff stick and those boys have got problems and their stuff is semi-granulation, which means half there, more or less.

I think that if you will talk to the people who have done a little bit of all, both granulation, the semi-granulation and maybe some of the Eyman type and just get their reaction from time to time, I think you will find out that I'm not too far wrong when I say that this Eyman type process is not what we would call a granulation, it's bulk or blending of solution and acid.

But it's not too far off because we're using nitro super, the nitro triple that's got ammoniation of 1.82 pounds per unit and you're comparing that with a man that's using triple that's got three to four pounds per unit.

Some of you don't know it, when you start getting granular super — and we make both super and triple — if you start using granular super you're getting six pounds per unit, you're going to slow down to two and a half to three pounds and it's just a horse of a completely different color and the boys who have tried what we call semi-granulation they've had cleaning problems and they've had dust problems; they've got fines problems.

CHAIRMAN REYNOLDS: I think as far as this type of discussion here on semi-granulation goes, as the opening speaker mentioned, I think it's a very relative term, as Mr. Walton's secretary couldn't find "scuffed" or whatever the word was in the dictionary. This is probably the same thing. (Laughter.)

It probably covers a broad band of fertilizer practices and products.

MR. EMERSON JONES (Allied Chemical Corporation): One comment on this build-up problem. We have found two little things that can be causing excessive build up. One of them Mr. Robinson mentioned; that is, if you do not get your end holes on your spray pipe spraying against the side of the mixer you're apt to cause excessive build-up.

The second thing is, you have a lot of down time and cause that mixer shell itself to cool down so that you get a salting out of the liquid phase right up there against the shell and it will cause excessixe build-up. We found a lot of times it actually pays to run right on through the noon period rather than shutting down and letting the system cool off.

CHAIRMAN REYNOLDS: Thank you. He makes a very good point.

Any other discussion? We have time for about one comment more or question or anything to be added.

(No response.)

If there's no other question, then we'll turn the program over to Dr. Sauchelli.

Thank you very much for your patience and your cooperation.

(Dr. Sauchelli then resumed the Chair.)

CHAIRMAN SAUCHELLI: This next item, as I mentioned earlier this morning, should be of interest to all of you. It's going to deal with a new, indespensable tool in industry; many industries are using it; we could perhaps use it to a greater extent in our own industry.

You folks have been very, very patient and kind and I know that you pay these speakers the finest compliment, the courtesy of close attention. We know that these speakers have put a lot of time and energy into their preparation and we show our appreciation by close attention.

I'm going to dare you to stand up one minute, then we shall resume with the next speaker.

(Stretch.)

The next speaker is a member of the Reliability and Statistics Group of the senior staff of Booz, Allen Applied Research, Incorporated, at Bethesda, Maryland. Before joining his present organization he was an assistant professor of statistics at the George Washington University and during his seven years in this capacity he served as a private consultant on industrial and government contracts.

He's had wide experience as a teacher and as a practical investigator. It gives me great pleasure to call on Dr. Chester McCall, who is going to give us a preliminary or more or less elementary discussion on statistics.

I suggested to him that he consider a mixed audience. There are many of you in the audience that undoubtedly are quite familiar with this subject of statistics; others are not quite, and it's very difficult for a speaker to project his talk to a mixed audience and I'm sure that we'll all appreciate what Dr. McCall has to tell us.

Statistical Quality Contol

Chester H. McCall, Jr.

It is my pleasure to discuss with you today a subject which is quite close to my heart. As trite a beginning as this might appear for a talk, I can state quite sincerely that my understanding and acceptance of the principles underlying the field of statistical quality control have proven invaluable during my seven years as a University professor and in my present work. When discussing a subject as esoteric as quality control to a lay audience, I find myself in the same position as when I was an instructor beginning a new semester. Before entering the classroom for the first time, one is inclined to ponder over the expected audience of students. Each one has a different background and each one is attending for a different reason. Some may be present because they have been forced to attend; others may be sincere in their desire to learn more of this subject. In your case, you may be curious about the mysteries of this thing called SQC (Statistical Quality Control). Before you, if I may do so, I would like to place myself in the position of a teacher. It is most important that I do not lose your attention, yet I do not wish to appear so basic as to insult your intelligence. Let me, therefore, make an assumption one is forced to make at the start of a new course. The student doesn't know me—I don't know the student. I want to be sure that he leaves the first session with the feeling that "maybe this subject isn't as bad as I've heard." Unfortunately, in discussing a subject with you in such a limited period of time, from my point of view there is no opportunity to do any long range convincing. Instead, I shall attempt to discuss with you some of the basic concepts of Statistical Quality Control and give you some indications of the significance of this field. Mr. Vance Ward, in a talk tomorrow, will relate the subject to your specific areas of interest.

I should first like to emphasize the difference between Statistical Quality Control and Quality Control. Perhaps I can best do this with an example. Recently, I visited a plant in New England and was introduced to the Quality Control manager. My purpose was to study the reliability program within the plant. However, during my stay, it became necessary to learn something about the quality control procedures which were used. It was most revealing to learn that quality control in this case meant process inspection and control, and nothing more than a culling process to weed out the obvious defectives in the production. No attempt was made to utilize a basic scientific approach to the quality control program. No attempt was made to feed back the information gained during sampling to provide improvements in production. This in turn would have given greater control over output and as a result increased the profit of this particular firm. I do not mean to criticize what was done in the name of quality control. But I do wish to emphasize the fact that this kind of quality control can work few miracles for the practitioners. One must utilize the scientific method in setting up the quality control program; one must take advantage of the techniques which have been made available to us in the theoretical statistical research being conducted throughout the country. And above all, one must provide some method to take corrective action as a result of the use of statistical quality control.

I have told you what statistical quality control is not; perhaps it would be well for me to tell you what I feel it is. A program which uses statistical quality control utilizes the most modern techniques of applied statistics to provide measures of production within a plant in such a way that problem areas can be indicated rapidly and with the desired accuracy. Such a program should also suggest the necessary channels through which appropriate action can be taken to rectify the areas which are dangerous from a production point of view. One must realize that the use of statistical methods in a quality control program cannot tell you when some action is called for. The manager—the person in a position of authority—should use the results of SQC as an indicator of the need for corrective action. In testing the reliability of a piece of equipment, statistics can tell whether the actual reliability is meeting standards; if standards are not met, it is rather difficult to conceive of a situation in which statistics will point to the exact part of the equipment (for example, a tube), and say "There your problem is; correct it and all will be well."

And so we see that Statistical Quality Control can serve as a tool for the manager of some phase of a plant's activities to assist him in deciding when corrective action is necessary. This corrective action may take the form of adjusting some serious defect or of noting how production has improved because of some other change in a process.

We may well ask "What is the foundation on which SQC rests its case?" It is my sincere belief that the area of SQC rests its case firmly on the foundation of probability theory. We rely on the fact that there is a regularity in nature and that we are capable of predicting behaviors when this regularity is occurring. A person could stand and watch a crap table for a long period of time. If the dice were unbiased, the laws of chance would, in the long run, be operating and we would be able to predict with some degree of success the occurrences of the possible combinations of the two dice. Similar things are true in studying the results in tossing a single true coin a large number of times. We are accepting the fact that statistical regularity will, in the long run, bring up heads about as often as tails.

Where, you may well ask, does this regularity crop up in production processes? Imagine the situation which occurs when several chemicals are combined to provide a fertilizer with a specific mix. Can we state that there is never any variation in the amount of each of the components going into the product? How many of you have ever opened a box of cereal and wondered whether you were getting the amount stated on the box? Certainly the government has expressed more than a passing interest in this problem. We must accept, therefore, the fact that variability is with us, no matter how much we should like to avoid this possibility.

Each bag of fertilizer can be likened to a large lot of shells which are ready for shipment. The procuring group is interested in the proportion of good shells in the lot; the buyer of the fertilizer is interested in the proportion of each chemical in the sacks. In the case of the shells, it would be a simple matter to fire all shells and give the procuring group a guaranteed statement about the proportion of defectives in that lot. But, what does it matter if we state with 100% confidence that 95% of the shells *were* good. The buyer wants to know with some degree of assurance that 95% of the shells *will be* good. In a similar way the buyer of the fertilizer wants to know with some degree of assurance that he is getting what is stated on the bag. If we assume that the chemists could make no mistakes in correctly indicating the proportions of the chemicals in the fertilizer, we might be able to make some statement about each bag after we had mixed it thoroughly and made the necessary laboratory evaluation. But this is too costly and time consuming, so we are forced to select a sample of bags for this study. And further, we often do not examine the entire bag, but select samples from several positions within the bag. This is done to enable one to examine the possibility of heterogeneity within the bag. As soon as we agree to study the proportions of each chemical within the samples which have been selected, we are faced with a decision problem. How far away from the requirements can our results be and still be considered consistent with the standards? This question is similar to the one which one could ask in tossing a coin fifty times—how far from 25 heads can my results be and still not cause me to suspect that the coin is biased? Here is where statistical regularity enters into the picture.

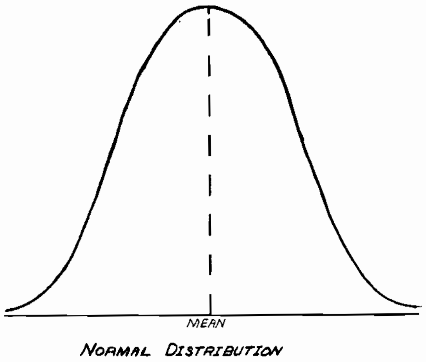


Figure I

Studies of processes of the kind involved in your industry have shown that variations about regularity seem to follow a pattern which resembles a distribution referred to as the normal distribution or the Gaussian distribution. Figure I indicates the typical normal curve. Assume, if you will, that we are examining the proportion of nitrogen in a fertilizer. If, in fact, we are meeting requirements, the results will be expected to vary from sample to sample. In general, we will find the largest number of sample results close to the requirement. Results more distant from this average will be less frequent. In fact, if we sampled often enough, we should be able to make some statement, for a process in control, about the proportion of times we would expect results to be less than a stated amount or more than a stated amount, or even between two stated amounts. This ability, when a process is in control, to predict results *in advance of sampling* is the basis of SQC. You will note, we do this predicting prior to sampling, not after the results are in.

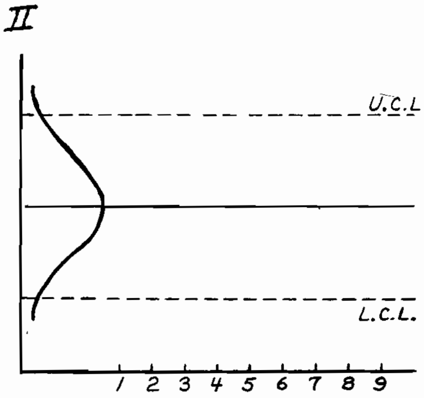


Figure II

If you will now allow me to turn the normal curve in Figure I on its side, we see that it appears in this form in Figure II. Note that the central or expected value is parallel to the base. If we were to label the base scale as sample number 1, 2, 3, and so forth, we could then set up a fairly simple graph, referred to as a control chart. The lines labelled U.C.L. (upper control limit) and L.C.L. (lower control limit) serve to indicate when corrective action may be necessary. At stated intervals of time, a sample, or samples, could be taken and the proportion of, say, nitrogen computed. This value could then be plotted on the chart. Successive values of the sample results could be charted and we would then have an indication, graphically, of the way in which production was progressing, with respect to the stated standard. The plot of these sample values would be sufficient to give us some indication of the consistency of our process. For example, if successive sample values kept increasing, one would suspect that something could be happening to the feed-in machinery. Corrective action would be called for. In **Figure III**, I have indicated one possible se-

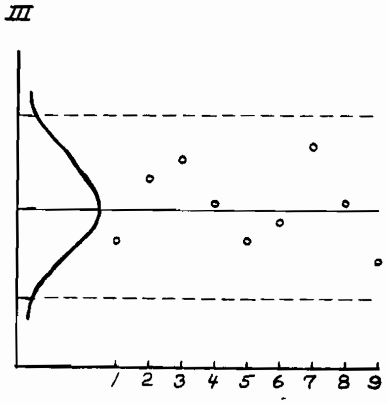


Figure III

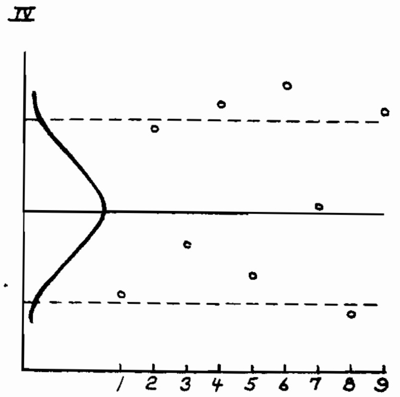


Figure IV

quence of results for a nine-day period. At the left is a drawing of the expected normal variation about the desirable proportion. You can see quite well that things are progressing according to laws of statistical regularity. However, if we examine Figure IV, even a novice in this area would suspect that something was wrong. Production is quite erratic; many of the sample values have jumped out beyond reasonable expected variation. Some action is definitely called for.

There are rules of the game by means of which one can say that this is too unusual a situation for me to continue production. It is time to stop and examine things before they get worse. These rules are too specific and in some cases too lengthy for me to mention now. One of these is obviously a search for a trend in the behavior of the sample results. However, these rules are such that we can state what risks we are willing to take in making the decision to investigate production. Obviously, when we base our action on sample results, we are drawing an inference from only a segment of the possible information. There are risks associated with generalizations from sample results. It is the use of probability which allows us to specify, in advance, the risks we are willing to accept.

If one uses the results from several samples to plot a control chart for averages, there is one most important item which is often overlooked. Things can, on the average, be quite satisfactory, while the results which make up this average tend to vary wildly. Averages are dangerous statistical beasts; without some measure of the variation of the elements going into these averages, we are often in considerable danger. A chain is as strong as its weakest link; no one would be crazy enough to accept a shipment of links which were, based on sample results, on the average quite acceptable. One would require some measure of the variability of these values; one would require some control over the variation within the process. A technique most widely used today involves setting up controls on the ranges of the observations which go into the averages being used for control. To illustrate this, suppose that we select five samples daily and make laboratory determinations of the proportion of nitrogen. The results of this would be five numbers. From these five we could get an average and so plot this figure on the normal control chart indicated in Figure II. It would also be of considerable interest to plot the range along with the average. This is most frequently accomplished by setting up a range chart, such as I have indicated here in Figure V. You will note that the averages are plotted on the upper part of the chart and the ranges are plotted along the lower part of the chart. Our process may, on the average, be within control, yet the ranges could be too great. Such a problem is of concern when we are faced with the necessity of meeting government specifications, for example, on the mixes. Too wide a range could conceivably mean that we are not controlling individual bags well enough to meet specs. On the average using enough fuel to successfully launch a missile is not enough. We want to be sure that we are close enough to that average to assure ourselves with a high degree of probability that a large proportion of the missiles will be successful. So, too, we must be sure that the variability about the average is small enough to be consistent with whatever restrictions have been imposed on the process.

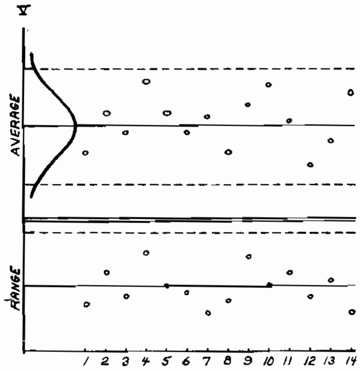


Figure V

The average and range (often referred to as X-bar and R) charts are the two original charts in the quality control area. Today we find new charts being introduced to bring the application more in line with the advanced findings in the statistical areas. It is of prime importance that we understand and accept what is indicated by the results of sampling. While at George Washington University, in my lectures I used control charts as illustrations of how the laws of probability behave, I have never found a case where I lost face with my students because probability just didn't work for me. I feel confident that one could make this same statement with the same degree of certainty in the applications within your industry.

Before closing, let me indicate other important areas where statistical quality control can play an important role. By properly designing the sampling procedure, it would be possible to exert a control over more than just the entire process. Raw materials could be subjected to acceptance sampling. Within the plant, it would be possible, for example, to pinpoint some of the trouble areas. I have seen situations where control charts indicated machines which were faulty, technicians who were in disagreement with the general results, work shifts which were inconsistent with each other. This has been accomplished by simply sitting down and defining the areas where trouble could conceivably exist. The techniques of SQC are then called into play so as to evaluate the possible effects of these areas of concern on the process. It may well be that the break of control in a process is only due to the hiring of a new individual who is just learning how to perform his function. SQC can be a useful tool to management. One should not cast it aside as a toy for "the others" to play with.

A chemist, with whom I was associated for several years, made the following statement about SQC. "Chet, you let me get into one of the smaller plants in our industry and let me apply the techniques of statistical quality control to their production and management functions. I would be willing to stake my reputation that this plant would soon have the others out of business or it would be forcing them to get into the statistical quality control area." He had reason to feel so strongly about the wonders of SQC: the basic factor which influenced his advancement in his profession was his acceptance of the methods of statistical sampling and the associated control.

I sincerely hope that I have conveyed to you today the strong feelings I have about the importance that statistical quality control can play in the modern industrial program. I will not go so far as to say he who does not use SQC will be lost; but I will state that he who uses properly the tools which are available will no longer be at the back of the pack but will be taking the long strides forward into the region of scientific management.

Where does one start in setting up an S.Q.C. program?

Here, the cooperation of management, production, and sales, with the aid of competent statistical help can indicate the channels to follow. Problem areas must be studied; sources of variation must be analyzed and isolated. These are but a few of the starting points. One must basically consider the entire process to ascertain where S.Q.C. can be most beneficial. S.Q.C. can be applied to any situation in which some characteristic (which can be measured or counted) is subject to variation.

* * *

It would be well for member firms to consider joining the American Society for Quality Control and its Chemical Division. For further information, write to A.S.Q.C., 161 W. Wisconsin Avenue, Milwaukee, Wisconsin.

* * *

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CHAIRMAN SAUCHELLI: Thank you, Dr. McCall. I know that your message is very much appreciated here. As our industry goes more and more into precision, into measurements, and into engineering, I'm sure that more units in the industry are going to take advantage of Statistical Quality Control.

We at National Plant Food Institute have just completed a study of sampling. The experiment was designed by two of the outstanding statisticians in the country and we just got a taste of it and I'm so enthusiastic about it I'd like everyone in the industry to become more familiar with it and also have a few more units in the industry adopt it as a tool.

We have a minute for questions.

(No response.)

You've been a very fine audience and a very patient one and this shows interest in the program.

Tomorrow morning we'll start at nine-thirty. We'll have a brief business meeting prior to the planned program. We hope that all will be on hand at nine-thirty.

(The meeting adjourned at four fifty five o'clock, p.m.)

Remarks by Mr. H. E. Hefler

Horizontal Drum Type Mixer in ½, 1, 2 and 3 ton sizes by Worthington Corporation.

Charging time in the Worthington Mixer is 30 seconds.

Mixing time in the Worthington Mixer is 1 minute.

Discharge time in the Worthington Mixer is 30 seconds.

So that the anhydrous ammonia and nitrogen solution is not sprayed directly on bare metal it is recommended the entire charge be in the drum before starting solution. Some operators prefer to allow the drum to revolve 5 to 6 times after charge before starting the solution.

Any system that will direct the spray toward the mass of dry materials and not toward the bare surfaces of drum and blades.

The Worthington Mixer is designed so that material can be fed through the charging side of the unit while the discharge door on the opposite end of the mixer is closed.

To keep the mixer tight during ammoniation and granulation the vents which are supplied must be put to proper use.

The rate of introduction of solutions will vary depending upon the type of system being used.

Friday, Morning Session, Nov. 6, 1959

The Round Table reconvened at nine-thirty o'clock, a.m.

Dr. Vincent Sauchelli, presiding

CHAIRMAN SAUCHELLI: Let's come to order now. Good morning. It certainly is a pleasure to see so so many alert faces in the audience this morning. This is a grand group; it certainly can take a lot of punishment and— (Laughter.) — they stay up most of the night and they are still bright and cheery on the third day. That's great.

We're going to start this session with a brief business meeting. As I emphasized in my opening remarks the other day, one of the characteristics of our organization is that it is very informal and I think the members have indicated that they want to keep it informal. We want to get away from the stereotyped sort of organization.

Our business meetings have always been brief and to the point.

The first order of business will be to ask the Treasurer to give his report.

Dr. Marshall.

(Dr. Marshall then presented the report of the Treasurer.)

CHAIRMAN SAUCHELLI: Thank you.

I'll hear from the Auditing Committee. Who's going to speak for the Auditing Committee? Joe or Frank? "Slugger" Nielsson.

DR. F. T. NIELSSON: Mr. Bosman and I have both examined the books of the Treasurer and found them in good order and believe that Dr. Marshall should be commended upon the efficiency and the general excellent conduct of his office.

CHAIRMAN SAUCHELLI: This is a job that takes a lot of time. We are fortunate in having a Treasurer who—he accuses me of having changed his whole relationship with the members here. He says that instead of being greeted with "How are you?" the greeting is "How much do you want?" (Laughter.)

He's sometime blunt but apparently you have to have a hard-boiled Treasurer in order to keep afloat.

The next order of business is on organization and I'd like to have an expression here—remember, as I said the other day, your Executive Committee serves as a handmaiden to the organization which acts as a Committee of the Whole. This is your meeting; we try to reflect your suggestions; your thinking and present on our agenda problems that all operating men are interested in.

So these meetings are your meetings. The Executive Committee does not try to impose its program on you; you are the ones that create the program and I hope you keep that in mind because we need ideas for next year and we'd like to have each and every one of you write in to me or to any of the members of the Committee, — Dr. Marshall or Al Spillman or Joe Reynolds, — your ideas as to what we should do, what items for next year's agenda and any suggestions as to how the meeting should be organized to be more effective and interesting.

I recognize our good friend, Wayne King. We didn't hear so much from him this year; last year he was very much on the job.

MR. W. W. KING: Thank you, Doctor.

I rise here to a point of order. You know a good meeting just doesn't happen; it's the result of careful planning and a lot of sweat and blood.

If you get a good horse, you know, you don't shoot him. He was talking about keeping him afloat; always remember, Doc, if you think you're drowning and you're going down for the third time, maybe you counted wrong. (Laughter.)

—I'll give you a little nautical story.

A friend of mine, this Admiral and his wife were entertaining a Captain in the Navy and the wife had an intriguing necklace on of signal flags. The Captain says, "Madam, what does that mean?" "Well that means," she said, "my husband gave me this necklace. It says 'I love you'." He looked at it pretty good and he couldn't remember any signal like that had anything about "I love you" business, so he gets home that night and he gets his book out, his signal book, and he remembered the sequence of the flags and by golly there it was all right. There it was, it says "Permission granted to lay alongside." (Laughter.)

I could go on like this for an hour and a half, but —I wish to place in nomination, Dr. Vincent Sauchelli as Chairman, Albert Spillman and Joe Reynolds, Jr. for a two-year tenure of office in the Fertilizer Industry Round Table. Now, of necessity, this has to be informal; we don't want to get incorporated; there's all kinds of angles to this thing. It must be informal and the members of this Committee must be readily available to each other; it's just an absolute necessity.

Now as a permanent thing— my friend that is'nt going to drown here—Dr. Marshall as the permanent Secretary and Treasurer. What is your pleasure here? Do you want someone—I'll entertain any kind of a motion, perferably one that we close the nominations.

(Laughter.)

(The motion was duly made and seconded.)

All in favor say "Aye."

(A chorus of "Ayes.")

Contrary?

(No response.)

I declare them elected. I think it's informal enough; we're allowed to do that.

Thank you, Doctor.

(Applause.)

CHAIRMAN SAUCHELLI: Joe Bosman.

MR. J. BOSMAN: I have to tell you gentlemen what an excellent job our officers have done this year. You see for yourselves the results of their endeavor, so I make a motion that we all give them a rising vote of thanks if that's in order.

(Standing ovation.)

Chairman Sauchelli: Thank you all and I know I speak for all members of the Executive Committee. We certainly appreciate your confidence and this vote of thanks.

The next thing to decide is on next year's meeting. We have that in mind. We can return to this hotel on November 2, 3 and 4 next year if that is your wish. We thought that the first week in November has ben apparently adopted by the Round Table as a desirable period in which to meet and the facilities here have been exceptionally good; the management has been very cooperative and apparently it was your desire, as indicated last year, to continue to meet in Washington. We assume that that's what you'd like to continue.

Now next year is an election year and about the second week would be a bad week in which to try to have a meeting and therefore we thought that November 2nd, 3rd and 4th; at one time we thought perhaps only a two-day meeting might suffice, but we've checked, we've had a randomized sampling of your wishes here and apparently those who have expressed themselves think that we still should continue as we have, a three-day meeting, terminating on the third day at noon so as to give folks an opportunity to get away conveniently; so if it is your wish to convene next year in this hotel on November 2, 3 and 4—that's Wednesday, Thursday and Friday, just as this year, let's have a show of hands.

(A showing of a majority of hands.)

Well, I think that will be it.

As one means of helping you for next year the hotel is prepared to take your reservations now. Those who have had difficulty in getting reservations this year just be wise. Usually November is a month of a lot of conventions and conferences here in Washington and it's a good idea to register in advance for next year's meeting; November 2nd, 3rd and 4th.

Now as to the question of exhibits and so on. We want this meeting, as I said, to be informal and to be a meeting where operating personnel have an opportunity to exchange ideas, to discuss their problems and all on a technical basis, on a non-commercial basis. We respect wholly and appreciate the sales departments in all organizations. I've always said the heart of any organization, is the sales staff.

But this is a meeting, it has been right from the start, a meeting for operating men, production problems and so on and there isn't much time to do justice to exhibits and the cost of bringing these exhibits here is tremendous.

Now it might be possible to hire a suite in the hotel and each have his exhibits there and full opportunity, to visit; but this—I've had several complaints—this idea of having just exhibits round the wall and very little time in which to discuss them and so on, has been sort of a handicap to the exhibitors. I don't know what your reaction is, so we'll have a couple of minutes discussion on it to see what is your feeling.—Is anybody willing to talk on that?

Mr. W. Jacobi: I'd like to express my feeling about it and see how all of you people feel about it. We've been in this Round Table thing, I guess, for quite a few years and we feel that it should be kept on an academic plane and as soon as you bring sales people—and Lord, I love them—into a thing like this, it immediately becomes competitive.

In other words, one fellow has a better looking booth than the other one. His sales manager comes down and says, boy, you flubbed the ball this year; next year we're going to have a better one. And the next thing you know you're going to start taking up half your room with exhibits.

Now I feel that practically every production man in this room receives literature from our company, from all our competitors, from people who make screens, from people who make elevators; I'm sure that everyone of you at some time or other gets that literature throughout the year, and everyone of you knows that if you drop the word that you're going to put in a new plant there are 9,000 salesmen who will descend on you.

So I actually don't think that the purpose of this meeting is to peddle your goods. I think the purpose of this meeting is for people like myself and others in other companies to be available for any questions which you may have of a technical nature to discuss informally at a time where you don't have your phone ringing and 9,000 trucks at the dock waiting to be loaded.

I think the whole purpose of this meeting is to be able in a relaxed way to get together with one another and say, all right, let's cut out all the sales talk; what are the basic facts of this, that and the other thing, and as far as we are concerned in our company, if money is needed in the way of whatever these booths cost to support the organization, I'm sure any of the suppliers and members of the Round Table, that if this extra money is needed, why have the booths? Say we ned $25 from you for this worthy cause and I'm quite sure that everyone of us in this room would be very happy to donate or set it up on a regular basis, but I do believe that if we allow this—and I can remember the first meetings where we had 20 people and it's grown to 400—If we start making an exhibit out of it, it's either going to drop dead in another couple of years or it's going to be so big that it becomes big business and we will lose this informal Round Table spirit which we have at the present time.

Chairman Sauchelli: Thank you, Bill.

(Applause.)

Is there any contrary opinion?

That's the answer, Bill, I think it expresses very definitely the will of the group.

Yes, Allen Jackson?

Mr. Allen Jackson: I'd like to say something on the other side. Some of us don't have quite the sales staff to get around to see all these people; to send all the literature that they might need.

I believe that a booth or a display of technical features, something special, that the various companies would have; you bring it to an atmosphere where people are going to talk between themselves on special features; not the salesman trying to sell everybody, but to bring machines or devices that may be different where you can discuss it with other people who may have used them.

I think it is the starting point for a lot of progress. I think these displays are simple enough; they are not offensive and I think they've helped a lot.

CHAIRMAN SAUCHELLI: I think we can make provision for a table for you to put your literature on, but I feel strongly that we want to keep this Round Table a forum for discussion of production problems by production men and in the limited time that we have to concentrate on those problems and on those informal gatherings here in the hall. Now any manufacturer that wants to hire a suite and display his goods and so on is perfectly welcome to do that, but the Round Table seems to me is not the place for it and I think the majority expression in applauding Jacobi's comments is indicative of the feeling.

We have a program—we want to try to terminate by noon and we have a very interesting program here this morning, one of major interest to all production men. We have a carryover from yesterday's agenda.

The next speaker is one of our good Canadian friends. Last year he missed an opportunity to come to Washington and give his lecture. I need not emphasize again what I emphasized last evening about the importance of statistical quality control. It's coming to be more and more used by all industry, by agronomists and by the chemical engineers and the business leaders and others; all beginning to get interested more and more in statistics.

There are not many units in the fertilizer industry that I know of that have employed statistical quality control in their plants as a tool to help the production man, but Canadian Industries, Ltd. of Montreal is one company that has employed statistical quality control and we are really fortunate in having with us this morning one of their representatives, Mr. Vance Ward, who has actually used it.

Last evening you had a preliminary discussion by Dr. McCall who gave you some of the definitions and just led you into it briefly.

This morning Mr. Vance Ward will show how SQC is actually applied.

Practical Applications of Statistical Quality Control in the Manufacture of Agricultural Chemicals

R. Vance Ward

YESTERDAY afternoon, Mr. Chester McCall of Booz-Allen Applied Research talked to you about some of the basic concepts of statistical quality control. In my talk this morning I would like to expand the subject by telling you how we have been able to use some of these basic principles with the Agricultural Chemicals Division of Canadian Industries Limited. I will discuss applications of S.Q.C.* to bag weight filling control, the problem of meeting guaranteed analysis, blending efficiency and the study and control of laboratory sampling and testing procedures.

A Simple Application of S.Q.C. Is Filling Control

In the chemical industry in general the simplest and often the most rewarding application of S.Q.C. is to control the net contents of packages. Chemicals are expensive and any system that can be used as an aid to control the filled weight and improve the overall product yield will provide a good return.

I would like to illustrate the application of S.Q.C. to filling control with a piece of quality control demonstration equipment called a Quincunx Demonstrator Figure I). It can be used to represent a production machine, and for the purpose of my talk this morning I will use it to represent a bag filling machine. The beads in the hopper at the top will represent bags waiting to be filled and the action of the beads falling through the hopper and the maze of pins into the vertical slots will represent the variation inherent in the filling operation.

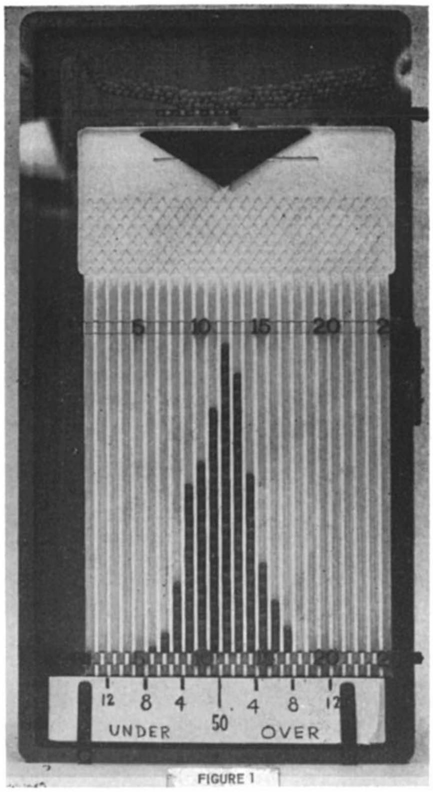

FIGURE 1

You will notice the normal bell shaped distribution of beads and how this compares with an actual distribution of filled bags (Figure II) that was obtained from an actual bag filling machine.

The Frequency Distribution is a Useful Tool

A frequency distribution is very simple to construct and it can be very useful to you in determining the precision of your bag filling machine. If you weigh approximately 100 consecutive filled bags from a machine and construct a frequency distribution, it will give you a good idea of the natural or inherent variation that you can expect.

This should be the first step in setting up an S.Q.C. system to control the net fill; that is, to make a frequency distribution to determine the "process capability" of the filling machine.

The Way We Used to Control Filled Level

Before explaining the S.Q.C. system we have introduced I would like to describe the control procedure that we used in one of our works to control filling and in fact still use in some of our locations.

Filling control was formerly

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*Statistical Quality Control.

maintained by weighing individual bags as they passed over a dial scale which was placed in the conveyor system leading from the filling machine. The machine operator was instructed to change the machine setting if he observed over or underweight bags.

Our S.Q.C. study pointed out that this procedure of controlling the filling level based on the results of individual bags was not sound. The statistical explanation is this: from the analysis of the frequency distribution (Figure II) it was shown that if the machine setting was held constant, individual bags would vary randomly about the mean by ± 1.0 pound. When the filling operator checked one bag chosen at random, this bag could be off the true setting by as much as a pound and yet this would not necessarily reflect a change in the machine setting—it could be the result of random filling variation. If the operator changed the setting, he would be changing the distribution mean i.e., the bags would vary by ± 1.0 pound about a different level. This overadjustment tended to increase the overall variation in bag filling.

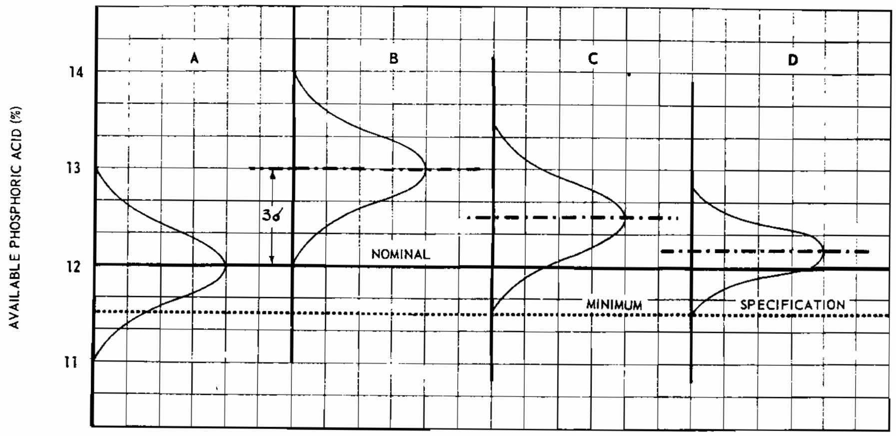
FIGURE II

DISTRIBUTION OF INDIVIDUAL MIXED FERTILIZER BAG WEIGHTS

LBS WT.	
1.0	𝍸 𝍸 II
.5	𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 I
0	𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 IIII I
- .5	𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 III
-1.0	𝍸 II
-1.5	I

FIGURE V

SETTING ADJUSTMENT FOR OPTIMUM OPERATING CONDITIONS AVAILABLE P_2O_5 IN 9-12-18 GRADE MIXED FERTILIZER

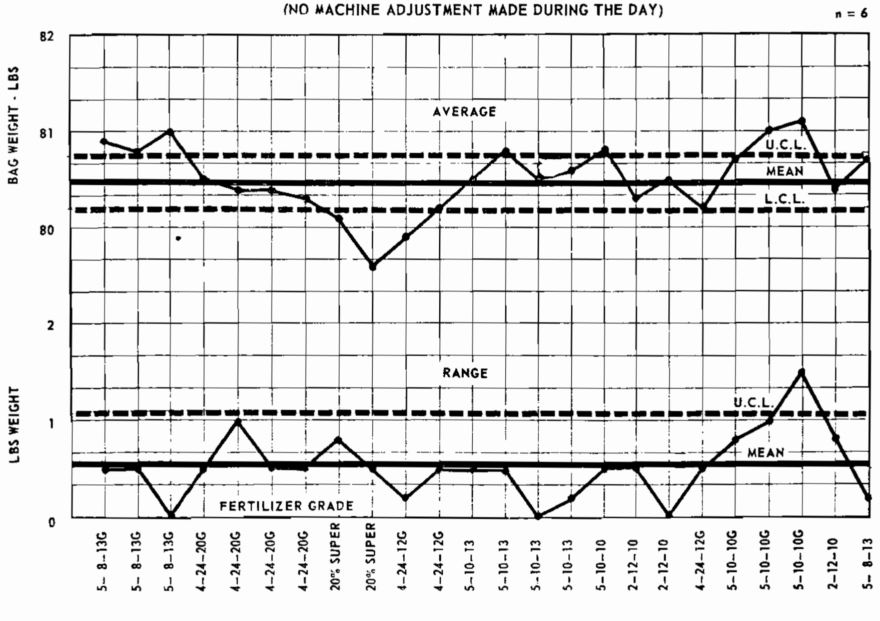


A Recommended S.Q.C. Procedure to Control Filling Level

To correct this problem of overadjustment we have taken the scales out of the conveyor system and discontinued the procedure of weighing each individual bag. The new procedure is to take a sample of six filled bags at random times during the day and to plot the average and range (heaviest minus lightest bag) on an S.Q.C. chart (Figure III). Provided the plotted points remain within statistically determined limits, the operator has been instructed not to interfere with the machine setting. However, points occurring outside the limits on the average chart indicate that the machine setting has changed and points occurring outside the limits on the range chart indicate excessive variation from bag to bag which would probably require machine maintenance to correct.

FIGURE III

S.Q.C. AVERAGE AND RANGE CHART FILLING 80 LB BAGS OF FERTILIZER (NO MACHINE ADJUSTMENT MADE DURING THE DAY)



This S.Q.C. system has resulted in improved uniformity of filling and has also had some secondary advantages such as increased throughput because of less adjustment and reduced scale maintenance.

A Statistical Approach to Meeting Guaranteed Analysis

In the remainder of my talk I would like to present a statistical approach which I feel could be used to determine the extent that we can use in-process statistical quality control economically in the agricultural chemicals field.

Just as weights of filled bags of fertilizer follow a normal frequency distribution, so will the sample-to-sample test results that we obtain from the chemical analysis of a mixed fertilizer. For example, the frequency distribution in Figure IV illustrates the batch to batch variation in the routine analysis of available P_2O_5.

FIGURE IV

BATCH TO BATCH VARIATION IN P_2O_5 ANALYSIS 9-12-18 GRADE FERTILIZER

1.0	II
.8	I
.6	III
.4	𝍸
.2	𝍸 𝍸 III
0	𝍸 𝍸 𝍸 𝍸 𝍸 𝍸 II
- .2	𝍸 𝍸 𝍸 𝍸 𝍸 II
- .4	𝍸 𝍸 𝍸 III
- .6	𝍸
- .8	IIII
-1.0	II

It should be emphasized strongly at that stage that this apparent variation in quality has four contributing factors 1) the sample, 2) the testing procedure, 3) the raw materials and 4) the process.

In Figure V this batch to batch variation is represented by the smooth curve "A." You will notice that, if on the average, the formula calls for an available P_2O_5 of 12%, there will be a sample variation in the analyses and some batches will appear to have as little as 11% P_2O_5 and some as much as 13% P_2O_5.

If it is mandatory that no batches should have an apparent plant food content below nominal, then as illustrated in curve "B," the mean level would have to be raised three standard deviations or 1% above nominal. This would assure that almost all analytical test results would appear above the nominal specification.

Now it seems unfair that the industry should have to be penalized with this amount of overage just to obtain an apparent test result above nominal. Raw materials contribute significant variations which for the most part are uncontrollable. We know that sampling and testing errors contribute significantly to the variation in analytical test results and that actually the true variation in quality is less than indicated. Finally in order to obtain the greatest benefit from high volume business, we must accept a certain amount of process variation.

These arguments support the case for providing a minimum specification and if this is done, then the mean plant food content can be lowered as illustrated in curve "C."

In order to lower the average overage still further, the problem becomes one of reducing the apparent batch to batch variation in test results and this is where statistical quality control techniques could be used.

Of this whole approach to the problem of meeting guaranteed analysis, one of the most important things to keep in mind is that control procedures cost money. If we must spend money to reduce these apparent variations in test results, then this cost must be offset by a saving which in this case would be the ability to reduce the overage closer to the nominal. This is illustrated in curve "D" where the test to test variation has been reduced and consequently we were able to lower the overage.

The Key to Quality Control is to Study Process Variations

As was pointed out earlier in this paper, the variation in the test results that we observed in diagrams, A, B and C is due to sampling, testing, raw materials and the process.

You have probably heard many times that the objective of statistical quality control is to build quality into the product—not inspect it. To phrase this statement a different way our main emphasis in applying statistical methods should be in finding ways to reduce and control process variation. It is interesting to me that from the literature I have read it would appear that the main emphasis in the application of S.Q.C. to agricultural chemicals has been in studying the problems of sampling and testing. Another basic cause of variation is the process and it should not be overlooked.

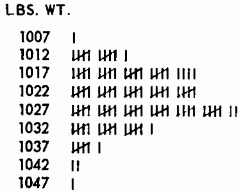
We Use S.Q.C. to Control Blending Efficiency

One application of in-process S.Q.C. is the control of blending efficiency. At the batch mixing operation, the raw ingredients are added into a weigh hopper from overhead storage bins. At some of our works the weigh hopper is suspended from a print weigh scale and as each ingredient is added, the weight is printed cumulatively on a paper tape. After all the ingredients have been added, totalling about 2,000 lbs., the batch is fed into a rotary batch mixer.

The importance of adding the correct quantity of each ingredient at mixing had justified the purchase of the print weigh scales. In actual practice these scales provided so much data that it was difficult for the operator and the foreman to assess rapidly and accurately variations in the additions of raw materials. We felt that possibly an S.Q.C. sampling and control chart procedure would present the data in a more suitable form.

A study of the weight data showed that the raw material weights were normally distributed. Figure VI shows the distribution of the amount of superphosphate in 100 batches of a mixed fertilizer.

FIGURE VI
WEIGHT DISTRIBUTION OF SUPERPHOSPHATE IN MIXED FERTILIZER



We decided that in order to obtain the optimum use of the raw maerial weight data we would check the batch weight results for 5 batches each day chosen at random. These random batch results were plotted on a statistical quality control chart which was kept at the mixing operation. From the results the operator was able to gauge his performance which resulted in a considerable improvement in batch uniformity.

S.Q.C. Can be Used to Reduce Testing Errors

There is a considerable amount of literature available on the application of S.Q.C. to study and control sampling and testing variations* and in my talk I do not intend to discuss this application of S.Q.C. Suffice it to say that these causes of variation are very significant and statistical quality control accuracy and precision charts can be used in the laboratory to isolate and control technician and testing equipment variations. Statistical sampling theory can help shed some light on the problems of bulk sampling.†

In Summary S.Q.C. is a Valuable Tool

I hope that the two talks that have been given at this Round Table on S.Q.C. have shown you that the various techniques of S.Q.C. can be of help to you in solving some of your quality problems. The techniques are not really difficult, they take a little understanding and probably the greatest obstacle in obtaining acceptance of S.Q.C. is in educating people as to what a valuable contribution quality control statistics can make. I hope I have been able to give you

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*See reference page, part 1.
†See reference page, part 2.

some insight into the type of problems that S.Q.C. can help solve.

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   c) "S.Q.C. in the Chemical Laboratory" by G. Wernimont (Industrial Quality Control, May 1947).
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   f) "Controlling the Control Laboratories in a Chemical Fabres Manufacturing Plant" by J. H. Reynolds. (The American Society for Quality Control, Conference Papers, 1952).
   g) "Application of Statistics" A.S. T. M. Manual Number S.T.P. 103 July 1950.
   h) "Effective Quality Control Programme for the Industrial Control Laboratory" by F. W. Knoll, Jr. Industrial Quality Control, December 1957).
2. "The Statistical Approach to Bulk Sampling Problems" by B. H. Lloyd. (The American Society for Quality Control, Conference Papers, 1952).

Chairman Sauchelli: Thank you, Mr. Ward. That certainly was excellent. It was one of the best lectures on the subject that I've ever listened to. We at the National Plant Food Institute have been making use of this statistical tool in that research project that is sponsored by the Institute on sampling.

Also he referred to the Magruder Fertilizer check sample that the control people are engaged in. We're making use of this statistical tool in that series of control tests. So, we've made a beginning. I sincerely hope that more in the industry will study this new tool and learn to apply it.

Is there any question that anyone wants to fire at Mr. Ward? We have just a few minutes.

Mr. W. Southworth: I don't have a question for Mr. Ward, but I would like to ask a question of the production people in the audience here. After hearing this presentation by Mr. Ward, I wonder how many of you production people feel statistical quality control might be of help to you. I wonder if we could have a show of hands?

Chairman Sauchelli: An excellent question. Would there be a show of hands on that?

(A showing of several hands.)

You know I've often thought that we might hold a seminar on this in order to familiarize more production men with this SQC and open it to suggestion sometime; I try to encourage as many people as possible to get acquainted with it and get their feet wet.

A question here?

Mr. Lortscher: In trying to get at your plant food variance there and using statistical quality on that and in the course of a shift's run, how many samples do you have in mind it would take in the course of an eight-hour shift to start to make this treatment valuable?

Mr. Ward: I would like to use past data on this as much as possible. This to me is not really SQC as a control on an hourly basis or anything. This is an example where you might use frequency distributions and variations to set up what your formulation should be.

Is that what you wanted?

Mr. Lortscher: Yes.

Mr. Ward: I wanted to get some idea of what my present variation is in my testing and my processing and my raw materials. You probably got this from some analyses that you've done. You just take your analyses that you have done on a certain, say, on one type fertilizer. If you make a distribution of past analyses that you've done you'll get a frequency distribution as a result. Is that right?

Mr. Lortscher: Right.

Mr. Ward: If you take that frequency distribution you can see how high you would have to set that above normal in order to make sure that no further results would appear below normal or below specification, if there's a lower spec below that.

Once you've got an idea of your variation—

Mr. Lortscher: In other words, you take a plant that perhaps takes one sample per shift out of production. You could use enough past history of that and still utilize this data and derive some meaningful—

Mr. Ward: Oh, yes. I'm quite sure that is a start. Now the thesis of what I had to say is once you see where you are—for example, if it tells you that you've got to put two per cent more plant food in for any one, because of variation, then you can begin to associate that with the cost of being able to get more precise answers and it's only worth whatever you can save in getting it down.

Mr. Lortscher: Thank you.

Mr. Marburger: You agree with one sample per shift here. How about one per week, What's the limit here?

Mr. Ward: I don't know; I haven't really agreed on one per shift either. (Laughter.)

I'm afraid I'm not a statistician that will come in and tell you you should take five or six from each batch. This is not the answer, but I think it must be associated closely with what food content you have to put in in order to meet the control procedure that you have at present.

If you do it once a week and it gives you a certain variation and you can set your mean two or three per cent over fill and you're happy with this against the cost of doing the testing, leave it at once a week. But if you look at it and you say it's too high and I've got to do something about it, then you've got to find ways of improving your control; in other words, reduce your variation, and as you reduce your variation you can reduce your average level of content, is that right? —your formulation. So what I'm trying to do is trying to find ways of counterbalancing the cost of this business on a practical basis.

Mr. Lortscher: But you're really getting—

Mr. Clegg: Could I interrupt you for a second? I think the question was, you know, we took a lot of samples, perhaps samples of each batch. We give you the data, you analyze the variation and you said we must sample every so many bags. I think that's the information they're looking for.

Mr. Ward: As to the number of batches.

Mr. Clegg: The number of samples you have to take to give you some idea of the variation.

We took samples of each batch and then you analyzed the data and told us how frequently we should sample.

MR. WARD: I'm trying to think what the exact number was.

MR. CLEGG: I don't remember a specific case, but I think that's the information you're looking for and that depends on the variation that you have in your equipment from batch to batch or hour to hour continuously. Could you analyze that data for us?

MR. WARD: Primarily though, if you look at the variation you got and you are willing to set your plant food content high enough you would still only need to take relatively less samples than if you want to get closer control of it.

MR. LORTSCHER: But to get a look at possible process control, am I right in assuming you'd have one sample per shift, probably wouldn't give you enough information to lead you to make improvements, process wise.

MR. WARD: Exactly. You're right. I think what we're confusing is two stages here; a preliminary stage to see where you would have to set your plant food content with your existing situation and secondly, what control—is it worth spending the money on to improve this level. Okay? And I think my first example was what have I got now and what kind of money have I got to play with to improve it.

CHAIRMAN SAUCHELLI: Thank you, thank you very much. I think we'll have to go on with our program.

With your indulgence I want to take one mintue here to welcome friends and guests from overseas. Increasingly, we have had attendance here of men from different countries.

At this meeting we have nineteen from countries outside the United States. Australia, India, China and a fine delegation from our neighbor to the north, Canada. We're always glad to have people from other countries visit us and it just shows the spread throughout the world of this great fertilizer industry in which we are interested and engaged in.

It's spreading and what we do here through our proceedings is eagerly sought by your colleagues overseas. Our visitors who are here, will they please stand.

(The visitors arose: Applause.)

Our next item is one that has aroused intense interest. It started at one of our Round Tables and since then there's been intense interest throughout the country. I'm talking about pre-neutralization.

What is "pre-neutralization" and its place in the mixed fertilizer manufacturing process? What are its advantages, disadvantages; what specific equipment is needed? These and other facets of this interesting subject are to be presented and discussed by our distinguished panelists.

We know the subject is very much alive among operating men and none of us is going to be disappointed in his expectation of a thorough analysis of the problems.

We hope that we'll have time to permit a discussion period and that there'll be many questions or pertinent comments that you will make.

The panel comprises the following: Philip Stone, Virginia-Carolina Chemical Corporation; Grant Marburger, Spencer Chemical Company; George Gilliam, Allied Chemical Corporation; Dr. Frank Keenan of du Pont and Fritz Alfrey of the W. R. Grace and Company, and we also have Art Hansen of the American Agricultural Chemical Company, who'll come in a little later and I hope we'll have an opportunity to hear his comments too.

Dr. Keenan is going to concentrate on the conditions required for the successful use of Uramon Ammonia Liquors in Preneutralization operations.

Fritz Alfrey of Grace is to tell us about the development of a concentric pipe reactor and of its design and purpose in pre-reactions.

Phil Stone will lead off with the place of the preneutralization process in the fertilizer plant.

Preneutralization

Philip E. Stone

V-C's development work on the pre-neutralization process and the application of the process to factory scale operation was completed in the early spring of 1957. Later that same year, at the Fertilizer Industry Round Table, the process as used by V-C was disclosed to the fertilizer industry. At this time the use of a pre-neutralizer or pre-reactor in the manufacture of mixed granular fertilizers had received very little attention from the producers. After the 1957 Round Table meeting considerable interest was developed in pre-neutralization. V-C, for example, received numerous inquiries about the process from the United States and also from abroad.

During 1958, studies of the different applications of the process were started by several different groups. Many nitrogen producers showed interest in pre-neutralization and some companies began extensive test programs at bench scale and pilot plant levels. As time passed, the knowledge and experience gained by these studies was applied to several factory scale operations across the country. The results of these commercial operations have not been generally reported. Today we hope to learn more about pre-neutralization as practiced at these relatively new installations.

The process which V-C uses today is essentially the same as that described by Floyd at the Round Table meeting in 1957. Only X-O-X grades, such as 14-0-14 and 8-0-24, are presently produced. Low free-ammonia solution is reacted with sulfuric acid in a preneutralizer tank, and the hot liquor which results is then mixed with potash and limestone in a rotary type ammoniator-granulator. The granules formed are dried, screened, cooled, and coated prior to bagging. Since 1957 thousands of tons of granular goods have been successfully produced with the V-C pre-neutralizer. Much has been learned and naturally some improvements have been made. We would like to discuss a few of these improvements, and also comment briefly on some of the problems which the pre-neutralization process presents.

Fan for Removal of Water Vapor. When the V-C pre-neutralizer was first installed, a fan was placed in the stack above the reaction tank to assist in the removal of water vapor from the building. Since then it has been learned that no fan is needed. The V-C stack is made of ¾-inch marine plywood. It is 18 inches square and approximately 25 feet high. There is a hood at the bottom of the stack just above the reaction tank. The original fan has now been removed. The stack stands up well against water vapor and other gases, and does an excellent job of removing the gases from the tank areas in the building.

Hot Liquor Distribution. To improve the uniformity of the granular product, both chemically and physically, V-C originally used an over-flow trough for distribution of the hot liquid on the rolling bed of solids in the ammoniator. The trough was about three feet long, and fitted with a cover to prevent solids from dropping in from above. The liquor flowed over the lip of the trough by gravity onto the solids bed in sheets or in several different streams. This method gave generally good distribution of the liquid but it was not very dependable. Frequent cleaning of the lip of the trough was needed to assure good distribution.

The method finally adopted by the factory operators and the one in present use still employs an open trough. The liquor does not overflow the trough but discharges to the solids bed through orifices drilled in the bottom of the trough. At present, eight orifices are used about six inches apart. The hot liquor flows through the eight openings continuously and little or no attention is required to keep them open and flowing freely.

The trough is nothing more than a six-inch steel pipe cut in half the long way with the ends closed to form a trough, and with half-inch holes drilled through the bottom about six inches apart. V-C plans to extent the length of the present trough to provide about sixteen discharge openings instead of the eight openings now in use. This will be done to improve still further the uniformity of chemical composition and the uniformity of particle size of the products.

Plants which mix hot liquor with solids in a pug mill are not too concerned with liquor distribution as an aid to mixing. The pug mill apparently does a very good job of mixing liquids and solids. Plants which already operate with a TVA type ammoniator, however, may have trouble justifying a pug mill for production of a few pre-neutralizer grades.

Sparger for Sulfuric Acid. In the beginning, the acid sparger was made from 316 stainless steel pipe. The openings in the pipe became enlarged after very short use. Several other materials were tried without success. Finally an acid sparger was made from Hastelloy C. This sparger has been in use for several months and there is no indication of excessive wear. The nitrogen solution sparger and the pre-neutralizer tank are both made of 316 stainless steel and these items are still in good shape after many months of use.

Caking Problems. To avoid caking, V-C dries pre-neutralization grades to about 0.5 per cent moisture. In addition to this, the granules are coated with anti-caking agent. As a further precaution against caking, 14-0-14 is not held in bulk storage for long periods of time. This grade is bagged soon after production to avoid moisture pick-up from humid atmosphere.

Reason for Pre-Neutralization. Low-cost nitrogen solutions are widely used by the fertilizer industry. Granular grades containing up to about eight per cent nitrogen are often formulated with all of the nitrogen requirement from solution. However, granular grades having nitrogen contents above eight per cent commonly utilize some sulfate of ammonia in the formula when conventional production methods are used. Sulfate is added to these high nitrogen grades to prevent the formula mix from becoming too wet and over-granulating. The sulfate content of the formula generally increases as the nitrogen grade increases. For example, a conventional 12-12-12 granular formula might contain 400 pounds of sulfate, whereas a 16-8-8 formula might contain as much as 800 pounds of sulfate.

Sulfate nirogen costs about 25 to 30 cents per unit more than solution nitrogen. Therefore, from the point of view of formula cost, the use of sulfate nitrogen is not desirable. The pre-neutralization process provides a method of producing high-nitrogen grades with *less* high-price sulfate nitrogen and *more* low-price solution nitrogen. Formula savings may sometimes amount to as much as two to four dollars per ton, depending upon such variable factors as formula in present use, fertilizer grade, and delivered costs of materials.

It is not difficult to determine the savings that are possible by switching from conventional methods of production to pre-neutralization methods. This can be easily done and the potential savings are often very attractive. The evaluation of the storage and handling qualities of the pre-neutralization products, however, is an entirely different matter. Most people in the business of producing mixed granular fertilizers are reluctant to make radical changes in formulation without knowing with certainty the effect of the changes upon the quality of the products produced.

Pre-neutralization methods can be expected to grow only as fas as factory experience proves that pre-neutralization products can be produced with consistently good storage and handling qualities. We believe that the Round Table members are more interested in the storage and handling properties of pre-neutralized grades than in any other phase of the subject. We are therefore hopeful that those people with firsthand information on the subject will come forward today and give us the benefit of their experience on this important subject.

CHAIRMAN SAUCHELLI: We'll wait until the end for the discussion period.

The next panelist will be Grant Marburger of Spencer Chemical.

MR. GRANT MARBURGER (Spencer Chemical): I admire the fine tone that Phil started this off in an so I'm going to follow him and give a brief definition of by intent here.

One, I have tried to pull out what are the important aspects of the processing itself.

Two, is to summarize a cross section of the few installations—the results that have been achieved on a commercial scale. In other words, none of this data is specifically tied down to a given case that all put together would come to some final answer for your application in your own plant.

The Use of Preneutralization

Grant C. Marburger

THE use of a preneutralizer to manufacture granulated mixed fertilizers is a recent development in the fertilizer industry that has followed granulation itself by only a few years. Neutralizers have been used for many years to produce ammonium sulphate, nitrate and phosphate. The preneutralizer is an adaptation of the neutralizer.

Preneutralization has a place in mixed fertilizer plants for the production of high nitrogen grades. Flat ratios such as 1-1-1 comes under this billing, and inverse ratios such as 2-1-1 and 3-2-2. Other developmental work will be reported today on a preneutralization process that includes low nitrogen grades.

In high nitrogen grades, overgranulation is the basic problem. If some of the excess temperature and liquid can be removed by prior reaction, a reduced liquid phase is achieved. Stated more directly, preneutralization removes heat and moisture from the reacting materials in the preneutralizer before the reacted material enters the granulator.

Now let's jump to the question, why use this process? There are two primary reasons that will determine its potential use in a plant. The first is economics and the second is the production of what I shall call "exotic" grades.

First comes the consideration of economics. Whether prenuetralization is advisable in your plant depends on a formula cost calculation to determine whether money can be saved on grades presently being produced. The economics depend on your delivered cost of raw materials, which is largely a function of the plant's location. An example will illustrate this point, and Figure 1 is a comparison between two plant locations. At Location No. 1 the calculated formula savings is 90¢ per ton, yet at Location No. 2 it reaches $3.52 using the same formula and varying only the delivered cost of raw materials. Obviously the possibilities of installing preneutralization equipment are enhanced where the savings are greater. There are many plants in the United States where no costs savings would appear on a grade like 12-12-12; the example shown in Figure 1 is to illustrate the differences between locations.

The difference in cost savings depends on three things (Figure 2):

1. The cost spread between solid and liquid nitrogen.
2. The cost spread in phosphates (normal and triple, or normal and H_3PO_4)
3. The cost of H_2SO_4.

In the second plant location there is a greater spread between solution and ammonium sulphate (82¢ vs. 41¢), H_2SO_4 is cheaper, and there is a greater spread between normal and H_3PO_4 (68¢ vs. 38¢). These three combinations created the greater savings.

The second of the two primary reasons for preneutralization is that it makes possible the production of inverse ratio grades. Examples are 20-10-5, 16-8-8, 15-10-10, 14-0-7, and 15-15-0. The need for these grades is a market consideration in your sales area. If a potential market exists, is developing or can be developed, the process becomes worthy of consideration. Figure 3 illustrates some of these inverse ratio grades and their formulation.

Beyond these two primary reasons for considering preneutralizations as applicable to your plant there are some secondary benefits that may be achieveable. As experience is gained, some of these secondary benefits may prove to be primary reasons for its utilization. In other words the full value of side benefits will be ascertained from operating experience in the industry. There isalready a growing fund of data from present production plants.

The first is less recycle. Recycle requirements to control excess liquid phase are drastically reduced by the use of the preneutralizer. By removing heat and moisture the need for so much dry material is removed. To the plant that utilizes recycle as a control for over-agglomeration, the effect of a reduction in recycle is to increase the production rate of the plant. In plants not so equipped, it allows the production of high nitrogen grades using all solution as the source of nitrogen.

For instance, in the production of 16-20-0, using H_3PO_4, a recycle ratio of aproximately 3 to 1 is required. In commercial production this has been dropped to less than 1 to 1. In the production of 12-12-12, obtaining all the nitrogen from solution, the required 2 to 1 recycle rate can be reduced. I don't have commercial data on this grade, but estimates are at less than 1 to 1, say ½ to 1. Commercial production data for two other grades might be of interest:

20-10-5	1½ or 2 to 1
14-0-14	½ to 1

Pilot plant data is as follows:

14-0-14	½ to 1
20-10-10	1 to 1 (Spensol, Triple, H_3PO_4)
18-36-0	2 to 1 (Spensol, Triple, H_3PO_4)
11-48-0	2½ to 1
16-48-0	2½ to 1 (Ammonia, H_3PO_4, H_2SO_4)

It is interesting to notice that the last grade was made with an all liquid formula, granulating on a bed of recycle only.

Another secondary benefit is less fumes. When the neutralization of ammonia is carried to completion prior to its introduction into a bed of materials containing potash, the formation of NH_4Cl is eliminated. Ammonium chloride aerosol is generally considered the most troublesome fertilizer plant fume.

You will not, however, escape

entirely from fume considerations when contemplating preneutralization. Excessive temperatures will create fumes and apparent decomposition of material. Also when the preneutralizer is run on the acid side, fuming will result. If the acidity is kept below 2% (as H_2SO_4), fuming can absolutely be eliminated. It is rather striking to see an exhibition of this from a preneutralzer plant stack. As the operator raises or lowers the acidity, the fogging increases or decreases. Originally our pilot plant operator glanced at the stack to control this acidity because it was visible from his platform, but a simple chemical test has now been put into commercial practice that can be performed by any plant man. It is also comforting on this fume question that we have not encountered any major fume problem on installations in operation today.

Less nitrogen loss can also be included in this list of preneutralization benefits as a distinct possibility. I would say the least on this subject because nitrogen loss is so dependent upon the basis of comparison used and other factors. But to pass on some "order of magnitude" that has been obtained today in commercial experience indicates ¾ unit loss on a 20-10-X grade and less than ½ unit on 16-8-8, 14-0-7, 15-15-0, 15-10-10 and 16-20-0. A more complete discussion on nitrogen loss becomes involved with equipment design, which other panelists are covering here today.

Itemizing these points, the advantages to be considered when evaluating the potential use of preneutralization in your plant are:

Primary Reasons

1. Economics
2. Production of "exotic" grades

Secondary Benefits

3. Less recycle
4. Less fumes
5. Less nitrogen loss

It's beyond my dreaming capacity that all five of these will ever be realized in any one plant. That's too much to expect from a few thousand dollars, a tank and some pipe.

Now let's cover a few subjects at random under the heading of production experience.

The thing that impresses a person when he first sees an operation of this nature is its simplicity and ease of operation by plant personnel. Specialists, or extensive training, or complicated control are not neded as many of us envisioned at first blush. Production rates that are ultimately possible are not known. Most generally there's a bottle-neck in some other part of the plant that doesn't allow the preneutralization section full performance. Today's experience ranges at 13 to 15 tons per hour, and higher rates have not had time to be fully resolved. We pacify ourselves with the knowledge that it wasn't long ago we hadn't even considered using 20 units of Spensol and 10 units of phosphoric acid in a formula or the recent application of granulating with an all liquid formula, let alone be concerned with high tonnages. However, production rates are a necessary concern, and the answers will be forthcoming in the near future.

There have been some initial problems limiting production rates, all concerned with this preneutralizer tank itself, that resolve around an inter-relationship of violency of reaction, splashing or spattering, surging, and mixing. Splashing itself is not of concern, for with a closed vessel it cannot hurt anything. Surging from the tank into the ammoniator is correctable by several methods. But the inter-relationship of these isolated problems with nitrogen loss, amount of mechanical mixing, and the sparger arrangement becomes a more complicated problem in equipment design.

Operating control is by temperature, which can be controlled by the amount of water added to the preneutralizer. The operator manipulates his temperature indicator with a water line and valve to the preneutralizer.

It's hard to separate production problems and equipment design considerations in a discussion of this nature. The production problems we have encountered relate directly to the tank size, and more specifically to water loading and ammonia loading. So let's look briefly at these considerations. The evaporative water loading (Figure 4) of a preneutralization tank is the water evaporated per square foot of surface per hour. The amount of water that must be evaporated has been calculated in Figure 5 from thermal data. It shows an excess heat balance of 287,900 BTU's per ton. This heat must be dissipated to maintain the proper operating temperature for that grade, and the calculated amount of water for evaporation is 256 pounds per ton. This amount of steam must be disengaged from the top of the tank. Translating this to tank size, Figure 6 shows that a 3′ diameter tank would require 362 pounds of water flashing from the surface per square foot per hour. Of course at a higher production rate the evaporation load goes up, but as the tank size increases its water loading decreases. Somewhere there will be an upper limit to the amount of evaporative water loading and a larger tank becomes necessary. Violent reaction and splashing are some of the penalties of an undersized tank.

Ammonia reaction loading (Figure 7) is the pounds of ammonia per cubic foot of liquid per hour. The example shown is for 16-8-8 at 15 tons per hour in a 5′ diamter tank with a 3′ liquid level, which results in 33 pounds ammonia per cubic foot per hour. Again (Figure 8) as the tank diameter increases the ammonia loading decreases.

Figure 9 summarizes these characteristics for two grades. To translate some meaning to these numbers, let's establish a range of permissible operation. Commercial operating experience on water loading indicates better success when operating somewhere in the 100 to 150 range. There are other variables that must be taken into account for final design. For instance, 150 has been unsuccessful in the more viscous slurry created with the use of phosphoric acid, whereas it has been operatable with an ammonium sulphate slurry. If a water loading of 100 to 150 is approximately the proper operating range, the size of the tank becomes important. Experience on ammonia loading might indicate successful operation at 35, and once again as seen from the table, the larger tank becomes indicative.

There are other considerations besides this approach that materially affect the equipment sizing problems, and consequently operating problems. However experience substantiates the importance of a large size vessel where high nitrogen grades or high rates of production are desired.

Comparison of Formula Costs at Two Plant Locations

12-12-12 Without Preneutralization

		Location #1	Location #2
Spensol	8 Units		
Sulfate	4 Units		
H_2SO_4	93 Lbs.		
Super	6.5 Units		
H_3PO_4	5.5 Units		
Potash	12 Units		
		$41.82 Per Ton	$40.31 Per Ton

12-12-12 With Preneutralization

Spensol	12 Units		
H_2SO_4	207 Lbs.		
Super	8.1 Units		
H_3PO_4	3.9 Units		
Potash	12 Units		
		$40.90	$40.31
Cost Savings		$ .92 Per Ton	$ 3.47 Per Ton

Raw Material Delivered Costs

	Location #1		Location #2	
Spensol	$1.42	41¢	$1.36	82¢
Sulphate	$1.83	Spread	$2.18	Spread
H_2SO_4	$ .012		$ .0075	
Super	$1.10	38¢	$ .80	68¢
H_3PO_4	$1.48	Spread	$1.48	Spread
Potash	$ .56		$ .56	

Inverse Ratio Grades

20-10-5	
Spensol	893
H_2SO_4	481
Normal	353
H_3PO_4	237
Potash	167
	2131

15-15-0	
Spensol	669
H_2SO_4	307
Normal	827
Triple	291
	2094

16-8-8	
Spensol	715
H_2SO_4	431
Normal	552
Triple	104
Potash	267
	2069

14-0-7	
Spensol	677
H_2SO_4	400
Potash	233
Filler	750
	2060

15-10-10	
Spensol	669
H_2SO_4	388
Normal	430
Triple	247
Potash	334
	2068

Water Loading

(Preneutralization Tank)

Evaporative Water Loading =

Lbs. H_2O Evaporated Per $Ft.^2$ Surface (per hr.)

Preneutralizer Design Calculations

16-8-8

Heat Input

(Heats of Solution, Reaction, Dissolution)

16 Units @ 31,610 BTU = 506,000 BTU/Ton

Heat Out-Go

(1) Latent & Sensible Heats, Materials

16 Units @ 13,230 BTU = 212,100 BTU

(2) Extraneous Heat Loss

$(Q = UA\Delta T)$ = 6,000

218,100 BTU

Total Heat Out-Go = 218,100 BTU/Ton

Excess Heat Requiring Additional Water = 287,900 BTU/Ton

Latent & Sensible Heats

of H_2O Evaporation = 1126 BTU/Lb.

Additional Water Needed to Evaporate =

$$\frac{287{,}900 \text{ BTU/Ton}}{1126 \text{ BTU/Lb.}} = 256 \text{ Lbs. } H_2O\text{/Ton}$$

Water Loading

16-8-8

Lbs. H_2O Evaporated Per $Ft.^2$ Surface (per hour)

Diameter Tank	10 Tons/Hr.	20 Tons/Hr.
3′	362	724
5′	130	260
7′	66	132

Ammonia Loading

(Preneutralization Tank)

Ammonia Reaction Loading = Lbs. NH_3 Per $Ft.^3$ Liquid (per hr.)

Example:

16-8-8 @ 15 T/Hr.

5′ φ Tank 3′ Liquid Level

16 Units × 92 Lb. NH_3/Unit=133.5 Lbs. NH_3/Ton

$$\frac{133.5 \times 15 \text{ T/Hr.}}{59 \text{ Ft.}^3} = 33 \text{ Lbs. } NH_3\text{/Hr./Ft.}^3$$

Ammonia Loading

16-8-8

Lbs. NH_3 Per $Ft.^3$ (Per Hour)

Tank Diameter	10 Tons/Hr.	20 Tons/Hr.
3′	83.8	167.6
5′	30.1	60.2
7′	15.4	30.8

Water Loading

15 Ton/Hr.

Tank Diameter	12-12-12	16-8-8
3′	405	543
5′	146	187
7′	74	96

Ammonia Loading

15 Ton/Hr.

Tank Diameter	12-12-12	16-8-8
3′	99	125.8
5′	35.6	45.2
7′	18.1	23.1

CHAIRMAN SAUCHELLI: Thank you, Grant. That was very interesting.

The next panelist is George Gilliam of Allied Chemical.

MR. GEORGE GILLIAM (Allied Chemicals): The two previous speakers have talked a good deal about preneutralization and as a request of our hard working committee to submit storage date I'm going to confine my remarks to big storage data on preneutralization grades made in connection with our pilot plant studies at Hopewell.

Storage Data on Preneutralized Grades

G. R. Gilliam

IN connection with our pilot plant studies of the use of a pre-reactor for manufacturing high analysis fertilizers in which all nitrogen is derived from solution we also included bag storage tests on products made. Grades made included 14-0-14, 16-0-8 and two 16-8-8 materials. Since formulation and process conditions may influence the handling and caking characteristics of a given fertilizer material, information on both will be described along with the storage data.

The 14-0-14 and 16-0-8 materials were formulated with a nitrogen solution having a composition of 19 per cent ammonia, 72.5 per cent ammonium nitrate, 8.5 per cent water and a total nitrogen content of 41 per cent. Sulphuric acid (66° Be) was used to neutralize the free ammonia. Regular non-granular muriate of potash was the source of K_2O and dolomite was used for filler. The dolommite particle size was about 80 per cent through 200 mesh. For the 16-8-8A formulation the same raw materials were used with the addition of normal and triple superphosphate to supply the P_2O_5. In the case of the 16-8-8B formulation a nitrogen solution having a composition of 25.3 per cent ammonia, 69.2 per cent ammonium nitrate, 5.5 per cent water and a total nitrogen content of 45 per cent was used. Normal superphosphate and wet process phosphoric acid were used to supply the P_2O_5. Other raw materials were the same as in the previous products. Of the materials charged, ammonium nitrate considered by many to be the most influential factor affecting caking varied from 502 pounds per ton for the 14-0-14 to 580 pounds per ton for the 16-8-8A material. Data are shown in Table 1.

Table 1.

Formulations—Lbs. Per Ton

Fertilizer Grade	14-0-14	16-0-8	16-8--8A	16-8-8B
Formula				
Nitrana 4M (19 NH_3 41N)	693	790	801	—
Nitrana 7 (25, 3 NH_3 45N)	—	—	—	735
Normal Super (18. 96 A. P. A.)	—	—	560	646
Triple Super (44. 1 A. P. A.)	—	—	133	—
H_3PO_4 (58.2)	—	—	—	89
H_2SO_4 (93)	412	465	353	389
KCl (60 K_2O)	473	270	270	270
Dolomite	490	555	20	20

Basic process equipment employed in making these four materials was a pre-reactor, pug mill mixer, dryer, cooler, screens and crusher. Total throughput capacity was about 1000 pounds per hour giving a production rate at a one to one recycle ratio of 500 pounds of product per hour. Product size was taken as minus 6 plus 20 mesh. For the 14-0-14 and 16-0-8 grades all of the nitrogen solution and acid were processed through the pre-reactor and over flowed continuously as a slurry to the pug mill at a temperature of 291 °F and a water content of 5.8 per cent. When this slurry was combined in the pug mill with the KCl, dolomite and recycle the resulting mix was at a temperature of about 185 °F and contained 2 to 3 per cent water. From the pug mill the mixture was passed to the dryer where the moisture was reduced to one per cent or less. Material from the dryer was fed to the cooler and out of the cooler to the screens where the oversize went to the crusher, the product size taken out, and fines returned to recycle. In the case of the 16-8-8 materials a portion of the nitrogen solution was fed to the pug mill to ammoniate the superphosphates. The remainder of the nitrogen solution, all of the sulphuric acid, and, in the case of the 16-8-8B formulation,

Table 2.
Operational Data

Fertilizer Grade	14-0-14	16-0-8	16-8-8A	16-8-8B
	Temperatures °F			
Pre-reactor Slurry	291	291	293	284
Dryer Feed	187	183	176	144
Dryer Product	187	171	199	185
Cooler Product	140	136	158	129
	Moistures %			
Pre-reactor Slurry	5.8	5.8	5.8	6.5
Dryer Feed	2.1	2.6	2.3	2.7
Product Out	0.2	1.0	0.4	0.3

the phosphoric acid were fed to the pre-reactor. Conditions in the pre-reactor for the 16-8-8A formulation were essentially the same as for the 14-0-14 and 16-0-8 grades. For the 16-8-8B formulations a somewhat higher slurry moisture and a slightly lower pre-reactor slurry temperature were used. Other process conditions through the dryer, cooler, screens and crusher were the same as for the other three products. To determine the benefit of a coating agent on the product storage characteristics sufficient product from each grade was coated with a two per cent clay coat to supply material for storage tests. In this particular case a kaolin type clay having a specific surface of about 16,000 cm^2 per gram was used. However, other clays or kieselguhrs with equivalent total surface would serve equally as well. Process operating conditions are shown in Table 2.

Actual storage data on these products were obtained over a six months storage period. All test bags were on hundred pound size and were placed in positions two and three from the bottom in piles twelve bags high. Five ply bags consisting of three paper and two inner asphalt liners were used in all cases. The storage area was a one story unheated cinder block building with a concrete floor. Each test bag was inspected and graded for degree of caking at the end of 30, 90 and 180 days of storage. (Actual time of storage between inspections was 30, 60 and 90 days.) The uncoated 14-0-14 and 16-0-8 products having an initial moisture content of 0.7 and 1.0 respectively, were found to be severely caked at each of the inspection periods. These materials had a hard bag set requiring two or more drops of the bag on the flat side from a height of about three feet to break the bag set. Both of these materials would be expected to cake rather severely in either bulk or bag storage. Somewhat lower initial moistures would be expected to improve storage condition. Both of these materials with about the same initial moisture and coated with 2% clay showed essentially no caking over the entire test period. The 14-0-14 was free flowing at the 30 day inspection and the 16-0-8 had only a slight bag set requiring only one drop of the bag to make it completely free flowing. At the 90 and 180 day inspections both materials showed the same condition as observed on the 16-0-8 at the 30 day inspection. The 16-8-8 materials, having a somewhat lower initial moisture, showed good condition with the exception of the uncoated 16-8-8B material at the 180 day inspection. Both the coated and uncoated 16-8-8A and coated 16-8-8B materials were free flowing at the end of30 and 90 days of storage and showed only a slight bag set after 180 days of storage. The uncoated 16-8-8B was free flowing after 30 days, had a slight bag set after 90 days, and was severely caked after 180 days of storage. Data are shown in Table 3.

Table 3.
Bag Storage Tests

Fertilizer Grade	14-0-14		16-0-8		16-8-8A		16-8-8B	
% Kaolin Coat (1)	0	2	0	2	0	2	0	2
Initial Moisture, %	0.69	0.60	1.01	0.90	0.44	0.48	0.28	0.40
Inspections and Grade (2)								
30 day	D	A	D	B	A	A	A	A
90 day	D	B	D	B	A	A	B	A
180 day	—	B	D	B	B	B	D	B
Final Moisture, %	—	0.93	1.03	1.05	0.79	0.86	0.93	0.97

(1) Kaolin Clay — Specific surface of about 16,000 cm2/gm.
(2) Grading Code — A — Free flowing when bag is removed from pile.
B — Free flowing after one 3-foot drop on flat side.
C — Lumps remaining after one 3-foot drop on flat side.
D — Two or more 3-foot drops required to break bag set.

These data would indicate that X-O-X fertilizers, made by the pre-reactor process, should be dried to a very low moisture level, preferably below 0.5 per cent, and should be coated with an inorganic coating material such as clay or kieselguhr in order to have good condition in storage. For the N-P-K grades, made by this process, good condition would be expected, provided product is dried to a low moisture level, preferably 0.5 per cent or less. The addition of a coating would be beneficial but not as important as on the X-O-X grades.

CHAIRMAN SAUCHELLI: The next panelist will be Dr. Frank Keenan of the duPont organization.

DR. FRANK KEENAN (duPont): Gentlemen, I think our preceding panelists have set the tone of this symposium in sort of two directions; one is that it's a very complicated subject; secondly, that we're all feeling our way a bit. It's better to say it that way than the blind leading the blind, I think.

But it is complicated and because of that and the early stage of development in which it finds itself in the industry, I think we ought to take one step at a time and as least insofar as I'm concerned this morning, I'm going to stick to the characteristics of the preneutralizer itself when we're talking about urea-ammonium-water systems, because in operating any preneutralizer, be it a tank or the other devices such as the mixing jet sparger that we'll hear about subsequently, I think what we first need to know are the characteristics of the solutions we're dealing with in the preneutralizer.

Performance of Uramon® Ammonia Liquors in Preneutralization

Frank G. Keenen

DURING recent years, processes for granulating fertilizers appear to be utilizing more and more acid (sulfuric and/or phosphoric). Introduction of this acid directly into the ammoniator-granulator has created problems. Some of these, at least, may be resolved by partially or completely preneutralizing the acid with the ammoniating solution before introducing it into the granulator. The operation of a preneutralizer—be it a tank, mixing jet sparger, or other mechanical contrivance—requires a knowledge of the physical and chemical characteristics of each particular ammoniating solution and the products resulting from its reaction with the acid. This paper deals primarily with the preneutralization characteristics of "Uramon" ammonia liquors (commonly called UAL). We do not propose at this time to delve into the complexities of the entire granulation process.

It seems especially appropriate to clarify the performance of urea in this process in view of the confusion that appears to exist. The confusion results from the fact that the chemical reactions involving urea under preneutralizing conditions are completely different depending on whether the system does or does not contain ammonium nitrate. Any attempt to apply data from urea-ammonium nitrate solutions to urea solutions containing not nitrate or vice versa leads to completely erroneous conclusions. Since both compositions are in general use by the fertilizer industry, it is obviously to the best interests of all concerned that this distinction be understood and all technical information be accurately identified.

Inasmuch as the breakdown of urea in aqueous acid systems in the absence of ammonium nitrate under preneutralizer operating conditions can only produce carbon dioxide and ammonia, the excitement about urea decomposition leading to evalution of toxic gases from preneutralizers is obviously unfounded and erroneous. This has been thoroughly covered in already published articles (Croplife —May 11, 1959) and need not be repeated here, since gas evolution other than carbon dioxide relates only to ammonium nitrate-urea combinations. Consequently, the operation of a preneutralizer using UAL is completely safe and practical, since no inflammable, toxic, or explosive gaseous products are evolved. Control of pH, temperatures ,and liquid ratios can be quite flexible in this respect.

A major point of concern is, of course, the stability of urea as it passes through the preneutralizer. It has been known for many years that during long periods of several weeks in hot storage piles (120 to 160 degrees Fahrenheit) urea partially "breaks down," i.e., hydrolyzes into ammonia and carbon dioxide. For example, in mixed fertilizers containing five to seven per cent moisture stored at 140 degrees Fahrenheit, the rate of breakdown is around five to six per cent per week. Reduction of the moisture content to one per cent or less, as is normally done in granular goods, limits the extent and rate of hydrolysis. Temperature also is a major factor—the hydrolysis rate being negligible below 120 degrees Fahrenheit storage temperature but increasing as the temperature rises. This has led many to jump to the erroneous conclusion that at preneutralizer operating temperatures (220 to 250 degrees Fahrenheit), urea should break down almost instantaneously. Such "conclusion jumping" has overlooked the time factor in this chemical reaction.

It has been found that in strongly acid solutions in the absence of ammonium nitrate the extent of urea hydrolysis during a seven-minute hold-up time in the preneutralizer tank is around two per cent at 220 degrees Fahrenheit, four per cent at 240 degrees Fahrenheit, and eight per cent at 250 degrees Fahrenheit. Doubling the hold-up time in the tank to 15 minutes approximately doubles the extent of this hydrolysis. The pH of the solution has relatively little effect on hydrolysis during these short-time intervals. Since cost and flexibility of operation require that preneutralizer tanks be as small as feasible anyway, it is not likely that hold-up time would exceed 10 minutes in any unit. In fact, the use of an eductor-sparger type mixing device appears quite feasible for UAL solutions. In such a device, the contact time is but a few seconds, and urea hydrolysis is hardly detectable.

While on the subject of urea hydrolysis, reference should be made to a paper presented by Ove Jensen at the 1955 Round Table Discussion. In that paper, he cited plant operating experiences with UAL in a typical granulation process that showed urea hydrolysis through a 10-minute granulator plus 15 minute dryer amounted to less than five per cent. This has been confirmed many times since 1955.

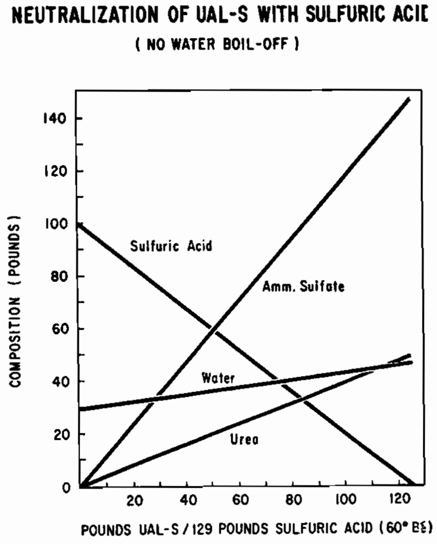


Figure I

Now as to what happens as sulfuric acid is neutralized with UAL, Figure 1 shows the composition range using UAL-S as typical example. This is based on 129 pounds of 60 degree Bé sulfuric acid which contains 100 pounds of

100 per cent sulfuric acid and 29 pounds of water. Complete neutralization is reached at approximately 126 pounds of UAL-S. The ammonium sulfate produced, of course, exceeds its solubility so that as neutrality is approached, the slurry becomes thicker. Simultaneously, water is being boiled-off by the heat of reaction. From the standpoint of plant operations, therefore, it becomes a matter of determining the optimum degree of neutralization that will give the slurry consistency best for handling and granulating.

The amount of water that is boiled-off in the preneutralization step is important, yet quite difficult to define at all accurately. Water boil-off is affected by heat losses through radiation which in turn, relate to equipment design, location (inside or outside the building), winter vs summer operation, rate of throughput, strength of acid, and other minor factors. Likewise, operation temperatures, plant lay-out, and granulation design, i.e. rotary or pugmill, and specific formula characteristics will dictate the kind of a slurry required. The inter-related complexity of these variables tend to discourage any attempt to carry performance data from one unit to another or from small to plant-scale operations. However, the following general information may be useful as a starting point.

Neutralization up to around 70 per cent of the acid in a continuous preneutralizer tank operating at 225 to 250 degrees Fahrenheit with any of the several UAL compositions yields flowable slurries. Under these conditions in an open tank, approximately eight pounds of water are boiled-off per unit of nitrogen added as UAL. For example, to each 129 pounds of 50 degrees Bé sulfuric acid can be added 85 pounds of UAL-S (1.8 units N) which would boil-off around 15 pounds of water. In addition to the water removed, the carbon dioxide present in the UAL as ammonium carbamate is evolved. This carbon dioxide improves the efficiency of steam removal and is particularly helpful in an eductor-jet sparger in keeping the slurry line swept open. Occasionally in a tank preneutralizer, it tends to cause some foaming, but this is readily controlled by adding an anti-foam agent.

Although water could be added to the preneutralizer to control slurry flowability, it seems that this vitiates the major objective of preneutralization i.e., to remove water. Sulfuric acid stronger than 60 degrees Bé is not recommended as the increase in concentration leads to undesirably violent reactions. If the acid source is 66 degrees Bé, some dilution would be desirable. Phosphoric acid may replace all or part of the sulfuric without adversely affecting operations, in fact they seem to run a little more smoothly with phosphoric acid present.

In conclusion, "Uramon" ammonia liquors have no properties or lead to any reactions that in any way preclude their use in preneutralizer operations. The breakdown or hydrolysis of urea is not excessive during the short-time intervals (under 10 minutes) in preneutralizer tanks even up to temperatures of 250 degrees Fahrenheit, and the absence of any flammable, explosive or toxic gases makes their safety particularly appealing.

CHAIRMAN SAUCHELLI: Thank you, Frank. It certainly shows how far we've advanced in our technology. You can see by these charts and these discussions how very few of the oldtimers—when Frank first started getting around the fertilizer industry, — conceived of such advances and such progress.

The next panelist is Arthur Hansen of the American Agricultural Chemical Company.

MR. ARTHUR HANSEN (American Agricultural Chemical Company): Thank you, Mr. Chairman, Members of the Round Table. The process which I am about to describe is called the automatic pug process. Why this name was chosen I do not know since the process is not automatic and we did not use a pug mill.

But actually a pug mill could have been used but we used a cuff light screw conveyor and found it to be quite satisfactory, but here on however, I will refer to as a pug mixer.

Pilot Plant Production of Mixed Fertilizers from Preneutralizer Fluids

Arthur Hansen

THIS process will describe the preneutralization of nitrogen solution with 60° Bé sulfuric acid, 75% phosphoric acid and mixtures of both acids and the blending of the resulting fluid with dry solids to form high analysis mixed fertilizer.

Objective

The objectives of this work were as follows:

1) To enable use of more liquids in formulas with little or no solids recycle. This would then result in lower formula costs and higher production rates.

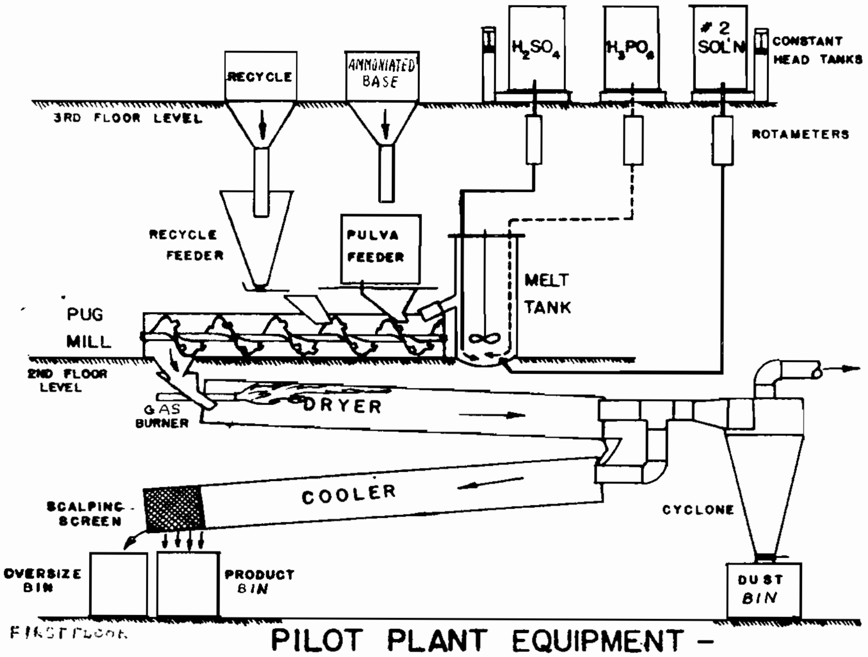
2) A better process for high analysis and X-O-Y type or nitrogen potash grades.

3) Low cost water soluble fertilizer.

Figure 1—This pictures the pilot plant flow diagram. Note that there are two points for addition of nitrogen solution for complete N-P-K grades.

The dry solid raw materials were fed into a ribbon mixer and batch mixed. The mixture was then passed thru a pilot continuous ammoniator to form an ammoniated base. This was subsequently blended with preneutralizer fluid — granulation was controlled by means of a small water spray. For most of the grades prepared—the sole source of nitrogen was a standard 41% NH_4NO_3 containing 22% NH_3, 12% water and 66% NH_4NO_3—very little granulation water was used (from 10-40 pounds per ton) —recycle was used to return cyclone dust (40-60 pounds per ton) only—except for the 18-18-18 water soluble grade.

Figure 2 — This illustrates the pilot plant equipment arrangement. This center unit point is the preneutralizer tank — which is designated as the melt tank. Except for that material entrained in the cyclone—there was no provision in



PILOT PLANT EQUIPMENT -
AUTOMATIC PUG MILL PROCESS

Figure II

the process for fines removal. At the time of this work, which was the year 1955, granulation was a secondary objective. Most of us will remember that, 5 years ago, the substitution of one unit nitrogen solution resulted in a dollar per unit cost saving. Therefore no sulfate of ammonia was used in this work.

The production rates used varied between 900-1400 pounds per hour. For one grade 18-18-18, the production rate was quite low (300-400 pounds/hour).

The piece of equipment which limited the production rate was the rotary dryer. I think this is true for many operating plants today.

The problem here with very high nitrogen grades is to prevent over-granulation and "puddling" in the dryer itself. It is probable that a counter current type dryer would be more efficient for very high nitrogen grades.

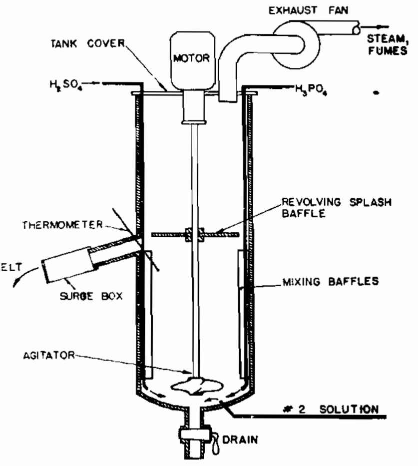


Figure III

Figure 3 — This presents a detailed view of the preneutralizer tank. The basic outline of this tank and the preneutralization scheme was obtained from TVA.

To improve mixing, the tank was modified with mixing baffles.

A Lightnin gear motor revolved the stainless steel agitator at 425 RPM.

The tank itself was constructed of mild steel which showed little wear after almost 1000 hours of operation. The tank was 4 ft. high.

The acid feed lines consisted of ¼″ stainless steel pipe (#316).

Note that the nitrogen solution line is located outside the tank. This caused some difficulty during the shutdown because the high density reaction fluid would back into the line. This problem was eliminated by means of a steam blowout arrangement. Originally the solution line was located inside the tank—but this caused flashing of the solution and erratic behavior of the rotameter. Various piping arrangements and air cushions were tried but we ended up by relocating the feed line outside of the tank.

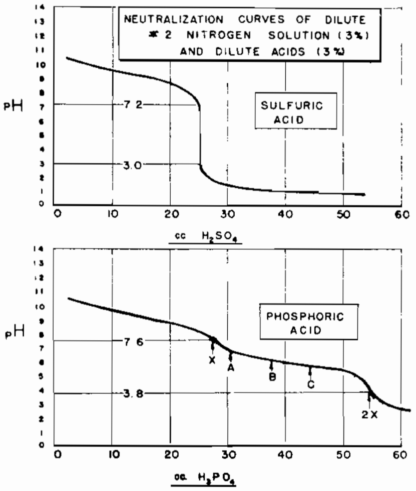


Figure IV

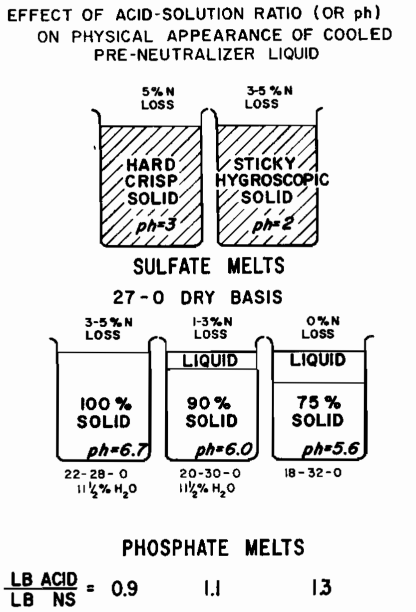
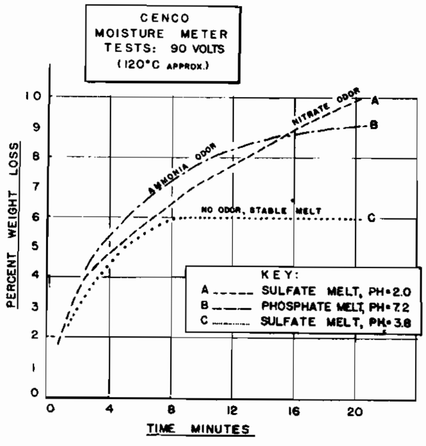


Figure V



TYPICAL DRYING CURVES OF MELTS

Figure VI

Another problem was that of buildup of stalactites and a thick layer of solidified fluid on the top and upper sides of the tank. Much of this was caused by the splash due to the violent reaction taking

Figure 7

Analyses of Mixed Melts

Experiment No.	**43-132**	**56-18A**	**56-18B**	**56-18C**
Figured—dry	(27.10-9.15-0)	(26.80-25.50-0)	(26.80-25.50-0)	(26.80-25.50-0)
Figured—10% H_2O	(24.40-8.23-0)	(24.10-22.90-0)	(24.10-22.90-0)	(24.10-22.90-0)
Expected—5%N Loss 10% H_2O	(23.20-8.23-0)	(22.90-22.90-0)	(22.90-22.90-0)	(22.90-22.90-0)
Actual— % H_2O	9.35	10.10	8.00	11.30
% N	25.00	23.25	22.67	23.00
% Am.N.	17.36	16.53	16.21	16.23
% P_2O_5	7.20	23.80	23.56	21.64
% SO_4	X	7.51	8.76	8.06

place. We did experience from 30-40 pounds of buildup during the first hours operation of a run. This problem was reduced considerably by insulating the tank, and installing a revolving screen just above the liquid level.

Because the preneutralizer fluid discharged in surges—between 5 and 10 seconds apart—a small weir box was installed by the discharge spout. This improved the subsequent blending of the fluid with the dry base.

The retention time in this tank, which had a 200 pound capacity was between 20 and 45 minutes—depending on the grade being made.

Figure 4 — This illustrates clearly the difference between H_3PO_4 and H_2SO_4. These pH curves were prepared using dilute solutions. From experience, it was determined that the desired pH values were 3.0 for H_2SO_4 and about 6.7 for H_3PO_4. These points represent a compromise between NH_3 loss and liquid fluidity.

Figure 5 — This shows the physical effect of pH on the fludity or solidification tendency of preneutralizer liquid.

The pH has a marked effect on the number of units N from solution which can be incorporated in a fertilizer formula without "puddling".

Figure 6 — These time-heating curves illustrate the thermal stability of sulfate and phosphate preneutralizer fluid.

Figure 7 — Here are some analysis of preneutralizer fluid prepared with mixed acids.

Figure 8—Typical formulas for the 8-0-24, 14-0-14, 10-0-10, and 12-0-10 grades are shown. The 8-0-24 is a common tobacco starter grade prepared with K_2SO_4.

Note that a small amount of superphosphate was added as a low cost conditioner for the remaining grades.

Figure 9—Typical formulae for 12-12-12, 12-24-8, 12-24-12 and 13-13-13 are shown.

Conclusions

Advantages

1. The key advantages of preneutralization is that the heat of reaction between the ammonia solutions and acids may be fully utilized to drive off excess water from these liquids. It is ideally suited for 60° Bè strength acid and for nitrogen solutions low in NH_3 content.

2. A larger amount of liquid may be incorporated in a formula, making it ideal for grades exceeding 12% N and X-O-Y type grades. Thirteen units from nitrogen solution of low ammonia content (22%) may be added to these grades without requiring recycle for granulation control. For phosphate containing grades, part of

Figure 8

Formulas — Actual & Recommended

	Nitrogen-Potash Grades			
Actual Formulas	**8.40-0-24.70**	**14.80-0-14.80**	**10.50-0-10.20**	**12.60-0-10.20**
Materials				
No. 2 Soln. (40.50% N)	415	722	518	623
H_2SO_4—59° Be.	322	584	407	503
Dolomite	368	0	0	0
Muriate of Potash (60% K_2O)	0	503	350	340
Sulfate of Potash	998	0	0	0
R.O.P.	0	200	400	200
Limestone	0	262	515	553
	2113	2271	2180	2218
Recommended Formulas	**8.40-0-24.70**	**14.80-0-14.20**	**10.50-0-10.20**	**12.60-0-10.20**
No. 2. Soln (41% N)	410	722	512	615
H_2SO_4—60° Be.	336	591	420	505
Dolomite	376	0	0	0
Muriate of Potash 60% K_2O)	0	472	340	340
Sulfate of Potash	998	0	0	0
R.O.P.	0	200	200	200
Limestone	0	287	709	558
	2112	2272	2181	2218

Figure 9

Formulas — Actual & Recommended

Complete Fertilizers

Actual Formulas	12.60-12.20-12.20	12.60-12.20-12.20	12.60-24.50-8.15	12.60-24.00-12.00	13.65-13.00-13.00
	(Dry Base)	(Pre-Ammo Base)	(Pre-Ammo Base)	(Pre-Ammo Base)	(Pre-Ammo Base)
No. 2 Soln (40.50% N)	500	388	388	384	458
No. 3 Soln (40.50% N)	0	231	236	238	217
H_2SO_4–59° Be.	273	304	220	147	359
Muriate of Potash	407	403	227	400	433
Sulfate of Ammonia	234	0	0	0	0
Triple	362	334	930	825	475
R.O.P.	414	491	0	0	222
H_3PO_4–75%	0	0	97	179	0
Limestone	0	53	64	20	40
Dolomite	0	0	0	0	0
	2210	2204	2207	2193	2204

Recommended Formuals	12.60-12.20-12.20	12.60-12.20-12.20	12.60-24.50-8.15	12.60-24.00-12.00	13.65-13.00-13.00
		(Pre-Ammo Base)	(Pre-Ammo Base)	(Pre-Ammo Base)	(Pre-Ammo Base)
No. 2 Soln. (41% N)	Not recommended from dry base	613	616	615	666
H_2SO_4–60° Be.	“	316	227	152	371
Muriate of Potash	“	403	272	400	433
Triple	“	334	930	825	475
R.O.P.	“	491	0	0	222
H_3PO_4	“	0	97	179	0
Limestone	“	48	67	21	40
		2205	2209	2192	2207

Basic Equipment Sizes — Pilot Unit

Melt Tank	Total Height	42 inches
	Liquid Heights	21 inches
	Inside Diameter	16 inches
	Wall thickness	¼ inches
	Material	Mild steel
	Capacity=130 lbs. water	195 lbs. melt
Pug Mill	Length	6 feet
	Screw Diameter	9 inches
	Discharge opening	9″ x 9″
	Material	Mild steel
	R.P.M.	42
Cooler or *Dryer*	Length	12 feet
	Inside Diameter	16 inches
	Wall thickness	¼ inch
	Material	Mild steel
	R.P.M.	14
	Inside volume	16.7 ft.^{3}
Cyclone— (for Dryer plus Cooler)	Diameter	16 inches
	Overall Height	7 feet
Fan	Melt Tank	⅛ HP
	Capacity=300 cfm at ⅛ SP	
Fan	Cyclone 2 HP (for handling dryer plus cooler)	
	Capacity=1000 cfm at 4″ static pressure	

this (5-6 units) is used to preammoniate the base. The remainder is added as preneutralizer fluid.

3. Since the acid is preneutralized, side reactions with muriate, dolomite and other solids are eliminated. All of the acid is thereby utilized, and also, fume problems are reduced.

Disadvantages

1. To obtain maximum benefits and formula flexibility, preammoniation of the superphosphate is required. This is not a serious drawback, since 4-5 units nitrogen solution may readily be batch mixed with the base.

2. Anhydrous ammonia cannot advantageously be used in the preneutralizer tank. Therefore the process is not suited for such grades as 4-16-16 and 5-20-20. This is not a disadvantage, however, if the preneutralizer is regarded as a supplementary piece of process equipment. It is entirely probable that the preneutralizer may be

used in conjunction with the TVA ammoniator — particularly if the TVA reciprocating pipe is used to distribute the fluid on the rolling bed.

3. There is a problem of startup and shutdown of the reactor. To avoid 1/2-3/4 hour startup time, the reactor may be shut down on the previous day's operation by adding some water to it, and keeping the contents hot with a small steam coil or electric heater. However, drainage of the reactor is necessary if a different acid is to be used.

4. Contact H_2SO_4 66° Bé cannot be advantageously used in the reactor. This is because it is necessary to replace all the water driven off by the reaction. Otherwise the fluid becomes so hot and dry that the ammonium nitrate will destroy itself and go up in smoke. If we replace all of the water we are driving off—we are then nullifying the basic purpose of the reactor.

5. In order to obtain the desired physical features of the process (i.e. prevention of puddling and overgranulation) it is necessary to sacrifice up to 5% of the total nitrogen input.

Production Rates

Output Capacity on 10 TPH Factory
Automatic Pug Process

Grade	Units N From No. 2 NS	Estimated Production Tons per Hour
8-0-24	8	14.5
10-0-10	10	13.5
12-0-10	12	12.0
14-0-14	14	10.0
14-0-14	12	12.0
12-24-8	12	10.0
12-24-12	12	10.0
13-13-13	13	10.0
20-0-20	12	12.0
14-14-14	14	9.0
14-14-14	12	12.0
15-15-15	14	8.5
18-18-18*	18	4.0

Production rates are limited by the tendency of a high-nitrogen fertilizer to melt, or "puddle" in the dryer.

*This water-soluble grade requires considerable recycle (66%), and could be made by using the recycle feed system for muriate base, and the base feeder for recycle.

Equipment Sizes

10 TPH Factory Unit

Melt Tank	Calculated	Recommended
Height	9.85 feet	10 feet
Diameter	3.28 feet	3 ft. 3 in.
Material		Mild Steel
Pug Mill		
Length	16.8 ft.	20 ft.
Screw Diameter	28 inches	24 inches
Discharge Opening		2 ft. x 2 ft.
Material		Mild Steel
Dryer & Cooler		
Length	22.6 feet	40 feet
Diameter	5 ft.	5 feet
Material		Mild Steel

Cyclone

Diameter—6 feet
Overall Height—24 feet
Inlet Duct—1½ ft. x 3 ft.
Outlet Duct—3 ft. diam.

Fan-Melt Tank 1 Hp

Capacity=4000 cfm at ⅛" SP

Fan-Cyclone (for hanling Dryer plus Cooler)

Capacity=15,000 cfm at 4" static pressure
HP=30

Comparison of Pug Process With Continuous Ammoniation Process

Units from No. 2 NS without recycle without pre-ammo. base. 12-12-12 grade	10 but not recommended	8
Units from No. 2 NS without recycle with pre-ammo. base 12-12-12 grade	5 + 7 = 12 batcher + pug	5 + 4 = 9* batcher + ammo.
Units from No. 2 NS with 30% recycle with pre-ammo. base 12-12-12 grade	Recycle not required	5 + 6 = 11*
Units from No. 2 NS without recycle 8-0-24 grade	8	4
Units from No. 2 NS without recycle 14-0-14 grade	12	5½*
Units from No. 2 NS with 30% recycle 14-0-14 grade	14	7*

*Estimated—perhaps slightly on the high side.

The unique characteristic of the pug mill process is that larger amounts of formula liquid can be handled. The 14-0-14 grades prepared in this work contained over 1300 pounds (722 No. 2 NS + 584 H_2SO_4) of formula liquid per ton of product.

CHAIRMAN SAUCHELLI: Thank you.

A word of explanation. When I was organizing this panel I'd already had the completed panel, then got into conversation with Arthur Hansen and thought he had something of interest, so we invited him to participate. I'm sorry that we had to rush him. He had a very fine paper; lots of work on there, but perhaps this whole subject seems to be of tremendous interest and maybe we'll have to continue it next year.

The next panelist is our good friend, Fritz Alfrey. He's going to tell us about this new development of W. R. Grace Research Laboratories.

MR. N. K. ALFREY (W. R. Grace and Company).

A Concentric Pipe Prereactor for Liquid Raw Materials in Mixed Fertilizer Manufacture*

N. K. Alfrey

MANY of you will remember the description of the Davison-Trenton granulation process presented at the 1956 Round Table by Mr. J. E. Reynolds. The process uses a double shaft pug mixer as an ammoniator-granulator. Usually each liquid raw material is introduced into the pug mixer through a separate set of pipes. The acid discharge pipes are paired with either anhydrous ammonia or nitrogen solution discharge pipes in several arrangements, all of which are designed to help assure that as much acid as possible reacts with ammonia in preference to the other raw materials.

Paired pipe liquid sparger systems are well known, not only in pug mixer amoniators, but also in the more common rotary drum type ammoniators. One major problem associated with the use of conventional liquid sparger systems is that some of the acids usually react with potassium chloride to form hydrochloric acid, which in turn reacts with ammonia to form ammonium chloride in the form of a dense white fume which constitutes an atmospheric pollutant. Another difficulty is incomplete ammoniation and possible decomposition of the various nitrogen compounds to nitrogen losses and a possible personnel hazard due to the toxic nature of some of the gases. Also, under certain conditions, the decomposition gases may cause flash fires in the ammoniator.

In an attempt to overcome these difficulties we developed a concentric pipe prereactor which permits all liquid raw materials to be combined and reacted, then introduced into the ammoniator in a single stream.

Figure 1 shows the general arrangement of the concentric pipe prereactor as it is now being used in three of our granulation plants. Each liquid raw material is metered and supplied to the prereactor through conventional instruments and piping which is not shown. Each supply line is provided as shown with a gate valve, a check valve and a pressure gage immediately upstream from the prereactor.

The water and nitrogen solution, supplied through 1½ inch pipes, blend in a 1½ inch pipe tee and subsequently discharge from a perforated 1½ inch pipe into a 6″ pipe section which is 42 inches long. Anhydrous ammonia is suplied through a 1½ inch pipe, then flows through a 3 inch pipe tee and enters the 6 inch pipe section where it blends with the nitrogen solution and water. The pre-blended liquids then flow through a 3 inch pipe section into a 3 inch pipe tee which is referred to as the reactor tee.

Sulfuric and phosphoric acids, supplied through 1 inch pipes, preblend in a one inch pipe tee and subsequtntly flow through a 1 inch acid discharge pipe which passes concentrically through the reactor tee and terminates in a simple showerhead arrangement 3 inches inside the reactor pipe section.

The showerhead is 1¾ inches outside diameter and has 25 drilled discharge holes, each 3/16 inch in diameter. Its purpose is to break up the acid flow into many small streams, thus assuring that neutralization by ammonia is largely completed in the 3 inch diameter by 4 foot long reactor pipe section.

The discharge pipe is 3 inches in diameter by 10 feet long and has eighteen ½ inch diameter discharge holes which are located along each side at 6 inch intervals. A 4½ inch inside diameter trough is welded into the midline of the bottom of the pug mixer for the purpose of supporting the prereactor discharge pipe. The preblended and prereacted liquids dis-

Figure I

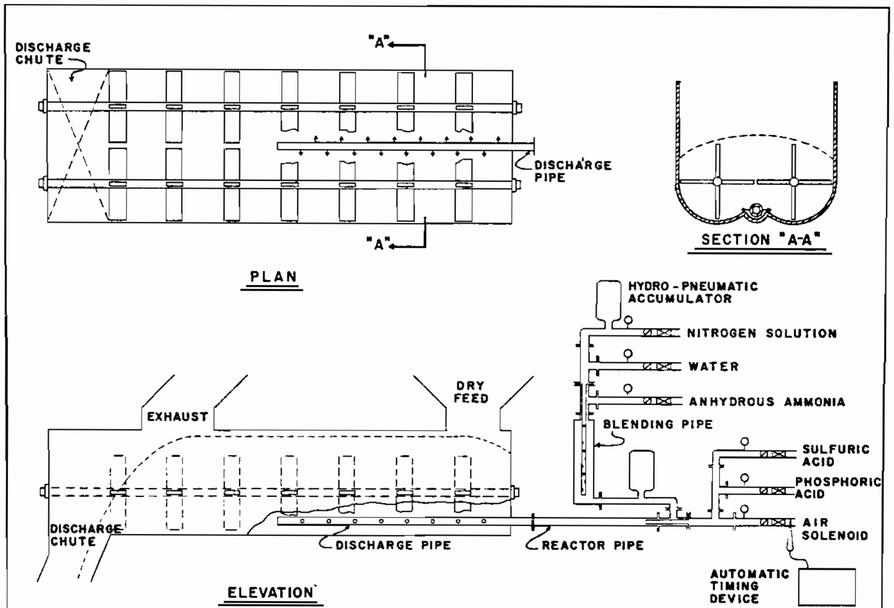


Figure 1. ARRANGEMENT OF PREREACTOR IN GRANULATION PUG MIXER

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*This description is based on information from the paper entitled "Prereactor For Liquid Raw Materials In Mixed Fertilizer Manufacture" by G. L. Bridger and N. K. Alfrey, presented at the ACS meeting in Atlantic City on September 16, 1959.

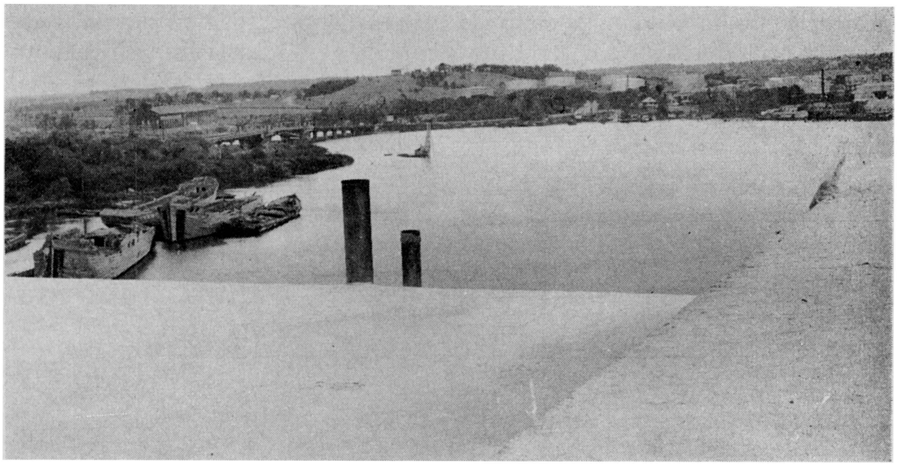

Fig. 3. Negligible visible fume from mixer exhaust stack.

charge laterally into the lower region of the pug mixer approximately 2 feet beneath the surface of the bed of dry materials. The design and location of the discharge pipe was made with due regard for efficient ammoniation of the superphosphates and uniform wetting of the dry materials.

A one inch compressed air supply line enters the blended acid line in a simple pipe tee. The air line is equipped with a solenoid valve which is arranged with an automatic timing device so that on a preset timing cycle which is usually five minutes, a short two or three second blast of compressed air passes through the reactor and discharge pipe sections and assures that the discharge orifices remain open.

Hydro-pneumatic accumulators (Greer Hydraulics, Inc., Brooklyn, N. Y.) are located in the nitrogen solution supply line and in the preblended nitrogen line for the purpose of dampening out pressure pulsations caused by the blending and reaction actions. Each accumulator is a 5-gallon steel vessel equipped with a spring loaded feed valve and contains a rubber bladder which is inflated to five pounds of pressure or about one-half of the line pulse pressure. In normal operation the line pressure pulses between 2 and 10 psig.

Sevaral corrosion resistant materials of construction are used for the various prereactor components. The pre-blending section is constructed of type 304 stainless steel. The reactor tee is type 316 stainless steel and the acid discharge pipe is Hastelloy C. The reactor pipe section is teflon lined mild steel and the discharge pipe is teflon lined type 316 stainless steel. The teflon lining has shown excellent resistance to the severe neutralization reaction environment. After 10 months of plant service, the original reactor section shows negligible deterioration.

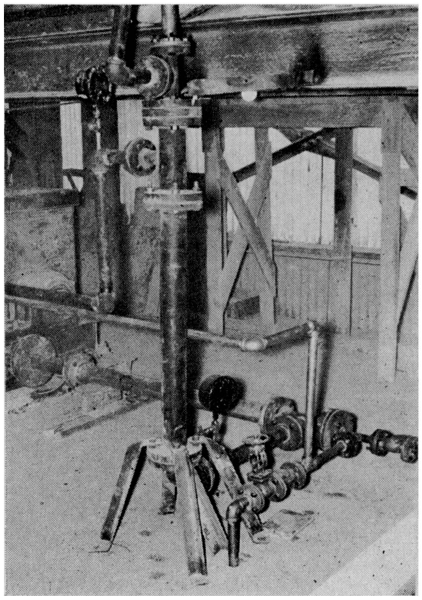

Fig. 2. An early plant prereactor installation.

Figure 2 is an overall view of an early plant prereactor installation before accumulators were added. The vertical section receives and preblends nitrogen solution, water, and anhydrous ammonia. The discharge pipe is visible entering the pug mixer and is flanged to the reactor pipe section.

Figure 3 shows the typical appearance of the mixer exhaust stack during granulating operations. The larger of the two stacks is not in use and acts as a control stack. The smaller is the mixer exhaust stack and a faint whisp of condensing steam can be seen emitting from it. The notorious white mixer exhaust fume cloud is virtually eliminated by the prereactor. No odors are normally detectable in the gases, however, at the higher ammoniating rates a slight odor of ammonia can be detected.

Tables I and II show typical liquid usages and plant operating data with prereactor. The prereactor is used for production of all grades required by the plant. This is believed to be an advantage over other preneutralizers. The different 5-20-20 formulations are shown to indicate the flexibility of the prereactor. Recent experience has indicated that when using wet process phosphoric acid, it is important to start the liquid flows in the order (1) water, (2) nitrogen solution, (3) anhydrous ammonia, (4) sulfuric acid (when used), and (5) phosphoric acid. Starting the wet process phosphoric acid

Table I. Typical Proportions of Liquids Prereacted

Lbs./Ton of Fertilizer Product

Grade	Anhydrous Ammonia	Nitrogen Solution*	Sulfuric Acid (66° Be' H_2SO_4)	Phosphoric Acid (75% H_3PO_4)	Additional Water
3-12-12	77 (3) **		105		210
5-20-20 (Formula A)	125 (5) **		125		350
5-20-20 (Formula B)	98 (4) **	50 (1) **	105		300
5-20-20 (Formula C)	77 (3) **	96 (2) **	55	130 (3½) ***	275
10-20-10	48 (2) **	410 (8½) **	80	150 (4) ***	None
12-12-12		510 (10½) **	115		None
15-15-15		750 (15½) **	225	150 (4) ***	None

* 41.4% N, 6% Urea, 19% free NH_3, 65.6% NH_4NO_3, 9.4% H_2O
** Units of N.
*** Units of P_2O_5

Table II. Granulation Plant Operating Data With Prereactor.

	3-12-12	5-20-20 (Formula)	12-12-12	15-15-15
Production Rate (Tons/Hour)	36	36	20	15
Recycle Rate (Tons/Hour)	25	25	25	40
Water Usage (Lbs/Ton Produced)	210	350	None	None
Material Temperatures, °F				
Recycle Entering Mixer	125	115	120	120
Material Leaving Mixer	205	200	170	175

flow last is apparently important to preventing internal clogging. This circumstance is a recent occurrence which was not encountered with electric furnace acid and our experience is thus limited. However, we believe that with the correct startup procedure and perhaps with minor changes to the discharge section, wet process acid can be used satisfactorily.

Both 60° Be′ and 66° Be′ sulfuric acid have been successfully used in the formulations. Nitrogen solutions containing both urea and ammonium nitrate are commonly used without apparent decomposition occurring. However, our tests for evidence of decomposition are not completed. We believe that since all ammonia formulated passes through the prereactor, the resultant high pH of the acid neutralization environment may prevent or greatly reduce decomposition of the nitrogen compounds.

The production rates, recycle rates and water usages shown are not appreciably different from those when we were using paired pipe spargers in these plants. The temperatures shown are also similar to our previous experience, except that with the 3-12-12 and 5-20-20 grades, the temperature of the material leaving the mixer is between 10 and 15° F. higher than with paired pipes. This is believed to be caused primarily by improved ammoniation and decreased ammonia losses which result in more heat being released from the neutralization reaction.

In conclusion, we don't believe that the concentric pipe prereactor is the final answer to the liquid distribution problem. Instead, we prefer to consider this presentation to be a progress report on the present state of development of a prereactor which has been shown in plant operation to have certain advantages over other systems. Further experience will undoubtedly result in improvements and may also reveal limitations which are not now recognized.

Chairman Sauchelli: Thank you, Fritz. That certainly was an excellent presentation.

Now we're ready for questions. Our panelists are prepared to answer your questions and I'm sure there must be a lot of interest in these discussions. Remember this discussion period is an important function of our Round Table and we look to the discussion period with a great deal of interest.

Mr. L. V. Clegg (Canadian Industries Limited): I understand that you must be careful not to use waste acid in this preneutralization system. Is this applied to all waste acids or just certain types?

Chairman Sauchelli: Who wants to answer that?

Mr. Gilliam: We do not necessarily say that waste acid can't be used, but if you use waste acid you'll want to know the composition of its impurities and what effect this will have chemically on your other materials in your preneutralizer or prereactor at the temperatures at which you propose to operate.

Chairman Sauchelli: Grant, do you have any comments?

Mr. Marburger: I think we have said in the interests of safety that spent sulfuric acid shouldn't be used in the preneutralization tank. This is based on just the fact that the organic material could become nitrated and accumulate in the system. There's no positive proof on this, but this is the worry.

Chairman Sauchelli: Art, do you want to answer?

Mr. Hansen: No, I don't think so.

Chairman Sauchelli: Any other comments? Phil?

Mr. Stone: I'd just like to say that I think spent acids vary too much in composition for us to say here whether they can or they can't. It just depends on the composition of the acid as has been pointed out. It might be certain spent acids that have certain acceptable compositions can be used very satisfactorily, but I think again, it's a matter of knowing the components that make up your spent acid.

Chairman Sauchelli: Frank, do you have something?

Dr. Keenan: No.

Chairman Sauchelli: What's the next question?

Mr. Richard Powell (International Mineral and Chemical Corporation): I'd like to ask Phil Stone—what is the size of the hose in the bottom of the trough; secondly, you mentioned you're using an anti-caking; is that coating agent—where do you have it?

Mr. Stone: The first question, the diameter of the hose in the bottom of the trough that distributes the hot liquor onto the tumbling bed of solids is a half-inch.

The second question had to do with the type of anti-caking agent that's applied?

Mr. Powell: Yes. where do you have it?

Mr. Stone: This is applied at the time—at the cooler discharge before storage of the product in bulk pile. We have been doing this right along because in the first place it offered a good point at which we could apply it and probably reduce caking in the bulk pile.

We have used two or three different coating agents. We are, right now, in the process of testing a third one. I'm leaning strongly towards the kiln and clay type of coating agent in preference to some of the others to cut down on this dusting that occurs in the factory, sometimes to the extent where our tractor operators can't see to run their vehicles. That's the type of coating agent we're using and we're using maybe, 20 pounds to the ton and sometimes higher.

Chairman Sauchelli: Any comments or questions on this side of the room?

Mr. Walter W. Whitlock (Texaco, Inc.): I have a comment I'd like to make. Several years ago several of my colleagues in the East St. Louis Plant of the Illinois Farm Supply Company did a little experimenting with what we called an external reactor. We were running a pilot plant at the rate of about a half ton or a ton an hour and we used a two-foot section of three-inch pipe for preneutralizing the solution B and sulfuric acid in the absence of an agitator or a flight mixer or ejector pipe agitator. In order to get a little more intimate mixing we filled the pipe with glass marbles.

Needless to say, the reaction was quite violent. We lost some of our marbles. (Laughter.)

I should be careful in saying that. Most of the fellows are here, Ben Anderson, Walt Monti, and Seymour and so forth. I say this in pointing out, after listening to all the fine papers and presentations here this morning that there is no one way to accomplish any particular end result.

I think that we all should be very careful and cognizant of these facts and know what we are attempting to do in order to follow the end result. If we want to use an all urea solution we go along with that; if we want to use an ammonium nitrate solution we have another type. We have a pug mill; we use the type of ejector that the Davison people have described and there is no standard pat answer as we can see here. the right line or track in getting the end result. These fellows have done a wonderful job, I think, in clearing up the muddy waters for a lot of us.

Chairman Sauchelli: Thank you, You're trying to stress the principles and as has been said, there are no two plants in the fertilizer industry alike and it makes it very difficult for standardizations in it.

Do you have a comment?

A Voice: The comment there brought a thought to mind that might be worth mentioning. Although we haven't done any test work in that direction yet, we know of no reason why the concentric pipe prereactor could not be used in rotary ammoniators.

Chairman Sauchelli: Other coments?

Mr. Bruce Sherman (Olin-Mathieson Chemical Corporation): I'd like to ask Mr. Stone how many units of nitrogen he's getting on 14-0-14 and what recycle rates.

Mr. Stone: We're getting about ten units of nitrogen from solution, four from sulphate. Generally, we have varied it back and forth, but this is about what we've settled on and on what we operate at present.

The second portion of your question had to do with amount of recycle. Recycle amounts to about a quarter to one. Sometimes a little higher, sometimes a little lower, but with good favorable particle size on the part of sulphate and other solids and a good operator you needn't have recycle any larger than 25 per cent.

Chairman Sauchelli: This gentleman has been recognized down here.

A Voice: I'd like to ask Mr. Alfrey—the water you're putting in your system now, are you controlling your reaction too as you also control your granulation? You show water, but you don't say how you determine what goes in as water.

Mr. Alfrey: That's an excellent question that I didn't clear up in the description. The water being used is based strictly on requirements for granulation. There are no other considerations given. One consideration we do have which is a policy generally insofar as order of introduction of the liquid goes. We usually start the water off first.

Chairman Sauchelli: I saw two hands here.

Mr. Charles Everhart (U. S. Industrial Chemicals Company): I'd like to ask Mr. Alfrey if he would discuss his problem with the wet process acid. Do you think the seriousness of it will prevent the use of it in his type of operation?

Mr. Alfrey: I'd be happy to hazard a guess in answer to your question. Actually the difficulty that we're encountering is with large ammonia dosages and reasonably large phosphoric usage and at least medium to small usage of water. Consequently the difficulties are tendencies of the sales, that are formed from the reaction between the acid and the ammonia, to collect on the inner surface of the pipe and act as an interference to the flow.

My own optimistic guess on this thing is that some of the other difficulties that we encountered previously during this first year of plant operation were a lot greater than this difficulty and some of those, I thought, we might not be able to solve and we ended up solving them; but this one looks rather small in comparison and I think we can solve this minor discharge pipe design arrangement, but I might be wrong.

Chairman Sauchelli: Question. Tom Hoshall?

Mr. Tom J. Hoshall (Farm Fertilizers, Inc.): I would like to ask Mr. Alfrey how did you get rid of the steam out of the pipes.

Mr. Alfrey: The photographs that you saw of the exhaust system with steam visibly condensing in the exhaust stack is the sole point of discharge of the steam for the system. The steam comes up through the mass of material and dissipates into the exhaust system.

Chairman Sauchelli: A question here?

Mr. Larry Lortscher (Spencer Chemical Company): I'd like to make an observation. I think it would help answer Mr. Hoshall's question. I notice in the recycle rate put on the slides that Mr. Alfrey's system has a tendency towards higher recycle requirements on some of the higher nitrogen grades than with the open tank system.

So that the steam which he may be talking about, may be the neutralizer flushing off and going out the stack. In his system, he's not got quite as low a recycle, although he's got other advantages.

Chairman Sauchelli: Do you agree, Fritz?

Mr. Alfrey: Yes, I sure agree with what he's speaking of there as a possible expectation. So far, I might comment, we have operated this device almost completely according to accepted plant experience and previous plant procedure. We're quite anxious to get into some test work with some 2-1-1 grades and some X-O-X grades to see what we can do, but I would expect, as a general comment, that what Mr. Lortscher here has said, would be true. To what extent it's true, I don't know.

CHAIRMAN SAUCHELLI: Any other comment?

MR. MARBURGER: I'd like to add a little more comment to that In the open tank we think of preneutralization in terms of coming out of the tank, and the slurry going into the bed of the ammoniator at a very low moisture content. This isn't an absolute necessity, but down into ranges of one per cent water in the slurry exit and when you're down into that low you do get into the large amount of steam flash off.

CHAIRMAN SAUCHELLI: Tom Hoshall.

MR. HOSHALL: I'd like to ask Mr. Alfrey if we can get some plans for this thing.

MR. ALFREY: I appreciate your question — I don't know if this is a fair answer or not. The paper that I took my description from was presented to the recent ACS meeting and now belongs to the American Chemical Society. We have submitted it to them for their choice of publication. They have the publication rights on the paper. Does that answer your question?

Maybe Dr. Bridger or somebody could elaborate on that; I don't know.

CHAIRMAN SAUCHELLI: Is Dr. Bridger here? I think we're going to have it for the proceedings, I'm sure.

DR. G. L. BRIDGER (W. R. Grace & Company): If anyone is interested, just write us and we will try to tell you what you want to know.

MR. RODGER SMITH (Eastern States Farmers' Exchange) I know there are some views among the panel on safe operating temperatures of these preneutralizers. It might be expanded upon a little more and secondly, possibly Mr. Alfrey might give a little information as to what temperatures they do experience in the pipe, the reactor pipe, in his unit.

MR. ALFREY: So far as precise data showing actual temperatures inside the pipe during operation with the various formulations are concerned we haven't gathered that type of data.

We have a general impression that the range of temperature we encounter varies, depending on concentrations of the acid, the amount of acid and ammonia usage and the production rate. But we think, speaking of the very low nitrogen grades up to the high acid usage grades, we're probably ranging from somewhere in the neighborhood of 220 degrees Fahrenheit to temperatures under 400 degrees Fahrenheit.

I think the life of the thin Teflon liner has indicated that we're not approaching temperatures that would begin to shorten Teflon life.

CHAIRMAN SAUCHELLI: Question?

MR. CLARENCE B. KIRBY (Allied Chemical Corporation): With the reactor pipe what would happen if sulphur were to stuff the holes up completely?

MR. ALFREY: I assume if the entire discharge system were clogged what would occur inside the pipe particularly with the temperatures?—That has happened to us, once, only. This was on a very early prototype where our Teflon lining was not anchored down on the discharge pipe and its rate of expansion was different from that of steel and it expanded over the holes and clogged them.

The pressures that we encountered were just equal to the maximum pressure on the supply system. In this particular circumstance we were using anhydrous ammonia in the formulation and the maximum pressures that we encountered were equal to the anhydrous ammonia pressures.

I might mention a safety feature on that point. We have a policy in our plant of offering a pressure relief vent on any anhydrous ammonia system between valves. A vent of this type just beyond the most downstream valve of the anhydrous in this system, I think, would take care of all pressure problems of that type.

Does that answer your question?

CHAIRMAN SAUCHELLI: Does that answer your question all right?

MR. KIRBY: Yes, sir.

CHAIRMAN SAUCHELLI: Question here?

MR. EDWARD A. HEISLER (Baugh & Sons Company): I have two questions for Mr. Stone.

First, is an agitator necessary in this type of reaction vessel? What would be the location of the preneutralizer? I presume you'd want it as close preferably over the ammoniator, but that's just my supposition and third, what sort of control tests are necessary to keep it running?

MR. STONE: Your first question, is an agitator necessary. We were very hopeful that an agitator would not be necessary and we tried to do without it, just as we tried to do, and were successful in doing without this little fan that we originally put in the stack to take water vapor away.

When we tried to operate without an agitator in the tank we had considerable nitrogen loss in the form of ammonia from the tank and we concluded that an agitator was necessary. But I would like to say that I'm not at this time completely sure that it isn't possible to operate without an agitator and our attempts to operate without an agitator have not ceased. We are hopeful that there will be some way to go about this. Right now our liquid level above the spargers in the tank is, in my opinion, too low. It's only about 18 inches and when we put in the new tank in another few months this tank will give us a deeper liquid level above the spargers in the bottom of the tank and at that time I'm hopeful that we can attempt again to operate without the agitator because this is a direction towards simplicity and low cost which I think in the fertilizer business we're all after.

The location of the preneutralizer is, as you suggest, on the floor level above the ammoniator-granulator that we use and located in such a way that the overflow discharges into a three-inch line and flows directly down into the entrance to the ammoniator-granulator where the solids enter and it is at that point that the hot liquor is distributed on the bed of tumbling solids.

Your last question, I think, had to do with the control of the operation. The only controls that we have for the operation is temperature and this is regulated by means of water added to the reactor tank. We prefer not to add water at this tank and many times we do not add water at the tank, but we have temperature that is recorded at the tank and registered on the control panel downstairs where the operator is situated.

Mr. Heisler: I'm sorry, Mr. Stone, my question really was pH control. I imagaine you have to operate slightly on the acid side to avoid ammonia losses. I was wondering whether you run periodic tests of that sort of thing on that?

Mr. Stone: We think pH is probably a very important problem. We're always after a simple, easy way to do things to keep it that way for our operators. We have no idea what the pH is on the hot liquor discharge from the tank. Our controls do not involve pH measurments; we, at one time thought about it and we still turn this over in our mind, but it's another function for a man to perform and we've been successful without the use of any pH control. The only thing that we do is to calibrate our liquid flowmeters with good accuracy and make damned sure that the liquids are entering the tank in the proper proportions to each other.

This may give us a little excess acid to make sure that we catch all of our anhydrous. We don't think this is a disadvantage in making X-O-X grades because we have plenty of limestone downstairs to take care of this little additional acid.

Chairman Sauchelli: We have time for a couple of more questions.

A Voice: I believe Mr. Marburger mentioned some test for acidity which was pretty accurate. Could you describe that?

Mr. Marburger: I'll touch on it. I have a procedure written up that explains it fully, but it's a matter of the operator taking a little dipperful of the slurry coming down and then with a 100 milliliter beaker; and he does need a pipette. These other chemicals involved are tenth normal sodium hydroxide and some methyl-red indicator and this will give him the per cent of acidity as sulfuric acid and he'll quickly find an operating range of maybe it's .2 or .5 or 1. I think we said originally two per cent. Above that you're getting into fuming problems, although that frequently is too high.

Chairman Sauchelli: Another question?

Mr. Everhart: Mr. Marburger or Mr. Gilliam, do you experience blockage in the discharge air line of the open tank, If you do, do you have any control device to cut off your system?

Mr. Gilliam: We would not expect any blockage in the discharge line of your slurry from your neutralizer tanks since we purposely overdesigned the size of that so as never to run over half full. Now you can picture under certain conditions of temperature that if for some reason you're shut down and material has a chance to salt out, that line can become blocked.

What little experience I've had with commercial units, most of them have an emergency overflow line installed above if such blockage should take place; but, I believe this is a minor problem and it's recognizing that you're dealing with a slurry that does have a salting out temperature at something above room temperature, depending upon the water content of the slurry.

Does that answer your question?

Chairman Sauchelli: How many folks here would think we ought to put this subject on our program next year? There would probably be more plant experience and so on. Let me see a show of hands of those that think we ought to continue.

(A showing of a majority of hands.)

It's of increasing interest and perhaps we'll consider it. That leads me to urge once more upon you all that we need ideas for next year's program. It's our concern to make next year a better program if possible.

I'm sorry we have to cut off here. You've been a grand audience and cooperative and on behalf of the Executive Committee I want to thank all speakers for their generous participation; their giving us their time and a lot of work put into these papers. I also want to acknowledge publicly my appreciation to the National Plant Food Institute for permitting me to devote time and secretarial service for work on the Round Table.

I think from the comments that I've heard, we've had a fine meeting this year and we all look forward to convening again next November. Let us all hope that we'll have as good if not a better meeting.

We stand adjourned then until 1960, November 2, 3 and 4.

(Applause.)

(The meeting adjourned at twelve forty-five o'clock, p.m.)

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PROCEEDINGS
OF THE
FERTILIZER INDUSTRY ROUND TABLE
1960

Held at the
MAYFLOWER HOTEL
Washington, D. C.
November 2-4, 1960