PROCEEDINGS

OF THE

FERTILIZER INDUSTRY ROUND TABLE

1960

Held at the
MAYFLOWER HOTEL
Washington, D. C.
November 2-4, 1960

Executive Committee

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Wednesday Morning Session, Nov. 2, 1960

The Tenth Annual Meeting of the Fertilizer Industry Round Table convened at ten o'clock a.m., in the Colonial Room, Mayflower Hotel, Washington, D. C., Messrs Reynolds and Spillman presiding.

Moderator Reynolds: It is my privilege on behalf of your Executive Committee to welcome you to your Tenth Anniversary Session of the Fertilizer Industry Round Table.

The previous nine Round Table meetings have been called to order by our mutual friend and colleague, Dr. Vincent Sauchelli.

Dr. Sauchelli is regretfully absent from this year's meeting due to an extended assignment to India and the Far East. We've become accustomed to the enthusiastic welcoming spirit with which Vince opened and launched the Round Table program.

For this reason your Executive Committee requested Dr. Sauchelli to record his personal greetings and remarks for the group prior to his departure.

To my knowledge no one has heard the transcription which we are about to hear. The contents are totally unrehearsed with your Executive Committee and known to Vince alone.

It is my privilege and honor to introduce Dr. Vincent Sauchelli's transcription.

Dr. Vincent Sauchelli (Chairman, Fertilizer Industry Round Table): Friends and fellow members of the Fertilizer Industry Round Table; this is Vince Sauchelli speaking.

Once more it is a real pleasure to greet you to our Annual Meeting. This is our Tenth Anniversary.

As I speak to you memories of previous meetings flood my mind. I had looked forward to being with you at this Tenth Anniversary, but circumstances have decreed otherwise; this is the first meeting I will have missed. Although I am in person in far off India, believe me when I say, I am here with you in spirit.

My sincere wish is that you make this tenth meeting memorable for the quality of the discussions and the worthiness of the information you'll freely exchange among each other.

Your Executive Committee has worked hard to organize an acceptable program. As the technology of our industry advances more and more into modern engineering and quality control practices it becomes increasingly important to establish standards in materials and products of manufacture; precision, reproducibility, define specifications; these attributes are insisting to be recognized and respected.

Our program reflects industry thinking on this phase. Phosphoric acid and phosphates have always been an integral part of our industry operations. The heightened interest in this subject is shown by the agenda we have set up and the numerous papers devoted to it at the American Chemical Society Meeting in September.

All in all I feel sure that my committee colleagues in particular and you members generally will do everything possible to make this, our Tenth Meeting, the best yet.

These meetings have been distinguished from the very first as a forum for the operational personnel of the industry. Here, practical information, based on plant experiences, has been frankly and honestly exchanged. Every one here in this audience is welcome, indeed urged, to take part in the discussions. You'll never know how helpful to others in a similar situation your remarks can be.

Furthermore, remember we need your suggestions for the 1961 program. Start thinking about that now and let the Executive Committee have them before you leave for home.

I shall hope to be with you at next year's meeting; perhaps I may be able to report to you at that time my observations on developments taking place in Asia in our rapidly expanding fertilizer industry.

Good luck and best wishes for a most successful Round Table.

Moderator Reynolds: Reference to our Tenth Anniversary causes us to pause and review the history of our group.

The Round Table is an outgrowth of the foresight and interest of an unselfish, very small group of men. We are indeed indebted to these men who had the courage to guide the Round Table through a period when exchange of information was practically frowned upon.

The early sessions permitted only limited note-taking and a very cautious choice of words were reflected in the discussion periods.

The Round Table has prospered and advanced because of the feeling of belonging and the realization of the individuals that exchange of information is a two-way street.

You also receive when you give freely.

The theme of spontaneous question and discussion for production men was instantaneously appealing. No group existed which permitted the individuality and informality that was possible with this new group.

The Round Table belongs to no group, nor does it have any group affiliation. Your Round Table is composed of mutually interested persons who volunteer to accept responsibility when called upon. No one has an axe to grind; no one is seeking personal limelight.

The Round Table is this group as a whole, accepting the challenge to be of service to your colleagues and your industry.

A review of the proceedings of the past five years reveals hundreds of pages of priceless technical and informative data; many hours of thought in careful preparation went into these discussions. Our industry has advanced further technically in the past ten years than any previous ten year period.

We are proud that the Round Table has played such a prominent role in this movement. Exchange of ideas and information often strikes home and aids in removing obstacles standing in the way of progress. We can be proud that practically every company producing raw materials and finished fertilizers has appeared on the program at some time in our history.

It would be difficult to select a breakthrough year when companies really opened up their secrets; however, the 1956-1957 period would come the closest to this transition. The Round Table was openly, gradually accepted, but the success of open discussions was indicated early by those persons attending the sessions.

We have come a long ways and can be proud of our accomplishments. We can not overlook the importance of the after hour sessions which are possible in the halls, rooms and corridors.

These personal contacts and exchanges of information are most valuable to our every day performance of duties. Ten years ago plant superintendents and operating people would not have the opportunity to meet other persons in comparable positions in the fertilizer industry, let alone exchange information with them.

The 1960 Fertilizer Industry Round Table has again been prepared from your oral and written comments and suggestions. Your Executive Committee has pursued all possible leads to reflect the actual interests of the group. Our theme of short, thought-provoking presentations has prevailed through these ten years.

We recognize that a group of this size does not afford the same informal atmosphere of the group of five to ten years ago. However, we believe that the general framework has been preserved.

We welcome and solicit suggestions of how we can keep the program appealing.

The subject themes for the 1960 program will be phosphates, type, source, characteristics, standard and uniform raw materials, instrumentation and chemical control, and preneutralization.

These subjects were considered timely for our everyday needs. The many forms of phosphates are returning in importance as the key to increased plant food concentration and as the anchor for increased ammoniation ratios. With the trend towards higher analysis mixed fertilizers it is imperative that we know the sources and characteristics of our raw materials. The day when we could remix an unbalanced formulation is passing. The analyses must be right the first time through.

Uniformity and consistency are vital to the manufacturer and ultimately to the farmer. We cannot overlook the increased control efforts of state fertilizer regulatory agencies who also demand uniformity.

Instrumentation has been included in the 1960 program to permit the exchange of information for better understanding between the manufacturer and the user of these instruments.

During the past two years case histories have been reported which indicates a misunderstanding between instrument selection, maintenance and operation. Chemical control remains as a yardstick for our accomplishments.

The importance of proper sampling and the statistical evaluation of potential nitrogen losses will be further illustrated. Preneutralization received considerable interest last year and a desire for a continuation of this topic was expressed with a background balance from research and development, technical service and operating people being highlighted.

We are indeed fortunate to include on our program so many highly respected and qualified representatives of the fertilizer industry. We are grateful for the high percentage of acceptance when these men were contacted.

The Executive Committee performs as a unit in the preparation of the program and we appreciate your encouragement and assistance. I personally wish to thank the other members of the Executive Committee, Doc Marshall and Al Spillman for their cooperation and untiring efforts to make the Round Table Program a success.

So without further remarks I declare the 1960 Fertilizer Industry Round Table in Session.

Our regular business meeting is scheduled for Friday; however, an auditing committee is required at this time and I would like to appoint Joe Bosman and Frank Nielsson to perform this task. I think they are familiar with this type of procedure.

The Friday Business Session will also entertain suggestions and ideas for the 1961 Round Table. As Vince highlighted in his talk we need your suggestions and we want them. Only by your continued outwardly expressed interest can we grow and advance. Do not hesitate to offer subject themes and questions.

MODERATOR REYNOLDS: Let us get into our program. Our first topic, as indicated on the program, is Phosphates. We will give practically the entire day to this subject; I believe there will be a lot of information here and once again we want your questioning. This is really the backbone of the group; it's one of the reasons why people attend, to get comments, no matter what the question is; if you feel like you have a question, speak up.

I might mention one other thing; as far as your questions and also the answers both of the mikes in the back are hooked up to the front so we would urge each one of you who have a question to go to the mike, state your question so that it will be recorded. Also give your name. In case the name is not given, we will try to fill in or someone pass the name up.

Also in the case where we do have a panel indicated, if those persons would come to this center round table, it would be greatly appreciated. In case of individuals, move as close to the front of the room as possible.

We'll get along with the program.

During the past ten to fifteen years we have seen inroads of nitrogen solution containing varying amounts of anhydrous ammonia, ammonium nitrate, urea and water and the use of larger amounts of anhydrous ammonia in the mixed fertilizer industry.

The potash industry received considerable attention during the growth of granulation, whereas prior to this period the influence of high grade potash materials was largely neglected.

Now we are encountering a swing to the hows and whys of phosphates. Of course superphosphate and triple superphosphate were discussed in the ammoniation sessions of the 1956 and 1957 Round Tables.

These considerations were based largely on physical characteristics and operating results. Now the fertilizer industry is engaged in multi-phosphate formulations using normal and triple superphosphates, wet and furnace grade phosphoric acid, wet and furnace grade superphosphoric acid, calcium metaphosphate and diammonium phosphate. Reference to mixed fertilizer, mixing equipment as dry mixing is now restricted to the mixing of dry raw material ingredients rather than the process for finished mixed fertilizers.

We further need to more clearly identify wet mixing between the production of superphosphate and that of production of granulated mixed fertilizer.

A new concept in processing has occurred within the short span of three to five years. With the continued increase in the tonnage of highly concentrated plant foods more concentrated forms of phosphates are imperative to achieve the desired level.

The opening session of the 1960 Round Table provides an overall discussion of phosphates, their forms, types and characteristics. The basic form of phosphate to our industry is phosphate rock.

Our first speaker, Dr. Engibous, Manager of Agricultural Chemical Research for International Minerals and Chemicals, will pinchhit for Dr. LeBaron who is presently out of the country.

Dr. LeBaron's subject is the "Present and Future Status of Phosphate Rock."

Phosphate Rock — Availability, Grade and Usage

I. M. LeBaron

Introduction

YOUR program carries the title of this paper as "Phosphate Rock – Availability, Grade and Usage." In the brief time alloted an attempt will be made to follow phosphate from the field to the plant where beneficiation by physical and chemical means is accomplished.

With your permission, I will use IMC's operations for my example. Major emphasis will be placed on the techniques employed to control the quality of products to meet the exacting demands of the customers for phosphate rock. The presentation might be titled "Phosphate Rock Production and Quality Control."

Prospecting and Mining

It is essential that the areas to be mined be carefully prospected in order to determine the depth of overburden, matrix, yield of phosphate per acre, as well as the grade of product which will be produced. A critical management decision, that of selection of areas to be mined, is based on the prospecting data. Here we see a prospecting team with the drilling rig and a prospect core sample being prepared for transfer to the laboratory for analysis.

The pebble phosphate deposits usually occur with an average of 15 to 20 feet of overburden which may contain some phosphate, and underneath that is the matrix which varies from 10 to 30 feet in depth. In addition to the phosphate pebble, the matrix contains large amounts of clay slimes and silica sand. In this slide we see that the overburden has already been removed by draglines and dumped into the previously mined out cut. Digging of the matrix can now be accomplished. By means of hydraulic guns typified in this slide the mined matrix is washed into the suction of sand pumps from which point it is pumped through pipes to the beneficiation plant.

Concentration

This is a very simplified flow sheet of IMC's Noralyn plant near Bartow, Florida. The feed matrix enters the washer section where preliminary screening is accomplished. In the sizing section, the hydro-separator removes slimes and the remainder of the product passes Akins screw classifiers. The +14 mesh product comes off as pebble concentrate. The –14 mesh feed proceeds to the spirals section where the +35 mesh fraction is removed as spiral concentrate. The flotation section handles the –35 mesh material to obtain the float concentrate.

Let's take a quick look at a few slides of this process. This is a 175-ft. hydro-separator, and here are the Akins screw classifiers. You will recall that the spirals give us the +35 mesh phosphate values, and the float cells treat the –35 mesh feed to give us the float concentrate.

This has been a hurried picture, and those interested in a more detailed presentation are referred to such sources as Waggaman[1] and Sauchelli[2]. The final concentrates are placed in shipping bins and ultimately all concentrate products are sent to wet storage facilities for storage by grade. Here we see the end of the storage pile tunnel and the technique used in loading from the pile by blending different grades which are then sent to the dryer prior to drying, grinding and/or shipment.

Note the final quality control check point as the car is loaded. The flow of rock is sampled one cut from each of three chutes for each hopper car loaded. No less than six cuts are made from the flow of rock at intervals uniformly spaced throughout the loading operation in the case of box cars.

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[1] Waggaman, W. H., 1952. Phosphoric acid, phosphates and phosphatic fertilizers.

[2] Sauchelli, Vincent, 1960. Chemistry and technology of fertilizers.

Florida Phosphate Shipments

The domestic consumption of Florida phosphate rock continues to increase (Table 1). Since 1959, the increase has been over 35%. Phosphate shipments from the Florida operations can be subdivided into three general categories (Table 2)—wet rock, dry rock unground, and dry rock ground. Wet rock shipments refer to concentrates which are sold in the natural state without being dried, and are usually used in the production of elemental phosphorus. Dry run-of-mine (Table 3) rock is sold both domestically and abroad, in seven base grades from 64 to 77% BPL. The dry rock grades are ground for certain applications and sold on the basis of fineness of grind (Table 4) in the five grades shown on the slide. As you know, the dry ground rock is used for both direct application and acidulation purposes.

Table 1. Domestic Consumption of Florida Phosphate Rock

Year	10^3 Long Tons
1949	6,816
1957	8,039
1958-59	9,162

Table 2.

Wet Rock
Dry Rock, Unground
Dry Rock, Ground

Table 3. Dry Rock, Unground

7 base grades (% BPL			
64	68	72	77
66	70	75	

Table 4. Dry Rock, Ground

5 base grades (% BPL)		
66	72	77
68	75	

Table 5. Guarantees

Dry Rock, Unground
    Max 3% H_2O
    ROM Size 1.5 in. to 200 M
    Max 4% I + A (5% at 66% BPL)
        includes $0.45/ton drying cost
        add $1.50/ton for 90% + 10M (pebble)
Dry Rock, Ground
    Add $0.40/ton grinding to 48/52%—200 M
        0.49   58/62%
        0.58   90%—100 M

For the benefit of those of you who are not involved in phosphate products, it might be well to quickly review the specifications of these materials (Table 5). The guarantees for unground dry rock carry a maximum of 3% moisture and 4% I and A values. The I and A maximum on 66 rock is 5%. Rock prices include a 45 cent per ton drying cost, and the ground rock prices include the grinding costs which appear in the silde. Rock phosphate prices begin at about $4.50 per ton.

Now let us look more critically at the product specifications on rock shipments. General domestic shipments (Table 6) for the base grades of 66 through 77 BPL product are shown in this slide. Note that the desired monthly average value is in the upper allowable range for shipments. A comparable situation exists in the direct application shipments, (Table 7) and in the acidulation rock shipments (Table 8). Of critical importance in acidulation material is grind. It should be pointed out that while the desired maximum +35 mesh specifications are low, it is often the practice in Florida operations to dress the car with a small amount of pebble to facilitate product discharge at the destination.

Chemical Analyses of Shipments

In general, this is the blending schedule (Table 9) followed in achieving the various BPL grades for sale. The major portion of my remarks and slides will deal with the measure of success achieved in meeting the grade and grinding specifications.

An analytical program was set up to check the variability in the major chemical constituents of shipped phosphate rock. Analyses were performed on the four most

Table 6. Domestic Rock Shipments

Contract Base Grade	Car Minimum BPL	Desired Monthly Average BPL	Contract % I and A	Maximum % Moisture	Allowable Range for Shipments
77%	76.00	77.25	4.00	3.00	76.90/77.30
75%	74.00	75.25	4.00	3.00	74.90/75.30
72%	71.00	72.25	4.00	3.00	71.90/72.30
70%	69.00	70.25	4.00	3.00	69.90/70.30
68%	67.00	68.25	4.00	3.00	67.90/68.30
66%	62.00	66.00	5.00	3.00	65.80/66.20

Table 7. Direct Application Shipments

30%P_2O_5	65.55	66.00	None	3.00	65.80/66.20
31%P_2O_5	67.74	68.25	"	3.00	68.05/68.45
32%P_2O_5	69.92	70.25	"	3.00	70.05/70.45
33%P_2O_5	72.11	72.25	"	3.00	72.05/72.45
34%P_2O_5	74.29	74.50	"	3.00	74.30/74.70
35%P_2O_5	76.48	76.75	"	3.00	76.55/76.95

Table 8. Acidulation Shipments

Contract Grind	Contract Minimum Grind	Maximum Grind	Desired Maximum + 35 Mesh
50%—200 Mesh	48%—200 Mesh	52%—200 Mesh	2%
60%—200 "	58%—200 "	62%—200 "	1%
90%—100 "	90%—100 "	92%—100 "	0.5%
85%—200 "	83%—200 "	87%—200 "	0.5%

Table 9. Blending Schedule

Grade (BPL)	Concentrate Mix
64	Pebble
66	Pebble
68	3 pebble, 1 concentrates
70	1 pebble, 1 spiral
72	95 concentrates, 5 pebble
75	3 float, 1 spiral
77	9 float, 1 spiral

Table 11. Variation in Chemical Analyses

Base Grade	% BPL	% Insol	% I and A	% CaO	% CO_2	% F	% shipped H_2O
72 BPL							
Average	72.73	4.01	1.87	48.45	3.73	3.74	—
$\mp \delta$	0.48	0.99	0.23	0.62	0.23	0.14	—
$\mp L_{95}$	0.20	0.42	0.10	0.26	0.10	0.06	—

Table 12. Variation in Chemical Analyses

Base Grade	BPL	Insol	I and A	CaO	CO_2	F	% shipped H_2O
75 BPL							
Average	75.14	3.09	2.20	48.71	3.19	3.65	1.05
$\mp \delta$	0.14	0.34	0.15	1.79	0.27	0.22	0.04
$\mp L_{95}$	0.07	0.16	0.07	0.84	0.13	0.10	0.02

Table 13. Variation in Chemical Analyses

Base Grade	BPL	Insol	I and A	CaO	CO_2	F	% shipped H_2O
77 BPL							
Average	76.99	2.79	1.90	49.70	2.95	3.73	1.08
$\mp \delta$	0.20	0.17	0.09	0.38	0.10	0.16	0.18
$\mp L_{95}$	0.10	0.08	0.05	0.19	0.05	0.08	0.09

frequently shipped grades, that is 68, 72, 75 and 77 BPL materials. Standard deviation and confidence limits were determined on each group of products. Of course we make the basic assumption that the variations in analyses follow a normal distribution. The standard deviation calculation gives us the range in which the majority of the analyses will fall, with 68.27% of the group of samples falling within the range of plus or minus one standard deviation from the mean. Similarly, 95% of the samples will fall within plus or minus 1.906 times the standard deviation from the mean.

For any particular process, and in this case we are considering a combination production-blending process, the standard deviation is a relatively constant perimeter. The problem in sampling is often one of determining a precise mean or average for a population which is quite variable. A reasonably good estimate of standard deviation permits us to estimate the population without increasing the number of observations.

A more useful reference point is the confidence limit, which is a mathematical computation based on the standard deviation. For example, here are the results of the statistical evaluation of the samples covering 68 BPL grade (Table 10.) While the mean of shipments was 68.33, we can be 95% sure that the average shipment would range between 68.17 and 68.49% BPL.

Table 10. Variation in Chemical Analyses

Base Grade	% BPL	% Insol	% I and A	% CaO	% CO_2	% F	% shipped H_2O
68 BPL							
Average	68.33	8.45	2.58	45.84	3.63	3.42	1.22
$\mp \delta$	0.37	0.54	0.20	0.61	0.12	0.15	0.22
$\mp L_{95}$	0.16	0.23	0.08	0.26	0.05	0.06	0.09

Similarly, in the case of 72 BPL material, (Table 11) the odds are 19 to 1 in our favor if we predict that the average BPL fell between 72.53 and 72.93, etc. Here are the data summaries for 75 rock (Table 12) and 77 rock (Table 13).

Time limitations do not permit a more complete discussion of the individual analyses involved on all samples, for I know you would be interested in the insol, I and A, fluorine and water values as well as phosphate analyses. The insol and I and A relationships to BPL are summarized graphically however. Below about 70.5 BPL, equivalent to 5.9% insol, changes in grades are attributable almost altogether to changes in insol. Above this figure, the importance of insolubles decreases rapidly, until no further improvement in insolubles can be expected past 76.5% BPL, or 2.8% insol. Removal of iron and alumina is steady over the entire range of products. It amounts to 0.067% decrease in A and I for each percent increase in BPL.

Florida Check Samples Data

For some time, since 1932, in fact, the control chemists in the phosphate companies in Florida have been doing something to check the accuracy of their chemical analyses. A program of check samples was instituted in which phosphate producers, fertilizer companies and private laboratories collaborated. A recent count showed 36 chemists participating in the program, representing 17 organizations.

Our analysis of 13 months' samples, and the data are presented here for your consideration (Table 14). These standard deviations represent the total number of samples. It is statistically valid to throw out any values which are more than three times the standard deviation from the mean. As you might expect in any large group of samples, some of the analyses fell wide of the mark. Out of 2,054 samples, 28 could be considered in-

Table 14. Florida Check Samples

Determination	No. Samples	Mean	Standard Deviation	Invalid[1] Results
H_2O	399	1.21	0.13	5
P_2O_5	399	33.49	0.17	5
BPL	399	73.18	0.38	5
Fe_2O_3	313	1.26	0.13	4
Al_2O_3	287	1.06	0.18	3
I and A	287	2.19	0.21	5
Sand	347	6.56	0.21	5
CaO	312	48.11	0.45	5

[1] Results are considered invalid if value is > 3 *6* from the mean. On a statistical basis, the anticipated number of invalid results in this study was 0.5; a total of 5 was found in most cases. Note also the large standard deviation, (on the order of 10%) for H_2O, Fe_2O_3, Al_2O_3, I and A, and CaO determinations.

Table 18. Screen Analyses for 50%-55% + 10 Mesh Pebble Product 31.13% P_2O_5 Sample

Size	% Weight Retained	% BPL
½ inch	1.2	48.54
7/16 "	1.8	59.78
⅜ "	3.3	62.14
3 mesh	7.4	66.65
5 "	11.9	67.34
6 "	4.2	67.34
8 "	9.5	67.49
10 "	11.1	68.62
14 "	11.1	68.89
16 "	3.4	67.93
20 "	2.2	67.18
–20 "	32.9	70.00
Head	100.0	68.02
% + 10 Mesh	50.4%	———

valid. The point is that removing these worthless analyses would considerably reduce the standard deviation values. Nevertheless, these data suggest that perhaps we are doing a better job of mining, refining and shipping standardized products than our chemical quality control laboratories are able to describe within the methods and equipment now available.

The statistical analysis also included data covering grinding efficiency. Let us examine these data. Beginning with the screen pebble grades (Table 15), we note the the market calls for four grades — 23 +3, 90 + 6, 50-55 + 10, and 70-80 + 20 mesh, (Table 16). The four typical samples covered in this slide of the 23 + 3 grade averaged slightly below grade, (Table 17). The 90% + 6 mesh averaged above grade with no samples falling below 90%. Typical (Table 18) screen analysis data for 50-55% + 10 mesh pebble product average 50.4% + 10 mesh. On the finer grades there are five major grinding levels (Table 19). Typical data for the 50% –200 mesh product (Table 20) show an average of 51.7% –200 mesh with no samples below specification. Similar results are noted for the 60% product (Table 21) with 61.8% as the average –200 mesh value. Again none of the samples fell below specification. With respect to the 70% product, (Table 22) a similar relationship holds.

Table 15. Screened Pebble Grades

23%	+	3 mesh
90%	+	6 mesh
50-55%	+	10 mesh
70-80%	+	20 mesh

Table 19. Ground Rock Grades

50%	—	200 Mesh
60%	—	200 "
70%	—	200 "
85%	—	200 "
90%	—	100 "

Table 16. Screen Analyses for 23% + 3 Mesh Pebble Product

Size	Sample 1	Sample 2	Sample 3	Sample 4	Average
		% Weight Retained			
½ inch	1.5	3.3	3.8	2.3	2.7
7/16 "	1.5	2.9	2.7	2.6	2.4
⅜ "	4.1	4.2	5.2	4.4	4.5
3 mesh	14.0	10.4	12.3	15.3	13.0
5 "	21.6	17.7	17.4	26.1	20.7
6 "	5.2	5.0	4.8	6.4	5.4
8 "	9.7	9.8	10.2	11.7	10.3
10 "	11.2	13.1	10.8	12.8	12.0
14 "	13.4	13.6	11.4	9.3	11.9
16 "	4.1	3.8	4.1	2.5	3.6
20 "	4.0	2.7	2.7	1.3	2.7
–20 "	9.7	13.5	14.6	5.3	10.8
Head	100.0	100.0	100.0	100.0	100.0
% + 3 mesh	21.1	20.8	24.0	24.6	22.6

Table 17. Screen Analyses for 90% + 6 Mesh Pebble Product

		% Weight Retained			
½ inch	5.4	3.0	1.3	4.6	3.6
7/16 "	4.6	5.5	3.9	6.0	5.0
⅜ "	7.2	10.9	7.3	7.6	8.2
3 mesh	29.7	30.0	25.4	25.3	27.6
5 "	39.2	38.0	47.3	42.4	41.7
6 "	6.9	5.5	6.4	7.2	6.5
8 "	4.1	3.8	4.8	3.8	4.1
10 "	1.0	1.0	1.4	0.9	1.1
14 "	0.5	0.6	0.5	0.4	0.5
16 "	0.1	0.2	0.2	0.2	0.2
20 "	0.1	0.2	0.1	0.1	0.1
–20 "	1.2	1.3	1.4	1.5	1.4
Head	100.0	100.0	100.0	100.0	100.0
% + 6 mesh	93.0	92.9	91.6	93.1	92.6

Table 20. Screen Analyses for 50% —200 Mesh Grind

Size	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Average
			% Weight				
+ 35 Mesh	3.2	6.2	2.7	3.5	4.8	3.2	3.9
— 60 "	88.2	81.8	89.5	86.5	84.8	87.9	86.5
—100 "	75.4	69.6	76.2	72.9	72.7	74.5	73.6
—200 "	53.6	51.1	52.0	50.2	51.7	51.5	51.7

Table 21. Screen Analyses for 60% —200 Mesh Grind

			% Weight				
+ 35 Mesh	1.8	2.0	1.3	1.4	1.4	1.5	1.6
— 60 "	93.1	91.4	83.5	92.6	92.6	92.7	92.7
—100 "	82.4	81.3	84.0	83.1	82.0	82.3	82.5
—200 "	62.1	60.9	63.0	62.6	60.8	61.5	61.8

Table 22. Screen Analyses for 70% —200 Mesh Grind

			% Weight				
+ 35 Mesh	0.2	0.5	0.0	0.5	0.1	0.1	0.2
— 60 "	97.7	96.6	98.7	97.4	98.8	99.2	98.1
—100 "	91.1	89.6	92.5	90.0	91.7	94.4	91.6
—200 "	72.5	71.2	72.1	70.8	72.0	71.9	71.8

Table 23. Screen Analyses for 90% —100 Mesh Grind

			% Weight				
+ 35 Mesh	0.1	0.0	0.0	0.1	0.1	0.1	0.1
— 60 "	97.9	98.9	99.1	98.7	98.4	98.1	98.5
—100 "	91.1	91.0	91.2	92.0	90.6	91.6	91.3
—200 "	71.6	69.4	70.1	78.7	75.2	76.0	73.5

Table 24. Actual Shipments vs Specifications

	68 BPL	72 BPL	75 BPL	77 BPL
Car Min.	67.00	71.00	74.00	76.00
Desired	68.25	72.25	75.25	77.25
Actual (Ave.)	68.33	72.73	75.14	76.99

Note: All shipments in sample met I and A, moisture and grind specifications

In our laboratories, the 85% —200 mesh product is not generally subjected to a complete screen analysis. For this study, however, ten samples were analyzed, with the average percent —200 mesh determined at 86.1%. Representative samples of the 90% —100 mesh grind product (Table 23) show an average value of 91.3%, again with none of the samples below specification.

Perhaps these data (Table 24) will summarize our experiences in quality control. The data cover approximately 20 monthly averages in each product grade. Actual shipment averages were well above minimum specifications in BPL values, and were well within I and A and moisture specifications. Grind specifications were met in all instances.

Summary

An attempt has been made here to present the story of phosphate rock from the point where it was laid down in nature to the point of embarkation from the Florida phosphate plants. I have used our own company as an example because I am most familiar with it. Gentlemen, the phosphate producers fully appreciate how important it is to their customers that the products we furnish be high in quality and consistently so. Our Noralyn analytical laboratory alone represents a $200,000 capital investment. The staff includes 17 chemists and technicians, which means that substantial operating costs are connected with this customer service. Over 40 chemical analyses are run on rock from the time it reaches the plant until it is loaded into railroad equipment. It has been so many years since samples of our products went to a referee chemist that I could find nothing in the record on this point. We have to take the phosphate where we find it. A tremendous materials handling task is involved in mining and beneficiating a low cost product. The industry undoubtedly could improve product quality if the markets were willing to pay the added cost. After all these facts are considered, I earnestly hope that the data we have reviewed here this morning will leave you convinced that a commendable job is being done to serve the agricultural and industrial markets.

MODERATOR REYNOLDS: Are there some questions, comments or any ideas we might pursue a little further?

DR. ENGIBOUS: I might add here, Joe, that the IMC people were threatened with severe penalties not to ask questions, but I didn't think they would pass the word around to everybody here.

MODERATOR REYNOLDS: Phosphate rock as you have heard here, many different grades, many different types, forms, as far as movement of material, it is quite a problem. Phosphate rock is processed in many ways. Perhaps the oldest and most widely known process is in the manufacture of normal superphosphate.

Through the years the chemistry of phosphate rock acidulation has not changed, although production methods and techniques have changed. Continuous superphosphate processing was introduced to the fertilizer industry several years ago and in the form of several different types of systems.

Our next speaker, Mr. Henry Wheless of Davison Chemical Division of W. R. Grace will discuss one of these systems for continuous superphosphate production.

Continuous Superphosphate Production

Henry Wheless

THE manufacture of superphosphate at Davison's Curtis Bay works in Baltimore began in the second decade of this century before our entry into the first World War. More than forty years later, in the spring of last year, the original batch process was finally abandoned in favor of a modern continuous process.

In the original process, measured batches of finely ground phosphate rock and sulfuric acid were introduced into a pan mixer which mixed these ingredients and discharged them into a 350-ton capacity pit or box den situated beneath the mixer and covered by the mixer platform. In charging a den this operation was repeated some 175 times by each of two such mixers on a mixer platform. Most of the reaction took place in the den and the slurry was transformed into a solid mass. Two such box dens were serviced by each of the three mixing platforms and after one was filled the platform was shifted on rails to the adjacent den. The superphosphate in the completed den was then removed by a clamshell bucket and transferred by bridge crane to a rasper or disintegrator at the other end of the building. This rasper broke up large lumps of superphosphate and discharged the product into the storage bays.

In this operation the ground rock was transferred from the suspended storage bunkers to the mixing platforms by a series of screw conveyors and a scale indicated to the mixer operator the weight of each batch he drew into the batch weigh hopper. Sulfuric acid flowed to the platform by gravity from a constant head tank and was measured for the batch by volume. If acid density or temperature changed, the volume still remained constant but the rock batch weight was changed accordingly. Fluorine and fluoride gases were transferred to the main gas flue by wooden ducts that passed through the mixer platforms.

Obviously, such a process had many shortcomings and disadvantages both with respect to operating costs and product quality and uniformity. Each completed den contained stratified layers of over-and -under-acidulated material even though the ratio of rock to acid for the entire den may have been proper. A reasonable degree of product uniformity was afforded by the considerable turning and rehandling of the stored product by the bridge cranes.

Faced with the higher and higher costs of operating and maintaining the six mixers on the three mixer platforms, three raspers or disintegrators, and the heavily scheduled bridge cranes, Davison elected to install a modern efficient continuous manufacturing process.

The first construction work was begun early in January, 1959, by the A. J. Sackett and Sons Company at Baltimore and our new Super-Flo process produced its first superphosphate about three months later on the ninth of April.

As I describe our process, I believe your understanding of it will be simplified if you will observe an artist's conception of it in a series of slides. While these slides are highly accurate with respect to function, the artist has purposely omitted a maze of pipes, conduits, intermediate decks, stairs and hand rails which only confuse the issue.

Davison's Super-Flo process was designed to tie into the existing ground rock storage bunkers acid supply, and fume removal system. Screw conveyors which formerly supplied ground rock to any of six locations for the batch process were modified to deliver the ground rock to a single downspout. The overall length of these suspended bunkers which span three huge crane bays is some 225 feet. The downspout discharges the ground rock into one of the old superphosphate dens which was incorporated into the ground rock handling system to provide a surge and overflow pit and to increase storage from some 480 tons to about 800 tons, thus permitting greater flexibility between rock grinding and acidulation schedules. An 80-ton per hour capacity elevator in a steel elevator shaft in one corner of the old den picks up the ground rock and discharges it into a constant head rock hopper, the overflow from which is returned to the surge pit.

By elevating a surplus of rock and returning the excess to the surge pit the scale feed hopper maintains a constant head of fully aerated dust, thus eliminating one of the variables which tend to impair accuracy or precision in a continuous ground rock weighing system. A variable speed screw feeder supplies the rock to a weigh feeder belt, the rate on which is controlled and indicated in the control room, Should the rock feed rate increase or decrease, the feed screw is automatically decreased or increased accordingly so that the rock feed to the cone is constant for any given setting. When the process is operating at its design rate of 90 tons per hour the rock feeding system delivers ground rock to the acidulation cone at a rate of some 55 tons or about one railroad carload per hour. For limited periods, the process has been operated smoothly at almost 100 tons of superphosphate per hour.

The belt feeder discharges the rock dust into a funnel which in turn discharges into a TVA-type mixing cone.

Sulfuric acid at a concentration of some 57° to 58° Baume and at a temperature of approximately 90° F controlled by indirect heat exchangers is supplied to an indicating-controlling magnetic flowmeter at some 25 to 30 pounds pressure and precisely metered to the mixing cone through a four-nozzled ring manifold. The mixing cone is also equipped with water nozzles for further dilution of the acid if necessary. At its design production rate of 90 tons of superphosphate per hour the process consumes about 33 tons of acid per hour basis 100% H_2SO_4 or over 100 gallons per minute at 90° F—58° Baume.

The ground phosphate rock is mixed in the swirl of sulfuric acid in the cone and discharged into a single shaft pugmill or puddler. Although considerable mixing of rock and acid occurs in the cone it is believed that more intimate contact and ultimately better conversions are achieved by the additional puddling or churning of the slurry during its liquid or semiliquid phase. The puddler finally discharges the mix into the solidifier. The solidifier consists essentially of a steel slat conveyor which moves very slowly on its supporting rails and conveys the solidifying superphosphate toward the helical cutter or excavator. Being turned up on each end these slats actually form a pan which is six feet wide and three feet deep and which confines the fresh super and prevents a sliding contact between it and any stationary part of the equipment. A dam at the feed end prevents the still fluid mass from pouring out the rear of the solidifier. The center to center distance between the drive and tail sprockets is approximately 70 feet and with a retention time of about thirty minutes the load on the solidifier when it is operating at its design capacity is about 45 tons. The superphosphate is transformed into a fairly-dry and porous solid before it is conveyed into the cutter or excavator. This cutter has six helical cutter bars so shaped that the drive motor load is virtually constant. Each bar has eleven stainless steel teeth that shave the solid superphosphate from the cake and discharge it onto a belt conveyor which ascends across the in-plant tracks to the storage bays. This belt discharges onto a reversible belt at right angles to it which discharges the product into either of two bays. Further handling is performed by the bridge cranes and after a reasonable cure in the pile the superphosphate is transferred by these same cranes to the milling, screening and shipping center.

The process is connected to the main gas flue by two fume ducts, one 24″ and one 30″ in diameter. The larger of these is tied into the puddler and the other is tied into the hood over the cutter. The entire system from the puddler to the cutter is hooded and under a negative pressure so that all of the evolved gases are removed to the fluoride recovery plant. All of the fume ducts, the puddler, the entire solidifier cover, and the cutter hood are rubber lined for protection against corrosion.

Under the old batch process, most of the gases liberated during mixing and during the filling of a den were removed to the fluoride recovery plant, but during the reaction in the deep den, some gases were generated which were not liberated from the mass until after the platform had been removed and the clamshell began its excavation. These gases and those liberated during rasping or disintegration were lost.

In the new process the helical cutter replaces the den digging and the rasping operations and since it is incorporated in the fume removal system, fluoride recovery as well as working conditions have been significantly improved.

As for raw materials, the process uses rock analyzing about 74.5% to 75% BPL which is received by vessel from Davison's Florida mines and which is ground to some 75% to 80% minus 200 mesh. Sulfuric acid is a blend of selected spent acids and some virgin acid. For each part of ground phosphate rock, acid consumption is about 0.6 parts by weight basis 100% H_2SO_4.

The moisture of the superphosphate as it is discharged to storage is about 10% and after a 3 to 4 week cure it drops to approximately 6%. Conversion is in the range of 98.5% with a typical analysis being 20.2% APA and .25 to .3% citrate insoluble. Higher conversions can be attained but at the expense of increased moisture, increased acid consumption and some impairment to product physical condition.

Some eighteen months of experience have shown us that most of the savings and other benefits we had hoped for in our modernization have actually been achieved.

Moderator Reynolds: Do we have some questions?

A Member: What is the free acidity of your product?

Mr. Wheless: The free acidity of the product after it is cured at the time of shipment runs something in the range of three and a half to four per cent H_3PO_4 by the acetone method.

A Member: Is it necessary to still use a hot pot in your system with the cone for mixing? Some batch systems use the TVA cone; however, they do not have any other means of mixing; is it necessary to have the puddler go along with the cone?

Mr. Wheless: As I said in the paper we believe it provides additional intimate contact between rock and acid. Some processes, continuous and batch, use only a TVA cone. This is the usual thing; as a matter of fact I know of no other way of doing that in the TVA cone process as it applies to triple superphosphate, but in normal superphosphate where we have a fluid phase for a little bit longer time we do not think it does any harm to mix this a little more thoroughly.

A Member: What is the hold up time in the pug mill?

Mr. Wheless: The hold up time in the pug mill I would say not over a minute and a half or so.

A Member: Could you give us again the efficiency of conversion of rock to available P_2O_5?

Mr. Wheless: About ninety-eight and a half per cent is what we shoot for. Now, of course, we have run higher than that. We have been above 99 per cent with citrate insoluble in the final product analyzing as low as .11. This is not the goal that we are striving for here. That is, in my opinion, over-acidulation. This gives a little higher moisture.

As a matter of fact, you make more tons that way, but the moisture of the final product being slightly higher tends to dilute it back and lower the analysis to a point that the finished APA of the material is not higher significantly than it would be if you had a little lower moisture and a little higher citrate insoluble.

A Member: What is the approximate maximum temperature of the acidulation?

Mr. Wheless: Harry Velker can correct me here if I'm wrong, but I think in the den at about the point of solid phase it is up to about 250 to 260 degrees Fahrenheit.

Mr. Walter Horn (MFD): Will you please describe that rock feeder again after the constant level tank?

Mr. Wheless: The rock feeder comes off the bottom of the constant level tank with a screw conveyor. This discharges to a scale, a belt scale, and the load on this belt scale if it exceeds the set load actually slows down the screw conveyor feeding the belt. If the load on the scale should be light then the automatic mechanism speeds up the screw conveyor which is supplying the rock to the belt.

MR. W. J. TUCKER (G.L.F. Soil Building Service) : Mr. Wheless, in the first paper the standard deviation of confidence limits was applied to the superphosphate being the factor; the phosphate rock people given the handle therefor to work with.

I was wondering if superphosphate producers have established what the standard deviation is and what the confidence limits might be on individual shipments of superphosphate to the manufacturers.

MR. WHELESS: I have no statistics with respect to the standard deviation on our superphosphate with me here. We maintain an analysis that is above 20 per cent available P_2O_5. As far as deviation goes it can not deviate below that because we assure ourselves that it will be that high. It is not going too much higher than about a 20.4 ever because it is just not in the rock.

A MEMBER: How long a curing period does it take to get that 20 per cent flux?

MR. WHELESS: Approximately three weeks. Three to four weeks.

A MEMBER: Mr. Wheless, can you tell us why you use a mixture of virgin and reclaimed acid?

MR. WHELESS: We are also in the sulfuric acid business and sulfuric acid producers and marketers must in order to sell virgin sulfuric acid receive back from the customer spent sulfuric acids. We will not use spent sulfuric acid in our product which will impair its quality.

A MEMBER: Can you give us the proportion?

MR. WHELESS: It varies depending upon the rate that the spent sulfurics are coming back, but some spent sulfuric acids we cannot use at all.

A MEMBER: What are the materials and construction of your pug mill, of your corrosion pug mill?

MR. WHELESS: We have not had a corrosion pug mill and I am not sure there is steel in that pug mill on those paddle blades—can you help me on that, Walter?

MR. WALTER SACKETT: It's a chrome alloy steel.

MR. WHELESS: Mr. Sackett says it is a chrome alloy steel. I do not know exactly what the specification is.

A MEMBER: Mr. Wheless, would the equipment in your bin lend itself to the production of fortified super say of 23 per cent?

MR. WHELESS: Yes, sir, I think it would. You know we did some work on that not long ago, just looking into it, thinking that one would assume offhand that if using phosphoric acid for acidulation the set time is reduced in comparison to using sulfuric acid for normal superphosphate that a blend of the acids might fall somewhere in between. But this is not necessarily the case. Making, for example, a 28 to 30 per cent P_2O_5 product is kind of tricky, to say the least, and conditions have to be pretty favorable, we believe, or you will not get a set time in the retention time in this unit.

This unit does have in the solidifier driver, a controllable speed, which will permit it to be speeded up or slowed down. This would affect, of course, the retention time.

This same unit has been, and this one can be used in the manfacture of triple superphosphate, in which case the doors on the bottom of the pug mill open up and the freshly acidulated mass falls through the cone right on past the pug mill shaft and directly into the solidifier.

This is a bypass on the pug mill.

MODERATOR REYNOLDS: We have time for two more questions.

Mr. Tayloe.

MR. RICHARD TAYLOE (Smith Douglas) There was a question over here somewhere about using the fortified super. We are currently using one of these units; they pilot 20 tons a hour on 27 per cent superphosphate making our own phosphoric acid, sulfuric acid and phosphate rock which works pretty well.

MR. WHELESS: Yes.

MR. JOE SHARPE (Spencer Chemical Company) : How does the density of your product made this way compare with the old bin process?

MR. WHELESS: I don't think it is significantly different. It is about the same.

MODERATOR REYNOLDS: Thank you very much, Henry.

The discussions are good and I hate to halt this temporarily here, but we must move along.

Phosphoric acid provides the key for increased plant food concentration. Production of phosphoric acid has increased manyfold during the past few years with the main outlet going initially into triple superphosphate.

Recently the quantity of phosphoric acid directed towards ammonium phosphate production and towards mixed fertilizer granulation plants is substantial.

We are beginning to realize that we have possibly oversimplified the role triple superphosphate plays in the chemistry of mixed fertilizers.

Recent investigations reveal complicaed differences between types of phosphoric acid. The behavior of these variables contained in phosphoric acid ultimately influences the mixed fertilizer product and its behavior in triple superphosphate and/or wet process phosphoric acid.

Our next speaker is closely associated with the production of phosphoric acid and is well qualified to provide an insight into the processing techniques.

Mr. Bill Weber of the Dorr-Oliver Company will discuss Phosphoric Acid—Types, Sources and Characteristics.

Phosphoric Acid

Wm. C. Weber

Introduction

PHOSPHORIC acid production in the United States now exceeds 2.6 million tons of P_2O_5 annually. Plant expansions now under way or planned by present producers along with new entries into the field promise to significantly increase the supply of phosphoric acid within the next year or so.

Some of the factors responsible for the increased interest in phosphoric acid are:

1. Improved plant design and better materials of construction have reduced cost of production.
2. Continuing trend to higher analysis fertilizers.
3. Development of liquid fertilizers.
4. Fertilizer compounds made from phosphoric acid have good storage and handling characteristics, and are compatible with most other complete fertilizer ingredients.

In the light of the increasing importance of phosphoric acid to the fertilizer industry, this subject seems appropriate for this meeting. My discussion will deal mainly with the types and characteristics of phosphoric acid available to the industry.

Types

As you are no doubt aware, there are basically two types of phosphoric acid — electric furnace acid and wet process acid.

Electric furnace acid is obtained by reduction of phosphate rock with carbon in an electric furnace to produce free phosphorus which distills from the furnace and is condensed in water. The phosphorus is subsequently burned in an excess of air to produce P_2O_5, which is in turn dissolved in water to give phosphoric acid. Furnace acid is more costly to produce than wet process acid and consequently, it has not been as widely used for fertilizer purposes as wet process acid. Most of the furnace acid production goes into the manufacture of polyphosphates and food grade phosphates.

Wet process acid is produced by reacting sulfuric acid and phosphate rock to produce phosphoric acid and gypsum. The gypsum is separated from the phosphoric acid by filtration. The resulting phosphoric acid normally will be approximately 32% P_2O_5, and must be evaporated if a stronger acid is required. Wet process phosphoric acid is used almost exclusively in fertilizer manufacture.

Characteristics

By nature of the production process, furnace acid is invariably pure, water white, regardless of the type phosphate rock used to produce it. On the other hand, wet process phosphoric acid may vary in composition significantly, depending on the grade and type of phosphate rock used in its manufacture and also depending on the manufacturing process.

A typical minima - maxima analysis of concentrated wet process acid made from commonly used phosphate rocks may be as follows:

% P_2O_5	52-55%
Combined I & A	2.0-5.5
Undissolved Solids	0.1-2.0
Fluorine	0.8-1.8
H_2SO_4	2.8-4.7
Specific Gravity @ 60°C.	1.58-1.72
Viscosity @ 25°C. C.P.	25-80
Total Impurities (Anhydrous H_3PO_4 Basis)	12-20%

The impurities in wet process acid are not necessarily objectionable but their presence and their effect on the end product should be recognized.

The following discussion will illustrate some of the influences of wet process acid impurities:

1. *Iron and Aluminum* — About 80 to 90% of the iron and aluminum in the phosphate rock is dissolved in the phosphoric acid. Hence, the higher the I & A in the phosphate rock used to produce the acid, the higher the I & A will be in the acid. I & A will inactivate approximately an equal amount of P_2O_5, thus reducing the total acidity of the acid. In addition, the I & A will, of course, lower the strength of the acid in the same manner as any inert filler. The amount of I & A normally present in wet acid is not objectionable; however, excessive amounts may affect grade, physical condition, and conversion when used to make triple superphosphate. Water soluble P_2O_5, ammoniation rate and product grade will be lowered if the acid is used to make ammonium phosphates.

2. *H_2O* — The water content of wet process acid can vary significantly for two acids containing the same percentage P_2O_5. The P_2O_5 content of a phosphoric acid depends on the total water plus total impurities, and therefore if the impurities in the acid change, a corresponding change in water content is necessary to maintain the same P_2O_5 percentage.

3. *H_2SO_4* — Free H_2SO_4 increases the acidity of the phosphoric acid and will affect grade of product, ammoniation rates and acidulation rates when making ammonium phosphates and triple superphosphate respectively. Similar effects will also result in the case of fluorine impurities.

4. *Insoluble Solids*—Insoluble solids in phosphoric acid can be particularly objectionable because of attendant problems from sludge formation during storage and handling. Proper design of storage vessels will be discussed in detail later.

Superphosphoric Acid

Superphosphoric acid, developed by the Tennessee Valley Authority, has roused considerable interest, and commercial plants are now under construction to produce this acid from both wet process acid and furnace acid. The furnace acid product contains about 76% P_2O_5, and the wet process product about 68% P_2O_5. These materials, contain a significant percentage of their P_2O_5 in the form of condensed phosphates. The condensed phosphates have the property of complexing calcium, iron, aluminum, and other heavy metals. Ammonium phosphates made from superphosphoric acid are considerably more soluble than their corresponding ortho phosphates, which permits making more concentrated liquid fertilizers.

The special properties of superphosphoric acid make this acid especially attractive to liquid producers because of the wider market area possible from a more concentrated product. Moreover, cheaper wet process acid can be used since the impurities remain in solution when superphosphoric acid is ammoniated under proper conditions.

This thumb-nail sketch of the types and characteristics of phosphoric acid is by no means complete, but may perhaps provide a basis for later, more detailed discussions.

MODERATOR REYNOLDS: We have time for some questioning.

A MEMBERS How does the viscosity of superphosphoric acid wet process compare with the viscosity of conventional wet processing?

Mr. Weber: I will ask my associate Mr. Lutz, if he knows the answer to that. Do you, Bill?

Mr. W. Lutz: No, I don't really.

Mr. Weber: There are other producers of phosphoric acid here. Are there any comments; does any one have information on this question?

Mr. A. B. Phillips: I don't have any actual viscosity figures in mind, but we have done some pilot plant work on producing wet process super acid and it is considerably more viscous than the electric furnace super acid because I presume the impurities that are present in the wet process acid that are not in the electric furnace acid.

When you get to above 70 per cent P_2O_5 with a wet process acid it is very viscous; about like molasses.

A Member: Could you give us some idea of the range of water content and sulfuric acid content and fluorine content that you might find in the run-of-the-mill wet process acid?

Mr. Weber: I was speaking of that before, but I will be glad to do so again.

The water content—I had that in here—I thought we did—do you know that figure, Bill?

Mr. Lutz: It was 15 to 20 per cent.

Mr. Weber: I think it is in here someplace. The sulfuric acid of course depends on the manufacturing process. But it will run somewhere between, in the concentrated acid, from 2.8 to 4.5 per cent.

The fluorine, again it depends a little bit on the rock used, and the manufacturing process for the phosphoric acid, whether you are recovering fluorine, will run from about .8 to 1.8 per cent in the 54 per cent acid.

A Member: That figure of 15 to 20 per cent, is that based on approximately 52 per cent P_2O_5?

Mr. Weber: Yes.

Question?

Mr. Bourne: What per cent of solids in a 54 per cent wet processed acid could be used, in making triple, without affecting the curing time and so on?

Mr. Weber: The solids in the acids do not significantly affect the curing time as far as I know.

I don't think that the insoluble or inert solids in the phosphoric acid from our experience materially affect the curing time or the triple superphosphate. They only act as a diluent and reduce the grade.

They are inert in the reaction.

A Member: One more question. Do you have any idea about the chemical analysis or composition of the insolubles, insoluble impurities in the wet process acid concentration?

Mr. Weber: It varies. It varies a little bit with the handling of the acid at the manufacturing plant, the degree to which it has been clarified and to some extent the manufacturing process. It is—it will consist most substantially of gypsum and fluosilicates and some iron and aluminum phosphates.

Moderator Reynolds: I believe there has been some work performed on this by the American Cyanamid Company. I think that that was reported at the ACS meeting; would Dan Walstad care to comment on that question?

Mr. D. O. Walstad (American Cyanamid Company): I do not have the data with me, but it is in this paper; it shows you that the solids which remain in the 54 per cent acid are primarily iron and aluminum fluosilicates; that the solids which drop out duringing the evaporation period are primarily gypsum and the composition of the solids are naturally quite complex.

We have not been able to identify all these different complexes that you have in the solids which remain either suspended or post precipitate after shipment.

Mr. Weber: Yes, I mentioned that the composition of the solids is affected again by the handling of the manufacturing process for making phosphoric acid because there does seem to be a significant difference in the composition when the phosphoric acid has been evaporated by vacuum evaporators or direct heat evaporators.

Mr. Walter Horn: Mr. Weber, is there solubility data available on iron and aluminum phosphates in 32 per cent acid?

Mr. Weber: Yes, I think we have such data.

I think we can supply you some data of that type if you will write in to us.

A Member: I understand that the new solvent process has been used in Israel and Japan, which is a wet process, is a very much clean acid altogether. Have you any figures available?

Mr. Weber: I don't have any figures available. I know about that project, but the way we understand it, it does make a very much purer acid.

A Member: What is this IMI process?

A Voice: We didn't hear the question.

A Member: What is this IMI process?

Mr. Weber: That is a process developed in Israel by the Israel Development Company, I think it is.

I don't think it has been more than pilot planted so far.

Mr. E. N. Mortenson (Swift and Company): What is the name of it?

Mr. Weber: I don't know that it has any name.

A Member: IMI process.

Mr. Weber: Israel—it is titled by the name of the company.

A Member: For those who are interested in that it was written up in 1959 by work done at the University of London.

Moderator Reynolds: One more question.

Mr. George Reid (Western Phosphates): Joe, just a comment on the wet process superphosphoric. It is our opinion that the viscosity will be great enought so that it will have to be handled always in a positive displacement pump rather than in centrifugals.

Moderator Reynolds: Thank you.

Moderator Reynolds: We are moving right along here from phosphates, superphosphates, phosphoric acid production; now we come to the usage of phosphoric acid in mixed fertilizers.

One of the most discussed items in the fertilizer industry today is the use of phosphoric acid. The availability of economically priced, wet process phosphoric acid has permitted the introduction of a new raw material which possesses both anhydrous ammonia neutralizing values and a P_2O_5 plant food value.

Based primarily on experience and existing equipment the choice between phosphoric acid and triple superphosphate is largely governed by the economic balance between sulfuric acid, normal and triple superphosphates.

Instruments are now available to meter thick phosphoric acid and process techniques can be varied to control granule formation and the degree of wetness. Mechanics of formulation must be adjusted to compensate for the realistic moisture in the phosphoric acid.

Our panel for this discussion has been assembled from the phosphoric acid users. We are pleased to introduce the participants of this panel; Mel Leach of Indiana Farm Bureau, Rodger Smith of Eastern States Farmers Exchange; we have had a cancellation of Phil Stone of Virginia-Carolina due to illness. We were advised that he was unable to make this meeting. So we will turn the panel over to either Rodger or Melvin, which one would care to come first. I see Rodger has retreated to the back of the room.

Melvin, would you care to kick this off?

MR. MELVIN LEACH (Indiana Farm Bureau): I would like to make a few remarks here in regard to our use of phosphoric acid and I, like Mr. Weber, am going to stick just to what I have written here because if I start off and get into all the details of our use of phosphoric acid I could keep you here well after lunch.

We are not necessarily old-timers in the use of phosphoric acid in the granulation of fertilizer; however, we do feel that way.

We started in 1955 with an experimental car of phosphoric—furnace grade phosphoric acid and have had trouble using the stuff ever since.

I just want to go into a few of our difficulties. And, of course, some of the good points. I think it's—let me see how many experts we have in here—I think possibly we are talking to a group of experts. A show of hands of those that are using phos acids in fertilizer and mixed goods productions.

Joe, we've got a bunch of experts.

It is good to get off—I mean, it is nice to get off to a good start, so if some of you fellows are thinking of getting into it, if we make some comments here that will help you, well I think our time is well spent.

In 1955 when we first got into this business we purchased a ten foot by eighteen rubber lined phosphoric acid tank. It was delivered by truck and unloaded by a crane from the truck to our concrete foundation.

We ordered a carload of phosphoric—this is furnace grade, now —and proceeded to run several tests using phosphoric acid with various nitrogen solutions and on various grades. These tests, some good, some bad, indicated that this is what we needed. This was the thing.

One half of the car or acid was still on hand and we saved it for some test runs of grades using the combination of phosphoric acid and anhydrous ammonia. Our anhydrous ammonia car came in about a week and on Monday morning when we went to work we found our phosphoric acid had somehow found its way through the sides of the tank—acid was everywhere and still coming.

It had flooded the general area around the tank, buried our fuel oil storage tank, ran over the scrubber area and so forth.

Now this is a handling problem.

Joe, I don't know whether this is the one you referred to on your agenda, but it is a handling problem.

You take the fire hose and begin to scrub; we scrubbed for half a day; we finally got what we thought was most of it away, so we dug down to our fuel oil tank, washed it off good, throwed lime over it, washed it off again and went and hunted up some good clean dirt and covered the tank over—and by the way, it's all right—well, back to the tank.

The point I wish to make is this. Be sure to have your rubber lined phos acid storage tank spark tested after it is installed on your property. Ours was spark tested at the factory and somehow was damaged in transit or installation.

By the way, we did get a new liner for this tank; we got the tank repaired free of charge and the company did spark test it after the new liner was installed on our job site.

Phosphoric acid has a place in granulation of plant food, no doubt about it, in my opinion. I will not attempt to say how much because in our four plants in Indiana the rates and grades will vary from plant to plant. Part of this is due to economy since one of our plants is located on the Ohio River and another one just out of Chicago. Here the freight rates play an important part.

Using the same formula in computing the plant costs of these four locations on 5-20-20 the savings of phosphoric acid over sulfuric at Indianapolis amounted to 74 cents. At Jeffersonville, 51 cents; at Columbia City, 8 cents and at Hartsdale, $1.42.

Now these savings can be quickly consumed if in the process or in the formulation of phosphoric acid you need to add some sulfuric acid to aid in the granulation and this happens in some of our plants; we have to add a little bit of sulfuric in addition, just to get enough heat, to get it dry.

So if you have to use sulfuric to get this job done there goes your savings.

Plant equipment plays an important part in usage. Open flame dryers with no combustion chamber and counterflow air will take different rates than the same dryer with co-current air.

Different reactions are also experienced with dryers with combustion chambers. Therefore the equipment and other factors, such as temperature, humidity, and so forth, must be considered making it impossible to say this it it, this is the rate for this grade.

Grades which show the biggest savings in material costs by using phosphoric acid are the high phosphate grades.

In our Indiana plants these are 6-24-24; 6-24-12; 8-32-05; 5-20-20 and so forth. However, other grades, such as 12-12-12 and 4-16-16 can be included even though the savings is much less.

It just depends on plant location and freight rates and due to this geographic point in regard to raw material costs I will not go into specific formulation.

We have used both volumetric and magnetic meters. Here temperature of the acid plays an important part. At the present time all of our plants have magnetic meters. This metering is no particular problem.

Volumetric meters were not too successful for us. If you do use these volumetric meters you must compensate for the reading — to allow for specific gravity changes due to temperature. Of course this is the same as the changes which would be necessary with any liquid raw material.

As long as our pumps are delivering the phos acid to the magnetic meters at the right pressure we have no trouble with rate control.

We have used several different types of pumps. Last year we started using the positive displacement type pump and are very satisfied with their operation.

I would like to say a few more words on the storage facilities. We have used tank liners with very unsatisfactory results; we prepared the inside of some old tanks the best we could by sand blasting and grinding the rough surfaces, particularly the weld seams and even so, one year was the limit of the use.

We have tried spray coat linings. At one plant this spray coat lining was applied a year ago. On inspection we found some failure spots. These are being repaired and we believe the tank will be all right for service.

How long the spray type lining will last is anybody's guess. This is a relatively low cost lining when compared with other types of tank lining and we hope it works.

We have tried using phos acid both below bed and sprayed on top the bed and find no particular difference. However, technically speaking, we would prefer application under the bed. Actually we apply on top.

One of the very obvious reasons is because the spray pipes last longer. One of our main objections to using wet process phosphoric acid — and there are several — the main reason is the lack of uniformity of analysis. Every car or truck is different, either different water contents, solids and so forth. This necessitates continually changing the formula with resulting change in control problems.

Another problem is solids left in the car or truck and resulting inventory problem. With the best control methods we have been able to come up with we still do not know just how to handle this one. When a car is unloaded and we do not know the weight left in the car until it is returned to the shipper, weighed and notice is received on weight return. This notice being received a week to a month after the car has been used means that in the meantime we have been wondering what was wrong with the meter, or why the grade was a little off in analysis.

We do measure all tanks after they are unloaded, but even so there is plenty of room for improvement in the method of inventory control.

One car, for instance, we measured two inches of sludge left in the car and received credit a month later for 10,000 pounds returned in the car. So I think you can see the problem.

I have rather hurriedly given you a story of our use of phosphoric acid; if there are any questions I will be glad to attempt to answer them during the question period.

Moderator Reynolds: Thank you very much, Mel.

I think we will hold our questions until Rodger gets through his presentation.

Phosphoric Acid Usage in Mixed Fertilizers

Rodger C. Smith

THE use of phosphoric acid can be very useful in the granulation of mixed fertilizers. The benefits of substituting phosphoric acid for sulfuric acid in a formula can be summarized as: increased concentration, usually lower cost mixture, better granulation and less formation of noxious gases of nitrogen compounds. The beneficial effect of phosphoric acid is evidenced by the rapid increase in its use during the past three years.

The reactions of phosphoric acid during the mixing process are as follows:

If there is twice the units of P_2O_5 in the formula from ordinary superphosphate as from phosphoric acid, the ammoniation rate can be as high as 10.8. The balance of the phosphoric acid has to be calculated at 7.2 pounds ammonia per unit of P_2O_5. Experience in our plants, as I am sure is the case in many of yours, that these figures are a sound basis for use in formula calculation. That is not to say that it is practical to assume retention of ammonia in these exact amounts in all equipment, at all production rates and under all operating con-

Without the presence of superphosphate:

$$H_3PO_4 + NH_3 \longrightarrow NH_4H_2PO_4$$

The rate of ammoniation resulting from this reaction is 4.8 pounds ammonia per unit of P_2O_5.

$$NH_4H_2PO_4 + NH_3 \overset{\longleftarrow}{\longrightarrow} (NH_4)_2HPO_4$$

Assuming 50% completion of this reaction, the combined rate of reaction is 7.2.

With the presence of adequate ordinary superphosphate:

$$NH_4H_2PO + CaSO_4 + NH_3 \longrightarrow CaHPO_4 + (NH_4)_2SO_4$$

$$3CaHPO_4 + NH_3 \overset{\longleftarrow}{\longrightarrow} Ca_3(PO_4)_2 + NH_4H_2PO_4$$

$$2CaHPO_4 + CaSO_4 + 2NH_3 \overset{\longleftarrow}{\longrightarrow} Ca_3(PO_4)_2 + (NH_4)_2SO_4$$

ditions. You may find it expedient to arrive at one appropriate figure for day to day use by plant managers based on the amount of superphosphate generally occurring in your formulas, the operating conditions and the record of analyses. We are currently using 8.0—our grades being largely high analysis and containing only limited amount of ordinary superphosphate.

The advantages of being able to ammoniate P_2O_5 to these levels as compared with those used for superphosphates are evident.

The heat of reaction is as you know, about one third less for phosphoric acid than for sulfuric acid. The amounts are as follows.

Sulfuric Acid
2940 BTU's per pound NH_3
Phosphoric Acid
1780 BTU's per pound NH_3
Triple Superphosphate
1540 BTU's per pound NH_3
Ordinary Superphosphate
1430 BTU's per pound NH_3

I do not believe that it is necessary to go into a discussion of heat requirements for granulation. It can be said, however, that in most grades a slight adjustment of the water content or dryer temperature will compensate for the lower heat of reaction. The most satisfactory alternative is production of more concentrated grades which entail reaction of more ammonia and acid enabled by the use of phosphoric acid.

The choice of nitrogen solution may be affected by the higher water content of phosphoric acid. Choice of lower water content solution is particularly applicable to high nitrogen grades. Solutions 430 and 440 containing 6% urea and 6% water are examples.

A typical formula is the following 8-16-16. In this 40 unit grade, three sources of phosphorus are used, moderate rates of ammoniation are employed and there is at least the minimum heat of reaction satisfactory for granulation.

The following table contains three grades in each of three ratios which demonstrate advantages of higher concentration and of using phosphoric acid. Costs per unit of plant food are indicated using representative materials operating and transportation costs. The advantageous use of phosphoric acid is apparent.

Grade	Pounds Sol. 440	Pounds Amm. Nit. & Urea	Pounds Acid Phos. 54.0	Pounds Acid Sul. 93%	Cost*** Ton	Cost*** Unit
6-9-9 Pulv.	250	180			$53.50	$2.23
8-12-12 Gran.	358	258		100	66.00	2.06
10-15-15 Gran.	370	266	160		74.15	1.85
7-7-7 Pulv.	194	140			51.50	2.45
10-10-10 Gran.	378	272		100	63.25	2.11
13-13-13 Gran.	380	274	220		75.00	1.92
9-6-6 Pulv.	164	118			52.75	2.51
12-8-8 Gran.	306	220		100	63.00	2.25
15-10-10	360	259	220		73.00	2.09

***Includes $4.00 delivery charge

Without reference to specific data on quality of granule formation, it is our uniqualified opinion based on several years experience that it is better with phosphoric acid. The lower heat of reaction and resulting lower rate of drying, the physical structure of ammonium phosphate as compared with ammonium sulfate, and the slightly higher moisture level used tend to result in smoother, better formed granules. Stability of granules is good and recycle is low assuming adequate drying is provided.

The other advantage is less stack plume particularly, less evidence of nitrous oxide. This is very real. In the case of plants located in congested areas, a serious community relations problem can be avoided in this way assuming satisfactory dust collection is practiced. This is certainly preferable to installing scrubbing or other fume control equipment.

I have been asked to mention metering equipment. Any problems concerning this as well as storage and handling are reduced as a result of the steps taken by manufacturers to remove a larger portion of the calcium sulfate present in wet process phosphoric acid. The viscosity of even relatively clean phosphoric acid, varying widely with temperature, makes impractical the use of certain equipment, particularly the rotameter. Fortunately there is the magnetic-type meter which is not influenced by viscosity and is virtually foolproof mechanically. This type is excellent. There may be others which are suitable such as the displacement pump or volumetric measurement.

With any meter, provision in the installation for calibration is important. A satisfactory way is to put a tee in the line beyond the meter with appropriate valves and piping to a measuring tank. Volumetrically measuring the flow at a given meter setting for one minute provides a satisfactory check.

Another device which we find worthwhile is to have a reference booklet with tables of meter set-

Grade: 8-16-16 PLANT: York-Kittanning 1960-61 June 14, 1960

Pounds	Ingredients	Analysis Basis	N	Nutrient Units APA	Nutrient Units K_2O	MgO	Ammonia Input	Capacity Capacity	H_2O Units
396	Solution 430	43.0 N	8.51				79		1.19
140	Phosphoric Acid	54.0 APA		3.78				30	1.12
236	Triple Superphosphate	46.5 APA		5.49				22	.47
706	R/P Superphosphate	20.0 APA		7.06				42	2.47
488	Muriate of Potash	60.5 K_2O			15.09				
110	Sulfate of Potash-Mag.	22.0 K_2O							
		18.5 MgO			1.21	1.02			
3	Borate								
2090	TOTALS		8.51	16.33	16.30	1.02	79	94	5.25

tings for given solutions or acids and for given rates of production. The booklets also contains tables for use with the dry material feeder. Having these tables for quick reference by the plant manager when selecting a formula and planing a day's operation saves time and avoids possible calculation errors.

Phosphoric acid is making a significant contribution to mixed fertilizer production with the methods now commonly used and at current materials' costs.

MODERATOR REYNOLDS: Thank you, Melvin and Rodger.

We have some time for some questioning. I am sure there will be. Who is first?

Bill Tucker?

MR. TUCKER: Rodger, you mentioned in switching to phos acid the lower heater reaction might be compensated for by drying and water. Could you give us a range or some idea of how much water you are talking about per ton or per hour; in 8-16-16, for example, were you using 140 of acid?

MR. SMITH: Well, with the formulation—formula such as the one that was on the screen yet with the solution which is demonstrated, 440 or 430H which contains 6 per cent water—it would not be normal to have to add let's say over five gallons, three to five gallons of water per ton. I think we all recognize that this is dependent very definitely on the screen range of your classification screen and it also depends upon the cargo size of the ingredients, particularly the potash which is used.

With the general run of materials which we use and also the screen range that we are working with largely within six to 20 mesh, I would say three to five gallons per ton.

MODERATOR REYNOLDS: Another question?

MR. R. D. YOUNG (Tennessee Valley Authority): Rog, I wonder if you could give some idea of the maximum rate of phosphoric acid that can be used per ton without exceeding a recycle ratio say of one to one?

MR. SMITH: I had better add first that I do not have plant experience up to a recycle ratio of one to one or anywhere near it.

We, like everyone else,—or not like everyone else—but the majority of the industry, are trying to find short cuts that avoid the necessity of going to such a high recycle, so I cannot answer it categorically. We have had experience, however, with the use of phos acid up to about 300 pounds per ton without undue level of recycle. I'm afraid that isn't a very good answer, or a very definite answer to your question. Perhaps Mel can do better.

MODERATOR REYNOLDS: Does anyone else have comments on that question?

We have another question, I believe.

A MEMBER: Would either of the panel members care to comment on the materials of construction for pumps or pipes, metering equipment, et cetera, in using phosphoric acid?

MR. LEACH: Normally the pumps that we find satisfactory for phosphoric acid are the stainless steel or some of these alloy materials, normally stainless steel.

Now on the pipes itself we have used successfully both plastic and stainless steel. Plastic works very well. You have to watch the pressure pumps; these pumps will build up tremendous pressure and your safest bet is to go to stainless steel.

MR. SMITH: By plastic, I assume you are referring to PBC.

Incidentally, Dr. Batson who will be on the program a little bit later I am sure will go into this phase of it in much more detail.

Our experience is similar inasfar as Mel I believe mentioned sparger pipes, we have gone to the Hastelloy C which is suitable for both sulfuric and phos acid.

MR. RICHARD KULP (Dorr-Oliver): I think Mr. Smith probably answered the question. I was interested in the pipe, introducing the acid below the bed. Mr. Leach had mentioned the problems of corrosion there and the materials they were using. I am wondering whether this Halstelloy was also deteriorated?

MR. LEACH: We have not used Hastelloy on phosphoric acid. We do use Hastelloy in our sulfuric lines with good results, but we have not tried on phos acid—it does not seem to make too much difference whether it is on top the bed or below the bed so we have been staying on top the bed either using just plain stainless or PBC pipe.

MODERATOR REYNOLDS: We have time for one more question.

MR. KULP: I have a question on measuring these materials. When you have a formulation you have the phosphoric acid, for instance as in triple super and the superphosphate is there any — in making this formulation are these feeders interlocked in any way? For instance the rotor meter, the genetic flow meter for the phosphoric acid and the gravimetric feeders for the drive?

MR. LEACH: We pre-weigh the raw materials. It is weighed on a batch basis and it preferred to a continuous feed and of course the solution meters are manually controlled based on whatever flow we set for that day's operation.

Does that answer the question?

MR. SMITH: On this matter of interlocking a feeder at the meter or two meters together or all three together that can be done. We do not have it in any of our plants. I believe it is done and someone else in the room can probably contribute to that, but I know that it is the sort of thing that is done regularly in many chemical industries, but we have not found it necessary or practical to do.

MODERATOR REYNOLDS: We are going to delay the next speaker until this afternoon. I think we have a chance for a few more questions. I previously indicated only one more question, but I think we can switch this around a little bit and we will have some more.

MR. RICHARD POWELL (International Mineral and Chemical Corporation): I would like to ask Rodger Smith is the phosphoric under the bed or on top the bed?

MR. SMITH: The question is: Is the phosphoric under the bed or on top of the bed. We are putting it in on top of the bed—I'm putting it in under the bed.

We do have a batch plant where we are putting it in—spraying it in on top, that is, the solution, but in this ammoniator, under the bed. We have not tried the cutting on top. The indication here—I know I've talked to the people who seem to indicate it works perfectly satisfactorily.

MODERATOR REYNOLD: I saw another hand.

MR. JACK FREDERICK (Sohio): On 5-20-50 do you have to add any sulfuric acid at that particular rate to get the necessary heat?

MR. LEACH: The question is, do we have to add some sulfuric acid to 5-20-20 formula figured from all phosphoric acid, no triple, right?

MR. FREDERICK: Yes.

MR. LEACH: This depends entirely, I think, on the equipment. At some of our plants we do add sulfuric acid; some we do not have to. At the Indianapolis plant where we have a big dryer, dryer ample enough to take out the water, we do not add sulfuric acid.

At some of the other plants where we have open flame type dryers with either the counterflow or co-current air we do find it necessary to add some sulfuric acid up to sometimes as high as 60 pounds which you can see does affect the cost of the formulation considerably. Our big problem with the phosphoric acid at these plants that do not have sufficient drying and that is the problem. We do have the one big problem of drying to get the material down to two per cent moisture.

MODERATOR REYNOLDS: Any other comments on that subject?

MR. ALBERT HENDERSON (Wilson and Toomer Fertilizer Co.): Wouldn't spraying acid on top of the bed produce some serious clean out problems of ammoniators?

MR. LEACH: It does on some days and I think that probably is a factor. However, we do not seem to have the lumps or the balling effect in the ammoniator when we spray on the bed so we generally have to weight one against the other.

A lot of times we have tried spraying this acid under the bed and come up with these big balls—those big granulars, you know—in the ammoniator; where we spread it on top of the bed we do get away from this problem and of course you do have to have the knockers or scrappers on the ammoniator to keep the walls clean.

I think in this regard the pressure of the pumps supplying the acid plays an important part. If we get this material atomized on top the bed we have a lot better results than if we don't have sufficient pressure. These pumps operate up to 165 pounds of pressure at the meter in order to get this phosphoric acid atomized in the sparger.

MODERATOR REYNOLDS: This comment concerning increased ammoniation ratios that Rodger brought out calls to mind a couple of years ago this same subject came up on ammoniation ratios and there was practically no indication that we would go above that 7.2. I wonder how much actual practice is behind this. I know that in the last two or three months we have had some reports of successful attempts to go higher on ammoniation ratios.

Does anyone care to expound in that experience of Rodger's? I believe 8 was mentioned as a figure and I think the theoretical was even up around 10.

Is anyone brave enough to try 10?

A lot of experts put their hands up earlier—

I don't know whether we are being bashful.

Rodger?

MR. SMITH: Just to elaborate a little further. I certainly wouldn't indicate that we, or anybody else, would have no loss at those higher levels. Perhaps I could put it in a practical way best by saying that by following these rules of ammoniation, or levels of ammoniation, it is our experience that the ammonium loss is no more, or I would say in most cases less than with sulfuric acid under similarly good ammoniation techniques. I believe that you have considerable basis.

A MEMBER: Since you mention the ammonia losses would you care to comment on what is the degree of loss; how much ammonia is lost in this production, the range?

MR. SMITH: Well I will comment first and perhaps Mel can later comment. Of course the most practical answer to that is the amount of overformulation which is considered necessary. That formula that I had on the board has a half unit overage which is on the high side with an 8 unit—we do not usually find it necessary to go quite that high.

We find it necessary to go in the range—on low nitrogen grades—as low as two to three per cent overage on total nitrogen and out to a maximum of four to five per cent on high nitrogen grades and that's going up as high as 15 units of nitrogen.

MR. LEACH: I think that's a good question and it is really one that we do not have any data on at all so all I can say is this: there is, we think, a little bit of difference in retention between say 5-20-20 or 1-4-4 made from sulfuric acid or from phos acid.

We believe we have a little bit less retention of nitrogen when we use phos acid than we do when we use sulfuric. There again this may be because we are spraying phos acid on top of the bed.

I think our quality control boy—and he's here—and I think he will say that on phos acid where we use all anhydrous ammonia in 5-20-20 we group about a pound or half a pound per minute on the meter.

Now, as Rodger said, we do overformulate on 5-20-20. We will overformulate to 5.2. However, we do not lose this. There is a lot of other factors involved in nitrogen losses other than whether you are using phos acid or sulfuric acid.

One of our—the best ways to retain this nitrogen is to get a good sparger system with the correct pressure on the sparger bar. In our opinion that does more than what materials you are using in this nitrogen retention thing.

MODERATOR REYNOLDS: I think we are going to have to call this session to a halt here. You've been a very patient group.

We will resume here at two o'clock.

(The Session adjourned at twelve-thirty-five o'clock, p.m.)

Wednesday, Afternoon Session, November 2, 1960

The Round Table reconvened at two o'clock p.m., Messrs. Joseph Reynolds and Albert Spillman presiding.

MODERATOR REYNOLDS: The first question confronting a potential user of phosphoric acid is: How do I store and handle phosphoric acid?

Many advances have been made in the field of lower cost storage and improved and lower cost piping. Techniques of handling wet process phosphoric acid in cold weather have also changed in the last few years. The next speaker, Dr. F. M. Batson, General Chemical of Allied, has been asked to summarize the subject of Phosphoric Acid Storage and Handling.

Phosphoric Acid Storage And Handling

F. M. Batson

WET process phosphoric acid has been used by the fertilizer industry for many years in the manufacture of triple superphosphate. As a result, equipment and materials used to store and handle the acid have been pretty well developed and proven out. This branch of the industry has encountered no unusual problems in handling this acid.

In recent years the dry fertilizer industry has started to use phosphoric acid for granulation, and the liquid fertilizer manufacturers are using wet process acid increasingly in various liquid formulations. With this increased usage, some new types of equipment for handling the acid have come into being. Problems initially experienced in handling the acid have been largely overcome as acid manufacturers improve their product, and the fertilizer manufacturers improve their techniques.

This paper presents a brief resume of types of equipment for storing and handling phosphoric acid, and a few comments on handling procedures. It should be pointed out that references made to equipment or materials of construction are simply reflections of present industry practices and are not intended to favor any particular equipment. Materials of construction and operating procedures will vary from plant to plant, and there may be equipment and procedures employed other than those described here. This presentation will attempt to cover the practices which appear to be the most widely used, with some consideration being given to their serviceability and economics.

The intention is to try to cover the items which are most frequently of concern to those handling phosphoric acid. If anyone has questions which are not covered, or additional information that may be of help to the industry, it is hoped these will be presented during the discussion period.

Storages

The storage most commonly used, and the one that will give the best overall service at reasonable cost, is the rubber-lined steel tank. A storage of this type will cost on the order of $0.40 to $0.50 per gallon, plus installation costs. It should last twenty years or more.

Tanks of type 316 stainless steel may be used, although this material is more subject to attack by wet process acid. A tank of stainless steel would cost roughly $1.00 per gallon, plus installation costs, for plate 3/16 inches or more in thickness. Thinner gauge sheet is not recommended.

Polyvinyl chloride (PVC) bonded to steel has worked out satisfactorily for vertical tanks, but is somewhat less attractive for horizontal tanks. The cost of this type of storage is about the same as the rubber-lined tank.

Another type of storage used for phosphoric acid consists of a plastic bag inserted as a loose liner in a steel or wooden tank. Costs for this type of storage will run perhaps $0.20 to $0.25 per gallon. Although this combination of materials is less expensive than a rubber-lined tank, it may be less durable. Such storages have not been in use long enough to get a good idea of their service life. Plastic liners for storage tanks can be obtained with either open or closed tops. For use with wet process phosphoric acid, we would suggest an open top liner, or at least one which would allow entrance for cleaning purposes. For maximum service the plastic liner should be a heavy one. One type now in use is about 30 mils thick. The tanks in which they are inserted must be smooth and free from rough edges that would tear or puncture the liner. It would be interesting to have comments from any here who have used this type of storage.

Rubber containers, made in the shape of a large bag and closed except for an outlet at one end, have found service in transporting phosphoric acid on flat bed trucks, but these are not as practical for storage as the more conventional types of tanks.

Finally, there has been some use of the pool-type storage. This offers a large volume of storage at a low cost, about $0.05 to $0.10 per gallon. A storage of this type can be built on top of the ground by moving in earth to form its walls, or it can be built partly below ground level. The interior of the storage is lined with an acid resistant material, and a roof built to keep out rain and dust. One type of lining material that has been used consists of several layers of asphalt and burlap, on a base of heavy screening.

The site for such a pool storage must be chosen to eliminate ground water problems and to provide for drainage facilities. Where a pool storage is considered, it is recommended that an engineer be brought in to thoroughly study the ground characteristics and make certain that the location is suitable.

Experience with pool-type storages is somewhat limited as yet. We have one that has been in use nearly a year and it has performed very well. We have three others under construction. There were difficulties with some of the earlier pool-type storages but these problems have now been corrected. The pool storage should be regarded as a secondary type of storage, a low cost auxiliary reservoir, to back up one or more primary storages such as rubber-lined steel tanks. They might be considered where there is a large seasonal demand for acid, or where a manufacturer wishes to insure against acid shortages.

Pipes, Pumps, Valves

A variety of pipe materials have been used for handling phosphoric acid. The material most commonly used for rigid pipe lines is unplasticized PVC. This is very serviceable, and of relatively low cost, and has the added advantage of being easy to install. Rubber-lined steel gives good service, but is more expensive. Installation is more of a problem in that the sections must be ordered and fabricated to the proper length. Epoxy-glass piping stands up very well in phosphoric acid service and is finding increased usage. Type 316 stainless steel is satisfactory, but is more costly than the other materials mentioned.

Several types of valves have been used in phosphoric acid service. Probably the one most commonly used is the Saunders Patent diaphragm valve, with either a steel or PVC body. These give good service, at relatively low cost. Valves made of FA-20 alloy, or type 316 stainless steel, give excellent service but are more expensive. Teflon-lined plug valves of FA-20 alloy fall in this same category.

For pumping phosphoric acid the common practice is to use a centrifugal pump made of FA-20 alloy or type 316 stainless steel. The extra cost of the FA-20 alloy pump is more than justified by its longer service. Rotary gears pumps can also be used where self-priming features are desirable, or for high heads. Pumps suitable for phosphoric acid are available as standard items from many manufacturers.

Agitation In Storage

As is generally known, wet process phosphoric acid tends to drop out solids on standing, and this in time causes a build-up of precipitate in the acid storage. This has generally been more of a nuisance than a problem. The usual course of action is to do nothing about this until it becomes convenient or necessary to clean the storage, usually about every year or two. The question has sometimes come up concerning agitating the storage, thus keeping the solids is suspension and removing them along with the acid. This is not particularly practical. It would conceivably work if the acid was agitated continually, seven days a week. If agitation is interrupted the solids will settle and will probably not resuspend.

Acid producers are aware of this condition and have expended considerable effort toward reducing the drop out from wet process acid. In our own product, a considerable reduction has already been made. There is little doubt that this deposition could be virtually eliminated, but to do it economically enough is a real problem. It may be expected, however, that as time goes on, the precipitate from wet process phosphoric acid will become less and less, and the question of storage cleaning will, at least, not be a pressing one.

Acid Heating

In most areas there is no problem with phosphoric acid freezing. While the freezing point is generally given as about 0°F., we have actually cooled acid to —50°F. without it freezing.

At lower temperatures the acid does become more viscous, making centrifugal pumps less efficient, and making it difficult to get accurate measurements with the rotameter type flow meters. For this reason a number of plants have gone to the magnetic type flow meter which is not affected by changes in viscosity. Most fertilizer manufacturers use the acid without having to heat it.

If heating is necessary it could be accomplished in several ways. The simplest method would be to install the storage tank in a heated building. Another method would be to insulate the tank, and to pump the acid through an external heat exchanger, diverting part of flow back into the storage. Generally, heating should not be necessary, except for possibly the very cold parts of the country.

Deposits In Piping

Very infrequently questions arise concerning the formation and removal of deposits in piping. Generally, under normal operating conditions, using a pipe line 1½-2 inches in diameter, this has presented no problem. It is possible that a deposit may develop under certain conditions, for example, where the pipe is under size, and where the flow rate through the pipe is very low, or where acid stands in a pipe for long periods without use.

If a pipe line does become clogged, the best procedure is to try to force hot water through the pipe. If this procedure is not successful, the pipe should be dismantled, cleaned by physical means and flushed with warm water.

Unloading Tank Cars

Phosphoric acid tank cars are usually constructed of rubber-lined steel and have no bottom outlet. They are unloaded through the dome by means of compressed air. This method is entirely satisfactory for transferring acid from the tank car to storage. If, however, the acid is to be transferred from the tank car directly to process, sometimes through long lines, or to higher levels, it is preferable to blow from the car to a nearby pump which then moves the acid to process. This method insures a full and uniform flow of acid. Any fluctuation in air supply caused by use elsewhere in the plant would not affect the flow of acid since only a small amount of air is needed to get the acid to the pump.

Occasionally difficulties have been reported in unloading tank cars of wet process phosphoric acid. The operator has applied air pressure slowly to the car, up to 30 pounds maximum, without getting any acid flow through the dip pipe. Usually what has happened here is that, on prolonged standing, the deposit from the acid has filled the small sump below the end of the dip pipe, effectively plugging the end of the pipe. In such a situation it is generally advisable to contact the acid supplier for advice or assistance.

The first suggestion is to try to blow back the dip pipe. The air hose is connected to the dip pipe, leaving the normal air connection on the car open to the atmosphere. Air, up to full plant pressure, may then be applied to the dip pipe. Before applying air, it may be helpful to try to loosen the deposit in the lower end of the dip pipe by forcing with a round-ended wooden rod. During the blowing operation care should be taken to protect personnel against any acid that might come out the air inlet connection in case the dip line suddenly clears.

If the blow-back procedure is not successful, then it will be necessary to actually remove the dip pipe and clean it out. All lines to the car should be disconnected before carrying out this operation.

Conclusion

If any fertilizer manufacturer is considering the use of phosphoric acid for the first time, it is suggested that he contact an acid manufacturer, who will be able to advise on all phases of acid storage, handling and unloading.

MODERATOR REYNOLDS: Thank you very much, Dr. Batson. That was most interesting and most informative. I know we will have some questions now. The first question.

MR. THOMAS PEARCE (Swift & Company): You indicated that you have improved your wet process acid by increasing the storage so that you have dropped out more solids. Would you care to make some comments about how you have done this?

DR. F. M. BATSON (General Chemical): This is a manufacturing problem. I would correct a statement. I said we have improved the acid; I did not say we had done it by storage. In the early days of phosphoric acid, it was new to us, this fertilizer use, it was new to you people on how to handle it. As Mr. Leach mentioned this morning, there were problems. Over the last several years, we have worked on our acid, as I am sure the other manufacturers have. At the same time, fertilizer manufacturers have learned more about how to handle this. But, to answer your question, I am going to ask our Mr. Vik from our production department to comment, if he will, on what they may have done on improving acid.

MR. OLAV VIK (General Chemical of Allied Chemical): This is normally called passing the ball. We have been trying to reduce the so-called after-precipitate, which, of course, is what is giving us the problems and we are working on this problem. I do not think, to be frank, that we are ready as yet to tell how we do it.

DR. BATSON: Thank you, Olav. I got out of that one.

MODERATOR REYNOLDS: David Long of Miller Chemical, would you like to comment on the use of the fibreglass epoxy types for storage in addition to the use of these plastics for pipelines?

MR. DAVID LONG (Miller Chemical & Fertilizer Corporation): I have seen some small tanks used for storage. We, ourselves, have not used them. We are using epoxy glass piping and it is turning out very well. I know that it is more expensive than the PVC. Some figures I was looking at the other day would suggest to me that epoxy glass might also be as expensive, or possibly a little more expensive than rubber lined steel. It may be about the same; it may be a little more. It does stand up. I frankly have not seen enough of them in use to say any more.

MR. BEN GILES (California Chemical Company): You mentioned heating and agitating in tanks in connection with the heat exchanger. Would you recommend against an internal steam coil with agitators?

DR. BATSON: I believe we would. The question there becomes one of leaking, if the steam coils spring a leak, and you would have to have the right materials for a steam coil so as not to have corrosion. In some of our experiences where we had internal coils and agitation, both erosion and corrosion leaks occurred. I think we would generally recommend against internal heating.

MR. GILES: I was wondering couldn't you use the material, say, that you use in your evaporators, 317 or something like that?

DR. BATSON: Olav, you're on again.

MR. VIK: If you are talking about heating, Carbon D-20 should last you for several years. It's when you get up to boiling point and fluorine is driven off it is actually the fluorine attack that hurts you.

MODERATOR REYNOLDS: Any more questions?

A MEMBER: You mentioned that you would recommend that you allow the sludge to just remain in the tank and accumulate until such a time as something has to be done. That is your present recommendation, is that my understanding?

DR. BATSON: The question was: Did we recommend that sludge just be allowed to accumulate in the tank and then take it out when something has to be done?

Perhaps I should qualify this a bit by saying that when and how often you clean out tanks is going to depend upon the acid, on the history of the acids. Acids are made by different processes and at different times have different amounts of mud in them, so I cannot speak for everybody. However, we have customers who are using acid and letting it accumulate. Again, I would say this would depend on your storage, the design, where the take-off is and so forth and whether it gives you any problems.

One I have in mind, mentioned just today, is going to be slack in a couple of months and we think we will clean it out, but I do not believe there is much there. This is a thing that you will have to find out for yourself how fast this builds up, what the construction of your tank is, whether you can still get acid out. If you do have a slack period, take a look at the tank, if the mud is appreciable, clean it out, if it's not appreciable, let it stay.

MODERATOR REYNOLDS: Let me ask a question. With reference to the plastic bag lined steel tank, have you any experience with agitators in those tanks?

DR. BATSON: In the first place, our experience with plastic lined bags—you're talking about the loose liners, now?

MODERATOR REYNOLDS: Correct.

DR. BATSON: I am not aware of anyone using agitation in tanks with a loose bag type liner, are you, Roger? You haven't seen any either. No, we have not encountered any.

A MEMBER: I have a question, please. At the risk of seeming nosy, I would like to ask: Is this improved wet process acid produced at your Claymont, Delaware plant?

DR. BATSON: Yes, we are also going to produce it in St. Louis in a few months.

SAME MEMBER: Yes, I'm being nosy, or, yes, that's the answer?

DR. BATSON: Yes, we are producing this acid at our Claymont works and yes, we will be producing it in St. Louis in a couple of months.

Thank you.

MODERATOR REYNOLDS: We are now to the subject of Behavior of Triple Superphosphate in Mixed Fertilizer Formulation. What are the problems in ammoniation, granulation, formulation, variation in P_2O_5 recovery, influence of moisture and so on. We have assembled a panel of experts to discuss this subject. We have had some slight revisions to the names of the people to appear on the panel. However, it is my pleasure to introduce the team of Frank Nielsson of International Minerals, Joe Markey of Tennessee Corporation, Dan Walstad, American Cyanamid, and Charlie Franklin of International Minerals.

MR. FRANK NIELSSON (International Minerals & Chemical Corporation): We have assembled here a small group, and I am just a leader like Joe so I do not do anything but give orders, I guess, but each fellow will give his speech and later we would like to have you come up with your questions, if you have any.

Generally speaking, we are all agreed that triple super is an important source of P_2O_5 for the nation's agricultural economy and its growth has been substantial.

It is easy to say that a product has jumped 200 per cent in sales, these guys in sales always tell me that. They sold one last year, this years they sold two, and they have 200 per cent.

We are in a little bit different category. Triple has jumped 1,860 per cent in the last 20 years and right now it stands at about the same place that ordinary super did in 1930; that is, the 1959 triple was up around 800,000 tons. We think that the use of phosphoric acid may increase the use of super a little bit more—it has been on the decline a little bit—but I believe it will not be too long before the production of triple and the downhill use of super will probably intersect at about a million tons per year.

We talk in millions and it is a great big number and unless you boys are figuring out income taxes or fool around with government taxation you do not have much idea of what the devil we are talking about. So to give you an idea at at the other end of the line as to what we mean when we talk about a million or a part per million, I will repeat something I heard in Atlanta recently.

Imagine a real soak drinking two martinis at each meal, three meals a day, since the time of Abraham. That's a long time ago. Now, if he started out with a bottle of Vermouth and he was adding one part of Vermouth per million parts of gin, he would still have at this time one half of the original bottle of Vermouth left after drinking martinis three times a day for 5960 years.

I just want you to know that when we talk about a million tons, we are talking about a lot.

We have Dr. Markey the Director of Development for Tennessee Corporation, Charlie Franklin from I.M.C. Corporation, Technical Service, and Dan Walstad from American Cyanamid, Technical Service.

Dr. Markey will be the first speaker and, if you will please reserve all of your questions until after the three experts get through, we will then try to go along in a pretty orderly fashion.

DR. J. MARKEY (Tennessee Corporation): You know, it is with a great deal of mixed emotions that anyone has the nerve to come up front of this group and attempt in any way to tell you anything new, because this Round Table has, in my opinion, and I think in the opinion of every man here, become both the technical and the practical institute for the entire fertilizer industry. But about this mixed emotions I just spoke of, I just heard a new definition for mixed emotions as the feelings that one develops in oneself while watching his mother-in-law back over a cliff in his brand new Cadillac.

As I say, it isa little presumptuous to think that I could come before a group like this and cover anything that was new to you.

Since World War II, and particularly in the last 10 years since the Round Table has been having these annual meetings, there has been a tremendous group of developments. As a matter of fact, I think it would be very safe to say that in the course of the last 10 years the fertilizer industry has practically revolutionized itself. These developments have come in high analysis, in granulation, continuous processes, increased ammoniation rates, to name just a few. Day by day more and more new developments are on the horizon.

During the course of the last 10 years a tremendous amount of excellent work from our colleges, most certainly the U.S.D.A., by our good old friends the T.V.A. and, not the least, the researchers and the technical service groups of the entire industry. These findings have been coming out at the ACS meetings, other meetings, and most of all right here at the Fertilizer Industry Round Table. I cannot help but feel that in the course of this 10 years and as all of these developments and findings have come forth that in many instances it has almost caused a panic in the fertilizer industry. I think that people have come and they hear about continuous ammoniation and they think, "My goodness, I've still got a batch mixer; what am I going to tell the boss?" We are coming today with these problems of the use of phosphoric acid, we have heard about increased rates of ammoniation, and I remember at these meetings people in the back of the room said, "I get two pounds," and the man in the front of the room insists he got six.

I think that everyone has been coming to these meetings through the course of the years expecting to hear an answer, expecting to find out what is going to happen here, what is high man on the Totem Pole, what is going to come forth from this whole thing? Is the whole industry going to granulation, is everything going to high analysis, is my batch plant going to be obsolete, do I have to go continuous?

I cannot help but believe that too many people have come to the Round Tables and to these other meetings hoping to hear the one answer, the panacea, the be all and end all. I think through the last year and I think this year the answer to those kinds of problems and those kinds of questions is simply this. In my opinion there is no one answer, and anybody who thinks he is going to find one single answer that is going to be the high man on the Totem Pole I think is looking for the wrong thing.

To go back again, being that there is not one answer, I think Joe Reynolds hit it in the keynote this morning when he said that there are a number of things to be considered. I think the driving force of the whole thing is, of course, economics, and I think in this question of economics there are a number of questions which come up: the demand in your own particular area, the existing circumstances around you, yourself, personally, at your own mixed goods plants, the state of the technology, both in your plant, within your company and within the entire industry, and, of course, this ever-driving thing: what in the world is the competition doing?

Going back again to this thinking, that there is no one answer, I hope here this afternoon to accomplish just one thing and that is not to bring up anything new but merely to refresh your minds and to bring back into your recognition just one small facet of this tremendously large, varied and complex industry that we are all a part of. This one facet is the use and problems of triple superphosphate in mixed goods. Again, the point that I really want to make is that there is no one answer.

In the problems of triple superphosphate, I think that the largest single problem, at least is from the point of view of a producer, is to out-guess you fellows both from your consumption point of view and from the point of view of just what you want in a triple superphosphate.

I would like here, speaking of consumption, and Frank Nielsson brought this out, I have a figure that I would like to throw on the screen at this time relative to the consumption of these various phosphate materials.

(Slide) As Frank has already indicated, you will notice the top line is the total P_2O_5 in tons, not tons of materials. This is all on a comparative basis. This indicates that there is some two and a quarter million tons of P_2O_5 used in the United States today.

You will notice the second line from the top is the P_2O_5 that is carried in normal superphosphate, and, as Frank has already indicated to you, you can very easily see that through the years, and the first year on the left is 1952 and the last year on the right is 1959, even in this last decade there has been a very marked decline in the total normal superphosphate.

Coming down another line, as Frank has already indicated, this indicates the P_2O_5 carried in triple superphosphate and it has gone from some 400-thousand tons to almost a million tons of P_2O_5.

Another big factor that came in in the mid-thirties with these other concentrated materials and you can see that they came from a very scant beginning and are now marketing at an appreciable amount, between a quarter of a million and three hundred thousand tons of P_2O_5.

Again, I do not think that any of us should sit and draw a conclusion that any particular thing has happened. I think that the big thing that we can say in the phosphate field is that the total use of P_2O_5 in the United States is very definitely on the up. Triple is very definitely on the up. I am not too certain that I would like to draw the conclusion that superphosphate is on its way out; I don't believe that at all.

Back on this other large problem of triple superphosphate, and that is on the type of material that you, the mixed goods manufacturer, want, there is some question at times as to whether we really know what each other want. This is very easy to understand if you look at just how complex this entire mixed goods field is. In the first place, you can have pulverized mixed goods, semi-granular mixed goods, full and complete granular mixed goods, you can have direct application of granular raw materials, then you can also have mixes of granular raw materials. You can complicate this by running them through either a batch plant or a continuous plant. On top of this, you can complicate this by using very high analysis materials. You can also prefer to have high analysis final goods or you can prefer to have low analysis goods. I think we are prone to forget when we are talking about our 12-12-12's and our 15-15-15's and our 100-100-100's that there is still a substantial tonnage of things like 6-8-6 on the market.

You can complicate this even further, and this has already come up, as to the additions of special materials like sulfuric acid, phosphoric acid and so forth. So you see there is no easy one answer.

This business of supplying raw materials to the mixed fertilizer industry has contained within it a number of joint responsibilities. Most certainly the supplier has a group of responsibilities, and I would like to name a few of those. Most certainly any manufacturer of any raw material, in this particular case triple superphosphate, must be responsible for its chemical and its physical properties. He must also assure that it is as consistent material as is humanly possible to deliver. He must also constantly be vigilant to improve this material and to offer the best product possible to the customer.

With specific reference to triple superphosphate, I think the manufacturer has responsibilities in providing adequate surface area for ammoniation, this comes both in particle size and in porosity. In addition to that, I think that there is a definite responsibility for providing a material with the proper moisture content, and so forth, again for ammoniation, and proper amounts of free acid.

For success, there must also be certain responsibilities that the mixed goods manufacturer must assume for his own success and this most certainly is beyond the hands of any materials producer. This would be led off by operational control, of course, within your own plant. The proper handling of the raw materials, for one. In the case of mixed goods in ammoniation to establish proper ammoniation temperatures and ammoniation conditions. Developing that further, the proper distribution of nitrogen solutions and so forth, providing you are scheduling your production on adequate reaction time. There again, in the quality of your mixed goods, this is further complicated by drying and/or cooling, your operations are also modulated by the formulations that are available to you, but most cardinal of all your responsibilities lay in economy and in the economics of your operation.

Back to triple superphosphate for just a minute, back in the earlier days, and triple has been available through some 30 odd years, but back in the early thirties I think all triple superphosphate was plagued with much the same problems that normal superphosphate was back in those days, excessive dusting, caking, pile set, and in the case of bags, bag rot and whatnot due to the excessive free acid, certainly a very poor handling material, but I think probably the hardest and most difficult problem to resolve was the setting up in transportation. I think probably many of you have experienced the case of receiving a car and practically have to dynamite the car loose to get the triple superphosphate out of the car.

Through the course of the years the industry has improved their production procedures, they have been constantly vigilant in attempting always to get a material of better handling properties.

My comments here are limited, I think, to our particular material which is produced at U. S. Phosphoric Products. Through the years and the process development and so forth you will all recall where we put in, in an attempt to better this handling ability, a drying step and, of course, then we went to the other extreme. I think there were a lot of complaints received and people were scratching their heads over the material that was case hardened.

Again, through the years of development, I think that further process improvement for the milling of these materials and so forth has now produced a product and a material which is a fairly good compromise between all of your varied requirements.

I would like to put a blackboard up here and point out a few factors about the types of materials of triple superphosphate that are available today (drawing on blackboard).

As I attempted to point out, there is no one triple superphosphate that can possibly satisfy all your varied needs and demands.

In the course of process development and so forth, particularly through the past ten years, three particular types of triple superphosphate have not become available: the ROP, the run-of-the pile, the coarse material and what is known as a granular material. I will just quickly try to point out a few little highlights to you.

In the ROP, the total 48 per cent with an available APA of 47. The coarse is quite similar to this, 48 per cent with an available 47.4, substantially the same. There is a little difference, though, with the granular. You will notice that the total is 47.6 with an available 46-plus.

I call your attention to moisture content of these particular superphosphates. Again, the ROP and the coarse being very similar with a 4 per cent moisture and a 3.8 per cent moisture. The granular again being substantially drier with a 2.9.

I call to your attention the phosphoric acid free acid in this material, the ROP and in the coarse again running significantly the same, 3.5 and 3.6. Again the granulation being somewhat lower, 2.8.

Why do we have all these different types of triple superphosphate? Let me call to your attention the screen analysis which is set up with Tyler screens. I think we can very quickly point out the big difference between these various products. You will find in the run-of-the-pile that there is an average distribution, practically nothing in the 4 to 6 mesh range, from the 6 to the 20 and from the 20 to the 35, the 35 to the 60, and the minus 60 fairly uniformly distributed, 25 to 38, 16 to 23, 10 to 22, 30 to 38. So that the run-of-the-pile is fairly well distributed through all these various size ranges.

The course is a specially developed material that you find will fall immediately off this pattern in size distribution. Again there is practically nothing in the fraction of 4 to 6, but you will find here that there is a substantial difference in the fraction of minus 6 plus 20, practically all of the material lays in that fraction; between 65 and 78 per cent of the material falls there.

Between the minus 20 and the plus 60, a small fraction, 20 to 28.

One of the other significant things is that in the minus 60 range almost all the fines are now gone, only 2 to 5 per cent of the material falling in that class.

The granulated, of course, is a breed of cat all to itself, 95 to 99 per cent will fall in the minus 6 plus 16 fraction, and only from 1 to 5 per cent will fall in the fines.

Why all these varied materials? The why of all the varied materials is to meet your varied needs.

Before going down to what you would want to use each of these materials for, I would like to point out some other factors and facts just to refresh your memory on these. I have some slides on some of the problems that you run into on ammoniation and I would like to touch on those next very quickly. There are a number of effects.

(Slide) This slide is to show the effect of particle size in ammoniation and this, in itself, partly answers the why of the different type triple superphosphates. We find here that as the size increases there is a very sharp drop-off in ability to amoniate. These all are ammoniations at 5 pounds of free ammonia per unit of APA and these would apply to a 41 per cent nitrogen ammoniating solution. The vertical axis shows the efficiency of the recovery of nitrogen, whereas the X-axis carries you out through the various sizes. You can very easily see that it would be wonderful to have a minus 60, you could get practically entire absorption of nitrogen. But this isn't entirely practical because, as I said, this is the type material that is hard to handle, it is dusty and sets up in a car, so there is some compromise answer.

I would just like to point out that the coarse material would fall in this general vicinity (indicating), whereas the run-of-the-pile is a little different compromise and will run you much higher on the graph and is approximately in this vicinity (indicating).

Again, there is no one answer; it's a compromise and each of you are going to have to study the facts and analyze for yourself just what best suits your needs. There are other things that affect ammoniation and the next slide shows the effect of moisture.

(Slide) Again the vertical axis shows the per cent of recovery and this is at an ammoniation rate, again, of 5 pounds of ammonia per unit of APA. The X-axis shows the effect of moisture. As the moisture increases to 5 or 6 per cent the ability to absorb nitrogen also increases.

There is a compromise here because the more moisture you ship out, the more freight you have involved, the lower the APA, but, again, you could not have a material so dry that it would be unusable to you, so the compromise is reached. As you will notice, both the ROP and the coarse fall in this general area where the moisture ranges from 3½ to 4.

The next effect on ammoniation is in the next slide (slide) which is the effect of temperature on ammoniation. Of course, you are all only too familiar with the differences between the ammoniation of normal super and triple super, which has been so adequately described in other meetings here, but now this is certainly something that falls strictly within the province of good operating procedure in a mixed goods plant. Here again, this is something that is certainly beyond the control of a triple manufacturer.

You will notice that the efficiency of ammoniation greatly improves and increases as the temperature rises. Again, the point which has been made so many times before is that with the triple superphosphate it is a must to operate your ammoniators at a higher level, certinly 150 degrees to 175 degrees at a minimum, and I think the general procedure is most often to have it up in the neighborhood of 200 to 212 and slightly higher.

There is still another factor affecting ammoniation and this is ammoniation rate. That is the next slide.

(Slide) You will notice that as the ammoniation rates increase the efficiency of recovery of nitrogen drastically decreases. I think through the years there has been a lot of discussion as to what the different rates of ammoniation are or what they should be. Of course, in normal superphosphate the theoretical is some 9.4 but it is good practice to obtain anything from 5 to 6. With triple superphosphate we are told the theoretical is 7.3. We find that it is good practice to get anywhere from 3½ to 4 with the run-of-the-pile and from 2½ to 3 with the coarse.

Phosphoric acid, which has been touched on earlier, its theoretical ammoniations can be as high as 9, 8.9 to 9, whereas good practice limits it to 6.5 to 7.2. I think that is brought out quite clearly here because it certainly would not be good practice to go up to the 6 and 7s and taking a beating on the loss of nitrogen. Again, the compromise is taken and one would stay in this region to keep up with good absorption.

Having pointed those various factors out to you, I would like to call your attention again to the blackboard which shows the different types of triple. In here, as I pointed out, the highest ammoniation rate is the one which has the smallest particles in the material. You can get the higher rates from the 3½ to 4. If on the other hand you have a compromise that requires using the coarse, you are taking out the fine particles and you are maximizing this larger particle fraction from minus 6 to plus 20, therefore you must penalize yourself to an ammoniation rate of 2½ to 3 in good practice.

On the granular material, however, where 95 to 99 per cent of this is a solid particle in the minus 6 plus 16 range one penalizes himself with the fact that that material will practically not ammoniate and it would be good practice to obtain between ½ and 1 pound of ammonia per unit of P_2O_5.

Why would one want to use these various materials? I would like to point out some of the other things besides ammoniation that would dictate which of these materials you would want to select.

Before passing on to that, there were some other factors which I did not want to slight and I will just quickly mention them, other factors which are entirely within the control of the manufacturer, such things as the distribution of solution, the type of sparger and the way the holes are in, very critical. The reaction time, I think is most generally conceded, and we have heard it a number of times here, should be in the neighborhood of three minutes, cooling and drying. These are important things, particularly in your high analysis grades, particularly in your granulation material where you have been operating at high temperatures and during many past sessions we have heard discussions on reversion and so forth, and then the cooling. Of course, those are still there and good operating practice calls for one to pay attention to those.

How should these various different materials be used and why are these factors to be considered? Run-of-the-pile would, of course, be chosen in the event that one would be interested in a pulverized material, and I do not think that too many people today in a high analysis field using triple would be interested in the pulverized material. But, again, should one be interested in pulverized high analysis, it most certainly would be the run-of-the-pile that you would be interested in.

If you were interested in full granulation in any particular grade, the type that you would be interested in would be the run-of-the-pile. Here again, this gives you this wide variety of particles. It gives you enough of a small sized fraction to wrap up a good granule to provide a good uniform, granular material.

The ROP again points out the features of having enough fines there to provide a good ammoniation rate. I might even suggest that one of the things, and I am certain there is a possibility you are doing this within some of your plants, and even the ROP could be improved within your own plant by further milling down to a smaller particle size to make a more uniform and perhaps a better final, fully and completely granulated mixed goods.

Why would one want to use the coarse? The coarse material does have advantages and it is found and has been shown to be best for this class of material which has come to be known as the semi-granulated. Herein there are enough coarse materials to act as the nucleus for the granule for the other fine materials to wrap up around. Here again, in the semi-granulation processes you certainly do not have the totally and completely uniform particles, each particle, as you do in the complete granulation, but this has become very popular in the industry and a nice material and a nice product and a nice mixed goods is meeting the market in this area.

If in your particular set of circumstances, this is the type of plant that best suits your area, then in triple superphosphate you would prefer to use the coarse.

On a granular product there are occasions and times when direct application is needed. In a situation where you are not interested in ammoniation, you are not interested in mixing this with nitrogen solutions, perhaps in some of your plants you are interested in mixtures of granular raw materials and in that case your choice and what you would be interested in is in the granular product.

Here, now, as you can see there is not a thing that I have told you that each of you do not already know and have already been practicing. The entire thought here was to just bring these back to your mind, refresh them in your memory.

Here again, one cardinal thing to be pointed out is that there is no one way. There are just as many different ways as there are men sitting in this room. I think that the triple superphosphate manufacturers, as all raw material people, are going to attempt to provide a versatile material to fit your various needs. I think then each of you, knowing the facts and being in an informed position, can arrive at the best decision and the proper decision for each of your own particular situations.

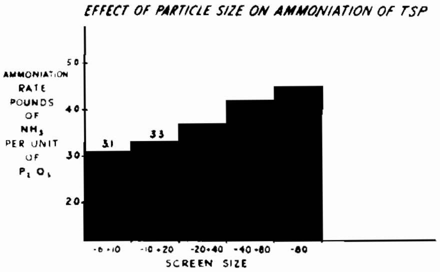
MR. NIELSSON: Thank you, Joe. We now have Mr. C. E. Franklin of International Minerals & Chemical Corporation:

Ammoniation Characteristics of Ammonium Superphosphate

C. E. Franklin

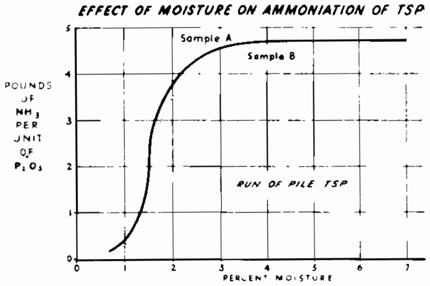
FIRST, I would like to review some of the more important factors, which influence the ammoniation properties of triple superphosphate. These are particle size, porosity, moisture content, temperature at which the reaction takes place and time of reaction.

Particle size of your phosphate material is a very important factor in its ammoniation properties. This is due to the fact that particle size is related to surface area. The smaller the particle the larger or more surface the phosphate particle will have per pound of material. And since ammoniation is essentially a surface reaction, the more surface area that is exposed the higher the ammoniation rate will be in that the ammonia can reach more of the phosphate material. To demonstrate this effect of particle size let us look at this slide which shows the results of the ammoniation of different sizes of triple superphosphate. Here a random sample of ROP triple was obtained. The material was then screened into the different size fractions indicated. Each size was ammoniated in a small laboratory-sized closed ammoniating drum with an excess amount of ammonia. The minus 6 plus 10 mesh material absorbed 3.1 pounds of ammonia per unit of P_2O_5. Each succeeding screen size ammoniated had a higher ammonia absorption rate. The smallest size fraction, or minus 80 mesh material, absorbed 4.3 pounds of nearly a 40% increase in the ammonia absorption capacity. Incidentally, the moisture content of each of these sizes was essentially the same, four per cent.



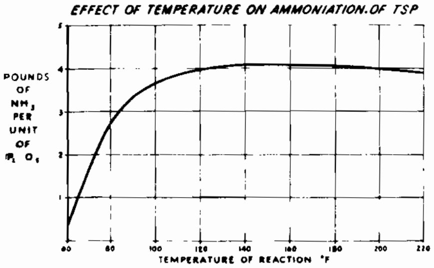
The second and one of the most important factors which influences the phosphates ammoniation properties is that of porosity. Porosity by definition is the ratio of the empty volume of a material to the mass or a measurement of the porosness of the material. We often speak of phosphates having a honey-comb or sponge-like structure. That is each particle has many small holes or void spaces within it. This in turn increases the surface area of each particle by increasing the depth in which the ammonia can penetrate, giving more area in which the ammonia may react. Therefore, the greater the porosity of the phosphates the higher the ammoniation rate. The degree of porosity of a phosphate material is a result of its method of its manufacture. This is one variable the ultimate user has little or no control over.

The third factor effecting ammoniation is the moisture content. This slide shows the result of data on the effect of moisture content on two different samples of ROP triple. You will note here that the essentially dry triple, 1 to 2% moisture, ammoniates very poorly while above 3% moisture there is not noticeable increase in ammoniation rate. There appears to be a



definite critical moisture content between 1 and 2% where ammoniation starts. The ammoniation rate increases until 3% moisture is reached then appears to level out. Therefore, from the results of this page for good ammoniation characteristics your triple super phosphate should have a minimum of 3% moisture. Moisture must be present to initiate the reaction between ammonia and the phosphate material.

The temperature is the fourth factor to be considered, that is the temperature at which the ammoniation reaction is carried out. This slide shows graphically how controlling the temperature effects the ammoniation rate of triple superphosphate. For this particular laboratory test the small ammoniation drum was put in a container of ice to help control the ammoniation temperatures. You can see at temperatures below 100 degrees F. ammoniation rates were low. The maximum ammoniation rate appeared to be reached between 120 and 130 degrees F. For most all grades this will not be an important factor since ammoniators usually operate at a temperature much above 120 degrees F. Note on this slide the slight decrease in the ammoniation rate as the temperature exceeds 200 degrees F. No data was collected at temperatures beyond 220 degrees F. However, in extrapolating this curve it would give the indication that a further decrease in the ammoniation rate would take place with increasing temperatures. This may be caused by the increased vapor pressure of ammonia at these higher temperatures or possibly the incomplete reaction between monoammonium phosphate and ammonia to form diammonium phosphate.



Now the last factor to be considered is that of time. We know that every chemical reaction requires time and especially that of ammoniating phosphates since the ammoniation process is essentially a series of stepwise reactions. We like to recommend that solution or ammonia be added to the phosphate material over a minimum of 2 to 2.5 minute period. In continuous processes where the ammoniated material goes from the ammoniator to either a dryer or cooler drum and a blast of air is blown through the fertilizer material, a four minute residence time in the ammoniator or granulator is recommended before going into the dryer or cooler drum.

Now that we have reviewed the factors which influence the ammoniation properties of triple superphosphate, let us now consider factors which enter into commercial production or why it is possible to ammoniate the same triple at a 3.8 pound rate in one unit and maybe only a 3.4 pound in another.

Let's first consider overglomeration. In the factors which effect ammoniation we pointed out two major items, particle size and surface area. These two items were important because we want the maximum surface area practical for our triple. However, getting a material with a good particle size and maximum porosity is not going to do us one bit of good if we immediately roll this material up into large granules in such a way that the ammonia being added either in the form of anhydrous or nitrogen solution cannot reach the surface of the triple superphosphate particles. This is particularly true when we are working with formulations which have a high liquid phase which may result in excessive agglomeration.

In Mr. Hignett's report on *BETTER NITROGEN RECOVERY* the results of tests or ammonia losses in the manufacture of 12-12-12 were given. This report stated that when granulation was well controlled ammonia losses were low, in the range of one per cent. An overall ammoniation rate for both the superphosphate and triple of 5.5 pounds of ammonia per unit of P_2O_5 were achieved. However, when overgranulation occurred ammonia losses were 6 per cent giving an overall ammoniation rate of only 3.9 pounds per unit of P_2O_5.

Results of this nature point out that we should take steps to keep overgranulation from taking place in the ammoniator. This may be accomplished by adjusting or correcting formulations to keep the liquid phase from becoming too high in the ammoniator. When economically feasible recycle will aid in reducing overgranulation. Good even distribution throughout your ammoniator for all your liquid materials is always important for good granulation.

The slide showing the effect of temperature on ammoniation indicated a decrease in ammoniation absorption as the temperature in the ammoniator increased from 200 to 220°F. This decrease in ammoniation efficiency should be remembered and taken into consideration if using formulas which give excessively high bed temperatures.

High temperatures also have the tendency to increase the plasticity of the fertilizer. This can result in the decrease of the available surface area of the phosphate material thereby decreasing the ammoniation efficiency. Also if the sulfuric acid and solution spargers are located too close together localized over heating may result reducing the ammoniation efficiency.

Another operating factor which effects ammoniation efficiency is the speed on RPM of the mixer. Bill Harwood has demonstrated using his laboratory ammoniator that increasing the speed or peripheral velocity of the ammoniator decreases the effective density of the rolling bed. The less dense the bed the faster the ammonia vapors can escape through to the top of the bed without reacting resulting in ammonia losses.

In conclusion remember that the phosphates physical characteristics, particle size, porosity, and moisture content, are not the only controlling factors on the ammoniation rates. Operating conditions, degree of liquid phase, time, temperature, location and condition of spargers, and rate of rotation of the mixer, all play a very important part in determining the ammoniating efficiency of the phosphate material.

MR. NIELSON: Thanks Charlie. I would just like to have you men remember that when you see these curves that are put on the charts that these are theoretical curves, because if I put in 6 pounds and only hold 3, I have shown on the curve 3 pounds. I do not know what would happen if I put in 3. So, just use these as guides, because I am in the Plant Food Division of IMC and I am listening to material experts right now, and so far I still want to find out something. So, Dan Walstad, will you come up and help me out.

Granulation of Mixed Fertilizers Containing Triple Superphosphate

Dan Walstad

MUCH of the difficulty in granulation of high analyses grades containing triple superphosphate stems from the fineness and porosity of the material. Granulation characteristics of triple superphosphate are poor if the particles are both fine and porous.

As described very clearly by Newitt and Papadopoulos (4) granulation depends on wetting the surface of each particle with liquid phase. When the particles of triple superphosphate are fine and porous, a relatively large amount of liquid phase is needed. Attainment of the required amount of liquid phase, by increasing heat or soluble salt content of the mixture, is sometimes difficult to accomplish, and invariably increases cost of production.

Yet these two properties of triple superphosphate—fineness and porosity—are needed if the triple superphosphate is to have a high capacity for ammonia. The relationship between ammoniation capacity and fineness and porosity has been reported by Kumagai, Rapp, and Hardesty (3) and in previous meetings of this group. Raw material costs are lowered in proportion to the amount of low-cost ammonia (as anhydrous or in nitrogen solutions) that can be reacted with triple superphosphate.

Because of the properties of triple superphosphate that permit high ammoniation rates are exactly opposite those which aid granulation, the particle size and particle structure of triple superphosphate represents something of a compromise. Generally these properties favor a high ammoniation capacity. There is a good reason for this. Poor granulation characteristics can be compensated for completely by either proper formulation or by the design and operation of granulation equipment; but no methods exist for off-setting a low ammoniation capacity.

What are the methods for successfully granulating a formula that contains a high proportion of fine, porous triple superphosphate? If we bear in mind that the controlling factor is liquid phase, we can either take steps to insure maximum utilization of existing liquid phase, or change the formula to increase the amount of liquid phase.

One way to accomplish better utilization of liquid phase is to provide more uniform distribution of liquids in the TVA ammoniator. More uniform distribution will reduce the amount of liquid phase that is wasted in the production of oversize material. The factors that affect distribution, (1) sparger placement, (2) sparger design, (3) speed of rotation of drum and (4) length of drum need to be carefully reviewed by the manufacturer.

We believe that spargers should be placed as close to within 3 to 4 inches of the shell of the drum as practical where the particles are moving most rapidly. Spargers and their supporting structures should be designed to offer minimum obstruction to the flow of particles. Buildup on a sparger support, which affects distribution adversely, can be minimized by placing the supports outside of the area of maximum plasticity. The pattern of holes in the sparger may need to be revised to give more uniform distribution of liquids.

Speed of rotation of the drum should be as high as practical. Brooks (1), who investigated this factor quite thoroughly, recommends 50% of critical speed for a unit in which the spargers are placed above the bed. When the spargers are submerged we recommend 40% of critical speed. For a drum 7 feet in diameter, this speed would be 11½ rpm. Longer drums permit longer spargers which provide more uniform distribution of liquids into the solids. Many drums which are now divided into an ammoniation section and a granulation section would do a better job of granulation, if the ammoniation sections were extended and the granulation section eliminated.

Existing liquid phase may be utilized more effectively if loss of heat from the ammoniator is reduced by keeping the air flow through the ammoniator at a minimum. Another method of limited effectiveness is to use potash of a larger particle size.

Often, because of equipment limitations changes as outlined above cannot be made. Then changing the formula to increase the amount of liquid phase in the mixture is an effective way to improve granulation. This can be done by adding more sulfuric acid to increase heat; or by obtaining more nitrogen from nitrogen solutions and less from anhydrous ammonia, to increase the soluble salt content of the liquid phase, However, the increase in liquid phase is invariably accompanied by higher raw material costs, and sometimes by higher drying costs. For this reason every effort should be made to correct equipment deficiencies before changing formulas.

In calculating the above formulas the ammoniation rates and heats of reaction as shown on the next slide ammoniation rates are those recommended by T.V.A. Heats of reaction are derived from data given by Hardesty and Ross (1). It has been our experience that most granulation difficulties generally occur in the manufacture of a 5-20-20 or similar grades. To illustrate various ways of adjusting a formula to increase its liquid phase several formulas for grade

5-20-20 are shown on the first slide. Formulation A in which all the nitrogen is derived from low cost anhydrous ammonia is often used by fertilizer manufacturers. Enough sulfuric acid is added to react with the ammonia not absorbed by the superphosphates. The indicated heat of reaction, 243,000 B.T.U.'s is normally sufficient for good granulation.

The liquid phase might be increased by the addition of sulfuric acid to raise the heat content—up to the maximum shown in formula B. But to do so it would also be necessary to increase the amount of triple superphosphate. In practice, very little improvement in granulation is obtained by this approach.

In formula C the amount of soluble salts has been increased by obtaining one unit of nitrogen from ammonium nitrate and urea, through the use of nitrogen solution 440 (22-66-6). Enough sulfuric acid has been added to react with the ammonia not absorbed by the superphosphates. Although the increase in soluble salts favors an increase in liquid phase, the heat of reaction in formula C (172,000 B.T.U.'s) is insufficient to provide good granulation. The maximum heat that could be supplied (279,000 B.T.U.'s) is indicated in formula D. As this is more heat than desired, the sulfuric acid addition has been adjusted to give a heat content of 238,000 B.T.U.'s, and this formula is shown as E. Formula E, which has about the same heat content as formula A but contains more soluble salts, usually provides the correct amount of liquid phase needed for a good granulation. Some plants with good heat economy are able to granulate a 5-20-20 at indicated heat content of under 200,000 B.T.U.'s.

It was mentioned previously that formula changes to obtain a higher amount of liquid phase result in increased costs. The raw material cost of formula A is $45.14 per ton of product. This figure is increased by $1.42 to $46.56 for formula E. In this particular example drying costs are not increased.

Although a fine, porous triple causes some difficulties in the granulation of low nitrogen formulas, these properties are an advantage in the manufacture of high nitrogen grades. The high ammoniation capacity permits the maximum use of low cost nitrogen solutions. As the fine, porous particles require more liquid phase before they tend to agglomerate a higher amount of liquids (such as nitrogen solutions) can be tolerated before over agglomeration occurs.

Rates and Heats of Ammoniation

Material Reacted With Ammonia	Rate of Ammoniation, Lbs. of NH_3 Per Unit of P_2O_5	Rate of Ammoniation, B. T. U.'s Per Lb. of NH_3
Normal Superphosphate	5.8	1430
Triple Superphosphate	3.8	1540
Phosphoric Acid	7.2[a]	1780
Sulfuric Acid, 66°Be'	0.324	2940

[a] Rate of ammoniation can be increased in the presence of normal superphosphate. As a general rule each unit of P_2O_5 from phosphoric acid that can be paired with 2 units of P_2O_5 from normal superphosphate will react with an additional 2.4 pounds of ammonia.

Formulation of Grade 5-20-20

Moisture	Analysis, % Material	Analysis, % Plant Food	Formula, Pounds A	B	C	D	E
Anhydrous Ammonia	—	82.3% N	125	125	84	84	84
Nitrogen Solution[a]	6.0	44.0% N	—	—	77	77	77
Normal Superphosphate	7.0	20.0% P_2O_5	439	25	514	98	262
Triple Superphosphate	5.0	46.0% P_2O_5	696	876	663	844	773
Muriate of Potash	—	61.0% K_2O	656	656	656	656	656
Sulfuric Acid[b]	6.8	—	118	348	43	275	182
TOTAL			2034	2030	2037	2034	2034
Ammonia Supplied, Pounds			125	125	101	101	101
Ammonia Capacity, Pounds			125	189	102	169	143
Heat of Reaction, 1,000 B.T.U.			243	350	172	279	238

[a] Solution 440 (22-66-6)
[b] 66°Be' H_2SO_4

References

1. Brook, A. T., "Developments in Granulation Techniques," Proc. Fertilizer Society (London) *47*, 63pp. (November 1957).
2. Hardesty, J. O., and Ross, W. H., "Heat Developed in the Ammoniation of Superphosphates with Anhydrous Ammonia," Ind. Eng. Chem., 29, 1283-90 (November 1937).
3. Kumagai, Rikio, Rapp, H. F., and Hardesty, J. O., "Physical Factors Influencing Ammonia Absorption by Superphosphates," Agr. Food Chem., *2*, 25-30 (January, 1954).
4. Newitt, D. M., and Papadopoulos, A. L., "The Mechanism of Granule Formation," Proc. Fertilizer Society (London) *55*, 35 pp. (February, 1959).

MR. NIELSSON: Thank you, Dan.

Would you gentlemen gather at the round table?

For you newcomers here, Dr. Sauchelli always wanted this to be a very informal discussion. Initially everybody got around the table and yak-yaked their problems back and forth, and now that this doggone thing has gotten so big you just cannot do that. To carry out the spirit of the meeting Dr. Sauchelli always wanted to have, the experts will be down there to still give this feeling of informality.

We have gone through some triple superphosphate discussions. I imagine most of you have gone through the same thing that I went through. Phos acid came along, we got awfully excited, ammonia recoveries were way up, formulation costs were down $2 and $3 a ton, everything looked rosy. All of a sudden the field came back and said, this stuff makes one big puddle in the bag.

Since that time we have found out that if you go overboard on phos acid you might be in trouble and that backtracking a little bit and going back to some triple and formulas that used to be all phos acid will give you a better condition. I even found one manufacturer who makes 3-12-12 by putting in 50 pounds of triple. It sounds a little screwy at first, but he makes awfully good looking 3-12-12 and without the 50 pounds of triple he can't do it.

So triple is quite versatile and if any of you down there now have some working problems, we will endeavor to have our experts give you the answers to them.

Dr. Hignett.

Dr. T. P. Hignett (Tennessee Valley Authority): I should like to ask a question about the effect of moisture content on triple super. Two of the speakers have emphasized that the ammoniation rate depends on the moisture content. They have cited experiments in which presumably the moisture supplied by the triple was the only source of moisture, whereas in another case some moisture was brought in by the solution. However, in granulation processes very often moisture is added as wanted to assist in granulation. I should like to ask if any experiments have been carried out in which the moisture contents of mix during ammoniation are kept constant while the additional moisture content in the triple super per se and additional moisture supplied as required by spraying on the surface of the bed?

Mr. Nielsson: Dr. Hignett wants to know if anybody has had any experiments where moisture was a variable?

Mr. Franklin.

Mr. Franklin: First, I'll say no, I know of none that have been conducted in that manner.

Mr. Markey: I'm afraid I also must answer no. I do not know of any such experiments.

Mr. Nielsson: I guess then that the same thing that bothers Dr. Hignett bothers me and that is you have granular triple and it ammoniates at 1½ pounds then what happens after 2 pounds at 2 per cent moisture? You ammoniate another pile and it ammoniates 6 pounds and it has 4 per cent moisture. Therefore, you have to have 4 per cent moisture to get good ammoniation. He is probably wondering, is that the philosophy by which you arrive at your data, or has anyone actually taken moisture as a variable?

Mr. Franklin: Probably what we are facing here is that you have taken the first step in the answer to that question but we still have more work to do.

Mr. Nielsson: Are there any more questions?

Mr. Donald De Lapp (American Cyanamid Company): I would like to speak to Mr. Hignett's last question. We have run experiments at the Stanford Laboratory ammoniating under conditions where good granulation will occur in a laboratory scale granulator and it makes very little difference to the rate of ammonia absorption. In other words, the per cent efficiency, whether the original moisture in the triple is 3 per cent, or 8 per cent, it is about constant within the experiment average.

Mr. Nielsson: I hope everybody heard that. It makes no difference whether it is 3 or 8.

Are there any other questions.

Mr. William J. Brown: (Elecric, Reduction Company, Ltd.): Perhaps I am adding a new factor to this ammoniation end of the business, but have any of you gentlemen had any experience as far as ammoniation rate is concerned with the pan granulators as compared with TVA type granulators?

Mr. Walstad: I'll be the first to answer no to that question.

Mr. Nielsson: Pan granulators. There are only two people who can talk expertly about pan granulators, and then our pal, Gus Mautner, is the other expert and I don't think he would care to talk about pan granulators at this time.

Mr. Mautner: I didn't hear the question.

Mr. Nielsson: Gus, the Reduction man from Canada would like to know do you have any data on the effect of triple variables on ammoniation in the pan granulator; that is, particle size, moisture, degree of porosity?

Mr. William G. Mautner: (Baugh & Sons Company): We do not have anything on what you are talking about—but in the first place the ammoniation rate of a pan is not anywhere near the same degree as you could make in a TVA ammoniator.

There is one thing, though which I will mention in passing, that you can granulate low nitrogen grades at temperatures under 100 degrees and make a product that will run 95 per cent on size with no fines removed which you cannot do in a TVA ammoniator on a 5-10-10. That's the only advantage it has, plus headaches.

Mr. Nielsson: Are there any other questions?

(No response.)

Moderator Reynolds: Thank you very much.

We are going to move along here and turn the chair over to our good friend and colleague Mr. Al Spillman who will take care of the introduction of later speakers.

Moderator Spillman: Gentlemen, welcome to our Tenth Annual Round Table Meeting. We are delighted to have this large audience and hope that each of you enjoy your stay here.

Our program continues to carry out discussions on the suggestions and topics submitted by the membership. Finalization of the program is the result of many conferences by your Executive Committee, a great deal of correspondence and by personal contacts with many of our members.

This morning we had the pleasure of listening to a tape recording by Dr. Vincent Sauchelli, father of our Round Table, wishing us a successful meeting. Vince, we all thank you for your kind expressions and we are looking forward to have you back with us for our 1961 session.

Our Discussion Leaders are capable and experienced Operators, Technicians, Researchers, Chemists and Engineers. They represent Fertilizer Manufacturers operating all types of fertilizer facilities, Suppliers of raw materials, Personnel from the TVA and USDA-Beltsville. All of the subjects to be discussed during this meeting are timely and of equal importance to the mixer and to the Raw Materials Suppliers. During the past decade good progress has been made in all phases of fertilizer manufacturing technology. Refinements of processes, formulation and equipment have been progressively improved. We undoubtedly in the near future can look for more rapid improved technology changes in operations formulations and for more improved chemical and physical raw materials.

Tonnages of high analysis fertilizers are increasing. The demand for granular fertilizer is more each year. XoX grades are up in tonnage. High nitrogen 3-1-1 ratios and 2-1-1 ratios are now moving in substantial tonnages. The use in mixtures for triple superphosphate and phosphoric acid is tremendously on the increase. Ammonium phosphates are becoming important raw materials in mixtures for conventional, semi-granular, granular and for blending. We need more research and more breakthrough into the sciences for the reasons of adverse behaviors in formulating these high analysis materials. With optimism, enthusiasm, continuous study, experimentation and cooperation by all of us, we are sure to move forward to produce better quality fertilizers and more efficient operations.

Raw Material Suppliers, realizing the tremendous change in fertilizer technology during the past decade, have organized at great expense technical services to help the user-mixer. A good many of these service people are present at this meeting. The technical people at TVA and Beltsville are also available to us. We should use these services to the utmost.

Ammonium phosphate production seems to be on the increase. TVA has done considerable pilot plant testing on ammonium phosphate manufacturing in conventional equipment. We are honored this afternoon to have Mr. A. B. Phillips discuss ammonium phosphate experiments at T.V.A.

Talk on Diammonium Phosphate

A. B. Phillips

JOE Reynolds has asked me to tell you this afternoon about some pilot-plant work we have been doing lately at TVA on the production of diammonium phosphate from wet-process phosphoric acid and ammonia.

We started this work for several reasons.

First, we already had quite a stake in diammonium phosphate. We make it in one of our demonstration plants from electric-furnace acid by a crystallization method and have promoted its use as a high-analysis fertilizer. We piloted a process several years ago for making it from wet-process acid by vacuum crystallization, but this process has not been adopted by industry.

Second, some general studies of preneutralization, that will be described by Mr. Young later in this meeting, indicated that preneutralization might be a means to a simpler way of making diammonium phosphate.

Third, because of the recent surge of interest in diammonium phosphate in the industry, we rereceived many inquiries on how diammonium phosphate, or grades rich in diammonium phosphate, could be made in the TVA continuous ammoniator.

In our pilot-plant study, we have made several different grades of high diammonium phosphate content, but we have concentrated mainly on diammonium phosphate itself. The grades of diammonium phosphate produced ranged from 21-53-0 to 18-46-0, depending on the amount of impurities in the feed acid. The higher grade was made with electric-furnace acid.

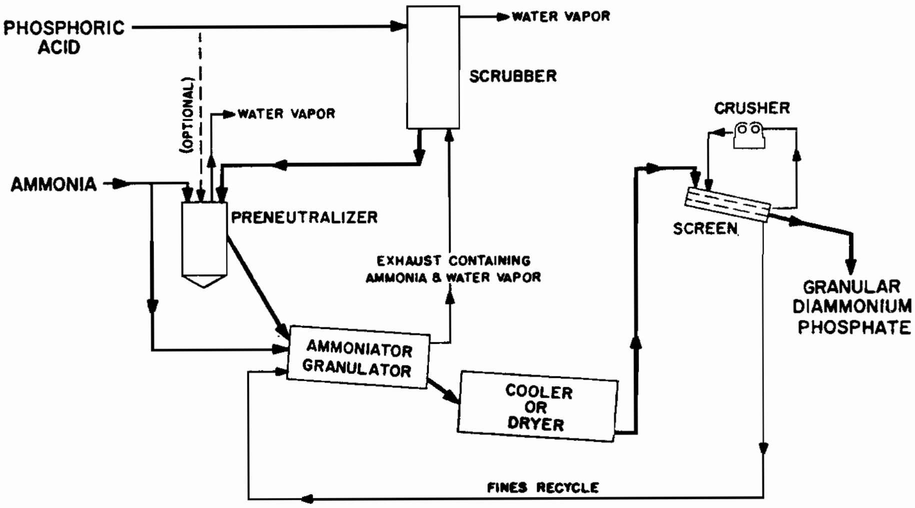
Also, we have studied the production of 16-48-0 which is a mixture of roughly two-thirds diammonium phosphate and one-third monoammonium phosphate. In a few runs, we have added potash to make a 14-35-14 grade, and in others we have produced 18-18-18.

In describing the process, I will first run through the flow diagram quickly and then talk about individual pieces of equipment. May I have the first slide, please?

You can see that we have a standard continuous ammoniation plant with two major additions—a preneutralization tank feeding the ammoniator, and a scrubber to recover ammonia from the ammoniator exhaust.

In our preferred method of operation, all of the phosphoric acid is fed to the scrubber. The effluent from the scrubber is pumped to the preneutralizer where it is partially neutralized with ammona. The slurry from the preneutralizer — a mixture of monoammonium and diammonium phosphate—flows to the ammoniator-granulator where it is distributed on top of a bed of recycled fines. Ammonia is added underneath the bed to complete the neutralization to diammonium phosphate. This takes some excess ammonia which is recovered in the scrubber by the incoming acid.

The product from the ammoniator is dried mildly in a rotary dryer and screened. The over-



size is crushed in a chain mill and rescreened. The undersize is returned to the ammoniator.

While describing the process in more detail, I will point out the operating conditions used in a recent run making 18-46-0. The production rate of 0.5 ton per hour. The acid used contained 46 per cent P_2O_5, 1.4 per cent Al_2O_3, 1.1 per cent Fe_2O_3, and 0.8 per cent CaO. The SO_3 was 4.9 per cent.

Preneutralizer

The preneutralizer is a stainless steel tank 14 inches in diameter and 3½ feet high. It contains a turbine-type agitator and is fitted with a hood and exhaust stack to remove water vapor. Both the acid effluent from the scrubber and the ammonia are fed through circles of perforated ¼-inch pipe near the bottom. The slurry overflows from the tank at the 2-foot level.

There are several important points about the preneutralizer operation I would like to emphasize.

1. The ammonia rate should be controlled to maintain the mole ratio of $NH_3:H_3PO_4$ at about 1.3. This is important because the ammonia loss becomes excessive at higher mole ratios. Operating much below a mole ratio of 1.3 makes the slurry thicker and more difficult to handle. The pH of the slurry is about 5.0 when the mole ratio is 1.3.
2. The slurry leaving the preneutralizer should contain 15 to 25 per cent water. This will result when acid containing not more than about 40 per cent P_2O_5 is fed to the process, or enough water is added to dilute the acid to this concentration. Evaporation of water in the preneutralizer controls the temperature at about 235° to 250° F. When liquid anhydrous ammonia is fed to the preneutralizer, we find it best to add a small amount of water with the ammonia, otherwise the slurry in the bottom of the tank will solidify.
3. The slurry is allowed to flow to the ammoniator by gravity to avoid difficulties that would surely arise if it were pumped. Surging of the slurry is a minor problem that probably would merit some attention in the design of a large unit.

Ammoniator

The piolt-plant ammoniator is a 3- by 3-foot drum rotated at 20 revolutions per minute. The slurry from the preneutralizer is distributed above the bed through a horizontal pipe with a saw-toothed slot. It is necessary to rod or steam this distributor occasionally to maintain uniform distribution. Anhydrous ammonia is fed under the bed through a slotted distributor, the type we normally use for ammoniating superphosphate. An excess of about 5 per cent of ammonia must be fed in order to ammoniate the slurry to diammonium phosphate. This excess ammonia is recovered in the scrubber. The temperature in the ammoniator is about 195° F. and a considerable amount of water is evaporated. It is necessary to use a high air flow through the ammoniator to remove this water vapor and prevent its condensation in the scrubber. We estimate about 600 cubic feet per minute for each ton per hour of product should be used. This is considerably higher than the air rates normally used in continuous ammoniators.

Granulation is controlled primarily by recycle. The minimum recycle rate used in the pilot plant was about 2:1 and ranged up to about 3:1 when acid containing about 40 per cent P_2O_5 was used. The recycle requirement increases sharply when the acid concentration is decreased, 5:1 with 35 per cent acid and 8.5:1 with 32 per cent acid.

The recycle rate necessary can be minimized by (1) maintaining a high $NH_3:H_3PO_4$ mole ratio, (2) keeping granulation on the fine side to assure that the recycle will be of small particle size, and (3) using warm rather than cooled recycle, to assure a high evaporation rate in the drum.

Scrubber

The pilot-plant scrubber is a Type 316 stainless steel tower 2 feet in diameter by 10 feet high containing 8 feet of wood grid packing. The gas rate to the tower from the ammoniator is about 350 cubic feet per minute and the temperature is 120° F. The acid needed in the process is added to the top of the tower along with water, if needed, to reduce its P_2O_5 content to about 40 per cent. Also, effluent from the tower is recirculated at about 10 gallons per minute to keep the packing wet.

Under these conditions, the water content of the effluent from the scrubber is about 35 per cent and the pH is less than 1. It can be readily pumped to the preneutralizer. The ammonia loss from the scrubber is usually less than 0.5 per cent of the total fed to the process.

Drying and Screening

The pilot-plant product is dried rather mildly in a 3- by 24-

Typical Data From Diammonium Phosphate Pilot Plant

Feed Rate, lb./ton	
Phosphoric Acid (46% P_2O_5)	2050
Water	395
Ammonia. To Preneutralizer	281
To Ammoniator	202
Preneutralizer	
$NH_3:H_3PO_4$ Mole Ratio	1.29
Slurry Temperature, °F.	237
Slurry Moisture, %	21
NH_3 Loss, % of Total Feed	2.5
Ammoniator	
$NH_3:H_3PO_4$ Mole Ratio	2.00
Temperature, °F.	188
Recycle Ratio, lb./lb. Product	2.2
NH_3 Evolved, % of Total Feed	5.2
Product Analysis	
N	18.5
Available P_2O_5	47.2
Moisture	1.7
Overall- NH_3 loss, % (including dryer)	6.1

foot countercurrent dryer. The dryer product temperature is maintained at 180° to 210° F. Loss of ammonia from the dryer has varied from 2 to 4 per cent and increased as the dryer temperature was increased. The product is screened on double-deck screens at 6 by 10 mesh. The screening of this material has given no dofficulty.

Some typical data from a pilot-plant run are shown in the next slide.

The pilot-plant products had good physical properties. After 3 months' storage in two-ply asphalt laminated bags, those dried to less than 1 per cent moisture had no lumps after one drop of the bag even unconditioned. When dried between 1 and 1.6 per cent moisture, they were fairly good without conditioner and excellent when conditioned with 2 per cent calcined dolomite or clay. Undried material with 2.2 per cent moisture was satisfactory when conditioned.

Other Grades

As I mentioned earlier, we made a few runs to study the production of 16-48-0. The main difference in operating technique was the reduction of the amount of ammonia fed to the ammoniator to give the lower mole ratio. Operation of the ammoniator was much poorer than when producing 18-46-0. The recvcle ratio required to control granulation was about twice as high. (5:1 vs. 2.2:1). The material in the ammoniator had a greater tendency to form balls and to stick to the equipment. We believe this was due primarily to the fact that mixtures of mono- and diammonium phosphate have a greater solubility than diammonium phosphate alone, so there was more liquid phase in the ammoniator.

In another run we made a 14-35-14 grade merely by adding potassium chloride to the ammoniator while operating under the same conditions used to make diammonium phosphate. This worked quite well. The only change was a reduction in the recycle rate to about 1.5:1.

We tried one other variation of the process. This was the production of the flat grade 18-18-18. For this operation we used ammoniating solution 448 to supply all 18 units of the nitrogen. About 75 per cent of the solution was added to the preneutralizer. The balance was added to the ammoniator. The formulation called for some sulfuric acid to fix a part of the free ammonia in the solution. This was added to the preneutralizer. Otherwise the operation was the same as was used in making diammonium phosphate.

The recycle ratio required to control granulation was about 1.7 to 1. Ammonia loss from the preneutralizer was about 2 per cent and only 1.8 per cent of the ammonia was evolved in the ammoniator. The actual product grade was 18.2-18.6-19.0 with 0.7 per cent moisture.

In conclusion, we believe this process is considerably better than the vacuum crystallization method developed earlier at TVA. It does not require a purification step for the west-process acid. The equipment required is simpler in design and is of a type more familiar to the fertilizer industry. Also, the investment required should be lower. The process requires a relatively low recycle ratio compared to some processes now used to make grades rich in diammonium phosphate, and produces a closely sized product having good physical properties. We think it should merit a place in the industry to supply the increasing demand for diammonium phosphate.

I put a number of copies of an interim report on this work on the table in the back of the room and it contains more detailed data on some earlier runs than I have described here.

If all of those copies are gone, if you will drop me a line in care of TVA at Wilson Dam, I will be glad to send you a copy and, if any of you are sufficiently interested, we would be very glad for you to come and visit us and talk to us about this work and we will give you all of the pilot plant data that we have available on it.

MODERATOR SPILLMAN: Thank you, Mr. Phillips. Questions please.

MR. WALTER R. HORN: I didn't understand whether you used vaporous ammonia or liquid ammonia?

MR. PHILLIPS: In most of the work we used liquid anhydrous ammonia. The question was did we use vapor or liquid ammonia and most of it we have used is liquid ammonia. In some tests we have used vaporized ammonia in the drum. That raises the temperature some and increases the amount of water evaporated somewhat.

A MEMBER: How does the overall ammonia yield compare? Is there any difference from the effect of the vacuum evaporation?

MR. PHILLIPS: I think that the losses are higher in this process than in the vacuum evaporation. I do not recall any figures on it, but I do not think there is much ammonia loss in the crystallization method.

A MEMBER: In your process were the gases from the dryer scrubbed?

MR PHILLIPS: No, they were not.

SAME MEMBER: Why?

MR. PHILLIPS: Well — The amount of ammonia in the gas was not large, the loss from the dryer was not large and it would be a lot of gas to scrub.

SAME MEMBER: I thought the loss in the ammoniator ran around 3 to 4 per cent, and I think you had an overall recovery of about 94 per cent on the ammonia.

MR. PHILLIPS: In that particular run I mentioned it was somewhat higher than normal. I think that in most of the runs the ammonia loss from the dryer would be somewhere around 1½ per cent of the total ammonia fed to the process. Is that about right, R.D.?

MR. R. D. YOUNG (Tennessee Valley Authority): That is right. We feel that you ought to keep the temperature on the dryer as low as 180 degrees F., and you will find it about 1½ per cent, if you go up to 200 it will go up 2½ or 3. But you cannot do a good drying job as low as 180.

A MEMBER: I think you said you used a counter-current dryer. Would you elaborate as to why countercurrent as opposed to cocurrent or were you just set up that way at the time?

DR. PHILLIP: We started in this work that way and it seemed to work out pretty well. R.D., would you have any ideas? I do not know of any particular advantages in this.

It might have some effect on the ammonia loss.

Mr. Young: We have used both countercurrent and cocurrent in different formulations, actually. The only real advantage we have found for countercurrent is where you have a high percentage of ammonium nitrate and have a plasticity factor. I believe the cocurrent probably would be preferred.

Moderator Spillman: How does the handling characteristics of that triple 18 compare with the 18-46? Would you comment again on those moistures? I believe you had 1.7 in the 18-46, and do you consider that adequate? In other words, is that low enough to give a free-flowing product?

Mr. Phillips: I think the 1.7 moisture in the 18-46 you would have to condition the product. Of course, the 18-18-18 you would not expect to have as good physical properties as the diammonium phosphate grade because of the ammonium nitrate in it. What was the moisture in that, the triple 18?

Mr. Phillips: 7 tenths. In regard to that particular one it was 7 tenths. We have not completed our bag storage tests on that yet. It did not look bad after one month, however, after conditioning, but we certainly want to look at it after 3 and 6 months.

Moderator Spillman: Yes, sir.

A Member: If you have a variable ammonia loss, how much ammonia feed—or how do you know how much ammonia feed to add to keep a 2 mole ratio?

Mr. Phillips: You can get these pretty well studied down and know what you are doing. It will not vary too much during a run. Another thing is we just add excess ammonia and it goes that far and that is as far as it is going. The more excess ammonia you add to the ammoniator, the higher the ammonia loss will be. It is more or less of an automatic thing, I think.

Moderator Spillman: Al Henderson.

Mr. Albert E. Henderson (Wilson & Toomer Fertilizer Company): What was the particle size of the recycle, return to the ammoniator?

Mr. Phillips: That was all minus-14, I believe. I don't know whether I have any data in addition to that, except it was all smaller, of course, than the lower screen. It was 10 mesh in this case. In other words, we put all of the crushed material back over the screen.

Moderator Spillman: Thank you, Mr. Phillips.

Moderator Spillman: During the past several years at our Round Table we have had discussions touching briefly on the manufactur of nitric phosphates. We are happy to have with us today Mr. Clem Giles representing the Ortho Division, California Spray Company, California Chemical Company, Richmond, California, to discuss the California Chemical Company nitric phosphate and spheroidizer process. Mr. Clem Giles.

Mr. Chairman, members of the Fertilizer Round Table. With all the experience that is represented by this distinguished gathering I must confess I feel at a great disadvantage standing up here. This is the first time I have had the pleasure of attending the Round Table so I trust you will bear with me.

To some of you, I should probably mention that the name of our company has been changed recently. You probably remember us as Cal Spray. We have now been incorporated in one operating subsidiary of the Standard Oil Company of California under the name of the California Chemical Company. The agricultural chemicals are handled by the Ortho Division and the Industrial Chemicals by the Oronite Division.

Nitric Phosphates and Spheroidizer Process

C. B. Giles

PROBABLY the best way to discuss the nitric phosphate and Spheroidizer process is to trace the development of the manufacture of nitric phosphate complex fertilizer in our company. Our company entered the fertilizer manufacturing business in 1956 with the installation of a complex fertilizer plant at Richmond, California alongside the Richmond Refinery of the Standard Oil Company of California. An ammonia plant and a nitric acid plant were built as part of the refinery complex and ammonia and nitric acid were piped to the fertilizer plant adjacent to the refinery. The major unit in the fertilizer plant was the complex fertilizer plant of the PEC (i.e., Potasse et Engrais Chimiques) process. The PEC process was chosen after conducting extensive agronomical testing in California and other Western states to determine the suitability of using the citrate soluble P_2O_5. These agronomical tests were extremely succesful and it was decided on the basis of these tests to proceed with the construction of the PEC complex plant. The plant consisted of the regular PEC reactor system, which I will describe in somewhat more detail later on, and a PEC drying system which received the slurry from the reactors and with returned fines from the dry system granulated the material in a pugmill to pellet size followed by drying, screening, cooling and coating to the warehouse. The plant had a nominal capacity of 200 t.p.o.d. on 20-20-0 and 300 t.p.o.d. on 14-14-14. Besides the two complexes mentioned, the plant also makes 20-10-0 which is a sulfo-phosphate and 16-16-8. In 1958, when it appeared that the Richmond plant could no longer guaranteed fertilizer to the Northwestern states including Washington and Oregon, it was decided to build another complex plant at Kennewick, Washington. This plant receives ammonia from Phillips Chemical plant and was designed to manufacture its own nitric acid ammonium nitrate and complex. Based on the experience and know-how developed during the Richmond fertilizer plant, it was decided to use the PEC reactor system as installed at Richmond but to modify the drying section and use the Chemical and Industrial Corporation's Spheroidizer process which was being developed at that time. This plant is now on stream and in this discussion I will make particular reference to design concepts, and the design and operating experience we have received with this complex plant at Kennewick and particularly the Spheroidizer. Our company has recently announced the installation of a fertilizer plant at Fort Madison in Iowa and ground has recently been broken for this project and we plan to have the whole plant in operation by the end of 1961. This plant will consist of an ammonia plant, nitric acid plant, ammonium nitrate plant and complex plant.

At this point I think it would be opportune to discuss or rather to describe the PEC reactor system. The system consists of three basic stages: the acidulation stage, the ammoniation stage and probably the third stage could be called the addition of potash, although this is not necessarily part of the PEC system. In the acidulation stage, which can be performed in one, two or three reactors depending on the rate and the retention time required by the rate, the phosphate rock is fed from some form of controlled weighing facility into the reactor and nitric acid is fed into the reactor. All the reactors in the PEC system are the same, which is one of the advantages of the system inasmuch as whenever you want to increase production it is necessary only to add more reactors to the train, since they are all the same.

The nitric acid acidulates the phosphate rock, making the P_2O_5 available in the dicalcium form which is the citrate soluble P_2O_5. Also produced is calcium nitrate, which if left in the product would produce a highly hygroscopic salt which could not be stored in bulk. Therefore, the calcium nitrate has to be either removed or converted, and in the PEC process this calcium nitrate is either converted to dicalcium phosphate by the addition of phosphoric acid or to calcium sulphate by the addition of sulphuric acid or to calcium carbonate by the addition of CO_2. The phosphoric acid or the sulphuric acid is added gradually to each of the remaining reactors together with the ammonia. The chemistry of the reaction is very complex but the end result is a mixture of ammonium nitrate, dicalcium phosphate, some ammonium phosphate and, if sulphuric acid is used, some calcium sulphate.

The reactors are designed to give thorough stirring of the whole mix, whether this be liquid or thick slurry, and therefore to remove nitrous vapor formed during acidulation. There is no risk of the formation of nitrates at a later stage, and local supersaturation during the course of ammoniation is presented. The reactors equipment is arranged on a single floor level and no circulating pumps are required.

It is easy to increase the capacity of the plant. Supplementary reactors may be added either to the end of the reactor line, or to the intermediate section if a U-arrangement is employed. The latter system has the advantage of maintaining the ends of the reactor line unchanged.

The introduction of ammonia is carried out by direct injection into the mix. Careful distribution to the various reactors makes possible a high rate of absorption, easy control, and eliminates local supersaturation.

At Richmond and Kennewick muriate of potash is added at the end of the reactor train. The advantage of the PEC system is that with this one set of pots almost any of the high-analysis complex fertilizers can be produced and in most cases the majority of the P_2O_5 is made available from rock instead of acid and they are produced in a homogeneous slurry without any possibility of the formation of nitrate. In fact, in the Richmond and Kennewick experience, no nitrates have ever been detected in the product. We feel this is due, as PEC states, to the high agitation and consequently large contact obtained during the acidulation and ammoniation. During the four years of operation of the Richmond plant and now lately of the Kennewick plant, we certainly have developed a lot of know-how in how to operate with the PEC reactor system, and as a result we now know its potential and are very pleased with its performance. We have used the PEC reactors for making ammophos of 16-48-0 and 18-46-0 which the process can do very easily, but we realize that one would not want to purchase the complicated and expensive reactor system for manufacturing these, but once you have the PEC reactors installed to produce nitrophos then it is certainly easy and efficient to produce the ammophos with the same system.

The Chemical and Industrial Corporation had been working on the development of a Spheroidizer between the time we built the Richmond plant and when we built the Kennewick plant. They had visualized that a step could be saved if the drying and granulating of complex fertilizer could be handled in one stage. They set up a pilot plant operation in Cincinnati to determine if this was possible, and after experimentation were able to operate the pilot plant satisfactorily and produce the spherical pellets which they had hoped to. The first plant was installed by C&I at Western Phosphates plant in Garfield to produce ammophos fertilizer. The next plant to go into operation was the one at Kennewick, although this was followed very closely by a plant at Cuba. We worked closely with C&I during the design stage of the complex plant at Kennekick and based on the Richmond experience, established design concepts which we felt would improve on our Richmond operation. We wanted to take advantage of the simple drying and granulating step to avoid as much as possible conveyors and elevators within the plant. In so doing we were able to cut down the size of the complex plant for producing basically the same rate from a plant approximately 150 feet by 150 feet to one 80 feet by 80 feet. We were also able to cut down the number of elevators and conveyors and reduce very appreciably the amount of fines recycle required. In so doing, we were able to eliminate the large crushers required at Richmond to produce the fines required for granulation.

Since it was a new process, naturally we were confronted during the design stage with several design decisions which could only be proven in practice. We did feel that it would be necessary to have a fines bin to control the fines feed to the Spheroidizer. We also took a calculated risk that we would not need elaborate of expensive pumps to pump the slurry. Basically, the process consists of the PEC reactors producing slurry which drops into a tank after having had potash added to it in a mixer at the end of the reactor train. The slurry is then pumped from this tank, which is maintained agitated, to the nozzles of the Spheroidizer and sprayed onto a dense curtain of falling recycled pellets. The Spheroidizing is performed at the first section of the drum and the majority of the drum is used for diffusion drying. The outlet from the Spheroidizer is then take by an elevator to a screen where the oversize is returned to a crusher in circut with the Spheroidizer, the product is coated and taken to the warehouse and the fines also fed back to the Spheroidizer. As I mentioned previously, the fines bin was considered necessary during the design stage but after operation it was felt that this fines bin could very well be bypassed, and as a result the operation simplified. This is how we are operating the Kennewick plant.

As would be expected the secret of success with the Spheriodizer is the design of the spraying system. During the plant startup, all nozzles and spray methods which showed any success in pilot plant were tried. As a result of the experimentation, a spray system has been developed which is easy to control and with which we can control the particle size without fouling the drum.

We have centralized all operating controls for the Spheriodizer at the Spheriodizer operating platform. This includes control of the spraying system, control of the furnace air temperature and amount of air and control of recycle. The operating controls for the reactor are located at the base of the Spheriodizer platform; also centralized at this location for ease of control.

(At this point a simple process flow diagram was drawn and explained by using the blackboard.)

The Kennewick Spheroidizer has met its design requirements in every way and during this past summer we have made other products than these for which it was designed. It was designed for 200 t.p.o.d. 20-20-0 and 20-10-0 and 300 t.p.o.d. 14-14-14. We have also made 16-48-0 and 18-46-0.

One real manufacturing advantage is the ease that products can be changed. This change can be made in as little as 8 hours between being on specification with one product to changing to another. Of course, going from nitrophos to ammophos takes longer.

Finally, we can say Spheroidizing together with the PEC process produces hard spherical pellets of consistently homogeneous analysis with minimum losses of raw material and under easily controllable conditions.

Since this is a new process, I took the liberty of bringing a few samples to pass around. I would draw your attention to the structural strength of the pellets, I have 16-48-0-, 20-20-0 and triple 14.

I also have here with me Dr. Makower in charge of our fertilizer research, the one who has been asking all the questions, and maybe he can help me answer a few now.

Albert Spillman: Thank you very much Mr. Giles for a splendid description of your processes. Question please.

Mr. Walter Horn: Mr. Giles, I was wondering what the fineness of rock grind was and also at the beginning of your digestion ammoniation circuit what is your percent of solids and slurry?

Mr. Giles: I think Dr. Makower can answer that better than I. As regards the phosphate rock, we use about 80 per cent through 200 mesh; that's pretty fine pulverized.

Dr. Ben Makower: In regard to the question you asked regarding the amount of solids in the slurry. I will say that at the end of the ammoniation chain we run something of the order of 20 to 25 per cent water. In the acidulation reactor, of course, you have much higher water content because we are feeding about 57 per cent nitric acid, which is 43 per cent water. The rock, of course, dissolves the undissolved material. But your primary interest is probably at the end of this acidulation and ammoniation chain, that is the order of magnitude. In some cases, we may find it necessary to add additional water but it is usually a very small amount to obtain good granulation or spheroidizing action, as we call it.

Albert Spillman: Mr. Giles, do you produce phosphoric acid at Richmond and Kennewick?

Mr. Giles: No, we don't.

Albert Spillman: Pardon me. You spoke of removing calcium from your process, at which point in the process do you remove the calcium?

Mr. Giles: No, I did not say we removed the calcium; I said the product of acidulation is calcium nitrate and we convert that replacing the calcium nitrate with dicalcium phosphate, we use phosphoric acid.

The basic PEC process, the one they are very proud of, is by injecting CO_2 which you get as an off-gas from an ammonia plant, then you convert the calcium nitrate to calcium carbonate.

It is possible to remove the calcium nitrate, and some of the European plants do that. They take the products of acidulation and remove the calcium nitrate and then put back in what is left and as a result you can get a much higher analysis. It means the addition of more phosphoric acid, but I think it is possible to get an 18-18-18 by doing it that way.

Albert Spillman: Is that acidulation a batch process?

Mr. Giles: Oh, not; it's all continuous.

Albert Spillman: Everything is continuous?

Mr. Gile: Everything is continuous.

Albert Spillman: Are the nitric acid and the phosphoric and the ammonia added at the same time?

Mr. Giles: The nitric acid—well, they are all being added at the same time. The nitric acid is added in the first reactor and the ammonia is added in the ammoniator reactors, but it's a continuous process.

We are thus provided with excellent chemical control because, by taking samples of the slurry, the wet slurry entering into the tank, we know what the product is going to be, and by varying the addition of phosphoric acid which is controlled with a magnetic flow meter, the addition of nitric acid is controlled and measured with a rotometer, the ammonia is controlled so that by a simple adjustment in the control room the whole operation can be maintained in very close chemical control. It does not require too much of the operator's time, either.

MR. HORN: What are your recycle rates, please?

MR. GILES: The plant was designed for a 1 to 1 recycle rate and in recent acceptance tests I think we probably get 1 to 1 but the next plant will be designed for a higher recycle rate, maybe about 2 to 1.

MR. THOMAS PEARCE (Swift & Company): Do you have any particular method of injecting or in the design of your reactor system and therefore the ammonia and the nitric acid—I mean, you seem to be rather proud of the reactor system.

MR. GILES: I am proud of them, but this is essentially the property of PEC and I just do not feel at liberty to disclose this. They consider this to be their knowhow of how they do inject, how they sparge and how they agitate, so I would prefer not to get into the details of the reactors.

A MEMBER: Have you had any foaming problem there in your nitric acid acidulation with that rather fine rock.

MR. GILES: No foaming problem. This is again one of the boasts of PEC, that there is no foaming problem and this is a fact.

A MEMBER: In your 1-1-1 ratio product, what portion of the phosphate is dicalcium and what part the plain phosphate, roughly?

MR. GILES: Dr. Makower can answer that better than I.

DR. MAKOWER: In the 1-1-1 combination, or perhaps I had better express it differently and say that something of the order of 10 to 15 per cent of the P_2O_5 is in the water soluble stage and 90 to 85 per cent of the P_2O_5 in the citrate soluble stage. If you want to call the citrate soluble the dicalcium phosphate you may make it that way. The chemical identity of these solids is still sort of a mystery.

MR. HORN: What kind of a coating agent do you use, could you disclose that, and how much?

MR. GILES: The coating agent?

MR. HORN: Yes.

MR. GILES: Again, Dr. Makower.

DR. MAKOWER: I would say triple 14.

MR. GILES: The question is how much conditioning or coating agent we apply?

DR. MAKOWER: We apply something between 1 and 2 per cent of a coating agent.

MODERATOR SPILLMAN: Thank you, Mr. Giles.

MODERATOR SPILLMAN: Our next speaker is your good friend and my good friend Dr. K. D. Jacob from the U.S.D.A., Beltsville, who will give us a summary on phosphates. Dr. Jacob.

Thank you, Al. Ladies and gentlemen: I was asked a few weeks ago to summarize the position on phosphates at the meeting here today. It seems to me that the situation has already been pretty well summarized by the very excellent contribution that you gentlemen have made to the subject today. The field has been pretty widely covered.

I would like to say that I do think that this is a real advance in the relationship of the technical folks in the fertilizer industry to get together at these Round Table Meetings. It was not long ago, as you all very well know, and as was mentioned this morning, that this sort of situation would not have been possible, and I certainly know it would not have been possible when I first became acquainted with fertilizers, which was also a few years ago.

Perhaps a good way to make us appreciate where we are today on phosphate fertilizers is to indicate briefly where we were not a few years ago. I am very sure that John Bennett Laws and Justus Von Liebig would have certainly had their eyes bugged out here today at this meeting if they could have been here and heard of the things that have happened in phosphate fertilizers since they first entered the picture 120 years ago.

I am not quite as old as Laws and Liebig but there have been a lot of things which have happened in phosphates since I first came on the scene. Maybe you would just be interested in knowing briefly what the situation was, let us say, 40 years ago. At that time, throughout the world, the old original product of Laws and Liebig was still almost the sole source of available chemical processed phosphate to agriculture over the world. It only had one serious competitor and that was basic slag in Europe resulting as a byproduct from the processing of steel.

Some 40 years ago almost our entire production of phosphate fertilizer in this country was in one form of superphosphate or another, and almost entirely that form was the old ordinary superphosphate known as superphosphate running at that time 16 to 18 per cent and 18 per cent was a pretty good grade.

We also had a little production of triple superphosphate and in 1920 we had two plants. One of those plants was in Montana and it was owned by a copper mining company at that time and it continued in operation in the production of triple superphosphate until just about a year ago when they switched entirely to ammonium phosphates, as I understand, and discontinued triple. The other plant was started in 1907 and that plant operated, certainly I am sure, for nearly 50 years. It was the old plant of the Virginia-Carolina Corporation at Charleston, South Carolina.

In 1920, we also had one other product, a superphosphate product which some of you fellows, I am sure, are not familiar with. I know some of you in the south, some of the older folks certainly remember it. They still make a little. It was the old wet mix base goods which was a superphosphate made by acidulating phosphate rock and nitrogenous organic matter such as leather scrap and wool waste and hair and all kinds of feathers and nitrogenous material of that kind. If any of you folks have been around a plant where they were making wet mix base and had occasion to stay there a short time—you did not have to stay very long. You will recall that that odor just got permeated through your clothes and you did not get rid of it for quite a long time. I suspect that probably wet mix base maybe was one of the things in fertilizers that prompted the city council of Atlanta, and this must have been about 1908 or 1909—I ran across it a short time ago in an old volume of American Fertilizer—to consider the institution of an ordinance in Atlanta which would prohibit anybody who worked in a fertilizer plant from riding on the streetcars in Atlanta. I do not know whether that ordinance was ever passed; I did not find a record of it having been passed, but it was certainly proposed.

In 1920 or very close to that time we also had the beginning of production of ammonium phosphate in this country. I think maybe there are a few of us who are not aware that ammonium phosphate has been a commercial fertilizer in the field in this country for quite a long time, out of the plant of the American Cyanamid Company at Warners, New Jersey. I am not sure of the time now, when it started, but I believe that plant was in operation in 1920 and it did produce ammonium phosphates up there, 11-48 and 16-20 grades, until about 1940 when that plant was closed down.

In the meantime, we had had essentially no other production of ammonium phosphates in this country and virtually all, or at least a greater part of Cyanamid's production throughout that time was exported. We had not gotten to the point where ammonium phosphate was catching on over here yet.

I might point out too that in the twenties, the late twenties, there was production started on a material which we were inclined to think was a very new material and that was the nitric phosphate product. The initial production was in the United States but the material was produced in Germany and the first production that came over here was not nitric phosphates, but shortly afterward they did begin the production of nitric phosphates and they were imported still under the name of nitric phosphate into the United States for several years.

The original nitric phosphate that came over here had practically no calcium and no sulfur in it. They unfortunately started to institute the use of those materials and tried to get them started in the United States down in the Carolinas, I believe, or down in that area, and they soon got into some very serious trouble where the absence of calcium and sulfur in those materials was a serious matter agronomically, and that caused them to go to the nitric process which did introduce some calcium and they certainly must have used some sulfuric acid along with nitric to get the sulfur.

In the meantime, we had been thinking about and there had been some work and experimental production of phosphorus by an electric furnace, Dorman Fertilizer, the old Federal Phosphorus Company down in Anniston, Alabama, later Swann Corporation started in producing phosphoric acid, not primarily for fertilizer, but it was essentially the first direct production of phosphoric acid by electric furnace where they oxidized the phosphorous vapor as it came to the furnace and collected the phosphoric acid in cotrell precipitators. Some of that electric furnace acid did get in the fertilizer. For a short time the Federal Phosphorus Company made some small quantities of triple superphosphate with electric furnace acid. I should say, however, prior to that time there had been some production of triple superphosphate, called double superphosphate, at Mt. Holly, North Carolina, also more or less experimental with electric furnace acid.

Of course, as you all know, when the Tennessee Valley Authority came into the picture about 1933 or 1934, when they were getting underway, they made great contributions to the production of elemental phosphorus and to the use of elemental phosphorus and furnace acid in the manufacture of fertilizer, originally triple superphosphate, but I believe that probably their use of electric furnace acid to produce triple was the use of a sufficiently strong acid to make triple without necessity of subsequently drying the product and was probably the first such use perhaps anywhere in the world.

As time went on up until the time of World War II, we did get gradual expansion in the production of triple superphosphates, yet ordinarily superphosphate and normal super still held the stage.

After World War II, things began to break, normal superphosphate production and consumption went up very markedly. Triple superphosphate was coming along and we were beginning to hear of production of other types of phosphate materials, calcium metaphosphates and defluorinated phosphates, and the nitric phosphates came into prominence. Ammonium phosphates came back on the scene again, I believe, with the first production of Olin-Mathieson or probably at the time that they first started in production at the Southern Acid and Sulphur Company at Pasadena. That was the second production after American Cyanamid ventured into that field.

In the last ten years things have really changed in the phosphate picture and are continuing to change and they will continue to change in the future. I am not a crystal ball expert and I just do not want to go out on a limb and predict where it is going in the future. I do not think anybody can. I suspect there are many things that we do not dream of today in the way of phosphate fertilizers that may be quite a commonplace not too far in the future. I think it would be of interest to just note briefly some of the things that have happened since World War I and most specifically since 1950.

If you will look at Table I,

Table 1.—Capacity for Production of Phosphorus Fertilizer Materials in the United States, as of January 1, 1,000 Short Tons of Available P_2O_5[1]

Year	Normal superphosphate	Concentrated superphosphate	Other concentrated phosphates	Total	% of 1952
1952	2,951	440	(2)	3,391	100
1954	2,951	580	200	3,807	112
1956	3,174	870	250	4,294	126
1958	3,130	1,090	400	4,620	136
1960	3,141	1,068	570	4,779	141

[1] Plant Food Review *6*, No. 3, 33-34 (1960).

[3] Included with concentrated superphosphate.

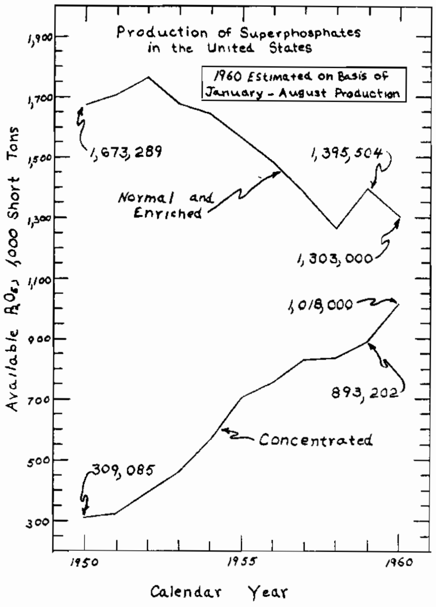
please, you will note that ordinary or normal superphosphate, this is the capacity of production of that material, has been almost static since 1952. We have had changes. Plants have gone out of production and other plants have come in. In 1951, for example, we had 204 plants that were actively producing super. In 1960, January 1, 1960, we had 217. On the other hand, concentrated super, which essentially is triple superphosphate, was stepping up rapidly and very markedly, from 440,000 tons of available P_2O_5, this is capacity, up to 1,068,000 tons. Other concentrated phosphates such as ammonium phosphates, the so-called complex fertilizer, phosphate fertilizer materials and calcium metaphosphate and other things also included in that category stepped up very markedly. During a period of about eight years we had a 41 per cent increase in capacity to produce these materials.

As to the consumption figures, I think it might be enlightening for us to just look at that. These are capacity figures. In Table II you will notice that the production of normal and enriched superphosphates and the proportion of enriched superphosphates in those figures is very small and had been going down for 1957, 1958 and made made some recovery for 1959 You can see that picture better if you will turn over to the next page and look at the figure which shows you the situation on production of normal and concentrated superphosphates over a ten years period

While the normal superphosphate is certainly going down, I think we will probably continue to see some decline, but I am rather of the opinion that the curve is not going to be a steady decline, but there are going to be some wiggles in the curve as is indicated for 1959 and 1960. Somewhere along the line we may strike a plateau where the production of normal superphosphate tends to even off. I think that one factor in that is wet process phosphoric now available to granulators. That may tend to lessen the rate of decline in the use of ordinary super since it should in many plants enable—at least for preparation in the manufacture of certain grades of mixed fertilizers—the use of more ordinary superphosphate.

Concentrated superphosphate is certainly going to continue to increase, I believe, but it is continuing accelerating competition from other materials, as you will notice back in Table II; whereas concentrated super has continued to go up in this period of four years, as indicated there, ammonium phosphates have increased very markedly and also have the other phosphatic fertilizers. However, our old friend normal superphosphate still accounts for nearly 50 per cent of our total production of phosphate fertilizer. This is chemically processed phosphates and not the P_2O_5 from ground rock applied directly to fertilizer.

The most interesting thing that has happened, I think, at least to me, in this phosphate fertilizer picture in very recent years is the great advance and the great strides that have been made in the manufacture and sale of phosphoric acid as such to fertilizer plants. I speak particularly for wet process methods because, while furnace acid has loomed rather large in that picture, the quantities of furnace acid that have gotten into fertilizers are really surplus production over and above the needs of phosphate chemicals, and think it is quite likely that that will continue to be the case with furnace acid.



Let us look at Table III for just a moment which shows the shipments of wet process phosphoric acid for a period of five years, from 1955 to 1959. I think those shipment figures are a very good indication of the quantities that are

Table 2. — **Production of Phosphorus Fertilizer Materials in the United States, Calendar Years, Except Ground Phosphate Rock — Years Ended June 30**[1]

Material	Available P_2O_5, short tons 1957	1958	1959	1960[2]	% of total 1957	1958	1959	1960
Normal and enriched superphosphates	1,380,009	1,265,258	1,395,504	1,303,000	56,2	53.1	53.5	47.6
Concentrated superphosphate	831,510	833,529	893,202	1,018,000	33.9	35.0	34.2	37.2
Ammonium phosphates[3]	171,351	158,849	200,287	279,000	7.0	6.7	7.7	10.2
Other phosphatic fertilizers[4]	72,227	123,227	120,599	138,000	2.9	5.2	4.6	5.0
Total	2,455,097	2,380,863	2,609,592	2,738,000	100.0	100.0	100.0	100.0
Ground phosphate rock[5]	24,919	25,383	24,938	----	----	----	----	----

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[1] U. S. Bureau of the Census and U. S. Dept. Agriculture.

[2] Est'mated on basis of production in January-August.

[3] Includes chemically processed nitrogen-phosphorus materials comprising liquid and solid fertilizer grades of mono- and diammonium phosphates and their processed combinations with ammonium sulfate, primarily produced for sale or use as such. Excludes the P_2O_5 content of ammonium phosphates produced in compination with potash salts to make complete mixtures.

[4] Includes chemically processed materials such as ammonium phosphate-potash mixtures, nitrophosphates, calcium metaphosphate, etc.

[5] Consumption of ground phosphate rock and colloidal phosphate used directly as fertilizer.

Table 3.— Shipments of Wet-Process Phosphoric Acid[1]

Calendar Year	P_2O_5, Short Tons
1955	19,678
1956	24,170
1957	41,571
1958	98,119
1959	150,098

[1] U. S. Dept. Commerce. Includes interplant transfers.

going to fertilizer plants; that is not being used as captive acid but moving out to other palnts, because shipments of wet process phosphoric acid have been almost used entirely, as far as I know, for fertilizer. Generally as a direct application as phosphoric acid out in the west, which, after all does not account for a great portion of the total use.

I feel certain that these figures are a pretty good indication of the amounts that might be moving perhaps into granulation plants or into other phosphate processing plants not connected with the plants that produce the acid. Of course, some of this acid may be going into triple superphosphate.

As to the future, I think that is anybody's guess. I am sure we can be positive ammonium phosphates and nitrophosphates are going to increase. All of the other sources are going to increase. We may have metaphosphate production sometime in the future, at least in limited quantities for special purposes. I think there is a big field in the future for the so-called complex fertilizers aside from the so-called nitric phosphates. I mean by that, fertilizers that are produced by processing phosphoric acid, ammonia and potash salts with any other nitrogen compound that might be necessary into complete fertilizers. That is one group or one type. The other type which I think there is a considerable future for is the direct treatment of phosphate rock with phosphoric or sulfuric or the two, with the addition of ammonia and the addition of potash salts to make complex fertilizers in that way, which do not involve the addition or use of either nitric acid or the use of phosphoric acid, except perhaps in minor quantities, produced as a separate material.

With that, I think that is about all I can offer to you this evening and I thank you very much for your attention.

MODERATOR SPILLMAN: Thank you, Dr. Jacob, for bringing that very fine summary on phosphates.

This ends our meeting for today.

We meet tomorrow morning at nine o'clock. Thank you, gentlemen.

(This meeting adjourned at five twenty o'clock p.m.)

Thursday Morning Session, Nov. 3, 1960

The Round Table reconvened at nine o'clock a.m., in the State Room, Mayflower Hotel, Washington, D. C., Messrs. H. L. Marshall and Albert Spillman presiding.

MODERATOR MARSHALL: The Thursday morning session will please come to order. This morning we have quite an intensive program that is going to take quite a while and we want to get going as fast as we can, so we will start.

Our subject today is Standardization of Raw Materials. This subject has been submitted to us, Your Executive Committee, over the years in question form. So many of the questions back about three or four years ago when we asked for questions did not specifically come out and say "standardization," but they led up to it and then stopped. Your Committee felt that we might as well have a session on standardization this time and get all this backlog of questions in our files out on the floor for answering.

To start this thing off, we had better lay down some ground rules. The first thing is that we ask anybody who has factual information that they want to toss into the discussion to get up, identify yourself if you care, make your statement and let us have it. But remember, it is to be facts, not fancy, facts. In regard to new materials, give us a concrete statement. That is number one.

Number two, we want you to feel free to give your statements and we ask you not to identify the supplier of the materials, so there will be nothing on that.

With those little niceties out the way, the other thing is relative to the conduct of the session. We hope you all keep your shoes on, no pounding on the table with a shoe.

The first little discussion is: Why Uniformity? You're getting too much of "How Much" here this morning, but that's all right. Why Uniformity?

For many years I have been subjected to a lot of information regarding materials, their chemical composition and their physical composition, and when you take that data and try to use it you either get the ouija board out or you get a frown and a headache before you get through because information from different sources just doesn't go together. That has been a prevalent problem everywhere and your questions have asked us to try to get a language that we can work by. Therefore, that is one of the points of uniformity.

The other point is: we have to live with our friends, and we have some friends who are pretty intricate to live with, speaking primarily of the judge, jury and convictor, prosecutor, the state officials. Many of you people have had cause to meet with the state officials because you are a little bit short in one component or another in your fertilizer. Why that shortage? Everybody, we know, and everybody honestly figures their formula to come out right, but where you have five,—six carloads of material coming into a limited storage building you have to mix it up and if those cars vary there are going to be times while you are mixing with that material when your calculation may be low as much as one to two per cent. Some of that data shows that. It sounds shocking but it is true; that some of the data shows you can get a series of cars and analyze the individual cars and you will be short. That is another reason why we need uniformity.

Going back to the uniformity of expression, we have a need—a lot of people are granulating and they need to know the water content of their system, be it combined or free water, but if you look at your analysis certificate; on most of them you cannot tell how the moisture was determined. Maybe we need to state that moisture figure and the method of arriving at it.

Another thing, with the use of the AOAC methods which have been studied by everybody, we can skip the citrate insoluble P_2O_5 and of course everybody can subtract and get available. The K_2O is all right; the nitrogen is all right, and the AOAC has been outstanding in giving us methods and shortening the methods. Those figures nobody has a quarrel about, because they are a part of the book, but when you come to this value that everybody is interested in, free acid, well, free acid can be anything you can think of. A lot of it has been reported as phosphoric, (H_3PO_4) Our greatgrandfathers reported it as H_2SO_4. Then we see some figures as P_2O_5. Brother, when you figure how much ammonia some of those reports are going to take, you had better go get your transquilizers, you'll need them.

So we ask or suggest that you give consideration to getting a uniform figure of reporting and stating what it is. Going further with the free acid, we need to know how that free acid was determined, because if you take it off your water extract of your phosphate determination and it is fresh superphosphate you are going to titrate some of that monocalcium phosphate and get a higher free acid. If you have a lot of fluorine release, you are going to have fluorine in your free acid. If you use acetone, that is the closest, probably, according to everybody's experience, to the absolute ether determination of free acid. Of course, ether is too long, too hazardous and everything else.

What we need to do is ask the analysts to state how they arrived at their free acid figures, then you can have a base to go to work on.

We are talking a little bit about analysis here, and I would like to read to you some analysis of mixed fertilizer which points up the lack of uniformity. Our good and honorable and respected past friend Dr. W. J. Gascoyne, Sr. made an analysis of a 2-10-3, or 2-9-3, I guess it is. He came up with ammonia—that was in the days when nitrogen was expressed as ammonia —2.21, available phosphoric acid 10.39, insoluble .61, potash 3.38. Our dear friend and starter of the fertilizer industry Dr. Liebig, working in Baltimore, analyzing the same sample: ammonia 2.07, available phosphoric acid 9 per cent, insoluble phosphoric acid 1.41 per cent, potash 3.48.

This was done in 1878 and they had the same troubles we have today, but they went further on another series, these same two gentlemen, and analyzed a sample. I do not think there was too much time between the two results and they came up with the following: Liebig analyzed the sample as 1.03 nitrogen, available 9.30 phosphate, potash 1.13 and total P_2O_5 13.10. He sent the sample on to Bill Gascoyne. Bill Gascoyne came up with: ammonia 1.10 per cent, available phosphoric acid 9.60, potash 1.18, total phosphate 13.40. So they checked each other pretty close back in 1878. So how far have we come?

For that reason we plead with you to get a little uniformity in reporting these things, uniformity in technique of analysis and uniformity eventually in production.

In keeping with this uniformity scheme of affairs, we found that there was quite a variation in some of the received materials. Actually, your Committee did not believe some of the data they were getting. We took this subject up and talked about it in Brown's Bar for a couple of Saturday mornings, in Al Spillman's office for a couple more hours the same day, and the more we talked the more confused we got, *but it was only coffee,* and we invited the lady Editor of *Agricultural Chemicals* to come in and sit in on one of those conversations and offer us a suggestion as to how we could find out what is going on.

Out of that conference came a questionnaire which every one of you probably got and answered. This happened back in April. We sent them to you and we have replies and, in fact, we waited until the middle of June or the first of July for a cut-off date to go in and study those replies. There has been a summary prepared thereof and I will just let Miss Eleonore Kanar give the summary of that survey which she and her magazine so kindly put forward to the Fertilizer Industry Round Table people and the industry people. Miss Kanar.

MISS ELEONORE KANAR (*Agricultural Chemicals*): Thank you, Dr. Marshall. Members of the Fertilizer Round Table: I think the reason we are all here and the reason why the Fertilizer Industry Round Table has grown from a handfull of individuals to the 400 or more representatives here this morning is because you have common problems to discuss and, as Dr. Marshall pointed out, it is rather difficult to discuss common problems unless you have a common language. Recognizing this need, the Cabinet, as some of the manufacturers in the Baltimore area call themselves, suggested that it might be a good idea to find out what other manufacturers are doing throughout the country and how they are discovering such data as free moisture, free acid, sample preparation, particle size classification and so forth. Our company mailed out questionnaires earlier this year to fertilizer production plants asking them a series of questions dealing with their particular operations and my function on the program today is to present the results of that questionnaire.

I have most of them on slides and I am going to go over them fairly rapidly because reprints have been prepared and are available for you here today and you can read them at your leisure just as carefully as you would like.

A Need for Uniformity in the Fertilizer Laboratory

Eleonore Kanar

EARLIER this year, (March, 1960), *Agricultural Chemicals* published an article in this column under the title "Uniformity in the Fertilizer Laboratory and Plant." Subsequently we conducted a survey of fertilizer manufacturers to find out how different companies conduct routine testing of raw materials and finished products, and how they report the results of such tests. The response to the survey was good, and findings are summarized on these pages.

An analysis of the survey emphasizes the need for some standardization of raw materials and uniformity in nomenclature to cover the complete description of processed raw materials. The survey also shows that:

1. The term "Moisture Value" (free or combined) has no real meaning unless further defined as to method of determination.
2. Free Acid is expressed in several ways, and determined by several methods by various manufacturers . . . thus this term cannot be used freely without further identification.
3. Particle size and particle size classification offer the greatest opportunity for variety. Of all questionnaires returned, no two listed the same set of screens for particle size classification. Only 15% of the group showed any agreement, in that screens 6-8-10-20 (presumably Tyler Standard) were included in their series. No interest was shown in determining fines of minus 60 mesh.
4. In general about 80% of the participants in the survey used a pulverizing mill for grinding the sample in preparation for chemical analysis.

In discussing results of the survey with several fertilizer manufacturers, it was emphasized repeatedly that fertilizer manufacturers are greatly handicapped in production planning by inadequate and nonuniform raw material specifications. They stressed the importance of having correct data on chemical and physical characteristics of raw materials, presented in such terms as to be readily usable in plant formulations.

Moisture Data

In the case of moisture data, fertilizer manufacturers who granulate or agglomerate, need to know the amount of free water in the raw materials with a reasonable degree of accuracy. They point out that 100°CMoisture includes some combined water along with some volatile components. Hence, with this information, they can arrive at a free water figure from data supplied by the raw materials producer. They specify that composition data should clearly state how moisture is determined.

Free Acid

In ammoniation and granulation, identification of "Free Acid" is most important. The Free Acid in superphosphate is very likely present as phosphoric acid, and its role is to hold a portion of the free ammonia, which may be present in the formula. A chemical reaction takes place between these two components, yielding monoammonium phosphate. Since the system is phosphatic, many manufacturers prefer to express the Free Acid as "phosphoric." The questionnaire reported on the facing page shows that over 50 per cent of the fertilizer manufacturers surveyed report this value as "phosphoric acid."

Some raw material suppliers report Free Acid as "phosphoric," others identify it as sulfuric or as phosphorous pentoxide (P_2O_5). Fertilizer manufacturers interviewed point out that when the Free Acid is reported as sulfuric or as P_2O_5, it is necessary to make conversion to ascertain how much ammonia will be reacted with the free acid.

Another factor which affects the Free Acid figure, is the method of extracting the acid from the sample. Acetone extractant is sometimes used, because the value so obtained is approximately that obtained by the Absolute Ether method. Water may also be used as extractant, and according to the survey, 51.5% of the fertilizer producers reporting to the questionnaire, do use water. It should be cautioned, however, that if water is used as the extractant this value must be carefully evaluated. Factors influencing results obtained include the solubility and titrateability of mono calcium phosphate, particularly if the superphosphate is freshly made. However the Free Acid is determined, fertilizer manufacturers urge that they be informed of all factors affecting this value.

Particle Size

Since process efficiency is related to the Particle Size of materials going into the formula, fertilizer manufacturers agree that some uniformity should be developed if possible, in evaluation of this physical property. Although this is one of the most important factors in evaluating process efficiency, — it is almost ironical that fertilizer manufacturers do not agree on what screen sizes should be used in classification. No two returns listed the same series of screens, and only 15% showed any agreement, in that they used screens 6-8-10-14-20. Others replying to the questionnaire used some of this series, adding either a 4 mesh, 60 mesh, and/or 100 mesh. Personal discussion with some fertilizer manufacturers indicates that a desirable standard series might be 4-10-20-60-100 (W. E. Tyler series). Any additional screens desired for specific plant conditions could then be added to the "standard" set. These observations, of course, are just suggestions, and any agreement or standardization must necessarily be of a voluntary nature.

Replies to the questionnaire show some general agreement among fertilizer producers on size of sample used, method of reduction, and equipment used for screening. As reported in item number 5 of the survey, 50% replying use a 100-gram sample; allowing 3 minutes for granular products in shaking and 10 minutes for pulverant materials. As shown in replies to items 5 and 6, there is also some general agreement on screening and equipment for screening.

Fertilizer manufacturers observe that if chemical and physical data relating to fertilizer materials can be reported uniformly, the fertilizer manufacturer and mixer would then be able to formulate more closely. The plant superintendent would also be able to exercise closer control, and could conduct a more efficient operation, yielding a more uniform product.

Survey On Uniformity

Replies from 23 States and Canada are included.

1. Moisture Content

How is moisture determined and reported in your control laboratory?

Materials

70.0% Oven @ 100° C.
10.2% Vacuum
12.9% Other (not identified)
6.9% Air flow

Mixed Fertilizer

66.6% Oven @ 100° C.
7.0% Oven at temp other than 100° C.
10.2% Vacuum
13.1% Other (not identified)
3.1% Air flow

2. Free Acid

a) How is free acid content reported? (Expressed as:)

51.3% Phosphoric acid
37.8% Sulfuric acid
8.3% P_2O_5
2.6% Other

b) If free acid is reported in any category, other than as "phosphoric acid," is there any reason why expressing this determination as "phosphoric acid" would be inconvenient?

83.3% No
16.7% Yes

c) What is the extractant used?

42.4% Acetone
51.5% Water
6.1% Other

3. Sample Preparation

(13.7% of the replies did not identify sample preparation)

Raw Material

Mill — 80.0%
Other — 6.3%

Granulated Mixed Fertilizer
Mill – 81.4%
Other – 4.9%

Ammoniated Mixed Fertilizer
Mill – 76.7%
Other – 8.6%

Dry Mixed Fertilizer
Mill – 74.4%
Other – 11.9%

b) What is the mesh sample ground to?

35 mesh – 36.0%
30 mesh – 24.0%
20 mesh – 16.0%
over 35 – 24.0%

4. Particle Size

a) What are the testing sieve sizes used in determining particle size?

There was no uniformity in replies to this question. All replies listed a different series of screens.

5. Sample Information

a) Size of Sample
50.0% 100 grams
21.1% 200 grams
15.6% other
6.3% no reply

b) How is sample reduced?
89.2% Riffling
10.8% other

c) What equipment is used for screening?
72.2% Ro Tap
8.4% Hand
8.4% Tyler Portable Shaker
11.0% Other

6. Time of Screening

Granular Materials
61.4% 3 minutes
26.9% 5 minutes
11.7% other

Raw Materials or Powdered
65.6% 10 minutes
24.1% 5 minutes
10.4% other

MODERATOR MARSHALL: We thank Miss Kanar for her presentation of this, and there are reprints available of this particular report on the surveys. Incidentally, it appears in the November issue of Agricultural Chemicals.

As you probably noted, the data was as confused as it could be. There was quite considerable confusion particularly in the particle size delineation both as to method and to the particle sizes that they wanted to determine. Particle size is a question which means screening. There is only one fellow who knows anything about screening around here with half a degree of certainty and he is willing to tell us about it. He said he could do it in five minutes, but we decided that maybe an hour and a half or two hours were necessary, but we'll let Wayne King of the W. S. Tyler Company give us a little education as far as screening is concerned. But we have got a ground rule for Wayne; he cannot throw candy out into the audience to keep from having questions asked today.

Wayne King.

MR. WAYNE KING (W. S. Tyler Company): Thanks Doc. I just now wrote my little talk and it won't take long. The two hours I asked for, Doc, were to talk about my grandson. This won't take long. As a rule—pardon me for reading this thing, but I want to get my verbs in the right place on accounta they're recording this. As a rule I am pretty careful and use some effort to reduce my exposure to what might be termed "Open Season on Me," you see. We are talking about units of measurement. Of primary import, I would say, would be the chemical values. My subject here is in regard to particle size measurement, more particularly, if you can use this phraseology, I am referring to particle size distribution. This is going to be all printed and you will get a copy of this and Doc also has some data that I have from my laboratory which will be included and which I do not have on me.

Another word jumps in here, "Segregation." Segregation means different things to different people and different things to the same people, geographically, I guess. In other industries this has been solved, segregation has been solved and we will do it in this industry too, if they give us time; defense in depth, I guess.

I am equally interested in the commercial proper sizing of the raw materials and our finished goods, but this morning I confine my discussion to multiple gauges known as testing sieves. I want to explain to you, and it is absolutely an astounding thing that a simple thing like a testing sieve, as long as you know the opening of the sieve, I don't care whether you call it X, Y, Z, A,B,C, or what it may be, if you know the opening of the sieve, it is totally irrelevant to any of us who have spent over half a century playing with these things why we cannot get together on this thing.

Tyler Testing Sieve

Wayne W. King

WE are here to discuss specification standardization. I am interested in the physical measurement of particle size. Equally interested in the measurement of production classification of fertilizers as well as the application of multiple gauges, known as Testing Sieves. These testing sieves will show us the particle size distribution in our materials and in our finished products, also the efficiency determination of our plants on stream.

Specifically, the executive commission of F.I.R.T. has suggested the use of the Tyler Testing Sieve Series for our work in this field, so that we can simplify these test reports and make them comparable and fully understandable. The U. S. Series sieves can be used; but report with the Tyler Mesh Count, so that we have the desired uniformity. Also, make sure you have a standard sieve.

To each of you, as registered here, we will mail a copy of the Tyler Bulletin #608. The Tyler Series is shown on page #4. Also will be included suggestions for specific test procedures. This Tyler Series is of the 4th root of 2 and for our work only a few specified sieves are involved, except for more intricate research and experimental efforts.

For instance to refer to the preceeding "Paper," I will list the following sieves that should be used with identification by opening.

Tyler Standard Screen Scale Testing Sieves

4	Mesh	.185″	Clear	Opening	4.76	mm.
10	“	.065″	“	“	1.68	“
20	“	.0328″	“	“	841	micron
28	“	.0232″	“	“	595	“
35	“	.0164″	“	“	420	“
60	“	.0097″	“	“	250	“

These sieves are our suggestion for Superphosphate.

For Granular fertilizers, we would usually use the Tyler sieves between 4 and 35 mesh, including the sieves at point of separation.

For phosphate rock, obviously we would use the finer sieves from 60 mesh finer.

Perhaps at our next meeting in 1961, we can be more explicit, after additional data has been accumulated. It is my considered opinion, that a thorough study of particle size distribution, will reveal much important information as to efficiencies of acidulation in the ease of phosphate rock and amoniation rates on mixed goods.

In conclusion, may I state, that we are hopeful. that this start in uniformity of Testing Sieve analysis will give all of us a clearer picture of this physical measurements. Thanks for your indulgence.

MODERATOR MARSHALL: Thank you, Professor King. Has anyone any questions for the Professor?

There being no questions, we will now proceed with the next lecture hour and that is what is known as the Users' Panel. In my opening remarks I told you folks that we wanted to get the facts and for that reason we have, your Committee asked six people who use relatively large, in some cases, and in other cases moderate tonnages of raw materials to make mixed fertilizers to be a panel and to lay their case before the bar and tell us their experiences. As I said, we ask everybody to do that and for that reason there is a stack of forms to write your answers down on and turn in to the panel or to the Executive Committee, anybody who would like to do that.

Without further ado, I am going to ask Rodger Smith of Eastern States to come forward and start the parade. Rodger.

Address By Rodger Smith

Leader, User Panel

He crossed me up to the extent that I understood Al Spillman was going to chair this and I still think he should. Will the panel come forward and take their places at the table.

Good morning, gentlemen. We do have a Users' Panel of the men who are assembled here at the Round Table. The group got together Tuesday evening to discuss their respective parts in this morning's part of the program, and, after having prepared some notes on the overall topic myself, they have asked me to more or less introduce this subject, or at least the subject from the viewpoint of the users and so I have prepared a few notes along that line. This is a little bit off-the-cuff, so please bear with me.

The other members of the panel will follow up with more specifics. I will refrain from talking about specifics in the problem but give the overall problem as users see it.

As I say, I have been asked to open up the subject from the users' viewpoint. Actually, I think the three speakers have done a very fine job of that, Dr. Marshall with his statement, Miss Kanar with her report of the survey, and Professor King's important contribution on a very important phase of this whole thing, which are a pretty good introduction. But here goes with the problem as I see it.

Let me say first that it is a growing problem and it is a genuine one. We have come here today with not a gripe at all but a genuine problem that we are sure can be handled as we get this thing out in the open and discuss it, probably a matter of communication being the primary problem.

You might get the impression off hand that the mixed fertilizer manufacturers are trying to form a union of something to that effect or, perhaps even worse, that this is an uprising of Dark Africa against its European masters or governments, but I assure you it is nothing of that sort. As I said before, we have a genuine problem which we want to discuss with you all collectively.

I would say that this is merely a stage of growing pains in a rapidly growing industry. We are a rapidly growing industry as the tonnage figures and the growing technology that is recorded in this and other meetings indicates, and it is quite natural that growing pains of various types come to the front, and what we are talking about this morning is one of them, in my opinion.

It is a step necessary in the development of the industry from a scavenger one to a chemical one, to a truly chemical industry, which we are approaching, and this is one of the things that needs to be ironed out in order to progress with that development.

As I say, we have a problem and we are glad that this forum is available to discuss it and we know that you primary manufacturers or suppliers are also glad that this forum is available, because it is a matter of communication very largely and it is difficult to get together.

I know in talking with representatives of the various material industry they talk with 100 people and they get 99 slightly different viewpoints on specifications, and we are not going to finalize our specifications here this morning—that is too much to expect in this panel which, although a representative panel, still does not have the information nor the right to speak for the full mixed fertilizer industry, or finalize anything for the full mixed fertilizer industry.

As we approach this problem we know that you will be understanding and we know that you want to meet the needs; we know that, but you want guidance on what needs and that is what we will perhaps get a little closer on this morning.

We can only make a start on this, we can approach it at this time. Actually, our message today is not so much to indicate product types specifically as uniformity of types; uniformity within certain classifications is perhaps even more important than the specific types.

Our opening speaker yesterday morning, Dr. Ingebous, of the International Minerals and Chemical Corporation, in his presentation on rock phosphate production presented considerable statistical information to us. He talked tolerances and standard deviations and so on. I think this emphasizes to us that there is control being exercised in certain parts of the total industry, here, here and here, and there is a considerable amount of control and certain factors here and there are utilizing statistics and getting exercised about this whole overall problem, but we do not have continuity all the way from the rock or the potash ore and so on out to that final dry good fertilizer that is moving over the road from the mixed fertilizer plant to the country point or to the farm. We need more continuity and having great care for statistical control at one stage in this overall process of fertilizer manufacture is not too effective unless there is continuity and that is what we are trying to approach.

Let us take a look at this matter of continuity in attempting to define the problem. I would try to express here the viewpoints of some of the factors or people who are interested parties in this process of getting the basic raw material to its final, ultimate user. It seems to me that when approaching this chore, the logical place to start is out there at the farmer-purchaser and to work back. He is the last man to lay money on the barrelhead for fertilizer. He is not only —he is in this position, I think— he is not only convinced that he is making an investment in a farm production supply that will turn him money. We boast, of course, that his dollar spent for fertilizer will average about three dollars in return. He not only has apparently become convinced of that by the fact that he does make the purchase, but he also expects analysis to meet guarantee. It's a reasonable assumption, just the same as when we go to the grocery store and buy five pounds of sugar or the farmer purchases a ton of peat or any other unit of measurement, it is a reasonable assumption on the part of the farmer that when he buys a fertilizer and the salesman has expressed it to him in certain terms, units of measurement which in this case are the percentage content of nitrogen and phosphorus, potash and perhaps magnesium, boron, or other nutrients, that it does actually analyze to that. It is a reasonable assumption, approaching it from a strictly lay point of view.

The farmer has a guardian as has been expressed here already this morning. This guardianship has been established by law in, I think it is 47 of the 50 states. There is a fertilizer law in these states and that law establishes a control service, a control official and it is his responsibility through the techniques, the procedures we are all familiar with to protect the interest of the farmer.

These individuals, as you know, have developed here a few years ago a model fertilizer bill which is being gradually adopted, or with some modifications, in a growing number of states, and this bill, if adopted, as originally written, as the model, has teeth in it and it means that we have to in fact come closer and closer to meeting this guarantee if we are to avoid the consequences of the bill.

This control official is a responsible individual, a person with training, or he would not have that responsibility, normally he at least has a chemical degree, he reads the technical magazines with which we are all familiar and in these magazines he reads of the technical developments in the fertilizer industry. He reads articles by our friend John Hardesty, by Dr. Hignett, by Technical Service Directors like Elmer Perrine or Joe Sharp, all of these people, and he is impressed, obviously, with the information that is available to the industry and developments that are occurring. When he tries to stack that up in his mind, I expect — making a few assumptions, but I do not think too many—with the scorecards in his files, alongside his desk—he thinks there is a gap there, a serious gap between the apparent technical developments in the fertilizer industry and the record of the industry as indicated by the analyses of individual manufacturers, according to the analyses he has.

I am sure there is a growing impatience, as we look at the thing objectively here today, and perhaps understandably, on their part as to why the fertilizer industry with this very rapidly growing technology available to them still maintains the record that it does.

We have to ask ourselves today, I think, why?

These control officials, as we all know, publish their information and in some cases they go somewhat further with articles and get their viewpoint across to the public in the state, and the farmer who hears of a rather poor, or what appears to him a poor record of the industry, becomes very unhappy. He gets the impression, rightly or wrongly, that he has been cheated, the same as he would if he got a given poundage of some material and he found that it was something short of that. It is the impression he gets, although it may be well over in two nutrients and down in one; he still gains the impression by the bold type in that book or wherever he has gained the information that he had been cheated.

It is not only a problem for him but it is bad for all of us. It is bad for the total fertilizer industry, mixed fertilizer people and suppliers and, in fact, to all facets of this total picture. It is bad for the agricultural experiment stations which are doing things to encourage fertilizer use. It is bad for the total economy, because it discourages overall sales because if the impression is spread that the fertilizer industry is shortchanging the public that is not the sort of information which encourages participation by the ultimate user. It is only when we get participation by the ultimate user that the industry can grow and agriculture can become more stable.

Let us move from the position of the farmer to the mixed fertilizer industry. There is a major problem of analysis control, and the reasons appear to be as follows: I think there are about these five major reasons of why analysis control is a major problem of the mixed fertilizer industry.

The first is—and perhaps this morning as far as this discussion is concerned is one of the most important—lack of uniformity of ingredients. That is a major reason. Lack of uniformity of ingredients.

Two, lack of information on ingredients. These two go hand-in-hand but are important.

Third is ammoniation practices which we are all familiar with and realize it is an important problem and one that a great deal of time has spent in Round Table Meetings on and needs no further comment at this time.

Fourth is this matter of segregation which has been mentioned this morning. That is important and is not unrelated to the first one I mentioned: Lack of uniformity of ingredients.

Fifth is operating errors.

Let me repeat those. One, lack of uniformity of ingredients. Two, lack of information on ingredients. Three, ammoniation practices. Fourth, segregation of ingredients in the mix. Five, operating errors.

As far as my part in this program, there are more details and members of the panel will fill those in.

The causes of number one and two, that is lack of uniformity and lack of information, I would put as follows: These are causes of the problem on the part of the mixed fertilizer manufacturers.

One is undue averaging on his part. He has a given ingredient, varying considerably in analysis from different shipments and almost always he finds it, as a practical matter, necessary to put them all in the same bin. Undue averaging.

Two is financial loss on ingredients. He purchases most ingredients on a unit basis and he feels that he cannot, if he has analyses that vary, let us say, two units, or materials that vary a total of two units he feels that he cannot go down to the minimum and use that. He has to strike some sort of an average and use it because of the financial loss that he would otherwise incur.

There are instances of calculating too high on ingredients. This goes with the previous point, but due to lack of information or because of trying to avoid too great a loss on his materials, why, he calculates too high, assumes too high an "average"—and I put average in quotes there—and the result is low analysis of certain nutrients in the mixed fertilizer.

Fourth—all of these relate to each other, but the necessity for undue overages. The cost of overages as practiced in the fertilizer industry—I have checked this point with several producers—and I believe it is practice that they vary in the range of $1 to $2 per ton. It may be that some low analysis grades fall below that, but as a general statement I think it can be said that overages are costing the mixed fertilizer manufacturer a dollar to two dollars per ton.

I got a little substantiation for this point when I was a member of the NPFI's Committee on In-Plant Shrinkage last year or so and a group of industry people discussed this point in detail, ran a survey and you have seen publicized in the press in the last few months the results of this Committee's information and it is a major problem.

The next point that I have, the next cause of this problem or these off-analyses is inability to match particle size. Here we have segregation due to variation in the individual shipments, or between shipments, or between suppliers.

Causes: Undue averaging, financial loss, instances of calculating too high, necessity for undue overages, and inability to match particle size.

This is not only a problem to a person calculating formulas but it is a real problem to the man out in the plant, to the plant superintendent, to the foreman, the people who are actually controlling the valves out there on the meters and so on who are looking at the product coming out at the ammoniator hour upon hour during the day.

This variation in materials, differing screen size, and other characteristics lead to the common statement by plant operators that this thing with all the developments and all of the improvements is still to a considerable degree an art rather than a science, and to an unfortunate degree this is true. In other words, what they are saying is that one and one is not two. We all know that whether we are talking about making fertilizer or what we are doing here, if we put together hour after hour the same materials under the same conditions the results would be the same and in that case one and one would be two, but as it is now, because of variation in materials and, to a lesser degree, probably water and other variables in your process, why, we have to agree that one and one today are not two and that this thing is still art as well as a science and we encourage him, of course, to cultivate that art and we would be in a bad way under present conditions if we did not have people who could exercise judgment with those valves or in other ways to maintain a reasonably uniform final product.

The result of this situation is a less uniform product both chemically and physically than we would like to have and the farmer and the control official would like to have.

It is not just those of us here who represent production management, supervision or development but top management gets into the act also, and very rightly so. They ask this question: Why do we have lack of uniform analyses? Why is there such a large charge for overages? When they look at the profit picture or look at the buildup of costs, they very rightly ask: Why is it necessary to have such a large figure in there for overage? Why do we have a problem with the state chemists?

Those are some of the questions that top management ask us and very rightly so.

The sales department has a real problem. If morale on the part of sales personnel is lowered to the extent that information gets abroad that the product is not up to guarantee, why, it is demoralizing to the men out in the field and we can well appreciate their problem.

Furthermore, when you rack up the record for a year or as analyses come back to the plant from the laboratory, this sort of thing is discouraging to plant operators. For the most part, they take a certain interest in their work and they plan to do a job and they look at analyses that are sometimes way beyond reason and the record is discouraging to them. They are trying to do a job, they look at this record, and its is discouraging, because this in only a small way, in all probability, reflects upon their performance. Actually the causes are some of these other things that are beyond their control and things that we are trying to review with you now.

I would say that our need today is specifications, uniformity within tolerances, information on analyses and methods, uniform or similar terminology. Let me repeat those. Specifications, uniformity within tolerances, information on analyses and methods and uniform or similar terminology.

Together I am sure that we can work this matter out, work out the specifications and the terminology. In some cases, for example, 20 per cent superphosphate manufacturers may have to go to your suppliers in purchasing rock, for example and work out specifications in a way that will permit you to maintain uniformity. Others, of course, are working with the primary raw material and it is within your control to develop this thing.

I won't go into detail on screen analyses, but let me just comment a bit there. Screen analyses have become very important, much more important in the last few years than previously. Perhaps it is unfortunate, because we have violated a principle of good analysis control, and we might as well admit it. There is one person in this audience who feels very badly about this, and rightly so, and that is our good friend John Hardesty of USDA at Beltsville, because he has been telling us all for years that if we would grind our materials to minus-40 mesh that whether we are making pulverized fertilizers or granular fertilizers our problems of segregation of ingredients would be for all practical purposes eliminated, or be minimized to a point where it would not be a practical problem. We all know that he is right, he has been right, but we have been looking for short-cuts and trying to maintain certain rates of production in a given plant and still improve the product and so we have complicated a problem. As evidence that we have complicated the problem, I think we only need to look to the pesticide field. There they are working with materials that are very fine, uniformly fine and, of course, we admit they are working in most cases with lesser amounts, which makes it somewhat easier, but they do not have a segregation problem to the best of my knowledge. In talking with someone in our own organization, they hardly know by sight these control officials. You and I wish we were in that same position and we wish we did not have the problem that makes it necessary for us to come in contact with these people.

We have complicated this problem, but we have probably gone too far for the present anyway to retract and we have to find ways to minimize the thing and in this regard we need to have certain uniformity, certain ability to balance one material against another and information on what we have.

This may be something for the present, it may be supplemented by other processes as we go on which may come into the picture which will supplant this problem, but for the near future it is with us and I am afraid we cannot avoid it; we had better recognize it and attempt to do something about it.

As I wind up these notes, let me say that a balance is needed in our approach on this thing. I have mentioned here some monetary values of the benefits, but there are other factors than just the overages, the dollar to two dollars which I have mentioned, there are other benefits in operation and other factors. But there are benefits, but as we approach this problem we want to keep them in balance with the monetary cost of improving the uniformity of the raw materials. We have to strike a balance and I am sure that we can make the improvements necessary without undue costs. I am sure of that, based upon the steps that have already been taken by some factors in the raw materials industry.

One word of caution, as I close, and that is that standards can be current only. They are changing gradually as adjustments in processes of granulation and fertilizer manufacture evolve. In other words, we are talking about the near future look and I think that is a practical one and should not minimize our efforts, but I just want to caution that we are not talking about—in other words, we cannot establish today finality for ad infinitum specifications on materials.

That completes my remarks. I gather from what Al has said here that it falls to my responsibility to introduce the other members of the panel and I will proceed to do so now. I understand that our first speaker on this panel is Ed Kingsbury who I am sure needs no further introduction to you. Ed.

Standardization of Materials

E. D. Kingsbury

BEFORE we can discuss standardization of materials, we must first ascertain what products are most advantageous taking into consideration all facets of production, costs, and the distribution of plant food within the product. We must, it seems to me, justify our present selections of granular, coarse or standard material and then recommend changes that we would like to see made. If we can agree in the main, standardization should be no problem.

Now to discuss potash, and a few mechanical changes that have influenced our thinking regarding particle size.

Using granular potash in low nitrogen grades, aggravated separation in the screen ranges with respect to phosphate and potash, while nitrogen is relatively constant. The end product will be high in K_2O in those ranges closely approximating the size of the potash. If we were to select granular potash for low nitrogen grades, we should screen our end product approximately —4 +14 to minimize separation. Formula costs, as many of us know, can be minimized and production rates maximized.

Using coarse potash follows the same pattern, but to a lesser degree. We feel that if this selection is made an end product approximately —6 +16 should be maintained.

With standard potash, there is less separation in the screen fractions; however, there is a tendency to build up potassium in the recycle. In addition, a wider screen range may be necessary, such as —8 +24, and this within itself promotes a degree of separation.

A few manufacturers have done an excellent job of controlling plant food distribution using standard potash in low nitrogen grades, but we note that when deficient, potassium is consistantly low; moreover, in 12-12-12 where almost all manufacturers are using standard, there is still a great deal of separation between P_2O_5 and K_2O.

Since 1956 we have developed a gear reduction for our weigh-belt to increase accuracy, co-ordinated solid and liquid flow with an electric timer, developed a new sparger, and will soon complete our transition to electromagnetic meters. We feel that these improvements have a direct bearing on the selection of raw materials used in manufacturing.

With the advent of the O.K. sparger (O'Neill-Kingsbury), we have been able to increase our flexibility in formulation. We can, merely by turning valves, effect a difference in plasticity equivalent to 200 lbs. of water per ton. Three pipes under the bed and two on top have replaced the conventional T.V.A. design. Potentiometer readings taken at many locations under the bed indicate good distribution of the liquid phase, especially with regard to minimizing temperatures throughout the complete mass. This new sparger, as you would expect, has allowed us to decrease the particle size of the potash used, if we so desire.

Currently we are trying to "have our cake and eat it too." Since we produce granular fertilizer for two plants, 30 tons per hour of end product becomes necessary on some grades during the height of the season. Coarse potash is used in low liquid phases, combinations of coarse and standard in medium liquid phases, and standard in high liquid phases.

In an effort to improve our product *chemically,* as well as physically we have recently changed to 7 and 8 mesh tyrod screens on the top, and 14 and 16 mesh on the bottom to effect an end product that will screen 95% —6 +16.

Separation of particle size within our bins has been greatly reduced by new "desegregators." Small reversible belts traveling 800 feet per minute are minimizing the cone pile. This, we feel, will go a long way towards offsetting the disadvantages of using coarse potash.

In an effort to re-examine the separation of plant food within the end product, we have, for the past three months, been sampling the 6-10 range and the —10 range for nitrogen, phosphate, and potash. While there are still fluctuations in plant food, improvements have been made. In addition, samples received from the State this fall tend to confirm our hopes that we can "have our cake and eat it too," provided there are adequate controls.

Now what do we want, or more correctly what do we *think* we want with respect to particle size and where do we think improvements can be made.

In our opinion, there has been an effort on the part of potash producers to standardize their coarse material, 100% through an 8 mesh, 20% on a 10, 40% on a 12, 60% on a 14, 80% on a 16, and 100% on an 18 is, we feel, ideal, and some companies are approximating these specifications. If you move the screen size up 2 mesh, for a final product, theoretically at least, you will have 50% of the potash in the 6-11 range and 50% in the 11-16 range. Currently our end product is running a little higher than 50% in the 6-11 fraction, but if we use a potash with too high a percentage held on a 10 mesh, problems of coverage occur.

Differences between refining processes are not significant with regard to granulation in our system. There is, however, a significant difference in the amount of dust contained in various products, and we note that some producers exercise better control over particle size throughout the season than others.

One company is producing an almost dust free grade of potash with the approximate specifications. 5% 10-16, 10% 16-20, 75% 20-32, and 10% —32. We believe that this, at least with present formulas and equipment, is an ideal standard grade. With the replacement of 5-20-20 and 4-16-16 by 6-24-24, requirements for coarse potash will probably be reduced. In the near future then, we suggest that more *dust* free standard be considered with the approximate specifications previously mentioned.

With regard to phosphates, I shall limit the discussion to normal super, triple, and phosphoric acid.

Superphosphate is manufactured at our Indianapolis plant, with 77 BPL rock and virgin acid. After experimenting with various Baume inputs, we have settled on 607 lbs. of 100% H_2SO_4 to 100 lbs. of rock, utilizing 56½° Be acid.

This, we feel, is the highest degree Be that can be used without producing an excessive amount of small pellets. Tests of *ex*-den super averaged for this spring: 8.2% moisture, 19.9 available, 1.1 insoluble, and 3.0 free H_2SO_4. Starting the cut acid into the pan before the rock, and dropping the batch just prior to the top of the rise, has promoted optimum honeycomb. Screen analysis of the super prior to going into the ammoniator is approximately as follows: 2% on a 6, 10% 6-10, 10% 10-16, 12% 16-20, 20% 20-32, and 45% —32. Ammoniation rates are calculated at 6%.

We have used coarse triple in certain formulations containing nitrogen, but due to low ammoniation, we have standardized on R.O.P. Run of pile triple produced today, we feel, needs to be improved. Screen tests on a recent car showed that 60% passed through a 35 mesh screen. It's too bad we didn't have a 325 mesh screen at the time. It is not the —35 but rather the —100 and—200 mesh material that is causing the dust problems. If the Vice-Presidents in charge of triple production were required, as a part of their training, to unload one of these cars, I'm sure changes would be made rapidly. We would like to suggest, therefore, that producers *remove* these very fine particles just prior to loading.

In addition, standardization of triple specifications between producers is lacking. Summarized screen analysis of three triple cars received recently are as follows:

1.	39.3	—6 +16	with	53.8%	—20
2.	22.9	—6 +16	with	71.5%	—20
3.	13.2	—6 +16	with	78.5%	—20

While these specifications do not represent an arithmetical average of the products, they do, nevertheless indicate a lack of uniformity between producers. Moreover, there is a lack of standardization between products manufactured by the same producers. Part of this problem is probably due to the old culprit "pile separation," and while a cure for this problem will be difficult, improvement should be made.

The first triple car mentioned contains, in our opinion, too much material in the —6 +16 range; the second was not a bad car, fine, but not too much dust; the third one we should have saved for that Vice-President.

If moisture can be *held* to 4 to 4½%, the 6-16 range *held* to 20%, and the dust held down there in Florida, we can ammoniate to 3.5 or 4% and still live with the "stuff."

Several years ago we made a few experimental runs using wet process phosphoric acid with a water content of approximately 30%. While this material was satisfactory in a 1-4-4 ratios, it did not lend itself to 1-1-1 ratios. Due to high water content, slightly better distribution was accomplished in 4-16-16 and 5-20-20 runs, but this was more than offset with the problems created when manufacturing 12-12-12. Trial runs using electric furnace acid have also been made but do to the higher water content and purity of the product, granulation was more difficult. We have concluded that if the water content can be held to 19%, the material is satisfactory, but water contents below 10% are desirable in some ratios. High analysis acids, with about 15% water are preferred in high liquid phase grades.

So long as we can pump the material out of the tanks, *purity* of the acid is relatively unimportant; however, we have received our share of tanks that have been returned, minus about 12 to 14″ of stand pipe, and as Joe Prosser would say "this is not good."

Since we do not have storage facilities for phosphoric, the problems of handling a tank testing 52% and one testing 55% are minimized, and with a few exceptions viscous material has not been a major problem.

Di-Ammonium phosphate was used experimentally a few years ago. and efforts were made to determine product breakdown at that time. According to results obtained, no appreciable loss was incurred; however, inputs were held to 300 lbs. per ton, and temperatures were not allowed to become excessive.

With regard to sulphate, we feel that there is one outstanding product produced in the east. It is, in our opinion the only consistently free-flowing material that we have used in our plants. Sulphate that has been kiln dried is usually superior physically to that which has been dried in a centrifuge.

In discussing standardization of solutions, we have assumed that reference should be made to the *number* of solutions on the market and *not* the differences in specifications between suppliers offering the same solution.

We like many other companies have experimented with a number of solutions. In the fall of 1958 and spring of 1959, we were using 440 (22-66-6). Following is a summary of our observations:

1. Given the same end-product moisture content, a 1-1-1 ratio made with a 6% urea solution reduces the pile set by as much as 50%.
2. 1-1-1 ratios can tolerate at least .25% more moisture and still produce less pile set than a straight nitrate solution. This, in our opinion, confirms experimental data with respect to the change in crystalline structure of ammonium chloride.

During that time we experienced a dilemma when drying the product: Breakdown of granules with too much heat, and without enough heat, too much moisture in the end product. This caused a shift back to nitrate solutions, and while we have not given up on the use of these solutions, we have, for the time being, discarded them until better control can be obtained.

We have used "essentially anhydrous" solutions and consider them superior to other solutions when granulating 1-1-1 ratios, but limited supply, at present, is a factor in their disfavor.

Since we're here to discuss standardization of products, we'd like to muddy the water a bit and suggest that a 433 (22-72-0) solution be produced for the Midwest. This solution could be used about April through September and be the summer component of 448 (25-69-0).

More standardization with respect to solutions is warrented, and many *obsolete* combinations should be eliminated; however, all of us are on the lookout for better combinations of ammonia, nitrate, urea, and water, and new solutions that are superior to former combinations should not be sacrificed for the sake of standardization.

Mr. Smith: Thanks very much, Ed. That gets off to a fine start bringing out before the group some of the specifics of this problem on the part of the mixed fertilizer manufacturers.

Our next participant in this panel is Eugene Reichard of the Robert A. Reichard, Inc., a mixed fertilizer manufacturer of Allentown, Pennsylvania, one of the progressive organizations of the northeastern area. Eugene.

Address By Eugene Reichard

Gentlemen of the Round Table: I am going to do better. One fellow took thirty-five minutes, he took fifteen, the second fellow, and I think I will take about five.

Through some correspondence Mr. Marshall insisted that I get on the panel, so I hope I can answer his question. We represent a small outfit in Allentown. We do most of our business in eastern Pennsylvania and western New Jersey. I think we are a little unique in this group; in that we have produced three types of fertilizer: one is the conventional powder, full granulation and the third, the nasty word, complete liquid fertilizer. With these three we have quite a few problems in connection with raw materials.

As far as the solutions are concerned, and that is our main source of nitrogen in our powder and granular fertilizers, we have standardized on the ammonium nitrate ammonia solution in the summertime mainly because of our humid conditions and also in the summer months our main production is in the lower brands of nitrogen like 5-10-10, for example. In the wintertime we swing over to the ammonium nitrate ammonia and urea solutions which we feel give a little better granulation. As I said before, because of humid conditions in the summertime, we go back to the ammonium nitrate ammonium solution.

Our big problem, and I think as with most of the small manufacturers, is the lack of control of incoming tests, especially on superphosphate. As most small plants, we do not have the storage to store several hundred tons in an ammonium bin and take a test, and then the next bin, we have to take it from current use, especially in our heavy mixing season. Consequently—by the way, we do not make our own superphosphate, we buy it, mainly from Philadelphia or Carteret, New Jersey, which means that coming in truckloads, and most of the triple superphosphate comes by truck—we do get some by car, and in the car, of course, we have a little better chance for control because the car is tested. The trucks are also tested but of necessity perhaps of the supplier we do not get our tests until possibly three days or a week after we have the truckload coming into the plant. We try to average out the tests, but, as can be understood, if we are mixing a heavy schedule and a truckload comes in in the morning and we need it in the afternoon we cannot wait for a sample, we have to keep the plant going. So with most small fellows this is a real problem in that respect.

I know suppliers would have a problem of giving us a guaranteed analysis, but they also must understand that we have a problem trying to use it with such a difference.

Mr. Smith brought out the fact about the control officials. I would like to have them come into our plant and run it for about a couple of weeks and then go back and see what they have to say about it.

Pennsylvania is not too bad, they give us a little leeway, but you all know in New Jersey they are rough on tests. Naturally we have to protect ourselves by, let us say, over-formulating.

On these nitrogen solutions we formulate about three tenths of a per cent over, which pretty well takes care of us, and our controls of our tests coming back from the chemists are pretty good. But on the acids and the potashes, we tend to be over just in safeguarding ourselves because we cannot take a chance on a ton of a particular fertilizer which is produced and sold. Maybe it is sold in Georgia, maybe it is sold in Pennsylvania; we do not know. So naturally we overformulate and that, of course, costs money.

On the potash, the difference is not too bad, but nevertheless we have to stay on the high side—or on the low side, rather, to protect ourselves, and when the high potash comes in, of course, there again we cannot keep the potash separate, our storage will not allow it.

We took some tests of superphosphate samples from three suppliers of normal superphosphate from whom we get it and for six months, from January to July, 1960, it ran from a low of 18.60 to a 19.13, which is .45 per cent per unit.

B ran from a low of 19.24 to a high of 20.67.

C ran from a low of 19.45 to a high of 19.95.

The first two were bad. The worst of all was the triple.

The triple ran from a low of 44.74 up to a high of 48.31. We had actual cases where from day by day deliveries it ran as high as 2.67 difference on a unit basis.

We have a full granulation system; that is. we take all the raw materials and mill them, screen them before they go into the batch weighing tank, then from the batch weighing tank to a rotary mixer, pre-mixer, over to a bulk scale, and the liquid materials are all metered. The phosphoric acid we use in the heavy meters gives pretty good control. I think you can all understand, especially the suppliers, that we cannot change our bulk scale every hour or every day, it is hard to keep set and when you get it set once the way you want it, you do not want to change it, because your acid changes on you. Nevertheless, this is our problem and we are hoping that some time in the future this can be straightened out. I think it does cost most of us a few dollars to keep our tests up where they should be.

On the phosphoric acid that we use, fortunately our supplier of acid, I think the trade agrees, is one of the better ones, but we do have a sludge problem. The sludge problem does not affect our product, it affects our storage. Being a liquid fertilizer business, we have learned the best type of tanks to use. In our granular plant, of course, we have a flat bottom vertical tank, and that is the fellow that gives us trouble. We have electrically, from the pipeline into the building, into the stainless steel pumps, wired insulated pipe line which keeps the acid warm coming into the plant, but the sludge does collect. Down in Newark in the plant we have horizontal tanks. Horizontal tanks—by the way they are all rubber lined—are all right, the only thing is that you have to get down in boots and shovel it out, but at least you can get it out, whereas a straight vertical bottom tank you cannot.

We find that the cone bottom vertical tank is the best one to use in liquid phosphoric acid storage. You tap out your acid where the cone starts, at the bottom of the cone you have probably a three or four inch valve to clean out, and when your foam car fills up with insoluble matter and the time comes, you see, that you have to get into your tapoff pipe, you just tap off the insoluble matter then what do you do with it? Can't you work it back in the product? Well. I guess you could, but we just like to take it out and get rid of it, put it in the dump.

We think the vertical storage tanks with the cone bottom are the best storage for phosphoric acid.

On the potash, of course, we naturally use the standard red type of potash in our dry mixes; in the liquid, of course, we have to use the 100 per cent water soluble white potash, the red potash will not dissolve in liquid fertilizer. That is as far as I can help you. I told Mr. Marshall, we do not have any scientific department and graduate chemists; we just turn out a couple of tons of fertilizer every once in a while.

Mr. Smith: Thank you, Eugene. That continues on our expression here of genuine problems which are confronting all of us regularly.

Our next participant in the panel is one of the more colorful as well as competent figures in the mixed fertilizer industry, our good friend Gus Mautner of the firm of Baugh & Sons of Baltimore, Md.

Address By Gus Mautner

Gentlemen, I have not prepared anything specific, but in the discussion that we had prior to this meeting we felt that on the subject of uniformity and product designation the people who supply our materials should give us enough information which would be at the plant upon receipt of the car so that we at least would know what we were using and make the necessary adjustments and changes in our granulating plant. In other words, if you know the potash is high in the plus 10 material you might not want to make a certain grade with that. Maybe you would be better off, say, instead of making 8-16-16 to make 4-16-16, and maybe you can adjust to the materials, especially in small plants of which we have a few where the storage is limited and we work out of cars in a lot of them. I have seen—everybody seems to have it in for the triple boys, so I will sink that harpoon in too. The prime example is we had two cars that came in from a supplier who shall be nameless and the one looked like gravel and the other one, as Ed says, I wish they had left in Florida, the dust was so bad you could not see the outside of the plant, much less inside.

It is things like that that not only irritates me but they irritate my people too, because, after all, just because you work in this stuff you don't have to eat it.

I am going to make a suggestion on what perhaps the potash people and the triple people can do something like the solution people have done. In other words, regardless of what they call this stuff, in back of that fancy name is in parenthesis 34-60-0, 22-65-0, so you know what the composition is regardless of what they call it.

If the potash people, when you specify—now they have coarse, special granular, all sorts of mishmosh like that. If they would—if you want potash of a certain size, all right, you get a potash, say, 10-20 which means that it would run maybe 90 per cent minus-10 plus-20. Now, if all the potash people did that, then you would know when it came into your plant what you were getting.

Another peeve I have is that they will send you — now, this is what we're making, you see. So you run screen tests on it. Unfortunately, we have to use those miserable Tyler sieves—with the unmentionable wire, and so the first two cars met the specifications beautifully. In other words, our specification was 10 per cent plus 10 and not over 15 per cent minus 28, which we got. Then the next car the plus 10 was up to 15 per cent, the next car was up to 25 per cent, and it finally was up to 40 per cent. So we wrote them a nasty letter and probably it was filed in 13, in the wastebasket somewhere. But the next car was all right, you see. You have to bellyache. You have to crab continually—otherwise they foist all this miserable stuff and all their mistakes off on you and we have to use it whether we like it or not. But I think that first, instead of our trying to tell the producers exactly what we have to have, because what he needs in his plant, what Bill Jones needs in his plant, and what we need in our plants are not the same, believe me. You cannot even put the same material in your own plants, because one operates one way and one operates another.

I think that what they should do is to tell us what is in the cars and make some effort to—I know that when you ship out run a pile of stuff you can get most anything in it, but they should be a little more careful, I think. I think it would help us all. We have enough trouble with the stuff as it is. Even if it is the right size you are using, you have trouble.

I think I have talked too long already.

Mr. Smith: Thank you, Gus. You see what I mean by saying Gus is a colorful figure as well as a very competent one. You have added considerably to getting this problem out on the table.

Our next participant is Mr. Grayson Morris, Production Manager of Southern States Cooperative at Richmond, Virginia. Grayson.

Address by Grayson Morris

I am going to confuse the issue, I'll just read an extract from one of the Department of Agriculture Units in one of the states in which we operate and they say this, that one of the most significant developments in the fertilizer work was the increase in plant food violation over 1950 to '59. There was an increase of 44 per cent reflecting 8.6 per cent of samples collected compared to 6.3 the previous year. The explanation is selective sampling of those brands and grades which have shown a higher percentage of violation during preceding months. The increase in sales of high analysis grades has created more opportunity for selective sampling and, unfortunately, manufacturers are experiencing more difficulty in manufacturing these grades. The figures also show a substantial increase in amount of penalties assessed and percentage refunded to purchaser. Under the selective sampling program, samples are collected in proportion of percentage of violation, rather than in proportion to volume sold, therefore it is not logical to conclude that 8.6 per cent of all fertilizer sold is deficient. Many of the grades sampled represent a small percentage of the tonnage, yet it is our responsibility to locate these violations.

Now, if all of us could bypass those people and get it to the farmer, he will bury it and we wouldn't have any trouble.

What these other fellows are saying is that we should have a standard to go by and the greatest problem that we have today is segregation, whether we like it or not. We need more integration and less segregation.

Mr. Smith: Thank you, Grayson.

Now we will have a summary statement by the final member of our panel Mr. Bill Jones of Northwest Cooperative Mills in St. Paul, who will give us a summary statement for the panel. Bill.

Address By W. E. Jones

This is it; you're looking at the anchor. I hope that when I am through—as most of you know the anchor is supposed to be a holdback device, and I hope that some way we may have stimulated a little bit of thought.

Ever since man came out of the boondocks and stopped living his hermit type life he has found as the groups grew larger some sort of rules of behavior, regulations for living have to be found. It is in this spirit that we hope to have accomplished something today.

It has been brought out very ably that half of the group of fertilizer consumers and producers, if you will, have some rule, some protective device, legislation, in their favor. I say to you that we who represent the production people do not have such a similar device. I think it is time for us, and particularly you who are producers of prime supplies for this industry, to sit down and hold for yourself a constitutional convention, if you will, and come up with some sort of rules of the road by which you as an industry can live and from this tell us standards that we as consumers of your materials can expect to be adhered to when we buy your products.

Probably one of the biggest problems that we have in this aspect is the language barrier. We do not seem to agree, to cite the biggest example — somebody says mesh. We ought to agree on what we mean when we say mesh, and let us then all use the same definition. These are some simple things. There should be no conflict. When you say mesh, and I do not care whether you say it in Washington or Florida, it ought to mean the same thing to both the sayer and the listener.

We have a communication problem, because of this language barrier. Analytically, we have come a long way in some phases with modernization of our analytical techniques to bring them up-to-date. This has been brought out, and this also is a problem.

I am sorry, but I too, I guess, am going to throw a little dagger at the phosphate industry. When I make a 5-20-20 and sell it, if I put more than 5-20-20 in it, you know who pays for it; if I put less than 5-20-20 in it, some guy taps me on the shoulder and it winds up the same guy is doing the paying again.

Now, you tell me that you are going to ship to me 46 per cent triple, and then all of a sudden I see an invoice and, not really, but if you read between the lines, there is a phrase therein that says, "Oops, we slipped, it's 47 and, Buddy, we gotta bill you for it." Maybe this should receive some consideration. Maybe some of the people and the the phosphate people should sit down together, decide what specifications you can in your own plants realistically make and tell us what those are and we will live with them, but let everybody tell us the same thing. Do not tell us something you cannot do, and what you do tell us you are going to do, do it.

I wholeheartedly concur with Gus, get a damn Vice President out to look at it, by all means.

Do we need so much exotic phraseology? Names. You know we're getting to be worse than Revlon. Yes, my lady's lipstick. We have more names in our industry going on right now and almost monthly it seems we are finding more. Why can't we get together? Potash, phosphate people, as an industry, come up with a name and have the name synonymous with a specification, or skip the name entirely and simply give us a specification.

Last, but not least, most of the people who are the raw material customers of the raw material producing industries are reasonably pleasant people. Keep us that way. Don't make crabs out of us, we don't want to be that kind of people.

In conclusion, all that we have hoped to do, and I know certainly on behalf of our panel we would not at all presume to be able to be knowledgeable enough to speak for all segments of this industry, but we just hope that by presenting to you a few little isolated problems that we can get some orderly system on the part of our great supplier faction and come up with some uniformity, because we are to the point now, as the analyses of the materials grow, where we no longer can live with a haphazard system as it presently exists.

Thank you very much.

Mr. Smith: Thank you, Bill.

There you have it, gentlemen, the expression of this Committee or this Panel this morning. It may be disappointing, possibly, to some of you in that this panel has not come up with specific standards, particular materials, specific screen analyses, standards for these same materials, but this group is only a panel, a representative panel of the mixed fertilizer industry and, as Bill has indicated, we have not thought that we had license to go that far. It has been our mission, as we understood it, to bring this problem out before the group, and I hope that it has been brought out before the group.

Just a word of summary. I might just repeat our need as I see it today and I think has been amply brought out and was expressed earlier, but let me repeat, is for specifications, for uniformity within given tolerances realistically arrived at, that is, information on analyses and methods and uniform or similar terminology. Specifications, uniformity within tolerances, information on analyses and methods and uniform or similar terminology.

You have had this expression from the panel, gentlemen, and we have a few minutes and I am sure that other members of the mixed fertilizer industry have either questions for the panel or supplementary statements which they would like to make, likewise there may be, and undoubtedly are raw materials producers who have questions which they would like to pose to this panel at this time. We would welcome them now. Who has the first one? Who has a comment they would like to make? Gus?

Mr. Mautner: In regard to state control, in the State of Indiana and the State of Ohio they made a survey in which they rated all of the companies based on the number of deficiencies that they had. They also took the entire grade, like a 12-12-12 and lumped all the tests together and came up with a percentage of deficiencies. This might shock you, gentlemen, but in the State of Indiana 50 per cent of the 12-12-12 samples that they tested were deficient in nitrogen; 80 to 85 per cent of the 16-8-8s were deficient in nitrogen. The industry average was 25 per cent of the samples tested for the companies were below guarantee, and some companies had as high as 85 per cent up to test, others had down to only 30, 35 per cent. Those are not outfits from which they take three or four samples a year; there are hundreds of them. Unless we do something about it, they are going to do it for us. As far as state control, I do not know how many people here operate in North Carolina. We have a plant to which we ship a considerable amount in North Carolina and the State of North Carolina takes anywhere from six to seven hundred samples a year of our fertilizer, and, if they get one that is bad, the order goes out to all inspectors to hunt and track down every lot of that particular grade they can find and sample it. When you pay the penalties on that – of course, fortunately, they use such red hot grades as 4-9-3 and 3-9-9 and it isn't too hard to meet your specifications, but if you were shipping 6-24-24 and 6-24-12 down there, you would be broke inside of six months.

Which again points up the fact that we do not know what we are using to start with and you do not have time to look around either.

MR. SMITH: Thank you, Gus. Gentlemen, as I said, this meeting is open to questions, or supplementary statements. Questions or statements on the part of mixed fertilizer manufacturers or questions from raw materials producers are in order for the panel before us. Who has the first one, and let me ask that any who are near those mikes out there use them, otherwise you will have to speak up as well as possible.

Who has the first question? Don.

MR. DONALD WARREN (B-I-F Industries, Omega Division): I would like to direct this to Mr. Reichard. Which materials are used in the construction of the phos acid tanks you are talking about? You mentioned some cone shaped tanks for the storage of phos acid, what material is it constructed of?

MR. REICHARD: It is a mild steel, rubber lined.

MR. WARREN: When you scrape these deposits or drain these deposits, have you had any problems with material coming loose?

MR. REICHARD: These tanks are in Harrisburg, and the Allentown plant is now producing granular fertilizer and that is the vertical flat bottom tank, but the horizontals with the cone bottom we have been using with phos acid but the pressure of the acid will push the sludge down to the bottom of the pipe to the bottom valve, the feeder valve.

MR. WARREN: It is not the type of material that sticks to the inside of the tank?

MR. REICHARD: No.

MR. SMITH: The question concerned the phos acid tanks. Who has the next question or statement?

There must be something. Surely this panel has not exhausted this topic by any means, it was intended just to open it up?

MODERATOR MARSHALL: Certainly everybody is not satisfied and happy. Let's have a few statements from you fellows on the floor. We only picked six and there are more manufacturers of mixed fertilizers than that. Questions? We don't want to have to run an auction sale to get questions. This is your chance. You fellows gave us questions, repeat those questions you wrote in a couple of years ago.

We have just about finished with the users' side of this story. The next section will be devoted to the raw materials producers and we, the Committee, have asked the raw materials producers to come forward and tell what they are doing and what they are delivering to the consumers and lay before this group their specifications as of now or in the near future and tell us, the users, what we can expect to get.

To start this out the Committee has divided this into the three major groups of materials used: nitrogen, phosphate and potash. Of course, in the nitrogen situation we have the liquid and we have the solid. I know that the ammonia and ammonia solutions association has done a wonderful job in getting together their information. There has been presented through the magazines tabulations on nomenclature as regards the nitrate ammoniating solutions and their composition. For the sake of getting all this information together in one place, we will ask you to bear with us and we will have a review of the liquid nitrogen components delivered to the users.

To start this off, I will ask Bert Tucker of Sohio Chemical to give us his tabulation that he has formulated and used. Bert.

Address By H. H. Tucker

Mr. Marshall, Mr. Chairman, Members of the Round Table: If I looked at the program right I thought this was supposed to be after lunch. I did have a slide or two upstairs that I was going to use, but I think I can get along without them. As I came up here a minute ago I met Joe Sharp in the aisle and Joe's comment was, "Take your time, I've got to go get my slides," so apparently he thought the same thing.

To get this started, as an introduction, I would just like to make a few statements. I have written this down so it will not take too long, and I would like to add some comments as I go along.

The comments I have written down are: standardization of raw materials is the theme of this panel. We have heard already what some people would like to have. I have been asked to discuss nitrogen materials from this point of view. The use and types of nitrogen materials in fertilizer manufacture have changed very rapidly with changes in technology and manufacture. The low nitrogen grades of fertilizers of years past relied primarily on ammonium sulfate. This was a fairly standard product and has remained standard throughout the years except for improvement, as far as moisture content is concerned, particle size, and the resultant physical properties that are connected with these improvements.

Undried sulfate was guaranteed at 20.5 per cent while newer improved products take advantage of the lower moisture content and are now mostly guaranteed at 21 per cent.

Aqua ammonia, once used in the fertilizer manufacture, known as B-liquor, very largely has been replaced by nitrogen solutions. Nitrogen solutions have certainly experienced great changes through evolution processes since their first inception in use. These changes have been rapid and are continuing. In fact, we heard a suggestion this morning as to a new solution that was needed in the midwest. Nitrogen solutions producers are plagued with producing too many solutions, often with very nominal differences, much as the fertilizer industry, fertilizer manufacturers are plagued with an excess number of fertilizer grades, often with very slight variations or differences.

Some ten years ago only four ammonium nitrate ammonia solutions were in general use and only two urea ammonia solutions were produced. That right now is in the order of 20 to 22, 25, depending on which area of the country you are operating in.

The ammonium nitrate ammonia solutions were numbered or named by varying companies as solutions: 2, 3, 4 and 6, or A, B, C, and D, respectively. This first brought about the need for standardization of nomenclature, standardization of naming. This need increased as the number of solutions produced increased and the naming system became more confused. When our company came into the industry in '55, there were two main systems of numbering or naming, as was mentioned here, and in addition there had been a one, two, three, four. We tried to go along with the majority and finally there was some action taken.

Your industry, through the National Plant Food Institute, was instrumental in standardization of nomenclature which has been accepted by the majority of solutions manufacturers.

There has been some reference here in the meeting, in fact, yesterday there were solutions referred to as 41 per cent, and actually in production there are a number of 41 per cent solutions. There was reference to solution 4-40 or 44 per cent nitrogen material, likewise there are a number of these solutions as well.

The naming system developed including the nitrogen content of the solution in whole numbers and tenths, leaving out the decimal. In other words, the reference this morning to solution 4-40 with the other numbers in the bracket meant it had 44.0 per cent nitrogen. The naming system developed included the nitrogen content of the solution in whole numbers and tenths.

This was followed by the chemical analysis rounded off to the nearest whole number and expressed in the following order:

First, inside of a bracket would be the per cent ammonia, then the per cent ammonium nitrate and then the per cent urea. Thus a solution which was labeled or named 440 (26-66-6) would contain 44.0 per cent nitrogen, 22 per cent ammonia, 66 per cent ammonium nitrate and 6 per cent of urea. It is very easy from that to calculate the water content, because when you add your ammonia, the ammonium nitrate and the urea you come up with 94 per cent, the balance being water, in case the solution was not over-formulated.

I might add right there that in general over-formulations have not been charged for and under-formulations are in general reported and credit given.

Solutions containing additional salts, as was planned in this committee meeting, additional salts such as ammonium sulfate would show this percentage content in whole numbers following that of the urea content. Actually, what we have—that ammonium sulfate, if it were in there, would be in the bracket also, in the bracket of the name. This solution name, as you can see is in reality a pedigree, and like that of individual names supplies information as to the family and what might be termed as its genetic makeup.

In other words, if we see a name that says 440-22-66-6, we know that contains urea, we know it contains 6 per cent urea, we know it also contains ammonium nitrate and ammonia. That is really its genetic makeup, or, having been raised on a dairy farm, it is sort of a pedigree as we used to have pedigrees for Holstein cows, and still have.

Like all names, there is often need that they be shortened, thus people have developed the use of nicknames. I was talking with Mr. Crosley yesterday and his name being Edward, and, incidentally, we came to realize that we both have the same name that we do not normally talk about and somebody said to me, "I have known your name was Bert for a long time, but I did not know it was Hubert," well, there has been reason for a nickname, but I will not go into details as to why on that, so many black eyes took care of that.

The same is true of nitrogen solutions. Companies using the complete, correct name have found that their sales departments could not be bothered with such long versions which are time-consuming and often difficult to relay by telephone. In other words, if a customer would call up and say, "I want 440-22-66-6," the fellow on the other end of the phone would say, "What did you say?"

Some companies have accepted the nitrogen content of the solution as its nickname. In other words, 440-22-66-6 has come to be referred to in the trade as solution 440, which is its nitrogen content.

Thus, as I have just explained, 440 would be its nickname.

In practice, several different solutions would have this same nickname. In other words, there are about 4 different solutions that are currently sold that have 44.0 per cent nitrogen. They have a different makeup as far as their ammonia ammonium nitrate urea is concerned so in reality you need to use a whole name to be completely sure as to what you are ordering. In practice, the nitrogen companies, when somebody calls up and says I want solution 440 know pretty much which solution has been used in the past and we sort of check and make sure we are giving them the same 440 they have been getting in the past, or we use other types of nicknames which cannot be very well duplicated.

Another point which might need standardization. I think we have accomplished something on standardization in nomenclature, but another point which might need standardization, as far as nomenclature is concerned, is the field of physical properties of the solution. We all have our product cards which give information about these various solutions. Specific gravity, pounds per gallon and vapor pressure per square inch gage at a given temperature would seem to be accurately descriptive. The property normally reported as salting-out temperature, however, is reported rather ambiguously on most product cards. The salting-out temperature usually reported is actually the saturation temperature; it is not actually where it salted out or where you might expect it to salt out, but it is where you would probably have to warm it back up to put it back in solution once it were salted out.

The definition of saturation temperature could be the lowest temperature where no salts will form under any condition. The actual temperature at which a product will salt out is, of course, lower than the saturation temperature, depending on the degree of supersaturation experienced and, of course, cannot be given as a single figure for all conditions. So on the product card, in general, what is termed salting-out temperature is really the saturation temperature and I think maybe the industry could well consider changing their nomenclature and listing that as saturation temperature, not salt-out temperature which would give you a little better indication that this was the absolutely safe figure and that if the temperature went somewhat below that you may or may not get salting out, but at least you would have to bring it back to that point to get it back in solution.

The Nitrogen Industry therefore need well consider listing what is commonly called salting-out temperature as saturation temperature.

Much has been done by the National Plant Food Institute through the Chemical Control Committee on Standardization of Fertilizer Analysis. This group has a Subcommittee on Analysis of Nitrogen Solutions which is contributing much to the standardization of nitrogen products.

We now come to the subject of standardization of nitrogen materials through reduction in the number of solutions. You all have that problem on grades and you like to cut down the number of grades, but what might be done along this line through a reduction in the number of solutions and elimination of minor and insignificant differences in composition?

Reduction in the number of solutions manufactured will no doubt come with further improved technology, manufacturing technology of fertilizers, and the recognition of the savings which can be made by using specific solutions. The need for the three types of nitrogen solutions currently used, that is the ammonia-ammonium nitrate solution which Ed referred to here this morning as one that he felt he would like to have, the ammonia urea solutions and the combination or ammonia-ammonium nitrate solutions containing a small amount of urea will likely continue. In fact, you really have three families of solutions: ammonia-ammonium nitrate being one, ammonia-urea being another, and the combination or ammonium nitrate ammonia with a small amount of urea.

That urea normally is put in there for two reasons. One reason is that you can lower the saturation temperature of the solution by putting in a limited amount of urea, secondly, that urea may change the crystal structure of the ammonium chloride in the end product from a fern shaped crystal that will knit the product together to a cube shaped crystal.

Ammonium nitrate, ammonia-ammonium nitrate solutions have long proven themselves as desirable for nitrogen in complete fertilizers and as aids in granulation. The desirability of ammonia to ammoniate superphosphate was established even before the use of nitrogen solutions. Ammonia free ammonia urea solutions also have their advantages. Some companies particularly like the ammonia urea solutions. The use of urea in ammonium nitrate solutions, as I just explained, is primarily for changing the saturation temperature of the solution and, secondly, for some other advantages.

The reduction in the total number of solutions, then will likely come about largely through a reduction of the number of solutions within one of the categories, or one of the families, or within the families rather than a reduction or elimination of any one family or category.

This reduction in the number of solutions within a category will likely be the result of recognition within the industry that minor differences in the composition of solutions are insignificant as compared to variables of other materials used. You have heard some of the discussion on variables of other materials.

Variations in manufacture often cause greater differences in the manufactured product than do slight variations between nitrogen solutions.

Larger fertilizer manufacturers no doubt will tend toward the use, looking to the future, of one particular solution that probably fits best for their high nitrogen grade and then anhydrous ammonia and then perhaps they will blend the two materials together and make their own tailor-made solutions.

I think we are seeing some change, some standardization in use within the industry of solutions. I know that Mr. Adams of the USDA is making a study, a survey of which solutions are being used of the various nitrogen companies. Most of them, I understand, have sent him a compilation of about what percentage of their sales are in certain solutions. I hope that that is compiled and I understand it will be available very soon. But we in the industry, and our own company, and I know from talking with some of the other people, are beginning to see a trend toward some rather universal solutions that seems to be developing. In other words, some solutions are increasing in volume, others are beginning to pass out of the picture. It could be that Ed Kingsbury's solution that he mentioned this morning might walk in and be the universal solution; I do not know.

I think that about covers all I have to say.

Moderator Marshall: Thank you, Bert, for your discussion and now we will ask Joe Sharp of Spencer Chemical to come forward and give his nomenclature of ammoniating solutions that they use in the middlewest. Joe.

Nitrogen Solutions Used To Produce Both Powdered And Granulator Fertilizer Mixtures

Joe C. Sharp

Ammonia, Ammonium Nitrate Solutions

TABLE I lists fifteen combinations of nitrogen solutions made by dissolving ammonium nitrate in ammonia and water. The fifteen solutions listed do not cover all the combinations now on the market. It does cover a wide range and gives sufficient information to allow easy interpretation for any other combination.

The only thing complicated, or that gives the impression of being complicated about these solutions, is the number of them and the vast amount of data listed in the the tables put out by the producers of them.

Ammonium nitrate is soluble in both ammonia and water. It is more soluble in ammonia than it is in water. When a lot of ammonium nitrate is dissolved in a small amount of ammonia and water, the solution must be kept hot to prevent the ammonium nitrate from salting out. On the other hand, if a small amount of ammonium nitrate is dissolved in a large amount of ammonia and water, salting out of the ammonium nitrate is not a problem. Liquid anhydrous ammonia exerts a vapor pressure of about 212 psig at 104°F. This volatile compound is responsible for the vapor pressure shown in the vapor pressure column in the table. Water and ammonium nitrate lowers the vapor pressure of ammonia with water exerting the greater affect on a pound for pound basis.

The nitrogen content of ammonium nitrate is 35%. The nitrogen content of ammonia is 82%. Solutions that contain large amounts of ammonium nitrate and small amounts of ammonia have relatively low nitrogen contents. Conversely, solutions that contain small amounts of ammonium nitrate and large amounts of ammonia, have relatively high nitrogen contents, unless the solution is diluted.

To illustrate what I have just said, the sequence of listing the solutions in Table I was based on the percentage of the total nitrogen derived from ammonia. The smallest percentage (least ammonia) is listed first.

Since ammonia must be reacted with an acidic material to form a nonvolatile compound, the ammonia content of the solution used must be related to the acidic materials in the fertilizer formula. Fertilizers with high P_2O_5 content derived from superphosphate and phosphoric acid make it possible to use high ammonia nitrogen solution. For high nitrogen, low P_2O_5 fertilizers, a low ammonia, high ammonium nitrate solutions is generally selected. Exceptions are when chemical drying is required and sulphuric acid is used to neutralize ammonia and develop heat, or when high ammonium nitrate containing fertilizers are believed to cause unsatisfactory mechanical condition of the finished product.

Ammonia, Urea Solutions

Table II lists six combinations of nitrogen solutions made by dissolving urea in ammonia and water. The same sequence of listing was used as with the ammonium nitrate solution in Table I. Because urea is not as soluble in ammonia and water as ammonium nitrate is, it is not possible to make a solution containing as high a percentage of nonvolatile nitrogen in this type solution as it is with the ammonium nitrate solutions with comparable salting out temperatures. You will note that the solution at the top of the list on this table has 55% of the total nitrogen derived from ammonia whereas the top of the list of Table I has only 36.9% of the total nitrogen derived from ammonia. The carbon dioxide in some of these solutions lowers the salting out temperature and replaces water. The carbon dioxide tends to increase the vapor pressure.

Ammonia, Ammonium Nitrate, Urea Solutions

Table III lists eight nitrogen solutions made by dissolving ammonium nitrate and urea in ammonia and water. These solutions are more complicated from a salting out standpoint, because of the formation of double salt eutectics, etc. Because of these properties it is possible to get a high percentage of nonvolatile nitrogen in these solutions at favorable salting out temperatures.

In addition to this advantage, the urea in these solutions changes the crystal structure of ammonium cloride and potassium nitrate, which is formed in mixed fertilizers when the formula contains muriate of potash and ammonium nitrate, from long needle and fern-like crystals to cubical crystals which tends to reduce caking of fertilizer.

Table IV shows one solution containing urea and sulfate of ammonia dissolved in ammonia and water. In addition to the conditioning effect of the sulfate of ammonia, it also lowers salting out temperature as compared to a solution containing only urea as a solute.

Table V lists two specialty solutions. One containing ammonium nitrate, urea, and formaldehyde and carbon dioxide dissolved in ammonia and water. These solutions form water in-soluble nitrogen when reacted with superphosphate. Their main use is for tobacco fertilizers and lawn and garden type products.

Conclusion

The thirty-two solutions listed in the accompanying tables do not begin to cover all of the combinations that have been made and used. Many nitrogen solution producers will custom make solutions to specifications requested by the user. Based on our companies past experience, the solutions listed probably cover 98% of nitrogen solution consumption, for use in producing solid fertilizers. This does not include solutions used for the production of liquid complete fertilizer.

Table I. Ammonia, Ammonium Nitrate Solutions

		Composition by Weight					
Solution Name	Free Ammonia Nitrogen % of Total	Ammonia %	Ammonium Nitrate %	Water %	Specific Gravity 60°F/60°F	Vapor Pressure psig at 104°F	Salting out Temperature °F
370 (17-67-0)	36.9	16.6	66.8	16.6	1.182	3	48
414 (19-74-0)	37.7	19.0	74.0	7.0	1.198	8	64
462 (24-76-0)	42.8	24.0	75.0	0.5	1.162	25	19
466 (25-75-0)	44.1	25.0	74.5	0.5	1.150	29	5
440 (24-70-0)	44.5	23.8	69.8	6.4	1.151	17	26
410 (22-65-0)	44.5	22.2	65.0	12.8	1.142	11	21
471 (26-74-0)	45.4	26.0	73.5	0.5	1.137	31	—9
448 (25-69-0)	45.9	25.0	69.0	6.0	1.124	17	6
490 (30-70-0)	50.0	30.0	69.5	0.5	1.094	44	<—30
470 (30-64-0)	52.0	29.7	64.5	5.8	1.089	34	—30
410 (26-56-0)	52.6	26.2	55.5	18.3	1.079	15	—14
509 (34-66-0)	54.8	34.0	65.5	0.5	1.055	57	<—30
490 (34-60-0)	57.1	34.0	60.0	6.0	1.045	48	—49
585 (50-50-0)	70.4	50.0	49.5	0.5	0.930	144	<—30
530 (49-36-0)	76.0	49.0	36.0	15.0	0.907	104	—99

Table II. Ammonia, Urea Solutions

		Composition by Weight					
Solution Name	Free Ammonia Nitrogen % of Total	Ammonia %	Urea %	Water %	Carbon Dioxide %	Specific Gravity 60°F/60°F	Vapor Pressure psig at 104°F	Salting out Temperature °F
455 (31-0-43)	55.0	30.5	43.3	16.2	10.0	1.064	52	34
453 (31-0-43)	55.5	30.6	43.1	26.3		0.972	47	45
528 (36-0-50)	56.0	36.0	50.0	14.0		0.952	95	28
455 (37-0-33)	66.4	36.8	32.5	23.3	7.4	0.999	61	5
555 (45-0-40)	66.5	45.0	40.0	15.0		0.910	113	10
454 (37-0-33)	66.7	36.8	32.5	30.7		0.926	57	16

Table III. Ammonia, Ammonium Nitrate, Urea Solutions

		Composition by Weight						
Solution Name	Free Ammonia Nitrogen % of Total	Ammonia %	Ammonium Nitrate %	Urea %	Water %	Specific Gravity 60°F/60°F	Vapor Pressure psig at 104°F	Salting out Temperature °F
414 (19-66-6)	37.7	19.0	65.6	6.0	9.4	1.186	8	36
410 (19-58-11)	38.1	19.0	58.0	11.0	12.0	1.160	9	7
430 (20-68-6)	38.2	20.0	68.0	6.0	6.0	1.165	22	39
440 (22-68-6)	41.1	22.0	66.0	6.0	6.0	1.149	27	14
444 (25-55-10)	46.1	24.9	55.1	10.0	10.0	1.114	22	—20
444 (26-50-12)	48.1	26.0	50.0	12.0	12.0	1.086	25	—7
440 (28-40-12)	52.3	28.0	40.0	12.0	17.0	1.053	26	—1
490 (33-45-13)	55.4	33.0	45.0	13.2	8.8	1.034	44	—10

Table IV. Ammonia, Urea, Sulfate of Ammonia Solution

				Composition by Weight				
Solution Name	Free Ammonia Nitrogen % of Total	Ammonia %	Urea %	Sulfate of Ammonium %	Water %	Specific Gravity 60°F/60°F	Vapor Pressure psig at 104°F	Salting out Temperature °F
430 (28-0-39-10 S)	52.5	27.5	39.3	10.0	13.7	1.126	49	20

Table V. Specialty Solutions

		Composition by Weight							
Solution Name	Ammonia %	Ammonium Nitrate %	Urea %	Water %	Formaldehyde %	Carbon Dioxide %	Specific Gravity 60°F/60°F	Vapor Pressure psig at 104°F	Salting out Temperature °F
370 (13-53-16)	13.0	53.4	15.9	9.4	8.0		1.235	0	36
370 (25-0-35)	25.0		35.3	23.3	8.0	8.4	1.090	—1	34

Moderator Marshall: Thank you, Joe Sharp.

Now, does anybody have any questions regarding nomenclature or what-have-you on ammoniating solutions? Any questions going once, any questions going twice. I have a suspicion you are hungry.

If there are no questions, we will adjourn this session of the Round Table and take up at two o'clock with the discussion on Solid Nitrogens.

(The session adjourned at eleven fifty-five a.m.)

Thursday Afternoon Session, November 3, 1960

The Round Table reconvened at two o'clock p.m., Messrs. H. L. Marshall and Albert Spillman, presiding.

Moderator Marshall: We will continue the discourse on raw materials and what the producers have to offer to the consumer. Of course the producers want the consumers to think plenty of it, so they are going to tell us how good or how well they make their materials.

Continuing in that vein we jumped the gun a little bit this morning and got the two liquid nitrogen producers to come forward and give us their nomenclature. That takes care of the liquid phase of it.

Does anybody else want to discuss the liquid phase of the nitrogen situation, nomenclature-wise or chemical-wise?

I've asked you once; I've asked you twice; it's all over.

Now we'll get on to the solid nitrogen carriers and we have the good fortune to have gotten Dr. Waters, Allied Nitrogen Division to present some information regarding the products that they produce and possibly some products, specifications of products that he has been able to get hold of.

Dr. Waters, please.

Solid Nitrogenous Fertilizers

C. E. Waters

The subject assigned to me on this Nitrogen Producers' Panel is "Solid Nitrogen Carriers." I have decided to discuss the various materials without particular regard to whether they are used primarily for mixing or for direct application. The various grades of ammonium phosphate are purposely left out because they will be considered by the Phosphate Panel.

The latest Department of Agriculture Report on "Consumption of Commercial Fertilizers and Primary Plant Nutrients in the United States, Year Ended June 30, 1959" gives an idea of the amounts of nitrogen involved. According to this report, about 2,672,000 tons of nitrogen were used in the United States during this year, including a million tons in mixed fertilizers. No breakdown of the million tons is given, but it is believed that ammoniating solutions formed the most important single source, with ammonium sulfate in second place.

Slightly over half of the 1672 thousand tons of direct application nitrogen were provided as solids, divided as follows:

Material	Tons of Nitrogen (Thousands)
Ammonium Nitrate	429
AN-Limestone Mixtures	69
Ammonium Sulfate	110
Calcium Cyanamide	9
Calcium Nitrate	8
Sodium Nitrate	78
Urea	50
Phosphate Products	83
Potash Products	3
Other Chemical N Carriers	3
Natural Organics, including Bonemeal	15
	857

The main effect of including solids used in fertilizer mixing would undoubtedly be a substantial increase in the relative importance of ammonium sulfate.

The principal solid nitrogen carriers are thus seen to be ammonium nitrate and ammonium nitrate-limestone mixtures, ammonium sulfate, sodium nitrate, and urea, plus ammonium phosphates, which will be considered later. At the request of the Secretary, a table has been prepared to show certain properties of these materials, especially in the forms produced by my company. This will be available when the proceedings of the Round Table are written. The following remarks give highlights from the table, together with additional information about the materials.

Ammonium Nitrate

Pure ammonium nitrate contains 35.0% nitrogen, but for fertilizer use the pellets are usually coated with finely divided mineral, such as kieselguhr or kaolin, which reduces the nitrogen content to about 33.5%. No other commonly used solid, except urea, even approaches this figure. Half of the nitrogen is present as nitrate, half as ammonium ion.

To minimize caking in storage the moisture has to be kept low, a suitable value being 0.3%. As for screen analysis, a typical sample will have a few percent retained on 8-mesh sieve, over 90% passing this sieve but retained on a 20-mesh, and practically non passing a 35-mesh sieve. The majority of manufacturers produce ammonium nitrate as substantially spherical particles, commonly called prills.

Because it is so very soluble in water, and even more soluble in ammonia, ammonium nitrate is the principal solid nitrogen compound in the majority of ammoniating solutions. It is also supplied in solutions containing substantially no free ammonia, used mainly for direct application and manufacture of liquid mixed fertilizers. Of course, ammonium nitrate in such solutions is outside of the scope of this discussion.

Whether added as pellets or as a constitutent of the ammoniating solution, ammonium nitrate has particular value in granulating mixed fertilizers. The basis for this is the very high rate of change in water solubility with temperature. At working temperatures it will dissolve in much less than its own weight of water, thus forming enough liquid phase for granulation. As the mixture cools, most of the ammonium nitrate crystallizes, converting each soft pellet into a hard, rigid unit. In some mixtures the ammonium nitrate reacts with potassium chloride, forming potassium nitrate and ammonium chloride. Fortunately potassium nitrate also has a steep solubility-temperature curve, and acts somewhat the same as ammonium nitrate itself.

Ammonium Nitrate-Limestone

A typical product contains 20.5% nitrogen, and comprises about 60% ammonium nitrate and 40% mineral matter, the latter mostly calcium carbonate or dolomite. With these proportions the material has practically no equivalent acidity or basicity. Besides, it furnishes calcium, needed in some soils, and if made with dolomite it also furnishes magnesium, which is frequently in short supply.

One familiar product is made with dolomite, and has a guaranteed nitrogen content of 20.5%. It also contains 4.2% magnesium (7.0% magnesium oxide) and 7.5% calcium (10.5% calcium oxide). A typical moisture content is about 0.35%. Practically all of the pellets will pass through an 8-mesh sieve, yet about half is retained by a 10-mesh sieve; only a fraction of a percent will pass through a 35-mesh sieve.

These ammonium nitrate-limestone products are intended primarily for direct application, but may occasionally be of service to the fertilizer mixer.

Ammonium Sulfate

The theoretical nitrogen content is 21.2%, but the commercial product contains less, because of impurities. A nitrogen content of 20.5% is commonly assumed for the by-product material and 21% for synthetic. The nitrogen is all in ammoniacal form. From an agronomic point of view, the only drawback is the relatively high equivalent acidity, corresponding to somewhat over 100 pounds of calcium carbonate per unit of nitrogen. Ammonium sulfate contains 24% sulfur, an element that is essential to plants, and is missing from some highly concentrated fertilizers.

Ammonium sulfate is marketed in a variety of physical forms, depending upon the materials from which it is made, and the method used for converting it to salable condition. As produced by Allied Chemical, it is a crystalline product with a guaranteed nitrogen content of 21%. About 85% is in the size range through 12-mesh, retained by 35-mesh.

Although the relatively low nitrogen content is a disadvantage in high analysis fertilizers, ammonium sulfate is still the principal nitrogen compound added to mixed fertilizers in solid form. It is favored for its low hygroscopicity, and its generally good effect upon the condition of the fertilizer made with it. The solubility-temperature curve is quite flat, compared to that of ammonium nitrate.

Sodium Nitrate (Nitrate of Soda)

The pure salt contains 16.5% nitrogen, and commercially produced material will frequently run better than 16.3%. However, the common guarantee is 16%. This nitrogen is all in nitrate form. In contrast to ammonium sulfate, sodium nitrate has a basic effect in the soil, equivalent to 36 pounds of calcium carbonate per unit of nitrogen. The sodium has special value for certain crops, serving as a partial replacement for potassium. Complete replacement is not possible, however.

Sodium nitrate from natural deposits is imported from Chile, and the compound is manufactured for fertilizer use by at least two companies in the United States. In contrast to the other producers, Allied Chemical markets a crystalline product, the crystals being almost cubical. This product is marketed in three size ranges: *fine* (3A), with no more than 24% retained on a 28-mesh sieve, *medium* (2B), with at least 60% retained on a 28-mesh sieve, and *coarse* (1C), at least 95% of which is retained on the 28-mesh sieve. There is also an extra fine grade, seldom used in fertilizers.

Sodium nitrate is used for direct application, and also in certain grades of mixed fertilizer that are required to have particularly high proportions of their nitrogen in the nitrate form. For example, fertilizers intended for use on tobacco fields shortly after fumigation are required to have definite proportions of nitrate nitrogen. The coarse grade is favored for direct application, and the medium and fine grades are chosen for use in mixed fertilizers.

Urea

Urea has a higher nitrogen content than any other commonly used solid, the theoretical value being 46.6%. It has been marketed at various actual nitrogen contents ranging from around 42% almost to the theoretical value. By official definition of the Association of American Fertilizer Control Officials this is "Synthetic non-protein organic nitrogen."

Urea is sold in several forms, crystals, very fine pellets, and coarser pellets, which may be bought with or without a mineral coating. Special forms are sold for use in animal feed supplements. The table referred to previously gives information on one brand of crystal urea, as well as pelleted urea, coated and uncoated. The crystal urea and the uncoated pellets contain over 46% nitrogen; the coated pellets somewhat over 45%. Typical samples of the pellets run better than 95% in the range, through 8-mesh, retained on 28-mesh. The crystal urea is the finest of the materials discussed, nearly all passing through a 35-mesh sieve, and several percent passing through even a 100-mesh sieve.

The coated pellets are commonly used for direct application and for mixing. For making liquid mixed fertilizers, it is of course preferable to use uncoated pellets, or even crystal urea.

Urea is second only to ammonium nitrate as the solid nitrogen compound in ammoniating solutions. It is also used in nitrogen solutions that contain substantially no free ammonia. It has particular value in liquid complete fertilizers.

Urea is of value in mixed fertilizers because of its exceptionally high nitrogen content. It is also of special value in nongranular goods in making them less likely to cake. This anti-caking property probably depends partly upon the well-known effect of urea upon the crystal form of ammonium chloride made by reaction between ammonium nitrate and potassium chloride, and partly upon its preventing excessive drying of the fertilizer in storage. It is usually best to avoid making fertilizer that contains high proportions of both ammonium nitrate and urea, since the combination is likely to result in excessive hygroscopicity and stickiness.

Other Solid Nitrogen Carriers

This discussion has covered the solid nitrogen carriers that are of greatest importance from the standpoint of amounts of nitrogen supplied. It has been necessary to omit a number of other materials, both organic and inorganic, that are interesting, and also valuable in specific cases.

Properties of Some Important Solid Nitrogen Carriers

Material	Guaranteed Nitrogen %	Typical Moisture %	Typical Screen Analysis—Tyler Sieves Cumulative Percentage Retained On										Through	Through
			8	10	12	14	20	28	35	48	65	100	35	100
Pelleted Products														
Ammonium Nitrate	33.5	0.3	5	47	—	94	98	99.4	100-	—	—	—	Trace	—
Ammonium Nitrate-Limestone	20.5	0.35	3	51	—	74	91	93	99.5	—	—	—	0.5	—
Urea—Coated	45	0.35	2	30	—	70	97	99	100-	—	—	—	Trace	—
Urea—Uncoated	46	0.2	2	30	—	70	97	99	100-	—	—	—	Trace	—
Crystallized Products														
Ammonium Sulfate	21	0.15	—	—	10	—	79	92	96.5	97.5	—	—	—	—
Sodium Nitrate—Fine (3A)	16	0.3	—	Trace	—	Trace	Trace	8	43	81	95	100-	—	Trace
Sodium Nitrate—Medium (2B)	16	0.3	—	2	—	11	37	67	81	90	96	100-	—	Trace
Sodium Nitrate—Coarse (1C)	16	0.3	—	8	—	18	68	96	99.6	99.6	100	100-	—	Trace
Crystal Urea	46	0.25	—	—	—	—	—	—	—	30	62	92	—	8

MODERATOR MARSHALL: Thank you, Dr. Waters.

Before we open for questions, let's have Mr. Sedlack of U. S. Steel present his data that he has gathered on solid nitrogen products.

MR. D. F. SEDLACK (U. S. Steel): Gentlemen, I might say that I am quite glad to talk about solid nitrogen bearing materials because I believe that you will agree with me we do not have any nomenclature problem here.

I think that ammonium sulfate is always referred to as ammonium sulfate, ammonium nitrate as ammonium nitrate and urea as urea. However, that does not take away the fact that we as age old producers of ammonium sulfate and more recently ammonium nitrate woke up one morning and found out that we were presented with a — or confronted rather with several very important and real problems.

These problems were associated with the physical characteristic, particularly of ammonium sulfate as well as the chemical composition.

In the case of the physical characteristics of ammonium sulfate we were faced with two questions. What should we actually have and what we thought we should have.

We took these two questions to a consumer and solicited his preference. He, through his kind cooperation, gave us a preference, which preference we translated into a form of a specification, which specification was included in design. That design was the basis to make that type product which the consumer wanted.

That design was used to control particle size, so that ammonium sulfate that had been produced with 75 per cent through 30 mesh was changed to material that had 5 to 10 per cent through 30 mesh.

This type product enabled that consumer to use this type ammonium sulfate in either blending or mixing or direct application. However I would like to point out one thing at this stage. We used the consumer's advice to give the consumer what he wanted.

Today ammonium sulfate is produced in wide ranges so that you can make your selection. In the case of the chemical composition that was quite easy. All we had to do was neutralize a little better, dry a little better, resulting in three things.

The moisture content had a tenth of a per cent maximum, the acidity was practically nil and I must disagree with Dr. Waters, the ammonium content, or the nitrogen content is 21 per cent, not 20.5.

In the case of ammonium nitrate, it is common knowledge as to how it is mixed or how it is made, rather. It does have a very narrow range particle size; the guaranteed nitrogen content is 35.5% coated with diatomaceous earth to keep it from setting up and getting hard as a rock and that particle size consumer can select to fit his own needs.

Thank you.

MODERATOR MARSHALL: Thank you, Dr. Sedlack.

Now are there any questions about the solid nitrogen compounds that are offered for use in mixed fertilizer.

Do we have any questions?

Anybody once; anybody twice; it's all over.

Thursday Afternoon Session, November 3, 1960

Remarks by Albert Spillman

The excellent discussion this morning by "The Users' Panel" clearly and precisely covered the major problems and reasons asking for urgent, desired improved standardization of Raw Materials from the standpoint of Chemical, Uniformity and Nomenclature. The subject was thoroughly covered by all of the User Panelists, therefor, any additional comments that I can think of at this moment would be superfluous. I am confident all of you here, representing suppliers of materials, are desirous for your respective Company to produce and ship materials carrying specifications to do the most suitable job for our various manufacturing needs, therefore, you will recommend to your Management the importance for complying to the utmost possible to improve your respective materials to help alleviate the major problems brought out by the User's Panel. I commented yesterday that the Raw Materials Suppliers are definitely trying to help The User. Suppliers have at great expense organized Technical Services for our use and this phase of Technical Service, in itself, will have much influence with Raw Material Suppliers to improve Uniformity both Chemically and Physically. Our next Panel represents Phosphate Material Producers who will discuss their respective products, Viz: Single Superphosphate, Triple Superphosphate, Ammonium Phosphates and Phosphoric Acid. The Panelists are E. F. Carnell, Thomas J. Pearce, W. W. Harwood, Ray L. Jones, D. O. Walstad and H. P. Tatum. Our good friend Dr. W. L. Hill, U.S.D.A. Beltsville will give us his comments upon completion of The Phosphate Panel. The Phosphate Format is as follows: Presentation of product data, general comments regarding specifications, Uniformity as to physical, analysis, fines control, moisture variations, shrinkage factors, free and fixed water and dissociation of diammonium phosphate. Panel Chairman E. F. Carnell.

Address By C. F. Carnell

Thank you Mr. Spillman. I don't know whether this Panel ought to be flattered or frustrated. So many of the things that we have to present to you have already been touched on in various other talks that a lot of it is going to be duplication. We are under orders from Our Vice President not to be frustrated so we will count it as being flattered. At least it indicates that we are going pretty much in the same direction. Our formal talks covering the papers we have here will take only about 30 minutes and hope this will leave a lot of time for discussion. When we get thru we hope that any other producer representatives of phosphates present understand they are welcome to participate with us. Please save all of your questions until the end of this panel; also save all of your Tomatoes and Rotten Eggs and things for the last speaker because that will be Barney Tatum. Barney will be expecting. We appointed him to take that part.

I. Specifications of Materials Now Offered.

When we learned that we were to participate in a panel for standardization of raw materials it occurred to us that it might be well, if we are going to discuss what is required and what users want in the way of product specifiactions, to start out by showing the characteristics of the materials that are now available, or at least to consolidate as many of those that we can get together on one sheet so that we may have something in front of us as we talk.

To accomplish this, we sent a copy of our data sheets, along with a form letter to most of the major producers, asking them to reciprocate the information to us. The replies were summarized and the result is the tables that you are now being given.

A few general comments will be made in order to clarify some of the data on these sheets.

(1) As usually happens when information comes from 15 to 20 different sources, it comes in all different shapes and sizes — different companies feel that different things are important to show on a data sheet; hence one company will include the sulfate to show the benefit of the sulfur content as a plant nutrient while another feels its is important to show aluminum because of its effect on mechanical condition. For this reason there are blanks in the sheets that indicate that the information was not provided — not that the material didn't contain any of a given element or that a condition did not apply. Also, there is a wide variation between screen sizes used. To keep from confusing the sheets with too many specifications with litle data on each we used the screen

fractions that were most popular and expressed all the fractions as percent retained on.

To keep from identifying individual companies, we labeled the products "A," "B," and "C," etc. and I am sure you will recognize your own company's data as most of it is taken from published and widely circulated information. For the users of materials, I am sure your material salesman can point out the specifications on his material if they are shown here.

As you can see, this is certainly not offered as an all-inclusive list. Some companies did not respond to our request and we were forced to omit some of the information because it did not conform to the general outline of the sheets but it is believed to be a pretty good cross section.

After looking at all the different specifications on the same product offered by different companies, one wonders if an effort might not be well spent to go in the direction the nitrogen people did a few years ago and standardize our categories as a preliminary step to standardizing the product. After all, the large majority of specifications for raw materials now are based on two things: (1) What the captive mixed fertilizer plants of the various raw materials producers think they should be, or (2) what the technical service and sales people of the various producers find the needs in the customers' plants to be and, incidentally, as many of the developments and improvements in materials probably came from cooperating superintendents as were developed in the laboratory or pilot plant. So perhaps this end can be accomplished through these Round Table meetings which seem to be a mutual meeting ground for such problems. These suggestions are meant to apply to materials that require further processing, i.e., ROP Superphosphate, Phosphoric Acid, etc., not finished products designed for a specific purpose. These are supposed to be standard now.

Then, when we finally get specifications on a cut-and-dried basis, we can see what can be done to control the critical variables.

II. What Are the Variables That We Want to Control?

We would list them as follows, not necessarily in order of importance.

(1) The universal and one of the most important complaints is varying *analysis of APA,* posing a problem in storage and formulating.

(2) Important from a condition, formulating and shrinkage standpoint, is *moisture,* both combined and free. One important question that we have come upon which is out of the hands of the materials producer is what new compounds are formed on ammoniation and what are their water complexes?

(3) Free acid is also an important consideration to condition — especially in raw material handling.

(4) Trace element compounds, as well as causing difficulty in raw materials, carry right on through to the product forming gels and water systems that are least conducive to good handling and storing characteristics of finshed fertilizer. We wish to point out here that, while iron and aluminum phosphates are the ones generally credited with most of the trouble, they are by no means alone —Mg, Na, and probably others are important.

(5) Particle size. Many conflicting opinions appear in this area probably because it is so important to granulation and because so many different combinations seem to be necessary for different facilities.

III. What Can We Do to Get This Control?

It is chemically and physically possible to make fertilizer materials to any degree of purity and to any size specification that is desired. Triple superphosphate can be in the shape of aspirin tablets. It can be nonchalking, uniform encapsulated pellets, twelve to a package or sixteen as the need may be. The only drawback, as you all well recognize, is that this kind of processing costs money. On the other hand, if anyone of you would be willing to accept it we could ship you phosphate rock and sulfuric acid and you, in turn, could furnish these to your distributors and tell them to mix them in their fertilizer drills. They could get it cheaper that way but we don't think your customers would be satisfied with the condition and uniformity. So it boils down to how much control and processing are justified to provide a product that will give satisfactory qualities for shipping, storing and processing into mixed fertilizer of acceptable quality.

Some systems, of course, are more conducive to control than others—our Dorr system in which we make granular triple for instance, with its twelve to sixteen passes of product through a thin slurry of acid, rock mix and long retention gives a product that can be controlled to one-half unit of P_2O_5—still a cure is effected in the pile which results in variation if new material is shipped but even this accuracy is not ordinarily possible on run-of-pile because making run-of-pile is generally a one-shot proposition. You mix the acid and rock, den it, pile it and hope for the best—the best at this time usually meaning that the sales department can control shipments to the extent that a reasonable time can be allowed for cure.

There have been many points that we have been able to control to one degree or another—and these have been employed wherever possible—for instance, we learned soon after commencing the production of ROP triple superphosphate that agitation or plowing on the setting belt is an important factor in controlling porosity and density of the product — now this is applied in varying degrees to control structure, size and density. Incidentally, our experience has been that 20 to 25% minus 60 mesh material is preferred for a granulation plant that truly granulates while a less amount of fines is required for marginal systems and that brings us to another point—you want "standard materials" but by whose standards?

These we believe are the main items and I am sure my colleagues here will elaborate on various points in this presentation. These will be:

(1) Mr. T. J. Pearce of Swift & Co. will discuss uniformity of size.

(2) Mr. W. W. Harwood—I. M. C. will discuss uniformity of analysis; also shrinkage as it relates to free and fixed water in materials.

(4) Mr. R. L. Jones of Armour will discuss moisture variations in phosphate.

Mr. Dan Walstad of American Cyanamid will go through his plant on a flow sheet and show you some points that can and cannot be controlled.

Mr. Barney Tatum, U.S.P.P. will tell you something about dissociation of diammonium phosphate and summarize the discussion.

It is my pleasure to introduce to you Mr. T. J. Pearce of Swift & Co., who will talk about size.

Typical Analysis
Granular Triple Superphosphate
From Specification Sheets of Various Producers

Data Collected by *C. F. Carnell*

	Producers							
	A	B	C	D*	E	F	G	H*
Moisture	2.93	2.6	3.20	1.0	3.65	5.86		2.
Total P_2O_5	47.62	48.19	47.63	56.6	48.60	45.50	46.6	53.
Citrate Insol. P_2O_5	1.25	2.09	1.63	2.0	1.85	.35	.6	1.
Available P_2O_5	46.37	46.10	46.00	54.6	46.75	45.15	46.0	52.
Water Soluble P_2O_5	81-86% of APA		39.47		86-90% of APA	40.90		
Free Acid (H_3PO_4)	2.76	3.53	.19	2.1	1.8-2.5	2.40		3. (Max.)
Calcium (CaO)		20.41	21.57	23.	20.9	20.80	16.6	
Fluorine (F_2)		2.55	2.34	.8	2.	3.49		
Iron (Fe_2O_3)		1.85	.92		2.	1.62	1.32	
Aluminum (Al_2O_3)		1.80	1.20		1.61	1.90		
Sulfur (SO_4)		3.84	3.84		2.55	3.89	5.95	
Bulk Density Llbs./Ft.^{3}	65	75-80	60	65-70	64-67			75-80

Screen Size (U. S. Mesh)	Range Direct	Percent Retained—Cumulative						
On 6		.5		1.5	1.	2.5		2.
On 8	95-99%	27.4	12.5	20.5		28.		25.5
On 10		59.4		55.		60.		
On 12		82.4						
On 14					90.	79.50		
On 16		98.4		94.6				98.5
On 18								
On 20	1.5%	99.2	93.5	99.3		88.40		Virt.100
On 65					99.8			

* High strength triple from superphosphoric acid.

Typical Analysis
ROP Triple Superphosphate
From Specification Sheets of Various Producers

	Producers					
	A	B	C*	D(a)*	E	F
Moisture	4.	6.3	2.0	1.0	4.75	4.42
Total P_2O_5	48.22	47.8	53.	56.6	48.35	47.94
Citrate Insol. P_2O_5	1.20	.9	1.	2.0	1.35	1.15
Available P_2O_5	47.02	46.9	52.	54.6	47.00	46.79
Water Soluble P_2O_5	83-90% of APA				86-90% of APA	41.41
Free Acid	3.47	3.85	5. (Max.)	2.1	2.5-3.8	2.65
Calcium (CaO)		18.69		23.	20.	
Fluorine (F_2)		2.4		.8	2.	2.5
Iron (Fe_2O_3)		1.64			2.	1.34
Aluminum (Al_2O_3)		1.68			1.61	1.95
Sulfur (SO_4)		2.69			2.55	
Bulk Density Llb./Ft.^{3}	72	65-75	65-75	65-70	59-65	60-65

Screen Size (U. S. Mesh)	Direct Range	Percent Retained — Cumulative				
On 6	0-1	1.8	1.5		1.	
On 8		7.2	10.		11.	
On 12	26-38	14.8		Virtually all —14 largely —20	25.	
On 16		23.4	30.			
On 20		31.9	40.			
On 30		43.9				
On 35	16-23				60.	39.2
On 40						
On 60	10-22	76.3				
On 65	30-38				75.	70.6
On 100		88.	90.			

(a) Fines from granular
* High strength triple from superphosphoric acid

Typical Analysis
Ammonium Phosphate
From Specification Sheets of Various Producers

	Diammonium Phosphate and DAP/Map Producers				Mono-Ammonium Phosphate Producers		
	A	B	C	D	A	B	C
Moisture	1.04	1.2	3.30	.07	3.50	.75	.84
Nitrogen	18.20	16.38	16.01	21.	11.13	11.4	11.0
Total P_2O_5	46.90	48.27	48.48		50.66	50.9	48.15
Insol. P_2O_5	.62	.17	.09		.45	1.	.09
Available P_2O_5	46.28	48.10	48.39	53.6	50.21	49.9	48.06
Calcium (CaO)	.97	.08	.38			.13	.57
Iron (Fe_2O_3)	3.04	1.40	.87		1.94	1.40	.91
Aluminum (Al_2O_3)		1.38	2.10		1.82		2.28
Water Solubility	88.	91.35					
Bulk Density Lbs./Ft.^{3}	63	65	60	47			60

Screen Size (U. S. Mesh)	Direct Range	Percent Retained — Cumulative				
On 8		11.9	17.		7.3	13.2
On 12	95.99%	56.0		47.		
On 16		89.3		78.	99.0	
On 20	1.5%		99.3	90.	99.8	99.5

Typical Analysis
Phosphatic Fertilizer Solution
From Specification Sheets of Various Producers

	Producers							
	A	B	C	D(a)	E	F	G	H
Moisture (H_2O)	16. (Approx.)	22.3	18.75		19.5	19.		16.19
P_2O_5	52-54	54.1	52.06	76.	54.6	52.5	53.3	54.33
H_3PO_5	71.6-74.52				75.3			
Iron (Fe_2O_3)	1.4	1.74	.84		1.5-2.25	1.0	.58	1.25
Aluminum (Al_2O_3)	1.4	2.70	2.70		1.4-2.0	.85	1.53	1.70
Fluorine (F_2)	.6	.50	.94	.05	1.0-1.5	.4	.39	1.25
Sulphuric Acid (H_2SO_5)	4. to 5.	1.51			1.5	3.0		4.21
Solids	1. (Max.)	.72		Negligible	1.4	.5 (Max.)		1.17
Specific Gravity*	1.68-1.74	1.67	1.7	1.92	1.71	1.69		1.710

(a) Superphosphoric acid—ortho P_2O_5 50% of total
* At about 75-80° F

Address By Thomas J. Pearce

MR. THOMAS J. PEARCE (Swift and Company): With all this talk this morning about taking shots at and sticking it to the phosphate producers I feel more or less like General Custer must have felt just before the Indians moved in on him.

Actually the problems that the phosphate producers have are almost identical to the one expressed by the mixer producers, that of having a uniform raw material supply.

Mother Nature did not lay down the phosphate deposits with any uniformity at all, whether you speak of the geological formation or its chemical composition.

Since Mother Nature does not have any panel or representatives that we can shoot at, then I think we must be honest about it and admit that the variations do exist.

I am not going to attempt a defense on the subject of particle size because uniformity of this particle size depends on so many other factors within and among the producing plants that a whole review of these operations would then be necessary.

Because there are as many variations in producing the end product of triple as there are in the number of producing plants it should not be expected that cars of triple from different companies would have similar particle size distributions.

When examining products from individual companies more size uniformity would be expected.

My experience is limited to Swift and Company's production and I can only speak on this basis.

With the uniformity of size, however, uniformity of ammoniating qualities is also expected as some of the other speakers pointed out this morning.

We maintain the ammoniating qualities by producing triple that has thousands of small holes or pores formed within the walls of triple. The effect, of course, is similar to the cross-section of a honeycomb or sponge, as has been previously mentioned.

Chemically speaking this is excellent for the absorption or reaction of the gas such as ammonia because the rate of reaction is dependent upon the amount of exposed surface.

Mechanically speaking this is not the best condition to have, because like the honeycomb, the thin walls can be broken when subject to crushing or shear causing a finer product to result.

Variation in screen analysis can occur when loading a car from the edge of the pile or the center of the pile since about 1,400 pounds per square foot can occur at the bottom of a thirty foot pile. I know you are also all aware of the size segregation that occurs as the storage pile is being formed.

Screening out of the courser material does not necessarily guarantee the uniformity of size at the mixing mill because loading, shipping, unloading, handling will induce some additional fines to be formed.

These then are some of the reasons as to why we do not have exact uniformity of product size. It is our greatest desire to ship you the triple that is 100 per cent within the size that you request. I believe this is the ultimate objective of every producer and we are working towards that goal just as fast as money, time and technology will permit.

Thank you.

E. F. CARNELL: Our next speaker, W. W. Harwood will discuss the uniformity of analysis and also tell us something about the shrinkage as it relates to free and mixed water in the material.

Address by W. W. Harwood

In answering this question, let us discuss first the composition of Triple Superphosphate and the factors which influence its composition. Any variations in these factors may result in variations in the percentage composition of the material, such as the available P_2O_5 content.

Triple Superphosphate is, in reality, a mixture of chemical compounds, predominantly phosphates, present or formed from the chemical reactions between phosphate rock and phosphoric acid, as shown in the following table.

Percentage Composition of Triple Superphosphates

Available phosphates:	
Phosphoric acid	2-5
Monocalcium phosphate (water-soluble)	63-73
Dicalcium phosphate, iron and aluminum phosphate complexes, etc. (water insoluble)	13-18
Non-available phosphates:	
Unreacted phosphate rock	2-6
Diluents:	
Calcium sulfate	3-6
Silica, fluosilicates, organic matter, etc.	3-6
Moisture	3-6

The variations in percentage composition which establish the available P_2O_5 percentages of the Triple Superphosphate are affected by variations that may occur in any or all of the following factors:

I. *Process*
    1. Phosphate rock compositions in the production of phosphoric acid as well as in the production of the Triple.
    2. Phosphoric acid composition used as the acidulant in the manufacture of the Triple.
    3. Acid and rock metering equipment including instrumentation.
    4. Acid and rock mixing equipment and the efficiency of mixing.
    5. Acid temperature.
    6. Den reaction temperature and retention time.
    7. Efficiency of disintegration and mixing after den.

    8. Many others, including sampling, analysis, etc.

II. *Curring*
    1. Length of cure.
    2. Depth and size of curing pile.
    3. Location of material in the pile.
    4. Climatic conditions.
    5. Others.

III. *Shipping—Origin and Destination*
    1. Handling and conveying equipment.
    2. Sampling and sample preparation techniques.
    3. Analyses
    4. Climatic conditions.
    5. Others, such as in transit conditions, storage, etc.

Some of these factors can be controlled while others are random and interdependent upon other factors and conditions. As an illustration of the effect of only one of the listed variables — let's assume that the Triple was produced under very closely controlled conditions in order for it to analyze within a close range of available P_2O_5 after a certain planned curing time. Due to weather conditions, market situations or other factors the length of cure was necessarily extended prior to shipment, therefore, the resulting available P_2O_5 would be higher than planned. Likewise, a decrease in curing time would result in a decrease in the available P_2O_5. Also, the material close to the outside of the pile will be lower in available P_2O_5 than the material towards the center of the pile due to the accelerated loss of moisture which retards the curing processes appreciably. This is only one of many reasons that large curing piles are preferable. There are many other variables within the curing patterns alone but time does not permit further discussion of these and other variable factors.

In conclusion, it can be stated that a very close range of available P_2O_5 from car to car is desirable from a producer's standpoint as well as by the users but it can be achieved only through very exact controls of all the factors involved. These are neither possible nor economically feasible at this time. I feel that I can speak for all of the producers on this panel in saying that efforts are being directed continually towards improving the controls of the different variables and that definite inprovements have been realized in the uniformity of quality. With further advancements that are being made in equipment, technological "know-how" and process controls, additional improvements in uniformity and standardization of raw materials will be forthcoming.

As has been pointed out by some of the other panel members, closer controls, greater demands, naturally, will require more money; it will require also a higher price received for the materials. You must keep this in mind that, 1—Super phosphates, both triple and normal, are one of the—and probably about the only raw material that you use—where a chemical process is continuing on. In fact in the manufacture of triple superphosphate your curing starts immediately after mixing and that curing continues in decreasing amounts actually until it goes into your mixer. 2—To stop a chemical reaction this suddenly does require quite a bit of factors which are not justified as yet.

Question: To enable closer formulation to help eliminate some overages and "In Plant Shrinkage" is it possible to get a definite free water and fixed water determination?

The official methods of analysis of the Association of Official Agricultural Chemists include procedures for the determination in fertilizers of total water and two procedures for the determination of free water, namely the air-flow method and the vacuum dessication method.

The total water method covers the percent loss in weight of a 2 gram sample heated for 5 hours at 99-101 degrees C (210-214F). The aid-flow method calls for the use of a manifold in an oven at 60 degrees C (140F) using a 15″ vacuum for 2 hours. The percent loss in weight is calculated as free water after cooling the sample in a desiccator for 30 minutes. The vacuum desiccation method employs the use of a 2 gram sample dried at 25-30 degrees C (77-86F) in a vacuum desiccator over a suitable desiccant, under not less than 20″ of vacuum for 16-18 hours. The percent loss in weight is calculated as free water.

For several years one of the triple superphosphate producers in Florida, who is represented on this panel, has been distributing a monthly check sample of their product. There are 20-25 participating laboratories in this program, which is conducted in a similar manner to the monthly N.P.F.I. collaborative sample program; however, this program is conducted at no cost to the participants. With one exception the moisture analyses have been reported by the various laboratories as total water. One laboratory has reported the moisture as free water, employing the vacuum desiccation method. An examination of these results over the past two years reveals the following information for comparative purpoes:

	Range	*Mean*
Total Water	3.13-6.74	4.57
Free Water	2.24-5.01	3.18
Difference	1.00-1.81	1.39
Free Water (100) Total Water	59.0-78.1	69.6

It was indicated that there was a general trend for the difference between the total water and free water to increase proportionally as the total water increases. These differences could be explained in the following manner.

In the manufacture of triple superphosphate, the tricalcium phosphate, when reacted with phosphoric acid, is converted mainly to anhydrous mono-calcium phosphate with some anhydrous di-calcium phosphate being formed. Also, there are small quantities of complex iron and aluminum phosphates formed in this reaction. The sulfuric acid present in the phosphoric acid acidulant is converted to anhydrous calcium sulfate.

The theory advanced by investigators is that the phosphate-bearing compounds are converted principally to hydrated compounds during the curing process. Also, the hydrated calcium sulfate is formed. Therefore, the free water present in freshly acidulated triple is used to satisfy the water of hydration required during curing. During the same time that the water of hydration requirements are being fulfilled, some of the free water evaporated slowly. This phenomenon is borne out by the increases in total P_2O_5 that occur in triple between "green" and "cured" triples.

During a natural curing period of 4-6 weeks, the increases in total P_2O_5 will generally be 0.5-0.7%, while the decreases in total water will probably be 4-5%. If the free water merely evaporates, then the increase in P_2O_5 would be proportional to the decrease in free water content. This does not happen; however, if the triple is cured in small piles, the free water will evaporate before the water of hydration requirements are satisfied, resulting in increases in total P_2O_5 which are more closely proportional to decreases in free water. This condition also occurs around the edges of large piles of triple. This type of curing in undesirable, since the rate and degree of curing is greatly decreased when the free water is decreased. When the length of cure is extended and the water of hydration requirements have been satisfied, then the increases in total P_2O_5 will be more closely proportiond to further decreases in free water.

Since the reaction temperatures generally reach 190-230 degrees F in most ammoniation-granulation units, it would appear that a knowledge of total water content, as defined by A.O.A.C. procedures, would be more desirable than free water content for calculating moisture balances in formulations. However, which water percentage is preferable to you is dependent upon your own operations and requirements.

E. F. CARNELL: Thank you Bill Harwood. I am sure we will get some discussions on that one. The next talk is by Ray L. Jones who will discuss moisture variations in superphosphate.

Address by Ray L. Jones

MR. RAY L. JONES (Armour Fertilizer Company): Members of the Round Table, I have been asked to discuss moisture variations. The question is "What does the moisture value mean" and you've heard the pros and cons of free water and combined water discussed by the previous speaker.

Right or wrong our laboratories have continued to use the oven method. We believe that the results so obtained will more nearly approach the loss of water that occurs in granulation.

My comments will be based on results obtained by this method and my experience with triple has been limited to our own process which is a dry product; therefore I will try to speak only of run-of-pile superphosphate.

I would like to offer the following comments with the hope that the user will better understand the manufacturers' problems.

These comments are based on the observation of analysis of shipment from our own plants and from super received from some dozen other manufacturers at various of our plants.

There are two types of variations. One is the day to day or car to car type which was harpooned this morning; the other is seasonal. The day to day variety probably causes more difficulty for the user.

Some of these variations are inexcusable and could be controlled with closer control in the plants. Other variations are beyond the control of the manufacturer. These I will try to set forth.

The reaction within a pile of superphosphate continues for months after mixing. The aeration which occurs during shipping, loading and unloading, results in some evaporation of water, but the change from pile moisture to unloaded moisture is not always accompanied by an accompanying change in the total phosphoric acid.

Changes in free acid and insoluble indicate that aeration has sped up certain reactions which probably include change in the amount of combined water.

This change may never be noted by the user.

Evaporation does concern the user. The amount of water lost by evaporation will be affected by the relative humidity and the temperature of the superphosphate. Cars loaded on humid days or in early humid hours of the day will not lose as much moisture as those loaded on dry days or in the drier hours of the day.

The superphosphate at the edge of a pile will be cooler and lose less water than the hotter center of the pile. If many cars are loaded in the day quite a range of moisture results will be found. If one user receives more than one car several moisture results could be found.

Cars received on successive days if loaded at different times of the day or from different locations in the pile could contain superphosphate of differing moisture values.

The other variation is seasonal. While of greater range, probably two to three per cent and perhaps causing greater formula change, the changes can be more easily anticipated by the user.

The nature of the fertilizer business with its high seasonal peak does not permit storage of sufficient superphosphate to meet the commitments. The manufacturer usually begins with an acidulation program which will provide cured superphosphate for his customers and himself with a little surplus; so his storage will be filled when the peak hits; then bang.

Old well cured low moisture superphosphate and more recently made high moisture superphosphate will be moved out in just a few weeks. Then freshly made super must be milled. Having anticipated that the manufacturer has modified his formula to slightly over-acidulate for faster insoluble drop. This further increases the moisture, three to five per cent.

The alert user who has followed this trend should not be unduly hurt by these changes. However, he may criticize the manufacturer for not having adequate storage, but let's look at the economics of this.

It very often happens that almost half of the commitment moves in the final period; to provide sufficient storage would necessitate an increase of 50 per cent to 75 per cent of the present space. This would increase—this would result in an increase in the cost of plant food units.

Also in the face of lower superphosphate consumption such a move does not seem justifiable.

These variations are those whick would exist within a single plant. There is other variation from manufacturer to manufacturer or from plant to plant.

There are at least five types of dens manufacturing superphosphate. Each of these will produce superphosphate of differing moisture contents. Acidulation is carried out with acid ranging from 54 to 58 Baume. This, too. would affect moisture content of the super produced.

The changes in dens, in strength of acid, evaporation will affect free acid. Rock received by car may vary at least one BPL from car to car. This will affect free acid and to a certain extent, moisture.

All of these are factors the manufacturer of the product has no control over and which we hope the users will take into consideration in setting up their standards and tolerances which they require the manufacturer to deliver.

Thank you.

E. F. CARNELL: Thank you very much Ray Jones. I don't know whether our next speaker is a victim of scheduling or a case of working a good horse, but anyway we enjoyed his talk very much yesterday and I am sure can expect the same from this one. Dan will go through his plan on flow sheet and show us some of the points at which quality and condition can and cannot be controlled.

MR. DAN WALSTAD: Obviously I was a victim of circumstances here because I was scheduled on Frank Nielssen's program better than 3 months ago and at the last minute I found out I was supposed to be on this program. Back in the good old days when mules were prevalent a farmer was having trouble driving his mule down a country road and the mule would not budge so he got out and went over the fence. into the woods, cut himself a big stick and started beating the mule unmercifully. Down the road came a car that stopped and a little old lady got out—later we happened to know that she was a member of the S.P.C.A.—and she was horrified at this terrific beating the mule was taking. She cried out to the farmer, "Sir, you will never get the mule to move if you beat him with that stick," and the farmer stoped and look at her and he said, "You know you are right, I have got to get a bigger one." Laughter.

Properties of Triple Superphosphate

Address By Dan Walstad

In any discussion of the properties of triple superphosphate, it is helpful to know how the material is made and what procedures are used to control the quality of the product. In the first slide a flow sheets of a process for run-of-pile triple superphosphate is shown.

Finely-ground phosphate rock and wet-process phosphoric acid are brought together in a TVA-type cone. After the mixture has set it is passed through a disintegrator and chain mill before being placed in storage. Here the chemical reactions are allowed to continue. After three weeks or more in the storage pile the material is removed, crushed to pass a 5-mesh screen, and shipped to customers.

The numbers on the flow sheet indicate points of sampling and measurement.

Control Points in Triple Superphosphate Manufacture

(1) A sample of phosphoric acid is taken from a full storage tank and analyzed for P_2O_5 and Acidulation Value, before the acid is used. A full tank holds enough acid for 14-16 hours of operation.

(2) Samples of ground phosphate rock, taken from cars being transferred from the drying plant to the triple superphosphate plant, are composited on a daily basis. The composite is analyzed for Moisture and P_2O_5.

Based on the analyses of samples (1) and (2) a mix ratio is calculated.

(3) Flow of acid is measured by electromagnetic meter, which is calibrated periodically to insure that its accuracy is within ±1%.

(4) Flow of rock is measured by an Omega feeder. Periodic calibrations of the rock feeder are made to insure that its accuracy is maintained with ±1%.

(5) (6) Shift composites of acid and rock entering the mixing cone are obtained and analyzed to provide a check on the valves used in calculating the mix ratio.

(7) A shift composite of green triple superphosphate is obtained and analyzed for Moisture, Total P_2O_5, Citrate Insoluble P_2O_5, Free Acid and CaO. The analytical data serves two purposes:
   a. It provides a means for determining the mixing ratio that was actually used.
   b. It indicates the efficiency of mixing of acid and rock.

(8) A composite sample of triple superphosphate is obtained from each car loaded for shipment, and analyzed for Moisture, Total P_2O_5, Citrate Insoluble P_2O_5, Available P_2O_5, Free Acid, and Particle Size.

The control system as outlined is quite effective most of the time in minimizing variations in the properties of triple superphosphate. However, variations still occur, caused by two factors over which we have limited control. One factor is the variation in impurities, mostly iron and aluminum, that are present in the rock and wet-process acid. These impurities influence amount of reaction and setting time of the acid-rock mixture. They also affect the amount of hydration, the conversion of free water to bound water, that occurs in the storage pile.

A second factor is the length of time the material remains in the storage pile. This may vary from three weeks to two months depending on seasonal fluctuations in shipments. As both chemical reactions and hydration continue during the storage period the length of the storage period has a marked effect on final available P_2O_5 content.

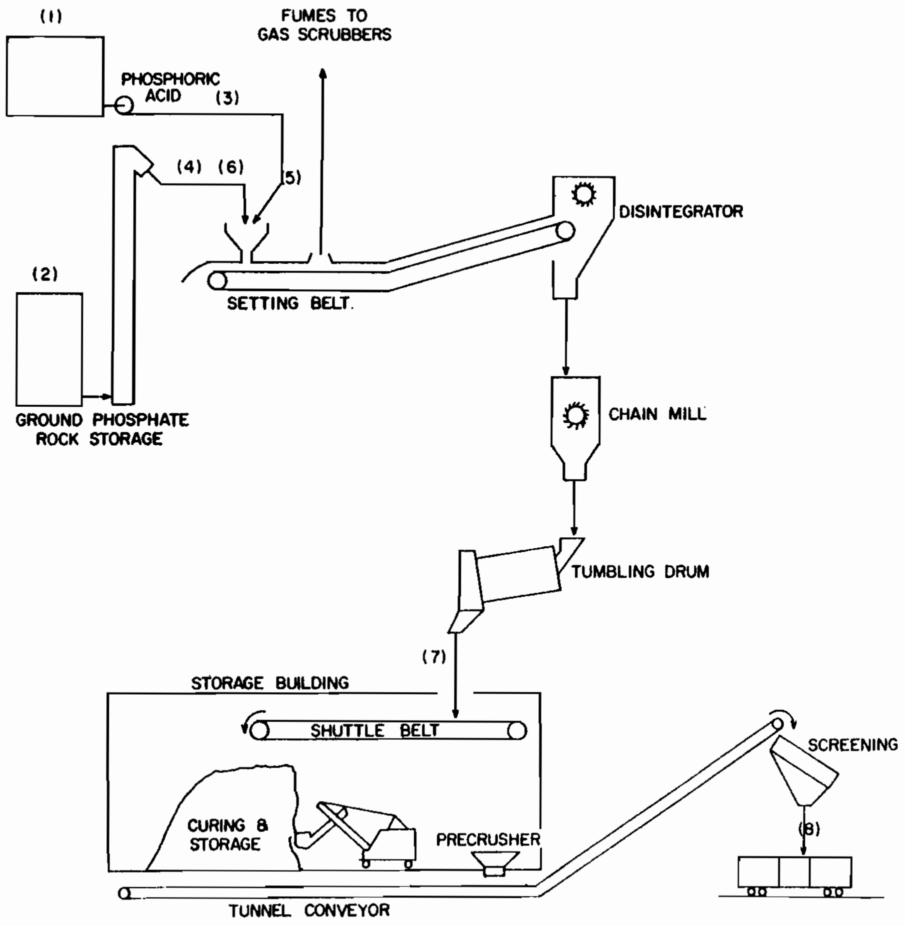
In the last slide variations in the chemical and physical properties of triple superphosphate are given. This illustrates the effectiveness of the control system as outlined above.

We do expect that improvements can be made as the other panel members have indicated, but it will cost money and time and will require some increases in our knowledge. The 3 areas in which we can make improvements in the matter of control: First Better Metering equipment that will be more accurate than plus or minus one per cent; secondly, we can learn different methods of evaluating the rock and acid before we apply a mixing ratio; third, we can improve our analytical procedures to eliminate the variation that occurs in the analytical results upon which we base our chemical control. All of these, I think, in time can be improved and all of us are endeavoring to do so. Thank you very much.

Mr. E. F. Carnell: Thank you very much Dan Walstad, you did it again. The next speaker, and last on this panel, is Barney Tatum who will tell you something about dissociation of Diammonium Phosphate.

Mr. H. P. Tatum: Gentlemen, from that session this morning I came up with one conclusion and that is that to the finest mixed goods manufacturer on earth we ship the *finest material.* Well, I mean "fine." Laughter. If you really want to crab about this thing and be a real old crab, why let us know a little bit more about it and I am sure that we can get you some better materials. I am not going to get into the triple superphosphate manufacturing procedure, nothing like that. We are getting into some other fields. Triple has been ably covered here. I do want to talk with you for a few minutes about a modified diammonium phosphate.

TRIPLE SUPERPHOSPHATE MANUFACTURE



Analysis of 30 Consecutive Shipments of Triple Superphosphate

CHEMICAL ANALYSIS:	High	Low	Average
Moisture	5.46	2.46	3.87
Total P_2O_5	49.02	47.48	48.35
Citrate Insoluble P_2O_5	1.68	0.76	1.28
Available P_2O_5	48.06	46.12	47.07
Free Acid	4.19	1.78	2.82
SCREEN ANALYSIS:			
—10 Mesh (Tyler)	90.6	74.8	84.6
—35 Mesh	70.4	46.0	60.8
—65 Mesh	38.0	22.4	29.4

Dissociation of a Modified Diammonium Phosphate —Physical Characteristics of a Modified Diammonium Phosphate In Various Types of Plants.

Address By H. P. Tatum

Several of you have written to the Roundtable in regard to the physical properties of modified diammonium phosphates. The question centering around dissociation seems to prevail. What happens to high temperatures? Is it stable? Will it melt? Does it remain hard on cooling? Is the normally high water solubility affected? What is the effect of high moisture? How much NITROGEN is lost? Gentlemen—these questions are most pertinent—and some answers will be forthcoming.

Chemical and physical studies of modified diammonium phosphates have been made in the laboratory and in the various types of mixed-goods plants. In thermal stability experiments the results indicate that a modified diammonium phosphate is relatively stable at temperatures up to 275° F.—retaining most of its nitorgen at this temperature, even with an extended exposure time. To graphically illustrate may we have the first slide.

The object was to determine the thermal decomposition characteristics of the N-P grade at elevated temperatures for periods of time coincident with those encountered in the manufacture of mixed fertilizers.

On this slide the temperature in degrees F is plotted against loss of nitrogen in pounds of nitrogen per ton of N-P grade. The red curve indicates a relatively small loss of N at normal ammoniator operating temperature. At 300° F. the loss is in the range of 4 pounds of nitrogen per ton of modified diammonium. Please note—the exposure time was 30 minutes which should be ample for a particle to travel through the ammoniator and dryer. Initial moisture content was 1.25%.

The next slide shows the effect of different initial moisture levels, ranging from 0 to 10%, on loss of N. The loss is small, yet measurable. The exposure time was 30 minutes; the temperature of the N-P grade was constant at 230° F. Maximum loss of nitrogen under these conditions proved to be 1.2 pounds per ton of N-P grade.

In batch, semi-granular operations wherein sulfuric acid is used, the temperature is expected to rise higher with low initial moisture in the mix. The fully-integrated plant operation presents the opposite picture—higher moistures in the ammoniator coupled with air swept beds lower the temperature. These compensating conditions in each type of plant hold dissociated nitrogen losses to a minimum, and for all practical purposes a minimum of 99.5% of the original nitrogen in the N-P grade is retained in the mixed goods.

Using granules of modified DAP in the fully-integrated systems allows a reduction in overall heat loads in the ammoniator. You recall that balanced mixed-goods formulas having a heat load of 125,000 to 140,000 BTU's normally have good chance of producing a large percentage of on-sized granules. Successful experiments have been performed in fully-integrated and semi-granular plants wherein the heat load was reduced to a total of 75,000 BTU's per ton of mixed-goods producing an on-sized range, minus 6 plus 20, of about 80%. The reduced heat load lowers the fertilizer temperature in the ammoniator to the 175 to 200° F. range.

Again, let us turn our thoughts to moisture and temperature. The N-P grade is more susceptible to particle size reduction in an ammoniator wherein the mix contains a high moisture and medium temperature. The high moisture, temperature and the attritioning action forces a particle size reduction by putting a portion of the surface in solution. As the smaller particles roll to the top of the bed other small materials are affixed to the surface. The induced air, sweeping over the bed momentarily chills the surface, evaporating enough water to tighten the mechanical bond for another cycle through the reaction zone. The alternate heating and wetting in the reaction zone followed by cooling and partial drying in the air-swept zone tends to build laminated surfaces resulting in strong hard granules. In the meantime, that portion of liquid phase, formed by the dissolution of the surface, is busy gathering other particles to form granules.

In the semi-granular plants, the same physical action takes place, but to a lesser degree because of the lower initial moisture.

Calculating a practical problem from the moisture and temperature graphs — should a formula contain 550 pounds of modified diammonium phosphate per ton of mixed goods at a mixing temperature of 230° F and at 10% moisture it would require an excess ammonia absorption capacity of approximately 0.3 pounds. Should the temperature be increased to the scope of good standard operating procedure, the dissociation could amount to 1.5 pounds of free ammonia. The adsorption of 1.5 pounds of free ammonia requires slightly less than 5 pounds of 66° Baume sulfuric acid.

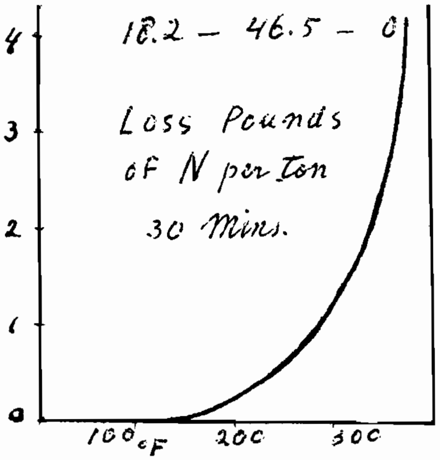
A general rule may be established — for very 100 pounds of modified diammonium phosphate used in a formula add one (1) additional pound of sulfuric acid. Thank you gentlemen.

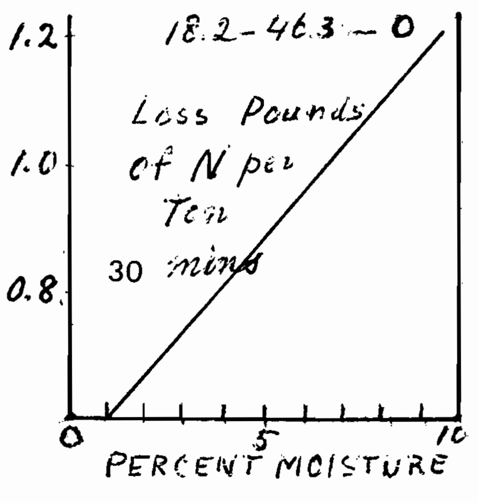
MR. CARNELL: Thanks very much, Barney.

I think Barney didn't get the tomatoes and eggs, so don't throw them now.

The panel is now open for questions and again I would like to say if there is anyone else who is in the phosphate producing business, any of these materials, he'd like to join us we would certainly be glad to have you.

Last night we got around in Barney Tatum's room there to kind of sketch this thing out and figure





out what we were going to say and Mr. Al Spillman and Doc Marshall dropped by and we got into one of the biggest discussions and arguments you ever heard, so we said that if that happens multiplied by about 400 today we might all turn tail and run, but anyway we are open to questions and we hope some of the same thing will develop that did in Barney's room last night.

Do you have any questions?

A MEMBER: I wonder if the previous speaker would comment on the effect of this nitrogen loss on the moisture determination in 18-46-0.

MR. CARNELL: To whom are you directing that question, please, sir?

SAME MEMBER: To Mr. Tatum.

MR. CARNELL: Mr. Tatum.

MR. TATUM: I think I have an understanding of the question. My understanding is, if we had different moisture levels in the initial diammonium phosphate would we have different losses of nitrogen. Is that essentially the question?

SAME MEMBER: Yes.

MR. TATUM: That's essentially it?

Would you come on up here, sir, so that we might all hear?

SAME MEMBER: My question is, if you were running a routine control on the moisture content of the finished 18-46-0 in your production how does the loss of nitrogen affect this determination. Are you getting a relatively true value of the moisture or are you incorporating just a total loss which includes nitrogen or ammonium loss.

MR. TATUM: That, in effect, has been one of our studies in the laboratory. We have given it considerable consideration in the analysis of this nitrogen that it does not interfere with the moisture determination. It has been a very difficult thing for us to separate moisture and nitrogen but we have been successful in doing that in our laboratory, after a period of study of about six to eight months.

MR. CARNELL: Barney, if I may and he doesn't mind answering I think there is someone else here who can certainly give some good ideas on that.

Mr. Caro of USDA have looked at this effect.

MR. JOSEPH H. CARO (U. S. Department of Agriculture): I have done some work recently with the vacuum oven as a proposed method for moisture and I have also included the official AOAC methods for moisture and I have used diammonium phosphate as one of my test materials.

If you are going to run total water as given by the AOAC at 100 degrees in the oven you will lose nitrogen from diammonium phosphate, but if you run the moisture methods, that is free water with the vacuum oven at 50 degrees you will not lose nitrogen. Nor will you lose it if you run the official method in a vacuum desiccator for free water, but the total water situation is a difficult one, as Barney said, to separate out the nitrogen from the water.

MR. CARNELL: Thanks very much.

A MEMBER: I wonder if you tried the air flow method there.

MR. CARO: Yes, I tried the air flow method and the air flow method does show some loss of nitrogen at two hours which is the official time, the 60-degree air flow method at two hours, begin to lose some nitrogen but it is not terribly great; if you go beyond two hours, up she goes, you lose nitrogen.

However, the vacuum desiccation method, which is quite simple; anybody can toss a sample into a vacuum desiccator for overnight, will behave quite satisfactorily in this respect.

MR. CARNELL: Thank you.

MR. NIELSSON: The only thing that bothers me here. I don't know if Mr. Caro is working with diammonium or modified diammonium and by past experience the vapor pressure of ammonia over a DAP solution is much higher than a solid. Therefore, when you get DAP 21-53-0 wet you lose ammonia in a hurry at elevated temperatures.

Normally then as an arbitrary factor we assume that from liquid fertilizers you can ammoniate to a one to three nitrogen P_2O_5 weight ratio making 8-24-0, that gives you 8.2 pounds per unit of P_2O_5. If you take 21-53-0 and back it off you don't lose any nitrogen if you ammoniate in making 8-24-0. This is a very liquid solution.

If you take that data and take 21-53-0 then you come back by our method of calculation we say that DAP 21-53-0 has a minus equivalent or has an equivalent of minus four which would mean that you have to take care of four pounds of ammonia per hundred pounds of material and roughtly one pound of ammonia means three pounds of 66° acid so on 21-53-0 we would add 12 pounds of 66° acid for every hundred pounds of 21-53-0 to make sure that we don't lose any nitrogen during granulation and we are assuming there that we are ending up with a 8.2 pounds per unit ammoniated P_2O_5 (electric furnace product.)

Electric furnace or 21-53-0 is fine and finally goes into solution in a hurry.

Is your figure of one pound of sulfuric acid for this modified DAP so low because modified DAP is essentially different from true DAP or is it because your particles are so large that you don't get the solution or you don't get the ammonia loss?

MR. TATUM: It is without a doubt tied up with particle size. I feel that if you were to take this material and grind it to those small crystals that you might find in 21-53-0 that you would have essentially the same thing.

I feel that without a doubt that itis particle size that helps to control the nitrogen loss in this case.

MR. CARNELL: Yes, sir.

A MEMBER: On this figure of one pound of acid decomposition how is that affected if your mixed muriate of potash with ammonium nitrate and some of the other things we have in a regular fertilizer mix. Will the decomposition occur at a lower temperature or just what happens?

MR. TATUM: We have used this material in all types of plants at all different temperatures and with all types of potash and we find that there is no relative difference in the loss of the nitrogen as we would have this compared with the laboratory results.

Does that answer your question?

MR. CARNELL: Wouldn't you

say, Barney, that there should essentially be no difference between this and using diammonium and sulfuric acid in conventional material and any other way. The fact that you just mentioned one pound —many people have been formulating products on their conventional formula except substituting diammonium, haven't they?

MR. TATUM: Yes, that's right.

Another question, too, that came up that since you saw on the slide that this was essentially 18-46 the question that has come up to us is "What effect—or suppose we were to change this analysis to 16-48?" Now, we do not have that experience with 16-48, but we feel that the results would be essentially the same, provided that you know that you have the same particle size. I can see some strange things on that but I feel as though the loss would be—if we could consider this loss—would be just the same.

The diammonium phosphate content in 16-48-0 is about two thirds and we would have one-third monoammonium phosphate and with this 18-46-0 we would have essentially 85 per cent diammonium phosphate and 15 per cent mono.

In other words, you are really up so close to diammonium phosphate that you could hardly be able to tell the difference anyway.

I don't think we could tell that much difference.

MR. CARNELL: Any more questions?

MR. ROBERT WILLS (Chemical Construction): I don't have as much interest in the uniformity as I do in process and I would like to ask Mr. Walstad, between the disintegrator and the transfer conveyors on his triple flow sheet he shows a chain mill, I believe, and a rotating drum. I wonder if he would care to say what prompted him to do that; whether it is purely to improve product or actually what his reasons were.

MR. WALSTAD: As you know, the Florida Pollution Commission is very interested in fluorine leaving the phosphate plants and this equipment was put in to remove fluorine from the fresh green triple before it was put into our storage piles. We had a choice of either removing it there or putting a collection duct the full length of the storage building which is 1,000 feet long in order to catch fluorine coming off the pile. We thought this was the better approach.

I think Mr. Chesson, our Plant Manager is here; would he care to comment on that?

MR. WALSTAD: We would like to have you comment on the use of the chain mill and the tumbling drum for fluorine removal.

MR. CHESSON: Actually this equipment went into operation about two weeks ago. We are very well impressed with the function. The chain mill was a piece of equipment we selected thinking that it would be one that would not cake seriously and it has proved to be that way. It is successful in disintegrating material to essentially minus half inch.

The rest of the equipment, the chutes and the like, you gentlemen are familiar with the trouble we have with chutes, it seems like you can't make them straight enough or wide enough. There have been some problems in this respect.

The tumbling drum, again we have have scaling in the tumbling drum; its basic function is good. We are scrubbing the fluorine from the triple before it goes into the curing pile. This was a primary objective and it is successful in that respect. The physical counting problems I don't think will be so severe that they will plague us indefinitely. They will be worked out.

A MEMBER: What about the fluorine?

MR. CHESSON: It does not have the fluorine content at the time of shipment that it had before, but without this equipment we were removing the fluorine from the curing pile and it accentuated our fluorine pollution problem and in Florida you are well aware of the intensity of this problem.

MR. CARNELL: Thanks very much.

I am told that that is all the time we are going to have for questions and I certainly want to thank each of you for your interest and participation.

MODERATOR SPILLMAN: Thank you, Ed Carnell and your associates for the discussion on phosphates.

MODERATOR SPILLMAN: Our good friend Dr. W. L. Hill, will discuss his studies on variability of Superphosphate shipments.

Address by W. L. Hill

Mr. Chairman, Members of the Roundtable. Here I stand precisely in the position described by the clergyman, Viz: "You will find when I get up to preach I don't know, and I have serious doubts that God Almighty or the Devil knows what it is I am going to say." Now I know what I would like to talk about, however, I haven't got it figured out as to how to say it. I would like to call your attention, however, to certain changing attitudes on the part of the State officers under whose surveillence your fertilizers move to the farm. I have been sitting around many State Control Chemists Association Meetings. They are talking in private, they have not talked in public yet. What they are saying is this, that they would like to judge the fertilizer man on the basis of the performance of the plant, saying take his shipments, the analysis of his consecutive shipments over the season or over a period, the average that they would get would be what the man's objectives are; that is what he is shooting for. Then they would be concerned about how much he varies from that; how much he goes up or down, which is measured in terms of the standard deviation of that average.

I began wondering then how the Superphosphate People are doing in that respect. I asked some of our Producers and Users for their shipper's analysis, of shipments.

You know these folks, when they give this information, they want to make sure that you are going to attach the name of the producer to the results that are reported, therefore, I am going to do that and the first one will be identified "No. one." Laughter.

Variability of Superphosphate Shipments

Plant No.	Date	Consecutive analyses by producer Number	P_2O_5 %	Stand. dev. of average %	Coef. of variation %
		Total Phosphoric Oxide			
1	April, 1960	55	20.76	0.11	0.53
1	May, 1960	55	20.79	0.16	0.77
1	July, 1960	55	20.78	0.10	0.48
1[a]	3 months	165	20.78	0.13	0.63
2	1960	50	20.50	0.10	0.48
3	1960	50	47.65	0.44	0.92
4	1960	50	48.91	0.31	0.63
		Available Phosphoric Oxide			
2	1960	50	20.20	0.05^5	0.27
3	1960	50	46.72	0.55	1.18
4	1960	50	46.70	0.44	0.94
5[b]	1960	10	46.70	1.11	2.39

[a] Den analyses over same period: Total P_2O_5 19.86%, S.D. 0.29, C.V. 1.46%.
[b] User's analyses of 10 car lots from same producer.

You will note on the blackboard I will show a number of observations and the averages and the standard deviations of the average. Note — Plant one for April 20.76, May 20.79 and July 20.78 and on the standard deviation, which is the measure of the variability there was respectively .11, .16 and .10. Up a little in May and dropped down in July. Folks, that is on straight superphosphate and I am reminded of the little boy that took his report card home. He had all "A's" on it. He showed it to his daddy and his daddy said "Well, son, that is pretty good." The little boy replied "Pretty good, hell, dad, that is perfect." Laughter. I think that is about perfect. I would like to indicate here that we also had the den analysis over the same period and they did not do too badly on the dens. Total P_2O_5 analysis of the den averaged 19.86 with standard deviations of .29. That is pretty good for 20% superphosphate. Well I thought that was too good, we go to plant #2, shipments during 1960. We had 50 observations and what do you think he did? We got 20.50 with a standard deviation of .10. That is the result with ordinary superphosphate. I suppose most anybody can do that. Everybody knows how to make ordinary superphosphate.

Plant #3, shipments 1960, triple superphosphate, 50 observations. This plant had an average 47.65 with a standard deviation of .44.

Plant #4, shipments 1960, triple superphosphate, 50 observations. This fellow had an average 48.91 with a standard deviation of .31 just the reverse of what I thought it would be, however, that is the way it came out. That is pretty good, too, of course. These results are total P_2O_5. I wanted results on available P_2O_5 because that is the way you market it. Plant 2 showes 20.20 A.P.A. with standard deviation of .05, plant 3 showes 46.72 A.P.A. with standard deviaton of .55. Plant 4 shows standard deviations of .44.

What does all this mean folks? This is what the shipper says each year. "We took the samples and analyzed them and this is the summary of the work that was performed on those analysis. That is all it is."

But there is a rule—this is the variability now among shippers. There is a rule in statistical theory that examins variability that says the variability within the shipments is one thing and the variability among shipments is another thing and it says that the variability among shipments must be or will be at least as big as the variability within the shipment.

I have #5 on the chart. He is not a producer of superphosphate. He is a user. He had ten carloads. He is saying what he got and I suspect that he received his material from this "Bird" up here — right there. Now you see when we sent it to him it had a standard deviation of .44 but when he got it it looks like the standard deviation was 1.11.

Most of you have already done a pretty good job for ordinary superphosphate and there is at least one fellow in this summary not doing too badly with triple superphosphate. I don't know what the others are doing but if one can dot it, the rest of you can.

I have heard a lot of talk about the reasons why you cannot do things, you do not have the money or you cannot control variables. All I want to say is this: Another little story, the colored fellow in the deep South. He got put in jail and he sent for his lawyer and his lawyer went around and said "Slim, what did you do to get in? He told him. He said, "Why they can't put you in here for that." He said, "I don't know about that, boss, but they is doing it." Laughter.

Moderator Spillman: Thank you very much Dr. Hill. We are going to deviate from our program somewhat. The Potash Panel will be first on the program tomorrow morning. Joe, I will turn this meeting over to you for your topic on instrumentation.

Moderator Reynolds: Thank you very much, Al.

Why don't we take a couple minutes of standing here. I know that the chairs are getting a little bit on the hard side. In the meantime, can the Instrument Panel come on up to the front and we will be set for that discussion.

Instrumentation

Moderator Reynolds: During the initial period of technological growth in the granulated mixed fertilizer field instrumentation features have received their ups and downs of approval. Serious error in recommendations of improperly protected equipment and application have resulted in the drop back to selection of extremely simplified systems which sacrificed accuracy.

Many hundreds of thousands of dollars were spent in the selection of process equipment with only a small percentage of the appropriation directed toward establishing adequate controls.

This choice was not necessarily the desire of man, but possibly because accurate instruments had not caught up with the needs of our particular industry.

An evaluation of our process equipment reveals now that we are basically sound in our arrangement, sizing and selection. The fertilizer industry has been busy training personnel, establishing operating techniques, increasing daily production and eliminating bottlenecks in bulbs and of course we all know there is no substitute for these efforts required.

Now we are in the position to reduce the inefficiences and losses which will persist. Also, but this time, advances have been made in the availability of instruments for our particular industry. A better understanding of mutual problems exist between supplier and user of equipment.

Perhaps rough accuracy was sufficient initially, but now we must narrow the range between our highs and our lows. Plant food variations must be controlled. To be specific, this section of the program will discuss operation, maintenance and selection of proper instruments to measure liquid flow, dry raw material flow and provide efficient moisture removal.

Control of liquid flow has been discussed in many ways. One of the most widely endorsed instruments of the past few years is the magnetic meter.

Our first speaker, Mr. A. G. Simmons, District Manager for Fischer & Porter Company, will discuss magnetic flow meters, application, selection and installation and new developments.

Address By A. G. Simmons

Mr. Reynolds, Gentlemen of the Round Table.

The previous speakers have, on occasion, mentioned the magnetic flow meter which I feel is a good testimonial to the acceptance that this meter has had in fertilizer plants in the past several years. We would like to discuss here today primarily some of the few problems that have been encountered with the magnetic flowmeter; primarily because of its newness and the lack of familiarity of plant personnnel on all levels with the meter and with the advantages that it has.

First, before getting into these practical aspects of the magnetic flowmeter, let me review for some of you who may not be completely familiar with the meter some of the basic operating principles.

The magnetic flowmeter is essentially a simple AC generator in which the flowing fluid is the armature and the electrodes act as brushes. You see here the fluid, a moving conductor passing through an extremely uniform electromagnetic field.

The magnetic flowmeter follows Faraday's basic law of electromagnetic induction which states that the movement of a conductor at right angles through a magnetic field produces a voltage in a plane mutually perpendicular to the field in the direction of the movement.

The voltage produced is directly and linearly proportional to the velocity of the conductor. The conductor here is your fluid; the magnetic field is created by electro magnets shown above and below the moving conductor and flush electrods on the side of the fluid container; a piece of pipe, pick up the induced voltage as this conductor passes through the field.

Since with a given pipe size the volumetric rate of flow is dependent solely on the flow velocity. The induced voltage then from a magnetic flowmeter is directly proportional to volumetric flow.

Therefore, we have in the magnetic flowmeter a meter which will give you a volumetric flow independent of influences of viscosity or density or solids content of the fluid itself.

In the actual concept of the magnetic flowmeter the actual production concept as we know it today the unit is constructed of a stainless steel tube, which is lined with a non-conductive material. In the fertilizer industry this has primarily been teflon with on some occasions a vitrious enamel liner being used to insulate the fluid from the metering tool.

The flush electrodes, which are the only conductive piece in contact with the fluid, can be constructed from very inert materials so that an essentially corrosion resistant meter can be easily made.

Generally, the construction, as I said of fertilizer industry meters has been teflon liners and Hastelloy-C electrodes with only an occasional deviation to something more exotic in electrodes such as tantelum or platinum for pot acid service.

In this diagram you will see the electromagnets in a saddle shape above and below the tube and in the cutaway in the center you can see how the electrodes come through the liner to carry the induced voltage into the calibration circuitry.

This, of course, is a picture of the primary element only; the element which actually senses the flow and its induced signal is then passed on into a potentiometric circuit which does the indication for recording of flow and of course accomplishes the additional functions of totalization or control.

We look here briefly at a block diagram showing the components of a complete magnetic flowmeter recording system showing the flow signal going into a different amplifier and on into the circle amplifier and into the circle motor panels.

The second signal that you see on the botom is the balancing signal; actually the output of the magnetic flowmeter is not taken as only the induced voltage, but is taken as a difference between the induced field voltage and the output voltage in order to take care of some of the deviations in magnetic flux density in the AC current system.

This, then, briefly is how the magnetic flowmeter operates, what it looks like and as I have mentioned, it has been widely used in the fertilizer industry because of its basic characteristics. A higher accuracy than has heretofore been available in a meter suitable for this type of service and immunity to viscosity changes in the fluid, a particular advantage in handling phos acid, a completely clean design with no moving parts in the fluid allowing for fluids which have in them solids or materials which might coast out on moving parts and influence the reading of the meters.

This flowmeter will pass solids and in the event that there is some settling out which coats the entire pipeline it can be cleaned in the same manner as the pipeline.

To go on then to the practical aspects of using magnetic flowmeters and some of the problems that we have found in case histories of many, many magnetic flowmeter installations in the fertilizer industry.

We find that among the principal problems with the magnetic flowmeter has been the obtaining of absolute accuracy which is inherent in this meter. The magnetic flowmeter has an accuracy of plus or minus one per cent of the maximum flow rate for which it is originally sized. This means, however, that when operating at low flow rates you do not obtain this accuracy. The accuracy of the meter is always rated as a percentage of the maximum flow.

In many installations we find that these meters have been oversized to provide for some future condition or it has not appeared important to pin down the flow rate very closely. We then have the problem of the customer coming back and rightfully saying this meter is giving us an accuracy of four per cent when you said it was accurate at one per cent.

We find that without an understanding of just what this meter can do and how it should be sized that we cannot give the one per cent accuracy. When a customer talks in terms of having obtained four per cent from the meter we invariably find that he is operating down around 25 per cent of chart and if you figure out one per cent of maximum flow at 25 per cent of chart is four per cent of his rate of flow.

The way to get around this is, as I said, to size the meter so that your operating condition is as close as possible to the maximum. However, this is not always possible in that you run various grades of material and you run different flow rates for these grades.

The answer to this has been a range adjustment in the secondary instrument itself. This illustration shows this dial adjustment in the lower left hand side of the case reading in feet per second allowing a setting of velocities between one and 30 feet per second as the maximum full scale reading.

This, then means, that for differen tgrades and different operating conditions the plant operator can readjust the instrument to take full advantage of this one per cent accuracy. In each case where we have found a plant complaining about accuracy it has only been a simple adjustment of this range knob to narrow down the range so that the operating condition is up around 80 or 85 per cent of the chart reading that brings them back to one or one and one half per cent accuracy.

Over on the blackboard—which now in the dark you can't see—is the basic formula for calculating just what your setting should be on this range knob in order to obtain any given gallons per minute flow as full scale on the meter.

In other words this dial, which reads in feet per second, is set according to this formula; the gallons at which you are operating are on the right hand side of the formula and are divided by the square of the meter diameter. Now this meter diameter is stamped on the flowmeter itself, on a tag attached to the flowmeter, each one varies just slightly and therefore they are individually measured and this information supplied with the meter.

The factor of .408 on the right is a combination to make this come out directly. Now this adjustment, as I said, is extremely important from the standpoint of getting the accuracy from this meter. Here is a meter which will give you accuracy, which will let you turn out the same type of product time after time, so we urge you all to look at this, ask questions about it of the suppliers, but try to get your meter in the proper operating range because it can really benefit you having this additional accuracy.

Onto the other problems which we have found in different cases. The second group deals generally with the installation of the magnetic meter itself. We show here in our first slide—these are all cartoon versions, incidentally—in this first slide we show a preferred installation system for a magnetic flowmeter.

You will note that it is arranged in a bypass so that it is very easily cleaned. In some of your services you do have a material being deposited on the walls of the pipeline. Eventually, as this deposit changes the diameter of the inside of the meter you will have flow error in the meter, therefore it behooves you to periodically clean it out and this is the simplest type of installation to accomplish that clean-out.

Likewise in this installation the meter is shown in a vertical direction—vertical installation, which is the preferred installation from two standpoints. First, although this is not a great problem in fertilizer plants, it insures that the meter section is always full of fluid. In a horizontal line it is possible to get a partially filled meter which reads the velocity of the fluid properly, but it also figures in the air that is going through along with the fluid and therefore for the liquid alone you are getting a slight error in reading.

In this installation is pointed out the one problem to look out for if you are installing your magnetic flowmeter in a horizontal direction. That is, that the electrodes should be positioned not on top and on the bottom, but on the sides, because when they are on the top and bottom the one at the top tends to get all those air bubbles clustered around it and the air is, of course, quite an effective insulator.

The only thing that can possibly happen in your horizontal installation is that after you have shut down the fluid any solids in the material will then tend to settle to the bottom of the magnetic flowmeter and more cleaning than necessary might be encountered in a horizontal installation.

The next slide deals with an old fertilizer problem—it's nothing new with magnetic flowmeters—but it deals with dust and corrosive films and vibration and all of the problems that the instrument makers have been beating you people over the head with for years, I think; but, obviously, this unit as well as any other instrument has to be located where it will perform and where it will have a reasonable life. You cannot attach the recording device directly to a column which is also in contact with heavy vibrating equipment.

Those of you who have tried it know that you can not even keep the ink in the recorder and it goes all over the place, so that if you have vibration problems and you do not have a suitable location for it, then it becomes very wise to try to rig up some sort of a shock mounting to take care of these vibration problems.

The dust and corrosive fumes, of course, can be taken care of by simply running an air purge into the instrument case. This assures that the air will be constantly bleeding out of the case and the fumes and dust cannot get in. This will make a considerable difference in the life of the instrument.

Next we go back to problems which are unique with the magnetic flowmeter and we deal with the problem of protecting the liner in the magnetic flowmeter. This liner is the insulating piece between the fluid and the metering elements themselves and if it becomes cut the meter is shorted and inoperative.

Therefore, when you are doing any moving of the meters—in other words, if you have a system where possibly you may be running several materials through the flowmeter, you might be changing your lines from time to time, it would be wise to do as is shown in this illustration, attach spool pieces to the end of the meter which then will take all the bouncing as this meter is moved around, rather than exposing the ends of the liner which overlap the flanges and can be damaged to accidental damage.

Many people have, in moving their meter from the crate to the installation site, attempted to walk this meter on its end such as you would with a barrel.

This cuts the lining. We have even had some users who have actually snipped off the pieces of liner which come out around the flanges.

This may sound silly, but it has happened.

So these are things to avoid, and incidentally, in one of the further things that we will discuss, the grounding of the meter, having these spool pieces on here you can connect your grounds directly to those spool pieces and therefore in moving the meter you will not be constantly connecting and disconnecting grounds.

The next item that we will take up is the grounding of the magnetic flowmeter, which has proved to be a very essential part of satisfactory operation of the meter.

Actually the real necessity of grounding this meter completely was not even realized by the manufacturers until they went into some of the chemical pipes at which point we found we had no contact whatsoever with the fluid and it was necessary to get some sort of a ground between the meter bodies and the fluid in order to have the system operate properly.

Therefore, on all of the meters that we are supplying today we have ground flange straps attached to the flanges and to the body so that your operators will realize that this must be done.

In addition, almost all of these things that we are talking about at the moment, are discussed in a small tag which is attached to each one of the magnetic flowmeters that is shipped out.

Naturally these things are also in our instruction bulletins, but we find that this tagging procedure, again in this cartoon form that we're showing here, has been very effective in guiding operators to proper use of the meters.

For grounding itself it is necessary in a metal pipe line simply to attach the ground straps on the meter to the metal pipeline either to the flanges or the pipeline itself and then to a good ground such as as a cold water pipe.

In plastic pipe it is necessary to provide an insert flange which will go between your meter flange and your piping flanges and acts—this must be a metal flange which will give you a grounding contact with the fluid.

On the same subject of electrical precautions with magnetic flowmeters immediately comes to mind the handling of the signal cable from the magnetic flowmeter which is carrying quite a low-voltage induced signal back to your secondary instrumentation. This signal cable must be the special signal cable supplied by the manufacturer and it must be run inside a conduit for protection and for isolation from stray electrical pickup.

This means that in that conduit you do not run any of the power sources either to the meter or to any other type of equipment.

Again a final problem to look for on installation is to make sure that all of the equipment that has been supplied with the installation kit is used. We have discussed most of these pieces; we supply in addition a dusting talc for use on the flanges and on the gaskets so that when you disassemble the meter at a later time it does not tend to pull the liner away from the meter.

We supply also seals for the conduits so that you can keep moisture out of the inside of this meter where it will damage the coils.

This covers most of the installation problems that we have found in our different operating histories of the magnetic flowmeter in the fertilizer industry.

One further thing that might be mentioned is in the start up of the meter after a shut down—this is something that has caused people troubles—after having the meter shut down for 24 hours or 8 hours or what have you, the operator zeros his instrument again before starting and finds that he is getting errors.

Well, the answer to this is that the meter should be flushed out before this zeroing procedure is tried because of what has settled out and the fact that you do not have your normal process fluid in the meter at that time and filling the meter.

All of these things that I mention are in the instruction bulletin; they are on the tags which we are now supplying with the meters, but these are the things that have come to light during our several years of experience in the fertilizer field with this meter.

We have on the table by the door some prints of what we call helpful hints to the housewives which are these fine points which have been overlooked in so many installations. These are in a pile on the table by the door and are available on your way out.

In further assisting in the service of this instrument there are many other hints in this little bulletin.

To go from problems of what we consider our problems of the past we think that most of the people who are now using meters have been informed and that the precautions which are now being taken with new meters will prevent this sort of thing in the future, let me just dwell briefly on the newer things in the magnetic flowmeter line.

First of all you will see in this coming years transistorized or solid state secondary units for the magnetic flowmeters. There is already available an indicator which is transistorized and during this next year a recorder also which is transistorized will be available. This, of course, eliminates any of your problems with electronic tubes as well as reduces the size of the secondary instrument considerably.

The magnetic flowmeter is also pushing into much lower conductivity fields, initially limited to a minimum conductivity of 20 micromhos per centimeter, it is now available for conductivity as low as one tenth of a micromho per centimeter.

This means that it is within the realm of possibility that by the end of this year there will be a number of installations handling anhydrous ammonia in addition to these other more conductive fluids that have been handled in the past.

Also the magnetic flowmeter is getting into mass flow measurement. In other words a measuring device which will give you pounds without regard for density changes in the material. This is accomplished by using it in conjunction with a radiation density gauge, both of which read out on a single recorder in terms of mass flow.

These then are the things which we have found with the magnetic flowmeter and which we look forward to with the magnetic flowmeter and we feel that this device will be around for many years helping you people and other industries as well.

Thank you.

MODERATOR REYNOLDS: Thank you, Al.

The next speaker on the program is also taking a big interest in our industry and its future. Mr. Bill Strauss, Manager, Chemical Industry Sales, Foxboro Company, will discuss case histories and new developments in instrumentation for the fertilizer industry.

Case Histories and New Developments In Instrumentation for the Fertilizer Industry

W. I. Strauss

IN today's forward-looking society it is certainly not appropriate to dwell at length on past achievements. However, it is always profitable to look back and see what we have accomplished so that we can better acknowledge what the future has in store for us.

A brief look into the fertilizer industry of yesteryear would show that our production methods were aided by such simple but very useful forms of instrumentation as pressure gages on tanks and outlets of pumps, thermometers in vessels measuring liquid temperatures or placed in dry feeds on conveyor belts, thermocouples measuring the temperatures of various points in a process such as at the dryers, and flow instruments which indicate quantities of various ingredients fed into our processes. If there were records, and in many cases they were necessary, a man with a log sheet would record the various values at specified time intervals. These records, although better than none at all, were far from sufficient when previous operations were to be analyzed because they only showed random point readings and not trends.

It is at this point that we can make a sharp division from the past to present even though some of the time-honored methods of measurement are still used in non-critical locations. The instrument companies, along with some users, realized the existing limitations and developed an instrument recorder. Whether the records are on round or strip charts is of little consequence since we can now have daily continuous records for the use of production people. If laboratory analysis shows that a product does not meet specifications, it is now a very simple matter to look at the records of the operation and find out where corrective action should be taken. The recorder also provides the operator with constant data on how his operation is proceeding.

This instrumentation worked very well up to a point. When the variables that were being manually controlled changed only gradually, it was perfectly satisfactory for an operator to look at the record on the instrument, see if a change was required, and turn a valve in the process line for a correction. If the correction was not enough or too much, there was still time for another try at it. As specifications became tighter, this type of manual control did not suffice. Instrument people then developed an automatic controller. In this discussion it is not necessary to cover the evolution of electric or pneumatic control functions. As we all know, we have available proportional, reset and derivative control. What did this control do for use? It simply did automatically those process adjustments the operator once had to do manually. The instruments were equipped with internal feedback which enabled them to sense if the change it was making was correct; and if it wasn't, to make an additional adjustment.

A specific example is the use of instruments on phosphoric acid or nitrogen solution. In the beginning a rotameter was used, with corrections for temperature and viscosity. Soon indication was not enough and a recorder was used. Along came the development of the magnetic flowmeter which eliminates the need for temperature and viscosity corrections for volume flow measurements. However, operators still made corrections manually. Finally, with the reliability of the measurement established, automatic control was utilized, with the result that what was formerly a production headache is now a routine operation.

Where to next? First of all, as some manufacturers are aware, more and more variables should necessarily be recorded and controlled on one floor, another several hundred feet away and still another on the floor above. Efficient operation would require too many operators. With automatic control and the advantage of measurement transmission, a centralized control panel is not only practical but a necessity. From the control point generally one operator can control all variables in the plant: flow, temperature, level, pressure and analysis. It might be advantageous to add a flow or graphic presentation of the process in the centrallized control room to assist the operator. Certainly, industrial instruments, although ruggedly built and designed for plant operation, work more efficiently and have less maintenance requirements when housed in the clean atmosphere of the control room. Some producers of fertilizers now have centralized control rooms that have dust-free atmospheres, probably the only spot in the plant to commend such gracious living. It is, however, a necessity for efficient plant operation.

Automatic control has become routine for instrument manufacturers. For example, exact ratios of two liquids fed into a process can be controlled automatically within highly accurate tolerances. Certainly better quality product should result. An article written recently by Mr. Paul F. Truitt of NPFI states "Fertilizer manufacturers overformulate at an annual loss of an estimated 6 to 8 million dollars." Instruments, correctly applied, will assuredly help reduce these losses.

So much time is lost between the sample taking and the laboratory report that this technique can not cope with high speed processing. Our production rate per hour is too high to afford the luxury of laboratory analysis for production control. Continuous in-line anaylysis instruments, on the other hand, offer vast improvements in production control and product quality. There are many proven and tested analysis instruments on the market today. pH, ORP, conductivity and capacity are all routine. Relating troublesome variables to any of these measurements affords a rather simple solution to what may appear on the surface an unsolvable problem in production.

This does not mean that instruments will obsolete the laboratory. Instead, the laboratory will be relieved of routine production checks and could devote more time to fruitful research. Nor does it mean that we have reached Utopia and that as soon as a production problem is discovered or an improvement desired one merely obtains the counsel of an instrument manufacturer. Granted many problems could be solved this way. However, there is a great need for certain instruments that are not available today. The need for continuous in-line analysis instruments for moisture content of both feed and product would be most advantageous, as would continuous analysis of nitrogen. These are a few of the instances where new solutions are needed.

Now that we have looked into the past and present accomplishments and peered into the crystal ball of the future, it can be seen that we are only on the threshold of what is in store for us. There are many successful production improvements ahead and these can only be realized by a joint effort on the part of both fertilizer and instrument manufacturers.

Moderator Reynolds: Thank you, Bill.

To go on to our next speaker. Throughout this program we have heard considerable emphasis placed on better control of liquids going into process. Likewise we must maintain the same level of controls with the feed of the dry materials.

We requested one of the gravimetric feeder manufacturers to provide an insight into how improved weight accuracies can be achieved.

It is my pleasure to introduce Mr. Don Warren of the Omega Machine Company.

Discussion of Gravimetric Feeders In Fertilizer Formulation

D. L. Warren

I AM glad that Joe Reynolds scheduled this talk under the heading of Instrumentation because the Gravimetric Feeder as well as meters, controls, recording devices, etc., is an instrument. Unfortunately, too many people have used Gravimetric Feeders more as rock crushers in the past.

Several reports earlier in this program have given you information regarding the wide chemical variations in fertilizer ingredients. While they are reasonably wide from a percentage standpoint, they are probably not nearly as wide as physical variations, and in particular, the bulk density. Here are a few examples of the wide variations which can be found in a few well know fertilizer ingredients.

This will immediately indicate to you the importance of having feeding equipment which can compensate for density changes. When this is done an accurate flow is maintained by weight and this, of course, adds up to formulating closer to grade—more profits. Gravimetric Feeders offer a great deal of flexibility in formulation if they are of a type which have a wide

Bulk Density Range—Lb./ Cu. Ft.

Mat'l.		Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4
Super	HI	54.5	62	73.7	79
	LO	45.5	50	58.8	65
Ammonium Sulphate	HI	68.7	58.7	67.9	63.4
	LO	58.5	47.6	58.7	49
Ammonium Nitrate	HI	72	55	55	68
	LO	62	50	47	62
Potash	HI	76.7	72	76	
	LO	66.2	66	63	

range of rates. They can generally be added to existing plants without taking up too much extra space. If they are set up as a multiple application, by that I mean one feeder for each of the generally used ingredients, they can all be set to deliver their particular part of the formula directly to the ammoniator and eliminates a batching step. With these feeders we can give you secondary instrumentation, such as totalizers and recorders. Another advantage is the built-in alarm system and they can also be used for pacing secondary flows if flow responsive blending is desired.

The features to look for in Gravimetric Feeders are:

a. Variable speed belt—allows direct-reading totalizer, heavy beam load and simple weight check.
b. Positive drive weigh belt, wherein slippage cannot occur. some sort of toothed drive
c. As accurate as possible commensurate with a decent service life.
d. Supplemental power for the operation of the control gate—if the actual weight of the beam through leverages is expected to control the gate then friction can frequently cause inaccuracy.
e. Stainless steel parts in contact with the material being fed.
f. Suitable for suspension type installation where head space or other reasons limit the available floor area.
g. Shear pins are a must. Incidentally, we recommend that as a part of supervision sheer pins be inspected. We frequently find that people use oversize shear pins and needless to say, this can cause a great deal of damage.

Here are a few installation tips:

a. Avoid excessive vibration. Over vibration is not harmful; in fact, I feel that it even helps. It keeps the feeder operating freely. Excessive vibration, however, which can be imparted to the mounting structure under the feeder from pulverization equipment, conveyors, screens, etc., can be detrimental to accuracy.
b. *Never, never* mount directly over the ammoniator. This method of installation has caused more inaccuracy and probably lost more money than any mistake that can be made in the application of equipment in a fertilizer plant.
c. A bin gate for isolation of the feeder from the hopper is recommended. If a belt is to be changed, or other maintenance has to be done, it is always nice to out the flow from the hopper.
d. Always leave room to change the belt, catch samples and do routine maintenance around the unit.
e. Have the unit accessible to operators and maintenance men. Occasionally, we see these suspended from rafters, in almost impossible positions, and is it obvious that feeders in these locations will not get the attention they need.
f. There are several methods of hopper agitation, such as pulsating sides to the hoppers, plate type agitators, or vibration. Vibration is mentioned last here because it is only effective if carefully controlled. It should be integrated with the feeder switch so that it cannot vibrate when the feeder is shutdown. Even better, a simple system of contractor on the feeder belt in conjunction with a time delay relay allows, not only control of the duration of the time the vibrator is on, but also can make the vibration intermittent and proportional to flow. Obviously, if vibration is continuous the fertilizer can be packed into the throat of a feeder with the resultant troubles.
g. A bar screen close to the inlet chute of the feeder is a must. Even though the materials may have been re-pulverized and screened, it is possible for the product to build up in the corners of a hopper, etc., become caked and then drop down into the feeder. Of course, the shear pins should give if a large lump gets wedged under the gate and protects the equipment, but, if, as mentioned earlier, oversize pins have been used — watch out. In operating the feeder let me repeat:

1. It is not a rock crusher;
2. When all else fails — read the instruction book;
3. We like to use the D. W. T. Theory (Distance, Weight and Time) of calibration. This is just simple arithmetic and we know that a certain weight of material is carried per foot of belt and when this is multiplied x time, we, of course, have rate. When checking or calibrating a feeder the three parts of this equation should be checked separately. If we run off, for example, 10 feet of belt and the belt load is set for 30 pounds per foot we should have 300 pounds. If, after three samples are taken it is found that the weight is not 300, but that the difference from the desired load is about the same on each of the three samples then we know that it is merely a question of changing the tare weight. If the three samples show wide variation then it is not tare and other points should be checked, such as friction in the system, breakage in the scale leverage system, frozen bearings, etc. Once we have established a definite load per foot of belt then by varying the speed of our belt through our speed changer it is very simple to come up with a rate based on tons per hour, or pounds per minute, whichever is more convenient.
4. When taking samples the longer sample that can be taken the better. This reduces the human error involved.
5. Once a feeder has been checked out using the sample catching and weighing technique it is very simple to set up some test chains which can be fastened at the inlet chute and allowed

to lie flat on the belt, as the belt is run. Working from the scale beam on the feeder it is simple to come up with a value for these chains and thereafter these can be used to simulate the live load.

6. We repeat—don't use heavier shear pins than the manufactuer recommends.
7. Watch out for build-up around the weigh belt, scale leverage, feeder linkage, drive, control gate. Because in these areas any build-up will not only cause inaccuracies, but will contribute to excessive wear in the equipment.
8. Try to limit the personnel authorized to operate and maintain the feeders to a few who have been checked out on the unit and know what they are doing. One of the greatest problems in this field is people with "fingeritis." These are the people who don't know what they are doing, but walk up and turn a knob, or reset a rate changer, move a tare weight, before they have analyzed the problem and isolated it according to the D. W. T. Theory mentioned above.

In conclusion, I would like to tell you a little story that happened in a fertilizer plant which indicates the way some segments of the industry regard their equipment. I was starting up a plant and the manager had told be not to use any tools because of the union. He said I will send over some maintenance and production men and I want you to train them very carefully. Well, when the maintenance men showed up it was obvious that they had been used to working on pieces of equipment wherein welding torches, heavy hammers and big pipe wrenches were required, because that was what each of three men carried with them respectively.

I told them that I would show them the calibration technique and asked one of them to remove the guard from the chain drive on the side of the feeder. This guard is made up of laminated fiberglass with polyester resin reinforcement and is strong functionally. Many of you may have seen me lay this guard on the ground and jump all over it; during demonstrations of the equipment. We think it is fairly rugged. The man removing the guard, however, thought it was a piece of steel and exerted quite a lift on it when removing it. Of course, it was much lighter than he had anticipated and he looked around, surprised and asked me what is this material. I told him it was laminated fiberglas. He then turned to one of his other co-workers and said "Joe, did you know this was fiberglass?". Joe said "I sure did, I tried to weld a bracket on to it last week."

MODERATOR REYNOLDS: Thank you, Don.

There is one more person on this particular panel.

Of equal importance to the production of high quality granulated mixed fertilizers is the moisture removal techniques that are used.

The principals of drying are well known, but how do we control the application of this heat?

Mr. Bill Law, Industrial Sales Manager, Minneapolis-Honeywell, will discuss this subject.

The Application of Heat

W. J. Law

I WILL discuss today the use of instrumentation or temperature control and the measurement of temperature on granulator drying kilns.

Moisture content of your product is the variable to be controlled and all our efforts on recommendation of suitable control systems, has this end result in mind.

It is difficult if not impractical to measure and control continuously the actual moisture content of your products as it is discharged from your drying equipment. This is usually done in your laboratory on a spot test basis.

This requirement of moisture control was fulfilled by controlling the exit air temperature from dryer at approximately 250°F. for a particular product specification. It was shown that if this exit air temperature was controlled within plus or minus 5°F. the moisture content of product would hold within specifications.

It is possible to control exit air temperature by insertion of a thermocouple or measuring element in breeching or ducting immediately as it leaves the exit end of dryer. This measuring element is connected by wire to a circular chart recorder potentiometer graduated 0 to 600° F., which throttles the fuel to burner in very small increments on a slight change in temperature away from set control temperature point.

A control of this type, either electric or pneumatic operated, connected to a control actuator on fuel line, should give a straight line control of exit air temperature.

Such a system has a distinct advantage over hand control as the operator can preset his exit air temperature say at 265° F., for brand (X) product from a previous record of successful operation after moisture check—this control point index can be increased or decreased by operator to obtain results desired. Once established this temperature can be controlled without too much attention from the operator.

This is the practical method of temperature contol on most dryer units.

In order to guard against excessive air temperatures from burner chamber, an indicating control pyrometer is installed to measure temperature of throat or inlet air to dryer. The burner could raise combustion and throat temperature beyond a point of safety and harm the firebrick, sheet metal or the product itself. This controller can take over and cut the fuel to a low or off position until the temperature is lowered to a safe operating condition. Normally this instrument will not operate except as a high limit or a watchdog. In some installations a recorder is used for this measurement or just an indicator without control.

On some critical products the temperature of the finished product is measured, recorded, and controlled. This is accomplished by a thermocouple in the product itself as it leaves the dryer.

Other applications only require the recording feature with a top limit control to cut burner to low position.

Whenever fuel is fired on industrial ovens or dryers, it is necessary to install flame safeguard equipment to cut-off fuel on flame failure or main burner or pilot. During operation it is possible to lose flame for a short duration, due to momentary fuel failure, excessive drafts, dirty burners, failure of combustion air. If this occurs flame is lost, and fuel comes back on, there is a possible explosive hazard due to accumulation of raw fuel in combustion chamber, especially when an attempt is made to relite. With this flame safeguard equipment consisting of an ultra-vision detector, sighted on flames, as soon as flame is lost—the unit will immediaely cut-off fuel and require a sequence on manual start-up when fuel is again available.

(Applause.)

Moderator Reynolds: Thank you very much, Bill.

We are really indebted to this group of experts here for their presentation. We are sorry we don't have time for some questions.

We are going to cover one more topic. Mr. Frank Nielsson is going to pinchhit here for our good friend, Jim Archer, who was unable to attend this session. He is going to discuss the subject of Sampling. The program indicates "Automatic Sampling" but I believe that Frank has a little different version here on sampling and I am sure it will be well received.

Sampling and Analysis

F. T. Nielsson

THE industry is conscious of the importance of shipping quality fertilizer, that is, fertilizer which will meet analysis guarantees, satisfy customers, and comply with state control laws.

Now, how do we go about the job of determining that our products meet these standards?

The answer would appear to be rather simple — take a sample and make a chemical analysis.

I wonder if the answer is as simple as it sounds.

The late Dr. W. F. Hillenbrand, chief chemists, U. S. Bureau of Standards, made this statement: "The sampling of the material that is to be analyzed is always a matter of importance and frequently a more important operation than the analysis itself."

The comments which I will make will be confined to the taking of samples and the problems involved and the technique which the control man must prescribe as he goes about the business of determining whether or not the fertilizers of his company meet standards.

Perhaps, the logical approach for the control man is to refer to the section on sampling, as it appears in the publication of Official Methods of Analysis of the Association of Official Agricultural Chemists, and determine if these instructions are adequate for the job and, for the present, let us confine our thinking to the first sentence of the section which reads: "Use *slotted single or double tube,* or *slotted tube and rod,* all with pointed ends." This sentence deals with sampling instruments but details as to design and dimensions are not given. Therefore, the control man may use one of many instruments and still comply with the instructions.

For example, either of the two instruments having the following dimensions should be satisfactory:

	Length	Tube Diameter	Slot Width
⅝" Double tube	26"	⅝"	½"
1" Double tube	20"	1"	⅞"

The slot width or width of opening which receives the sample is ⅞" for the larger tube compared to ½" for the smaller tube.

Sample men are human and will ordinarily select the smaller tube, since the sample can be taken with less effort.

A double tube sampler of the so called "Indiana design" is difficult to operate, as fine particles of fertilizer sift between the two sections and the instrument will not open to receive the sample. However, the instructions state that a single tube is satisfactory. Therefore, samples are often taken with the single tube section of the sampler.

When only the single tube section is used a portion of the sample core can easily be lost as the sample is withdrawn, because the opening in the tube which receives the sample is exposed to the torn edge of the bag.

The following analyses were made on duplicate samples taken with the single tube sampler and shows the effect of sample loss:

0-20-20 Fertilizer

Nitrogen	A.P.A.	Potash	
0.00	19.50	15.90	First Sample
0.00	21.90	17.80	Second Sample

The A. P. A. is more than 2 units higher in the second sample and the potash approximately the same.

The large diameter double tube is shown on the attached sketch and its design is such that the problem of freezing of the double section does not occur.

This example gives the screen analysis comparision of samples taken with the small (Column C) and large diameter sampler (Column A) and of a sample taken by riffling the entire bag to laboratory size (Column B).

12-12-12 Fertilizer

	% Retained on		
Screen Size	1" Sampler (A)	Bag (B)	⅝" Sampler (C)
4 Mesh	0.6	0.9	0.4
6 Mesh	13.2	15.1	8.7
8 Mesh	33.6	31.7	21.2
10 Mesh	53.1	51.0	37.1
16 Mesh	92.3	89.9	83.7
20 Mesh	95.4	93.3	83.3
–20 Mesh	4.6	6.7	11.7

The screen analysis of the sample taken with the large sampler is essentially the same as the bag;

whereas, the sample taken with the smaller sampler differs from the bag; hence, we can conclude that the smaller diameter does not receive or loses large particles.

If the fertilizer was completely homogeneous, the proper proportion of particle size would not be of importance. However, few fertilizers are completely homogeneous and the next example gives the effect on analyses caused by the sample not containing the proper proportion of particle sizes.

Granular 20-0-20 Fertilizer

Sampled With	Nitrogen	A.P.A.	Potash
Double Tube	20.00	—	20.75
Single Tube	16.53	—	23.56

The sample taken with the larger diameter double tube has a very good analysis; whereas, the sample taken with the smaller diameter single tube indicates a substantial underrun in nitrogen and an overrun in potash.

After a sample is taken it must be reduced to laboratory size and this, according to the instructions, is accomplished preferably by riffling or by mixing thoroughly on a clean oil cloth.

Riffling is the preferred method. Several types of riffles are commonly used, many of which are not satisfactory in the hands of a non technical sample man. A riffle which does not have a guide or other means for determining that the sample is evenly distributed over the dividing section is not satisfactory.

The next example demonstrates what can happen to an analysis when the sample is not properly distributed on the dividing sections.

10-2-10 Fertilizer

Nitrogen	A.P.A.	Potash	
9.01	2.25	10.80	Cut No. 1
10.90	2.14	9.79	Cut No. 2

The fertilizer collected in one pan (Cut No. 1) indicates an underrun on one unit in nitrogen; whereas, the fertilizer collected in the other pan (Cut No. 2) indicates an overun in nitrogen.

Riffles are available which have a feed hopper positioned above the dividing section which restricts the fertilizer thus providing an even distribution of the flow to the dividing section.

The next example gives the analysis of a sample which has been divided three times or reduced in size by means of a riffle having the feed hopper feature.

10-2-10 Fertilizer

Sample	Nitrogen	A.P.A.	Potash	
No. 1	10.44	2.37	10.20	North Portion
	10.46	2.37	10.06	South Portion
No. 2	10.50	2.39	10.08	North Portion
	10.40	2.37	10.10	South Portion
No. 3	10.53	2.38	9.90	North Portion
	10.53	2.37	10.01	South Portion

The North Portion represents the fertilizer collected in one pan and the South Portion represents the fertilizer collected in the other pan. Under Sample No. 1, the nitrogen is 10.44 compared to 10.46; under Sample No. 2, the nitrogen is 10.53 compared to 10.53. Observe the good agreement in A. P. A. and potash.

Finally, the laboratory sample must be taken. Usually the sample is placed on a cloth and rolled thoroughly to mix.

After rolling the sample is normally scattered over the surface of the cloth in a thin layer. A small representative portion must be taken for the analysis. This is done with a scoop by taking portions from the entire area.

Dense heavy particles lay close to the cloth and it is very difficult to obtain a representative laboratory sample from a thin layer and it is preferred to roll the fertilizer into a pile in the center of the cloth and flatten the pile to about ¾″ thick with a plasterer's hawk before taking the laboratory sample.

The next example shows the difference in analyses where the laboratory portion is taken from a sample scattered over the surface of the cloth compared to a sample assembled or rolled into the center of the cloth. Note: 4-12-12.

4-12-12

	Nitrogen	A.P.A.	Potash
Sample rolled to center before taking laboratory portion	3.70	12.23	11.89
Sample not rolled to center before taking laboratory portion	2.75	11.41	13.99

Observe that the analysis of the sample rolled to the center before taking the laboratory portion agrees reasonably well with the guarantee; whereas, the analysis of the sample not rolled to the center shows a substantial underrun in nitrogen and APA and an overun in potash.

Summary

Detailed procedures, in order to obtain better agreement between state and industrial laboratories, were called for by the past president of the Association of American Fertilizer Control Officials.

The data presented indicate that detailed sampling instructions are necessary in order to obtain representative samples.

A company's most precious asset is its reputation and ability to distribute quality products. When a laboratory fails to use sound sampling and laboratory technique—reputations suffer.

Thank you.

MODERATOR REYNOLDS: Thank you very much, Frank.

Remember we are meeting here tomorrow morning at 9 o'clock; we sure appreciate your staying with us; your cooperation and your attendance.

Thank you.

(The Meeting adjourned at five-forty-five o'clock p.m.)

Friday Morning Session, Nov. 4, 1960

The Round Table reconvened at nine o'clock a.m., Messrs. Joseph Reynolds and Albert Spillman presiding.

MODERATOR SPILLMAN: Good morning to all of you.

Today our Potash Panel will conclude our format on standardization of raw materials. We are confident the producers of raw materials will carefully consider the problems discussed yesterday by the User-Mixer panel and will do everything that possibly can be done to improve nomenclature and uniformity, both chemical and physical. The leader for our Potash Panel is our good friend, Dr. Ed Kapusta of U. S. Borax Company. Other panelists are: N. T. Wendt, American Potash & Chemical Company, D. J. Bourne, Duvall Sulphur, Ben Heck, International Minerals and Chemical Company, Dean Gidrey, Potash Company of America, and Van Rogers, Southwest Potash Company. Without further comment, I turn the meeting over to Dr. Kapusta.

DR. E. KAPUSTA (U. S. Borax Company): Thank you, Al. At this time, I would like the participants in the Potash Panel to take the front seats. We need as many people as we can get up here in front for moral support, so if the Potash people will please take their places.

Members of the Round Table: on behalf of my associates in the potash industry and myself, I would certainly like to thank you for this opportunity of appearing on your panel to discuss this topic. I see we have a wide awake, alert group here and we will certainly appreciate any questions or comments you might have on this after we have finished with our presentations.

You are all aware of the advances in fertilizer technology which have taken place during the past decade. You have had the development, application of granulation processes, you have had the continuing trend toward the manufacture of higher analysis fertilizers, you have had the growth of bulk blending, liquid fertilizers. These are some that are more prominent.

Some of these advances, of course, were made possible by the development of newer and improved types of raw materials by the basic raw material suppliers. In other cases, the application of these newer technologies brought about the need for different or improved types of products. We, the potash producers, as well as the nitrogen and phosphate producers, have invested considerable sums both in research and process development and in plant investment to provide you with what we hope are the types of products which you need in your operations and we, of course, continue to do research to develop newer and better products for your use. Accordingly we welcome this session to gain a better idea of the types of materials which you will be wanting now and perhaps in the future, as advancements in technology continue.

Many of you in attendance here are engaged in production and other technical activities. I am sure you are all aware of the problems which arise in attempting to make a wide variety of products in large tonnages as we in the potash industry do. As has been mentioned previously by speakers on the Phosphate Panel, although it is physically possible to make just about any kind of product you want, usually it is not economically practical to do so. Somebody has to pay for this thing. I think we have to be reasonable about stating the types of products which we think we need. I think we have to have a better idea of the types of products which we need for our operations before we can set down definite specifications.

The assignment as initially made to this group was that of describing to you the properties of the agricultural potash products currently being marketed.

In the past few days, the base of discussion has been broadened somewhat. There have been questions raised concerning tolerance in specifications, others have spoken on methods of manufacture as they may affect properties of their particular products, consequently I have asked the members of this panel to, if they wish, go beyond the description of the properties of their particular products and touch upon any related fields of production or use of these materials.

Our format for this morning is going to consist of each of the individual company representatives describing their particular products and making any other appropriate remarks. I would ask you please to defer questions until we have the presentation from each of the participants. In that way we may avoid any repetition and subsequent speakers may answer questions which may arise.

We hope to keep to a rather tight schedule. I have advised the speakers to limit their remarks to ten minutes, I think we can do it, and, again, we want questions; we would be delighted to answer them.

To get the ball rolling here this morning we are going to call upon Nelson T. Wendt with the American Potash and Chemical Corporation. As you all know, American Potash and Chemical is the only major producing company not located in Carlsbad. Nelson, it is a pleasure for me to introduce you to this group.

American Potash & Chemical Corporation Potash Materials for the Fertilizer Industry

Nelson E. Wendt

AMERICAN Potash & Chemical Corporation has been producing potash salts for the fertilizer industry since 1916 at its Trona, California plant, located on the edge of Searles Lake in the Northern part of the Mohave Desert. Incidentally the word TRONA, the town name and the long-established trademark for products of my Company, is of Egyptian origin and refers to a double salt sodium carbonate — sodium acid carbonate found in reefs on the Eastern edge of Searles Lake. At the Trona plant, the dense alkaline brine, pumped from the porous salt body of Searles Lake, is treated by evaporation followed by fractional crystallization to separate potash from the other brine components—mainly sodium chloride (salt), sodium sulfate (salt cake), sodium carbonate (soda ash), sodium tetraborate (borax), and a variety of other chemicals including lithium and bromine.

The potash is separated as muriate or KCl. Carefully controlled conditions of crystallization in Swenson crystallizers, washing, drying, and screening of a portion of this potash result in our present Standard (or crystalline) Agricultural Grade of Muriate of Potash for the fertilizer industry. Part of the potash mentioned goes to a battery of Komarek-Greaves pelletizers where it is compacted, crushed and screened, to give our present Granular Agricultural Grade of Muriate of Potash.

Some of the potash is also converted to Sulphate of Potash. For economic reasons, including our location, production cost factors, etc., our Agricultural Sulphate is marketed in only a limited sales area roughly comprised of the eleven western states. Finally, in passing, I would mention that we reprocess and recrystallize another portion of our potash to produce a high quality Chemical grade Muriate of Potash for a substantial chemical industry market.

Of primary interest here today, then, are the two grades of muriate which we have most recently developed for the fertilizer industry, namely:

(1) our Standard or Regular Agricultural Muriate, a crystalline product guaranteed 60.5 per cent minimum K_2O (95.8 per cent min. KCl equivalent).

(2) our Granular Agricultural Muriate, a compacted coarse granular product also guaranteed 60.5 per cent minimum K_2O (95.8 per cent KCl equivalent).

Our specification Sheet for the Standard material reads as follows:

Standard Agricultural Muriate of Potash

Specifications

Analysis	Wet Basis Guarantee	Typical*
K_2O	60.5 % min.	61.23%
Equivalent KCl	95.8 min.	96.93
$Na_2B_4O_7$	0.50 max.	0.31
H_2O		0.05

Miscellaneous Impurities**

KBr—1.56%; NaCl—1.45%; Na_2SO_4—.09%; Organics—.007%; Na_2CO_3—.25%; H_2S—.0073%

Cumulative Screen Analysis

U. S. Sieve No.	Guarantee	Typical*
+ 8 mesh		0.1
20		10
30		36
50	80% min.	90
100		99.2
200		99.8
Poured Density lbs./cu. ft.		70.6
Storage " " " "		72.7
Angle of Repose from horizontal		37°

* Typical analysis is unweighted average of 6 months production analyses.

** Unweighted average of September 1960 production.

Our Specification Sheet for the Granular material reads as follows:

Standard Agricultural Muriate of Potash

Specifications

Analysis	Wet Basis Guarantee	Typical*
K_2O	60.5 % min.	61.36%
Equivalent KCl	95.8 min.	97.13
$Na_2B_4O_7$	0.50 max.	0.31
H_2O		0.09

Miscellaneous Impurities**

KBr—1.29%; NaCl—1.10%; Na_2SO_4—.09%; Na_2CO_3—.22%; H_2S—.0072%;

Cumulative Screen Analysis

U. S. Sieve No.	Guarantee	Typical*
+ 6 mesh		0 %
8		0.4
18		70
30	90 % min.	95
100	99.2 min.	99.2
200		99.6
Poured Density lbs./cu. ft.		59.7
Storage " " " "		62.7
Angle of repose from horizontal		39°

* Typical analysis is unweighted average of 6 months production analyses.

** Unweighted average of September 1960 production.

Our present production specification for the Standard Grade is a screen analysis of 80 per cent or better on a 50 mesh screen. For the Granular, our present production specification is 90 per cent minimum on 30 mesh, and 99.2 per cent on 100 mesh (cumulative basis). Generally speaking, our plant is now accomplishing this. This is not to say that we have not had some deviations from time to time. In developing these newer grades and the processes to provide them, we have had our problems. One of these has been the friability of the Granular Muriate. We understand the problem and believe we are well on the way to controlling it.

In conclusion, in our contacts with you in the fertilizer industry we have found differences of opinion, and changing opinions, as regards acceptable grades of potash for specific processes, or certain grades, or individual operations. The situation has not been static. Nevertheless, we have had a move towards a fairly definite product goal based on generally expressed needs and our production process capabilities. During this year I believe we have made major progress in that direction. Furthermore, we have due on stream in mid-1961 a new evaporation plant which not only will increase over-all production capacity, including potash, but also will improve the efficiency of our operations. How much further we can economically go towards more exacting chemical or screen specifications, more grades for specific purposes, narrower screen limits, etc. is always an important question. We believe we can do most anything if the market justifies it and the customer is willing to pay the cost.

Thank you for this opportunity to tell you about our materials.

Thank you.

DR. KAPUSTA: Thank you, Nelson.

Our next speaker is Douglas Bourne with Duval Sulphur & Potash Company.

Douglas J. Bourne

MR. DOUGLAS J. BOURNE: Thank you, Ed.

I can remember when our instructions to our producing people involved only operation with the highest recovery we could get. We are using a basic flotation process for our principal separation and I think probably five out of the seven major producers in the country are using a similar process or an adaptation thereof. This is not ancient history, this is a scant eight years ago when product quality was of secondary importance to potash producers and efficiency in recovery was the primary aspect in our operation. This flotation process has a number of inherent faults, one of which is that in crushing ore you end up with material which will run perhaps 15 to 20 per cent minus 100 mesh. After we had been producing for two or three years our sales people advised us that it would be quite desirable if we could provide for our customers a granular material, perhaps 10 per cent of our production could be diverted into this market.

We put in the screening capacity to handle this 10 per cent of production and skimmed off the top part of our flotation product and we had what we understood was a quite acceptable product then as a granular material. Of course, you can imagine what happened to the remaining product which already had 15 to 20 per cent minus 100 mesh in it when we took the bulk of the extremely coarse material out of it.

We were then faced with the enigma or the dilemma that has faced us all at one time or another, what to do with the little ones, how to make big ones out of little ones, and as you know if you have followed potash processing at all there are number of different ways in which this metamorphosis can be brought about.

We had the rather dubious distinction of pioneering the high pressure Allis-Chalmers compacters on potash production. We now produce two potassium chloride products, both red products which is again typical with the flotation process: one is a standard material, the other a coarse. Our production has averaged about 60.9 per cent K_2O over the past several years. There is some variation, of course, day to day and car to car but I would estimate that it would be on the order of perhaps 2 tenths per cent K_2O above or below the 60.9 figure.

The screen analysis specification, which, as we have heard many times, is all important today, with respect to our standard we have perhaps 10 per cent plus 20 mesh and we hold the minus 100 mesh to less than 2 per cent. Our coarse product holds the plus 8 mesh to 1 per cent or less, if possible, and we have been told that the most desirable plus 10 mesh figure for the coarse product is between 10 and 15 per cent. We also try to hold to that, and at the same time keep 95 per cent plus 28 or better and less than 2 per cent minus 35 mesha. These are all Tyler screens.

When I mentioned that we are currently producing these products, I did that with a purpose. You all are well aware of the somewhat seasonal nature of potash shipments and while all producers ship a significant tonnage of material directly from production, we all have extensive warehouse facilities.

Just prior to the heavy shipping season, in the spring of the year, it is not uncommon for potash producers to have between 100 thousand and perhaps 3 or 400 thousand tons of material in storage. Now you can appreciate, putting 100 thousand tons of, let us say, a standard product in a warehouse at a given screen analysis and then attempting to withdraw all of this tonnage car by car at the same screen analysis, it is a difficult problem. We have other problems of course, almost as varied and complex as the manufacturers' problems.

I would like to say that we would be most anxious to join with the other producers and with the industry in general in standardizing nomenclature.

Again, at the risk of being repetitious, I would like to make perhaps a trite statement that we are most anxious to provide our customers with the most suitable product we can, obviously, if we can find out what they want and if we can make it at a profit.

Thank you.

DR. KAPUSTA: Thank you very much, Doug.

Because our program was

changed from yesterday afternoon to this morning, unfortunately, Dick Powell had to leave town, however he has asked one of his associates, Bob Heck to speak about International's potash products.

Mr. Robert Heck: Thank you, Ed.

Unlike some of the people who have appeared on this panel before in the last two days, I feel that I can come before you as an expert on potash and give you some of the specifications on IMC potash. The reason I say that I am an expert is because I have been with this industry since January and along with that I am more than 50 miles away from home, I carry a slide rule, and I have a generous expense account, and you can't be any more expert than that.

Since the processes for mining and recovering potash have already been briefly described and some of the problems associated with this have also been described, I will confine my remarks to the specifications on International Minerals products. Could I have the first slide, please.

(Slide) This slide shows the screen analysis on standard muriate of potash as processed by International Minerals. This product contains from 60 to 61.5 per cent K_2O, the typical product being 60.6 per cent and a guarantee of 60 per cent K_2O. The moisture varies between one tenth and 12 hundredths per cent with the typical product being one tenth. I won't go into detail on the screen analysis; you can see that on the slide.

The typical bulk density of this product is about 75 pounds per cubic foot, the angle of repose is 32 to 34 degrees.

This is a typical screen analysis on coarse muriate of potash. This product has a typical K_2O content of 60.7 per cent, guaranteed minimum of 60 per cent. Moisture content is in the same range as standard, typical bulk density is 68 pounds per cubic foot, and angle of repose 32 to 34 degrees.

This slide shows the typical screen analysis on the granular muriate of potash. This product has a typical K_2O content of 60.4 per cent, guaranteed 60 per cent,

I. M. & C. Potash Slides

Standard Muriate of Potash (KCl)

Chemical Specifications	**Range**	**Typical**	**Guarantee**
K_2O	60.0–61.5%	60.6%	60.0%
Moisture	0.1– 0.12%	0.1%	no guarantee
Physical Specifications (Tyler standard sieve sizes)			
+20 mesh	13–21%	17%	
+28 mesh	34–42%	38%	
+35 mesh	60–68%	64%	
48 mesh	78–86%	82%	
65 mesh	89–99%	94%	
+100 mesh	95–99%	97%	
+200 mesh	98–100%	99%	

Typical bulk density—75 lbs/ft^3
Angle of repose—32 to 34 degrees
Typical Chemical Analysis

K	50.54%	SO_4	0.56%
Mg	0.11%	Br	0.065%
Ca	0.05%	Insol	0.28%
Na	1.00%	H_2O	0.10%
Cl	47.26%		

Coarse Muriate of Potash (KCl)

Chemical Specifications	**Range**	**Typical**	**Guarantee**
K_2O	60.6 –61.3%	60.7%	60.0%
H_2O	0.10– 0.12%	0.10%	no guarantee
Physical Specifications (Tyler standard sieve sizes)			
+10 mesh	9–15%	12%	
+14 mesh	44–56%	50%	
+20 mesh	78–88%	83%	
+28 mesh	95–99%	97%	

Typical bulk density—75 lbs/ft^3
Angle of repose—32 to 34 degrees
Typical Chemical Analysis

K	51.06%	So_4	0.42%
Mg	0.09%	Br	0.065%
Ca	0.05%	Insol	0.24%
Na	0.63%	H_2O	0.10%
Cl	47.24%		

Granular Muriate of Potash (KCl)

Chemical Specifications	**Range**	**Typical**	**Guarantee**
K_2O	60.0 –61.0%	60.4%	60.0%
H_2O	0.10–0.12%	0.1%	no guarantee
+6 mesh	9–11%	10%	
+8 mesh	45–55%	50%	
Physical Specifications (Tyler standard sieve sizes)			
+10 mesh	74–86%	80%	
+14 mesh	94–98%	96%	
+20 mesh	98.5–99.5%	99%	

Typical bulk density—75 lbs/ft^3
Angle of repose—32 to 34 degrees
Typical Chemical Analysis

K	50.42%	So_4	0.51%
Mg	0.10%	Br	0.065%
Ca	0.05%	Insol	0.73%
Na	0.86%	H_2O	0.10%
Cl	47.02%		

and moisture range about the same as the other products described, typical bulk density of 64 pounds per cubic foot, angle of repose 32 to 34 degrees.

Sulphate of Potash (K_2SO_4)

Chemical Specifications	Range	Typical	Guarantee
K_2O	50.0 –52.4%	51.5%	50.0% K_2O
Cl	1.50– 2.18%	2.0%	2.5% maximum
H_2O	0.10– 0.12%	0.10%	no guarantee
Physical Specifications (Tyler standard sieve sizes)			
+14 mesh	5–11%	8%	
+20 mesh	6–16%	11%	
+28 mesh	7–23%	15%	
+35 mesh	9–27%	18%	
+65 mesh	27–43%	35%	
+100 mesh	42–62%	52%	
+200 mesh	79–93%	86%	

Typical bulk density—75 lbs/ft^3
Angle of repose—32 to 34 degrees
Typical Chemical Analysis

K	43.08%	SO_4	54.19%
Mg	0.69%	Br	0.005%
Ca	0.11%	Insol	0.33%
Na	0.10%	H_2O	0.10%
Cl	1.35%		

This slide shows the typical screen analysis on sulfate of potash. This product contains 51.5 per cent K_2O, which is the typical analysis. The moisture content is in the same range as the other products described. Chlorine is guaranteed at 2.5 per cent maximum. This product has a typical bulk density of 85 pounds per cubic foot, angle of repose 32 to 34 degrees.

This product, Sul-Po-Mag, has a typical K_2O content of 22.3 per cent, guaranteed 22 per cent, magnesuim oxide typical content of 18.5 per cent, guaranteed 18 per cent, chlorine maximum 2.5 per cent, water varies between 1200ths and 1500ths. The typical bulk density is 100 pounds per cubic foot and the angle of repose on this product is 32 to 34 degrees.

This last product described is a water soluble double sulfate potash magnesia and the magnesium in this product as described is water soluble.

At the present time International Minerals ships potash from Carslbad, New Mexico and in the near future we will be shipping from the Esper Hazey mine in Saskatchewan, and the screening specifications on products in this plant will probably be about the same as the ones described here. I noticed when I said this that Dr. Kapusta grinned. We are digging a good, deep well and we feel that we will be in production at Esper Hazey some time next year.

Sul-Po-Mag®

Chemical Specifications	Range	Typical	Guarantee
K_2O	22.0 –22.9%	22.3%	22.0%
MgO	18.0 –18.8%	18.5%	18.0%
Cl	1.5 – 2.3%	2.0%	maximum 2.5%
H_2O	0.12– 0.15%	0.14%	no guarantee
Physical Specifications (Tyler standard sieve sizes)			
+10 mesh	18–22%	20%	
+14 mesh	34–46%	40%	
+20 mesh	60–68%	59%	
+28 mesh	68–84%	76%	
+35 mesh	81–95%	88%	
+48 mesh	95–99%	97%	
+65 mesh	98–100%	99%	

Typical bulk density—75 lbs/ft^3
Angle of repose—32 to 34 degrees
Typical Chemical Analysis

K	18.68%	So_4	67.39%
Mg	11.30%	Br	0.005%
Ca	0.05%	Insol	0.33%
Na	0.76%	H_2O	0.14%
Cl	1.25%		

Thank you.

Dr. Kapusta: Thank you, Bob. The grin was not occasioned by the remark. I remembered a joke someone told at the Gaslight last night, really.

It is now my pleasure to introduce to you Dean Gidney of the Potash Company of America. As many of you know, Dean has been active in many phases of the potash industry. He is presently, of course, Vice President in Charge of Sales for PCA. Dean.

Dean R. Gidney

Thank you very much, Ed. Dr. Kapusta and members of the Fertilizer Round Table: I am somewhat embarrassed at appearing on this panel of technical experts. Probably the only excuse that I can give you for my being here is that all of the technical people in our company are busy in Carlsbad making the potash which you will require next spring.

In one respect I feel that I am qualified for this panel because my service in the potash industry goes back almost to the days when the fertilizer industry was an industry of cow manure and kainite. Now it is a highly complex chemical process industry. All of the interest in standardization, in nomenclature, in physical characteristics and analysis of potash is a fairly recent development.

At the time when the original producers began producing potash in Carlsbad, New Mexico and a California producer had been producing for many years, our customers could not have cared less about the screen size of the potash. They did not care a great deal about the actual analysis. The reason for this was simply that when the highest potash content of fertilizer was around 8 per cent and when the average was closer to 4 per cent it did not make much difference. The only reason that they cared about analysis was because of freight costs; it was cheaper to ship a 60 per cent material per unit of K_2O than it was a 25 or 30 per cent, but if you used 25 or 30 per cent potash you needed less Cape Henry sand, or Cape Henry phosphate. So, in those days, our sole interest was to make K_2O and make it as fast as we could. The customers were happy and satisfied and in many cases if you sold a man manure salts he was most grateful to you. This was particularly true when World War II cut off imports of Potash from Europe.

In the 1930s we offered such products as 25 per cent manure salts, 30 per cent processed manure salts, which was a mixture of 25 per cent and 60 per cent, 50 per cent processed muriate, and 60 per cent muriate. Our only problem with regard to physical characteristics at that time was the setting up of the material. Many of you in the room know some of the old timers who had very descriptive phrases for the way this material set up. These colorfully descriptive phrases are better left unquoted, but it is true that on some occasions when we shipped bagged potash we were shipping granular—one granule to the bag!

(Laughter.)

So, as you can see, this interest in screen sizes is recent and really began about the time of the Korean War with the accelerated trend toward high analysis fertilizers. It was the development of such high analysis fertilizers which led to the need for granulation of fertilizers. As you manufacturers know, as you made high analysis material in the powdered form, you encountered the setting up problem, which, incidentally, had been solved by the potash industry during the war. I will not say that the solution was 100 per cent complete, but the physical condition of your potash is much better than it used to be, because the potash companies discovered how to keep the material from setting up. The method which worked on potash did not apply to compound fertilizers and some other solution had to be found.

The apparent solution was granulation. This development was at first welcomed by the potash industry because we felt that the product which we had to offer you at that time was the optimum product for the manufacture of granular fertilizer. We believed that the best granular fertilizers were going to be homogeneous and we believed with USDA and other experts that the best homogeneous fertilizer was going to be made with fine materials. We soon discovered that at least for the time being we were in error, and you all know better than I that history of how, when you went to continuous ammoniation, you found that the fine materials did not agglomerate well, particularly in low nitrogen grades, and that you had to have coarser materials. This led to the development of some of the present products which the potash industry now offers you.

I might go back for a moment to say that prior to the war there was one other development initiated by the Carlsbad producers. That was the development of a potash for direct application, a potash which was then known as granular and first appeared as 50 per cent muriate and ultimately became 60 per cent granular muriate. This was originally developed as a direct application product.

We then had going into this period of fertilizer granulation, which really gained impetus in the early 1950s, two products: the standard material and the granular material. The new methods of granular fertilizer manufacture apparently called for a new product midway between the two old ones. From then on each potash company extemporized based on the requirements and requests of its customers. As a result we now have products which are substantially the same but which have different names and which we hope to standardize in nomenclature for your sake as well as for ours.

I have now been asked to tell you in general the products which the Potash Company of America offers to the trade. We have a very wide variety of products and rather than read off to you the analyses here, we have placed over on the table typical analyses of our various products. When I say typical, I am referring to the actual analysis of the material which we have shipped over the past six months.

We produce, as you know, basically by the flotation process. Potash Company of America was the original producer of potash by flotation in the world. Prior to the advent of Potash Company of America all muriate of potash had been made by crystallization. Since the construction of our original plant, there have been many adaptations and our product now is a combination of floated product, fused product and compacted product with some crystallized product as well.

Our three main products are the standard muriate of potash with which you are all familiar, the product to which we now refer as special granular, which is the middle range product, basically minus 8 plus 28, and the product we call coarse granular, and here is where nomenclature becomes confusing to us as well as to you. The coarse granular in our lexicon is the biggest product and is basically a minus 6 plus 14 product.

We also have a crystallized muriate of potash which runs between 62 and 63 per cent K_2O and is a white wholly soluble product whose application is primarily in the liquid fertilizer industry.

We also have sulfate of potash which is converted from muriate at two places, at Dumas, Texas by the Hargroves process and at Ft. Worth, Texas by Mannheim process.

The last, but not least of our products is our chemical grade muriate which is guaranteed 99.9 per cent KCl minimum.

Our differentiation of muriate products now is achieved by screening. There is a limit to what can be accomplished by screening and it is an expensive process. As Doug Bourne pointed out, screening material into a warehouse does not guarantee that each car coming out of that warehouse will have the screen analysis that went in.

Potash Company of America is anxious to produce its products in the forms which will be most satisfactory to the fertilizer industry. We realize that there are as yet no definite standards and that changes in the nature of the product required will continue to take place. It is impossible for any potash company to economically produce potash on a job order basis. For that reason any standardization of product must be on a very broad classification basis. On that basis we would welcome a standardization of nomenclature, with the realization that within the broad classifications there is still room for differences between various companies' products. It is generally true that the smaller the original range, the greater are the expenditures for capital and for operations. Our industry, like all others, must be guided by economists.

We have learned a great deal from you folks in the fertilizer industry. Thank you all very much for your time and your attention, and thank you, Ed.

(Applause.)

P. C.A. Potash Slides

Typical Analysis
Standard Muriate of Potash

Constituent		Percent
Volatile at 130°C		0.10
Volatile at 250°C		0.11
Water Insoluble		0.45
Silicon Dioxide	SiO_2	0.11
Hydrous Oxides	R_2O_3	0.26
Calcium	Ca	0.04
Magnesium	Mg	0.06
Sodium	Na	1.15
Potassium	K	50.43
Chloride	Cl	47.60
Bromine	Br	0.06
Sulfate	SO_4	0.17
Heavy Metals as Lead	Pb	0.01
K_2O		60.75
KCl		96.17

Typical Screen Analysis
Standard Muriate of Potash

Mesh	Cum. %
20	5.0
28	17.0
35	35.0
48	60.0
65	82.0
100	93.0
150	97.0
200	99.0
–200	1.0

Typical Analysis
Special Granular Muriate of Potash

Constituent		Percent
Volatile at 130°C		0.14
Volatile at 250°C		0.17
Water Insoluble		0.76
Silicon Dioxide	SiO_2	0.23
Hydrous Oxides	R_2O_3	0.35
Calcium	Ca	0.07
Magnesium	Mg	0.12
Sodium	Na	0.99
Potassium	K	50.31
Chloride	Cl	47.39
Bromine	Br	0.05
Sulfate	SO_4	0.31
Heavy Metals as Lead	Pb	0.01
K_2O		60.60
KCl		95.95

Typical Screen Analysis
Special Granular Muriate of Potash

Mesh	Cum. %
6	0.0
8	3.0
10	26.0
14	60.0
20	85.0
28	96.5
35	97.5
48	98.5
–48	1.5

Typical Screen Analysis
Coarse Granular Muriate of Potash

Mesh	Cum. %
6	1.0
8	45.0
10	90.0
14	95.0
20	97.0
28	98.5
35	99.0
–35	1.0

Bulk Weight of Products

	Lbs./Cu. Ft. Loose	Lbs./Cu. Ft. Packed
Standard Muriate	74.00	81.25
Coarse Granular Muriate	60.75	66.25
Special Graunlar Muriate	60.50	65.75
Chemical Grade Muriate	62.75	68.75
Soluble Muriate	61.25	67.25
Sulphate (Ft. Worth Prod.)	74.4	90.0
Sulphate (Dumas Prod.)	58.8	99.9

Typical Screen Analysis
Sulfate of Potash
Produced at Dumas, Texas

Mesh	Cum. %
10	10.0
14	30.0
20	45.0
28	60.0
35	70.0
48	75.0
65	85.0
100	88.0
200	95.0
–200	5.0

Typical Screen Analysis
Sulfate of Potash
Produced at Fort Worth, Texas

Mesh	Cum. %
20	5.0
28	7.0
35	10.0
48	15.0
65	30.0
100	60.0
200	92.0
–200	8.0

Typical Screen Analysis
Soluble Muriate of Potash

Mesh	Cum. %
35	1.0
48	15.0
65	60.0
100	90.0
200	99.5
–200	.5

Typical Analysis
Sulfate of Potash
Produced at Fort Worth, Texas

Constituent		Percent
Volatile at 130°C		0.03
Water Insoluble		0.45
Silicon Dioxide	SiO_2	0.11
Hydrous Oxides	R_2R_3	0.26
Calcium	Ca	0.04
Magnesium	Mg	0.06
Sodium	Na	0.74
Potassium	K	43.42
Hydrogen	H	0.01
Chloride	Cl	1.68
Sulfate	SO_4	53.58
Heavy Metals as Lead	Pb	0.01
Acidity as Free H_2SO_4		1.49
K_2O		52.30
K_2SO_4		96.76

Typical Analysis Sulfate of Potash

Produced at Dumas, Texas

Constituent		Percent
Volatile at 130°C		0.02
Water Insoluble		1.02
Silicon Dioxide	SiO_2	0.23
Hydrous Oxides	R_2O_3	0.26
Calicum	Ca	0.03
Magnesium	Mg	0.12
Sodium	Na	1.16
Potassium	K	43.00
Chloride	Cl	1.80
Sulfate	SO_4	53.37
Heavy Metals as Lead	Pb	0.01
Acidity as Free H_2SO_4		trace
K_2O		51.80
K_2SO_4		95.83

Typical Analysis Coarse Granular Muriate of Potash

Constituent		Percent
Volatile at 130°C		0.10
Volatile at 250°C		0.14
Water Insoluble		0.97
Silicon Dioxide	SiO_2	0.33
Hydrous Oxides	R_2O_3	0.44
Calicum	Ca	0.05
Magnesium	Mg	0.15
Sodium	Na	0.85
Potassium	K	50.39
Chloride	Cl	47.41
Bromine	Br	0.05
Sulifate	SO_4	0.18
Heavy Metals as Lead	Pb	0.01
K_2O		60.70
KCl		96.09

Typical Analysis Soluble Muriate of Potash

Constituent		Percent
Volatile at 130°C		0.02
Water Insoluble		0.01
Silica	SiO_2	0.005
Hydrous Oxides	R_2O_3	0.002
Calcium	Ca	0.002
Magnesium	Mg	0.0003
Sodium	Na	0.40
Potassium	K	51.89
Chloride	Cl	47.66
Sulfate	SO_4	0.007
Bromine	Br	0.025
Heavy Metals as Lead	Pb	0.0001
K_2O		62.50
KCl		98.94

Dr. Kapusta: Thank you very much, Dean.

Our next speaker is Van Rogers of Southwest Potash Company and comes to us from Des Moines. He doesn't sound like he belongs in Des Moines, he sounds more like he belongs back home in the south somewhere. Van, it is a pleasure for me to introduce you to this group.

J. Van Rogers

Mr. J. Van Rogers. Jr. (Southwest Potash Company): Mr. Chairman, Members of the Fertilizer Industry; Southwest Potash is proud to paritcpate in this program of the Fertilizer Industry Round Table. Privileged, because attending this meeting are a group of individuals made up of production personnel dedicated to the ultimate objective of producing the best agricultural fertilizers for use by the ultimate consumer, Mr. Farmer, so that he in turn may produce an abundance of fiber, food and meat for the world's population.

As a supplier of muriate of potash we in turn are charged with the responsibility of supplying the fertilizer industry of this nation with top, high grade muriate of potash.

This is a working group, and as a working group reminds me of the young paratroop lieutenant who was returned to this country during World War II and upon return the medical officers deemed that he had battle fatigue. He was, therefore, assigned to do administrative duties here in the Pentagon Building in Washington.

Southwest Potash Corporation produces at this time three types of muriate of potash: uniform standard muriate, coarse standard muriate, granular muriate. A typical analysis of our uniform standard muriate is 60.08 per cent. The range screen, typical screen analysis +28 to +100 using the Tyler screen.

In our coarse standard muriate our typical analysis is 61.02 per cent, our typical screen analysis ranges from +10 to +28 mesh.

In our granular muriate our typical analysis is 60.05 per cent with a screen range of +8 to +20 mesh.

Most of these products, as is the case of all the potash producers, were developed, as Mr. Gidney pointed out, as a result of many different factors entering into the fertilizer industry in the manufacturing of mixed goods. The potash producers have attempted to produce the type and quality of material which has been demanded by the industry and most of you over the years have written the type of material which we now produce, because it was through you that most of us learned what you wanted.

There have been great variations in the demand or the requests for different types of particle sizes and this again is due to the different types of processes used by you in the manufacture of mixed goods. In addition to that, it is quite common for a producer on a national basis to take the pieces of the information which has been developed and from them develop a particle range size, a product with a typical analysis which can be utilized throughout the whole country. This may not in individual phases fit your particular operation exactly but I daresay that overall the products which are produced today and shipped to you are very nearly the same type product that you desire.

It has been brought out in the panels preceding this that many of you would like to have information in advance as to what you are going to receive. In the case of Southwest Potash Corporation, each time a car is dispatched from Carlsbad a shipping memorandum is sent to you immediately containing the analysis of the car, the water content, the car number and the weight. If there is additional information which is required, I am sure that it will be brought out in the discussion to come. Thank you very much for your attention.

Dr. Kapusta: Thank you very much, Van.

Edwin C. Kapuska

Gentlemen, I want to tell you about some real potash products now. Here they are (exhibiting). We are the U. S. Borax and Chemical Corporation; we were the former U. S. Potash Company, the first producer in the Carlsbad basin.

You have heard some mention this morning of types of processes for producing potassium chloride. We happen to produce potassium chloride by a crystallization process, that is, the major bulk of our products are produced by crystallization. We also produce a product by a combination tabling and floating operation.

I do not know how familiar many of you are with potash, with how it is made. I will go through it very briefly. The potash ore as found in Carlsbad is essentially a mixture of potassium chloride and sodium chloride. The trick, of course, is to separate the two. There are two ways in which it can be done, two ways commonly used. The first is dependent on differences in solubility of potassium chloride and sodium chloride. This permits you to dissolve and re-cryctallize potassium chloride from a solution of sodium and potassium chloride.

The second method is dependent upon the differences in specific gravity between sodium chloride and potassium chloride. This is the principle used in flotation.

Crystallization gives you a white potash, pure potassium chloride is white. The color that you see in the red potash is impurities which have remained in the product simply because the method of extraction cannot remove them.

The U. S. Borax and Chemical Corporation markets three major types of agricultural muriate of potash. We also market a technical grade potassium chloride for industrial use. I would like very briefly to go through some slides now and discuss each of these individual products with you, if I may, please.

(Slide.) Our first product is Higrade muriate of potash, it is a so-called standard muriate of potash. Please note the analysis: 62 to 63 per cent K_2O. This product is produced by crystallization. It is a white crystalline product of uniform analysis. It is treated with an anti-caking agent to reduce caking and to assure free flow. You see the typical chemical analysis below it: KCl 98.5 per cent, on a K_2O equivalent that is roughly 62.3 per cent, principal impurities, sodium chloride, moisture content, and notice this. Actually, that is moisture in volatile at 150 degrees centigrade, .1 per cent. Below that, particle size analysis. You see that essentially it is a minus 35 plus 150 mesh product. Incidentally, do not bother to copy these numbers down, I have at the side table a quantity of brochures giving all of this data and in fact giving more detailed information. The bulk density, loose pack, 67 pounds per cubic foot, tightly packed, 72 pounds per cubic foot. Angle of repose 33 degrees.

U. S. Potash Slides

Higrade Muriate of Potash

62/63% K_2O

Standard

Description

Higrade Muriate of Potash is a white crystalline product having the highest purity and uniformity available for agricultural purposes. It is specially conditioned to remain free-flowing and resist caking—for trouble-free storage and handling.

Typical Chemical Analysis

Potassium Oxide Equiv.	(K_2O)	62.30%	Potassium Bromide	(KBr)	0.06
Potassium Chloride	(KCl)	98.52	Magnesium Sulfate	($MgSO_4$)	0.01
Sodium Chloride	(NaCl)	1.19	Moisture and Volatile	(150°C)	0.12
Potassium Sulfate	(K_2SO_4)	0.07	Other compounds		0.03

Typical Particle Size Analysis

Tyler Series	Cumulative Per Cent Retained	Tyler Series	Cumulative Per Cent Retained
28 Mesh	0	100 Mesh	84
35	2	150	93
48	19	—150	7
65	60		

Average Bulk Density (pounds per cubic foot) Loose pack: 67 Tight pack: 72

Higrade Muriate of Potash is a preferred source of potash in the manufacture of conventional mixed fertilizers and many types of granulated fertilizers. High purity and excellent physical properties make it an ideal source of potash for the production of moist fertilizer mixtures.

Our second product listed here is our Higrade Granular muriate of potash. It is essentially in the so-called coarse classification. Again, please note the analysis: 62, 63 per cent K_2O. It is a white crystalline material, very uniform analysis again, conditioned to prevent caking. Chemical analysis: K_2O average 62.4, moisture 0.2 per cent. Particle size analysis is listed below. Essentially this product is a minus 8 plus 35 mesh, or if you define it as 90 per cent through a given range of screen sizes, it would be a minus 8 plus 28 mesh.

Higrade Granular Muriate of Potash

62/63% K_2O

Coarse

Description

Higrade Granular (HIGRAN) Muriate of Potash is made in a special recrystallization process to produce coarse, white crystals of high purity and uniformity—both in chemicals and particle size analysis. It offers excellent free-flowing storage and handling characteristics.

Typical Chemical Analysis

Potassium Oxide Equiv.	(K_2O)	62.34%	Magnesium Sulfate	($MgSO_4$)	0.03
Potassium Chloride	(KCl)	98.67	Calcium Sulfate	($CaSO_4$)	0.02
Sodium Chloride	(NaCl)	0.94	Moisture and Volatile	(150°C)	0.28
Potassium Bromide	(KBr)	0.03	Other compounds		0.03

Typical Particle Size Analysis

Tyler Series	Cumulative Per Cent Retained	Tyler Series	Cumulative Per Cent Retained
8 Mesh	0	28 Mesh	93
10	13	35	98
14	46	—35	2
20	78		

Average Bulk Density (pounds per cubic foot) Loose pack: 58 Tight pack: 76

HIGRAN'S larger particle size makes it ideally suited for manufacturing practically all types of mixed fertilizers, incuding granulated mixtures of high potash content. It may be mixed with suitably sized nitrogen and phosphate materials to form stable mixtures of uniform analysis.

That is, incidentally, one thing I think we should clarify and that is definition of sizes. You have to put limits on that; you cannot say generally speaking a product is a minus 8 plus 28, because if you generalize in that way, I doubt very much that you are going to find anything that is 100 per cent within those limits. So you do have to define limits when you are talking about products.

This, we would call a minus 8 plus 28 mesh product. Bulk density: loose pack 68, tight pacfl 76. Angle of repose, about 30 degrees.

Granular Muriate of Potash

59/61% K_2O

Granular

Description

Granular Muriate of Potash is a reddish-pink crystalline product made by a combination tabling and flotation process: Larger particle size and dust-free condition assures free-flowing and non-caking properties during handling and storing.

Typical Chemical Analysis

Potassium Oxide Equiv.	(K_2O)	60.19%	Potassium Bromide	(KBr)	0.08
Potassium Chloride	(KCl)	95.08	Calcium Sulfate	($CaSO_4$)	0.08
Sodium Chloride	(NaCl)	3.48	Moisture and Volatile	(150°C)	0.20
Potassium Sulfate	(K_2SO_4)	0.17	Other compounds		0.82
Magnesium Sulfate	($MgSO_4$)	0.09			

Typical Particle Size Analysis

Tyler Series	Cumulative Per Cent Retained	Tyler Series	Cumulative Per Cent Retained
6 Mesh	0	20 Mesh	93
8	9	28	97
10	44	35	99
14	78	—35	1

Average Bulk Density (pounds per cubic foot) Loose pack · 67 Tight pack: 75

Granular Muriate of Potash is an ideal source of potash for direct application purposes . . . where potash alone is required for specific crop or soil conditions. It also is widely used in mixed fertilizers where a larger size potash particle is preferred.

(Slide) This is our third major agricultural muriate, our granular 60 per cent K_2O. The two previous products are made by crystallization, this product is made by combination tabling flotation operation. Note that whereas the two previous products were white, this is a red to pink crystalline material material, it is non-caking by virtue of some of the agents used in the flotation and tabling operations.

Chemical analysis—please note the difference again. This is essentially a 95 per cent KCl or 60.2 per cent K_2O product whereas the others were above 62 per cent K_2O. Moisture again here very low 0.2 per cent. Particle size analysis. Essentially the product is a minus 6 plus 20 mesh material by our definition. Bulk density: loose pack 67, tight pack 75 pounds per cubic foot. Angle of repose 31 degrees.

Those are all the slides.

I would like to now very briefly cover the major areas of use of these materials as we view them. The standard agricultural muriate, the finely divided product, we feel, is best suited for use in conventional mixed fertilizers, it is very useful indeed in the manufacture of granulated high nitrogen grades, some granular intermediate nitrogen potash grades. Again this would depend specifically upon the formulation you are using and, of course, the material is highly useful in the manufacture of liquid mixed fertilizers.

Our second product which I listed which was our coarse product, minus 8 plus 28 mesh material is most suitable for use in granulation of the low nitrogen-high potash grades. I might remark that the reason behind the manufacture of this product was the need for a material of that type for granulation of these grades. As you all know, granulation moved ahead most rapidly in the midwest. The principal grades in the midwest are the 1-4-4 ratios, which grades gave the most difficulty in granulation, consequently manufacturers desired a product other than the available standard materials for that use. We conducted a survey of a good number of manufacturers as to the type of product which they desired for that particular application and I might say that the answers we received depended upon the number of people we talked to. I think everyone had an idea, I do not think anyone was really certain as to what they did need. They knew what they wanted but they were not sure of what was needed. We attempted to help out a little bit in this regard. We did run several full plant scale tests to try to determine if we could what a proper particle size range might be for a product for that application.

The result was our Higrade Granular. The product, then, is essentially intended for use in granulation of the lower nitrogen, the more difficult to granulate mixed fertilizers. It has been used, of course, in granulation, and in other types of fertilizers as well.

Our third product, our granular 60 per cent muriate was originally intended as a direct application material. That is where its major use had been prior to the advent of granulation. With granulation, however, people began to use it in granulating, again, these low nitrogen-high potash grades. It is used in that area, still its principal application is as a Direct Application material. It is also finding use, of course, in the manufacture of physical mixes or in the manufacture of semi-granular types of mixed fertilizers.

Those are the major areas as we see them. We would appreciate any comments that you might have in that regard.

It is needless to say, it is our desire to provide you with the types of products that you want or need for your operations. We do welcome this opportunity to tell you about the kinds of products we have and there is one very important point I have overlooked.

On our coarse and granular materials we run a screen analysis on every shipment. The analyses which were presented to you in the form of these slides are averages of over a year's production and I might say that they are not — we have tossed out—of course, you are going to get one or two rotten apples where they deviate way away, but these are very good figures. We think they are very reliable figures. The important thing is that we do run screen analysis of every car. If you desire that information about any particular shipment, we would be delighted to send it to you and, naturally, of course, the chemical analysis is also run on every shipment. We feel we have to do that to be able to provide you with materials of top quality on a very consistent basis.

As far as our standard material goes, we do not run a screen analysis on every shipment; we do not feel it is necessary. The process has been perfected, it has been in operation for many years and I think, although we do check it, I think monthly, at the present time, and it does not deviate from either the screen or the chemical analysis which we have listed here.

I again thank you for this opportunity of talking to you and we would be happy to entertain any questions you might have.

Discussions:

A MEMBER: Most of the figures that you have given are closely in line with others. One of the fellows back here has a problem, though, that maybe somebody could help him solve. Several of you here have given the angle of repose as being below 30 degrees.

DR. KAPUSTA: Yes.

SAME MEMBERS One of the fellows back here has the problem where he can't seem to get his under 45 degrees, because it's on the roof—

DR. KAPUSTA: Say it again. I missed part of that, because it's on the —

SAME MEMBER: It's on the roof.

DR. KAPUSTA: It's on the roof?

SAME MEMBER: On the roof, yes. What techniques have any of the potash companies developed to classify potash into the various grades for the granulator?

DR. KAPUSTA: Actually, Doug, being more of an expert in the handling of the materials from the production standpoint, we do have a warehousing problem. I do not know the design of the warehouses, so I cannot really answer your question. I would prefer to refer this to Doug, if I might. Doug, do you understand the question?

MR. BOURNE: Do you mean that there is some release of finer particles while you are granulating?

SAME MEMBER: Yes, What has been done with regard to the ammoniator or what-have-you? Have you developed a technique for the liquid phase to help to operate with a particular grade of potash?

DR. KAPUSTA: I'm sorry. I misunderstood the question. As far as what have we done to maximize the use of standard potash mixed fertilizers; is that it?

To answer your question, what we have done, we have worked with manufacturers in an attempt to find out what the operating characteristics are when various types of fertilizers are used and I would rather, Doug—if you speak about the ammoniator, I do not believe that you have any excessive dust losses in the ammoniator. You may be referring, of course, to chemical reactions between sulfuric acid and potassium chloride producing hydrochloric acid combined with ammonia to make ammonium chloride.

We have attempted, in this particular field, to get the best information possible. We do not, I am afraid, conduct any pilot plant experiments on our own to try to reduce, or gather information for that purpose.

I do not think that answers your question, but we have worked with manufacturers in an attempt by means of changing formulations, by means of materials handling changes to permit them to use various of these materials in specific grades.

MR. ALBERT E. HENDERSON (Wilson & Toomer Fertilizer Co.): I think the problem, and it is a serious problem, is he trying to get the angle of repose back in the building and not on top of the building, using fine potash on a granular grade of, say, an 0-14-14. The first two or three hours there is not much trouble because you start out relatively cold and you do not have a lot of fine particle potash and it granulates real well.

DR. KAPUSTA: Are you using coarse potash or are you using standard now?

MR. HENDERSON: Standard.

DR. KAPUSTA: In 0-14-14. What are we formulating with, sulfuric acid and phosphate rock?

MR. HENDERSON: No.

DR. KAPUSTA: Are you just using moisture to wet down these materials, steam, hot water?

MR. HENDERSON: Hot water.

DR. KAPUSTA: Right.

MR. HENDERSON: After you have run, say, three or four hours, you begin to get a considerable amount of real fine potash in your ammoniator, a sort of a recycle, and dust from the cyclones, and it is very difficult to granulate. I have been told that you just cannot granulate potash.

DR. KAPUSTA: You can granulate straight potash, but I am afraid —that is in the type of system you are talking about, but I am afraid you are going to find exactly what you have found out, the stuff does not hold up after it has been dried. I think that is the problem you are running into, if you use water as a wetting agent. Of course, what is granulation? It is the sticking together of these particles through the interface and then crystallization. You do not effect a very strong bond between the potash particle and the superphosphate particle. I think the most successful attempts at granulating no nitrogen grades, using standard materials, has been through the use of some sulfuric acid and phosphate rock to make use of the heat of reaction and the plasticity which results from that. I think the people at TVA have conducted several experiments on that now and I might say I would agree with you, it is exceptionally difficult to do it the way you are doing it, because the crystals break down after a while and you do get dust.

Let me say this, I do not think all the dust you are getting is potash; I think that you are also geting some phosphate dust too.

MR. HENDERSON: Ed, I'm trying to offer a solution.

DR. KAPUSTA: Okay, have you got one? I'll listen.

MR. HENDERSON: If you get the mixture real wet in the ammoniator, it will granulate, provided you do not use normal standards. If you get it wet enough, it is simply mud and I presume that you form a lot of oversize in the dryer and as it comes back to your crushing equipment, you get enough seeds to begin to granulate again, so you do not really have a problem if you get it wet enough and run long enough.

DR. KAPUSTA: We thank you for the solution, Al, but practically speaking, some of the people—I see Al Phillips sitting there, and I know Al has done work on granulation of no nitrogen grades and I do believe that they have successfully done that in the TVA ammoniator and again through the use of sulfuric acid in conjunction with some phosphate rock; is that right, Al?

Mr. Phillips: That's right.

Dr. Kapusta: I believe several people are following that method rather than the old method of attempting to wet it down. Al may have a new and novel way of reducing or eliminating the need for sulfuric acid in this particular operation.

A Member: Ed, I apologize for my first question. I only wanted to get some sort of discussion.

Dr. Kapusta: You had me confused. I must say that I do not think I was completely awake and I did not quite get your point.

Same Member: I only wanted to get some discussion going on some of the products people use. If Al will stand up and tell us something about what TVA did, I am sure people will appreciate what they have done on use of acid in phosphate rock. Without figures, Al, just roughly what happens?

Dr. Kapusta: Al, we would be delighted to have you cover that, if you would, please.

Mr. A. B. Phillips (Tennessee Valley Authority): Thank you, I would be glad to try.

As Ed has pretty much summed up, in these no nitrogen grades in which, as it has been pointed out, it is difficult to granulate the potash that if they would use some rock and acid along with superphosphate and potash that we could get more heat and more plasticity and that would allow us to make stronger granules and better granules than we can make with granulating with steam and water alone.

We have some information in TVA on formulations that work best and we would be glad to send you some information on the work that we have done on this. We have gone from 0-14-14 on up to 0-26-26, if I remember right, by using various acids and various phosphates in this operation.

Dr. Kapusta: Are there any questions? There is one behind you, Al.

A Member: As this phosphate rock passes through the TVA Ammoniator, what —

Dr. Kapusta: Would you address your remarks into a microphone, please? I am afraid that otherwise many of these people cannot hear what you are saying, sir.

Same Member: This is just a short question. What percentage of the phosphate rock is converted to available P_2O_5?

Dr. Kapusta: Al, what percentage of the phosphate rock is converted to available P_2O_5?

Mr. Phillip: You can get very good conversion just like you do in superphosphate operations if you are careful to proportion the rock and acid properly, as you do when you make superphosphate, and if you have the moisture content right, we found it was quite important to use some steam also in these variants in order to get good conversion.

Dr. Kapusta: That is related to the need for moisture, is it not?

Oh, I see, you would have excess water anyway.

Are there any further comments on that?

Mr. Jack Frederick (Sohio Chemical Company): I was wondering if they had tried taking superphosphate that was fresh rather than one or two days old?

Dr. Kapusta: I really do not know. I imagine you would have some materials handling problems. Al Henderson.

Mr. Henderson: We tried using fresh super, as a matter of fact, we tried using some several hours old and it does granulate very well, but, as you mention you have a real serious material handling problem; in fact, it was so serious that we quit.

(Laughter.)

Dr. Kapusta: Do we have any other questions from the audience? How about some questions for these gentlemen seated around this round table? Are there any questions you would care to direct to them?

Moderator Spillman: I have a question. We have had a good many people ask us to convey to the potash companies whether or not the various processes of refining the potash have any effect on granulation processes. Some of the people say to me that certain types of potash will not granulate and other types will, and we were wondering if the various refining processes in the potash mines have any effect on this particular operation?

Dr. Kapusta: My opinion would be that it is based somewhat on actual test work where we compared the granulation characteristics of the 5-20-20 using as a source of potash the flotation potash and comparing that to the granulation characteristics using our high grade value which is a re-crystallized potash and essentially these materials, the two potash sources were about the same size; pretty close, in fact, and there was no real difference that could be noted in the granulation tendency of that mixture under those conditions. It would be my opinion that there would be no real significant differences. Others may care to comment on that.

I will tell you one thing, color did not have much to do with it.

I do not know whether that answers it, Al, but these were plant tests and that was one of the things that we did look into, and that is our conclusion.

Dick Tayloe.

Mr. Richard D. Tayloe (Smith-Douglas Co.): Ed, has there been any work done on granulation using a chemical grade of potash rather than one with a caking agent in it?

Dr. Kapusta: The answer again, I do not know what experience others have had, but I speak from our experience. We had a series of tests comparing the use of our technical grade with the use of our standard high grade agricultural muriate which is treated for non-caking, and the technical grade is not, however, in this particular test to insure arrival of the material in some sort of condition, we did put in some kaolonite. You have then really a comparison of two conditioning agents, kaolonite being one and the other being the organic. We found in that test again—we did a lot of work with 5-20-20, and in those particular tests there was no noticeable difference whatsoever.

Mr. Tayloe: What I was getting at there is that the flotation reagent which sticks on the potash interferes with the solubility of the potash and it might be that that is the thing that is interfering with the binding of the small particles.

Dr. Kapusta: I do not believe it is that significant, Dick; yes, that was the reason for running the tests, to find out whether or not the agent that was being used was adversely affecting the granulation characteristics of that product. Again, our conclusion was if it was we sure did not see it and the plant did not operate long on either one.

Are there any other questions? Al? We're conducting a wonderful dialogue here.

Mr. Henderson: Ed, to answer that question about no caking, anti-caking agents to potash, we have used various types of foreign potash which is much finer than domestic standard. As a matter of fact, when we first started doing this, everybody told us, "you simply can't granulate with potash that fine." When you use a foreign grade of potash with no anti-caking agent, although it is very fine, it is no problem to granulate with it. But in the last year and a half the foreigners have begun to add anti-caking agents and some of them really went overboard, like they put two to three times as much anti-caking agent in the potash as domestic people do, and that stuff is impossible. You cannot granulate it, even on 12-12-12 or 10-10-10, with the foreign granular or coarse potash you simply cannot handle it.

I think that answers the question.

Dr. Kapusta: Al, I think there is probably one point, of course, the reason they went to a caking agent, an anti-caking agent was the stuff caked like crazy.

Mr. Henderson: Oh, yes; there's no question about that.

Dr. Kapusta: Of course, I think the reason for that was probably the amount of magnesium chloride in it, which does a beautiful job in small quantities in promoting caking and it might possibly work that same way in granulation also.

I appreciate your comments on that.

Moderator Spillman: I'd like to comment on that, too. I can go along with Al on those comments, because we too use some foreign potash and definitely there are certain types of foreign potash that granulate beautifully and others you cannot do a thing with. I do not know what the reasons are for it, but that is our finding.

Dr. Kapusta: Of course, when you start to run granulation tests, I think one of the most important variables is particle size and you do have to compare materials of the same size or as close as you can come in size. That may be a more important factor, and I think it is, than the matter of an agent or not having an agent on the material.

Are there any other questions, please?

(No response.)

Thank you very much, gentlemen. Again, we appreciate the opportunity of appearing on your program and discussing these matters with you.

(Applause.)

Moderator Spillman: Thank you very much, Ed., to you and your panelists for thoroughly discussing the potash phase of our subject on standardization of raw materials.

Joe, our subject on standardization is through, and I will turn the meeting over to you.

Moderator Reynolds: Thank you, Al, and thank you, Potash Panel.

I think, of necessity, we need to go through with our business meeting. We are running a little bit behind but we will, as Vince has summed it up in the past, make it very informal and short.

BUSINESS MEETING

Moderator Reynoldss I think probably the first item of business is the Treasurer's Report, and the Secretary's Report, so I will turn the meeting over to Dr. Marshall.

Dr. H. L. Marshall (Secretary-Treasurer of the Fertilizer Industry Round Table): Members of the Round Table: Your Secretary-Treasurer's office, functioning for the year 1959 to 1960 has handled about 900 or better proceedings mailed out; it has also mailed a few 1958 proceedings, and it has prepared and distributed 3 mailing notices, 1 in January, another one in July and another one in October.

There had previously been some criticism about not enough notices, and we felt—your committee feels that these three notices would probably be sufficient.

As of ten o'clock this morning the total registration for this meeting was 425. If some lucky person here will step forward later and see me he will be rewarded.

Your Secretary, too, implemented the directive of last year has sent to the Agricultural Experiment Station libraries and, in the same institution, either to the Agronomy Department or the Ag Engineering Department copies of the 1958 and '59 proceedings to be deposited in their libraries. The response back from that has been gratifying and they are all very happy to have them and it is our hope that they will be used in some of the course work they do in these universities to orient it and get it standardized. If it is the pleasure of the group, we will continue this next year.

Your Secretary also must have tentative orders for any large number of proceedings of the 1960 Round Table, please, by January 1st, when we will start to print the proceedings.

We are at the present time storing quite a few of these things, rent free, but we do need to limit that amount of storage that goes into a person's basement. I will admit that we cannot give you a price, but we can give you a range of about $3.50 to $3.75. That is the best we can do at this point, because we do not know how many pages we have, the pictures and what this lady is going to charge us, and all that stuff, so that all goes into that.

Your Treasurer's Report is as follows: Starting with the date October 31, 1959 for audit purposes, because of the bank statements, I had to close on September 30, 1960 and take the indicated balances up to October 30, 1960. Last year on October 31, 1959, we showed a balance of $453.16.

The 1959 Registration was $3172.50. The 1959 Membership Lists were $216.00. The 1959 proceeding sales, and that includes some '58s, $841.10. The total cash handled from October 31, 1959 to 1960 was $4,682.76.

Your disbursements: 1959 incidental costs of meeting $93.70, 1959 Membership List $201.40. We made $14.53 on that deal. 1959 to '60 secretarial services $190.00. 1959 proceedings cost $2,846.51. 1959 to '60 secretarial office operation costs, that is mail, notices, addressograph plates and we had to buy a file, $227.86. The total disbursements through September 30 were $3,559.24.

Recapitulating, the income $4,682.76, the disbursements through September were $3,559.54, a cash balance of September 30, 1960 was $1,123.22, but during the month of October we had checks outstanding of $200.00, we deposited $26.50, which left to be deducted from the September 30 balance $173.50, which gives the treasury a cash balance of $949.72 as of Hallowe'en.

(Applause.)

Moderator Reynolds: Thank you very much, Doc. Doc Marshall does a really tremendous job on this Secretary-Treasurer's report and we are all indebted to him. I can assure you that it is a twelve months job with him, seven days a week. You are liable to get a telephone call at ten o'clock at night or seven o'clock in the morning, or just whenever he has something on his mind. We are really indebted to him for his fine efforts.

I guess also, from a formality standpoint, we should have an auditor's verification of all these facts and figures. Joseph Bosman, Frank Nielsson.

Mr. Joseph Bosman, (Auditing Committee): Mr. Chairman, I had the books, went over them and as far as I could see they seemed to be in good shape and so I put my signature on it and turned it over to Frank Nielsson and I think that he found the same thing and has his signature on the books.

Moderator Reynolds: That is correct. We have received them back with signatures so they must be in order. Thank you very much for helping us on that.

As far as other business, the Meeting Dates for 1961, so far as hotel arrangements are the 8th, 9th and 10th of November. You will notice that is one week later than this year. Hotel arrangements and accommodations are closely scheduled. In past years, we have gone back and forth between these two weeks. Does anyone have any objection or any conflicts that we are not aware of which will not permit us to hold those dates?

(No objection registered.)

Do I hear a motion from the floor concerning this?

Mr. Wayne King (The W. S. Tyler Company): I rise to a point of order. I move that we accept these dates, but I think someone's got their decimal point in the wrong place.

Moderator Reynolds: I have a Mr. Westinghouse calendar.

Mr. King: I move we accept the dates as outlined.

Moderator Reynolds: It is a Wednesday, Thursday and Friday. We have a calendar conflict here. I hear the motion; is there a second?

(The motion was seconded.)

Moderator Reynolds: The motion is made and seconded. All those in favor of the dates as mentioned, please indicate by the sound of "aye."

(A chorus of "ayes.")

Opposed.

(No response.)

The dates will stand, then.

Doc has just mentioned that the desk is being closed as far as official business. There are some notices and papers out there and those persons who might have missed please go by and pick those up.

As for any other comments, it is always our pleasure and privilege to have visitors with us from across the oceans. This year we have two persons here from Australia, one person from England. Are those persons still here, I believe the two people from Australia are with us, but I do not know whether they are in the meeting at this time or not. Would you please stand so that we may recognize you.

(Those visitors present arose, applause.)

We certainly extend you a welcome and hope you can come back next year.

Someone mentioned that we had quite a large group from our friends and neighbors in Canada. As my consideration, they're home folks, and we are always glad to have them and everyone else who can make the meetings.

Another item here, just for information. I believe that the Round Table will agree with this. We sent a cablegram to Dr. Sauchelli in far away India giving him a little report on the meeting in appreciation for his guidance in past meetings and regretted that he was not able to make this one.

(Applause.)

We are, of course, indebted to the press, the trade association and trade journals for their coverage of the meeting, we are always glad to have them and we try to work with them very closely and they with us. We also appreciate Miss Withers who has become part of our meeting, she has covered the last three or four sessions and she does a real fine job and we are indeed appreciative of her efforts.

One other comment here, as far as clarification, the papers and discussions, of course, are the property of the Round Table. There has been a question asked as far as circulation of these papers, or turning them over to publications, and we do reserve the right through your Round Table to control those at least until we get our proceedings out. Many of the papers will be reported upon in the trade journals, which is fine, but as far as the official discussion paper that is the property of your Round Table.

This is supposed to be a business meeting, so I would ask at this time: Is there any old business which should come before the meeting? Mr. Walton.

Mr. George Walton (Tennessee Corporation): I know that modesty forbids you from paying tribute to the people who are responsible for this, but I think we should all express our appreciation to those gentlemen on the committee, yourself and Doc and Al Spillman, who have given so much time to the organization of this. I think we would be very remiss if we did not pay some tribute to you.

(Applause.)

Moderator Reynolds: Speaking for your Executive Committee, we appreciate your comments. The way that we work this, just to give you a little example, when we get into difficulty on questions, someone sends in a question, we get hold of our friends, like you people there in the audience, and you would be surprised but we get about 85 to 90 per cent acceptance from this group which highlights the point that this is your meeting, you feel a responsibility for it as do we, and it is a real pleasure to be able to work with a fine group like this, I can assure, speaking for all of our committee.

Is there any new business?

(No response.)

As far as any other comments, I know our time schedule is set, but if you will all be patient with us, we are going to go right on through. We have a panel which is coming up now and then we have other people carried over from yesterday. Ed Glocker was very kind to go along with us and he will discuss his paper at the conclusion of this next panel. I will now turn the meeting back to Al Spillman.

Moderator Spillmans First, may I express my appreciation to those with whom I had contacts this year and to all the panelists for the wonderful cooperation given us.

Last year we had an excellent discussion on pre-neutralization given by Phil Stone, Virginia-Carolina Chemical Company, Grant Marburger, Spencer Chemical Company, George Gilliam, Allied Chemical, Frank Keenen, du Pont, Arthur Hansen, American Agricultural Chemical Company, and N. K. Alfrey, W. R. Grace & Company. Our 1959 proceedings fully covered these discussions, in addition to a good many questions and answers from the floor. Any of you who have not received a copy of our 1959 proceedings, and are interested to have this information, please write to Dr. Marshall.

Our membership has requested we discuss again preneutralization this year to bring the subject up-to-date on economics, formulation, smog problems and other pertinent available data.

The panel on Preneutralization today is comprised of: G. C. Marburger, Spencer; H. Garrett, Minute Maid; R. D. Young, T.V.-A.; Norval K. Alfrey, W. R. Grace & Company and George Gilliam of Allied Chemical. Will you gentlemen come to the table at the front, please.

Preneutralization

Grant C. Marburger

PRENEUTRALIZATION equipment was installed in the fall of 1958 at the Omaha plant of Farm Fertilizers, Inc., and has been used to produce 8,000 to 10,000 tons of fertilizer during the past two seasons. This experience creates worthwhile information on the preneutralization process that will be of value to the rest of us in the mixed fertilizer industry. Tom Hoshall, Production Vice President, has agreed to having their experience presented at the Round Table and the following report endeavors to present this story.

For background, let's review the process of granulation at the Omaha plant into which the preneutralization equipment was added. Raw materials are screened into a cluster hopper and weighted into a scale hopper. This mixture is elevated and conveyed to a feeder that meters it into a TVA type ammoniator granulator, 6' in diameter x 8½' long. From the granulator the material drops into a 5' x 32' dryer and then into a second dryer, 6' x 35', which is alternately used as a cooler. Exit this vessel it is elevated into a small cooler 5' in diameter x 10' in length. A double deck product screen is used exit the cooler. The oversize from the screen falls to a chain-mill type crusher and is returned to the screen. The fines are collected in a large recycle hopper from which they are removed by a gravimetric feeder and fed into the TVA ammoniator at a controlled rate.

SCHEMATIC OF PRENEUTRALIZATION
EQUIPMENT AT FARM FERTILIZERS, INC.

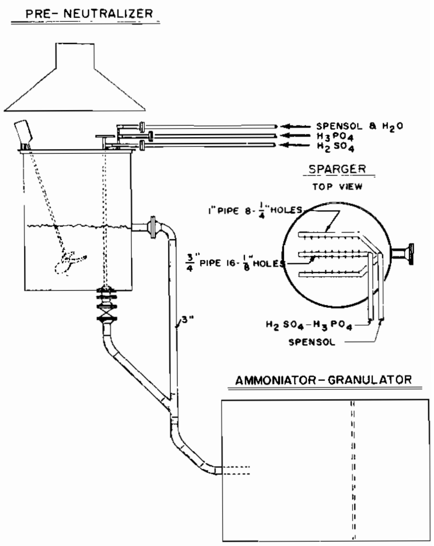


Figure 1.

Figure 1 is a sketch of the preneutralization equipment which later will be shown by photographs. The tank has a diameter of 5' and a height of 7'. It is constructed of 10 guage, #316 stainless steel. A 3" overflow outlet is located 3' from the bottom, and the preneutralized slurry flows by gravity through this 3" uninsulated line into the ammoniation section of the TVA ammoniator granulator, located one floor below the tank. The discharge into the ammoniator is made through the open end of the pipe. Better results probably would be achieved with some arrangement to give better distribution, but as yet it has not been attempted. At the bottom of the tank a second 3" outlet joins the 3" overflow pipe and is used at the end of the run to empty the tank. A 3" diaphram valve controls the rate of flow from this line and a plug is used to open or close this outlet. The valve originally installed left a gap between the bottom of the tank and the seat of the valve just long enough to fill with slurry and solidify during operation, and this salting out made the valve useless. The plug screws into a couple welded into the tank at the outlet, and has a handle extending to the top of the tank. A safety overflow is located 4' from the tank bottom. It's purpose is to prevent spilling over the top of the tank should the regular overflow become plugged.

All 3" lines were originally installed from carbon steel because of the cost difference between carbon steel and stainless steel in the 3" size range. It has since proven necessary to substitute stainless steel sections at the most corrosive points. Philip Shugart, Superintendent, and Mel Anderson, Assistant Supertendent, have reduced costs by making pipe out of stainless steel plate. They arc the plate and then weld it together.

In the original installations, two other 3" valves were included that permitted closing the flow into the ammoniator and directing it to a disposal tank in the event of a plant breakdown. These two valves were eliminated, primarily because of difficulty in plugging caused once again by salting out justahead of the valves, but also because the chances of their being needed seemed remote.

A hood and exhaust stack, constructed of lumber, were placed over the tank for removal of fumes and vapor. The 24" x 24" stack effectively removed fumes by natural convection without the use of a blower. A thermocouple well and a valve for sampling were also included in the tank. Temperatures are taken close to the bottom of the tank near the sparger outlets. A mixture is used as shown in the drawing, this one being a Model D3 Lightning, 1 HP.

The flow into the tank is made through supply lines that enter at the top and connect to the sparger located on the bottom. One line is for phosphoric acid, one for sulphuric acid, and the third carries a combination of SPENSOL and water. The sparger arrangement is a series of drilled pipes as shown in the drawing. A number of sparger arrangements have been tried, but this is the one presently in use.

Before preneutralization equipment was added to the plant, flow meters existed for all raw materials. With the addition of the tank, a second flow meter for SPENSOL was added permitting the use of ammoniating solutions simultaneously in the tank and in the ammoniator. In addition, all other existing flow meters were valved so that the material could be formulated into either vessel.

During the original plant startup some grades were produced, and some raw material used, that caused considerable turbulence and consequent splashing from the tank. To correct this splashing, an extension of two feet was added to the tank height as the simplest method to change the existing construction. This extension can be seen in the photographs.

From an equipment design viewpoint, one or two other equipment changes would be made if the installation were to be done over. A flat lid, closing off the tank, would be used with an opening that connected directly to the stack (eliminating the hood), and the mixer would be installed vertically in the center of the tank lid. An increase in mixer horsepower would also be made, for some grades and formulas create a highly viscous slurry. A caution should also be added, born from experience, to future installers of preneutralizers to make the slurry line as vertical as possible with no horizontal or relatively flat sections. These contribute to excessive surging in the flow of slurry to the ammoniator. Insulation placed on the slurry line also seemed to help this problem, reducing surging in the flow when dumping the preneutralizer at the end of a run.

Some method of stopping reverse flow in the acid and solution lines to the preneutralizer is needed. During a momentary shutdown, pressure was lost on the sulphuric acid tank. The acid control valve did not shut off tight enough and the backflow of acid to the sulphuric acid tank siphoned slurry up out of the preneutralizer. This of course solidified and was very difficult to get out. Check valves have been installed on the acid line at Omaha. Vacuum breakers might also be practical.

One other change that is more Spencer than Farm Fertilizers (in other words they may disagree), is to bring the SPENSOL line into the side of the tank instead of from the top. The seven feet of heat treatment this pipe received could be sufficient to flash the ammonia in the SPENSOL to a vapor, and when ammonia is released at the sparger as a vapor instead of as a liquid, it is more prone to escape and create nitrogen loss. Piping this supply line through the side of the tank instead of down from the top eliminates this possibility.

The cost of this equipment when originally installed included $1100 for the tank, $600 for the mixer, and $130 for the temperature indicator and capillary. Adding $450 to these figures for the additional flow meter brings the total for these parts to about $2300. Exact costs were not isolated for all the other items, but $800 would cover the cost of pipe, valves, and fittings for a total installed cost between $3,000 and $4,000.

Production

Four major grades are produced by Farm Fertilizers using the preneutralizer; and a considerable tonnage has been run of the grades 16-20-0, 15-15-0, 15-15-5, and 20-10-5.

The grade 16-20-0 was originally made from 11 units of SPENSOL and 5½ units of ammonium sulfate at 12 tons per hour. It is now produced using 16½ units of SPENSOL at the same rate of production with a formula costs savings of $2.50 per ton. The complete formula is shown in Figure II. Phosphoric acid has also been used in this formula, but triple works better. Figure III shows the preneutralization formula for 15-15-0 and 15-15-5 using all nitrogen from ammoniating solutions. Previously, 15-15-0 was produced with 10 units of solution and 5½ units of ammonium sulfate at 12 tons per hour at a cost disadvantage of approximately $3.50 per ton. All of these grades have been run at 15 tons per hour, but 12 tons per hour works better.

This 15-15-0 formula frequently is varied by substituting ammonium sulfate for one or two units of SPENSOL. It's hard to operate with 15 units from SPENSOL because the large quantity of normal superphosphate in the formula necessitates a lot of SPENSOL going into the TVA ammoniator instead of into the preneutralization tank.

When speaking of production rates obtainable with preneutralization, one penalty should be mentioned. A delay of approximately one hour is encountered in order to get the tank filled, so an hour of production time is lost.

The grade 20-10-5 runs well using as high as 19 units of ammoniating solution. The formula is shown in Figure V. The production rate is 10 tons an hour on this formula with recycle ratio estimated at less then 1 to 1. An over formulation of one unit of nitrogen is presently used.

Although this formula runs satisfactorily, the product doesn't store too well. Part of this may be due to the lack of sufficient cooling capacity at the plant. The granules are soft and a wet crust will form over-night on top of the pile.

As an interesting comparison of storage conditions experienced with a grade like 20-10-5, removing six units of SPENSOL from this formula and replacing three of them with anhydrous ammonia and three with ammonium sulfate reduces the pile set and improves storage conditions considerably. The moisture content of product is kept generally at ½% to 1%. Production rates are decreased to 8 tons per hour, more heat is created, and the required recycle ratio increases to approximately 1 to 1.

I don't think Phil and Tom were very anxious for me to mention this formula, and I agree that it doesn't follow the basic principles of good formulation practice. But yet it does present some interesting data on storage conditions of a high nitrogen inverse ratio grade.

Storage characteristics of this material are interesting. For any goods less than 20 units, no more trouble is experienced on mechanical condition in storage than was previously encountered before preneutralization. So for the grades 16-20-0, 15-15-0 and 15-15-5, the increase in ammonium nitrate content from the increased use of solutions has not created problems. If any hot material is bagged, trouble will be encountered. Remembering that this plant is undersized on cooling, 20-10-15 causes some trouble.

Operation Methods

Water is used as a control of temperature in the preneutralization tank. As water is added, the temperature decreases in the tank, and as it is reduced, the temperature increases. Temperature is held in the range of 275°-300°F. on the four grades produced.

Although it is possible to use water as a control of liquid phase in the granulator, this practice is not used at the Omaha plant. This is true because in the high nitrogen grades produced, the removal of moisture is difficult because very little heat can be used in the dryer. Therefore, adding water as a control becomes prohibitive. At the same time, control has to be close because granulation will swing quickly from dry to wet in this operation. Therefore, control is achieved by the alternate method of controlling fines recycle, and equipment to better accomplish this end has recently been modified at the plant.

Acidity control is also used extensively. A slurry sample is taken from the tank once or twice a day and analyzed through a pH meter. Acidity will effect the degree of granulation, amount of fumes created, and hygroscopicity in the product pile. For these and other reasons pH samples are used as an overall check of operations.

When starting a production run, water is first added to an approximate depth of 10″ which is just enough to cover the sparger pipes. SPENSOL and acid, or acids, are then turned on at full rate of feed according to the formula. When the liquid level reaches the overflow, the rest of the plant is turned on.

When the production run is ready to be shut down, the amount of preneutralized slurry in the tank is estimated in order to make the run come out even. Raw material flow to the tank is stopped, and the tank is dumped from the bottom valve and then flushed with water.

It was at first difficult to gauge the correct rate of flow when dumping the tank through the bottom valve, but with experience it has become relatively trouble free.

Contrary to early estimates, cold weather has not proven to be an operating hazard with preneutralization. The overflow line has plugged several times and will do so if the plant is shut down for about an hour. The slurry has cooled off in this length of time and the pipe has also become cold. The operators have learned, however, that the addition of water to the slurry helps prevent this trouble.

16-20-0

		Units		
	Pounds Per Ton	N	P	K
Spensol 448 (25-69-0	733	16.5		
H_2SO_4 (66°)	308			
Triple	718		16.5	
Normal	350		3.5	
	2109	16.5	20	0

FIGURE—2

15-15-0

		Units		
	Pounds Per Ton	N	P	K
Spensol 448 (25-69-0)	688	15.5		
H_2SO_4 (66°)	250			
Phosphoric Acid	176		4.9	
Normal	1017		10.1	
	2131	15.5	15.0	0

15-15-5

		Units		
	Pounds Per Ton	N	K	P
Spensol 448 (25-69-0	688	15.5		
H_2SO_4 (66°)	242			
Phosphoric Acid	272		7.5	
Normal	750		7.5	
Potash	175			5.25
	2127	15.5	15.0	5.25

FIGURE—3

20-10-5

	Pounds Per Ton	Units N	Units P	Units K
Spensol 448 (25-69-0)	844	19.0		
Sulfate of Ammonia	190	2.0		
H_2SO_4	430			
Phosphoric Acid	318		8.4	
Normal	159		1.6	
Potash	170			5.27
	2111	21.0	10.0	5.27

FIGURE—4

Moderator Spillman: Thank you, Mr. Marburger.

Our next speaker will be Mr. R. D. Young from T.V.A. giving their experience on preneutralization, Mr. Young.

Pilot-Plant Work On Preneutralization At TVA

R. D. Young

I WOULD like to thank Joe Reynolds and the Committee for this opportunity of discussing some of the pilot plant work at T.V.A.

After preneutralization was first discussed at the meeting of this group in 1957, we began to receive a large number of inquiries at TVA, both in correspondence and in discussions with visitors, concerning this new ammoniator-granulator tool. At first we were of the opinion that sufficient work was being done in actual plant-scale application that pilot-plant work on our part might not be desirable. As interest in preneutralization increased over the next year or so, it seemed advisable to do at least enought pilot-plant work to allow an independent evaluation of this phase of ammoniation-granulation work so that we could have first-hand information in reply to the numerous questions. This was the basis on which our preneutralization studies were initiated. As we got into the work, greater advantages became apparent and a wider versatility of preneutralization was realized. As a result, the greater part of our pilot-plant work on ammoniation-granulation for the past 18 months has been concerned in some manner with preneutralization. This discussion is designed to point out some of the over-all conclusions reached in this work as well as to report details of certain studies which seem to be of importance.

An over-all evaluation of this experimentation has demonstrated that very important advantages can be obtained through use of a preneutralizer. Removal of a large part of the water and dissipation of much of the heat of reaction in a preneutralizer can be very beneficial in:

1. Allowing use of more economical formulations for several conventional grades of granular fertilizer
2. Improving production and decreasing recycle requirements for special types of granular fertilizers such as the inverse ratio grades (20-10-10, etc.) and X-O-X grades (16-0-16, etc.)
3. New process applications such as the TVA process for production of granular diammonium phosphate

Description of Pilot-Plant Equipment

Most of the work has been in conjunction with our TVA ammoniator-granulator pilot plant at production rates of 0.75 to 2 tons per hour. Our earliest efforts were directed toward determining the most suitable type of prereaction equipment. First we tried premixing the acid and ammoniating fluid in a concentric pipe positioned tangentially on the wall of a tank. The idea was to obtain sufficient mixing due to a swirling and jet-like action that mechanical agitation would not be necessary. Mixing was not satisfactory and copious evolution of fumes was experienced. Later efforts were in the direction of greater dispersion of the reactants rather than bringing them together initimately. An agitator was installed in the tank and separate open-end spargers for acid and ammoniating solution were used. This gave considerable improvement, but fuming still was a problem. The most satisfactory arrangement which eventually evolved consisted of separate, circular, drilled-pipe spargers with holes directed toward the center of the tank. The ammonia sparger was positioned below the acid sparger and both were below the bottom turbine impeller. With this type of arrangement, "hot spots" or areas of intensive reaction were avoided and fuming was controlled at a satisfactory level.

The preneutralizer tank used in most of our work was 14 inches in diameter and 3-1/2 feet high. It was equipped with two turbine-type impellers on a single shaft driven by a variable speed air motor. The spargers, as mentioned previously, were the circular, drilled-pipe type of 1/4-inch pipe size. Type 316 stainless steel was used throughout for construction of the preneutralizer. The slurry produced in the preneutralizer was discharged through a side overflow pipe at a 2-foot level. Gravity flow was utilized from the preneutralizer to the ammoniator. The tank was equipped with a hood and an air ejector system for exhausting the moisture-laden off gases.

This type of preneutralizer, we believe, embodies several principles that should be important in design of commercial-scale units.

1. The equipment is comparatively simple, and use of gravity flow avoids pumping a hot, abrasive slurry.
2. Turbine-type agitators appear to be best for the viscous slurries usually encountered and for good ammonia absorption.
3. In sizing the preneutralizer tank, our work indicates a nominal retention time of 6

to 10 minutes. We have found, however, that surface area for evaporation of water and volumetric capacity for ammonia loading are of equal or perhaps greater importance. We have obtained good results with evaporation rates as high as 300 pounds of water per hour per square foot of surface. For large-scale design, not more than 200 pounds per hour per square foot would be recommended, however. Ammonia loading of 35 to 50 pounds of ammonia per cubic foot of preneutralizer liquid per hour has been satisfactory.

4. Adequate freeboard of at least 25 per cent should be provided.
5. Maximum dispersion of reactants to avoid "hot spots" is essential and can be obtained by the use of circular, drilled-pipe spargers.
6. A good exhaust system for handling the load of water vapor is essential.

Operation and Control of the Preneutralizer

At the start of our work we had heard of the necessity for close control of free acidity and other factors, so we were uncertain as to the proper guidelines for control. As the work progressed, we found that just as in the ammoniator, correct proportioning of reactants is the main concern. Proportioning became the primary control, with pH being used as a spot check. Our pH measurements were made with narrow range pH paper on samples of slurry diluted about 4 to 1 with water. Temperature in the preneutralizer was found to have a pronounced effect on loss of ammonia, on fuming, and on the amount of evaporation obtained and final moisture content of the preneutralized slurry. A most convenient means for control of temperature in the preneutralizer was through addition of water directly to the tank for evaporative cooling. The usual range of control of temperature was 250° to 275° F. After some experience, pH and temperature measurements could be used to ensure operation of the preneutralizer under the desired conditions. With proper control, loss of ammonia was low; fuming was moderate; and effluent of the lowest water content compatible with sufficient fluidity for gravity flow and good distribution could be ensured.

Distribution of Preneutralized Slurry

Because of the nature of the preneutralizer slurries, distribution beneath the bed in the ammoniator is impractical. Efficient distribution of the slurry on top of the bed is quite important, both from the standpoint of good granulation and for efficient ammoniation if further ammoniation of the slurry is desired. We have tried several types of distributors in pilot-plant work. The main points for consideration are that the distributor should be simple, free from restrictions that would lead to plugging with solids, and easily cleaned, preferably during operation.

The type of distributor that we have had the most success with is our so-called "saw-toothed" distributor. The original version of this distributor is shown in Figure 1. It is essentially a section of 1-1/2-inch stainless steel pipe closed at one end, with a serrated or saw-toothed slot along one side. The preneutralized slurry flowed by gravity into the open end of the pipe and the stream was divided into a number of individual streams that dripped onto the bed of material in the ammoniator. This distributor provided a fairly good pattern, but difficulty due to the individual streams of slurry combining in contact with the wall of the pipe was experienced. A modification of this distributor which largely overcame this problem by positioning the saw-toothed slot an inch or so outboard from the wall of the pipe is shown in Figure 2.

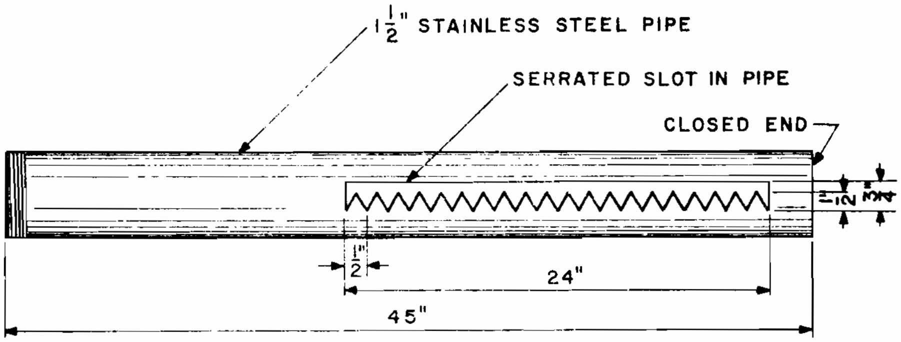


Figure 1.

Distributor for Feeding Nitric Phosphate Extraction Slurry in Pilot-Plant Ammoniator

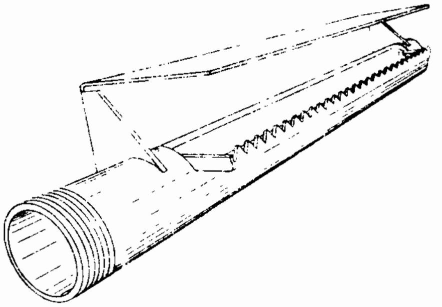


Figure 2.

Modified "Saw-Tooth" Distributer

Another type of distributor which showed promise used compressed air impinging on a stream of slurry flowing from an open-end pipe. For some slurries the cooling effect of the air was too great and caused too rapid crystallization of the material with resultant lumping. A pipe distributor with a series of holes along the bottom, of the type reported to give satisfactory service in plant-scale units, did not work well in pilot plant because of plugging of the small holes that were required.

Feed Materials Used and Grades Tested

All of the various acids—sulfuric, wet-process and electric-furnace phosphoric, and nitric—were used in our test work. These were preneutralized with nitrogen solution and/or anhydrous ammonia. Gaseous as well as liquid ammonia was tested. A large number of grades of fertilizer were produced in the various applications. These included:

1. Conventional grades - 13-39-0, 16-20-0
2. Inverse ratio grades - 16-8-8, 20-10-10, 30-10-0
3. X-O-X grades - 16-0-16, 20-0-20
4. Diammonium phosphate grades - 21-53-0, 18-46-0, 16-48-0, 14-35-14, 17-17-17

Conventional Grades: The main incentive for use of a preneutralizer in producing the convential grades is the ability to use lower cost formulations. The economy results primarily from the use of a larger proportion of nitrogen solution and ammonia. Good results in plant-scale operation have been reported for 12-12-12 grade with all nitrogen from solution. We did not test this grade in the pilot plant but did produce 13-30-0 with all 13 units of nitrogen from solution and ammonia and 16-20-0 with 14 units of nitrogen from solution. These grades were quite easily produced and granulation was controlled with a recycle ratio of about 1. Typical rata tests of 13-39-0 and 16-20-0 are shown in Table 1.

Table I. Production of Conventional Grades

Grade	13-39-0	16-20-0
Production rate, ton/hr.	2	2
Feed to preneutralizer		
Soluiton 370 (17-67-0)	9 units N	8 units N
Phosphoric acid	14 units P_2O_5	11 units P_2O_5
Conditions in preneutralizer		
NH_3:H_3PO_4 mole ratio	1.27	1.25
Slurry		
Temperature, °F.	276	277
pH	5.2	4.8
Water evaporated, lb./ton	50	50
Loss of NH_3, %	1.7	1.8
Water content of preneutralized slurry, %	10.7	10.4
Feed to ammoniator		
NH_3 (anhydrous liquid)	4 units N	—
Solution 370	—	6 units N
Ammonium sulfate	—	2 units N
Triple superphosphate	25 units P_2O_5	—
Ordinary superphosphate	—	9 units P_2O_5
Recycle ratio	1.2	1.0

Inverse Ratio Grades: Our studies of preneutralization in the so-called inverse ratio grades were primarily with 20-10-10 grade. Grades of this type are difficult to produce and require high recycle if preneutralization is not used. We produced 20-10-10 with 18 units of nitrogen from solution 370 and 2 units of nitrogen diammonium phosphate at a recycle ratio of 1. When all 20 units of nitrogen were supplied as solution 370, the recycle ratio was 1.9; a test without preneutralization indicated that a recycle ratio of about 6 would be required. Data for tests of 20-10-10 are shown in Table II.

Table II. Production of 20-10-10

Grade	20-10-10	20-10-10
Production rate, ton/hr.	1	1
Feed to preneutralizer		
Solution 370 (17-67-0)	16 units N	18 units N
Sulfuric acid (94%), lb.	461	—
Phosphoric acid	—	6.5 units P_2O_5
Water, lb.	83	50
Conditions in preneutralizer		
NH_3 fed, % of stoichiometric[a]	95	89
Slurry		
Temperature, °F.	268	285
pH	1.8	4.3
Water evaporated, lb./ton	183	161
Loss of NH_3, %	0.5	2.7
Water content of preneutralized slurry, %	5.5	9.0
Feed to ammoniator		2 units N
Solution 370	2 units N	
Diammonium phosphate	(2 units N, (5 units P_2O_5	— 3.5 units P_2O_5
Triple superphosphate	5 units P_2O_5	
Recycle ratio	1.0	1.9

[a] For ammoniation to diammonium phosphate and ammonium sulfate.

X-O-X Grades: Preneutralization of nitrogen solution with sulfuric acid has been used quite widely in the production of grades such as 14-0-14 on commercial scale. In our pilot-plant experimentation with these X-O-X fertilizers, most of the formulations were based on preneutralization of nitric acid with ammonia to produce grades of higher analysis. Grades ranging from 16-0-16 to 20-0-20 were produced with all of the nitrogen supplied as nitric acid and ammonia. Data for typical tests are shown in Table III. The nitric acid (58% HNO_3) was preneutralized virtually to completion with anhydrous liquid ammonia. The preneutralized slurry was fed to the ammoniator-granulator where it was mixed with potassium chloride and limestone filler required for the particular grade. Recycle fines were used to control granulation. A small amount of ammonia (about 3% of total) was fed to the drum to ensure removal of any trace of free acidity and to give a pH of about 7 for the finished fertilizer. The granules were particularly well rounded, very much resembling prills, and had good storage properties when a coating agent was used.

Diammonium Phosphate Grades: The new TVA process for production of granular diammonium phosphate utilizes a preneutralizer and continous ammoniator as the heart of the process. Mr. A. B. Phillips discussed development work on this process on Wednesday; therefore, I will only touch lightly on this phase

Table III. Production of X-O-X Grades

Grade	16-0-16	20-0-20
Production rate, ton/hr.	0.75	0.75
Feed to preneutralizer, lb./ton product		
Nitric acid (59% HNO_3)	1295	1617
Liquid ammonia	206	253
Input moisture content, %	35	35
NH_3:HNO_3 mole ratio	1.0	0.98
Loss of NH_3, %	2	0
Water evaporated, lb./ton	301	272
Slurry		
Temperature, °F.	258	260
pH	4.2	1.0
Water content	10.0	19.2
Fed to ammoniator, lb./ton product		
Liquid ammonia	6	6
Potassium chloride	524	652
Limestone	503	117
Recycle ratio	2.5	3.9
pH of product	7.1	6.7

of our preneutralization studies. In preneutralization phosphoric acid, the slurry is very thick at an $NH_3H_3PO_4$ mole ratio of about 1 and becomes increasingly fluid as the ratio is increased to about 1.5. Loss of ammonia increases rapidly as the ratio approaches 1.5. We used the highest ratio that could be used without exessive loss of ammonia; this ratio was in the range of 1.25 to 1.35, showing a pH of 5 to 5.5 when wet-process acid was used. If acid more concentrated than about 42 per cent P_2O_5 was used, sufficient water was added to the preneutralizer to give an effective concentration of 40 to 42 per cent P_2O_5. The temperature in the preneutralizer was about 250° F., and the effluent slurry contained 18 to 20 per cent water.

The straight diammonium grades 21-53-0 with electric-furnace acid and 18-46-0 to 19-49-0 with wet-process acid as well as complete grades such as 14-35-14 and 17-17-17 were produced. Recycle ratios ranged from 1:1 to 3:1. Without a preneutralizer the recycle requirements would have been much higher.

Conclusions

In summation, I would like to point out again that our work has shown that preneutralization can be used to advantage in several phases of ammoniation-granulation work. From one half to two thirds of the water and heat of reaction can be removed in a preneutralization step for many formulations. Economy in formulation can be realized, production of certain grades can be simplified, and production rates of high nitrogen grades can be increased due to a decrease in requirement of recycle. The reaction equipment is comparatively simple and inexpensive and control is not too difficult. We do not feel, however, that a preneutralizer is a "cure-all" or should be considered for use in all plants even though savings due to cheaper formulations are indicated. There is some additional complexity in control, another piece of equipment to operate, and another potential source of difficulty. Probably only those plants where technical help is above average and where present practice of granulation is well under control should consider use of preneutralization.

MODERATOR SPILLMAN: Thank you very much, Mr. Young.

Next, we will have Mr. Garrett from Minute Maid give us his story on preneutralization. Mr. Garrett.

Gentlemen: I have had several people ask me why a representative of Minute Maid was on a Roundtable discussion since they primarily think of us in connection with orange juice. However, we do operate enough groves that we thought our tonnage justified us going into our own production of plant foods.

Use Of A Preneutralizer

Harold Garrett

FIRST, I would like to give you a very brief background of Minute Maid Groves operations as it pertains to the Fertilizer Division. The Plant is located at Dr. Phillips, Florida, approximately ten miles Southwest of Orlando. We now operate 21,000 acres of groves throughout the State and are in the process of planting another 11,000 acres of young groves. So the operation of this plant is primarily for our own use. This is an enviable position in the Fertilizer Industry, in that we have a captive market, and since I have a voice in the recommendations, the number of analyses is held to the barest minimum, and the tons of plant food required can easily be determined. Consequently, 90% of our plant food is held to one analysis a year.

Our principal grade contains 14 units of nitrogen with varying amounts of potash and secondaries, for instance; the fall grade is a 14-0-7 with six units of magnesium. Where something different is required in the field, we generally add this by a supplementary application of straight materials.

As far as I know, the Minute Maid plant is unique in its operation, inasmuch as our entire production is based on making high analysis nitrogen, X-O-X grades, entirely from solution nitrogen through the use of a preneutralizer. The plant was originally designed to produce 20 tons an hour. With a 14% nitrogen fertilizer we have been able to produce up to 12 tons an hour but have a much smoother operation when we are on stream at 10 tons an hour. The chief limitations seem to be the capacities of the preneutralizer, dryer, cooler and hammer-mill. In this talk I purpose to confine my remarks to the preneutralizer and problems associated with its operation.

The preneutralizer is a vertical, cylindrical tank, 54 inches inside diameter, 80 inches high, with a three inch diameter overflow pipe placed 42 inches from the bottom of the tank. The tank is elevated so the product can overflow and feed by gravity into the granulator. The top of the preneutralizer was left open and stainless steel pipes carrying the acid and the solution were brought in at the top and down along the sides of the tank to horizonal spargers four inches from the bottom. The nitrogen solution sparger was U shaped consisting of two parallel pipes 12 inches apart with holes directing the solution toward the center. The acid sparger was placed in the middle between the two sides to direct the acid toward the solution. A large valve was provided in the bottom of the preneutralizer to drain the charge in case of unscheduled shut-downs. Fumes were removed by a stack approximately 20 inches square which was hung over the middle of the tank and vented through the roof of the building. Agitation was provided by a Lightnin mixer revolving at 1200 RPM's. This is a brief description of our preneutralizer as it was originally set up.

The agitator was originally hung over the edge of the tank. In a short time the motor was badly corroded and had to be replaced. Maintenance on the motor was reduced considerably by building a brace on the side of the tank at the top and by introducing the shaft at an angle through the side of the tank.

There has been practically no corrosion problem with the solution sparger. It is made of #316 stainless steel and the original sparger is still in operation. The acid sparger has given us considerable trouble. Black iron spargers were found to last about eight hours. Hastelloy B spargers lasted about six hours. After considerable trial and error we are now using Hastelloy C spargers which have not shown any appreciable wear after ten months continuous operation.

The solution line into the tank is made of #316 stainless steel and holds up very well. The acid line was originally #304 stainless steel which broke down in a short time. We are now using #316 stainless steel for the acid line and it is holding up satisfactorily.

The reaction between the acid and ammonia causes a considerable turbulence. We have found it necessary to bolt a lid on the top to prevent the hot material from spilling out over the top onto personnel on the floor. A large hole was left in the cover so the fumes could escape up the stack, which was hung so the end was about 12 inches above the hole in the cover. At times there was insufficient draft in the stack and fumes escaped into the building. This was corrected by lengthening the stack from 20 to 30 feet.

The most serious problem in the operation of the plant has been the excessive fuming and loss of material from the preneutralizer. Early in the operation it was extremely difficult to keep the preneutralizer from fuming. This caused a heavy, bluish smog which settled around the plant area. When the air movement was slight and the humidity high, this smog was a considerable nuisance to the surrounding community. There were many theories as to the cause and nature of the fumes and many changes were made to correct the condition.

I would like to briefly mention a few of the things we have done which have helped to reduce fuming. First of all we tried to reduce the violence of the reaction in the preneutralizer by changing from a 66° Be' to a 60° Be' acid. This more dilute acid was of some help but did not solve the problem. Various arrangements were tried with the spargers and finally we have placed the acid sparger on one side of the tank and the nitrogen sparger on the other side of the tank with the openings faced away from each other toward the wall of the tank. This has been the best arrangement of those to date.

In the early stages of the operation the acid content of the final product in the preneutralizer was controlled by analyses. With experience we have found that the acidity can be controlled by a trained operator without taking samples for analysis. We standardize the operation by using 41.4% nitrogen with 19% free ammonia, 60° Be' acid and water to hold the temperature at 275°-290° Fahrenheit. In practice we have found that the neutralizer will fume when the charge contains more than 0.5% excess acid. Thus by observing the fumes coming from the stack our operator is able to adjust the excess acid to within 0.2%-0.3%.

Fuming is now a minor problem in our operation. We are currently concerned with excessive oversize granules due to surging of solution from the neutralizer into granulator. This is due to the extreme turbulence from the reaction. Perhaps we may be able to modify the agitator to correct this condition.

We are now operating the plant with a minimum of Chemical controls during the manufacture. The operator, through experience, has the feel of the operation and by noting the condition of the product, the fumes from the preneutralizer, can make the proper adjustments to keep the plant running smoothly. The control of the finished product is through analysis in our Laboratory at Plymouth, Florida, and adjustments are made on our feed systems to take care of any inaccuracies.

Basically, I can sum up this discussion in this manner, as the personnel in the plant operation became better trained and reached the point where they were able to recognize the primary underlying reasons for the troubles, the big problems of yesterday became minor incidents of today.

Thank you.

(Applause.)

MODERATOR SPILLMAN: Thank you very much.

Our next speaker will be George Gilliam of Allied Chemical.

Preneutralization Equipment And Operation

George G. Gilliam

Thank you Al. Fellow members of the Round Table. Of course, my subject is also preneutralization, and I think from the remarks that have been previously expressed, we can look into the future and say preneutralization is going to have rather wide application in this ever changing fertilizer industry. As we originally investigated the preneutralization technique, it was mainly for the purpose of making X-O-X grades, and from there we went to, you might say, the inverse ratio grades. Somebody asked me why we call a 20-10-10 inverse ratio; that we should call that a straight ratio grade and a 4-12-12 the inverse ratio grade. When we consider standardization of nomenclature, maybe our Committee should take that under consideration.

The use of a preneutralizer or prereactor in the manufacture of mixed fertilizers was first disclosed at the Fertilizer Round Table in 1957. At this time, Mr. Floyd, of The Virginia Carolina Chemical Company, described VC's successful operation of a preneutralizer for making a granular 14-0-14 material. Again in 1959, preneutralization was included on the Fertilizer Round Table program. Discussion at this time covered such phases as design considerations, commercial experience, economics, product quality and the broad possibilities of the preneutralizer process for manufacturing high analysis materials. In view of the above it seems appropriate here that we consider equipment and some of the operating variables.

First Equipment. The basic unit is the stainless steel tank in which the acids (sulphuric, phosphoric or both) are neutralized with ammonia. This tank is normally located in a fertilizer granulation plant above and as close as possible to the fertilizer mixer. Mounted on top of the preneutralizer tank is the agitator drive motor with the shaft extending downward into the tank. Also, connected to the top of the tank is the vent stack for carrying off water vapor. Inside the tank mounted vertically along the wall and extending out through the top are the feed pipes for the two acids, the ammoniating solution and water. Horizontal pipe spargers with a single row of holes are installed on the bottom ends of both acid lines and the solution line to give better distribution of these materials in the tank. To reduce the possibility of direct contact of strong acid with solution, the solution feed line is located on the opposite side of the tank frfom the acid lines. The slurry overflow line is connected to the side of the tank about a third of the distance down from the top. The emergency overflow line is also connected to the side of the tank above the slurry overflow line. The thermocouple connection may be located in any convenient place on the side of the tank below the slurry level. Connected into the bottom of the tank is a flush bottom tank valve and drain line.

Now let us consider operations. There are several ways to start up and operate the preneutralizer unit and all are assumed to be satisfactory. The method which I will describe here has been proven in both pilot plant and commercial operation.

Start-up. First the equipment is checked over to make sure everything is in order. Then the automatic temperature controller is set at the predetermined operating temperature. Water is then fed manually into the tank until the level is two to four inches above the spargers. The top of the tank is closed and the unit is ready for operation. The agitator is turned on and the acid and solution valves opened simultaneously and gradually brought up to full rate. After the feed is started 15 to 30 minutes will be required for the preneutralizer tank to fill to the over flow line. This allows ample time for the other equipment to be put in readiness for immediate start-up as soon as the first slurry reaches the mixer. No additional water is added to the preneutralizer until after the slurry has reached the desired operating temperature, at which time the automatic temperature controller will feed water as required to control the unit at this temperature. Having reached the operating temperature in the preneutralizer tank and the slurry is overflowing smoothly to the mixer, the unit will operate continuously with minimum of attention. From time to time during operation the slurry pH or free acid content should be checked to make sure that the acid and the solution are being fed in the proper proportions.

Shutdown. The slurry in the preneutralizer will solidify if allowed to cool appreciably below operating temperatures. Therefore it is necessary to drain the slurry at the close of operation. This is accomplished by shutting off the feed to preneutralizer and hand feeding the slurry to the mixer through the bottom drain valve at approximately the same rate as it was being fed through the overflow line. When the preneutralizer tank is empty, solid feeds to the mixer are shutoff and the system closed down. As an alternate procedure, it is possible to dilute the slurry sufficiently to permit storage without solidification. However, about half the slurry must be drained to provide space for the required water, corrosion of the acid sparger is increased, and at start-up a large volume of dilute slurry must be worked off before operating temperatures are reached. Because of these reasons we would suggest dilution with water be used only when emergency conditions do not allow normal draining of the slurry into the mixer.

Operating Variables. For any preneutralizer installation there are a number of factors that can be varied which may affect the ease and efficiency of operation. These include the following:

Temperature. For preneutralization we normally consider operation with the slurry at the boiling point. Therefore, the operating temperature determines the amount of water in the slurry and this in turn will affect recycle and granulation. Formation of smoke and loss of ammonia also are determined in part by the operating temperature and may become significant as temperature is raised.

Ammoniating Solution. Technically any of the ammoniating solutions sold may be used. However, since the fixed to free nitrogen in these solutions is different the slurry compositions made from each solution will be different. Accordingly, we can expect a change in the amount of water and the viscosity of the slurry as we shift from one solution to another for a given operating temperature.

Acids. Neutralization is usually done by sulphuric, phosphoric, or a combination of both. Again slurry composition will vary as the ratio of these acids is changed and significant changes in viscosity and water content of the slurry can result.

Slurry pH. As slurry pH's are raised ammonia losses are increased, as the pH's are lowered fume formation increases. As a result the optimum pH for operation represents a compromise at which both losses and fumes are low. The best value of pH will depend primarily upon the solution and acids in the formulation and the operating temperature.

Throughput Rate. Throughput rate does not affect operation until it is increased to the point where adequate distribution of solution and acids can no longer be maintained or until the capacity of the surface for disengaging water vapor is exceeded.

In closing we can say that the use of preneutralization has now proven itself commercially for the production of high analysis materials of good storage characteristics. Future growth of this process will depend on the relative prices of solution and solid nitrogen and the increased demand for high nitrogen mixed grades.

(Applause.)

MODERATOR SPILLMAN: Thank you, George, for that very splendid information on preneutralization.

Our next speaker is Norval K. Alfrey, W. R. Grace & Company who will speak to us on preneutralization developed by the W. R. Grace Company.

Thank you Al. Members of the Round Table. This will be a description of recent improvements to the concentric type prereactor. It is a progress report since the details of the unit were described during last year's Round Table proceedings.

Norval K. Alfrey

THE concentric pipe prereactor is a simple piping arrangement which permits preneutralization of sulfuric and phosphoric acids with ammonia. It is a continuous flow sparging system which offers significant technical advantages when used in mixed fertilizer granulation. In our Davison Chemical granulation plants, its use has virtually eliminated the notorious ammonium chloride fume cloud. Also, it has reduced nitrogen losses from the ammoniator-granulator.

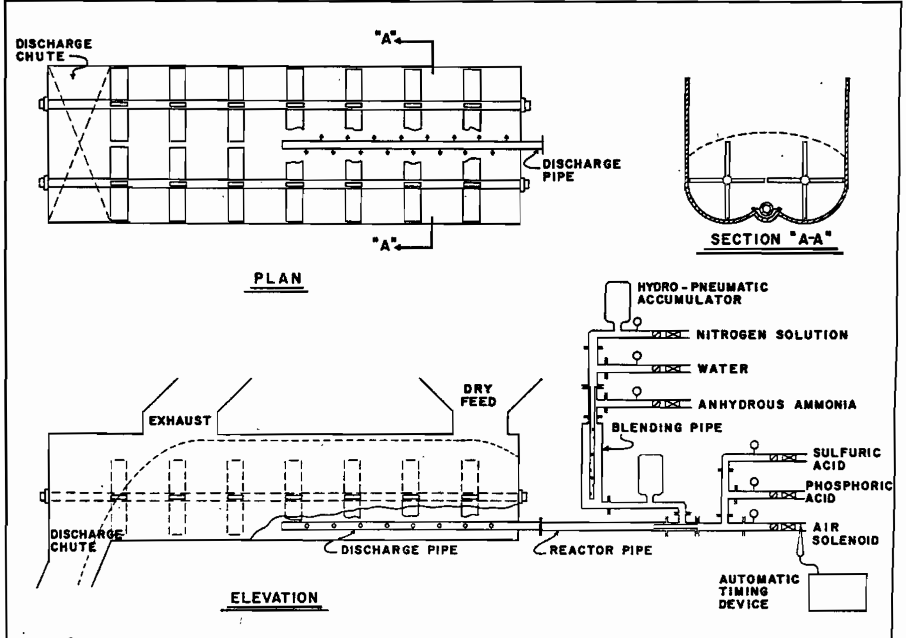


Figure 1. ARRANGEMENT OF PREREACTOR IN GRANULATION PUG MIXER

Figure 1 shows sketches of an early concentric pipe prereactor. Many of you have seen them in the 1959 Round Table Proceedings. The unit has a blending section, a reaction section, and a discharge section.

In the blending section anhydrous ammonia, nitrogen solution and water are blended together in a pipe. They are then discharged through a single pipe into the reactor tee. Sulfuric and phosphoric acids blend together in a one inch pipe which discharges into an acid nozzle. The acid nozzle passes concentrically through the reactor tee and utilizes a simple showerhead arrangement for discharging the acids into the reactor section. The reactor section is a three inch teflon lined pipe which is four feet long.

The acids are neutralized by ammonia in the reactor section which is flanged to a discharge pipe. The discharge pipe shown in figure 1 is simply a teflon lined three inch pipe drilled with one-half inch holes at six inch intervals along each side. The pipe extends eight feet into the mixer.

A one inch air supply line is connected to the acid line as it enters the acid nozzle. The air flow is controlled by a sodlenoid valve which is activated from an automatic timing device. The purpose of this arrangement is to permit the reactor and discharge sections to be given a periodic air purge to prevent the discharge holes from clogging.

Figure 2 is an actual photograph of the mixer exhaust stack during typical granulation operations. The larger of the two stacks is not in use and acts as a control. The smaller is the mixer exhaust stack, and a faint whisp of condensing steam can be seen emitting from it. The sulfuric acid being used in the formulation is neutralized by ammonia in the prereactor. Neutralizing the acid before it enters the mixer prevents the acid from reacting with muriate of potash. By avoiding this reaction, chlorides are not released and the ammonium chloride fume cloud is avoided.

The photograph was taken during a warm summer afternoon. A large volume of steam is being exhausted from the mixer. Because

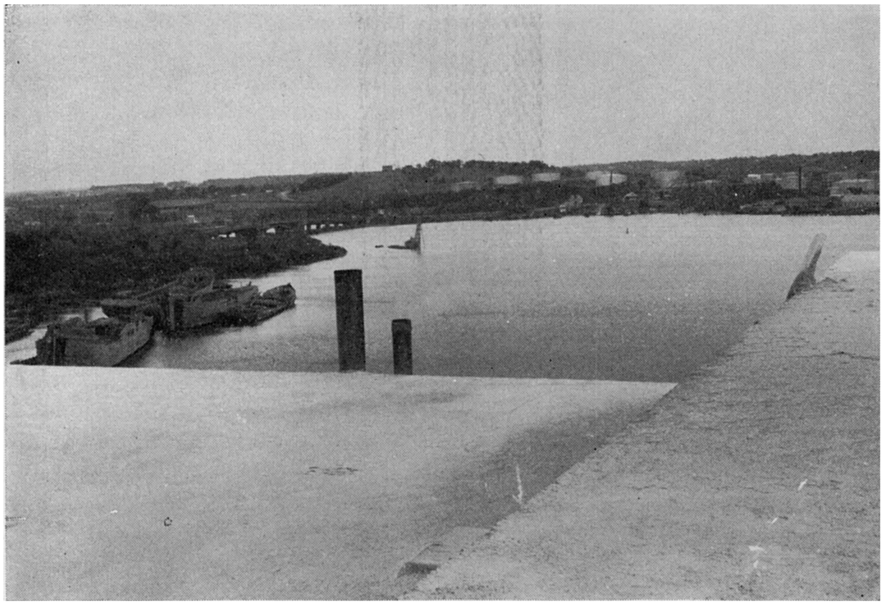


Figure 2.

of the warm day, it is not visibly condensing and consequently the effluent appears to be virtually invisible. Using the prereactor eliminates the need for a mixer exhaust fume scrubber.

Chemical analyses of the mixer exhaust gases were taken while prereacting ammonia, sulfuric acid, and nitrogen solution. The nitrogen solution contained both ammonium nitrate and urea. The analyses showed no evidence of decomposition having occurred. These results have strengthened our belief that since all ammonia formulated passes through the prereactor, the resultant high pH of the acid neutralization environment discourages decomposition of the nitrogen compounds.

The pipe reactor has drastically reduced nitrogen losses from the ammoniator-granulator. Its use prevents localized acid overwetting. It also assures a more carefully regulated sparging of the ammoniation fluids. These factors are instrumental in assuring more complete ammoniation reactions with less nitrogen loss. With several formulations, ammoniation is so complete that even the odor of ammonia is eliminated from the steam leaving the mixer.

Thus far only the advantages and technical potentialities of the system have been described. It is probably equally important to describe the problems which its use has entailed. Our early work at our Curtis Bay Maryland plant showed that all of the plant's usual fluids required in the granulation of any of their grades could be prereacted. These included 60 and 66 degree Baume' sulfuric acid, electric furnace phosphoric acid, nitrogent solution, anhydrous ammonia and water. Water was used only as needed for granulation control, thus of course in some instances it was not used at all. All of the fluids were successfully prereacted in whatever proportions were warranted by raw material economics.

Later, at another plant installation, difficulty in prereacting wet process phosphoric acid was encountered. Its use caused internal clogging of the discharge pipe. Also, with the low nitrogen 1-4-4 ratio grades, the prereactor caused startup operations to be more difficult. Heavier startup recycle was encountered and greater time was required to accomplish leveled out operations. Several months of operation went by before we realized that the prereactor caused this problem by doing too good a job. It preneutralized the acid so efficiently that only steam, hot salts, hot water, and ammonia were left to aid the granulation. This was entirely adequate in most instances, however, it was not adequate for rapid startup leveling out with the high potash grades. We needed the aid of a small amount of acid directly in the mixer to compensate for the cold equipment and materials during the startup.

This need, coupled with the problem of internal clogging from using wet process phosphoric acid led us to a change in the design of the prereactor. We have added a separate acid sparger which permits us to bypass the prereactor with a small amount of the acid. The bypass acid enters the mixer from a sparger located on top of the prereactor discharge pipe. We use this bypass only when needed to aid granulation and to prevent internal clogging from wet process phosphoric acid. Neither the phosphoric acid nor the small amounts of sulfuric acid bypassed create an exhaust fume problem. With most grades full benefit from using the prereactor can be realized. With the others, maximum operating convenience is achieved while still preneutralizing most of the acids.

Another improvement has been made to the prereactor in recent months. The discharge pipe with its one-half inch drilled holes has been replaced by a block sparger.

Figure 3 shows the arrangement used to adapt a block sparger as a prereactor discharge section. A smaller block sparger is shown located above and immediately adjacent to the prereactor block. The smaller block is for the bypass acid. Both blocks are adaptations of the original which was designed by Spencer Chemical Company. It was described by Mr. Joe Sharp in the 1959 Round Table Proceedings. We have found that the block assures more uniform distribution of the fluids. Its deep slots corrode more slowly than thin drilled pipe orifices. Also its use has so greatly reduced clogging that at one of our plants, the air purge system has been completely removed.

In conclusion, we feel that the principal of prereacting in the sparging system offers the advantages of fume elimination and reduced nitrogen losses. We have made significant progress in our efforts to accomplish trouble free performance. Nevertheless, we feel that further progress can and will be made in the future. I thank you.

MODERATOR SPILLMAN: Thank you very much, Norval.

This completes the preneutralization program. We have had an excellent review bringing us up to

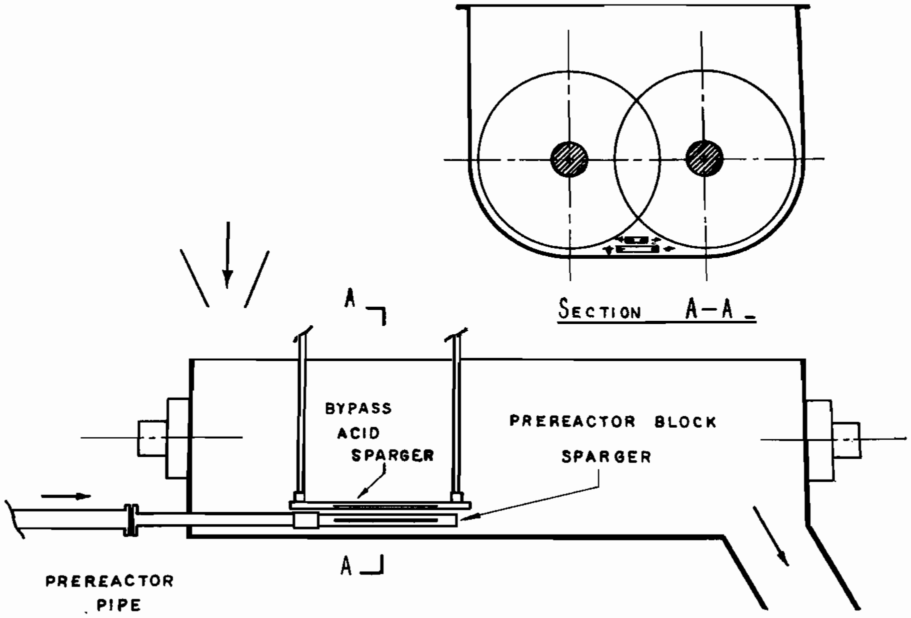


FIGURE 3_ PREREACTOR WITH BLOCK DISCHARGE AND BYPASS ACID SPARGERS_

date on preneutralization. Are there any questions for the panel?

A MEMBERS Yes, why does Minute Maid use a 14-0-7? You have to have phosphate even in Florida, don't you? How do you supply it?

MR. GARRET: Maybe that's a good question. There is an argument right now going on in Florida as to just how much phosphate is required in groves. Certain locations differ in the required phosphate. With others, it is questionable because we think we have enough phosphate in the soil. I mentioned the fact that we do apply straight materials whenever we think necessary.

MODERATOR SPILLMAN: Any other questions? (No response.)

There being no further questions, I would like to turn the meeting back to Joe and thank you all for your kind attention.

On behalf of our entire Round Round Table, many thanks to Tom Athey and his associates for the excellent treat Thursday evening.

MODERATOR REYNOLDS:

Our final speaker on the program was very gracious in holding off until today. You are all quite familiar with our schedule problem, but Mr. Ed Glocker of W. R. Grace & Company, Research, was gracious enough to hold over until today to discuss Statistical Evaluation of Nitrogen Losses in Drying. Ed.

MR. E. GLOCKER (W. R. Grace & Company): Thank you, Joe. Several centuries ago the alchemists had a few very definite goals. They wanted to transmute metals, they wanted the elixir of life, they wanted a universal solvent. As their successors, we have done pretty well on this transmutation problem. The elixir of life, we have just sort of scratched the surface with our vitamins and antibiotics, and we have given up completely on this universal solvent, because bag rot just isn't in it with the container problem we'll have there. So it has fallen to a group in another field entirely to come up with a universal solvent. The mathematicians have come up with some statistical methods that help us to attack these problems we have heard discussed for the last several days, and I couldn't help but sit here and say that when I first added statistics to my chemical vocabulatory some of these words were on the side that we said, well, we won't use these words: sigma, standard deviation, and that kind of thing. But it turns out that these things are very useful, and the fact that the mathematicians have come up with these tools means that we not only have a language problem, but I think we have an iron curtain sometimes. I was gratified to hear that much of the material that you have heard on statistics at this conference and other conferences has made some sense to many of you. There are a few others that I know who are still puzzled by all of this, so I have come here today to tell you how we have started to study our part in the $2,600,000 loss of nitrogen that Dr. Hignett has estimated occurs in granulation each year and how we have taken his advice to study this on the plant scale, of course, without disturbing the plant, but before I do I would like to just explain a little bit or try to poke a couple of holes in this iron curtain on the language problem.

First of all, statistics is essentially as simple as chemistry. In chemistry we have a nucleus and we have a cloud of electrons going around it; in statistics we have data which has an average and a whole cloud of individual points that have been part of making up the average and our samples are simply particular items out of the possible cloud. All the statistician does is to say, we will not only measure the average, we will measure the dispersion of the data that have gone into making up this average. Secondly, he helps the research man, the engineer, the chemist to establish the rules of the game before the game is played. We all have gathered data and then looked at it and said, what in the hell does this mean? So, if we establish the rules of the game ahead of time, we are much better off.

In our case, it was the old problem of comparing nitrogen in versus nitrogen out of the section of the system we were going to study, our dryer. From the statistical standpoint we said, we will have a difference which we will measure between in and out, if we do have a difference, and if we have an apparent difference, it has to overcome a handicap. Now I shouldn't mention that word because a handicap brings up golf, and if Joe Sharp is still here, he knows I'm not a golfer. It was only because the rain came down at the ninth hole that I could quit while I was ahead and I was ahead because I still had one ball left to play with. But anyhow, we have this handicap. The handicap is the degree of dispersion which our data must overcome before we can say that we have found a winner. In this case, a difference has to be significantly greater than the accidental dispersion or the random dispersion that always occurs in any measurements before we can say that we have a winner.

It happened then that we said, let's study our dryer; we suspect that in our dryer operation there may be some loss going on and we'll attack it from that point. We wanted to use a sufficient number of tests to get good precision and accuracy in our work, we wanted to find fairly small differences between the nitrogen in and out of the dryer. We decided then to take 12 samples of material entering the dryer over a period of a couple of hours to detect the fluctuations inherent in our process and our measurements and our sampling and in our analyses, and all the different things that help to add to the random variation and use this as an estimate of the randomness. We took a similar number of samples of the material out of the dryer at the same period of operation, but these we mechanically averaged by compositing. We said to ourselves, the variation out certainly can't be any greater than the variation in. Twelve samples of the material going in adequately measures the cloudiness of our data, the fogginess of it, the handicap. The compositing of the material out will reduce the number of analyses our laboratory will have to perform, so you can see that the tests involved 13 analyses. If we can have the first slide, I will show you what the combination score card and playing field looked like.

(Slide) The average analysis of dried product is the line through the center established in the particular case by the composite of outgoing material. The limits on the randomness of process samples were the boundaries within which we would expect points to fall if we have no loss. Now, obviously, if we have a loss, they are going to be above the outgoing average line, therefore, I think we should shift up for the ingoing points and be significantly above the average line for the outgoing product.

The way we set this thing up was to say that unless the difference between our average in versus our average out was above a certain point, we have demonstrated a difference. A handicap in the usual sense is something subtracted from our score. The statistician doesn't subtract a handicap, he divides by the handicap, and he divides by the adjusted standard deviation which is adjusted in terms of the number of points averaged or the number of samples composited. We were able, by the number of samples we took, to expect to detect differences in the range of the standard deviation which our individual samples measured.

The next slide (slide) will show how in the first plant these points looked. It looks as though we have a slight lowering of the points below the outgoing level. It looks like the material going in is a lower nitrogen level than the outgoing material. Obviously, we are not making nitrogen. We will come back to our interpretation of what this means, but here, at least, we have not demonstrated a loss and we seem to have demonstrated a gain. Statistically this was a significant gain. This is not showing anything except the way the data looked. It doesn't show you any of the statistical arithmetic at all. But this turned out to be a significant increase in nitrogen out of our dryer.

The second slide will show us how another plant looked. This was Plant A. (Slide) At B again we had something which was a significant change in nitrogen but in the direction of a gain. We were not making nitrogen, but here it shows that the nitrogen has gone up.

The third slide (slide) shows that in Plant C it rode along pretty much evenly, the seemingly slight increase in the individual dots above the discharge line was not significant in this case. So we do not have a winner in terms of our difference. The winner has not overcome its handicap, so in plant C, at least, there has been no change.

The next slide (slide) will show how we tried to interpret this seeming gain of nitrogen in the dryer by comparing the nitrogen in and out on the basis of the P_2O_5 in and out and on the basis of the nitrogen versus K_2O in and out. In this case, the interpretation that I have put on this is the following: that the absence of a significant change in relation to P_2O_5 and the absence of a significant change in relation to K_2O means that some non-nitrogen materials are being swept over to the side during a recycle so that the dryer is losing materials other than the nitrogen containing product and the recycle will come back after while and go back into the product, and the net result, of course, would be that at the start-up we will have a little higher nitrogen in our pile and at the end we will have a little lower nitrogen and our formulation will be off. Then it is a matter of which bags the state control officials pick up as to whether they think we have been deficient. So, all in all we have put in the right amount, but the separation of the material through the dryer could give us trouble.

In any event, we have not demonstrated a nitrogen loss, at least in Plant A. The next slide will show how it looked at Plant B.

(Slide) Again, we had a seeming increase in nitrogen through the dryer. We also have a significant increase in terms of nitrogen versus P_2O_5, no increase when we compare nitrogen in on the basis of K_2O. In this case, it indicates to me a loss of P_2O_5 fines to the cyclone which would go back through the system and again we would have the same trouble over the seeming deficiency at the end of the run in terms of nitrogen because it has been diluted by the recycle. It can only go in the direction of dilution which would throw our formulation off and the material in, but it is not being proportioned right through the run.

The final slide (slide) shows us how Plant C looked. If we recall something about the spread of these points, we can see that Plant C is doing very, very well in keeping the dryer discharge about even with the input material and also that the variation is slight. They have good control of the nitrogen seemingly but in comparison with the other charts the nitrogen P_2O_5 figures have quite a spread. It looks almost as if some change had taken place in the process.

This did not prove a nitrogen loss through the dryer, nor did it help us to uncover anything that might say there was separation in dust going to the cyclone, a differentiation among the components or any of that sort of thing. At least in none of the three cases should we have cause to worry about our dryer operation.

I think it is gratifying that, if we are going to try to pin down nitrogen losses in our plants, at least in these three places we should not keep looking to the dryer. We need to consider other possibilities, other points in the system and probably something about how the results that the plant manager sees may be due to this trend of the recycle continuing to dilute the nitrogen containing material so that we may find out that it's more apparent than real and we need to do something about the problems we have heard mentioned before of keeping the material integrated in our plant, not allowing this cyclone dust, if that is what one of the problems is, to cause the trend in the direction of a dilution of our nitrogen components. Of course, it can happen the other way too, because we can take nitrogen out along with phosphate for ammoniating, but since we are studying nitrogen losses I looked at it from the standpoint of how we could interpret these changes.

In conclusion, I would say that we should look elsewhere in our plant than to our dryer.

I would like to give a lot of the credit to Fritz Alfrey who was the one who was in charge of the project, to Joe Reynolds whose plants wanted the study made, and I simply helped as the man who tried to lay the ground rules before the game was played.

Thank you, gentlemen.

MODERATOR REYNOLDS: Thank you very much, Ed. Are there any questions? Ed has performed a real service to our industry. Ed has appeared before this group and similar groups during the last three or four years. I think all the way through this meeting you have seen reference to statistics and many of us a few years ago were really out in left field. I think we are getting a little bit closer now. As he says, we at least know what some of the ground rules are and some of the interpretations.

Are there any questions or any comments? This ties right in to our standardization and uniformity.

We have finally reached the end of our two and a half days' program. From your comments we have overheard in the rooms and the corridors and up and down the halls, it seems as if we have had a good meeting. We certainly hope we have. There have been many new ideas advanced and there have been a lot of new thoughts brought out in the meeting.

As far as trying to sum up the meeting, I am not going to do that. I think that some of the comments that were made initially on our opening day still hold. I think we all realize that this is our group and it is a group as a whole, everyone is important, everyone can come here, each and every person, express himself and that is what we want. You will be hearing from us very shortly as far as next year's program. We have some ideas and we have requested many people here in particular to accumulate ideas from your areas, because to keep your interest we have to have help, we have to have your ideas, so send them along to us.

As far as anything else before our group, I just want to express my appreciation for the opportunity of being Chairman this year. I have really enjoyed it, being most appreciative of your patients, your kindness in helping us through this one.

I want to thank the Executive Committee for also helping, everyone has pitched in and made this a good meeting. I am sure I will see many of you between now and next November, but don't forget November 8th, 9th and 10th, 1961, back here at the Mayflower. Our 1960 program is adjourned.

(The Round Table adjourned at 12:45 o'clock p.m.)

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Note: Many more participants who did not announce their names when asking or answering questions.

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PROCEEDINGS

OF THE

11th ANNUAL MEETING

FERTILIZER INDUSTRY ROUND TABLE

1961

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Held at the
MAYFLOWER HOTEL
Washington, D. C.
November 8-10, 1961