PROCEEDINGS
OF THE
19th ANNUAL MEETING
FERTILIZER INDUSTRY ROUND TABLE
1969

* * *

Held at the
MAYFLOWER HOTEL
Washington, D. C.
November 5, 6, 7, 1969

1969
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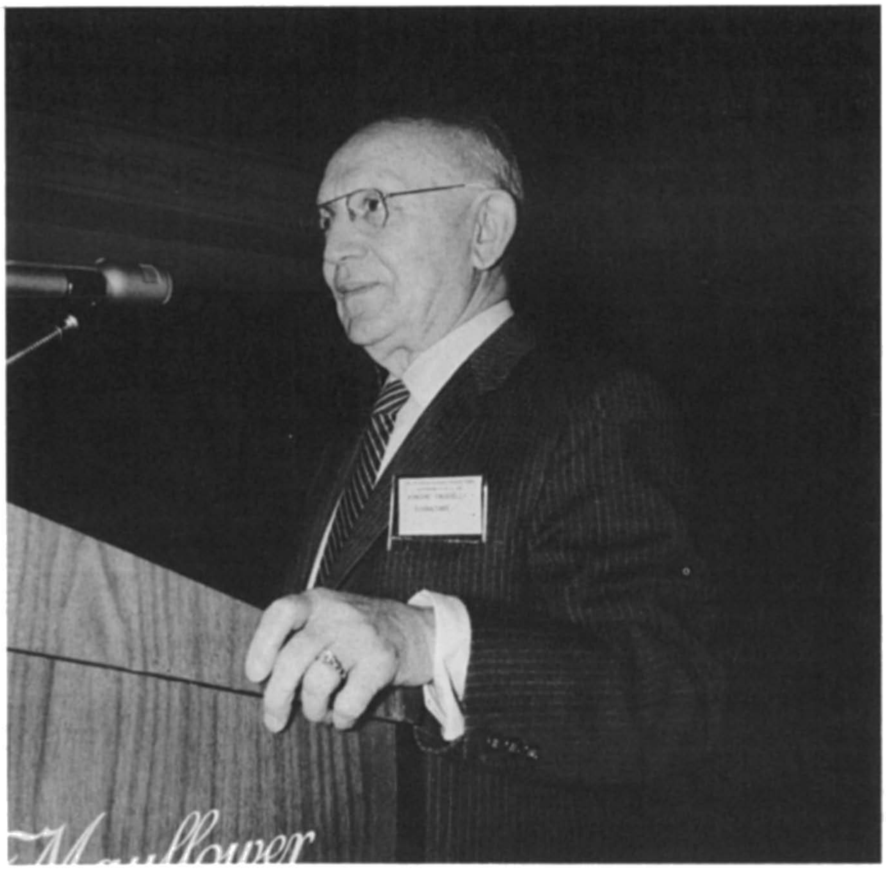


THIS ISSUE OF THE PROCEEDINGS IS DEDICATED TO THE MEMORY OF VINCENT SAUCHELLI OUR CHAIRMAN 1951-1969

Wednesday Morning Session, November 5, 1969

The 19th Annual Meeting of the Fertilizer Industry Round Table convened
at ten o'clock a.m. in the Grand Ballroom of the Mayflower Hotel,
Washington, D.C. Mr. Albert Spillman, Acting Chairman, presiding.

CHAIRMAN SPILLMAN: Good morning Ladies and Gentlemen. Welcome to our 19th Annual Meeting of the Round Table. With a heavy heart and great sadness I advise you of the death of our Chairman and Co-Founder, Dr. Vincent Sauchelli. Vince died at his home in Baltimore, this past October 1st, at around 9 p.m. of a sudden heart attack. Our Executive Committee was with him a short time before, going over the final arrangements and program for this meeting. Vince was very happy with the program and was eagerly looking forward to chair this 19th Annual Meeting. He has superbly chaired each of the previous 18 Annual Meetings and is mostly responsible for the success of The Round Table.

Our Executive Committee met in Baltimore on October 22. I was asked to be the Acting Chairman for this meeting. I accepted with much humility and shall try to do the best job I possibly can.

Before we start our first session of the meeting, it will be appropriate for all of us to stand in silent prayer for one minute to register our great sorrow and devotion for our departed friend Dr. Vincent Sauchelli. Thank you very much.

Dr. K. D. Jacob, U.S.D.A. (retired), a good friend and loyal supporter of The Round Table, has intimately known Vince for many years. Dr. Jacob has agreed to review the life and works of Vincent Sauchelli, 1892–1969, and express the warm feelings all of us had for Vincent Sauchelli.

Vincent Sauchelli 1892–1969

K. D. Jacob

Death has claimed, at the age of 77, the chairman of The Fertilizer Industry Round Table, who also was one of the Round Table's founders.

Vincent Sauchelli died of a heart attack on October 1st at his home, 303 Overhill Road, Baltimore. Besides his wife, the former Elizabeth Mason, he leaves a stepdaughter, Mrs. Rose Mary McKechnie of Baltimore, and four grandchildren.

Vince was born in Foggia, Italy, on July 11, 1892. He came to the United States at the age of two and grew up at Waterbury, Connecticut. He received a Bachelor of Science degree at Massachusetts State College (now the University of Massachusetts) in 1915. He attended the Massachusetts Institute of Technology in 1915 and 1916, during which time he also was employed as an analyst by Arthur D. Little, Inc.

From 1916 to 1923, Vince was director of research for the Societe Internationale de Plantations et de Finance in Kuala Lumpur, then a part of the Federated Malay States. He served with the British Army in 1916 to 1919.

He was employed by the Koppers Company, Pittsburgh, from 1927 to 1935. He then began some 20 years of service, 1936 to 1957, in Baltimore with the Davison Chemical Corporation (now a division of W. R. Grace and Company) in capacities ranging from assistant to the president, to director of agricultural research. He served as consultant in fertilizer technology to the National Plant Food Institute, Washington, D.C., from 1957 to 1961.

His activities in the consulting field, in which he was currently active, included service with the Agency for International Development in the Agency's program on fertilizer technology in India. In fact, he was anticipating an early trip to India in this connection at the time of his death.

The long-felt need for a medium through which industry personnel could discuss freely advances and mutual problems in plant operation and in fertilizer technology, distribution, and use led to the organization, in 1951, of The Fertilizer Industry Round Table. Vince Sauchelli was a founder of the Round Table, and he had served as chairman of its executive committee from the beginning.

Through the untiring efforts of its leaders, The Fertilizer Industry Round Table has gained international recognition as a forum for the Industry.

At the beginning of his service with the National Plant Food Institute, Vince was instrumental in organizing the Chemical Control Problems Conference in cooperation with the Association of American Fertilizer Control Officials. The 12th annual conference was held in August 1969 in Houston, Texas.

In introducing the program on the occasion of the 11th Chemical Control Problems Conference, Jim Archer of the International Minerals and Chemical Corporation, program chairman, said:

"Far-sighted people, led by Vince Sauchelli, saw the value of bringing state control people and industry together where mutual problems could be discussed, and both have benefited."

For the past 20 years, Vince had exercised leadership in directing the voluntary cooperative fertilizer check sample program sponsored by the Association of American Fertilizer Control Officials and the National Plant Food Institute. Generally known as the Magrunder Check Sample Program, this fine program will continue to benefit quality control laboratories, both state and industry alike.

These are examples of the foresight and leadership typical of Vince Sauchelli throughout his professional career. He anticipated the needs of industry, and he was most effective in bringing together the appropriate groups of people to work on mutual problems.

Vince was the author of several books and numerous articles on fertilizers. His books included "Manual on Phosphates in Agriculture" and "Manual on Fertilizer Manufacture". He edited and contributed chapters to two American Chemical Society monographs -- "The Chemistry and Technology of Fertilizers" and "Fertilizer Nitrogen: Its Chemistry and Technology". His most recent book, "Trace Elements in Agriculture", was scheduled for publication this October. For a number of years he had contributed a monthly column, "Views and News", to the trade journal Agricultural Chemicals.

He was a member of the American Chemical Society, the American Society of Agronomy, The Fertilizer Society of London, and Phi Kappa Phi fraternity, and he was a fellow of the American Institute of Chemists. He was active in organizing the Baltimore "Old Timers" luncheon at Christmas each year.

A scientist, a scholar, and a gentlemen of the old school, Vincent Sauchelli will long be remembered for his outstanding service in the fields of fertilizer technology and use.

CHAIRMAN SPILLMAN: Thank you Dr. Jacob for that most deserving praise for Dr. Vince Sauchelli.

Immediately after our last year's Round Table our members were asked to kindly send to the Executive Committee ideas and suggestions for this 19th Annual Meeting. Response was great. From the information received your Executive Committee was kept comfortably busy throughout the year, checking and analizing the many suggested technical subjects asked to be discussed. Many of you here for this meeting will note that the papers to be thoroughly discussed during our 3 day meeting are pursuant to your wants. We hope the program meets with your approval. Our discussion leaders are competent,experienced and each has the thorough knowhow for his assigned subject.

This morning we will have a key-note address titled "Wither Away Fertilizers" and two papers on "Phosphoric Acid Processes".

This afternoon "Papers" will cover Fluid Bed Phosphate Rock Driers and Calciners; Sulphuric Acid form Volcanic Sulphur Sources; Compacting and Granulation and Testing Particle Hardness for Granulated Fertilizers.

Thursday morning that all important subject "Air and Water Pollution" will be thoroughly discussed. Please do not miss this most important subject all of us must learn more and more.

Thursday afternoon will be a very important part of our program. I hope all attending this 3 day session will be present. The Secretary-Treasurer will give his report; voting for our expanded Executive Committee; Discuss the possibility of another location for our 20th Annual Meeting to be held in November, 1970; Financing; Printing of future proceedings and discussion topics desired for the next meeting.

Our final session on Friday will cover Shrinkage; Diaphram Valves; A New Granulation Process and Low Fluorine Triple Super Phosphate.

It has been the policy of The Round Table to serve all segments of the Fertilizer Industry on pertinent up-to-date technology. We will continue to give a great deal of attention "To Production People Problems" with interesting subjects for the entire Fertilizer Industry.

Mr. Hugh S. (Hoopy) Ten Eyck, Key-Note Speaker, has chosen to discuss "Whither Away Fertilizers"

Mr. Ten Eyck retired as Chairman of the Board of Occidental Agricultural Chemicals Corporation of New York in 1968. He is presently a consultant to the Fertilizer Industry and resides in Everglades City, Florida. In addition to his consulting activities he is a major participant in a "Resort Motel Marina Complex" in Everglades City.

Mr. Ten Eyck was born in New Jersey. In 1931, he was graduated from Lehigh University with a bachelor of science degree in metallurgical engineering.

After two years with Pyrites Co., Ltd., as a metallurgist, Ten Eyck returned to Lehigh under a fellowship for graduate study.

After holding positions as a research metallurgist, department foreman, assistant general superintendent, and chief research metallurgist, Ten Eyck was named assistant to the president of Southern Phosphate Corporation in 1941, and in 1944 was named vice president in charge of sales, production and for general administration.

In 1948, Ten Eyck joined International Ore & Fertilizer Corporation as vice president, and under his guidance, the company grew from a four-man operation to sales of $76,315,000 in 1965. In 1960, Mr. Ten Eyck was named president and held this position until his retirement.

In 1963, INTERORE was acquired by Occidental Petroleum Corporation. Mr. Ten Eyck was named a director of Occidental and executive vice president for the fertilizer group of the corporation. In May 1965 he became president of OXYCHEM. On March 14, 1967, Ten Eyck was named Chairman of the Board of OXYCHEM.

He is married to the former Pauline C. Sanders and the father of three children.

Mr. Ten Eyck has had a colorful, successful career. And now, friends, it is my real pleasure to give you the man who has made "Hoopy" in our industry, night and day, to our mutual profit and pleasure. Mr. Ten Eyck, please:

Wither Away Fertilizers

Hugh S. (Hoopy) Ten Eyck

Whither away is, of course, a little old-fashioned word for "where away". However, I use the word "whither" because of the other "wither" spelled without an "H", meaning to fade away or dry up. When you look at where something is going, normally all or at least part of what it leaves dries up. As we explore where fertilizers are going, we also have to jointly explore what's drying up or disappearing.

I think the next ten years, perhaps it is five years, perhaps it is fifteen years, will see the greatest changes occur in this industry. I am speaking now of the technical and operating economic phases of the industry and more or less ignoring the market side of the industry. I might add that the markets are in such a mess that any words I might add to the existing market confusion would perhaps only add to the confusion rather than to help it. So let us stay with the basic technical and production facts that appear to be in the offing and the effect that these changes will have on the existing industry.

We'll also overlook the effect of the minor elements, calcium and other necessary ingredients of fertilizer, and stay with the three basic components, nitrogen, phosphorus and potash.

First, let's set down some premises which I believe to be true.

1. The tendency towards production of the most concentrated products possible, and shipping these to the market for conversion, has become a fact in many instances and becomes more and more probable in the remaining instances.
2. The conversion within the market area of these concentrated products to finished goods and their distribution at that point has already started and eventually will replace the movement of semi-finished goods to these markets.
3. An intensification of the production of high analysis finished goods which will provide increased agronomic yields per dollar of cost and require new techniques of production and distribution as well as new actual farming techniques.
4. The intensification of basic concentrated raw material manufacture in those areas most economically suited for the production of such products.

The above premises are all tempered by the following:

1. Desire of many countries to be independent of foreign sources of materials and, coupled with national pride, resulting in the construction of unsound, uneconomic and unrealistic plants contrary to the above premises.
2. An almost complete lack of research within the fertilizer industry with the exception of pesticides, herbicides, etc., and with the exception of mechanical improvements within existing plants. This is understandable in one sense. Due to the low return in the fertilizer industry funds have been practically impossible to get for research, particularly pure research.
3. The inertia and lack of imagination present both in the industry and in the governments and farmers who use this industry's products.

It is hoped that the first set of premises will be strong enough to overcome the resistance encountered in the second set of premises. If the positive premises do not overcome the negative premises, the industry and the world in general is in for some very tough sledding in the years to come.

Let us take the three basic components now and attempt to analyze their future. The first and the most difficult product in which the future can be predicted is potash. Potassium chloride as produced today is at its practical maximum concentration. Small improvements may increase the K_20 content by fractions of percents, but that's about all. As fertilizer usage becomes more intensive, it is possible that the chloride content in muriate of potash will become objectionable from an agronomic viewpoint. Potassium nitrate is an improvement but it is still not a concentrated product. Potassium sulphate has certain agronomic advantages but, of course, is a dilution from the chloride. Mettallic potassium, while a possible product, appears at the moment to be uneconomical. Here research on this might provide some interesting possibilities. However, so far as I know, I don't think metallic potassium is being looked at in this vein.

The possibility of potassium polyphosphate or meta phosphate appears to present some possibilities but here again the industry is waiting for an economic form of phosphate to provide this product as a basic raw material. Thus, in the immediate future and perhaps some time in the future, it would appear as though potash will stay much as it is today.

The second component, nitrogen as ammonia, has perhaps reached its maximum concentration in an economic form. There will be improvements in operating costs resulting from mechanical improvements in processing. Transportation problems concerning ammonia have been for all practical purposes solved although, of course, they can still be improved.

There is, however, one major change in ammonia that is happening and that must happen in the future and that is the location of ammonia plants. There's a tremendous concentration of ammonia plants, for example, in the Louisiana and Texas Gulf area in the States. Long time contracts exist for gas supplies but to my knowledge most of them are hooked to a five year price clause and are subject to price negotations after the first five year period. With the natural gas shortage now apparent in the States, it would appear as though plants in this area must be faced with a rise in cost of natural gas.

New ammonia plants should be built in those areas in the world where there is no industrial market for gas, where there is a surplus of the product and where in the foreseeable future there appears to be no competitive situation that would change the basic price of cheap gas.

It is true modern manufacturing technology has lessened the importance of the price of natural gas in the total cost of the finished product. However, building a plant where 40c or 50c gas is a possibility within ten or fifteen years would not seem to make sense when there are areas in the world that gas can be contracted over a twenty year period for less than 10c. This is perhaps the most important change facing us in the nitrogen field and with the advent of big ammonia plants in the Maracaibo Area, the Arabian Gulf, and North Africa, the future of the producer in areas other than these certainly becomes questionable.

This leaves us with phosphorus which perhaps presents the greatest possiblity for change and in turn will have a great effect on nitrogen and potash as well as in the general field of phosphates. This industry has shown, in spite of itself, much progress towards the optimum product. We've seen ground phosphate rock, for example, decrease in popularity and its use isolated to limited areas throughout the world. We've seen single superphosphate practically disappear from the marketplace. We've seen triple superphosphate, as a more concentrated product, come in replacing single super and in turn being, at least in part, replaced by more concentrated ammonium phosphates. Here the change has taken place slowly but with a degree of certainty.

We have also seen the advent of merchant grade phosphoric acid into the world markets. We've seen the development of transportation facilities for this product and I would say that these transportation problems have been solved. We've seen the production of superphosphoric acid, which in theory should replace merchant grade acid. Its growth has been limited so far to its use in liquid fertilizers, particularly so if its feed stock is wet process merchant grade acid. With, of course, the development of superphosphoric acid has come some work in polyphosphates opening up the field of both its agronomic possibilities as well as new technical products.

Now the question is what comes next and that question is, of course, answered very simply by atomic energy located at the phosphate rock mines producing elemental phosphorus which can be competitive in the world of phosphate products. How far away is this? I really don't know, but I think it is within five year, ten years, maybe fifteen years from now.

It does appear that atomic power will be produced at a cost of somewhere between 3 and 3.4 mils and if this power can be produced on top of or at least adjacent to the mines, elemental phosphorus becomes the logical and economical source of P_2O_5 for the fertilizer industry. Shipping is definitely not a problem, both to port and aboard vessel to market, or by rail direct to market. This will be the most tremendous change that this industry has ever seen. This means that a market will import phosphorus and ammonia if it cannot produce ammonia economically within the country. Import potassium probably as a chloride, convert the phosphorus into polyphosphates or perhaps meta phosphates, combine it with potash NH_3 and come up with a whole new range of concentrated products and more efficient fertilizers from an agronomic viewpoint.

Let's take a look at how this will affect Europe, already a questionable nitrogen producer from a economic viewpoint, now would probably drop out of the world export market. It would import phosphorus and perhaps import ammonia, make fertilizers for its local use and that would be about it. It wouldn't be able to export products and compete with nitrogen and phosphorus produced at the sources of cheap raw materials. The same would be true of Japan.

Florida's immense investment in triple phosphate and ammonium phosphate production could in theory be limited almost entirely to local sales.

Developing countries with shortages of foreign currency and with an excess of national pride as far as being self-sufficient in finished fertilizers is concerned, would be in a position where somehow or other they would have to find foreign currency to provide for the importation of elemental phosphorus and ammonia. Otherwise their national economy would become even more shaky than it is now.

Hundreds of plants throughout the world would become economically obsolete literally overnight.

Tremendous amounts of capital would have to be developed to provide the cheap power necessary for the production of phosphorus.

Practically every plant now in operation is not only economically obsolete but a good bit of it would be technically obsolete.

Elemental phosphorus could be burned very easily to a very highly concentrated superphosphoric acid so the capital investment for the conversion of the phosphorus is relatively small.

This sounds like a chaotic condition that could prevail in the industry. However, the change is tempered by many things and they are basically the negative premises I cited at the beginning of this paper, plus the tremendous capital involved in such a step, not only in the atomic energy plant but in the transportation facilities and by the cost of the obsolesence of existing plants.

This affects not only the United States but every phosphate rock source in the world. It makes all sources of phosphate rock a little more competitive with each other. High grade rocks lose at least some of their today's value for if a low grade rock has the right silica content it needs no upgrading. Transportation cost differentials are minimized due to the concentration of elemental phosphorus.

In spite of all the factors, one thing for sure will happen when the first producer, and it will probably be a Florida producer, starts his first elemental phosphorus plant – every other producer is going to have to do the same thing so there will be a situation that will develop very rapidly, in terms of past progress in the industry. Planning for the future must certainly keep this in mind and be ready for such a change.

Summing up, you can say that phosphorus and nitrogen will be produced as elemental phosphorus and ammonia in those areas in the world where the basic raw materials such as natural gas and phosphate rock are cheap, assuming, of course, that atomic power can be economically constructed at the source of cheap phosphate rock.

Transportation costs of both ammonia and phosphorus will have less effect on location than previously due to the high concentrations of the products and the relatively lower cost per unit of plant food. As larger ships come into use this unit cost will naturally be lowered even further.

A further concentration of raw material production to the hands of fewer corporations because of the enormous capital investments both in plants and transportation facilities.

Areas without their own cheap sources of raw materials will be forced to alter their production facilities to plants adapted to the importation of ammonia and phosphorus as well as potash and conversion of these three components into end products for their own local marketing areas. This will call for a geographical rearrangment of a major part of the fertilizer industry.

The necessity for the industry to do some active research on the production of high analysis NPK compounds using elemental phosphorus as a source rather than wet process phosphoric acid.

A research program on the agronomic use of such compounds so that the consumer can extract the maximum benefit per unit of dollar cost of fertilizer.

An educational program probably undertaken by an agency such as the United Nations to demonstrate to developing countries the economic fallacy of trying to solve their national ego, or fears, as the case may be, by building their own fertilizer plants instead of importing the concentrated raw materials. This latter point is perhaps one of the key issues to heading off the so-called starvation period which still appears to be ahead of us and not too many years away.

I am certain there will be a lot of people who will disagree with the theme of this paper, but, if this paper generates that much thought, it has in one sense served its purpose.

I have just skimmed the surface of whither and wither, but perhaps some of these thoughts are worthy of your forward looking and forward planning. They will happen – it is just a question of when and to what extent.

CHAIRMAN SPILLMAN: Thank you Mr. Ten Eyck for your forward look into the future, during the next decade, on possible significant changes in "Phosphoric Production" thru use of low cost "Atomic Power" Who has a question for Mr. Ten Eyck ?

A MEMBER: What is the alternative of instead of producing ammonia where the gas is cheap, of transporting the gas to the place where the ammonia is desired? I think it is questionable whether the natural gas can be transported as cheaply as ammonia, however, there are developments going along on transportation of liquified natural gas. I wonder if it may come to pass that it will eventually be cheaper to transport the natural gas rather than the ammonia?

MR. TEN EYCK: It could well be. I think when you get the cost of liquification down to that stage, you can get so much more money for it as a fuel, I do not think too many people are seriously considering that this might be a major source of hydrogen for ammonia. However, it should be a rather simple essay in paper and pencil economics. None of these things ever are, but it is nice to say it that way.

WALTER R. HORN–Farmland Industries: Did I get the implication that maybe the ammonia plants on the lower Mississippe River will be losing somewhat their competitive advantage because of ammonia costs.

MR. TEN EYCK: Here again, it is very easy to say yes or no but then you have to qualify it with so many qualifications. It is how close they are to their local market. Some of the plants may have their own gas tie-ups through a relationship back to several other corporations. It could be that natural gas in Texas may go to 50 cents. On the Arabian Gulf it costs 5 cents. You can probably meet and beat ammonia delivery from Arabia, let us say, to England versus ammonia from a 50 cent gas plant to new modern large plants with a lessening inport of the cost of gas to the final cost of ammonia. I would say that it is a pretty tough situation that they may be looking forward to.

A MEMBER: Could I just add something about the cost of liquid natural ga. There have been recent contracts for gas from Africa to Continental United States. I believe that the costs are going to be about 50 cents a thousand. Also from Alaska to Japan at about 50 cents a thousand. The costs of liquifying and transporting natural gas are not cheap.

MR. TEN EYCK: Thank you, I agree.

MR. AI, Lumis Co.: You mentioned about elemental phosphorous production near those areas where they have sources of phosphate rock. When you are making furnace acid you need a supply of carbon or coke as a reducing agent. Would you care to comment: How in those areas where there are no coal deposits they can get by that kind of problem?

MR. TEN EYCK: I know somebody would think of that. This is one of the questions that has to be answered in the general economics of such a plant. Can you get coke in there? It is a raw material that cost the same as power. The whole thing has to balance out.

CHAIRMAN: Thank you Mr. Ten Eyck. I hope you can stay with us for the remaining program. An enthusiastic, long applaus was given by the membership to Mr. Ten Eyck.

CHAIRMAN SPILLMAN: We have two discussions on that important growing use of phosphoric acid. Our speakers will discuss phosphoric processes by"Kellogg-Lopker and Hemihydrate Process by Day and Zimmerman. First Mr. William Turner.

The Kellogg-Lopker Phosphoric Acid Process

L. E. Bostwick and William Turner

The chemical reaction between phosphate rock and sulfuric acid, to yield phosphoric acid and calcium sulfate, proceeds rapidly and nearly to completion when reasonably close control is exercised on the reaction environment. Separation of the phosphoric acid from the calcium sulfate crystals, however, can only be carried out efficiently if the crystals develop to such size and shape that the liquid will drain rapidly away from the crystals, and this in turn requires that the crystals be large and well-formed and that the particle size range be small. The overall conception of phosphoric acid production may thus be changed from one of attack on the rock by acid to one of crystallizing calcium sulfate. This change od concept is borne out in operation of a phosphoric acid plant since, if the calcium sulfate is crystallizing well, the whole system is functioning smoothly and correctly and is under good control.

Edwin Lopker's contribution to the technology of phosphoric acid manufacture is the invention of a new process in which phosphate rock rapidly and completely reacts with sulfuric acid and the resulting calcium sulfate crystals grow rapidly and to such size and shape that separation of the phosphoric acid from them is more rapid and complete than with any other process. Development of the method and apparatus of the new process was carried out at the phosphoric acid plant of the Marchon Division of Albright and Wilson, Ltd., in Whitehaven, England, while Lopker was acting as consultant to Marchon.

A pilot plant having a capacity of 60 long tons of P_2O_5 per day was constructed by Marchon to Lopker's specifications and under his direction adjacent to a conventional plant of the same capacity. Arrangements were made to feed one filter with slurry from either plant so that comparative testing of filtration rates and recoveries was possible. The pilot plant was started in December, 1966, and was operated nearly continuously until about August, 1968, when it was shut down for conversion into a demonstration plant for the process.

The successful pilot plant operation provided the needed incentative for Marchon to begin engineering and construction of a full scale plant having a nominal capacity of 240 long per day of P_2O_5 as 30-32 percent P_2O_5 phosphoric acid to be produced from 75-77 percent BPL Moroccan phosphate rock and 77 percent H_2SO_4 sulfuric acid. The first phosphoric acid was produced in the new plant in October, 1968. Startup was remarkably smooth and specification acid was produced. Production was slowly increased to design rate and beyond, with few problems. Since December, 1968, the plant operating factor and the overall P_2O_5 recovery have been at least as good as in conventional plants. Filtration rates have been consistently high and have met the design expectations of rates higher than those conventional plants.

Marchon management have concluded that the Lopker phosphoric acid process has been fully proved in full scale operation, that any operational problems are mechanical, not process, and that the mechanical problems are for the most part not connected with the process.

Meanwhile, Lopker and Kellogg concluded negotiations in May, 1968, for Kellogg's purchase of Lopker's invention, complete with world rights for exploitation. Shortly thereafter, Kellogg and Albright and Wilson, the parent company of Marchon, reached agreement whereby Kellogg would have complete access to all engineering and operating data concerning the process. Further, it was agreed that the 60 ton per day plant would be made into a true demonstration plant for large-scale testing of clients' phosphate rocks.

Of the many ways in which the apparatus may be arranged to carry out the concept of the Kellogg-Lopker process, Figure 1 will serve as an example. It should be noted that choice of this arrangment does not necessarily mean that this is the preferred one. In designing a plant, choice of alternatives is made to suit the requirements of the particular operation.

In Figure 1, two vessels are interconnected to provide a recirculating flow path, with the two vessels being offset vertically. The slurry is pumped from the lower vessel, called the "dissolver", into the upper vessel, called the "evaporator", and returns by gravity from the evaporator to the dissolver. The evaporator is maintained under vacuum, while the dissolver is at atmospheric pressure.

The recirculating flow of reactor slurry through the system is downward in the dissolver, to the pump, then through the pipe to the evapator, where it enters approximately tangentially and imparts a rapid swirling motion to the evaporator contents. The slurry flows downward in the evaporator and through the connecting pipe to the dissolver, where it also enters approximately tangentially to produce a rapid swirling in the upper portion of the slurry in the vessel. The downward flow in both vessels eliminates the possibility of any solids settling from the slurry.

Phosphate rock and the recycling phosphoric acid solution from the filter system are fed into the dissolver, and the swirling action is adequate to mix them into the recirculating slurry. In the evaporator, the sulfuric acid is distributed over the surface of the swirling slurry and mixes with it.

Sufficient height is provided in the dissolver to accomodate the increase in slurry level when the system is shut down and the vacuum is shut off, equalizing the levels in the two vessels. If desired, the pump may continue recirculation, although at a reduced rate due to the increased hydrostatic head imposed upon the pump. A shutoff valve is provided at the bottom of the dissolver so that slurry may be retained in the system. Thus, only the pipe from the pump to the evaporator need be drained if it is desired to inspect the pump. The slurry production, corresponding to the system feeds, is withdrawn through either of the valves shown.

As the phosphate rock enters the dissolver, it is immediately dispersed into and mixed with the large volume of recirculating slurry. An antifoam agent can be added, if desired, and any CO_2 formed is quickly removed from the vessel as shown. It should be noted that plant experience has shown that on the order of half of the CO_2 and other noncondensable gases are released in the dissolver. This effectively reduces the load on the vacuum system of the evaporator, with consequent economies in operation.

The phosphate rock rapidly dissolves in the liquid phase of the recirculating slurry and so raises the calcium content of the liquid phase by a small amount. As this occurs, the liquid phase becomes reduced in sulfate as calcium sulfate leaves the solution, largely by crystallization on the great mass of calcium sulfate crystals present in the recirculating slurry. The phosphate rock is added in a manner to avoid substantially increasing the calcium content in the recirculating slurry. It is this small change that insures growth of the calcium sulfate crystals and avoids precipitation of excessive quantities of fine crystals.

The addition of the sulfuric acid raises the sulfate content of the liquid phase of the slurry by a small amount and the calcium content is reduced by crystallization of calcium sulfate on crystals already present in the recirculating slurry. Again it is important to avoid substantial increases in the sulfate content. Allowing only a small change assists in insuring growth of the calcium sulfate srystals and avoiding precipitation of excessive quantities of fine crystals.

Recirculation ratio may be defined as the time rate of flow of slurry through the dissolver and evaporator circuit divided by the time rate of flow of slurry out of the circuit to the filter.

The effect of controlled recirculation ratio on variation in concentration of calcium and sulfate ions may be realized by reference to Figure 2. These curves were determined for a specific set of conditions of rock type and analysis and of desired excess H_2SO_4 content in the finished slurry. Use of other rocks or changes in desired excess H_2SO_4 level will change the position of the curves to some small degree. At a recirculation ratio of 50:1, for example, the maximum increase in concentration of CaO ions in solution in the liquid phase of the slurry is about 0.4% and the corresponding maximum increase in SO_4 ion concentration is about 0.7% Both of these figures assume that all of the CaO of the rock and all of the SO_4 of the sulfuric acid are momentarily completely in solution, with no compensation being made for removal of ions through crystallization. In contrast, if this recirculation ratio is reduced to 10:1, changes in ion concentrations show a corresponding increase by a factor of well over four. Similarly, doubling the recirculation ratio approximately halves the changes in ion concentrations.

The derivation of the curves of Figure 2 assumes that slurry passes the point of addition of rock or sulfuric acid and does not return until reactions are complete, crystallization has taken place and the slurry has returned to its original composition. These assumptions become facts in the Kellogg-Lopker system, since recirculation ratio is constant and slurry flow is positive around the circulation loop. This plug flow concept is a unique departure from other reactor configurations and leads to the conclusion that, with the elimination of the possibility of back mixing, maximum concentrations of reactants are predictable, the effects of external adjustments of reactant feed rates are predictable and the total system is therefore under close control.

Recirculation in the conventional types of reactor systems that are in commercial operation is promoted either by agitators in a tank or tanks, or by returning slurry from a later stage of the reactor system to an earlier stage, or both. Although the calculated theoretical slurry flow from a series of agitators in a single tank may be very large, the actual flow is uncontrolled and the interactions of the agitators are unpredictable. It is therefore possible that back mixing promotes local recirculation of small bodies of slurry which, in the vicinity of points of addition of reactants, may receive far more than the calculated average quantity of reactants. This has the effect of reducing the overall recirculation ratio for that portion of slurry with consequent detrimental results in crystallization.

In the multiple tank or multiple compartment systems, slurry is pumped from a section near the end of the series to the first section of the series and the slurry may be cooled before it reenters the first section. This pumping provides control of recirculation, but pumping costs are high and the ratio of recirculated slurry to slurry leaving the system as filter feed is rarely greater than 15 to 1.

In the all-in-one system recently announced, a single large agitator in a draft tube promotes recirculation in a single tank that is maintained under vacuum to provide simultaneous mixing and cooling. Calculated slurry flow in this system indicates recirculation ratios of 300:1 or more, but it should be noted that circulation in this reactor is dependent upon internally agitated and baffled flow rather than the positive, unidirectional flow provided by the Kellogg-Lopker system.

High recirculation rates have the additional advantages of reducing the temperature differential within the system. The heat of dilution of the sulfuric acid and the heat of its reaction with the phosphate rock are removed in the Kellogg-Lopker system as in some conventional systems by vaporization of water from the solution under vacuum. Figure 3 demonstrates the effect of varying recirculation ratio on the temperature difference experienced in any such reactor system. The curve was determined for the same rock and attack conditions as Figure 2. For example, with a recirculation ratio of 50:1, it may be seen that the temperature differential existing between the contents of the evaporator and the contents of the dissolver is approximately 2 degree F. On the other hand, if the recirculation ratio is reduced to 10:1, the temperature differential becomes 11 degree F. Such a drastic and rapid temperature drop usually results in excessive nucleation of calcium sulfate crystals and usually requires a long retention time in the system to allow the small srystals to dissolve and for the larger crystals to grow. If a long retention time is not specified, the filtration rate of the slurry will be poor. Further, deposition of calcium sulfate and other compounds on the internal surfaces of the apparatus becomes very troublesome.

It would seem that extremely high recirculation rates are most desirable to reduce changes in ion concentrations and temperature to very low values. It must be remembered, however, that a rock particle dissolves at a rate dependent upon its internal physical structure, chemical composition and diameter, and upon the hydrogen ion concentration of the solution. With the reasonable assumption that all sytem characteristics with a particular rock are constant, then the rate of solution is an inverse function of particle size, a premise that has been demonstrated by many investigators. If a rock particle enters a volume of solution containing on the order of 4-5% free sulfuric acid, the calcium ions in solution may be unable to migrate far enough from the surfaces and pores of the rock particle to avoid being precipitated within and upon the particle surfaces. This blocking phenomenon acts as an effective barrier against further reaction, and phosphate values in the rock are lost in the gypsum cake. Since it is mechanically impossible to instantaneously disperse the sulfuric acid in the circulating solution, in the Kellogg-Lopker system or in any other, time must be allowed for solution of the rock particle before the point of addition of sulfuric acid is reached. This time interval is determined by vessel arrangement and, most importantly, by the recirculation ratio. For a given vessel configuration, then, an exceedingly high recirculation ratio may not allow sufficient time for the rock particles to dissolve and would therefore be a detriment rather than an aid in the overall reaction. Grinding the rock finer would reduce this effect, but would add more rock grinding costs to the costs of maintaining the high recirculation ratio.

In the Kellogg-Lopker reactor system, rock particle size, retention time and recirculation ratio are carefully balanced for each individual rock for optimum process conditions, which also results in optimum process costs. For example, with ion concentrations and temperature varying within extremely narrow limits, the degree of supersaturation of the phosphoric acid in the reactor is held nearly constant and conditions are ideal for rapid reaction of the rock and rapid crystal growth. It is, therefore, no longer necessary to grind the rock to the fineness required by conventional processes. This has been proved in both of the Marchon Kellogg-Lopker plants, where the Moroccan rock is fed into the process at one millimeter. Further, since crystallization proceeds rapidly with limited and controllable nucleation, retention time required to grow the rapid=draining drystals is drastically reduced. And still further, the increased filtration rates that are characteristic of the Kellogg-Lopker process reduce the filtration area required per ton of P_2O_5 produced.

The high recirculation ratios are achieved in the Kellogg-Lopker system through use of a high-volume, low-head pump, which need only over-come pipe friction and entrance and exit losses, since the evaporator vacuum provides the differential slurry height in the vessel arrangement. Power requirements for slurry circulation in the Kellogg-Lopker process are on the order of 10% to 15% of the power required for agitation in a conventional process.

Figure 4 is the flowsheet of a typical Kellogg-Lopker phosphoric acid plant producing 32% P_2O_5 acid from 68% BPL Florida rock. The plant is conveniently divided into rock preparation, reaction and filtration sections.

Laboratory work in a simulator that duplicates plant operation with regard to retention time in the dissolver and evaporator, and to recirculation ratio, showed that rock ground to 95% passing 35 mesh was fine enough for reaching the required system goals of low retention, reasonable recirculation ratio, high filtration rate and high recovery of P_2O_5. Rock preparation for this example, then, consists of screening the raw rock at 35 mesh and reducing the oversize in a simple hammer mill or cage mill in closed circuit.

The ground rock enters the dissolver in the reaction section through a feeder whose output is varied by a signal from a sulfate ion analyzer-controller. It should be noted that this controller is a Kellogg-Lopker development and differs markedly from a Technicon analyzer. The phosphoric acid returning from the filtration section and the rock feed mix with the slurry entering the dissolver from the evaporator. The dissolver is sized for the retention time required for solution of the rock particles. The slurry is then pumped to the evaporator, where sulfuric acid is metered to distributors above the surface of the slurry. The acid stream is divided finely enough to yield good dispersion and mixing with the slurry but not so finely that droplets could become suspended in the vapor stream or that the surface area of the droplets is so great that appreciable quantities of water could be absorbed. In this latter effect, such absorption would effectively result in recycling of heat in the upper portion of the evaporator and could have detrimental effects on the reaction. Vapor velocities are limited by design to eliminate droplet carryover.

Continuous monitoring of the sulfate level in the slurry, for automatic application of corrections to the rock feed rate to maintain a constant sulfate level in the reaction system and constant reaction conditions is only practical with a reasonably high recirculation ratio and controlled flow throughout the system. This is an advantageous feature of the Kellogg-Lopker system that would be difficult to apply to other reaction systems.

A controlled flow of slurry is withdrawn and sent through a surge tank to the filter, where phosphoric acid at 30-32% P_2O_5 is separated from the gypsum crystals with the usual three-stage countercurrent washing method. At this point in processing, the results of the Kellogg-Lopker system become apparent. Although actual production figures and filtration rates in in the Marchon plant must be held confidential, the statement may be made that plant and laboratory testing of the Kellogg-Lopker systems have demonstrated production rates, in terms of P_2O_5 produced per square foot of effective filtration area, considerably above the rates experienced in a conventional system when both are using rock from the same source.

Filtration rates with rocks such as Florida 68% BPL, Florida "black", Florida calcined and North Carolina, as determined in the laboratory, vary from 0.6 to 0.9 tons of P_2O_5 per square foot of filtration area with 95-96% P_2O_5 recovery. As might be expected, the less reactive rocks, such as South African and Western U.S., filtered more slowly, but showed the same recoveries.

Large single-train plants are therefor possible, to produce up to 1,350 tons per day of P_2O_5 with filters now in commercial use and up tp 1,700 tons per day of P_2O_5 with filters now in the design stage.

Economies in plant equipment and arrangement that are possible with the Kellogg-Lopker system are reflected in reduced capital investment per ton of P_2O_5 produced. Economies in plant operation are apparent from the reduction in rock grinding, elimination of agitators, reduction in size and simplification of equipment, freedom from scaling and sludge buildup and reduction in operating manpower. An example of these economies is shown in the following table, which was developed for operation with Florida 68% BPL rock and 77% H_2SO_4 sulfuric acid feeds, in a battery limits plant for production of 1,000 tons per day of P_2O_5 as 32% P_2O_5 acid. Steam ejectors were used for producing the vacuum for the evaporator and filter.

ECONOMICS OF OPERATION

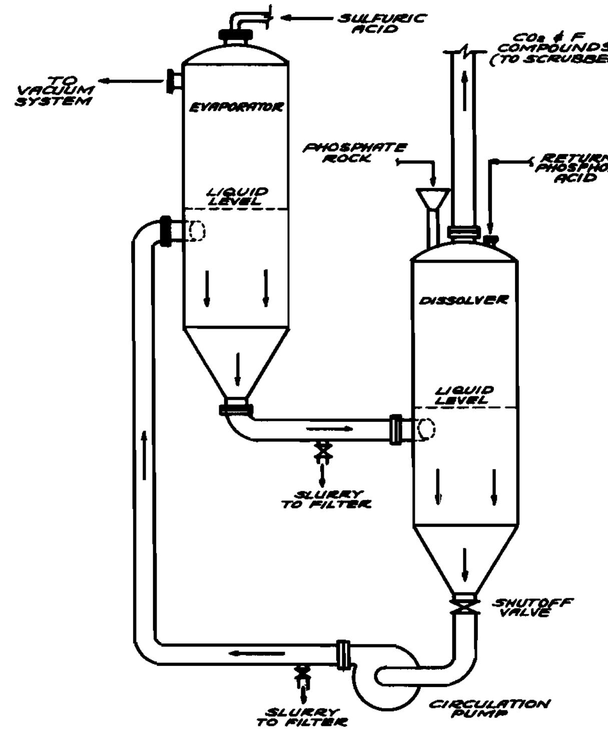
	Kellogg-Lopker	Conventional	Reduction with Kellogg-Lopker
Capital Investment Ratio	0.8	1.00	20%
Operating Manpower, Per Shift	3	4	25%
Maintenance, % of Capital	5%	6%	16%
Utilities, Per Ton of P_2O_5			
Power, KWH	53	145	63%
Process Water, Gal.	1,000	1,000	0%
Cooling Water, Gal.	12,000	12,000	0%
Steam, Lbs.	1,800	1,800	0%

The economics in the Kellogg-Lopker process can be calculated for any set of local conditions, taking into account the reduction of depreciation, taxes and interest, which are related to capital investment, and supervision and labor overhead. The overall reduction in manufacturing usually amounts to about 30%, equivalent to a cost reduction of well over two dollars per ton of P_2O_5.

The advantages of the Kellogg-Lopker process are not confined to operation in new plants, since existing plants can be easily and economically converted to the concepts of the process. The converted plants can enjoy the increased production that is possible with the high filtration rates that are a feature of the Kellogg-Lopker process. The incremental cost of conversion is considerably less than the cost of new construction to produce the same incremental production increase and therefore, the total manufacturing cost per ton of P_2O_5 is reduced.

It should also be noted that the concepts and apparatus of the Kellogg-Lopker process can be applied to production of 40% P_2O_5 and higher phosphoric acid through operation in the hemihydrate mode. Only alterations in the operating parameters are required.

FIGURE 1
THE KELLOGG-LOPKER REACTION SYSTEM



Acknowledgement

The permission given by the Marchon Division of Albright and Wilson, Ltd., for publication of those portions of this paper that concern operation of the Marchon Kellogg-Lopker system, is gratefully acknowledged.

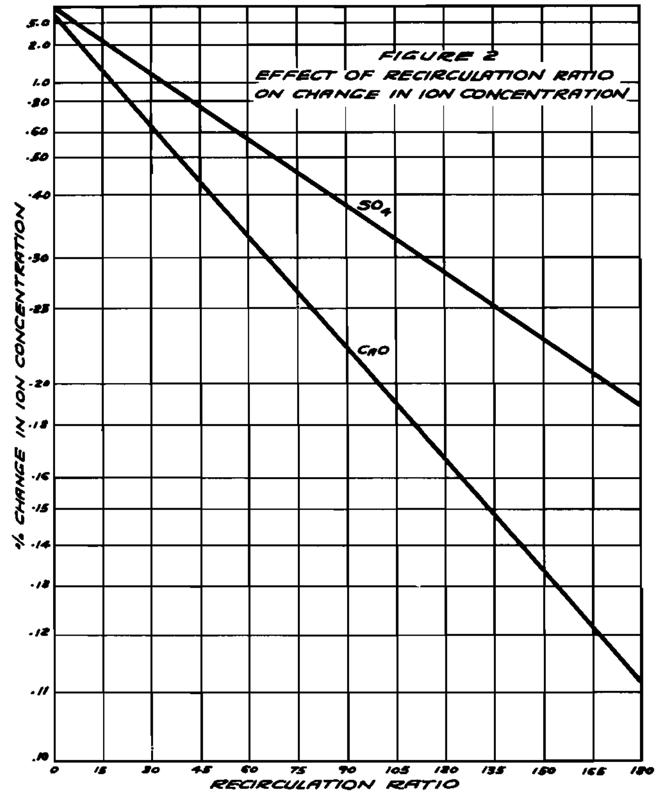


FIGURE 2
EFFECT OF RECIRCULATION RATIO ON CHANGE IN ION CONCENTRATION

MODERATOR SPILLMAN: Thank you. Mr. Turner, for that very fine description of the Kellogg-Lopker Process. Now we are open for questions.

MR. JOHN KRONSEDOR (Fluor Co.): In your description of the process you showed dissolving on one stage and the sulfuric acid addition in another stage. In most digestions you usually run with a sulfuric acid content of, say, 2 per cent to get the minimum amount of phosphate in the gypsum. In that case, in the Lopker process, what's the difference? The rock is dissolved in a medium which already has sufficient sulfuric acid to precipitate all the gypsum.

MR. WILLIAM TURNER: I know it seems hard to follow at times but this is the way it happens. Your figure of about 2 percent sulfuric acid is correct for the liquid portion of the slurry leaving the attack system. Our process differs from others in that changes in calcium and sulfate ion concentrations are very small, are controllable, are predictable and, most important, are separated in time as well as in space. This is the basic concept and the reason why our process works so well.

MODERATOR SPILLMAN: Mr. Turner, the audience may be interested to know the locations of some of these plants here in this country. Can you tell us where they are and if it is possible to get into the plant and see what they're doing?

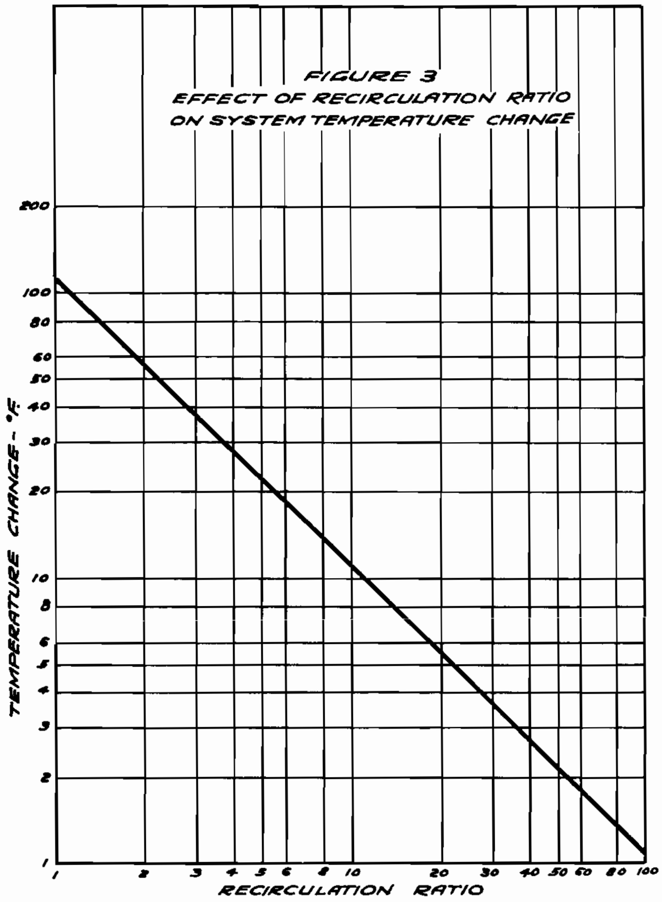


FIGURE 3
EFFECT OF RECIRCULATION RATIO ON SYSTEM TEMPERATURE CHANGE

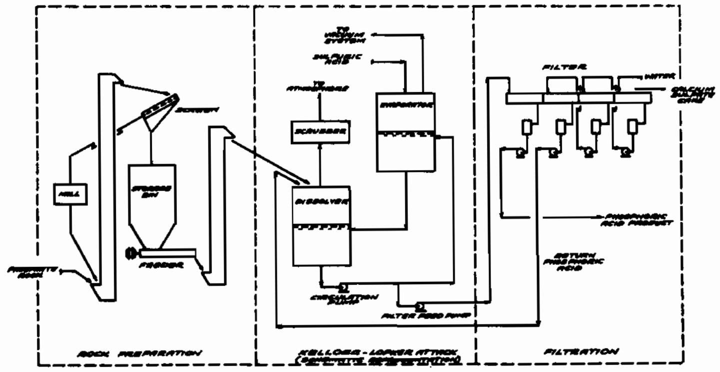


FIGURE 4
PHOSPHORIC ACID PRODUCTION WITH
THE KELLOGG-LOPKER REACTION SYSTEM

MR. TURNER: There are no plants so far in the USA. The only plant which is operating is the plant at Marchon at Whitehaven in England and this plant can be visited by arrangement with Kellogg, but we are very selective in that people have to be really interested in buying a plant before they get to see it.

MODERATOR SPILLMAN: Are there any other questions?

A MEMBER: At what size would you consider making such a plant, for what P_2O_5 production?

MR. TURNER: Any capacity. I'd say from 50 tons of P_2O_5 a day upwards.

SAME MEMBER: Yes, but what for the maximum size?

MR. TURNER: The maximum size of a single stream plant depends on the maximum size of filter that is available. With the largest size filter now in use we would design for just over 1300 tons of P_2O_5 a day. But we unserstand that a larger filter is now being designed, and this would allow us to design for about 1700 tons a day.

SAME MEMBER: So you think you are able to make a dissolver for 1700 tons a day of P_2O_5?

MR. TURNER: It would be possible.

SAME MEMBER: In one piece?

MR. TURNER: Yes.

SAME MEMBER: And what is the pump?

MR. TURNER: Oh, well, we don't go looking for one pump to do the whole job at once. It would be rather a beast.

MR. ROBERT SOMMERVILLE: My question is: What experience are you getting, what sort of scale problems are you running into on the filter? With the short retention time and probably relatively high temperature going to the filter, you would expect at least as severe and maybe a higher scale formation on the filter than with conventional processes.

MR. TURNER: No, this has not been a problem. Any scale which has been produced has been easily washed off the equipment.

MR. SOMERVILLE: What is your operating cycle on the filter? How many days or hours do you run before you shut down and wash the filter?

MR. TURNER: It varies. It's hard to give a number on this because the experience so far has been with belt-type filters, and washing has usually, so far, been done during periods of downtime for routine maintenance.

There's not enough long-term experience but it's been comparable with the conventional plant. It's certainly as good.

MR. SUMMERVILLE: Thank you.

MR. MORTENSON (Swift Agric. Co.): You indicated you had worked with some low-grade rocks. I'm curious to know whether you can operate with a lower amount of defoamer than you would ordinarily use in a conventional process.

MR. TURNER: This varies with the rock. That's all I can say. It's comparable.

MR. MORTENSON: Thank you.

A MEMBER: What type of retention time generally are you concerned with?

MR. TURNER: There isn't a general number. It varies with the individual circumstances. It's a case of balancing the recovery you want and the strength of the sulphoric acid available. the phosphoric acid strength you are trying to produce and the characteristics of the particular rock. There is no magic number.

MODERATOR SPILLMAN: It is my pleasure to introduce Mr. Richard M. Daniel, Senior Chemical Engineer, Day and Zimmerman, Inc. He will discuss "The Hemihydrate Process".

The Hemihydrate Process

Richard M. Daniel

One might ask, "Why the Hemihydrate Process?" Since there may be some confusion as to what this term means we will define it as a technique for manufacturing wet process phosphoric acid by digesting phosphate rock at a temperature which causes the calcium sulfate to form as the hemihydrate. In a subsequent step at a lower temperature the hemihydrate takes on additional water of crystallization to form gypsum which may be separated from the phosphoric acid bu filtration. The technique might be more accurately described as the "Hemihydrate-Dihydrate Process."

To answer the question, "Why?" in one word, it is simple a matter of economics. Japan is a country with insufficient deposits of minable gypsum and without deposits of phosphate rock. The Japanese, a very thrifty people, could not see the logic of importing phosphate rock *and* gypsum when the first in contact with indigenous sulfuric acid produces the latter in quantity. Hence their incentive to invent this process.

In the 1950's, Japanese researchers both in the universities and in industry set about to improve the efficiency of the wet process and to produce a usable gypsum. That they have been successful in achieving this is exemplified by the fact that three competing hemihydrate-dihydrate processes have been developed which not only produce a usable gypsum but also significantly reduce the amount of phosphate rock and gypsum imports. In fact, gypsum imports have been all but eliminated. These are the Nissan, Mitsubishi and NKK processes. Commercial plants utilizing each of the three have been built and are operating in Japan. All are producing usable gypsum. Some is going into wallboard and plaster manufacture and some into cement retarder.

The other objective of a more efficient process has also been achieved. The P_2O_5 recovery has been increased, the electricity consumption has been reduced, on-stream efficiencies have been improved and maintenance costs have been lowered. Detailed descriptions of the three Japanese processes have been published and undoubtedly most of you present are familiar with them. It will be the purpose of this paper to put an economic value on these features and to throw out a challenge to the U.S. phosphoric acid producers and gypsum users to develop an outlet for the millions of tons of chemical gypsum that are now going to waste.

Economic Savings

The improved economy of the process is due to the following: reduced consumption of phosphate rock and sulfuric acid per unit of P_2O_5 produced, reduced consumption of electricity and reduced maintenance costs. The most significant of these improvements is due to the high P_2O_5 recoveries that can be achieved. In all three of the Japanese processes 98 to 99% recoveries are attainable. The term recovery as it is used here means measurable P_2O_5 losses from rock addition through filtration.

It must be remembered that operation of the wet process to achieve the highest possible recovery is not necessarily the most economical. Higher sulfuric acid consumption may offset the economic savings in phosphate rock charged. However, the data indicate that for a comparable sulfuric acid consumption per ton of rock charged, recoveries that can be expected in the hemihydrate-dihydrate process will be 2% greater than in the dihydrate process. Thus in our example, we will compare a 98% with a 96% recovery.

In actual practice the source and BPL content of the rock can have a wide effect on specific operating conditions and economies. Since by far the larger part of our domestic industry is based on Florida rock in the range of 55 to 68% BPL, this discussion assumes that such a rock is the charge. In fact, the Sulphur Institute's Table No. 5 on page 50 of their Bulletin No. 8, 2nd Edition, which gives the estimated production cost of wet process phosphoric acid, is taken more or less as the standard for the dihydrate process. These data show a 95% P_2O_5 recovery overall. Since this figure includes handling, grinding and evaporation, the measured recovery figure comparable to the ones referred to above would therefore be in the 95.5 to 96% range. As stated above, 96% will be used in the comparison.

This improved economy may be expressed several ways. One way is to calculate a percentage reduction in operating costs. However, engineers differ widely as to what is includable in a typical phos acid plant. Some may include the rock grinding, sulfuric acid plant, and the evaporators. Others may exclude one or more of these normally necessary elements. Therefore, we have elected to express savings in total dollars per year for plants of various sizes. The range selected is from 200 to 1,000 short tons per day of P_2O_5 productive capacity. Extrapolation below this range would not be very meaningful and it is doubtful if any one in this country would even consider a plant smaller than 200 STPD. Figure I is presented to show graphically the cumulative values of the annual savings from all sources.

If the P_2O_5 recovery of the hemihydrate-dihydrate process is 2% higher than the dihydrate, then for our typical plant charging 66% BPL Florida rock approximately 2% less rock charge will be required and this saving can be directly calculated in tons per year. To convert this to dollars we have assumed our plant to be in central Florida and the rock cost to be \$7.00 per short ton. Thus the annual savings for a 600 STPD plant will be approximately \$100,000.

One of the basic conditions set forth for this comparison is that the consumption of sulfuric acid per ton of rock charged shall be the same. Thus if less rock is charged, a correspondingly smaller amount of sulfuric acid will be consumed. At a sulfur price of \$38 per long ton (\$34.60 per short ton) the annual savings for a 600 STPD plant will be approximately \$125,000.

Electricity is a major item of overall manufacturing cost. Digester agitators account for nearly half of the power consumed in the digestion through filtration steps. In the hemihydrate processes the large crystallizing tanks need be stirred only to prevent settlement. Thus agitation power requirements are reduced by over two-thirds.

Design data for a 600 STPD hemihydrate plant indicate a total power requirement of approximately 55 KWH per short ton of P_2O_5 produced. Bulletin No. 8 shows a figure of 200 KWH per short ton for a smaller plant which also includes a sulfuric acid plant. No grinding costs are included. In order to calculate savings we have used a power consumption figure of 120 KWH per short ton of P_2O_5 produced. Translated into dollars per year at 7 mils per KWH for a 600 STPD plant the savings are $90,000. In calculating the chart no allowance was made for variation in efficiency with throughput.

The fourth area of saving is in maintenance costs. Early writers on the subject of hemihydrate processes dismissed them on this score because the digestion temperature was in the range of 212 degrees F., which is some thirty degrees higher than the dihydrate process. All previous attempts to operate in this temperature range have come to grief because of the high corrosiveness of the reaction products.

This has not proved to be the case with Japanese hemihydrate processes for two reasons. First, the volume of the vessels in contact with the acids above 200 degrees F. in aggregate is relative small. These vessels can be protected by a synthetic rubber lining applied in the shop and cured in an autoclave for plants of smaller throughput. Premixers and digesters for larger plants can be protected with a carbon brick lining. Both of these designs have proven to be satisfactory. Since the crystallizers operate at temperatures below 150 degrees F., corrosion problems overall are actually less than those of the conventional plant.

The second reason for reduced maintenance is the inherently smoother operation of the two-step system. Once a plant has been started up and leveled out, upsets in the operating system are few. This is true even if there are fluctuations in the type or characteristics of the charge rock. This fact plus the almost complete precipitation of C_aSO_4 as gypsum reduces filter cloth blinding and subsequent evaporator scaling so that higher on-stream efficiencies are achieved.

In some hemihydrate plants a quantitative figure on maintenance as low as 4% on investment has been achieved. Since the lowest figure normally used for dihydrate plants is 6%, it is expected that an improvement of two percentage points can be realized on hemihydrate-dihydrate plants. These percentage figures are based on the capital investment for a phos acid plant that includes digestion, filtration, and concentration. Several studies for large sized hemihydrate plants indicate that at the present state of technological development, there is no essential difference in capital cost between dihydrate plants and Japanese developed plants. Costs used in preparing Figure I are based on data in Bulletin No. 8 referenced above and include an estimated 15% increase in construction costs between 1966 and 1969. Thus we calcualte the annual savings in maintenance for a 600 STPD plant to be approximately $100,000.

The total of the four types of savings mentioned above and shown on Figure I is $400,000 per year for a 600 STPD plant. At 1,000 STPD the savings become $650,000 per year.

At this point we would like to point out that since these processes have been developed in Japan, the emphasis was primarily on a usable byproduct gypsum. The improvements in P_2O_5 recovery, utility consumption, and maintenance were gladly accepted, but the equipment used in their plants has largely been of foreign design. Not only that, but there has been some over-design. What this means is that as more of the plants are built, improvements and economies of design can be achieved which could lower the investment cost below that of conventional dihydrate plants. The incentive to make these improvements will undoubtedly be present on the American scene.

Considering the overall effort of the Japanese to promote their hemihydrate-dihydrate processes, their success in selling the technique has been little short of phenomenal. Within Japan today, no other type of process stands much chance. It is estimated that there are at least 22 plants of this type in operation or under construction in Japan capable of producing a combined total of approximately, 1,600 metric tons per day of P_2O_5. Most of these plants are very small, the largest being slightly over 200 MTPD in capacity.

Outside of Japan the number of these plants built or being built in the last five years is eight. In the last year three have been sold: one in East Pakistan, one in France, and one in Belgium. This is a very creditable showing in a period during which the construction of wet process plants has slowed to a crawl. Table I is a listing of all of these plants.

With the acknowledged success of the Japanese in developing phos acid processes to produce a usable by-product, It has been assumed by many in this country that their recent success in selling the hemihydrate technique in other countries can be attributed to this feature. Although this may have been a factor, it is true that no hemihydrate plant outside of Japan is doing anything other than dumping its gypsum.

In actual practice it is seldom revealed exactly why certain processes are selected by producers in preference to others, but rarely is a decision made without competitive bidding. It would appear from the evidence at hand that recent sales of the hemihydrate-dihydrate plants have been dictated by two prime considerations, both of which have been discussed above. The first is better short- and long-term savings and the second is the reliability of this process in giving consistent savings over a long period of time.

We now come to the seond part of the story which has been entitled: "The Gypsum Challenge."

The Gypsum Challenge

Owing to the over-capacity of the wet-process phosphoric acid industry it is difficult to say how much P_2O_5 is actually being produced in the United States today, but this we do know: approximately 4.5 tons of gypsum are produced for every ton of P_2O_5. A very small portion of this gypsum may be used directly in field where there is a deficiency of sulfur which is required as a trace element. This is true in California where one small phos acid producer sells all of his gypsum to farmers. A plant has recently been announced to make ammonium sulfate fertilizer from natural gypsum in northern California. The present capacity of the industry would indicate that upwards of 25 million tons of gypsum are being sent to the scrap heap every year. Strangely enough, the United States is a have-not nation when it comes to this material. In 1968, we imported 5.8 million tons of it which was 58% of our output of crude gypsum.

Admittedly gypsum is a low cost – high bulk item with high transportation cost. This applies to both the raw material and the finished product. Because of this and the inertia of our own industry, only sporadic attempts have been made to use by-product or chemical gypsum. In this discussion these terms refer to gypsum derived from the manufacture of wet-process phosphoric acid and excluded similar material derived from other industrial sources.

Technologically, the reason for the impasse is that the impurities in chemical gypsum degrade the quality of standard gypsum products to the point where they are either unacceptable to the consumer or non-competitive. The most serious of these problems is caused by the residual P_2O_5. This reduces the structural strength of the building products. Typically, in the dihydrate process residual P_2O_5 ranges from 0.53% to 1.08% whereas hemihydrate gypsum ranges from 0.16% to 0.40%. Extra washing helps to lower the ultimate residual P_2O_5. Other impurities such as fluorine are also reduced. The actual figures for fluorine are from about 0.2% to about 0.8%.

Probably, the greatest obstacle to the development of uses for chemical gypsum is the almost complete lack of ties between the gypsum and the fertilizer industries. This is true not only in this country but also in Japan. Even though the latter country is now using over two-thirds of the chemical gypsum being produced, none of it is being processed by the phos acid manufacturers. The cement, wallboard and plaster industries purchase the chemical gypsum as is and process it entirely in their own plants. Production in 1965 is thought to have been 1,500,000 metric tons. Chemical gypsum from other industrial sources is considered to be 200,000 to 300,000 metric tons. Table II gives a breakdown of production and consumption of all forms of gypsum by Japanese industry for that year.

It is interesting to note here that about half of the gypsum is consumed by the cement industry whereas in the U.S. less than 20% is used that way.

From the American point of view, there is one major unanswered question in connection with the above and that is: “Have the Japanese accepted any degradation in product quality in order to utilize chemical gypsum?” We in the phosphoric acid business are unable to answer this question, but no doubt this could be determined very quickly by submitting samples of Japanese cement and wallboard to U.S. laboratories for testing.

In Australia some thought may have been given to using the gypsum, but decisions to use the hemihydrate process were based on other considerations. As in this country, the existing plants are based on mined gypsum and due to their different characteristics, chemical and mined gypsum cannot be charged interchangeably. Thus the technology for building chemical gypsum plants will have to be obtained from Japanese producers or U.S. producers will have to develop their own.

In Europe the problem of disposing of chemical gypsum is far more acute than it is in this country. Anti-pillution laws and the expense involved in dumping gypsum on the one hand and ever-increasing costs of mining natural gypsum on the other, point to the inevitability of chemical gypsum becoming competitive. Considerable effort has been expended. particularly in the United Kingdom and West Germany, to clean up dihydrate gypsum for use in building materials. Also, successful technology has been developed to produce sulfuric acid and sulfur from gypsum.

In Great Britain Imperial Chemical Industries is producing plaster and plasterboard at Billingham and Severnside. The combined capacity of these plants is reported to be 300,000 tons per year, which is equivalent to the output of a phosphoric acid plant producing 200 tons per day of P_2O_5.

A procedure of considerable current interest is the Giulini Process which was developed in West Germany. A plant has been built in Ludwigshafen which recrystallizes waste chemical gypsum to produce 165 tons per day of either hemilydrate powder or building blocks. Total production cost is reported to be $4.10 per ton and capital investment for a plant to take all by-product gypsum from a phos acid is equal to or higher than the cost for the phos acid plant itself (digestion through evaporation).

According to published information on the Giulini Process, the secret of its success is the formation of alpha crystals of hemihydrate rather than the beta from which results from calcination. The latter form is characterized by small needle-like shapes whereas the former are described as large crystals. The recrystallization process reduces residual P_2O_5 and fluorine to levels in the same range as the by-product gypsum from the Japanese hemihydrate processes.

Although it cannot be concluded from the information at hand that use of hemihydrate process by-product gypsum will eliminate the need for the clean-up procedures being followed by ICI and Giulini, there is a strong probability that costs could be reduced by starting with cleaner gypsum.

It would be well to mention at this point the status of another possible use for gypsum. Two processes have been developed in Europe to make sulfur or sulfuric acid from gypsum. These operations produce cement as a co-product. Elcor Chemical is reported to be using a similar process in West Texas. The recent high price of sulfur stirred considerable interest in this country, but the current depressed market for fertilizers has taken the pressure off sulfur prices. This fact, plus the impending large-scale production of sulfur by a modified Frasch process from West Texas deposits, certainly dims the possibility of disposing of waste gypsum via this route in the immediate future.

Due to the high concentration of U.S. phos acid plants in Florida and the lower Mississippi Valley, the logistics of handling waste gypsum have discouraged any serious interest in upgrading existing dumps or planning new facilities based on the availability of suitable by-product.

Obviously, building products such as wallboard and cement produced in these areas could not compete in price in the midwest or northeast. On the other hand, these products could compete if locally produced in the consuming areas. Thus, the best economics for a combined fertilizer-gypsum by-product operation might possibly entail shipping phosphate rock to fertilizer consuming areas.

Another factor that could become important is appearing now as a small cloud on the horizon. The air pollution emanating from fossil fueled power plants is setting off a major campaign to remove sulfur dioxide from stack gases. Use of low sulfur fuels is one way. Another is to use high sulfur fuels and recover the sulfur as sulfuric acid. The economics of this approach could very well hinge on a large consumer of acid being located nearby. Is there any potential customer larger than the fertilizer industry ?

In conclusion it is superfluous to belabor the point any further. One industry has a surplus of material that is being imported by another. What are we going to do about it? This is the gypsum challenge!

MODERATOR SPILLMAN: Thank you Mr. Daniel. Do we have any questions for that most interesting paper? I will ask you the same question asked Mr. Turner. Some of our members may be interested in going thru some of your plants. Are there any located in the U.S. and Canada?

MR. DANIEL: One is located in Melbourne Australia. I have been through that one. One plant in New Castle, Australia. I have also seen this one. And there is rather a small plant in Ireland. One of our men went there about a month ago. New plants, of course, are being built in France and Belgium, but are not available yet. For those who are interested in seeing the process, in operation, these trips can be arranged.

MODERATOR SPILLMAN: Thank you again, Mr. Daniel for that excellent discussion on "Why Hemihydrate Process"

This concludes our meeting for this morning. The afternoon session will start promptly at 1:30 P.M. Mr. Joseph E. Reynolds will be your Moderator. Thank you. Meeting adjourned 12:00 noon.

FIGURE 1

ANNUAL SAVINGS VS PLANT CAPACITY

HEMIHYDRATE PROCESS OVER DIHYDRATE PROCESS

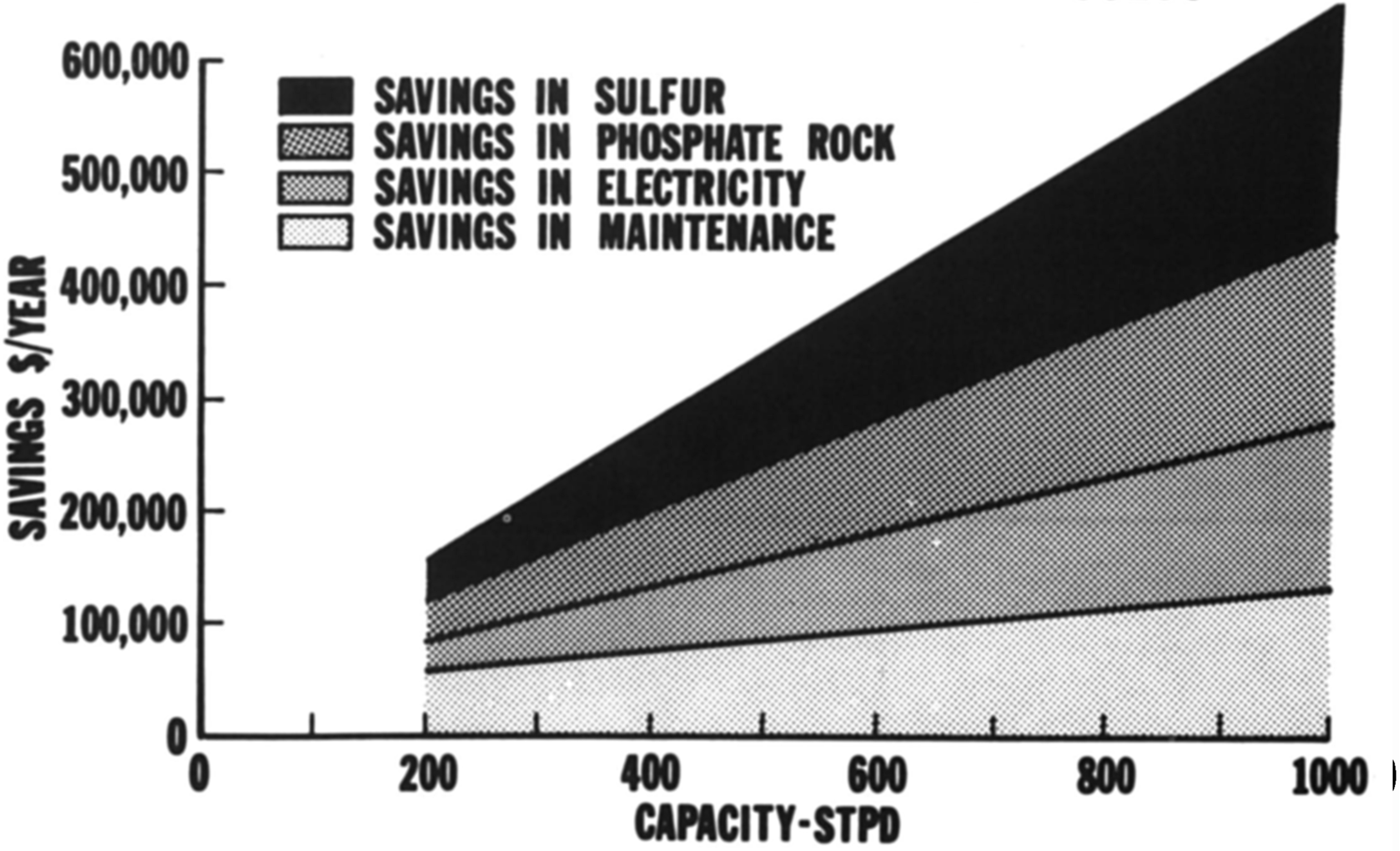


TABLE I
WORLD WIDE LISTING OF HEMIHYDRATE WET PROCESS PLANTS

PROCESS	COMPANY	LOCATION	COUNTRY	CAPACITY MTPD	INITIAL STARTUP
Nissan	Nissan Chemical Industries, Ltd.	Toyama	Japan	110	1955
	Niigata Sulphuric Acid Co., Ltd.	Niigata	Japan	60	1963
	Rasa Industries	Miyako	Japan	66	1963
	Rasa Industries	Miyako	Japan	100	1968
	Nippon Rinsan K. K.	Chiba	Japan	200	1969
	San Chemical Co., Ltd.	Niigata	Japan	230	1970
	ICIANZ	Melbourne	Australia	170	1964
	Greenleaf Fertilizers Ltd.	Newcastle	Australia	100	1966
	ACF & Shirleys Fertilizers Ltd.	Brisbane	Australia	100	1966
	Nitrigin Eireann Teoranta	Arklow	Ireland	60	1966
	Produits et Engrais Chimiques du Rhin	Ottmarsheim	France	150	1970
	East Pakistan Industrial Development Corp.	Chittagong	E. Pakistan	135	1970
	Produits Chimiques et Metallurgiques du Rupel	Sauvegarde	Belgium	300	1970
			Sub-Total	1781	
NKK	Japan Steel & Tube Corp. (NKK)	Koyasu	Japan	30	1961
	Nitto Chemical Co.	Hachinohe	Japan	30	1962
	Nitto Chemical Co.	Hachinohe	Japan	45	1965
	Sumitomo Chemical Co.	Niihama	Japan	35	1964
	Sumitomo Chemical Co.	Niihama	Japan	70	1967
	Mitsui Toatsu Chemicals, Inc.	Omuta	Japan	25	1964
	Mitsui Toatsu Chemicals, Inc.	Omuta	Japan	40	1967
	Kyushu Chemical Co.	Tobata	Japan	25	1964
	Toyo Gas Chemical Co.	Niigata	Japan	35	1966
	Taiwan Fertilizer Co., Ltd.	Kaohsiung	Taiwan	30	1965
	Asahi Glass Co.	Chiba	Japan	70	1969
			Sub-Total	435	
Mitsubishi	Tohoku Hiryo K. K.	Akita	Japan	40	1962
	Tohoku Hiryo K. K.	Akita	Japan	160	1967
	Mitsubishi Chemical Industries, Ltd.	Kurosaki	Japan	80	1963
	Mitsubishi Chemical Industries, Ltd.	Kurosaki	Japan	50	1968
	Nihon Godo Hiryo K. K.	Chiba	Japan	36	1963
	Nihon Suiso Kogyo K. K.	Onahama	Japan	32	1965
	Taki Seihisho	Befu	Japan	25	1966
			Sub-Total	423	
			Total	2639	

TABLE II
CONSUMPTION OF GYPSUM IN JAPAN, 1965

	Chemical Gypsum		Natural Gypsum				Total	
Application	**Metric Tons**	**%**	**Domestic Metric Tons**	**%**	**Imported Metric Tons**	**%**	**Metric Tons**	**%**
Cement	**450,000**	**20.1**	**680,000**	**30.4**	**nil**	**–**	**1,130,000**	**50.5**
Wallboard	**785,000**	**35.1**	**nil**	**–**	**nil**	**–**	**785,000**	**35.1**
Plaster	**290,000**	**13.0**	**nil**	**–**	**nil**	**–**	**290,000**	**13.0**
Plaster of Paris	**nil**	**–**	**nil**	**–**	**32,000**	**1.4**	**32,000**	**1.4**
Total	**1,525,000**	**68.2**	**680,000**	**30.4**	**32,000**	**1.4**	**2,237,000**	**100.0**

Surplus Chemical Gypsum (all sources): approximately 250,000 metric tons.

Wednesday Afternoon Session, November 5, 1969

The Round Table Meeting reconvened at 1:45 o'clock p.m.
Joseph E. Reynolds, Jr., Moderator

MODERATOR REYNOLDS: It is my pleasure to come before you this afternoon to moderate this session. I will try to keep the program on time. We have some interesting speakers who will give you some very helpful information concerning processing. The subjects are not necessarily tied together. They are actually widely diversified, however, are all pertinent to our Industry. We will now discuss the "Paper" Fluid Bed Phosphate Rock Driers and Calciners. I do not recall this subject having ever been presented at previous Round Table Meetings. As we go to increased plant capacities and increased size of operation the available space in the plant becomes very important. Fluid Bed Drying Systems are receiving more attention for all types of drying applications. This afternoon we are very happy to have Mr. D. W. Leyshon and Mr. R. J. Priestley representatives of the Dorr Oliver Company.

Fluid Beds For Phosphates

R. J. Priestley and D. W. Leyshon

Abstract

In only a decade, the fluidized bed reactor has become an accepted processing tool in the preparation of phosphate rock for fertilizer production.

This paper reviews the basic characteristics of fluidized bed drying and calcining of phosphate rock and describes features of these systems in their most advanced form. It reports two very recent phosphate rock processing developments: Beneficiation by fluidized bed sizing in a drying system, and calcining for calcium removal. Additional applications to the fertilizer industry, now under development, are suggested.

Introduction

There are now 30 FluoSolids* systems in 17 installations drying and calcining phosphate rock. If all of these were operating full blast they could dry 65 thousand tons of phosphate rock or concentrate each day, and they could calcine about 20 thousand tons a day.

Also, among the 400 FluoSolids systems are applications involving sizing, lime calcining, pelletizing, and other unit operations which are presently or potentially useful to the fertilizer industry.

Drying

First, let's look at the accepted practice of fluidized bed drying. The basic requirements of a drying system are:

1. Source of heat to vaporize the moisture;
2. Intimate gas-to-solids contact in the presence of the heat; and
3. Air or gas stream to carry the vaporized Moisture away from the solids.

Figure 1 shows the most economical means of meeting these requirements. The means for providing the gas to solids contact is the fluid bed reactor. It is used because of its extremely efficient gas to solids heat transfer rate. In the fluid bed system the source of air is a fluidizing air blower with enough capacity to provide the air required to burn the fuel. This blower must also overcome the pressure losses through the system. The source of heat may be any conventional fuel, but all existing dryers are fired with oil or natural gas. A belt feeder controls the feed rate and the discharge rate is controlled by a discharge valve.

The first such dryer was installed at the Norlyn Plant of International Minerals and Chemicals Company.

Since, that dryer, which is shown in Figure 2, started up 7 years ago, all but 4 of the 17 phosphate dryers that have been built in the United States have been fluid bed systems. The principal reasons for this acceptance are fuel and space efficiency, simple and flexible operation, and low cost.

LOW FUEL REQUIREMENTS

The fuel requirements are low. The quantity of air is minimized by heating it to 1800 degrees F. in order to carry the maximum amount of heat into the unit with each pound of air. Thus less heat is lost up the stack because there is less exhaust gas going to the stack.

Since the fluidized bed exhibits an extremely efficient heat trasfer, it operates at a lower temperature than a rotary, and since no differential between the drying temperature and the exhaust gas temperature is required, the exhaust gas temperature is much lower on this unit. These factors minimize both radiation and convection

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* FluoSolids is a registered trademark of Dorr-Oliver Incorporated describing fluidized bed designs, equipment and systems.

losses. Radiation loss is further reduced by the compact design.

COMPACT DESIGN

The principal drying chamber is a vertical cylinder, so that very little space is required for a FluoSolids dryer. Figure 3 shows a 300 ton per hour drying system. As you can see, the most prominent features are the feed bin at right and the scrubber at left. The dryer itself is the vertical cyclinder in the center, with the air heater extending to the right. Single units of rather gigantic capacity in the range of 500 to 1000 TPH can be built. The compact design also makes possible off-gases very close to saturation with water vapor without condensation in the ducts or cyclone system.

FLEXIBLE OPERATION

Each FluoSolids dryer is extremely flexible in its ability to dry either pebble rock or concentrates or any mixture of these. To accomplish this the reactor uses a fairly deep fluid bed so as to afford enough detention time to drive the moisture from within the pebble out to the surface, where it can be contacted with the gas stream. Moderate changes in size distribution in the feed are automatically compensated for, and wider swings are easily handled by adjustment of a single set point.

OPERATING SIMPLICITY

The FluoSolids dryer is very simple to operate. The major control systems are shown in Fig. 1. The air and fuel rates are set at design conditions to give 1800 degrees F. in the wind box at the design air flow. This establishes the drying capacity of the unit. The feed rate is automatically controlled by sensing the temperature in the bed and transmitting this to a panel in order to maintain a constant drying bed temperature. Therefore, if the moisture of the feed varies, the controller will automatically adjust the belt speed to maintain a constant drying load on the dryer.

LOW COST

Finally, two other important factors are the low cost and the low maintenance of fluid bed systems. These stem from the compact design, the lack of large moving parts, and the avoidance of moving parts in the high-temperature zone.

VARIATIONS FROM STANDARD PRACTICE

In the case of a dryer installed for International Minerals and Chemicals Company at their Kingsford Mine in Florida, electronic instrumentation suitable for computer control was installed. Because this unit is so simple to operate, it requires only the part-time attention of one operator.

Another variation on standard dryer practice has been adopted by Freeport Sulfur Company. Most rock that is shipped any distance is dried at or near the mine for the savings in transportation costs. Freeport, however, established their acid plant in Louisiana, where low-cost natural gas is available. Their rock, therefore, is shipped wet from Florida, and dried at the plant near Baton Rouge. In addition to the reduced fuel cost, this company benefits from the ability to store large quantities of rock in the open at their acid plant.

Drying Costs

Two years ago, when prices were relatively stable, we developed a set of reliable cost figures. Inflation has altered all prices since then, but each plant operator can apply his own factor for the increase.

We assumed the basis outlined in Table A.

TABLE A

BASES OF COST CALCULATION
Plant Requirements

Capacity, wet concentrates	350 TPH
Feed Moisture	12%
Product Moisture	1.5%
Fuel, per ton of wet feed	342,000Btu
Power, per ton of wet feed	1.5 KWH
Operating Labor, per ton of wet feed	0.006 Man-Hr.

Fixed Costs

Maintenance: 2% of Capital Cost Per Year
Depreciation: 15 Year Straight Line

Variable Costs

Fuel	$ 0.40 per Million Btu
Power	0.01 per KWH
Labor	3.75 per Hour

When we assigned economic data to those assumptions we came up with the costs listed in Table B, all based on 1 ton of wet feed.

TABLE B
COSTS OF DRYING
(per ton of wet feed)

Fuel	$ 0.137
Power	0.015
Labor	0.023
Maintenance	0.004
Depreciation	0.012
Interest	0.007
	$0.198

The figure of 19.8c per ton, which is equivalent to 22.5c per ton of dry product, compared very favorably with reported costs of drying by other methods.

Beneficiation By Sizing

These dryers are designed to remove the moisture and recover as much of the rock as possible.

But do you want to recover all of the rock?

At some plants the dryer feed shows a non-uniform distribution of phosphate content in its various size fractions.

Table C is an analysis of the feed to a proposed dryer in Morocco.

TABLE C

annalyses of feed material

Sample	%BPL	% Acid Insol.
+1/4"	24.2	15.2
−1/4"	68.1	3.41
Size Fractions:		
−4 x + 14 Mesh	61.9	3.48
−14 x + 35 Mesh	70.2	2.30
−100 x + 200 Mesh	51.9	17.7
−200 Mesh	29.8	

It shows that the most valuable material lies between 14 and 100 mesh. Obviously, this rock could be beneficiated by size separation.

SIZING BY FLUID BED

The fluidized bed performs a size separation. Fines always leave a fluidized bed reactor with the gas stream. Seven limestone dryers now operating were designed to make a separation at a specified particle size.

Instead of recovering the phosphate particles that leave the dryer with the gas stream, this material is fed into a second fluidized bed to strip off the fines, classifying it at about 140 microns. The flowsheet, much simplified, is shown in Fig. 4.

This circuit has actually been tested, and the results were such that a pilot plant is now being built in Morocco to test this beneficiation on a large scale.

OPERATION OF THE DRYER-SIZER

The dryer is fed a 68 BPL rock from which the +1/4 inch fraction has been scalped. The dryer scrubs the fines out of the bed, and the fluidizing air stream carries them into the exhaust system. This is a 70–30 split, with the bed product at about 71 BPL.

The elutriated fines, which analyze at 63 BPL, are removed from the gas stream by a gas cyclone and fed to a second FluoSolids reactor, where another 70–30 split is made. This time, the exhaust carries a 49 BPL dust which is discarded. The second bed product, which amounts to about 20% of the original dryer feed, is a 71.5 BPL grade.

About three percent of the dryer underflow is in a coarse fraction of only 57 BPL. This fraction is removed by a simple screening at 2.3 millimeters, or 8 mesh.

SCRUBBING ACTION

The scrubbing action of the fluid bed is shown in Figures 5 and 5a. The curves show that the product from the dryer test is slightly finer than the feed, and, with additional scrubbing, the sizer produces a further reduction. The size reductions are not very large, but the results show that it was the fine, low-grade product that was removed.

Note that the fraction between 8 mesh and 100 mesh in the feed contained 69.8 BPL, while the same fraction in the product contained 71.5 BPL.

To summarize we took a −1/4 inch rock containing 68% BPL product with an 89% recovery, and 93.5% BPL recovery.

The operation of the pilot plant now under construction in Morocco will establish the economics of this process. One big advantage of this beneficiation system in North Africa, of course, is the completely waterless operation. Even where water is cheap and available, however, the FluoSolids dryer/sizer combination may provide substantial economies in terms of equipment, power and chemicals.

Calcining

The fluidized bed was applied to calcining even before it was applied to drying, and the development has been similar.

The first FluoroSolids calciners were built in the Idaho-Wyoming phosphate fields, where hydrocarbon content may be as much as 28 gallons per ton of rock. There, well removed from industrial centers (shown in Black in Figure 6), simplicity of operation was second in importance only to organics removal.

Today calcining is an accepted procedure, as Figure 7 demonstrates and it is even being applied to the higher quality Florida rocks. The generalized flowsheet is shown in Fig. 8.

ELIMINATION OF DEFOAMER

Elimination of defoamer in phosphoric acid manufacture is, of course, one of the obvious advantages of calcination. Most Florida phosphate rocks of about 68 BPL take from 3 to 10 pounds of defoamer per ton of P_2O_5. Calcination eliminates this chemical requirement and some of the problems associated with it, such as premature rubber lining failures, poor process control, foaming, spillages, and slurry pumping difficulties.

INCREASED CAPACITY

The effect of using calcined rock upon phosphoric acid plant throughput depends upon many factors. However, in many situations both increased capacity and recoveries can be achieved.

Generally speaking, the calcination of dark phosphate rocks will increase the P_2O_5 throughput of a given filter due to the elimination of organic slimes which restrict filtration and tend to blind filter cloths.

The extent of improvement depends on the particular rock and certain characteristics of the plant. For instance, on one case, in a commercial multi-tank phosphoric acid plant normally processing 68 BPL Florida uncalcined rock, a 24-hour test run was made using rock calcined at 1560 degrees F. in a Dorr-Oliver FluoSolids pilot plant. Using this calcined rock in the phosphoric acid plant, it was possible to increase throughput by approximately 20% due to improved filterability. Recovery was also improved by about 2%, due mostly to an improvement in water soluble losses.

In a second case, we made two similar pilot plant tests, one using uncalcined black rock and the other using the same rock after calcination. As Table D shows, the use of calcined rock in this situation increased the yield by about 1.1% P_2O_5, and substantially higher filtration rates were also achieved.

TABLE D

PHOSPHORIC ACID PILOT PLANT TESTS

Test	Uncalcined		Calcined (1500 degrees F.)
Defoamer	6	lb/ton P_2O_5	None
Filtration	2.0	tons gyp./sq.ft./day	3.2
P_2O_5 Loss	4.2	percent	3.1
Acid Color	Turbid, Dark		Clear Green
Sludge	10%	after 1 yea	Slight

REMOVAL OF ORGANICS

Removing the organics produces a number of benefits.

FILTER CLOTH LASTS LONGER. An average cloth life using 68 BPL Florida dark rock is about 4 weeks. Filter cloth life in calcined rock situations generally is 8 to 12 weeks. For instance, Texas Gulf Sulfur reports an average of 90 days.

RUBBER-LINED VESSELS ARE BETTER PROTECTED. It is rather well known that many expensive rubber lining failures in phosphoric acid plants are due to antifoam reagents. These reagents are abosrbed by the rubber, which then loses its resiliency, becoming gummy and subject to deterioration by abrasion. This had been such a serious problem that antofoam reagents have been formulated specifically to try to minimize these effects on rubber linings. The problem is compounded by the tendency for operators to use too much reagent. Automatic controls using conductivity probes to sense a foam level, or other sensing devices, have been used to control reagent addition. But generally these are difficult to maintain. The permanent solution is to use calcined rock.

THE ACID PRODUCED LOOKS BETTER. There is an ever increasing use of and need for clear acid in liquid fertilizers. It is likely that a certain amount of the acid used for liquids will continue to come by the furnace route. However, it is also likely that expansion needs will be met by wet process from calcined rock. Wet process acid made from calcined rock is a pleasing light green or amber in color.

Super acid made from it has none of the black, tar-like appearance of that made from normal wet acid. Similarly, fertilizers both solid and liquid, made from clear acid, are light in color. In some areas of the world, this appearance is an important factor in solid fertilizer marketing.

SLUDGE FORMATION IS REDUCED. One of the plaguing operating problems of wet acid plants, particularly those producing 54% P_2O_5 acid or super acid, has been the precipitation of sludge during evaporation and storage. It is rather well known that most of this sludge is iron and aluminum phosphate, calcium sulfate, and fluosilicates which are less soluble in 54% P_2O_5 acid than in the disgestion acid. Using calcined rock is not likely to change iron and aluminum solubilities or eliminate post precipitation. However, it will significantly cut down the volume of sludge by eliminating the organic slimes which bulk this sludge. Therefore, calcining reduces the amount of P_2O_5 that is trapped in the sludge and must be rehandled, and which must be worked off in TSP or other materials.

LOWER GRADE ROCK MAY BE USED

The use of lower grade rock, after calcining it, can have great economic and even political significance. In low grade phosphate rocks, the proportion of organics and slimes to P_2O_5 is frequently much higher than in the usual rock of 68 BPL or higher, and there comes a point at which low grade rock becomes uneconomical to handle in a phosphoric acid plant. However, if this rock were calcined it could be treated at a profit. This is likely to be true for some of the large stockiles of low-grade pebble rock located in Florida.

CALCINING TO REPLACE FLOTATION

At the Lee Creek plant of Texas Gulf Sulfur in North Carolina, calcination is being used instead of the second amine flotation step normally used to make phosphate concentrates. The rougher concentrate is calcined and then used locally in T.G.S.'s phosphoric acid plant. Calcining raises the grade about 5 BPL.

They have eliminated the tailings loss that would have occured in the second amine flotation step. Beneficiation processing costs, such as reagents, maintenance and depreciation are, of course, lower. The rougher concentrate at T.G.S. is fine enough after calcination to handle in a phosphoric acid plant without grinding.

In some situations calcination can make possible the use of rock which could not be shipped economically, or rock which has undergone much less beneficiation than normal. The savings in the use of this lower grade rock and/or the higher recoveries in beneficiation can easily amount to between \$0.75 to \$2.00 per ton of rock, which in itself is more than the cost of calcination.

IMPROVED PROCESSING CHARACTERISTICS

Calcining improves the processing characteristics of the rock in several important aspects. One of these is a reduction in grinding requirements.

GRINDING CHARACTERISTICS ARE IMPROVED. Calcination produces a more friable and, of course, a bone dry product. One of the most frequent bottlenecks in the grinding process is moisture in the feed rock. This problem is eliminated in the use of calcined rock. Therefore in most cases less power is required to grind to a given mesh.

In some cases it is even possible to achieve a controlled size reduction of a phosphate concentrate during calcination, which in turn may make possible the use of the calcined rock to make phosphoric acid without any further grinding at all.

UNIFORM PRODUCT QUALITY. This effect shows up in improved process control. Very frequently operators of wet acid plants experience upsets due to changes in the feed rock. These take the form of loss of sulfate control or of foam-overs, frequently due to a change in the organic or CO_2 content of the rock. Fluid bed calcination will produce a uniform feed and eliminate these control problems.

Uniform product quality is a feature of fluid bed calcination which should be stressed. The "Black Art" that has been such a large part of wet acid plant operation can be eliminated!

CRYSTALLIZATION IS IMPROVED. Small amounts of surface active agents improve crystal formation in phosphoric acid production. But in black acid from uncalcined rock, additives of this type are absorbed by the organics in the rock. Calcining overcomes this difficulty. One such reagent, developed by Dorr-Oliver, has been successfully used in several plants to improve gypsum filtration and to reduce water soluble losses. Cost of such treatment is about 5 cents per ton of rock.

Calcining To Eliminate Lime

Now let's look at the most recent development in phosphate rock calcining. We have found that the FluoSolids calcining system can be used to reduce the calcium level efficiently and economically.

Sulfuric acid is used in the phosphoric acid plant to remove the calcium, and free the phosphate radical. But most rock also has calcium carbonate present, either within the crystal lattice or as discrete particles, or it may be present in both forms. Unfortunately, all the calcium is attacked by the acid. So the presence of excess lime in the rock increases the acid consumption.

Therefore, most of the *traded* phosphate rocks have a calcium to phosphate weight ratio of less than about 1.6:1 CaO to P_2O_5.

Where the carbonate is found as discrete particles, the rock can be beneficiated by calcination, slaking and sepration of the fine lime from the coarser mineral particles. In the case of CO_2 substitution for the phosphate within the mineral, the intimate condition of the $CaCO_3$ prevents practical beneficiation. In most cases, however, the amount of calcium carbonate contained within the lattice is limited, and most phosphate minerals that do contain high quantities of CaO can be beneficated to a calcium to phosphate ratio of 1.6 to 1 or less.

Phosphate deposits known to be high in CaO exist in Finland, Mexico, India, and throughout North Africa and the Middle East.

FLUIDIZED-BED LIME CALCINING

In studying this problem, we drew on our experience in other fields.

One of the accepted applications of the fluidized bed reactor is the calcining of various forms of lime. There are 7 FluoSolids systems calcining limestone with a total design capacity of 1140 tons per day, and 14 systems calcining lime mud, a finely divided carbonate produced by kraft pulp mills and water softening plants, with a capacity of 1412 tons a day.

It seems reasonable to apply this technology to a phosphate rock calciner.

A LIME-REMOVAL SYSTEM FOR PHOSPHATES

The phosphate rock calcining system for eliminating lime. shown in Figure 9, is similar to that already described for organic removal, except that the temperature in the calcining compartment is high – 850 degrees to 930 degrees C., depending on the particular phosphates, in order to evolve CO_2. Temperature control is rather critical. CO_2 must be evolved in order for subsequent slaking to occur. However, phosphate minerals soften at temperatures in this range, and therefore excessively high temperatures and over-burning must be avoided. The objective is to produce a reactive lime so that it can be slaked away and a maximum P_2O_5 grade produced. The close temperature control typical of a fluid bed is therefore important. In the fluidized bed of the aftercooler, the particles of rock abrade, and some of the softer lime is removed dry as fines in the cyclone.

The bed product is sent to a slaking station where the lime is extracted, reducing the calcium ratio to 1.6 to 1 or less. The flowsheet pictured in Figure 8 is rather general and would vary according to the requirements of the particular rock.

AN OPERATING LIME-REMOVAL CALCINING SYSTEM

At Djebel Onk in Algeria, the three Dorr-Oliver FluoSolids calciners shown in Figure 10 are in operation with capacity to calcine 3,000 tpd of concentrate to 75 BPL at about 1.60 to 1 CaO to P_2O_5. Rock fed to this beneficiation plant is about 55 BPL with about 1.75: $1CaO:P_2O_5$.

Table E analyzes the concentrate at various points in the process, showing the effective calcium removal. (For this rock a desliming step prior to calcination is used.) The rock is relatively high in alkalies, and a substantial reduction in these contaminants is achieved.

TABLE E

BENEFICIATION OF ALGERIAN PHOSPHATE

	BPL	CO_2	Na_2O+K_2O
Raw Phosphate	55-56	10%	1.5
Deslimed Phosphate	61-63	7-8	–
Calcined Ore	73-74	1.6	–
After Slaking	74	1.5	–
After Drying	75	1.3	0.6

Advantages of Fluidized Bed Calcining

We have seen some of the possibilities of calcining for both organics and lime removal. Now, what does the fluidized bed itself contribute?

LOW OPERATING COST

The first question is always economics. The FluoSolids System is a low cost method of calcining.

This is partly due to the nature of the fluidized bed. The violent agitation and uniform particle distribution create an efficient mechanism for both thermal and chemical activity. Fuel is burned with no localized flame. As a result, temperature is uniform throughout. Only a small excess of air is used. All these factors tend to hold fuel costs down.

In phosphate rock calcining, a further economy results from the utilization of the fuel value in the rock itself. The preheating section does not raise the temperature high enough to drive off the organics. Instead, they are burned in the combustion zone, where their heat can be used. Some of the reactors in the western United Ststes have been operated for significant periods without any fuel at all except the organics in the feed rock.

The reactor is compact and operates essentially as a closed system, so that both radiation and stack losses are minimized.

CALCINING COST CALCULATIONS

At the time we developed the dryer cost figures in Table B, we also worked up some costs for organics removal. They were developed on the same basis, and the same cautions apply.

The fixed and variable costs of Table A were used. The other assumptions are: capacity 3000 tpd product; feed mositure at 12%; power 12 kws per ton of product; labor – one operator and helper; and capital cost $2,100,000. Table F, on this basis, shows a total cost excluding fuel of 42.4 cents per ton. This should be the total cost for calcining providing the raw rock contains sufficient fuel value to be autogenous.

But the gross fuel requirement varies from 900,000 to 1,300,000 btu per ton of product, whereas the net fuel varies from 0 to 1,200,000 btu (the net fuel requirements for western rock is 0 in some cases). North Carolina rock requires 400,000 to 800,000 Btu per ton of product and Florida rock 900,000 to 1,100,000 Btu of product. So we took 500,000 Btu per ton for this example.

TABLE F

COSTS OF CALCINING
(per ton of feed)

Power	0.120
Labor	0.045
Maintenance	0.042
Depreciation	0.142
Interest	0.075
Total Excluding Fuel	$0.424
Fuel (At 500,000 Btu per ton)	0.200
Total Calcining Cost	0.624
Credit for Drying	0.198
Net Cost	$0.426

At this auxiliary heat requirement, fuel would cost 20c per ton of product, making the total calcining cost 62.4c per ton of product. Please note, however, that this calcining cost includes the elimination of up to 12% moisture; subtracting the normal cost of drying, which Table A shows to be 19.8c per ton of product, the net calcining cost is 42.6c per ton of product.

SIMPLICITY OPERATION

The fluidized bed system is simple and easy to operate. Automatic controls can be applied to any degree desired. Control systems are similar to those described for dryers, and even manual operation is simple and reproducibility accurate. This holds down manpower requirements and tends to reduce both the incidence and the effect of human error.

UNIFORM CALCAINED PRODUCT

The FluoSolids system tends to compensate automatically for variations in feed quality, and operating conditions are easily adjusted.

In the fluid bed, for instance, you can change the detention time without varying the capacity, or adjust the capacity without affecting the detention time. Either can be set to any point within fairly broad limits. Fifty percent of the design level is a reasonable range.

The result of this versatility is a consistent and uniform calciner product – and, therefore, uniform characteristics in subsequent processing despite variations in the feed.

PRECISE TEMPERATURE CONTROL

A unique feature of the fluidized bed is the precise temperature control available.

With other types of calciners, even the most accurate instrumentation can only control the temperature in the immediate vicinity of the sensor. The material being calcined is actually exposed to temperature conditions that vary over a broad range.

A fluidized bed, however, shows a uniform temperature throughout the calcining zone to within ± 10 degrees F. All the material in the bed is being calcined under nearly identical conditions. This permits you to adjust those conditions to accomplish precisely the results you want.

SCRUBBING ACTION

Another valuable property inherent in the fluidized bed is a scrubbing action that removes the fines which would otherwise cling to the coarser particles. Combined with the elutriating action phenomenon can be used to upgrade the rock. This is valuable where the fines consist mainly of clay or other low-grade material. A calciner now being built in Morocco was designed to take advantage of this effect.

Future Applications

The technology of fluidized bed processing is accepted, but by no means mature, even as applied to the fertilizer industry. We have shown two basic applications to phosphate rock, drying and calcining, and have described a recent development in connection with each: sizing and lime removal.

Now we would like to conclude with a few suggestions for the future.

BENEFICIATION WITH WEAK ACID

Since most calcining systems include an aftercooler, it is logical that the water spray could be replaced with a weak phosphoric acid spray as shown in Fig. 11 to upgrade the product.

As the world's supplies of high grade rock diminish, the differential in price between 70 BPL rock and 80 BPL rock tends to increase. AT the present time, depending on the source of the rock, this last 10 BPL carries a value of about \$65 to \$85 per ton on P_2O_5. This is based on the differential in sell price per unit of P_2O_5 between a high grade rock and a low grade material. Where low grade rock occurs and where sulfur is reasonably cheap, 30% phosphoric acid can be produced for less than this figure. The hot phosphate being fed to the aftercooler contains sufficient heat to evaporate the water contained in the 30% acid, with the result that 10% to 15% of the P_2O_5 in the final product may come from the acid. By this combination of calcination and use of phosphoric acid in the cooling step, rock can be produced that is 10 to 12 BPL higher than the feed. In addition, it should be possible to produce a rock with a CaO to P_2O_5 weight ratio in area of 1.35 to 1.

CONCENTRATE BLENDING

Another idea is to blend the calcine with an uncalcined concentrate.

In some situations a blend of calcined and uncalcined rock can be considered satisfactory if the objective of the calcination is to lower the foaming properties to a manageable level in the subsequent use of the rock for phosphoric acid or superphosphate.

Fig. 12 shows the wet feed rock split between the calciner and the aftercooler where the moisture in the feed rock is used to cool the calcine. Mixtures of up to about 50:50 by weight, are possible, depending on the moisture content of the feed. The combination of a FluoSolids calciner and a FluoSolids aftercooler is ideal because the units are conveniently situated for feeding from a common bin. Also, the fluid bed aftercooler serves as a blending device. Since no fuel is required for drying a substantial quantity of the final blended product, cost of production is only slightly higher than the normal cost to dry the rock.

PELLETIZED FERTILIZER

As a final suggestion for future development, let's look at the use of the fluidized bed to produce pelletized fertilizer.

There are several significant commercial applications of Dorr-Oliver FluoSolids technology outside the fertilizer industry where products are made in pelletized form.

One example is lime mud calcination in connection with recausticizing systems for the kraft pulping industry, or for regenerating lime from water softening sludge. There are installations of such systems producing a granular product shown in Fig. 13, which is essentially −6 +20 mesh in size. Another example of fluid bed granulation is in the combustion of spent liquor in the pulp and paper industry. In this process a fine granular product generally in excess of 90% Na_2SO_4 is produced.

It is suggested that such techniques could be applied to the production of fertilizers, such as MAP. This could be done by the process sketched in Fig. 14. The expected advantages over the MAP processes include the promise of particle size control. These processes make a rather fine product which is not suitable for bulk blending. Pelletized fluid bed products which is not suitable for bulk blending. Pelletized fluid bed products on the other hand, are generally dense, compact and uniform in size.

Normally the heat of reaction between the ammonia and the phosphoric acid feeds is enough to generate autogenous drying of MAP. However, in the event that only lower strength acid is available, the FluoSolids System offers an option of using fuel.

Other fertilizer products such as 1 - 1 - 0 grades, ammonium nitrate, urea, and ammonium sulfate are also possible candidates for fluid bed processing.

(See Figures 1 thru 14, beginning next page)

MODERATOR REYNOLDS: Thank you very much. We have time for questions.

ROBERT L. SOMMERVILLE: I have observed on one occasion, at least, distinct beneficiation of rock where iron and aluminium were eliminated by getting the fines out, so I think this works well. I have a couple of questions, however. In calcining the western rocks with high organic content, we have observed an awful lot of carbon and there seems to be a great deal of coking going on and the net result is the rock comes out as a gray color whereas in truth it's a light cream if you get the carbon out.

This carbon is probably the leading factor whcih contributes to or which causes the need for separan in the digesters or on the filter.

The other factor from this reducing atmosphere that we get, reducing condition, is that iron gets converted to iron sulfide in there and we get a lot of hydrogen sulfide generation in the digesters and I wonder if you have had any experience in eliminating either of those problems?

MR. LEYSHON: We could mention something with respect to the use of separan, for example, as a flocculant for gypsum filtration. I had never associated this really with the carbon char that's left in the phosphate. I think maybe it has become used because of the particular shape of the

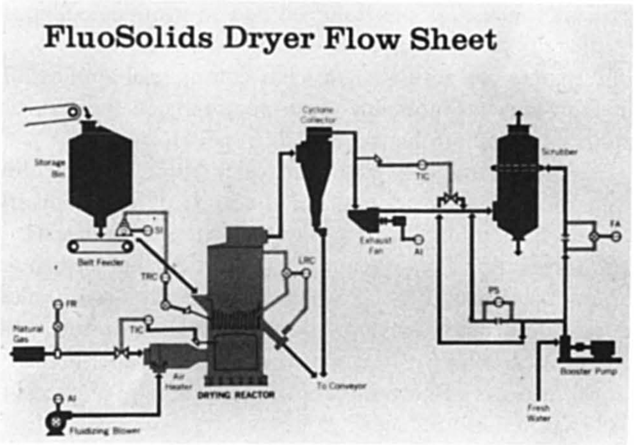


Figure 1
DORR-OLIVER FLUOSOLIDS
PHOSPHATE ROCK DRYER FLOWSHEET

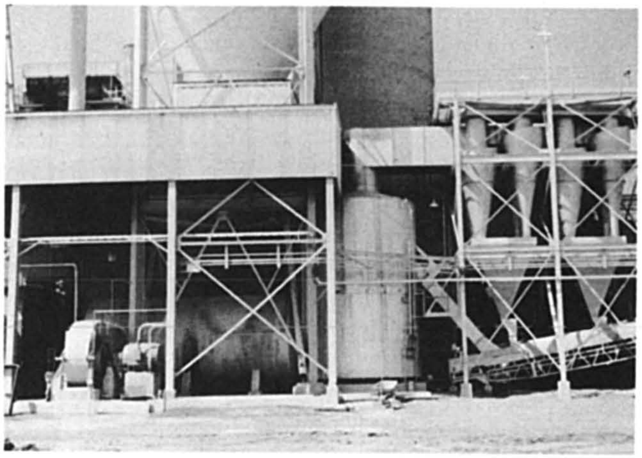


Figure 2
PHOSPHATE ROCK DRYER
AT THE NORELYN PLANT OF
INTERNATIONAL MINERALS
AND CHEMICALS COMPANY

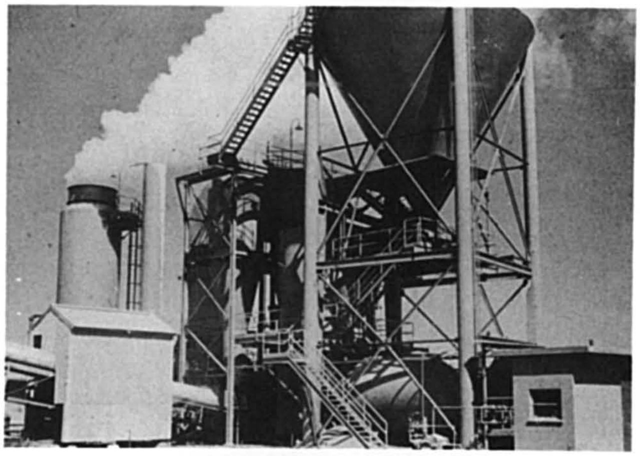


Figure 3
300 TPH FLUOSOLIDS PHOSPHATE
ROCK DRYING PLANT

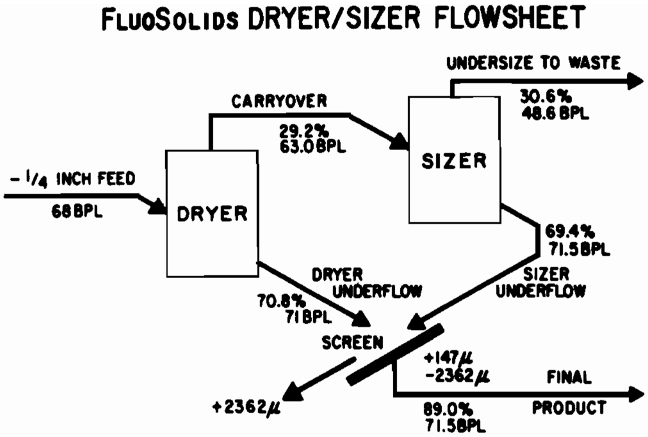


Figure 4
FLUOSOLIDS DRYER/SIZER FLOWSHEET

gypsum made from western rock.

I know that not all plants find it necessary to use separan on calcined western rock. With respect to the sulfide sulfur. there is a conversion of sulfur from perhaps an organic form in some cases, perhaps a mineral form too, to both an oxydized and a reduced form. I think on the surface of the particles you get a conversion to sulfate so that actually the total sulfur in a feed rock is partly converted to sulfate sulfur and to sulfide sulfur in the product. There are techniques for minimizing this sulfide sulfur production and actually you get some acid requirement reduction in the rock from converting sulfide sulfur to sulfate.

The problem is partly due to short-circuiting and can be eliminated and actually has been. So far as I know, the requirements or the specification for sulfide sulfur is usually something like a tenth of a per cent and it's quite easy to obtain much lower values than that.

MR. PRIESTLEY: I might add that in cases where sulfide sulfur is a problem it usually can be eliminated by increasing detention time and/or excess air and possibly adjusting the temperature of calcination. Also in one case it was found that the calcine compartment temperature

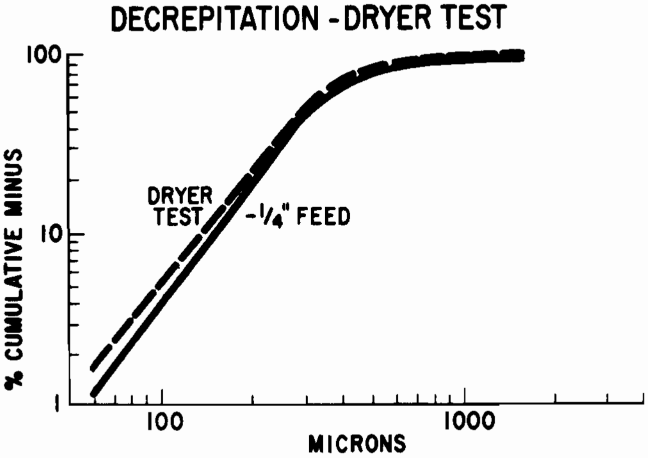


Figure 5
COMPARISON OF FEED SIZE
AND PRODUCT FROM DRYING TEST

controller, which controls the amount of fuel, was overshooting momentarily and putting more fuel into the unit than could be burned efficiently. This resulted in a temporary reducing condition which caused some sulfide formation. This condition was corrected by readjusting the instrument so as to limit the total amount of fuel injected. In rotary calciners there is a greater tendency for sulfide formation because the entire charge is not exposed to the oxidizing atmosphere.

MR HIGNETT (T.V.A.): I would like to ask 2 questions, First, What are the particle size requirements for use in a fluidized bed and what range of particle size can be used and how large or how small? Second, you mentioned the possibility of beneficiating calcined rock by removal of lime after calcining by slaking. Do you have any indication that magnesium can also be removed by that method?

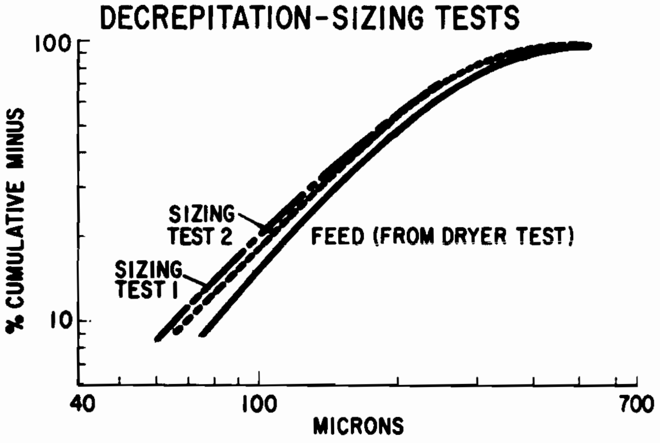


Figure 5A
COMPARISON OF FEED SIZE
AND PRODUCT SIZE FROM
SIZING TESTS

MR. PRIESTLEY: I will answer the first question Dave, and you can answer the second. The size of the particle used in a fluidized bed depends to a large extent upon what you are doing in the bed. If you are carrying out a simple drying operation, you can where the detention time is relatively low, use almost any size particle. We have dried coal, for instance, which is as big as an inch-and-a-half in size down to zero. It is necessary to have a range of particle sizes in order to get decent fluidization. We have no limit on the bottom size of the material to be fluidized.

MR. LEYSHON: In phosphate rock, would you say this is general.

MR. PRIESTLEY: In phosphate rock calcining we normally go minus four mesh and in drying we can go up to an inch by zero.

MR. LEYSHON: To attempt to answer your question on magnesia, we don't have a lot of experience on this subject. However, I would expect that the same problem exists with magnesia as exists with lime. If the magnesia is present in the matrix, in the lattice of the crystal, it's going to be darned difficult to get out. If it's present as discrete particles of dolomite I think it will decrepitate in the slaking operation and can be removed by size separation.

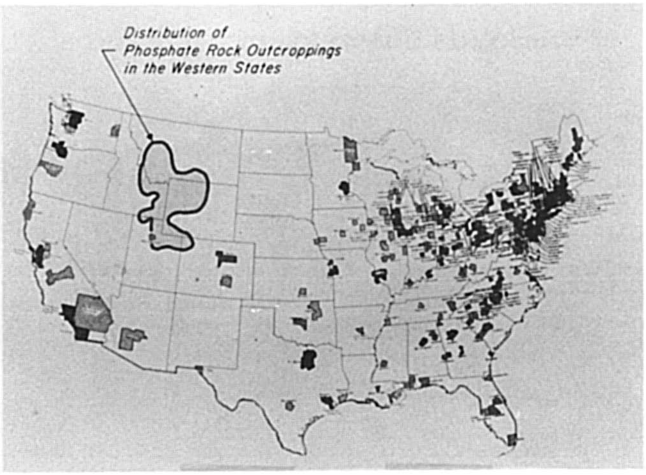


Figure 6
LOCATION PHOSPHATE ROCK DEPOSITS
IN WESTERN UNITED STATES

MODERATOR REYNOLDS: Do you have any cost figures to relate the cost of the rock that has been calcined into a unit of P_2O_5 cost in phosphoric acid?

MR. PRIESTLEY: We have given costs for calcining phosphate rock. These would generally be in the 40 to 60 cents per ton of rock range. Now, that means it's around $1.50 per ton of P_2O_5, roughly speaking, on a 33 per cent rock.

MR. PAUL POYNER (Continental Oil Company): I'd like to ask very briefly. You mentioned at the end of your talk your pilot plant facilities. What size unit did you say that you have?

MR. PRIESTLEY: The largest size unit we have is 4 foot in diameter.

MR. POYNER: Four foot in diameter?

MR. PRIESTLEY: Yes, two compartments.

MR. POYNER: Out of curiosity, concerning your pressure drops throughout these units when drying or calcining rock, what percentage of pressure drop is developed across the grid?

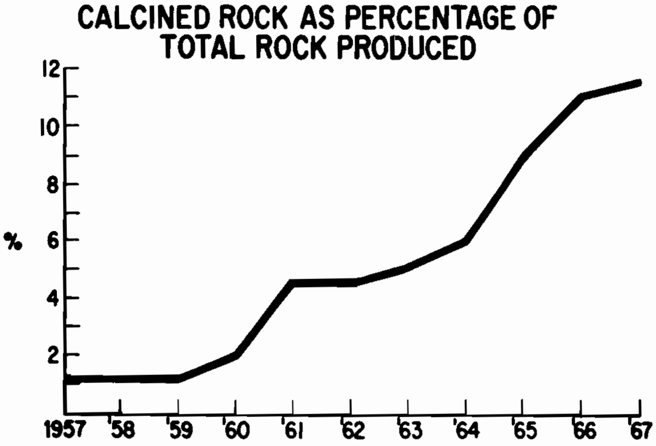


Figure 7
THE GROWTH IN PHOSPHATE
ROCK CALCINATION CAPACITY

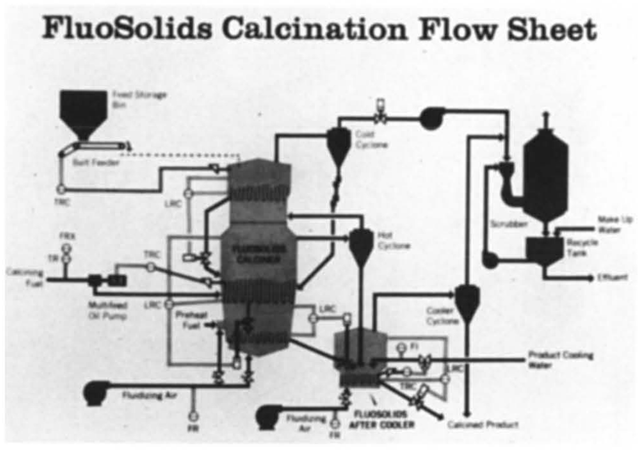


Figure 8
FLOWSHEET OF FLUOSOLIDS PHOSPHATE
ROCK CALCINING SYSTEM

MR. PRIESTLEY: It depends on what you are accomplishing and how big your vessel is in diameter. There is no stock answer that we can use to give you for that.

MR. POYNER: If you're trying to accomplish, let's say, calcining of phosphate rock with a four-foot diameter bed?

MR. PRIESTLEY: I'd say roughly 25 per cent of that pressure drop is taken through the grid.

MR. POYNER: Thank you very much.

MODERATOR REYNOLDS: Fine. Thank you, gentlemen.

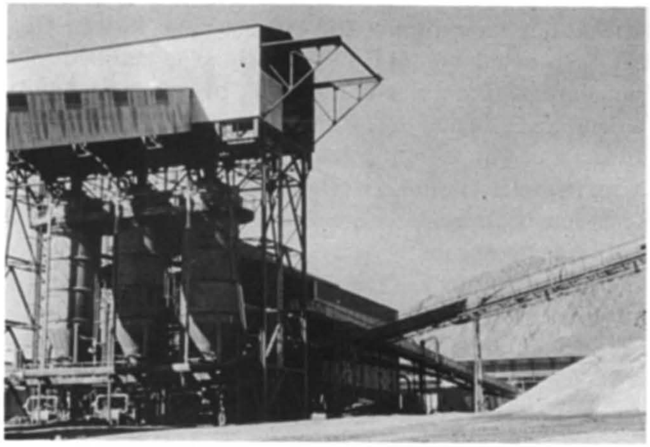


Figure 10
DORR-OLIVER FLUOSOLIDS
PHOSPHATE ROCK CALCINERS
AT DJEBEL ONK IN ALGERIA

MODERATOR REYNOLDS: Our next subject relates to sulfur and sulfuric acid. In recent years there has been considerable concern for the possibility of sulfur shortages and during a time like this we reactivate the studies and investigations that we have been looking into for other possible sources.

Our next speaker is going to discuss Sulphuric Acid from Volcanic Sulfur Sources. It gives me great pleasure to introduce Mr. Joseph L. Prosser of The Prosser Company.

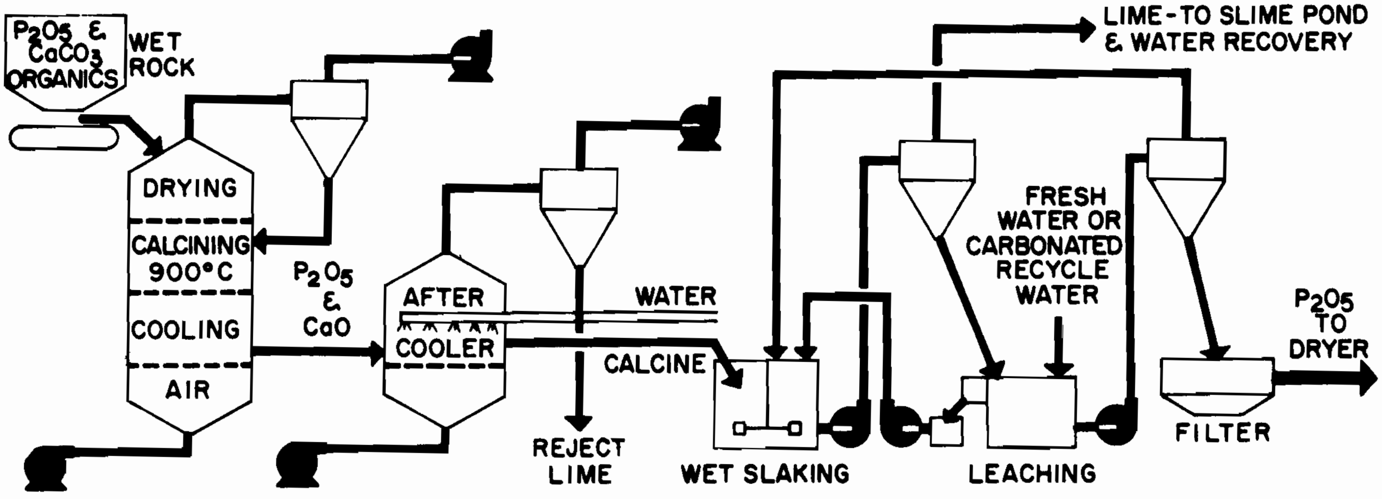


FIGURE 9
CALCINATION AND SLAKING SYSTEM
FOR LIME REMOVAL

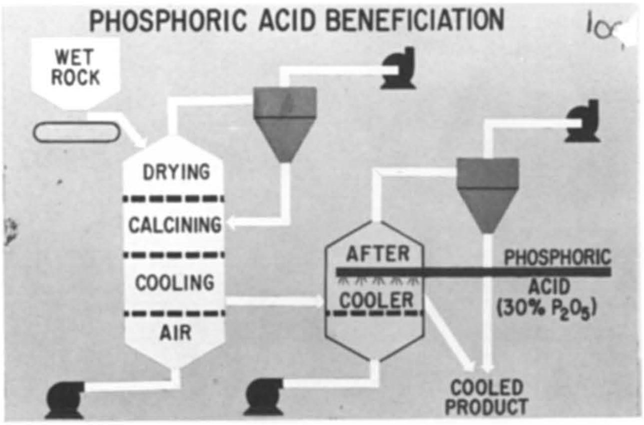


Figure 11
BENEFICIATION OF CALCINED
ROCK WITH WEAK ACID

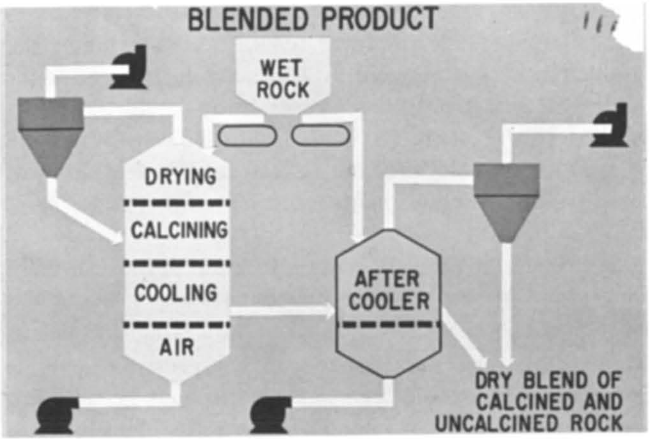


Figure 12
PROCESS FOR BLENDING CALCINED
AND UNCALCINED ROCK

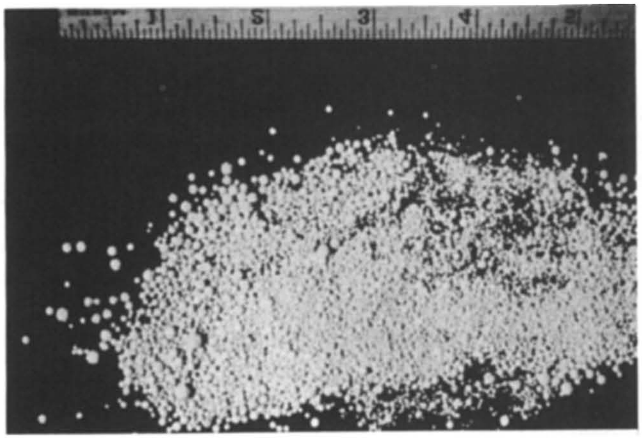


Figure 13
LIME PELLETS PRODUCED FROM
FLUOSOLIDS LIME MUD REBURNER

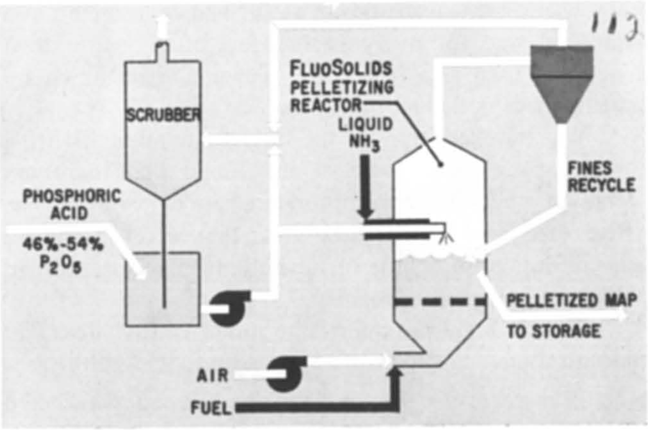


Figure 14
FLUID BED PRODUCTION OF MAP

Sulphuric Acid From Volcanic Sulfur Sources

Joseph L. Prosser

This talk is probably not as appropriate as it would have been two or three years ago. Then there was an acute shortage of sulfur in the world. Today that shortage is, at least for the present, a thing of the past.

However, alleviation of the sulfur shortage is not the whole answer for much of the world. There are very few countries that possess proven deposits of sulfur that can be mined to produce the 99½% sulfur that is required for use in the least expensive acid producing plants. These *have not* countries currently must buy sulfur from a few fortunate *have* countries. This is the case in many of the so-called developing countries. Such purchases mean increased balance of payment problems and absolute dependency upon availability.

At the same time, many of these countries have a possible source of useful sulfur. This source is volcanic sulfur, which occurs as an intruded material into the native rock. Such intrusion occurred ages ago, caused by the extreme pressure of the molten sulfur. The resultant ore might vary from fairly high to very low sulfur content. Since it is volcanic in origin it normally is found in mountainous areas. This has been one of the real problems with its use. It is costly to transport such low grade ore, especially from a mountainous area.

Volcanic Sulphur Ore
Mined in Mountainous Areas

Chile has made use of this volcanic sulfur for years. It is a common source of sulfur for all needs, including the production of sulfuric acid. In Chile the deposits of sulfur occur at very high altitudes – generally in the northern part of the country along its border with Bolivia. Mines are being worked at elevations of at least 15,000 feet. In most cases the beneficiation plants are at some lower elevation – say 12,000 feet.

Because of the very remote location at most of these mines, the Chileans up-grade the ore to nearly 100% by the use of auto-claves. These auto-claves are rather inefficient so far as recovery is concerned and are espensive to operate but they produce the high grade sulfur that permits low cost transportation per pound of pure sulfur. This low cost transportation is essential in the remote Andes locations of the mines.

Two or three years ago we ran into a similar situation in Ecuador. Frasch sulfur was scarce – almost not available. It was expensive. There was a place in the Andes (at 10,000 feet) where small quantities of sulfur had been mined in a haphazard way for many years. Most of the sulfur then mined was used as a bleach for "Panama" hats which are actually made in Ecuador not Panama.

We travelled up to the mine site along with a consulting economic geologist and found that there was literally a mountain of sulfur ore in a base of geyserite. The natives had only mined those areas that were 3" and 4" veins of pure sulfur. Quite fortunately, the national railroad passed the edge of the property.

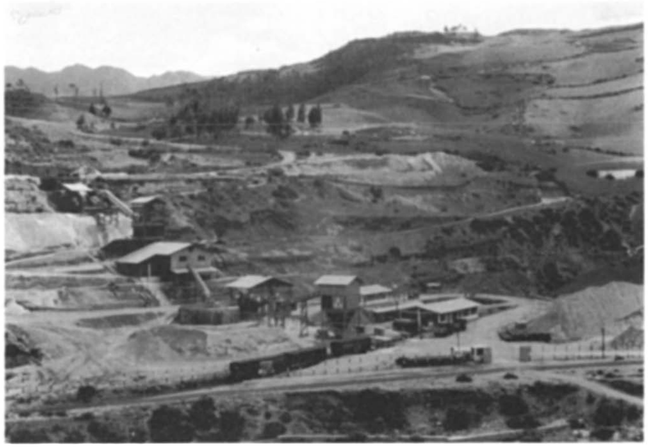
The ore body was quite large and of widely divergent concentration – ranging from 5 – 6% up to 25% sulfur.

Because of the proximity of the railroad, we elected not to use auto-claves but to concentrate to something in the range of 50% for rail transportation economies. This concentration is accomplished in a typical flotation plant which we erected adjacent to the open-pit mine and with a railroad spur serving it.

The erection of this mill was relatively difficult, since among other things it included the installation of a small hydro-electric plant with more than a mile of feed canal. Most of the equipment for the grinding and flotation unit was furnished by the Denver Equipment Company of Denver, Colorado.

In the mill the ore is concentrated from an average of 15% to an average of 50% - 60%. It is then loaded into rail cars or trucks for the 100 mile trip to the seaport city of Guayaguil. The cars reach this plant by means of a car ferry. We realize that this all sounds involved but the delivered cost at the plant is just $36.00 per metric ton of Sulfur. This is quite competitive in today's market. When the kinks are ironed out this cost of Sulfur will be reduced to $32.00 per metric ton. This includes rapid depreciation, interest and tolling charge.

But the problems to not end here. Conversion of this low grade ore into sulfuric acid poses some problems. We enlisted the aid of Hugo Peterson of Weisbaden, West Germany. This company has had years of experience in making acid from pyrites, refinery sour gas, and, of interest to us, volcanic sulfur. In fact their only business is the design of sulfuric acid plants and they have never designed one using Frasch sulfur as the raw material.

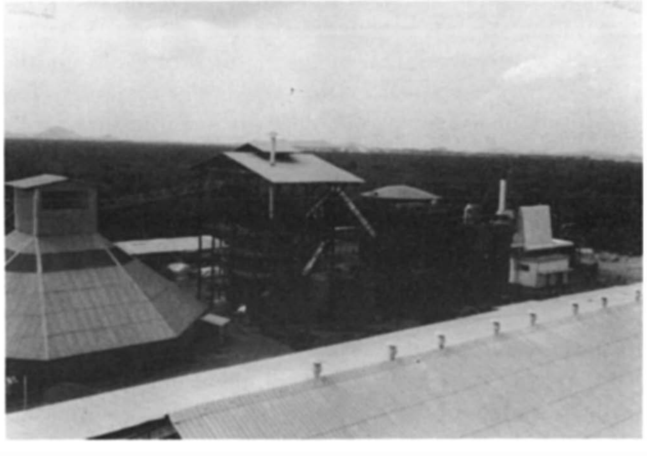


Mill where ore is concentrated and loaded in cars and trucks for shipment to seaport city of Guayaguil.

Dr. Gerd Petersen, son of the founder and present director of the company, designed the plant. He incorporated a seven tier rabble arm roaster similar to a Herreshoff furnace to produce the SO_2. This SO_2 contains fairly large volumes of entrained solids which must be removed before the gas reaches the converter. Removal is accomplished by passing the gas first through a bank of cyclones then through 2 wash towers and finally through a high pressure drop impingement type scrubber designed and patented by Petersen. This impingement scrubber is used in lieu of the normal electrostatic precipitator located at this point in the flow.

From the cleaning system the gas passes on to the drying tower then to a blower and then to the converter group where it is heated up prior to conversion to SO_3. The clean SO_2 delivered to the converter group is very cool – in the range of 150 degrees F. The temperature increase is accomplished in a series of heat exchangers whose source of heat is the exothermic reaction of SO_2 into SO_3.

After being elevated in temperature the SO_2 is passed over 3 stages of special vanadium catalyst being converted in the process to SO_3. Conversion rates are extremely good – normally in excess of 98%. The SO_3 gas is then cooled



Herreshoff Furnace and
Sulfuric Acid Plant

through a so-called hot gas cooler and passed to the absorption tower where it is converted to H_2SO_4 as 98% acid. The plant has a production capacity of 100 metric tons per day with a guaranteed operating range of from 60 tons per day to 120 tons per day.

In addition to the product acid the plant is equipped to make the small quantities of battery acid required for local consumption in Ecuador. This is handled in glass and glass-lined equipment and is stored in a heresite lined tank. The battery acid is diluted and packaged in car boys right in the plant for distribution throughout the country.

Also there is a little bit of sludge acid available for use coming off the 2 wash towers. This acid is very low analysis – testing only some 5% to 10% acid and is loaded with impurities. It is useful, however, in the fertilizer granulation process at the plant.

No economical use has been found for the large quantities of ash from the ore which is removed from the Herreshoff furnace. At the present time this ash is being wasted but it has been found useful for the construction of dikes around the gypsum pond of the phosphoric acid plant.

In order to utilize the sulfuric acid for the agriculture of the country the plant converts a good deal of the acid to phosphoric acid to produce ammonium phosphate sulfate fertilizers.

The source of ammonia for the plant (and for the country) is a 12,000 tons capacity anhydrous ammonia storage tank. This tank is supplied with ammonia by W. R. Grace & Co., from their Trinidad works.

In addition to the plant food products, sulfur is furnished for other industries. For this purpose a single auto-clave has been set up at the Guayaquil complex for the production of pure sulfur. This unit produces the few hundred tons of sulfur used annually in the sugar mills for production of "Sugarmill White" sugar, or to be used as a bleach in the local paper mill and by the aforementioned panama hat industry.

MR. CAMPBELL (Fertila): What capacity does the mine have? How much sulfur are you moving for that 34 a ton?

MR. PROSSER: At the present time we are processing about 300 tons of ore per day producing about 40 tons of S.

MODERATOR REYNOLDS: Thank you very much Joe. Our next talk will relate to granulation or compacting. We have had many papers in past years relating to granulation processes and particle size buildup, however, to my knowledge we have never had a discussion on compaction relating to processes for building up particle sizes for N. P. K. Today we have such a paper. There are two speakers, Mr. J.J. Boyce and Mr. J. R. Carney of the Komarek-Greaves and Company.

Compaction—Granulation Of Fertilizer Materials

J. J. Boyce and J. R. Carney

Granulation of fertilizers has been done by various methods for a long time. Granulation to most fertilizer people means agglomeration in that you are attempting to increase the particle size. This is normally done in the drum granulator or by prilling both for straight agglomeration and to complete or carry out a chemical reaction.

At this time we wish to explain the terms "compaction-granulation" as applied in this paper. "Compaction" is the agglomeration of finely divided materials, into a dense, continuous sheet; "granulation" is the subsequent size reduction operation. The raw materials are blended or mixed in the proper proportions for a specific NPK content. Here the smaller the particle size of the raw materials, the more homogeneous the final mixture. This mixture is then compacted into a sheet followed by grinding or granulation and screening to separate out the desired final particle size.

This procedure for producing a mixed analysis fertilizer is not followed at the present time in the fertilizer industry. However, some of your raw materials are compacted and granulated before shipment to you. Examples of compacted and granulated single analysis raw materials which you are accustomed to using are the potash salts, potassium chloride and potassium sulfate, also trace elements including the carriers for these trace elements which are pulp mill by-products and ceramic frits. Compaction and granulation is also used for carriers for pesticides and pesticide formulations.

Compaction and granulation can produce a granulator product from single materials such as urea, MAP, DAP, and others, or mixed and complete fertilizers in whatever combinations are desired. Compaction and granulation offers to the fertilizer producer advantages and a versatility of operation that is not abtainable under present blending and manufacturing methods.

(1) Large inventories of finished products in all combinations are not required. The fertilizer producer makes his estimate for the total sale of NPK materials needed then purchases these raw materials.

The system is easily and quickly changed from one NPK combination to another.

(2) The production rates are high, as well as will be detailed later. One compacter is rated at 20 tons of product per hour. This means that a trailer truck can be loaded out each hour, a 40 ton box car in 2 hours or an 80 Hopper car in four hours. All directly from raw material storage.

(3) Capital outlays and floor area required for production equipment per ton per day of production is comparatively small.

(4) The compaction-granulation system lends itself well to a completely automated operation. Therefore, production rates and product quality are not dependent on the experience and skill of the operator.

All of you are familiar with the mixers, granulators and screens involved in the compaction granulation process. However, the compacter would be a new piece of equipment and this will be explained before we get into processes and costs.

This is a cutaway view of a typical compacter or briguet machine. K-G machines are readily convertible from one operation to the other. However, we are concerned with smooth or corrugated roll operation in this application. The fine, blended fertilizer materials are fed to the machine hopper and, depending on the roll width, a single or multiple screw feeder starts the application of pressure to force the material into the nip of the rolls. At this point the hydraulic system on the rolls applies up to 300 tons of total pressure to the material. Since the point of contact is essentially a line, pressure is normally given in pounds or tons per lineal inch. As an illustration, the Model 300 MS 28-27 as currently used in production of granular grade potash operates at approximately 11 tons per lineal inch.

Since the pressure required for compaction varies from material to material, the pressure on the rolls is continuously variable from 0 to the full 300 tons and is supplied by the hydraulic system.

The simple compaction-granulation system would be composed of the compacter, granulator and screens. (Figure 1) The screens make 3 cuts on the material. Oversize recycles to the granulator, undersize recycles to the compacter, and the product is carried to storage or shipping.

An expanded, more versatile system would be used for fertilizer operations. This system would be composed of a mixer, compacter, flake breaker, pre-breaker, granulator, and screens. (Figure 2)

The Model 150 MS 20.5-11.2 compacter is proposed for a 7½ ton per hour of –6+14 blended granular fertilizer. Use of two of these machines and associated equipment will produce 15 tons per hour of –6+14 granular fertilizer.

The Model 300 MS 28-27 compacter with associated equipment will produce 20 tons per hour of the –6+14 granular fertilizer (Figure 3)

These production figures are average and will vary some depending on the materials used to give the proper NPK analysis.

Costs, both capital and operating, under present conditions, indicates that compaction and granulation costs would range between $1.05 and $1.88 per ton of fertilizer depending on production rate and length of production time. Costs have been calculated on both a full year and on a 6 months basis. We have made the following assumptions in these calculations.

(1) A 3-story building approximately 20 by 30 feet will be constructed to house the equipment.

(2) Storage facilities for the NPK raw materials are currently in place.

(3) Proportioning or feeding equipment for the blender is currently in use and can be utilized with minor modifications.

(4) The entire installation will be written off in 10 years with zero salvage and a 10% return on investment.

(5) Electrical power is available at 0.01 cents per KWH.

(6) Building and installation costs will be a round number that is approximately 60% of equipment costs.

(7) Annual maintenance on equipment other than the compacter will be approximately 10% of the equipment costs. The maintenance and repair parts costs for the compacter will be approximately 10 cents per ton of material produced.

(8) Calculated costs do not take into consideration the onsite costs of the raw materials, storage of these materials or moving the materials to the compacter or blending operation. The costs of bagging or storage of the finished product is not considered in cost calculation, although a chief operator in the general plant area could act as the part-time operator for the compaction-granulation equipment.

The first and least complex system is rated at 7½ tons per hour of –6+14 product. The K-G Model 150 MS 20.5-11.2 compacter is used for this rate. (Figure 2).

This plant is increased to 15 tons per hour by the addition of a second Model 150 MS compacter, a flake breaker, a pre-breaker, one additional granulator and another screen.

The 20 ton per hour plant is based on the Model 300 MS 28-27 compacter and associated equipment shown in Figure 3.

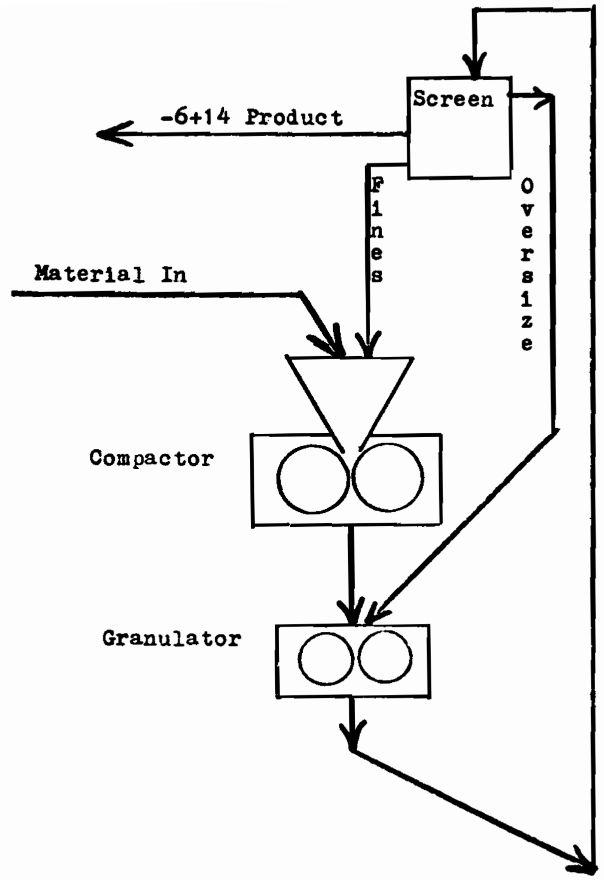
Table I explains the breakdown of the capital costs and operating costs for a 7½, 15 and 20 tons per hour plant.

We have dealt here only with large and medium size plants. Compacters and associated equipment are currently available for production ranges as low as 500 pounds per hour or any production rate up to these proposed at this time. As indicated in Table I, doubling the production rates does not double the capital cost. As an example, the 20 ton per hour plant could be increased to 40 tons per hour for approximately $225,000 more. Then on a full year of operation at 40 tons per hour the production costs would be less than $1.00 per ton.

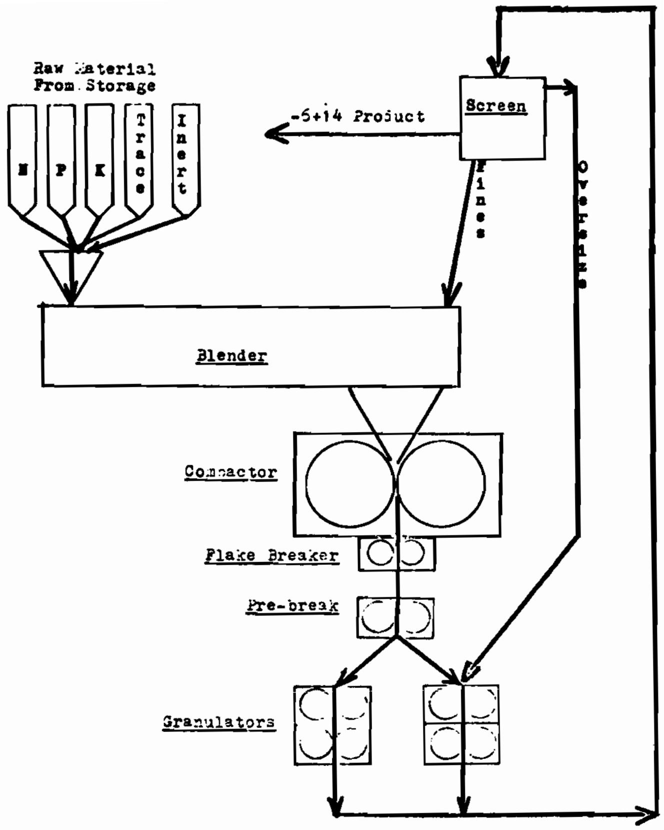
We believe that the compaction-granulation of blended fertilizers will solve many of the current problems of the fertilizer industry. The ability to quickly change production from one NPK blend to another will greatly assist the soild or dry fertilizer producer in meeting today's flexible market requirements. The increased sophistication of the farmer for his fertilizer needs places increased demands on the producer. These demands are approaching almost to a field by field fertilizer requirement. They have been specialized by crop for several years. The liquid fertilizer producer is able to quickly supply a field by field demand and we believe that the compaction and granulation system proposed will give the solid fertilizer producer a decided competitive advantage.

In many instances today, the low analysis fertilizers actually used for application have a transportation cost higher than the costs for the nutrients. Also, the new high analysis materials that are available and currently being developed require that the plants be located near the source of raw materials. The high analysis nutrients can be shipped long distances then blended with locally available inert materials to produce the low analysis fertilizer needed for application.

In closing, I want to paraphrase a slogan from one of the airlines: "When you are ready" to take this step and gain a decided marketing advantage, we at Komarek-Greaves "are ready" to assist you.



Compaction-Granulation Flow Diagram
Figure 1



7.5 Ton/hr. Compaction-Granulation System
Figure 2

MODERATOR REYNOLDS: Thank you, Mr. Boyce. I'm sure we'll have some questions generated here. I noticed that on most of your costs you discuss tons per hour and hours of operation. Have you run actual comparison here with some of the previous literature that would relate to a 20-tone-an-hour unit or a 40-ton-an-hour unit based on, say, 70,000 or 100,000 tons? Relative, in comparison with a conventional process?

MR. BOYCE: No. we have not. Since these costs would vary from various producers to producers, we decided it would be better this way rather than to get into discussions where someone might have $6 a ton, another one $8 and another $9, we'll let you use these figures to compare with your own present costs.

MR. WENDELL BIRCH (Continental Oil Company): You mentioned the pressure of 300 tons on the rollers. You didn't give any dimensions for the rollers. How does this break down in terms of pounds per square inch of the pressure that you are actually applying to the materials?

MR. BOYCE: On the one I was talking about the roller is 27 inches wide. Your point of contact between the rolls does not give you an area. This is then broken down into pounds per linear inch or tons per lineal inch that might be applied to the roll. This roll 27 inches wide gives you a little over 10 tons per inch.

MR. ARTHUR HANSEN (Wellman-Lord Co.): You may have mentioned this figure but I missed it. Could you tell me the battery limits installed cost of a 20-ton-per-hour granulation unit, that includes the granulator and sizing equipment and all? Do you have a figure on that?

MR. BOYCE: The compacting equipment as well?

MR. HANSEN: Yes.

MR. BOYCE: Yes, I gave that. Let me find it. At 20 tons per hour, you'd have capital equipment of $263,000. buildings and installation would be $150,000 for a total of $413,000.

MR. E. K. DRECHSEL (Duval Sales Corporation): Would you comment on the effect of anti-caking agents and so forth on the compacted materials and what, if anything, can be done about it?

MR. BOYCE: You mean the addition of anti-caking materials?

MR. DRECHSEL: Most of the fertilizers that you would use in such a compacter may have been treated prior to use. Does the equipment still work in spite of this additive or what can be done to eliminate the effects of it?

MR. BOYCE: We have worked here on the basis of not having anti-caking agents in there. Jack Carney, have we done any work with anti-caking material?

MR. CARNEY: Urea has been used. Various blends have been worked on. I think we went up to 29-29-0 blends.

MODERATOR REYNOLDS: Are they granulated products?

MR. BOYCE: In most instances they are finely ground.

TABLE 1

Capital and Operating Costs For Compaction-Granulation of Fertilizers

Production Rate	7.5 Tons/Hr.		15 Tons/Hr.		20 Tons/Hr.	
Equipment	$119,900		$222,800		$263,000	
Building and Installation	70,000		125,000		150,000	
Total Capital Investment	$189,900		$347,000		$413,000	
Hours of Oper.	8000	4000	8000	4000	8000	4000
Depreciation	$0.512	$1.025	$0.470	$0.940	$0.418	$0.745
Power	0.234	0.234	0.211	0.211	0.220	0.220
Compactor Maintenance	0.100	0.100	0.100	0.100	0.100	0.100
Maintenance, Other Equip.	0.098	0.196	0.086	0.171	0.070	0.141
Operator	0.333	0.333	0.333	0.333	0.250	0.250
Total Costs/Ton	$1.277	$1.888	$1.200	$1.755	$1.058	$1.456

MR. REYNOLDS: You take the crystal urea daimmonium phosphate and the potash and you grind it further.

MR. BOYCE: No, we do not grind. We take these materials as received. It can be done taking the materials as received.

MR. REYNOLDS: Most of the DAP that you would receive would be approximately minus 8 plus 12 mesh particle size.

MR. BOYCE: Yes, so going up to that size or even larger does not effect us.

MODERATOR REYNOLDS: Because you're going to reduce it?

MR. BOYCE: Yes.

CHAIRMAN SPILLMAN: In your formulation, do you use liquids, phosphoric acid or sulfuric acid?

MR. BOYCE: No.

CHAIRMAN SPILLMAN: Do you just use dry materials?

MR. BOYCE: Dry materials that you would use to make your blend of the high analysis such as urea, MAP, DAP, KC1, potassium sulfate or ground superphosphate. These types of materials.

A MEMBER: Have you had any experience with organic products such as slurry slag or components of garbage?

MR. BOYCE: We have compacted a lot of materials in all forms, including what you're talking about.

MR. THOMAS A. GRILLO (J.M. Huber Corp.): Do these compacted materials, since you don't use any anti-caking agents at all, have any tendency to cake or stick together?

MR. BOYCE: This would depend on the materials. Some of these materials are naturally sticky and compaction and granulation is not going to change that. There is a possibility that coatings would have to be applied to some NPK analysis.

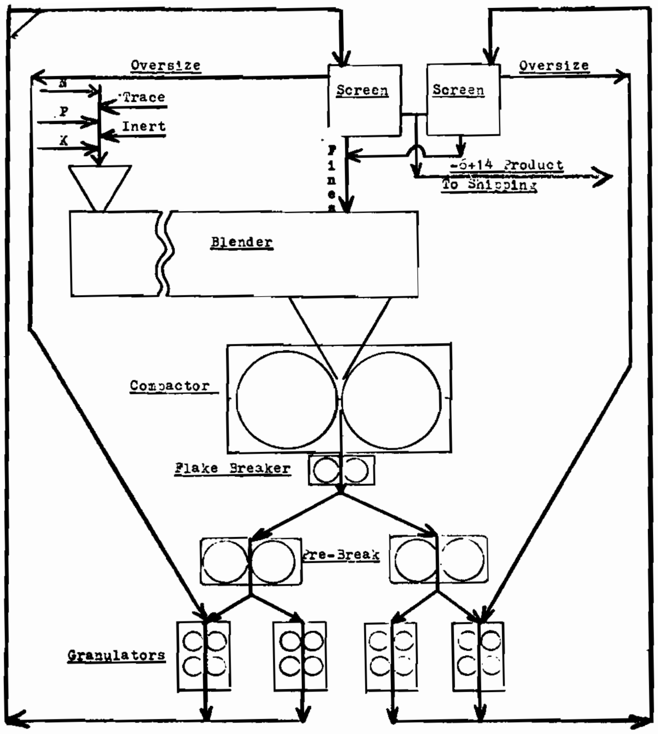
MR. GRILLO: There would be some provision then for adding material.

MR. BOYCE: This could be done by taking your product line coming off of the screen and using your normal coating equipment.

MR. GRILLO: Thank you.

MODERATOR REYNOLDS: Have you checked the uniformity of the resulting product that has been compacted and granulated?

MR. BOYCE: We've done this for some materials not all. This work is still going on. If you take a very finely



20 Ton/hr. Compaction-Granulation System
Figure 3

divided material such as minus-200 mesh or something like that, then as you go to an 8 by 14 or a 6 by 14, you have a more even distribution than if you blend NPK mixtures of a disired particle size.

MODERATOR REYNOLDS: I see. Most of your products as received are going to be coarse and in granular form.

MR. BOYCE: Yes.

MODERATOR REYNOLDS: So the thought being that one reason to go to this would be to get uniformity in your end product.

MR. BOYCE: That's correct. Most are crystals, if you blend these together, then when you apply the force, they are going to spread out and get on top of each other. You're talking about a quarter to a three-eighths inch, five-sixteenths thick cake. So you're going to have several particles in back and front of each other and the pressure will force them together, fill in your voids and give you a solid cake.

MR. SPILLMAN: Is there any attrition in handling the final product? How well does it hold together?

MR. BOYCE: This again would depend on the materials you are blending. Some would have very little attrition, others would have a higher attrition rate. There are variations and you could not put a definite figure on it for some materials.

MODERATOR REYNOLDS: Is there a commercial plant of this type under construction or in the design stages?

MR. BOYCE: For fertilizers, there is one under construction that will be in operation after the first of the year. I mentioned pesticides, insecticides and so forth, there are plants in operation. They are using some fertilizer, and inerts as the carriers for your pesticides.

A MEMBER: Is there any restriction as to moisture levels of the materials?

MR. BOYCE: Yes, there would be on the upper limits. Most materials will compact with as received moisture. Very rarely would you have to add moisture to it. Your potash today comes from a dryer into the compacter. Rarely would you find a material where you would have to add any moisture to it to get your compaction.

ANOTHER MEMBER: You have said there is one commercial plant under construction today in fertilizer. I assume you mean NPK mixed fertilizer or is it some straight material?

MR. BOYCE: It's NPK mixed fertilizer. This is being built in France.

MODERATOR REYNOLDS: Okay, we've had a very lively discussion. We have time for one more question.

A MEMBER: What can you tell us about the circulating load?

MR. BOYCE: The circulating load would depend on the type of granulation you are using. This would affect your final output of your compacter. If you own this 300-ton compacter we're talking about for 20 tons per hour production, you're talking a granulation efficiency of 70 to 80 per cent. This is using roll granulators. If you go to impact granulators, where your granulation efficiency is much lower, some of them are as low as 30 per cent, this will cut down your production of on-size product. So we can have a 70 per cent recirculating or use the more efficient roll granulators and you are circulating 30 per cent at the most.

SAME MEMBER: Do you have any problem of plugging of the rolls or adherence of buildup on the rolls and ejection of the particles?

MR. BOYCE: No, No. None to date in anything we've run. Now, some materials we have tested without water cooling on the rolls. Enough heat is generated that you will start melting. These are high analysis materials. You can do this. Urea, you can stick it to the rolls by generating enough heat to melt the urea actually in the compacter. Water cooled rolls would take care of this and it would be taken care of in the operation to prevent melting of the product.

MR. SPILLMAN: One quick question. You mentioned there is no lower limit on moisture. What about an upper limit?

MR. BOYCE: Here you run into problems. There are some materials, not necessarily fertilizer materials, that will become soupy at 5 per cent moisture, at 30 per cent moisture in other materials they are damp but they are not soupy. We prefer the materials to be dry. Some other materials we add a little moisture maybe to bring it up to 5 percent or something like that.

MODERATOR REYNOLDS: He's thinking of urea or ammonium nitrate, something like that. Most often it would be down about a half per cent but in storage it could go up higher.

MR. BOYCE: I don't think it would affect urea or ammonium nitrate. Some pick up in moisture in storage is not going to bother. Normal pick-up in storage, I should say.

MR SPILLMAN: What does your final product look like? Can it be stored in cone-shaped piles without segregation?

MR. BOYCE: Yes. It's cubes. You end up with a cube that would store in a cone-shaped pile. Since each cube is a complete fertilizer there is no problem with segregation.

MODERATOR REYNOLDS: Thank you very much. We will move on to our next speaker. I think we've discussed in the past many different standards, many different tests for our fertilizer related to physical and chemical properties. I think one subject that has always shown considerable interest relates to testing for particle hardness. As was indicated in the previous paper there is concern for degradation and dusting as material is handled. As we handle it more and more, through different systems, from production point to final distribution is now becoming of even more concern.

Out next speaker is Mr. Charles Dirdak of the American Potash and Chemical Corporation, who will discuss the subject: Testing Particle Hardness for Granular Fertilizer.

A Hardness Test

Charles Dirdak – American Potash Chemical Corp.

Abstract

A procedure is described for the determination of the hardness of granular materials. The significance of variables is explained in order to help users select appropriate adjustments to the procedure that make it suitable for a great variety of products. The precision of the procedure is expressed in terms of standard deviation.

The Need for a Test

The observation has been made many times that a particle of granular material does not retain its size. This is particularly true of granular fertilizers since many of them are rather fragile. A number of analytical procedures have been developed in an effort to measure the abrasion and shatterability of granular salts in order that products may be compared with one another for quality control and for competitive purposes. Some of these procedures measure the pressure required to crush a single particle, others tumble the product in steel drums. There are drop tests involving large samples.

There are impact devices that discharge the product into a stream of air and impinge the particles against a plate. All these procedures suffer from one or more of the following weaknesses: poor reproducibility, the need to average the result of many tests, usefulness limited to very soft materials, poor adaptability to a variety of products.

One Research Laboratory developed its own analytical procedure in 1956. It was later modified by our Control Laboratory and has been in constant use ever since. This procedure is highly adaptable to a great variety of products and is sufficiently reproducible for all practical applications that we have encountered.

PROCEDURE

A representative sample of the product is mixed and reduced through a Jones Splitter to a single weight of 100g ±5 g. The sample is transferred to the coarsest of a nest of 8" sieves. The nest is placed in a Ro-tap shaker for 5 minutes. The screen fractions are weighed. Each screen fraction is returned to its sieve, 200 steel balls are added to each sieve, and a pan is placed under each sieve. The nest is placed again in the Ro-tap, this time for 60 minutes. The screen fractions are again weighed. The total weight loss between the two weighings, as a function of sample weight, is reported as the breakage index (B.I.).

DISCUSSION

Almost every word in the procedure requires elaboration. Probably no single area of our work has been subjected to more intensive discussion than that of obtaining a representative sample. Since such a discussion would be somewhat foreign to the purpose of this paper it will suffice I suppose to state, with all the emphasis I am capable of, that we must have a representative sample. Failure in this area nullifies effectively the most diligent efforts made at any later stage of the analytical process. In our plant, all samples of granular products are taken by automatic devices that remove small cuts from the product stream, form 1 to 10 times per minute.

For our purposes an accumulated sample of from 5 to 20 lbs. is adequate. The chances are that the sample, even the best sample, will segregate in its container. It is therefore necessary to blend it into as homogeneous a distribution as is practical. Standard blending or tumbling equipment could be used for this purpose. However, since the determination of the fragility of a product is precisely our goal, we do not want to subject the sample prematurely to a damaging treatment. We found it both convenient and effective to pass the product repeatedly through a Jones Splitter. After each pass, the product is received in two pans, both of which are returned to the top of the splitter for another pass. For our purposes, 5 passes are adequate for sample blending.

After the mixing process, we continue to pass the sample through the splitter, but for another reason. We progressivley reduce the size of the sample by discarding the content of one pan after each pass, until a split weighs 100 grams, ±5 g. A practiced technician can handle such a reduction quite rapidly. However, a word of caution is here necessary. The sample weight should not be adjusted by withdrawing or adding small increments to the balance pan. It is quite impossible to perform such adjustments without causing some product segregation. Any weight adjustment must be made by the splitter process. When the sample weight is within its acceptable tolerance, it is weighed to 0.1g and the weight is recorded.

The selection of sieves to be used requires some precious knowledge of the product to be tested. Each separated screen fraction should be as narrow as possible. By that I mean that particle sizes within the screen fraction should be very uniform. Furthermore, it seems most practical to divide the product in not less than 2 nor more than 4 fractions.

One of the products that we test continually for its B.I. is Granular Potash.

The typical screen analysis is:

U.S. Standard Sieve No.	Typical Screen Analysis, % cumulative
6	0
12	60
16	92
100	99.8

The portion of the sample that is finer than 16 mesh spreads in small increments between the 16 and 100 mesh sieves and is therefore not suitable for the test. On the other hand the 6 to 12 and 12 to 16 fractions are narrow size bands and they describe more than 90% of the sample. These two fractions then are used for the B.I. test. It is our experience that fractions consisting normally of less than 10% of the product should not be used for the test because the data derived from them are not reproducible. It is also desirable that the total of the fractions considered constitute more than 80% of the sample.

100 grams of the original representative sample are thus placed in the coarsest of the nest of sieves. The nest is placed in the shaker for exactly 5 minutes. (We have determined experimentally that our product is adequately separated in its major screen fractions in that short a time, other products may require more time for the initial screening operation.) The amount of sample retained on each selected sieve is then weighed and the weights recorded.

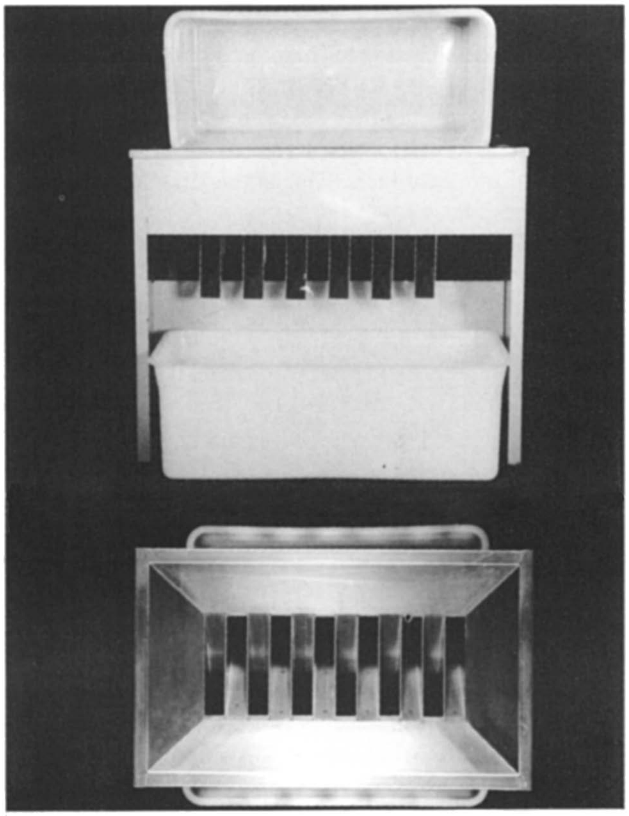


Figure 1 – Jones Splitter

The selected fractions, are returned to their respective sieves and 200 steel balls are added to each sieve. The steel balls have a diameter of 3/16", 200 of them weigh 86 grams, ± 1 gram. The function of the balls is to induce accelerated attrition in the product, and do this at least as violently as could be expected if this product made a cross-country trip in a box car or hopper, over poor suspension and flat wheels. A pan is installed under each sieve and the whole nest is placed again in the Ro-tap shaker, this time for 60 minutes.

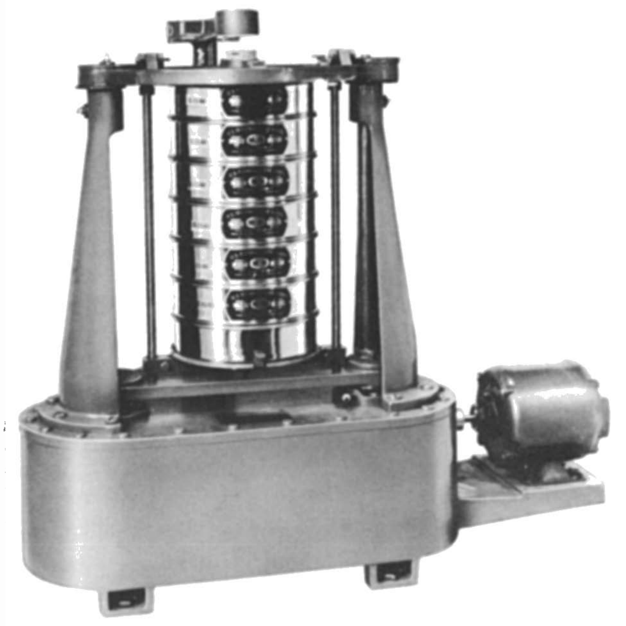


Figure 2 – RO-TAP Shaker

I am sure that most of you are familiar with the Ro-tap shaker. This piece of laboratory equipment is used almost as universally as Bureau of Standards certified standards. Because of the wear and tear of everyday use, it is essential to pay particular attention to certain operating conditions of this machine. The number of horizontal rotations per minute, the number of hammer beats per minute, the weight and height of the hammer, the type of wooden plug the hammer strikes, all these are operational characteristics that must be standardized and kept in good repair. We have also discovered that it is essential to fasten the nest of sieves securely in the machine. We use two rubber straps for that purpose, in addition to the clip supplied with the machine. We use 8" diameter, half-height Tyler sieves with U.S. mesh number identification. We have made our own additional pans by braising solid plates, instead of screen cloth, in old sieve frames. These pans are placed in the nest of sieves under the individual sieves selected for the breakage test.

After one hour of attrition, the amount of product retained on the selected sieves is then again weighed and the weights recorded.

A simple calculation completes the test. This is the formula we use:

$$\frac{\text{Total loss of selected screen fractions X 100}}{\text{Total original weight of selected screen fractions}} = \text{B.I.}$$

Here is an actual example:

1.	Original weights	
	On 12 mesh	70 grams
	On 16 mesh	25 grams
	Total of selected fractions	95 grams
2.	Weight after 60 minutes attrition	
	On 12 mesh	58 grams
	On 16 mesh	19 grams
3.	Loss = (70-58) + (25-19) = 18 grams	
4.	Breakage Index = $\frac{18 \text{ X } 100}{95} = 19$	

It should be noted that many options are available in this procedure. This is exactly what ensures its adaptability to a great variety of products that exhibit a great variety of characteristics. One further purpose in making judicious choices among available parameters is that the net result of the analytical operation should be a meaningful number. Generally speaking, when the B.I. of a product is less than 10, I would suspect that the analytical procedure is not sufficiently sensitive to product variations. It would probably be useful under such circumstances to modify test conditions in order to raise the B.I. value, say by increasing the number of screen fractions from 2 to 3 or 4, by increasing the number or weight of steel balls, or by decreasing the sample size (though not below 50 grams). Conversely, parameter modifications are also called for when the normal B.I. of a product is too high, say around 70 or 80.

In the course of the years, we have had numerous occasions to evaluate the precision of the procedure for our applications. We can generally state that it has a standard deviation of between 5 and 10% of the B.I. reported. For low breakage indexes, for example in the order of magnitude of 10 to 25 B.I., the standard deviation is very close to 1. For high breakage indexes, say a B.I. of 50 or 60, the standard deviation is 5 or 6. Of course, when a product is that fragile, a 10% error in B.I. is of no consequence.

In summary then, we have presented an analytical procedure that measures the fragility of granular products with adequate precision. The procedure is highly versatile, since it can be readily adapted to a wide variety of products and hardnesses.

MODERATOR REYNOLDS: Mr. Dirdak your paper was interesting and I am sure will be helpful information to our membership. We have time for questions.

MR. JACK CARNEY: Test procedure you said was 200 steel balls per pan. Did I miss the weight or the size of the steel balls?

MR. CHARLES DIRDAK: I indicated the dimensions. They are 3–16ths of an inch diameter and the 200 steel balls weight 86 grams plus or minus one.

MR. CARNEY: Right, but depending on your product, then you adjust the number of fractions and also the size and number of steel balls?

MR. DIRDAK: That's right. In order to have a significant number, you may adjust these parameters up or down.

MR. CARNEY: I see. Thank you.

MODERATOR REYNOLDS: Is this a published procedure or is this the first time it has been presented?

MR. DIRDAK: We have made this procedure available to our customers. This is the first time that it has been made public.

A MEMBER: Do you plan to present this procedure to some organization such as the AOAC?

MR. DIRDAK: I would hope that the presentation at the Round Table is sufficiently public to make the procedure available to anyone who needs it.

MODERATOR REYNOLDS: Fine. I'm sure that you'll find some application. Many people will be asking for it because it's very, very good.

MR. SPILLMAN: Has this been tested on product other than potash?

MR. DIRDAK: Yes.

MODERATOR REYNOLD: So you consider it equally applicable to phosphates and nitrates and so on?

MR. DIRDAK: It is. We have tested the procedure for some of our customers who make mixed fertilizers and we have given them information that they consider useful.

A MEMBER: What is the life span of these sieves?

MR. DIRDAK: The life span? It will depand on how tough the particles are. We use sieves for instance, for anhydrous borax, a very brittle and sharp edged material and the sieves wear quite rapidly. After a few weeks we

STANDARD DEVIATION OF BREAKAGE INDEX

DAY	SHIFT	DUPLICATE ANALYSES		DAY	SHIFT	DUPLICATE ANALYSES	
1	A	15	17	1	A	20	19
	B	20	21		B	21	21
	C	22	21		C	19	18
2	A	21	20	2	A	20	20
	B	23	23		B	20	19
	C	22	20		C	21	19
3	A	16	15	3	A	19	18
	B	18	19		B	20	21
	C	16	16		C	20	21
4	A	20	21	4	A	20	20
	B	19	20		B	18	19
	C	19	18		C	18	17
5	A	22	24	5	A	21	20
	B	17	18		B	19	19
	C	14	15		C	21	22
6	A	16	17	6	A	18	20
	B	14	15		B	19	20
	C	19	22		C	20	18
7	A	19	20	7	A	19	18
	B	17	16		B	19	18
	C	19	20		C	17	16
8	A	19	18	8	A	21	20
	B	17	20		B	19	20
	C	20	21		C	17	18
9	A	18	20	9	A	18	18
	B	21	19		B	20	21
	C	20	20		C	20	20
10	A	20	21	10	A	21	20
	B	21	21		B	20	21
	C	20	19		C	21	21
		s = 0.96				s = 0.75	

have to change sieves. But speaking of fertilizers, such as potash, the sieves will last almost indefinitely.

MODERATOR REYNOLDS: Thank you. Unless someone has a comment or anything else to bring before us or any discussion, then we will conclude our afternoon session and meet tomorroe morning at nine-thirty.

The meeting adjourned at three-forty-five o'clock p.m.)

Thursday Morning Session, November 6, 1969

The Round Table convened at 9:30 o'clock a.m.
Robert R. Heck, Moderator

MODERATOR HECK: Gentlemen, welcome to the third session of the Fertilizer Industry Round Table.

I don't think there is a person here who has not been made aware, through various means such as TV, radio newspapers, trade papers, and personal contacts, that industry in the United States is faced with a very severe problem in air and water pollution.

I think most of the companies represented here have had their share of air pollution problems. If you are in the phosphate, nitrogen, potash, fertilizer or limestone business, you have seen action being taken at various levels of state, county, federal and city agencies and even by neighborhood committees, to place limits on air pollution and water pollution.

As I look out across this group, I see quite a few people who have been directly involved, particularly at state levels in trying to help the various states write legislature concerning air pollution control. We know that the same action is taking place at the federal level.

There has been a great deal of confusion in the writing of the various laws. We have the problem that each state legislates more or less according to its own needs. During the last year or two, however, the various legislating bodies have indicated a willingness to work together to provide uniformity in the codes and regulations that are passed.

This morning we have the privilege of having a speaker with us who will discuss some of the ramifications of the air pollution codes and regulations, particularly at the federal level.

Our first speaker this morning is Rick Renninger with the National Crushed Stone Association. Rick is a graduate of Rensselear Polytech and is presently an engineer geologist with NCSA. He is quite experienced in the field of effluent control and has an excellent knowledge of legislative action concerning effluent control.

Air Pollution Control Regulations Of Concern To The Fertilizer Industry*

F. A. Renninger

INTRODUCTION

It is indeed a pleasure and I must confess somewhat of a surprise for me to be here with you this morning. Some of you may recall that three years ago, at your 16th Annual Meeting, I addressed the group on the question of the Statistical Concepts of Bulk Sampling. Apparently I must have succeeded in so completely confusing the situation or "polluting the atmosphere" that Mr. Marshall thought it appropriate for me to return again this morning and speak with you about Air Pollution Codes and Regulations. In other words, he felt that if I had been successful in so fouling the atmosphere three years ago, the least I could do would be to help purify it now. In any event, my purpose this morning is, in fact, to talk with you about the question of air pollution codes, regulations, and the current status of air pollution legislation in the United States.

WHAT HAS GONE BEFORE

The National Crushed Stone Association became interested initially in the question of air pollution from the standpoint of the dust generated at their processing plants as early as 1962. We launched a program within our Association which included the education of a staff member to investigate the dust pollution question in and around crushed stone operations. In conjunction with that task it became evident that a library or reference file covering the many aspects of air pollution control, codes, and regulations should be developed. Part of this task naturally involved following the federal legislative developments.

The first federal legislation in the air pollution field in this country took place in 1955. That Act was little more than a recognition of the potential consequences of air pollution. The passage of the Clean Air Act in December of 1963 served to truly focus public attention and opinion in this area. This bill also put "teeth" in the federal government's drive to reduce air pollution. By providing the funds necessary to finance needed research and the technical assistance, through the United States Public Health Service, necessary to train competent personnel, the Clean Air Act rapidly paved the way for the various states to strengthen thier air pollution control boards and to begin to establish air quality standards. At about this same time great increases in newspaper, radio, and television presentations relating to polluted air gave birth to a more informed population – more informed with regard to the

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* A talk given before the Fertilizer Industry Round Table, Washington, D.C., November 6, 1969

** Engineer-Geologist, National Crushed Stone Association, Washington, D.C.

perils of the situation and less informed with regard to the extent of the situation. The net result of this increased publicity was simply to arouse and create a public less willing to tolerate conditions which they now felt seriously affected their welfare. Articles such as the following which was taken from the Wall Street Journal of November 10, 1965 is an example of just this type of publicity:

"The annual damage air pollution does to crops, buildings, equipment, and other property is estimated at $11 billion, without even calculating the health hazards. Pollutants such as sufur dioxide and suspended particulates can aggravate asthma and other chronic respiratory diseases and in large enough concentrations can even cause death." Industry naturally became the prime target of these initial efforts to purify the air. A plant stack emitting large quantities of dense smoke, obnoxious odors, or injurious fumes was an excellent target, and probably rightly so, for such exercises of concern. This same concern has continued and is apparent today. However, by and large, the Clean Air Act of 1963 did little more than to provide the mechanism in general for control and to provide funds for research and development. Certain amendments made to it in late 1965 served primarily to intensify these initial efforts. Under the Clean Air Act of 1963 and its amendments in 1965 a number of the states and many communities began to establish air pollution control boards, air quality boards, or subsections of their public health departments charged with the problem of pollution control. In other words, under these acts, there began to develop a hierarchy with which to pursue at some later date the question of air pollution control.

WHERE WE ARE TODAY

In 1967 Congress of the United States began to consider what eventually became the Air Quality Act of 1967. This piece of legislation was indeed a meaningful and directed piece of legislation. It set forth the mechanism for eventual control of those pollutants recognized as potentially dangerous to human health or welfare, plant health and welfare, animal health and welfare, or general esthetics. The Air Quality Act of 1967 directed the National Center for Air Pollution Control, now known as the National Air Pollution Control Administration, a separate agency within the Department of Health, Education, and Welfare, to develop, publish, and circulate certain documents related to air pollution and to designate within the country various areas known as air quality control regions.

An air quality control region has been described as an area in which the climatic conditions, air movement conditions, or in general the potential pollution situation, is somewhat similar, and an area in which control may be exercised uniformly. Once an area has been designated, the political entities within that area are under notice to begin to develope the necessary bureaucracy, provided they don't already have it, by which to control air pollution. Step 2 under the Air Quality Act of 1967 states that the National Air Pollution Control Administration shall issue air quality criteria and control technology documents. These documents are to be basis for air quality standards developed by the local governmental bodies within each designated area. It is clearly emphasized in the Act that air quality criteria are not to be considered air quality standards. Air quality criteria are described and defined as quasi-health documents – that is, they are documents setting forth the limits of human, animal, plant, or materials exposure in terms of time and prevailing concentration of a particular pollutant. A standard, an air quality standard, on the other hand, is a document developed by the local authorities which specifies the prevailing control levels to be sought within the jurisdiction. Air quality criteria, therefore, are not to be confused with national emission standards. Most of you will recall that the question of national emission standards was quite a heated subject during the discussions which took place before the Senate Public Works Committee when the Air Quality Act of 1967 was receiving review in the United States Congress. The technology documents simply describe the status of control technology and the level of development within the control equipment field.

There is a very decided procedure by which one progresses from air quality criteria to air quality standards and finally to emission standards. If I may be permitted the liberty of quoting from a paper* prepared by Dr. Ralph I. Larsen, Office of Criteria and Standards, U.S. Department of Health, Education, and Welfare, National Air Pollution Control Administration, which was presented before the Annual Meeting of the Air Pollution Control Association in New York City earlier this year. Dr. Larsen had this to say with respect to air quality criteria, air quality standards, and emission standards:

> "Air quality criteria do not clean the air. Neither do air quality standards. The air is cleared only after air quality criteria have been used to derive air quality standards and the air quality standards, in turn, have been used to derive emission standards. Emission standards only specify how much pollution will be prevented from entering the atmosphere. The process of proceeding from criteria to clean air may be termed air management. Air management may be divided into about a dozen steps:
>
> "1. Determine the effects of various pollutant concentrations and exposure durations on people, plants, animals, and property
>
> "2. Decide which effects to prevent
>
> "3. Select ambient air quality standards that will prevent these effects
>
> "4. Measure and evaluate ambient air pollution concentrations
>
> "5. Calculate overall source reduction needed to achieve selected ambient air quality standards
>
> "6. Measure or estimate emissions from each source type in an area
>
> "7. Decide how much each source type could be permitted to emit and still achieve the desired ambient air standards
>
> "8. Select or develop means for achieving the necessary emissions reduction

"9. Decide the date after which each source type should be controlled
"10. Set emission standards
"11. Enforce emission standards
"12. Continue monitoring sources in the ambient atmosphere to ensure that adequate air quality is being achieved."

Dr. Larsen very clearly set forth the specific steps necessary for one to proceed from the federally issued air quality criteria to the locally enforceable emission standards. Under the Air Quality Act of 1967 there is a precise time schedule covering this process. Once the National Air Pollution Control Administration has issued air quality criteria and control technology documents covering a specific pollutant, each state or air quality control region has 90 days in which to file a letter of intent stating that they do, in fact, intend to develop air quality standards and emission standards with respect to the designated pollutant. The state and local control regions then have an additional 180 days in which to submit, for approval, their intended air quality standards. Following approval of the standards, the state or local control region has another 180 days to submit a plan to implement these standards. Assuming, therefore, that the time schedule is met, a little simple arithmetic leads one to the conclusion that following the issuance of air quality criteria by the National Air Pollution Control Administration effective air quality standards and emission standards must be in effect within a period of 15 months. If a state or local control region does not meet the specified time schedule, this is a signal for federal authorities to step into that specific area and to establish and begin to enforce federal control standards. Naturally, the state or local agencies are not about to permit this to happen for a number of very obvious reasons.

Where do we stand today with respect to the question of air quality criteria, air quality standards, and emission standards? To date only two sets of criteria and control technology documents have been issued by the National Air Pollution Control Administration. In February of 1969 NAPCA issued the control criteria and control technology documents for particulate matter or dust and the sulfur oxides. It is my understanding that to date all 50 states and all previously designated control regions have filed letters of intent and are now in the process of developing air quality standards. If one goes back to February, adds to it a total of nine months (90 days for letter of intent plus 180 days for standards) he very rapidly comes up with the month of November as the deadline for the submission of the air quality standard proposals by the states and the local control districts. Add to this an additional six months and we are looking at May 1970 as the date at which the implementation plans must be submitted for approval and enforcement must begin. Now, depending upon the time lags built into the implementation plans in the various areas, we are faced with control of particulate matter and the sulfur gases beginning in May of next year or at some point in the very near future.

WHAT TO EXPECT TOMORROW

The National Air Pollution Control Administration has established a proposed schedule for the issuance of future criteria and future control technology documents. During 1970 it is planned to publish documents relating to carbon monoxide, chemical oxidants, ozone, and hydrocarbons. In 1971 the federal government intends to publish criteria relating to the nitrogen oxides, the fluorides, lead, the organic carcinogens, and aldehydes.

As I understand the fertilizer manufacturing industry, the pollutants of primary concern are the particulates, the fluoride gases, the sulfur gases, and the nitrogen gases. Let's consider again, therefore, the schedule for the issuance of criteria and control documents by the federal government, and relate this to when one might expect nationwide control over those pollutants common to your industry. The documents for the sulfur gases and the particulates have already been issued and one can look for universal control, or nationwide control, beginning in 1971. The other two primary pollutants of concern to the fertilizer industry, as I understand it, are the fluoride gases and the nitrogen compounds. These are slated for treatment in air qulaity criteria documents and air pollution control technology documents by NAPCA in 1971. Therefore, with the aid of simple arithmetic once again, mid-1973 appears to be the target date for control standards and probably emission standards in each state and major air quality control region within the country.

In several areas there are already in existence control standards and in some cases emission standards dealing with the specific pollutants which affect your industry. The State of Florida, for instance, has a rather stringent control standard with respect to the fluoride gases. As I understand their control standards, their ambient air quality is judged by the fluoride content in foliage and in waters at some specified distance from the operation and emission is controlled by limiting the amount of fluoride gas which can be discharged per unit of process weight. To my knowledge this is the only state fluoride control standard in the United States at this time. It is somewhat restrictive and probably more restrictive than those which will appear once the fluoride criteria documents are issued.

When the sulfur oxide documents were first issued – I believe this was in 1967 or '68 – there was some serious concern as to whether they were not far more restrictive than need be. In fact, the National Air pollution Control Administration did re-examine these documents and make modifications. The question of the sulfur gases is the single most popular, if that is the correct word, pollutant as far as national concern and national attention. The most publicity has been leveled at this pollutant because of its very obvious effect upon human health in a number of air pollution episodes, namely, the most recent episodes in London and New York City. There are state and local

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* Larsen, R. I., Proceeding from Air Quality Criteria to Air Quality Standards and Emission Standards, Presented at APCA, New York, New York, June 1969

control standards already in effect covering the sulfur oxides.

To date, the nitrogen compounds have not received much publicity but, taken as a group, these three pollutants – the fluorides, the sulfur oxides, and the nitrogen compounds – are gases or very, very fine mists which are emitted from those stacks exhausting the processes in which they are produced. Since they are emitted from an identifiable source within an operation, they lend themselves to specific control, and also to the very specific and enforceable emission type standards. The emission standards which will attempt to control these pollutants will, most probably, be written in terms of the process weight. That is, it will be based upon the through weight of a process during a given period of time and one will be permitted to emit no more than a specified amount of each of the gases.

The problem of dust, or the particulate matter, however, is a somewhat different question. If fertilizer plants are anything like those we have encountered in the crushed stone industry, it will be almost impossible to control the emission of particulate matter on the basis of process weight. Such control is possible only when the emission takes place through some identifiable flue, chimney, or stack, or in general some specific point within the plant area. In the crushed stone industry this is not the case. The entire piece of property is a potential dust emitting source; that is, dust can be emitted during drilling, blasting, loading, hauling, crushing, conveying, and screening. While most of these processes are not now enclosed, it is possible to cover these operations (and this is being done in many areas.) but this does not entirely solve the problem. Due to the nature of our operations, and I understand yours also, it is necessary to stockpile large quantities of the finished product. Air moving across these stockpiles can generate and whip into the atmosphere enough dust to place the process in violation of most existing and proposed codes and regulations. The movement of heavy traffic in and around the area, in the yard, area and along the haul roads also generates dust very difficult to control. Because of the numerous sources of dust within a given quarry area, and I assume this is true also for the average fertilizer plant, we have been trying to have the control authorities look at the entire plant area as a single emission source; that is, to treat the entire area, whether it be 50 square feet or two square miles, as a single emission source and simply make ambient air measurements both upwind and downwind of the operation simultaneously. Compliance or control, then, would be based upon the difference between these two levels. A number of states have this type of control regulation – it is in effect in the State of Texas, it is in effect in the State of Pennsylvania, and it is currently being given very serious consideration by the consulting firm under contract to study the question of emission factors for the crushed stone, sand, and gravel industry for the National Air Pollution Control Administration. I trust that they will recommend this type of emission standard or emission factor analysis for these types of operations. Since the fertilizer manufacturers' concern with dust most probably parallels ours, such an approach should be encouraged by your industry also.

CLOSURE

This has been a very rapid review of the status of air pollution control legislation in the United States and certain of the proposed time tables now in existence which relate to the concerns of the fertilizer manufacturing industry. I am sure I have left out a good deal but I'll be most happy to answer any questions.

Gentlemen, thank you for your attention.

MODERATOR HECK: Thank you, Rick. Now, let's move to another source of pollution that has received as much attention during the last year as air pollution. I am referring, of course, to water pollution.

Those of you who live near the Great Lakes know that there is a constant fight going on, particularly now on Lake Michigan.

There is a lot of argument going on today; suits are being filed in courts now against cities, industry, townships, almost anything that connects with Lake Michigan.

Investigations are being conducted to determine the effect of "runoff water".

To discuss the subject of water pollution regulations as they apply to the fertilizer operations, we have with us this morning, Pete Cox. Pete is with Edwin Cox Associates. This company has done a tremendous amount of work in the area of both air and water pollution control, and I think Pete comes well qualified as an expert in this area. Pete.

A Summary Of Water Pollution Regulation As They Apply To Fertilizer

Edwin Cox III

It is a privilege to speak to the Round Table this morning. In this summary of water pollution regulations as they apply to the fertilizer industry, it appears best ***not*** to list many regulations and tolerances. Rather, a discussion of how effluent control is administered and an explanation of the laws and some of their terms will be more helpful.

At the outset, let me hasten to observe that there is nothing new or novel about water pollution. Those of us who grew up on farms know that septic tanks are down-hill of wells. Hundreds of years ago, laws were passed dealing with riparian rights.

In a detailed discussion of water pollution in the United States today, two expressions require definition – stream classification and stream quality. The *classification* of a stream is the assignment of probable use to it. For each use there is a minimum *quality.* Drinking water, for example, is a classification requiring high quality water. Conversely, agricultural use, in some areas for some crops, can tolerate many impurities.

There are three kinds of general controls of pollution which apply to the fertilizer industry – direct; damage awards; and indirect. *Direct regulations* apply directly to fertilizer plants – to mining and producing. These are the regulations governing works effluent. They are obvious and shall be discussed further.

Damage awards based on suits in civil courts, have been costly. As this type of suit increases, awards will be significant.

Indirect regulations are more interesting to us in terms of the future. The federal Water Pollution Control Administration (FWPCA) has recently stirred up a great deal of excitement by criticizing the excessive use of fertilizer, contending that this puts too much nitrogen and phosphorus into the waters. Eutrophication results, upsetting the ecology of the receiving streams. This was a subject of much discussion at the recent American Chemical Society meeting in New Yor. I refer you to those discussions. All sides of the point have been presented – from a denial of the effect to a strong presentation of the case.

Concurrently, however, another unique problem has arisen which has put the FWPCA and the USDA at cross-purposes. An approximate figure is that the waste from cattle in the United States is equivalent to a population of 800 million people. For years the USDA, through its county agents and other advisory personnel at all levels, is reported to have suggested that feeder-pens and like installations be established on hillsides so that they may be naturally washed and drained. The FWPCA does not look kindly upon this practice since it is the equivalent of such a large discharge to waters of untreated raw sewage. In the midst of this holocaust and conflict, I doubt that in the near future fertilizer usage will be regulated because of affect on water pollution.

But another *indirect regulation* that may apply to the fertilizer plants would result from the continued study being made of the use of phosphates domestically – primarily for detergents but also on other applications. Such uses are being discouraged. Should regulations be promulgated and enforced restricting the use of phosphates in the household, then a large abount of P_2O_5 will be thrown into the fertilizer industry at competitive prices. Unless fertilizer requirements increase more rapidly than is currently projected, producing plants will be affected by water pollution regulations applying directly to other industries. A further discussion of this point is beyond the scope of this paper.

Direct Regulation

Direct regulation of water quality (and pollution) occurs at three levels of government. Each publishes its own regulations, each has its own executive or enforcement power, but each is different. These are local, state and Federal.

LOCAL CONTROL

The local authorities are the easiest to understand. They may be either municipalities or several governmental units in a metropolitan area which have common waste treatment facilities. They are characterized by:

(a) the tightest control – since they have only a limited number of establishments of interest and normally have the capability of monitoring the effluents of each.

(b) the local regulations are associated with a local treatment facility. Since the locality operates the facility, it is interested in who is using it.

(c) a fee or license schedule is attached to the use of a treatment facility, normally with a maximum “tax free” use.

Fee schedules are established when effluent impurities rise above certain levels. These fees are considered to be equivalent to the increased cost to the treatment facility above the “average,” although these facts are subject to much debate. Local control does have one obvious merit. It is like a toll road. The user pays for what he gets. Further, combining wastes often permits several operations to share the benefits of a single, large, treatment facility.

STATE CONTROL

The second of the levels of control is by the state government. This is the level that is common to all control throughout the United States. There are many responsibilities of the state control agencies. They are under many different forms of organizations. Essentially four major functions are required of the state control agency:

(a) Classify streams.

(b) Establish stream quality standards.

(c) Issue permits for discharges to guarantee the quality of streams in question after a review of quality criteria, plant design and effluent projections.

(d) Enforce permits after issue.

Unfortunately, there is no common pattern in the United States. There are many different water groups in each state, each with control. Further, different govermental groups like different things – one group is for dams while another favors a high flow. (The construction of a dam often works against a down-stream operation which discharges into the stream in question.)

FEDERAL CONTROL

The third level of control is by the Federal Government. It is over all and uniform (hopefully) across the United States, but such control is impractical. Recently suits have been entered by the Federal government in Ohio. These were announced by Interior Secretary Hickle (under whom the FWPCA is) against four steel companies, a mining firm and the City of Toledo for alledgedly polluting interstate waters. This is a demonstration of potential power and may be regarded, although not so stated by the Federal government, as an example to the different states that they must enforce regulations because the FEderal government will move if a state does not. The problem is a difficult one because politicians at various levels seldom get together easily.

The November 1969 actions against Virginia are clear statements of this intent.

The Federal government practically has four missions assigned to it.

(a) Sponsoring and conduct of research and development.

(b) Coordination of the various activities of the Federal government as they influence water pollution.

(c) Review of states' programs to insure that water quality throughout the United States is consistently determined and insured.

(d) With the passage of recent laws, the power to act in specific cases, such as oil spills.

The Water Quality Act of 1965 provided for the establishment of the water quality standards. The Clean Water Restoration Act of 1966 provided the tools to make the earlier act effective. It also gave the government authority to participate directly with industry. Previously, Federal funding joint efforts had been possible with non-profit institutions only, and these efforts were restricted in scope.

Since 1966 the Federal position has been reinforced by specific laws concerning specific items (oil spills). There will be moreoof this type of legislation.

It appears that control must be at the state level. Above that level enforcement and knowledge of specific areas and problems is simply not possible. Below that level, the fragmentation of mission, duplication of effort and facilities, and the probable resulting arbitrary division of areas of responsibility would prevent effective control.

When the fifty state stream classifications and quality standards were reviewed by the Federal government, it was radily apparent that if any one state was not cooperative the Federal government could establish a stream quality for that state. Further, by isolating that state from the others, the Federal government could move in with its enforcement and actually penalize citizens by rigorous prosecution not typical of the nation – segregation, if you will. It should be noted that all fifty states have submitted plans to the government and that these have been approved. Now the FWPCA is going back and making the states adopt more stringent regulations.

One of the big threats which the Federal government does have over states involves money. The extent to which this applies varies among the states since not all are funded. Some states have, as you might expect, reached into the pork barrel of government money to get what they could. Others have refused to accept any. A third type has taken or sought such funds as it needs realistically to meet requirements it simply cannot finance by itself. This raises interesting problems for the Federal government in influencing state actions – no single program or method of operation can be universally successful.

Examples of Classification and Quality

So far I have discussed simply how the regulations are enforced. By taking regulations from two states, let me give you now an example of stream classification and then of stream quality.

After defining points of interest and making rules applicable to all water, one state establishes the following classes of water:

AA– meeting requirements for use for domestic and food processing purposes with disinfection as only treatment required.

A– meeting requirements for use as swimming waters.

B– suitable for domestic supply after complete treatment in accordance with requirements of the state Board of Health.

C– suitable for propagation of fish, industrial and agricultural uses.

C-1– suitable for fish survival, industrial and agricultural uses.

SA– suitable for shell fishing for market purposes.

SB– suitable for bathing and other usages except shell fishing for market purposes.

SC– suitable for crabbing, commercial fishing and any other usages except bathing or shell fishing for market purposes.

The above distinctions are typical of those of any state which would have both fresh and salt water. Thus are streams *classified.*

Another state established the following stream quality for a creek designated for agriculture and fishing:

Dissolved oxygen – 7.5 ppm
BOD – 6 ppm
Settleable solids – 0.1 ppm
Suspended solids – 5.0 ppm
Ammoniacal nitrogen – not to exceed 2.0 ppm
Residual chlorine
Coliform organisms – most probable number 100 per 100 milliliters with not more than 10 per cent to exceed 1000 per 100 milliliters
pH – between 6.0 and 8.0
Inorganic nutrients – maximum Nitrate N – 0.3 ppm
Phosphate as PO_4 – 0.6 ppm

Interesting enough the following specification in then added:

> "other physical and chemical constituents not specifically mentioned will be covered by additional specifications as conditions detrimental to the stream arise. The specific mentions of items 1 through 9 does not necessarily mean that the addition of other physical or chemical constituents will be condoned."

Above are examples of the classification of a stream by its ultimate use – drinking water, swimming, fishing, agriculture, other. After classification, stream quality is established – the minimum or maximum amounts of specific components.

The hard job of the control agency is to determine what effluent a plant will be permitted to discharge so that the plant operation can continue economically but at the same time permit the quality of the stream to be maintained. It is not an easy problem.

It is a pleasure to be able to state that most control officials – local, state and Federal – are aware of the complexity of the task of maintaining present stream quality or of restoring quality to streams which have become nothing but waste lines. Ultimately, the individual citizens of the U.S. must pay for pollution control – either directly, through public funding, or indirectly, by increased costs of manufacturing which are passed on to the consumer.

I would like to thank all of you for your courtesies. It certainly has been a pleasure to speak to you this morning.

MODERATOR HECK: Thank you, Pete. I think Mr. Reminger and Mr. Cox gave us a pretty practical level of working with the Federal, State and local Agencies on "Air and Water Polution Control". We are going to take a break for coffee. We would like to reconvene by 11 a.m.

MODERATOR HECK: Gentlemen, would you please be seated. This morning we heard two very good talks, two very good speakers, discussing legislation and agency action against air pollution. So we have sort of identified the problem. We have heard a little about what other people think about the problem, in particular those people who can legislate action.

Now we are going to discuss what can be done about some of these problems. Our first speaker, Mr. Jim Tomany, is with the UOP Air Correction Division and Jim has asked that I not tell you what UOP Air Correction Division is, he will explain that.

A Guide To The Selection Of Air Pollution Control Equipment

James P. Tomany

INTRODUCTION

Air pollution control is concerned with the removal of gases, vapors and particulate matter, either separately or in various combinations from process exhaust streams. Such emissions as solvents from drying ovens, nitric oxide gases from chemical processing plants, fly ash and SO_2 from power plants, ammonia and phosphate dusts from fertilizer operations represent some of the problems encountered in the air pollution control field.

The purpose of this discussion is to generally describe the equipment made for the solution of these various air pollution problems.

DEFINITION OF AIR POLLUTION PROBLEMS

Before any particular equipment item can be specified a definition of the type of contaminant, concentration, source and degree of control must be determined. The three equipment types available for the control of gases or vapors are combustion, adsorption and absorption devices. The four major types of equipment utilized for the collection of particulate matter are mechanical collector, electrostatic precipitator, fabric filter and wet scrubber. The relationship of the various equipment types for the removal of gases, vapors and particulates is shown in Figure 1.

In the control of gaseous and vapor pollutants a determination of the pollutants being emitted must be made. The emission rate is dependent on a measurement of the total gas flow and the concentration of the various pollutants in the gas stream. To permit the design of combustion, adsorption or absorption equipment for the removal of gaseous or vapor contaminants, the following minimum data are required; total gas flow (CFM), gas temperature (degrees F.), moisture content (%H_2O), contaminant identification and concentration (ppm, vol % or lb/min.) and degree of control (% conversion, lb/min. discharged). Different laboratory analytical methods are utilized to determine these concentrations. Devices developed by UOP/ACD for this purpose include a portable Total Combustible Carbon Analyzer and a Flame Ionization Analyzer Detector, which can be used in the field to determine CO, CO_2 and Hydrocarbon materials.

In the collection of particulate matter from a gas stream, the gas flow (CFM), gas temperature (degrees F.), moisture content (%H_2O), dust or fume identification and concentration (grains/CF, mg/CF), dust size analysis (micron, %) and degree of control *% collection efficiency, lb/min. discharged).

Some definition of these terms are in order.

Some definition of these terms are in order.

CFM	Ft. ^{3}/min., either ft. ^{3}/min. at conditions (ACFM)or standard ft. ^{3}/min. at 60^{o}F, 1 atm. pressure (SCFM); sometimes 70^{o}F or 32 ^{o}F.
ppm	Parts per million by weight or lb/10^6 lb.
% conversion	Percent of hydrocarbons oxidized in combustion unit.
grains/CF	Grains (700 grains= 1 lb.) of dust or fume per cu.ft. of gas; either actual ft.^{3}, standard ft.^{3} or dry standard ft.^{3}
mg/CF	Milligrams (454,000 mg = 1 lb.) of dust or fume per cu.ft. gas.
micron,%	Expression of dust size analysis; typical analysis follows.
+ 5 microns	40%
-5 + 2.5 microns	27
-2.5 + 1.5 microns	20
- 1.5 microns	13
	100%

This would mean that 40% of the dust particles by weight are greater than 5 microns in size, 27% are less than 5 microns but greater than 2.5 microns, 20% are less than 2.5 microns but greater than 1.5 microns and the remainder or 13% are less than 1.5 microns.

(micron) = 0.001 mm
0.00004 in. (approx.)

AIR POLLUTION CONTROL EQUIPMENT FOR GASES AND VAPORS

A great variety of gaseous and vapor air pollutants can be destroyed by combustion. These include most organic materials, particularly the hydrocarbons, plus inorganic combustibles such as ammonia, hydrogen sulfide and cyanide gases. To oxidize these pollutants it is necessary to either heat the gas stream to quote high temperatures (1200 to 1500 degrees F.) to accomplish thermal incineration or to use a catalyst to promote combustion at lower temperatures (500 to 900 degrees F.).

The basic design requirements for both catalytic and thermal incinerators are the same. Systems must provide thorough mixing of the combustibles with air, sufficient heat to raise the temperature of the gas stream to the desired level for oxidation, even temperature and flow distribution and sufficient dwell time at temperature to accomplish the desired degree of oxidation.

CATALYTIC SYSTEMS

Because a catalyst accelerates the combustion reaction, catalytic systems operate at lower temperatures and considerably shorter dwell time than thermal incinerators.

The basic components of a catalytic system are the preheat burner, catalyst elements, exhaust fan and control equipment. These are enclosed in an insulated sheet metal housing.

In Figure 2 there is shown a typical catalytic system for the treatment of exhaust gases from an oven wherein the major portion of the pollutants are solvent vapors at concentrations far below the flammable range. The exhaust gases enter the system at 400 degrees F. where they are mixed with flue gases from the preheat burner. The combined stream at about 700 degrees F. is discharged by the fan through the catalyst section where the oxidation of the vapors is affected. The actual dwell time in the catalyst bed can be extremely short; a few hundredths of a second or less. Combustion efficiency can be increased by increasing residence time, preheat temperature or the effective surface area of the catalyst.

The oxidation products, mostly vapor and CO_2, leave the catalyst at about 900 degrees F. and are discharged to atmosphere. Some utilization of the heat in this discharged gas stream can effect fuel savings and in Figure 3 there is shown an alternate system where the incoming exhaust stream is preheated by the hot combustion gases leaving the catalyst.

The most commonly used catalyst are of the platimum family. They are in the form of a thinly deposited coating on a supporting alloy ribbon. An important factor in the choice of a catalytic system is to make certain that there are no catalyst poisons or fouling agents in the exhaust stream. For the platinum type catalysts, heavy metals, phosphates and arsenic will act as poisons whereas practically any type of industrial dust will tend to coat the catalyst surface thereby slowly reducing its effectiveness.

Referring to Figure 2, the design and performance data for this catalytic system are as follows:

TABLE I

Combustion Equipment Design & Performance Data

Application	Metal decorating oven exhaust
Exhaust volume	5000 SCFM
Exhaust temperature	400°F
Contaminants	Naptha, Xylol, methyl, isobutyl ketone, isophorone
Concentration	1.25 lb/min.
Performance	Odor and color free, 90% destruction of organics

THERMAL INCINERATORS

Thermal incinerators require high temperatures for combustion; from 1200 to 1500 degrees F. for most pollutants. The upper end of this range is required for the more difficult-to-burn materials. Temperatures are furnished by a burner firing into a holding chamber where oxidation occurs.

In a thermal incinerator, dwell time is a function of the size of the holding chamber, which is predicated on the exhaust flow rate. Usually systems are designed with dwell times from .3 second to one second. Increasing either dwell times or temperatures will increase the combustion efficiency.

In Figure 4 there is shown a thermal incinerator for treatment of the identical solvent laden exhaust gases treated in the catalytic system shown in Figure 2. As before, the exhaust gases enter the system at 400 degrees F. They are circulated outside the firing tube by the fan, thereby increasing their temperature to the required level (1200 degrees to 1500 degrees F.). They are then mixed with air and fuel in the burner and pass through the firing tube at a rate to obtain sufficient dwell time to complete oxidation.

Conditions for this duty are shown in Table 1. In this case, either the catalytic or thermal incineration method was applicable so that the economy was the deciding factor. In Table II there is shown a simplified economic comparison for the two systems.

TABLE II

Combustion Equipment Economic Analysis

	Catalytic	Thermal
Installed equipment costs, $	15,000	13,000
Heat requirements, BTU/hr.	1.9×10^6	4.3×10^6
Annual fuel costs, $	3,800	8,600

ADSORPTION

Adsorption is the collection of gas and vapor molecules on the surface of a solid adsorbent material, such as activated carbon. Adsorption is most efficient when the gas stream is at relatively low temperatures, 150 degrees or less. This makes it a convenient method for cleaning a variety of ambient temperature exhausts. A typical example is the exhaust gases from dry cleaning operations.

Adsorption is a physical process, making use of attractive forces between the adsorbent surfaces and the gas molecules to accomplish collection and retention of the pollutant gases. To remove or desorbed by passing steam or hot air through it.

Adsorption is not as generally applicable as the combustion processes. This method is particularly useful under the following conditions:

1. The pollutant gas is non-combustible or difficult to burn.
2. The pollutant is ain very dilute concentrations in the exhaust stream.
3. The pollutant is sufficiently valuable to recover, either as a chemical or for its fuel volume.

For solvents, adsorption would have to be followed by either a disposal facility or a recovery system.

Some of the factors to be considered in the design of an adsorption system are the nature and concentration of the pollutants, their adsorption and desorption rates, the temperature, pressure and composition of the exhaust gas stream, the availability of a regenerative gas stream and the adsorbent characteristics. Either steam or inert gas is preferred for regeneration. The higher the regenerative stream temperature, the faster desorption can be accomplished.

WET SCRUBBERS

This topic will be treated in the next section.

AIR POLLUTION CONTROL EQUIPMENT FOR PARTICULATE MATTER

There are four major types of equipment available for the collection of particulate matter. These are the mechanical collector, electrostatic precipitator, fabric filter and wet scrubber. As shown in Figure 1, the wet scrubber also serves in the area of gas absorption.

The proper choice of equipment for any single particulate collection problem is determined by many variables; among these are process considerations, equipment ability and economic factors. Some of the process considerations and equipment limitations and their effect on the optimum selection of equipment are as follows:

1. Dust particle size –

The mechanical collector can collect dusts having particles 5 microns and larger at reasonable efficiencies. The precipitator, fabric filter and wet scrubber can be applied effectively to contaminants having much smaller minimum particle size.

2. Product recovery –

Usually dry collection for recoverable products is preferred so that the wet scrubber would be eliminated. However, in some cases where wet processing is practical, the wet scrubber can return a recoverable slurry to process.

3. Gas stream temperature –

If the gas is much over 600 degrees F., the fabric filter is eliminated. Although the wet scrubber can be used for handling hot gases, a sometimes objectionable steam plume will be emitted from the scrubber.

Operating costs for all four types of collectors are essentially power costs required to overcome the gas pressure drop through each type. This is true for the mechanical, precipitator and fabric filters collectors. In the precipitator there is an additional power cost required to maintain an electrical field of the proper density. There follows Table IV, a tabulation of average gas pressure drops for each of the four types of equipment with equivalent brake horsepower requirements for each 10,000 CFM capacity.

TABLE IV

Operating Pressure Drops and BHP for Particulate Collection Equipment

Type	Pressure Drop in. w.g.	Brake HP
Mechanical Collector	1.0 - 3.0	2.6 - 7.7
Electrostatic Precipitator	0.2 - 0.5	3.0 - 3.8*
Fabric Filter	2.0 - 6.0	5.2 -15.4
Wet Scrubber	2.0 -10.0	5.2 -26.0**

*Including electrical field power input
**For collection of moderate size particles; 1 to 5 microns

A general description of the operating principles, abilities, areas of application and the mechanical design of each of the four types of particulate collection equipment follows.

MECHANICAL COLLECTORS

A typical single tube collector is shown in Figure 5. These tubes are arranged in multiples as shown in Figure 6. The operation of each tube depends on exerting centrifugal force on the particles by introducing the dust laden gas stream tangentially into the body of the tube.

The dust is thrown to the outside wall while the clean gas is allowed to escape up through the outlet tube. The tube performs most effectively in the particle size range of 5 to 44 microns. A tabulation of collection efficiencies for a mechanical collector as a function of the average particle size for a fly sample is as follows:

4. Wet scrubber effluent disposal facilities –

This is becoming a greater problem in view of the efforts being exerted to clean up the water ways.

5. Gas stream dew point –

If the moisture content of the gas stream is too great, condensation will interfere most seriously with the operation of the fabric filter, mechanical collector and precipitator

6. Gas stream and particulate matter conductivity –

The electrostatic precipitator depends on a reasonable electrical conductivity of dust and if the conductivity is not in this range, the precipitator will not function properly.

7. Gas absorption and particulate collection –

This is a common cambination and the wet scrubber must be applied.

8. Gas stream flammable constituents –

This consideration would favor the use of the wet scrubber.

Capital investment and operating costs for the four types of particulate collection equipment vary considerably. For comparison an approximate selling price index expressed as dollars/CFM capacity is used. For carbon steel construction and moderate collection duty, approximately selling price ranges for the four types (today's market) are as follows:

TABLE III

Sell Prices for Particulate Collection Equipment

Type	$/CFM
Mechanical Collector	0.10 - 0.25
Electrostatic Precipitator	0.50 - 1.25
Fabric Filter	0.50 - 1.50
Wet Scrubber	0.20 - 0.60

TABLE V

Mechanical Collector Efficiencies

Particle Size, micron	Collection Efficiency, %
44	99
30	98
20	97
10	90
5	65
2	30

These prices are based on average gas flow rates, about 100,000 CFM for the electrostatic precipitator and 20,000 CFM for the others.

These data indicate the direct relationship between particle size and collection efficiency. The particle mass and tube diameter are also important variables, affecting the collection efficiency. This would be expected as the expression for centrifugal force (F) is,

$$F = \frac{MV^2}{r} \qquad \text{where}$$

M = mass of particle
V = particle velocity
r = radius of the dust particle path

This formula indicates that the centrifugal force or collection efficiency varies inversely as the tube radius. which it does. It also shows that the collection efficiency varies directly as the square of the velocity. Higher velocities are accompanied by increased pressure drop across the tube, as defined by the relationship $V = 2gh$, where "h" is the velocity pressure head and "g" is the acceleration due to gravity. Therefore, collection efficiency varies directly as the pressure drop across the tube. Most mechanical collectors operate in a pressure drop range of 1 to 3 in. w.g.

Tubular collectors are manufactured in a size range from one-half inch to 10-inches in diameter. A tabulation of these sizes together with their capacities is as follows.

TABLE VI

Mechanical Collector Capacities

Tube Diameter Inches	Capacity SCFM
0.5	0.8
0.75	2.4
1.0	8.6
1.5	16.8
2.0	30.0
3.0	67.2
6.0	220.0
10.0	857.0

Application of the multi-tube type of mechanical collector is restricted to granular materials or liquids. Major application areas are

Power Plants	Tubes in range of 5 to 10 inches in diameter used to collect coal and oil fly ash from boiler stack gases.
Rock Products	Large Diameter tubular collectors are used in cement, trap rock and sand processing operations.
Petroleum	In gas pipe lines, two-inch diameter tubes are housed in pressure vessels for the removal of entrained liquids and solids.
Special Applications	Air cleaners on railroad diesel locomotives (two inch diameter tubes). Gas turbine inlet air cleaners (two-inch diameter tubes).

ELECTROSTATIC PRECIPITATORS

The electrostatic precipitator relies on producing an electric charge on the particle to be collected and then directing the charged particle by electrostatic force to the collecting electrodes. A single tube electrostatic precipitator is shown in Figure 7. The electrostatic precipitator operation involves four basic steps –

1. Electrostatic charge is applied to each dust particle.
2. The ionized particle with its negative charge is attracted to the grounded electrode. This precipitation force is the product of the field strength between the electrodes and the acquired charge on the particle.
3. The acquired charge is discharged to the rounded collecting electrode.
4. The collected dust is gravity discharged to storage hoppers. The electrostatic precipitator has an extremely high collection efficiency over the entire dust size range. For a fly ash sample analyzing 40 percent less than 10 microns a precipitator can be designed for a collection efficiency of 99.5 to 99.8 percent at a draft loss of 0.2 to 0.5 in. w.g.

Most precipitators are presently designed to operate at efficiencies approaching 100 percent. High efficiency is the result of a careful balance between gas velocity and active length of the unit. A large precipitator with low gas velocity permits maximum efficiencies. Uniform gas velocity distribution is extremely important so that arrangement of inlet and outlet ducts requires careful study. For some installations a pilot plexiglass model is utilized to determine optimum arrangement.

The electrostatic precipitator collection efficiency is related to the time of particle exposure to the electrostatic field and the resistivity of the dust particle. The exposure time can be increased either by increasing the cross section area of the precipitator or its length in the direction of gas flow.

Particulate matter, having moderate electric resistivity, is easier to collect in an electrostatic precipitator than that having higher or lower values. Fly ash resistivities vary from 10^4 to 10^{17} ohm-centimeters.

A UOP/ACD plate type electrostatic precipitator is shown in Figure 8. It consists of reinforced steel casing and hoppers with inlet and outlet arranged for horizontal gas flow through the separating walls being the collecting or grounded electrodes. Suspended between each piece of plate electrodes is a high-voltage, small-diameter wire electrode.

To discharge the dust from the collecting electrodes a rapper system is provided. These are solenoid actuated and are mounted externally on the precipitator roof. Connecting rods pass through bushings and terminate at a point for delivering an impact blow to the electrodes.

Some major industrial applications for electrostatic precipitators include –

ELECTRIC POWER PRODUCTION –

In firing pulverized coal the residual ash is entrained in furnace gases as a fine dust or fly ash. Electrostatic precipitators are utilized to collect this dust. Gas flows up to two to three million CFM are handled in some installations.

METALLURGICAL INDUSTRY –

Precipitators are used to collect very fine metallic fumes. In some installations the recovery of copper, zinc and lead oxides with the precipitator has made their use an economic necessity.

STEEL INDUSTRY –

Removal of suspended particles from open hearth and basic oxygen furnace gases is an important application area for precipitators. A typical installation would handle total gaseous effluent of about 500,000 CFM at about 600 degrees F. with collection efficiencies up to 99.5 percent.

FABRIC FILTERS –

Fabric filters operate by trapping air borne dust by impingement on the fine filters comprising the fabric. As the collection of dust continues, an accumulation of dust particle adheres to the fabric surface. The fabric filter attains its maximum efficiency during this period of dust build-up.

After a fixed operating period the bags must be cleaned by some suitable method to prevent the build-up of excessive pressure drop. Immediately after cleaning, the filtering efficiency will be reduced until the build-up of collected dust takes place. The pressure drop also increases between "clean" and "loaded" operating conditions.

The fabric filter is particularly applicable where a high collection efficiency is required. It will handle dusts in a size range from 0.01 microns to coarse screen sizes at collection efficiencies usually in excess of 99.9 percent. The size of the collector is determined by the gas flow rate, particle size, dust concentration, cleaning period and allowable pressure drop. Draft losses for the fabric filter are in the range of 2 to 6 inches w.g.

Some of the limitations imposed on the use of the fabric filter are the temperature, humidity of the gas and the corrosive or flamable nature of the gas and/or dust. Maintenance resulting from fabric wear is one of the major problems associated with the fabric filter.

There are two general types of fabric filters, wherein the dust laden gas is passed through the woven fabric to remove the particulates while the gas is allowed to continue. The most common type is the tubular or bag filter, which is comprised of freely suspended fabric tubes usually fastened at both ends. The sleeve or envelope type depends on an internal wire frame for support. The frames carrying the bags are mounted in rows or banks to make up multiple units. In both types a mechanism is included to periodically remove collected particulates from the filter medium.

The UOP/ACD Fabric Filter is a modified sleeve or envelope type. This unit is shown in Figure 9. It is comprised of the casing, suspended fabric pockets, collecting hoppers, counter-current air-oulse cleaning system and support structure. A series of pockets are arranged to fit into an elongated rectangle. Each pocket is divided into sections or walls whose shape is maintained by spiral metal rings. These pockets are closed at the base and suspended at the top and are arranged in banks, the number required being a function of the process duty.

The process gas flows from the exterior to the interior of each pocket, the dust being collected on the outside surface. The clean gas goes inside the pockets and passes through a damper to the outlet duct.

Cleaning is accomplished by changing the position of the dampers so that cleaning air enters the filter. A pulsating air stream is produced by a fan which directs ambient air or cleaned gas through a venturi section of the duct. Its flow is intermittently interrupted by a partially perforated disc rotating normal to and cutting through the air stream at the throat. This air stream enters in interior of the filter pockets causing the dust to be dislodged and fall into the collecting hoppers.

The fabric filter can be applied in any process area where dry collection is desired and where the temperature and humidity of the gases to be handled do not impose limitations. Some of the industries and processes where the fabric filters are applied are:

Iron & Steel –
  Electric furnace
  B.O.F. process
  Cupolas
Cement Industry –
  Pneumatic conveying system
  Rotary kiln
  Grinding systems
Automotive Industry –
  Metal grinding and
    polishing
  Foundry sand
General Industrial –
  Room air ventilation
  Food spray dryer effluent

WET SCRUBBERS –

The wet scrubber removes dust from a gas stream by contacting it with a suitable liquor. A good wet scrubber is one that can effect the most intimate contact between the gas stream and liquor for the purpose of transferring the suspended particulate matter from the gas to the liquor.

The collection efficiency, dust particle size and pressure drop are closely related in the operation of the wet scrubber. The required operating pressure drop varies inversely as the dust particle size for a given collection efficiency; or for a given dust particle size, the collection efficiency will increase as the operating pressure drop increases. Because of this relationship, the major variable in the design of a wet scrubber is the operating pressure drop. It may vary from 3 to 100 inches w.g., depending for the most part on the size of the dust particles being collected.

Because, unlike the other particulate collection equipment, the wet scrubber employs another process stream to collect particulate matter, a scrubber usually performs additional process functions besides dust collection. Gas absorption, chemical reaction and heat transfer are some of these. The simultaneous removal of dust and noxious gases by the use of suitable scrubbing liquor is a very common duty for the wet scrubber.

A single scrubber can operate at varying collection efficiencies by varying the operating pressure drop. In one design a six-stage FB Scrubber operating at a pressure drop of 30 inches w.g. can effect a 99.5 percent collection efficiency with aluminum chloride having an average particle size of 0.2 microns. This same scrubber with only a single stage and operating at about 4 inches w.g. pressure drop can collect dust with an average particle size of 1.5 microns at collection efficiency of 98.5 percent.

There are three general types of gas-liquor contact designs which apply to the majority of commercial scrubbers. These are the venturi, surface area, and impingement designs. The following discussion covers two of these: the venturi (Aeromix and Ventri-Sphere); and the surface area design (Floating Bed and Turbulent Contact Absorber).

AEROMIX SCRUBBER –

The Aeromix Wet Scrubber is shown in Figure 10. The dust laden gases flow upward through the throat at high velocities where they are mixed with liquor being introduced from the feed tank. The gas-liquor mixture enters the diffuser tube whose area gradually increases so that as the gas flows upward, its velocity is decreased. A portion of the liquor and dust suspended in the gas stream falls back towards the throat where it is re-entrained and carried upward. This continuous internal recirculation provides intimate liquid-gas contact for high scrubbing efficiency.

The gas continues up into the entrainment separator where the entrained liquor is centrifugally thrown out and is seperated from the gases. The gases are then discharged to atmosphere.

The entrained liquor is returned by gravity to a three-compartment feed tank. The liquor overflows a weir into one compartment where it is mixed with fresh make-up liquor and then gravity discharged to the Aeromix throat. In the other compartment solid-laden liquor is discharged to waste or product recovery, depending on the nature of the process.

By regulating the make-up liquor flow, the concentration of collected material in the liquor can be built up to a high degree. For example, coal dust from pulverized bin systems has been built up to a 25 percent concentration and refired in the boilers.

The Aeromix is used –

1. To collect particulates in the range of 0.5 to 5 microns at pressure drops in the range of 3 to 8 inches w.g.
2. To collect and concentrate particulates up to 30 percent solids.
3. For absorption of relatively soluble gases with suitable liquors.

FLOATING BED SCRUBBER –

The Floating Bed Scrubber (FB) is shown in Figure 11. Dust laden gases flow upward through mobile packing consisting of 1-1/2 inch diameter plastic spheres (see example). Water or other suitable liquor flows downward from a liquor inlet header at the top of the scrubber. Under the influence of the counter-current gas and liquor flow, the spheres are forced upward in violent random motion. This action causes the spheres to impinge against each other and it is this self-cleaning action that distinguishes the FB Scrubber from competitive wet scrubbers.

The FB Scrubber can be provided with one or more stages, depending on the difficulty of the dust collection problem. Each stage is provided with a support grid and retaining grid, the latter acting as the support grid for the next upper stage. The grids are spaced about 18 inches apart and a static depth of spheres in each bed or stage is maintained at 12 inches. At nominal gas and liquor flow rates of 400 to 600 ft/min. and 10 to 20 gal/min. $ft._2$, the estimated pressure drop is about 3 inches w.g. per stage.

As the gases continue to flow upward, they pass through an entrainment separator where entrained liquor is removed and flows by gravity down through the various stages. The pressure drop in the FB Scrubber is increased by increasing the recirculation flow rate at the higher liquor rates, 20 to 30 gal/min. $ft._2$; the pressure drop can be increased to 6 inches w.g. per stage.

General Areas of application for the FB Scrubber are –

1. For particulate matter collection in the range of 0.1 to 5 microns, especially where high dust loadings are involved.
2. For the collection of viscid and flocculent particulate matter.

Turbulent Contact Absorber (TCA) –

The TCA is a further development of the FB Scrubber. By increasing the distance between the support and retaining grids, increased liquor and gas flow rates can be obtained at pressure drops equivalent to those abtained in the FB Scrubber. Referring to Figure 12, the TCA grids are spaced about 4 feet apart while the static depth of spheres in each stage is maintained at 8 to 12 inches. Average gas and liquid flow rates in the TCA are 1000 $CFM/ft._2$ and 15 gas/min. $ft._2$, respectively, at at pressure drop of about 3 inches w.g. per stage.

The advantage of the greater liquid and gas flow rates are two-fold;

1. The required diameter and the selling price per unit volumetric gas flow rates are reduced.

2. The mass and heat transfer rates obtained in the TCA are increased remarkably.

To understand the second factor, a discussion of the factors involved in mass and heat transfer must be considered. In most conventional fixed packing towers at liquor and gas flow rates of 5 gal/min. ft.$_2$ and 200 to 300 CFM/ft.$_2$, very low gas absorption and heat transfer rates are obtained. To increase these, greater flow rates and violent mixing of the two counter-currently flowing phases are required. However, as flow rates are increased in these packed towers, the pressure drop increases sharply until a condition known as "flooding" is reached. At this point, the gas upflow will actually attempt to support the liquor downflow, pressure drops will climb steeply, surging will take place and the tower becomes inoperable.

The diffused, mobile packing utilized in the TCA allows these high liquor and gas flow rates to be realized without exhorbitant pressure drops. At these higher flow rates absorption coefficients for SO_2 in NaOH have been increased five fold over those obtained in a fixed packing tower. It is this increase in transfer rate which makes the TCA idially applicable in areas of gas absorption, chemical reaction and heat transfer as well as particulate collection.

General areas of application for the TCA are:

1. For gas absorption, chemical reaction and heat transfer, especially in the presence of viscid and flocculent particulate matter.
2. For particulate collection accompanied by gas absorption.

VENTRI-SPHERE HIGH ENERGY SCRUBBER –

Because of the established relationship between perticle size and the required pressure drop for reasonable collection efficiencies, it is realized that for very fine particulate matter in the range of 0.02 to 0.5 microns, pressure drops in the range of 15 to 60 inches w.g. must be utilized. Furthermore, to obtain the most intimate contact between the dust laden gas stream and liquor, a venturi device is required. In this type of high energy scrubber the gas enters the venturi section and is accelerated to a high velocity at the throat where it impinges upon the liquid stream. This results in the atomization of the liquid into fine droplets. The high differential velocity between the gas and atomized droplets promotes impaction of the gas borne particles and fine droplets. As the gas decelerates, further impaction and agglomeration of the particles take place. These liquor agglomerates are separated from the gas stream in the separator.

UOP has developed an improved high energy scrubber of high efficiency and compact design called the UOP Ventri-Sphere High Energy Scrubber.

Referring to Figure 13, dust laden gases enter the venturi section of the scrubber, usually at elevated temperatures. In the venturi funnel the gases meet a

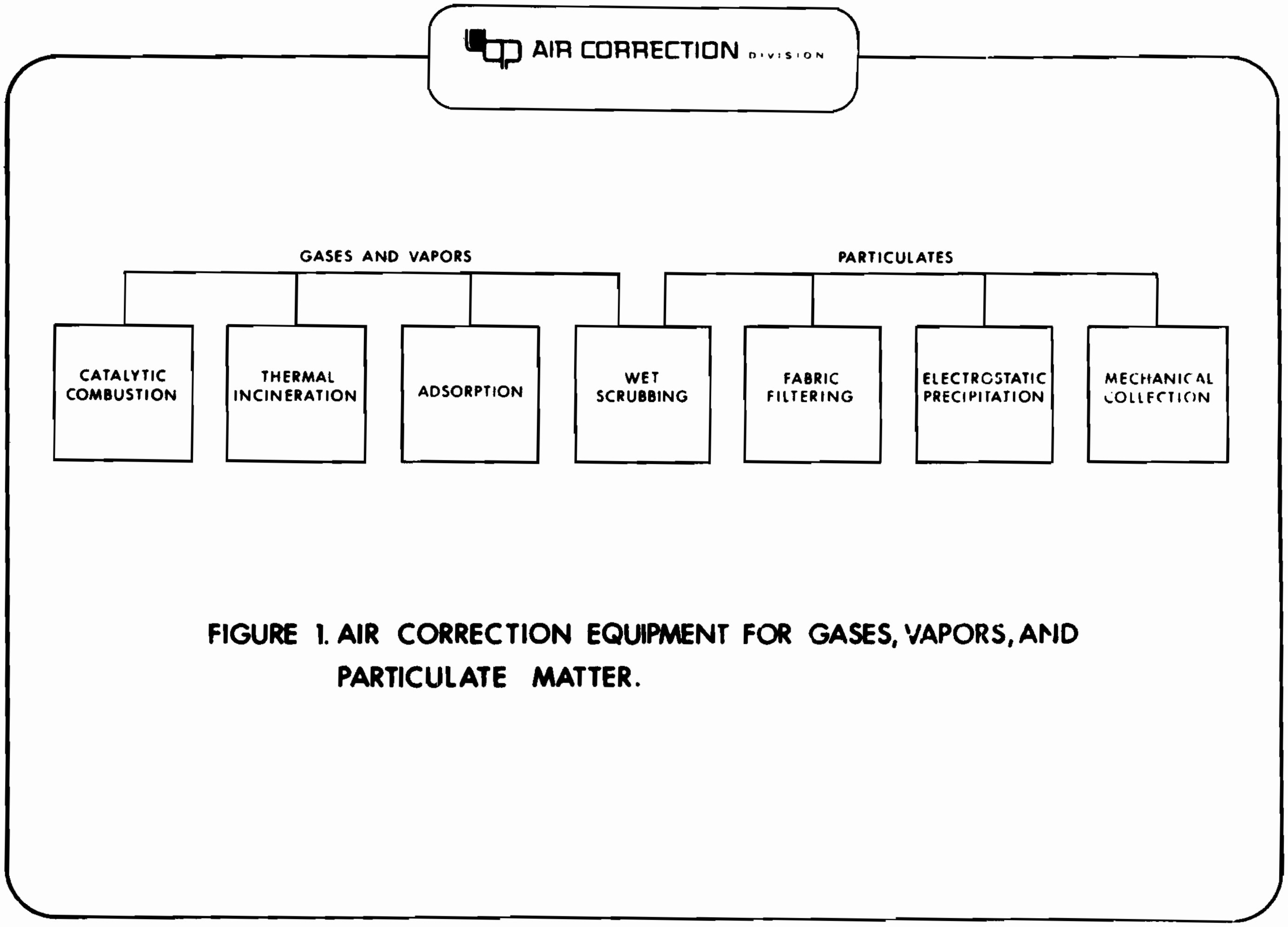


FIGURE 1. AIR CORRECTION EQUIPMENT FOR GASES, VAPORS, AND PARTICULATE MATTER.

continuous flow of recirculated liquor which overflows the weir and provides a continuous wetted surface so as to prevent solids build up. The liquor and gases pass through the venturi throat where the hot gas is cooled to its saturation temperature and the dust particles are forced into the liquor particles.

In this design the throat is fabricated of soft rubber which can be mechanically flexed to take care of gas flow rate variations so as to maintain a constant pressure drop.

The gas continues down through the difusser tube into the separator vessel where its flow is reversed. The gases rise up through the mobile packing stage where the remainder of the agglomerates are removed. The saturated gas then continues up through a helical spinner where final de-entrainment is effected.

Make-up liquor to the scrubber is introduced to a header located above the mobile packing section. Additional liquor is pumped to this header from the separator sump. Recirculated liquor is also intorduced to the reservoir where it overflows across the venturi funnel section.

This scrubber is ideally suited to collect submicron particulate matter. Where it might be applied to the collection of lime and soda fume from a lime kiln, it is expected that a 99.5 percent collection efficiency will be obtained at a pressure drop of about 15 inches w.g. The particle size distribution for this particular application would be 0.3 to 5 microns.

MODERATOR HECK: Thank you Jim for that excellent discussion on guides to the selection of Air Pollution Control Equipment.

MR. LIEBERMAN: (W. R. Grace Co.): What is this energy regain tube in your last slide 13?

MR. TOMANY: I am glad you asked that question. Actually, you see, from what I said, where you want the energy expended is across the venturi; that is where the work is done. By means of this diffuser tube, the 200 or 300 feet per second through the venturi is reduced to about 4000 feet per minute at the discharge of the energy regain tube. As a result of this velocity change, we actually obtain a regain of energy.

Now I will give you an example. On a paper mill installation we might have something like 20 inches in w.g. across the venturi. From the inlet of the venturi to the discharge of the regain tube, the total pressure drop instead of 20 inches in w.g., might read something like 14 inches in w.g. We have picked up 6 inches in w.g. in that regain tube. So that although the overall pressure drop across the scrubber might be 18 inches w.g., we might have put as much as 20 inches in w.g. of that, where it really counts, across the venturi. And this energy regain feature seems to be looking very well.

MR. GRANT MARBURGER (Kerr-McGee Co.): What is odor? Is it a sub-micron particle size or a molecular gas?

MR. TOMANY: You pick it an you can have it. If it is chlorine, it is strictly a gas. If it is smoke, like in municipal incineration, where odors are a problem, it is the sub-micron smoke particles that are responsible. So far as we are concerned, it can fall in either classification. We never attack odor per odor itself. But we try to determine, as you brought out, is it a gas or is it a particlate, and then use the solution we think best of it.

MR. HECK: I hate to cut this off at this point, and possibly later this afternoon we will have an opportunity to discuss some of these things with Jim.

We have gone through air and water pollution legislation and so forth, and we have seen an excellent demonstration of various means of controlling air pollution, controlling dust, controlling vapors and so forth. The next subject for this morning will be The Aspects and Costs of Air Pollution Control Within the Phosphate Fertilizer Industry.

Larry Hill with Poly-Con Corporation will deliver this paper. I am a little embrassed this morning to find myself introducing people I don't know. I felt like I had been around the industry so long, I ought to know everybody. The Poly-Con Corporation are manufacturers and engineers of air polution equipment. We are very glad to have Larry with us this morning.

(See Figures 2 to 13 Pages 51 Thru 62)

Aspects and Costs of Air Pollution Control Within The Phosphate Fertilizer Industry

Lawrence J. Hill

When the operator or management of a fertilizer plant contemplates the purchase of pollution abatement equipment, he must be aware of more than the technical and physical aspects of the equipment required. He will become involved with the capital and operating costs of such a system. He should also concern himself with the depreciation allowances and what further costs will be incurred when forthcoming pollution code requirements are to be met.

The extent to which the manufacturer of fertilizers will involve himself in the specification, evaluation and selection of pollution control equipment will depend upon his organizational structure. The ideal situation is to have an internal enviromental group which would be totally familiar with the company's products and processes, industry techniques, control methods and the status of existing and pending air pollution regulations. Many firms have such groups or individuals and they contribute valuable knowledge and experience to the application of various pollution control devices to the phosphate fertilizer industry. Other companies, because of their size or operation, will not have this special effort for defining and solving their problems. The knowledge may exist within the personnel of the plant, but their responsibilities are in the area of management or production.

Here it is that the role of the pollution control equipment manufacturer has to come to the assistance of the fertilizer producer. By having complete knowledge of the problem, the manufacturer's engineer can select and design the air pollution equipment or system which will best suit the application. To understand the air pollution*

* Continued on Page 63

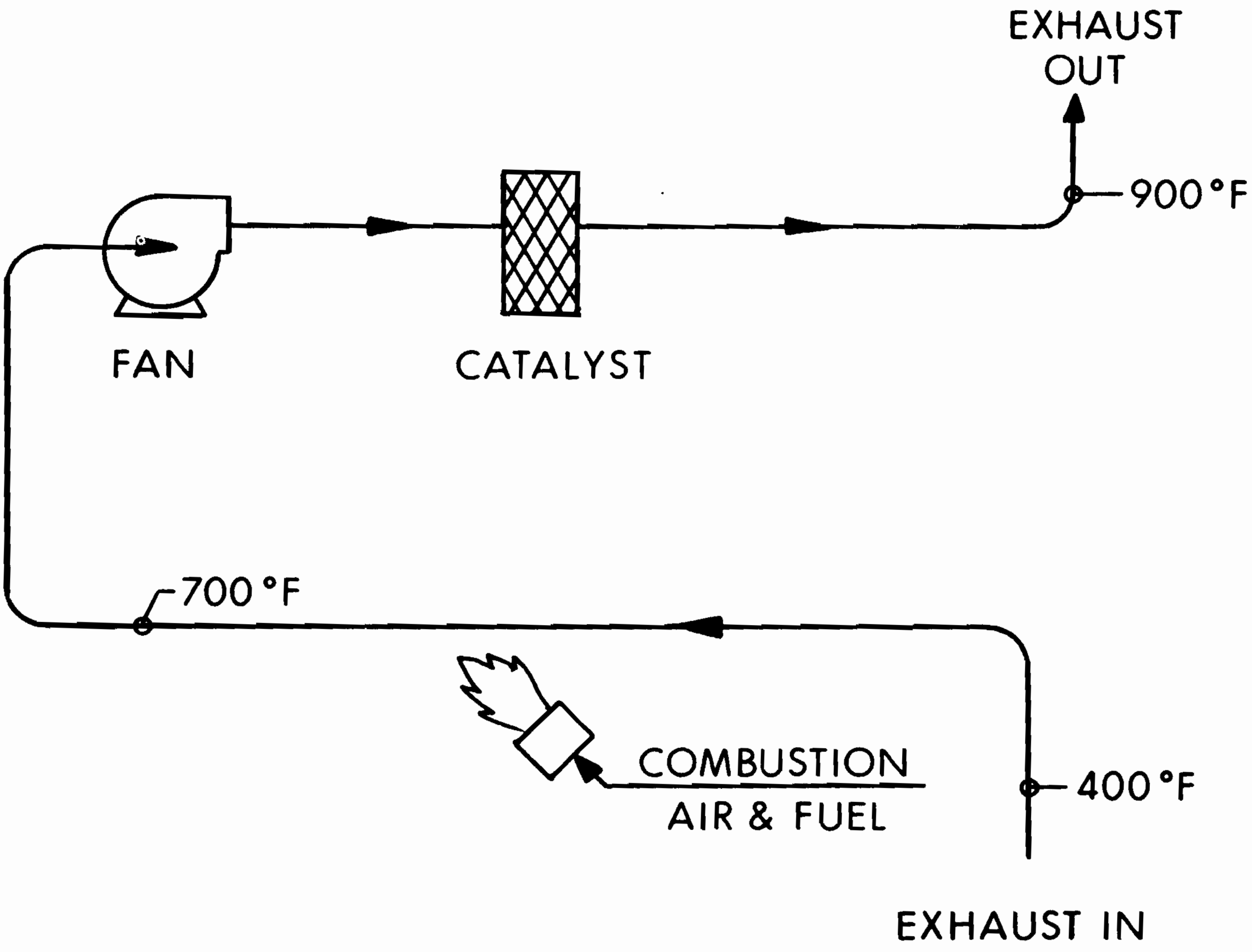


FIGURE 2. CATALYTIC INCINERATOR.

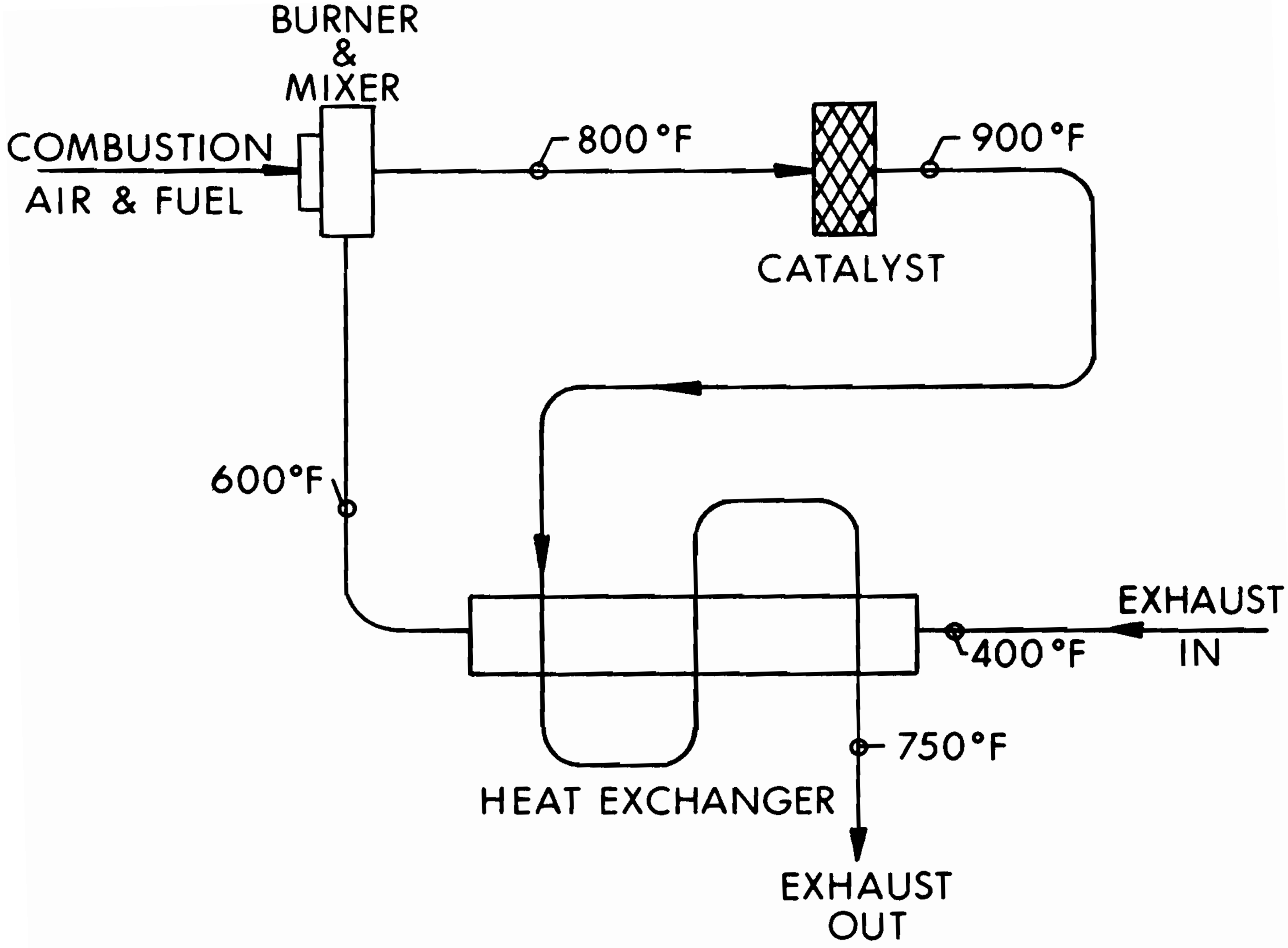


FIGURE 3. CATALYTIC INCINERATOR WITH HEAT RECOVERY.

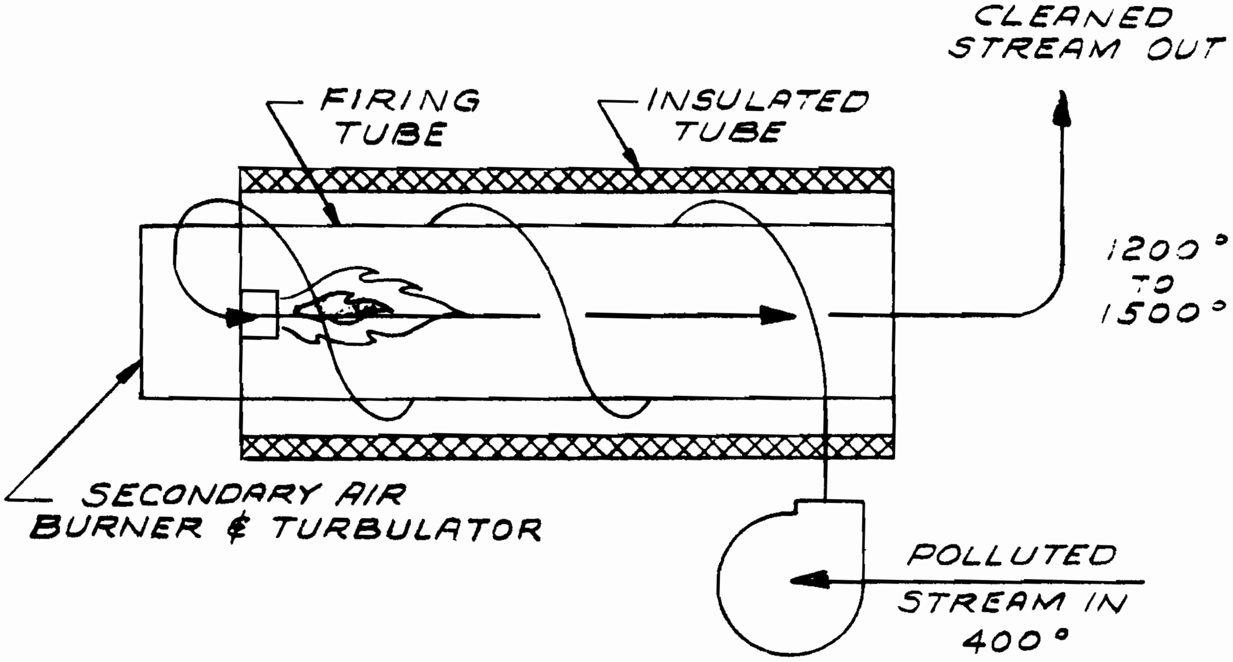


FIGURE 4. THERMAL INCINERATOR.

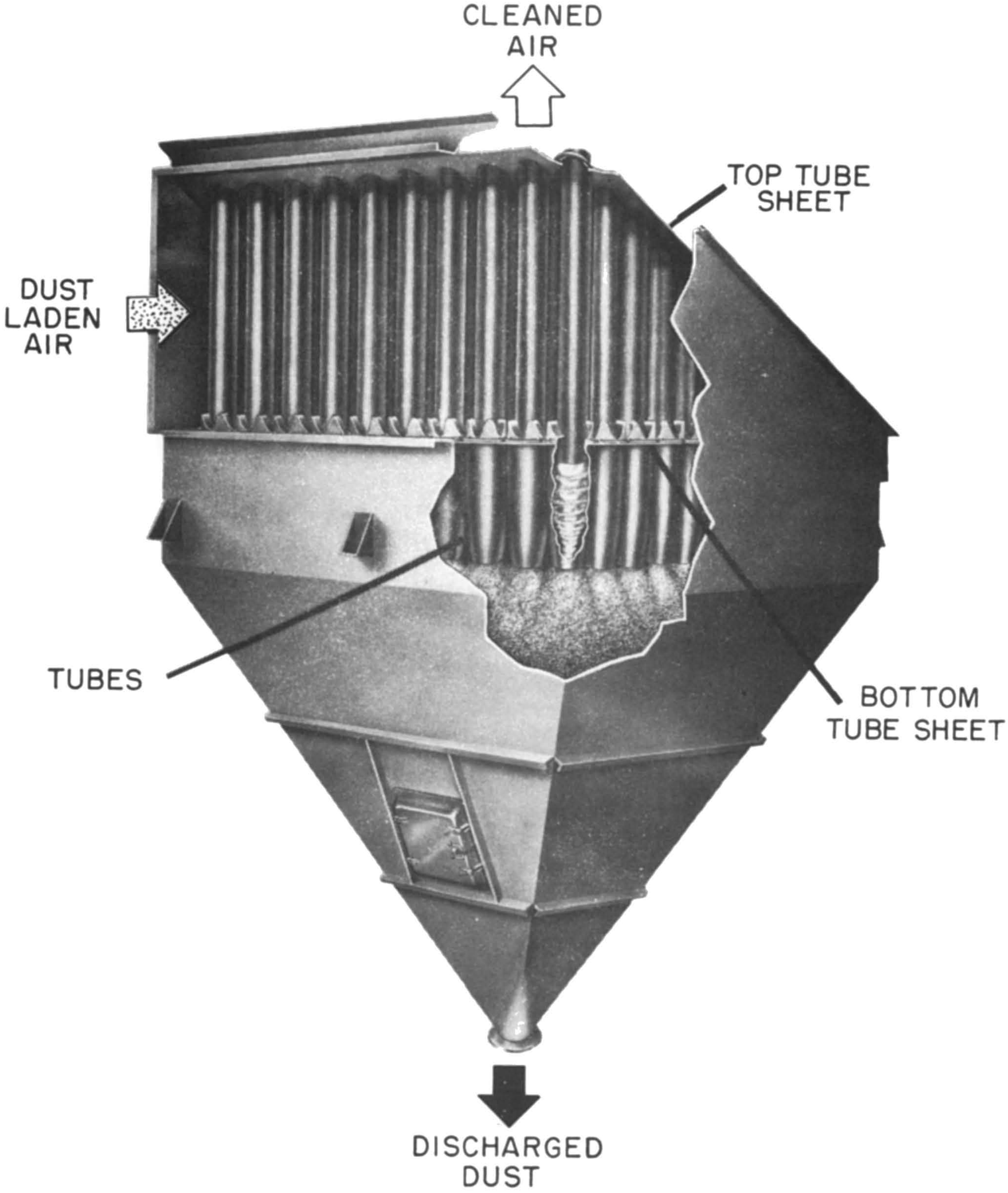


FIGURE 5. TUBULAR DUST COLLECTOR ARRANGEMENT.

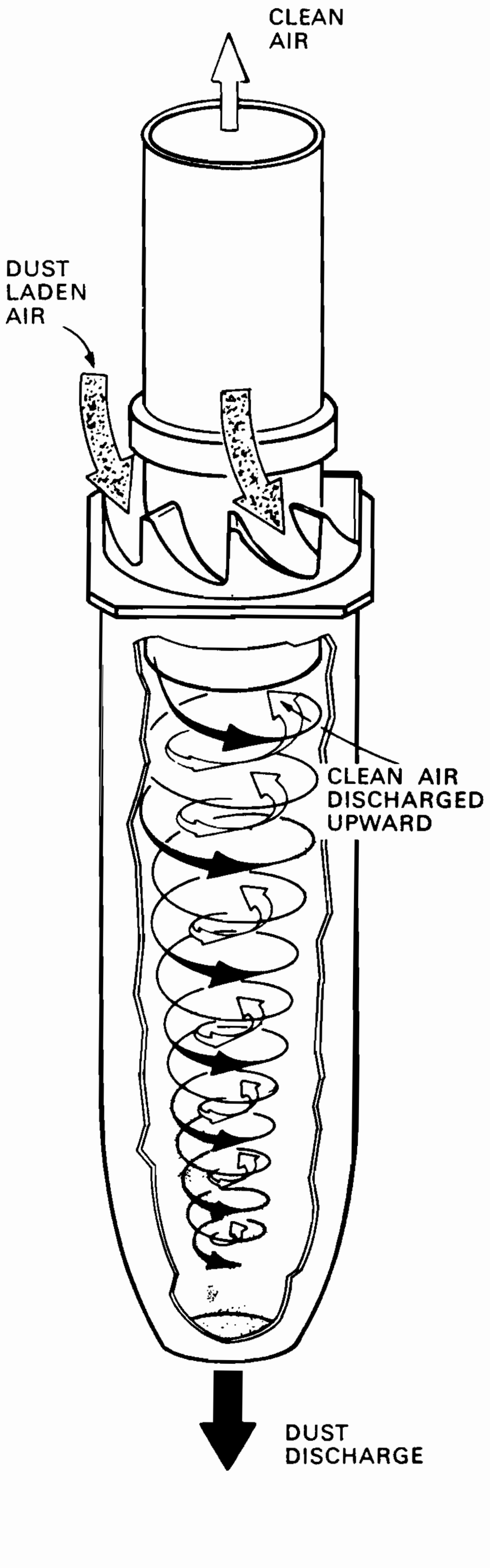


FIGURE 6. CUTAWAY OF DUST COLLECTOR TUBE.

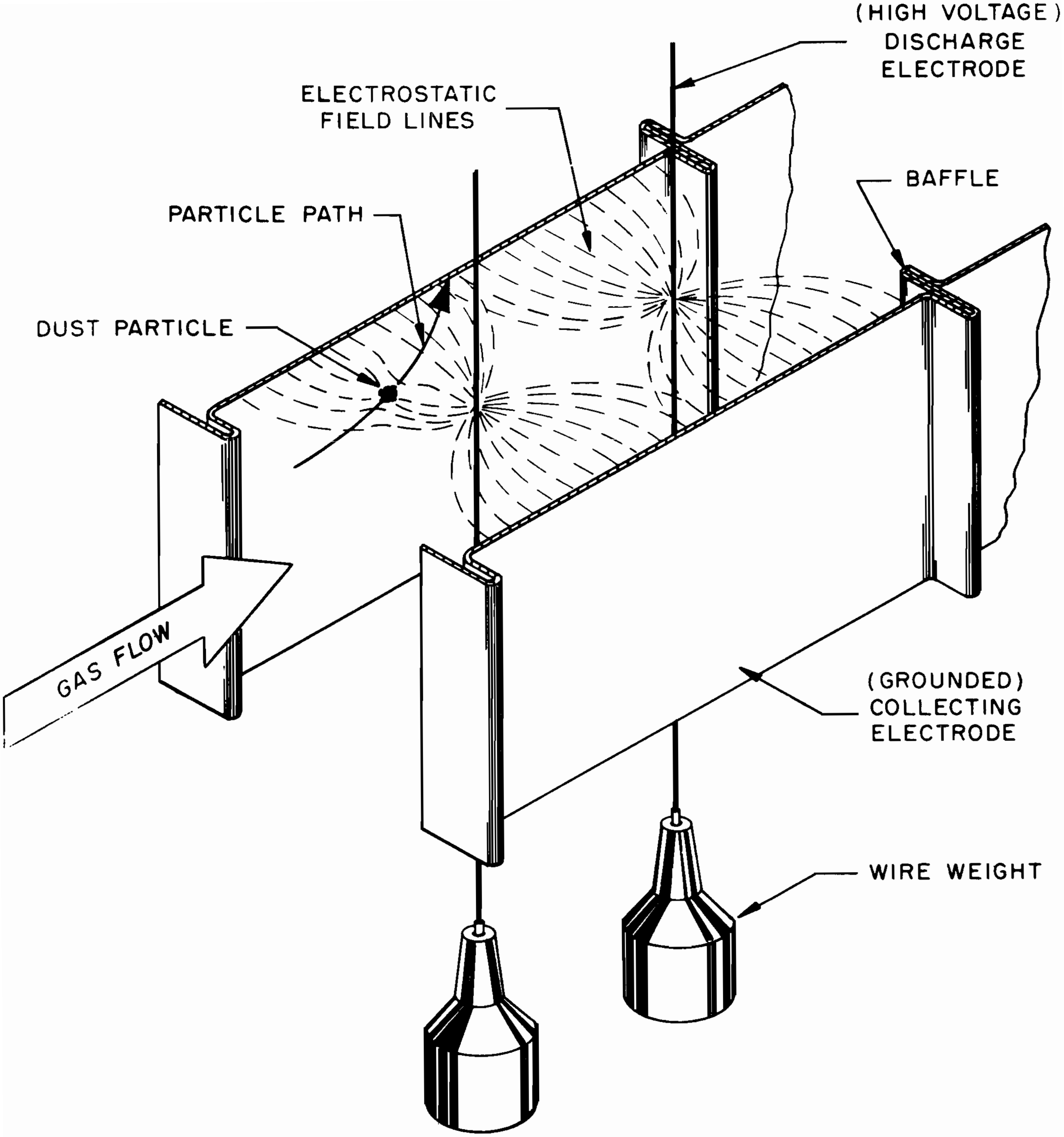


FIGURE 7. ELECTROSTATIC CHARGING OF DUST PARTICLES.

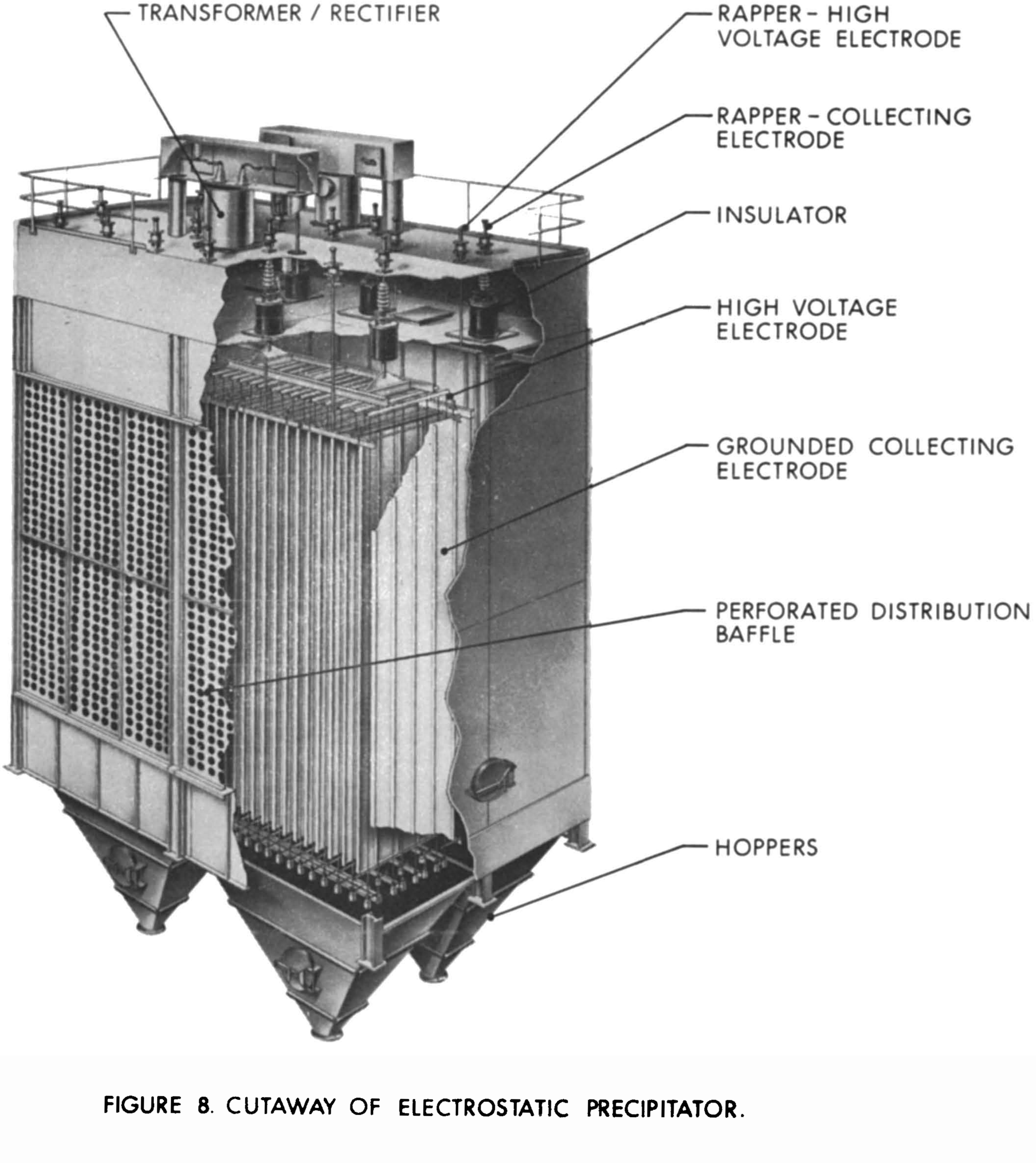


FIGURE 8. CUTAWAY OF ELECTROSTATIC PRECIPITATOR.

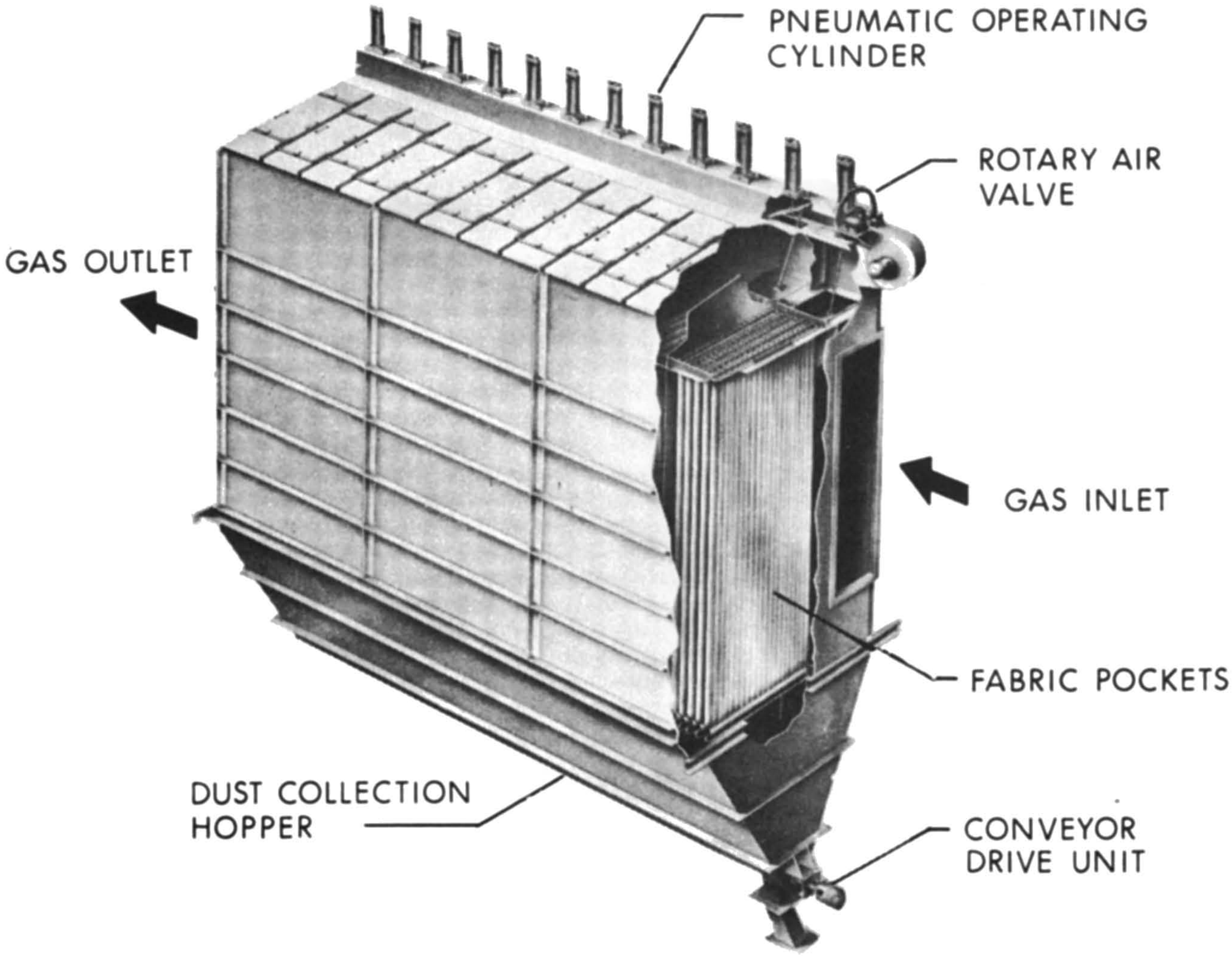


FIGURE 9. FABRIC FILTER.

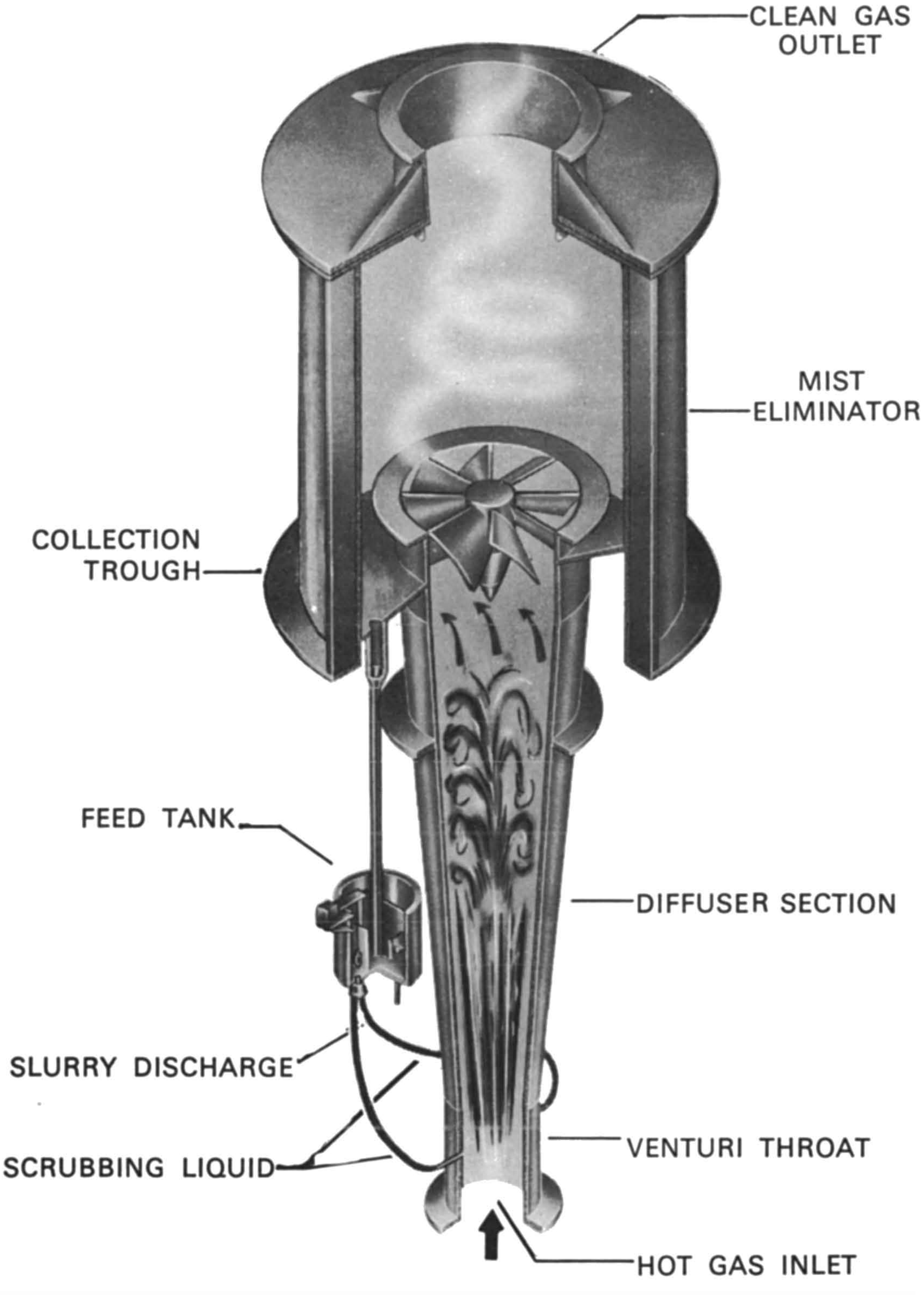


FIGURE 10. AEROMIX WET SCRUBBER.

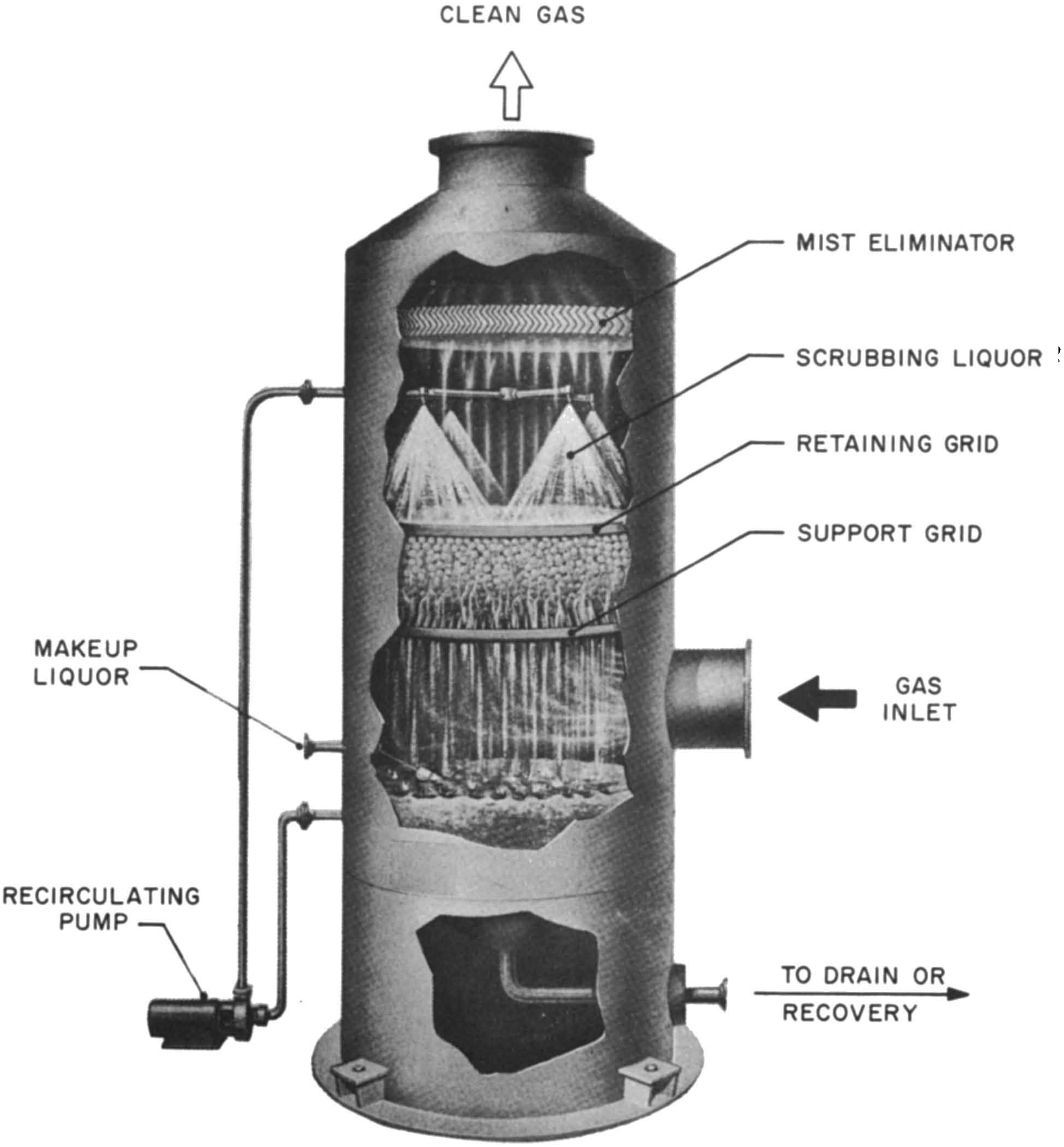


FIGURE 11. FLOATING BED WET SCRUBBER.

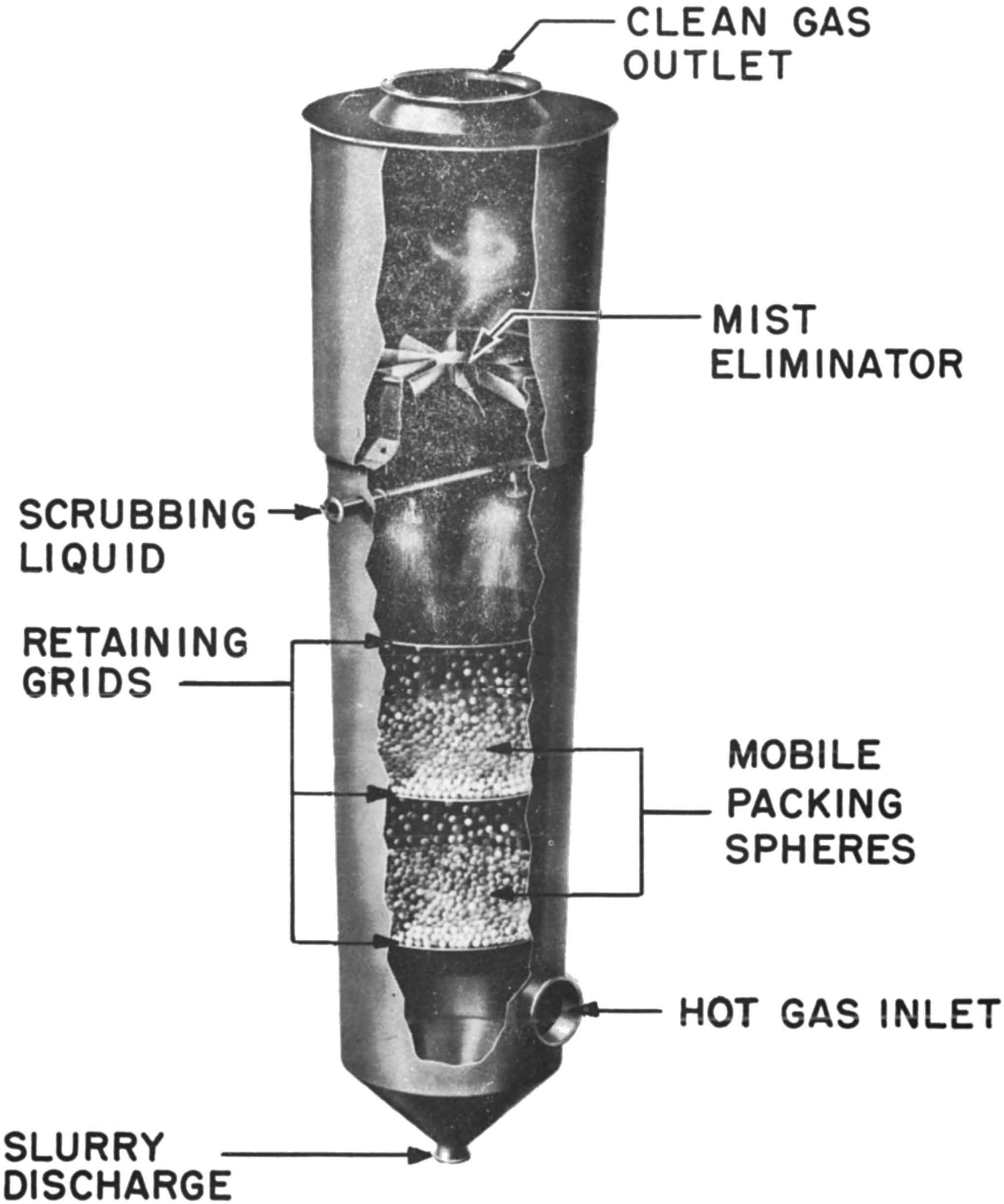


FIGURE 12. TURBULENT CONTACT ABSORBER WET SCRUBBER.

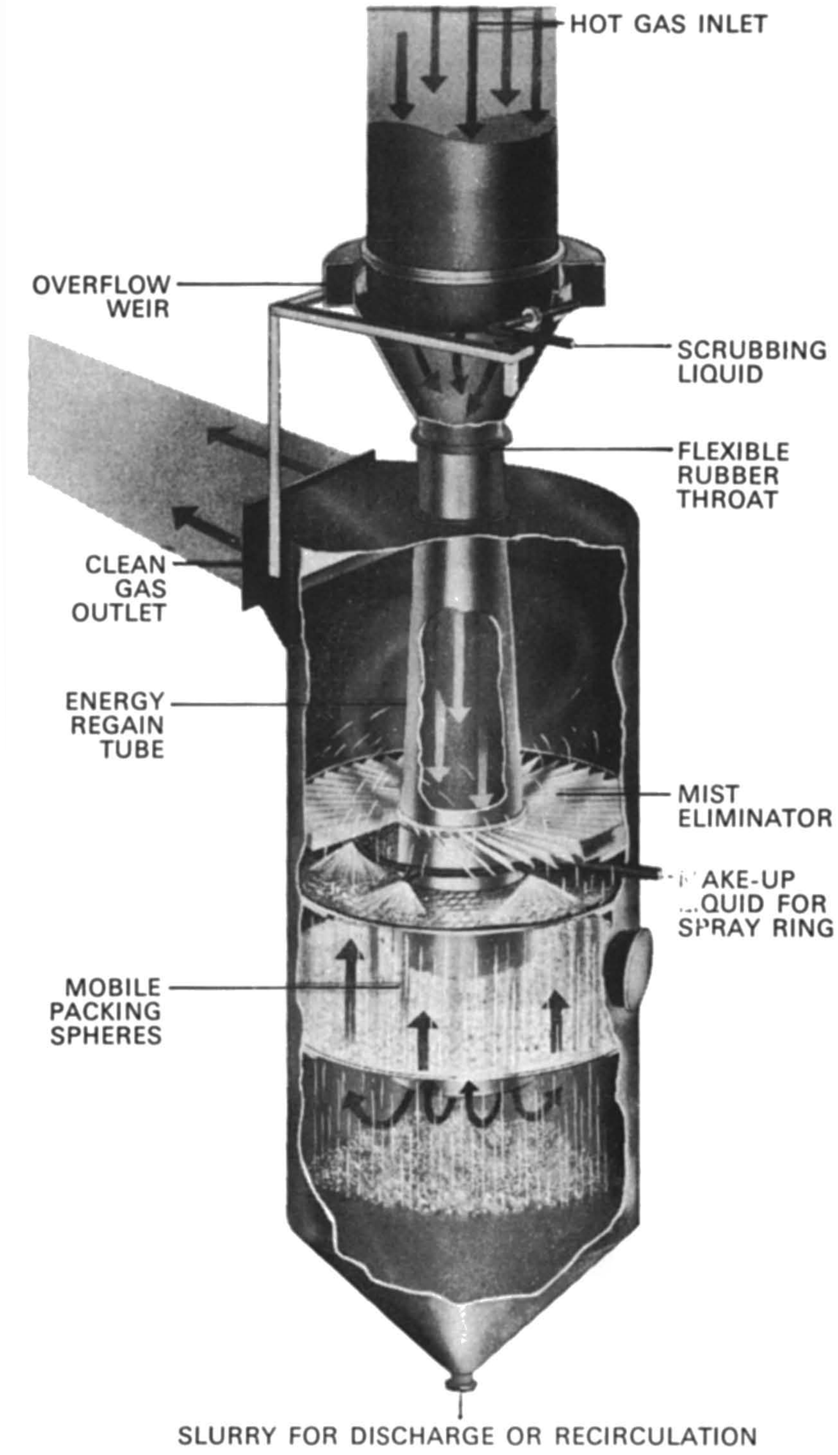


FIGURE 13. VENTRI-SPHERE WET SCRUBBER.

problems within the phosphate fertilizer industry, the engineer must have an understanding of the fertilizer manufacturing process and the experience of application of various control practices related to those processes.

We shall first review and define the types of pollutants normally associated with the manufacturing process of the phosphate fertilizer industry. Then we shall discuss briefly the role of air pollution legislation and codes and their effect on the design of plants for this industry. Lastly, we will discuss the principals of operation and the sources of pollution for the various mining and manufacturing processes in the industry. Along with this, some representative capital and operating costs will be presented for some typical plants and the effect of their costs relative to the product will be reviewed.

POLLUTION CODES AND REGULATIONS

In determining the type and extent of controls for air pollution, a prime factor is the existing and pending control legislation.

Since fertilizer plants are not all located in the same state and county, the air pollution codes which apply can very greatly in technical definition and stringency of control. In Florida, for instance, the codes state total emissions on fertilizer plants as pounds per day as F per ton of product on a P_2O_5 equivalent basis. Other states might class the fertilizer operation on a process weight basis with no distinction between industries other than processing and manufacturing.

The three examples below are taken from existing state codes and they show the extent to which these states define pollution emission levels for fertilizer plants. The amount or lack of definition may be due, in some part, to the number of plants within that state.

1) Fluorine Emission (3) Unit emissions of fluoride expressed as pounds of fluoride per ton of P_2O_5 equivalent shall not exceed 0.4 (four tenths) pounds.
2) Chemical Fertilizer Manufacturing Plant (4) The rules for emissions of particulate matters as listed in Rule 3-3.111 (a general ruling covering all process ventilation) shall apply to fertilizer plants as follows:
   (c) The total particulate matter emission from dryers and coolers shall not exceed either .05 grain per standard cubic foot or the standards for emission of particulate matter as listed in Rule 3-3.111.
   (f) The total amount of fume emitted from reactors, agglomerators, or any combination thereof, shall not exceed 2% of the total particulate matter allowed under Rule 3-3.111.
3) Contaminant Emissions From Processes and Exhaust Ventilation Systems. (5)
   187.3 Prohibitions. (a) No person shall cause, permit or allow the emission of air contaminants from an emission source resulting from an operation begun or modified, after the effective date of this Part, which exceeds the permissable emission rates specified in tables 2 and 3, for the environmental rating as determined in accordance with table 1.

The above three examples have shown the extent or lack of extent that different codes have taken to define the control requirements in relation to the fertilizer plant. However as some States take care of their more pressing sources of pollution they may start to be more specific concerning the emissions from the fertilizer plant.

FLUORINE ABATEMENT SYSTEMS

One of the most severe air pollution problems associated with the manufacture of phosphate fertilizers is the emission of fluorides that are contained in the fluorapatite type rock. These emissions are both gaseous fluorides as SiF_4 and HF as well as those fluorides contained in any dust that escapes. The fluorine content of most commercial rock is between 3.4 to 3.9% as F.

The gaseous fluorides are readily absorbed by scrubbing with water, provided the scrubbing device can create enough surface area on the scrubbing media, and provided there isn't any acceptance problem with the scrubbing media due to the presence of fluoride already in solution.

Removal of the particulates evolved however is more complicated because of the processes that generate these dusts. The ability of any dust scrubber to remove a range of particulates will be dependent upon the size of the particles and the energy expended across the device.

Most fertilizer manufacturing process will evolve both certain amounts of dust and gaseous fluorides, usually as siF_4. It then becomes important to select a scrubbing system that will provide the intimate contact between the (SiF_4) silicon tetrafluoride fumes and the scrubbing liquor while at the same time not be susceptible to plugging.

FLUORINE RECOVERY AND ABATEMENT

In many installations it becomes practical to recover the fluorides as hydrofluosilic acid for a process by-product. This recovery device cannot, however, be considered as pollution control equipment since it will evolve considerable amounts of fluorides as a result of the partial pressures over the percent acid being manufactured. Further scrubbing to remove fluorine is required. The venturi scrubber offers some distinct process and operational advantages over other devices, particularly when handling the recycled hydrofluosilic acid and the silica slurry.

1. It will have a high number of transfer units.
2. It will allow the use of a high-silica slurry content in the recycle liquids.
3. It is not susceptible to internal plugging.

Figure 3 on page 73 , shows a three stage scrubbing system on a ROP Triple-Super-phosphate den. This system is designed to concentrate to a 12 to 16% hydrofluosilic acid in the primary, 2 to 5% in the secondary and have an overall efficiency of 99%. The factors affecting fluorine removal are:

1. The inlet concentration as F.
2. The outlet or saturated gas temperature.
3. The amount of fluorides in the scrubbing water.
4. The number of transfer units associated with scrubber.

See Figure 1 on page 71 , for charts relating to fluorine removal. By proper use of these as governing criterias, it then becomes possible to design a scrubbing system to meet any pollution code requirement. In general, lower outlet temperatures will result in better Fluorine removal. Additional stages will increase overall efficiencies. Higher pressure drop can result in higher transfer units, but there is still much research work in this area before making any statement of how much or if the amount is significant to warrant the extra energy required.

ROCK DRYING

Within the mining operation of phosphate is the necessity of preparation of the rock either for shipment or consumption nearby. The material has a moisture content between 12 to 15% when it comes from the washing and screening operation and this is reduced to 1 – 2% in a process dryer.

This dryer is either of the Fluid Bed or Rotary types depending upon the processors operation or desire.

The type of pollutant associated with this operation is phosphate dust which is contained in the dryer exhaust gases. This dust can contain 3.4 – 3.9% Fluorine as F. This can result in as much as 85 – 100 lbs./day as F emitted in the dust losses for a 250 TPH dryer scrubber.

The amount of the pollutant will vary depending upon the type of rock being dryed and this will result in different pollution control requirements or results.

Florida pebble will result in outlet loadings twice those of concentrate blend when dryed collected and scrubbed in the same system.

Present pollution control methods are usually low drop scrubbers and there are some applications where the gases are exhausted direct from the cyclone collectors to the satisfaction of local authorities.

The present equipment being used is capable to reducing outlet loadings to between 0.08 to 0.15 grs./scf. (dry) for concentrate or Florida pebble.

Typical costs for a 250 TPH plant would be:

250 TPH DRYER (0.08 to 0.15 grs./scf. (dry)

Purchased Equipment:

Scrubber	**$ 25,000**
Fan with Motor	**12,000**
Pump	**2,000**
Accessories	**5,000**
Piping	**5,000**
Power Wiring	**2,000**
Erection	**16,000**
Foundations	**6,000**
Total Direct Capital Cost	**73,000**
Indirect Capital Costs 20%	**14,600**
	$ 87,600

Direct Operating Costs - $/year

Electricity	**$ 10,990**
Water Treatment	**4,390**
Maintenance	**7,570**
	$ 32,950

Total Annual Costs:

Direct Operating Cost	**$ 32,950**
Taxes and Insurance	**1,750**
Administrative Costs	**2,190**
Depreciation and Interest Cost	**16,650**
Total Annual Costs	**$ 53,540**
Total Production 250 TPH; **24 hrs./day; 330 days/yr.**	**1,980,000 tons/y**
Cost Per Ton	**$ 0.027/ton**

250 TPH DRYER (0.05 grs./scf. (dry)

Scrubber	**$ 30,000**
Fan with Motor	**25,000**
Pump	**1,500**
Ductwork	**6,000**
Accessories	**5,000**
Piping	**5,000**
Power Wiring	**6,000**
Erection	**17,000**
Foundations	**6,000**
Total Direct Capital Costs	**101,500**
Indirect Capital Costs 20%	**20,300**
	$121,800

Total Annual Costs:

Direct Operating Cost	**$ 42,260**
Taxes and Insurance	**2,440**
Administrative Costs	**3,040**
Depreciation and Interest Costs*	**22,900**
Total Annual Costs	**$ 70,640**
Total Production	**1,980 000 tons/y**
Cost Per Ton	**$ 0.035/ton**

The Scrubbing Equipment costs are based upon drying with gas as the primary combustion fuel with fuel oil standby. The equipment being carbon steel constructed with an interior protective coating of coal tar or other epoxy lining of 20 mils thickness.

If conditions or the customer's preference require it, the equipment can be rubber lined or fabricated entirely of stainless steel.

Scrubber and Fan prices would increase by 35% for RL and 65% for stainless steel.

The accessories include access/testing ladders and platforms, stub stack for mounting direct atop scrubber. Installation is assumed outside.

DEFLUORINATED PHOSPHATE ROCK

Defluorinated phosphate rock is produced by driving almost all of the fluorides contained in the rock off at a high temperature of 2400 degrees F. This material is used primarily as a feed for cattle and poultry.

Most of the product requirement specify a phosphate to fluorine ration of 100. With the amount of phosphorus in the product ranging between 18 to 21%, this will require 0.18 to 0.21% as F in the defluorinated rock. Feeding a rock having 3.9% as F will result in, for example, on a 500 TPD plant on loading to the scrubber of 37,200 lbs./day. With three stages of scrubbing, it will be possible to produce a fluosilic acid in the primary stage and reduce the total emission of 100 lbs. of fluorides per day.

CALCINED PHOSPHATE ROCK

The calcination of raw phosphate rock for product benefication is achieved in rotary or fluid bed calciners at temperatures of 1200 to 1800 degrees F. The amount of fluorides evolved will depend upon the temperatures of calcination and the type rock being fed to the calciner. Outlet loadings from calciners have varied between:

500 – 2000 mg/cu. ft. as gaseous fluorides
100 mg/cu. ft. as F contained in dust.

The gases are first treated by cyclone collectors where most of the air swept product from the calciner is separated from the gas stream and transported to a product cooler. The scrubber or scrubbing stages consist of a venturi or other similar high energy scrubber where the fine particulates and fluorides are removed. If it is desired to build up an acid concentration within the primary stage for purposes of by-product recovery, it will be necessary to add additional stages of scrubbing for fluorine abatement.

The scrubbers for these applications are usually construction of stainless steel over other corrosion resistant materisls due to the possibilities of loss of scrubbing water and a temperature run-away.

Typical costs for a calciner scrubbing system follow. These costs are for a single stage scrubbing system used for fluorine removal only. The scrubbing liquid is lightly recycled until the fluorine content reaches 3,000 – 5,000 ppm, the bleed rate is determined and the unit continues to operate at this rate.

Purchased Equipment:	
Scrubber	**$ 30,000**
Fan with Motor	**12,000**
Pump	**2,000**
Accessories	**5,000**
Stack - 100 ft.	**10,000**
Interconnecting ductwork	**5,000**
Piping	**5,000**
Power Wiring	**4,000**
Erection	**30,000**
Foundations	**8,000**
Total Direct Capital Costs	**111,000**
Indirect Capital Costs 20%	**22,200**
	$133,200

Direct Operating Costs - $/year	
Electricity	**$ 15,900**
Water Treatment	**9,500**
Maintenance	**5,250**
	$30,650

Total Annual Costs	
Direct Operating Costs	**$ 30,650**
Taxes and Insurances	**2,660**
Administrative Costs	**3,330**
Depreciation and Interest Cost*	**25,000**
	$ 61,640

THERMAL-PROCESS PHOSPHORIC ACID

Thermal Process Phosphoric Acid is a high purity acid that is used primarily for food, drug and detergents. A few plants produce fertilizer grade acid but they account for very little of the total output of these plants. However, should there accur any technical changes, increases in the cost and/or decreases in the supply of sulphur or a loss of markets for thermal process acid, due to decreased uses of phosphates in detergents this source of phosphoric acid might become more attractive for fertilizer manufacture.

The emissions from Thermal–Process Phosphoric Acid will occur as a phosphoric acid mist in the absorber exhaust gas.

WET-PROCESS PHOSPHORIC ACID MANUFACTURE

The manufacturing of wet-process phosphoric acid refers to a product made by the digestion of phosphate rock with sulphuric acid. The two principle products of this plant are a 30 – 32% P_2O_5 equivalent acid and the 54% P_2O_5 equivalent acid.

The ground phosphate rock and sulfuric acid is fed into a multi-compartment reactor or set of attack tanks. The digested rock slurry is fed to a filter where the gypsum is removed and the acid liquor is washed and filtered. The under-flow from these filter sections go to filtrate seal tanks as an intermediate step in the manufacturing process. This filtrate, or filter grade acid is our 30 – 32% P_2O_5 equivalent which is used for both a fertilizer blend raw material or 54% acid system feed. The chief sources of pollution are a result of the vent cooling of the reactors and venting of the filter and seal tanks. The type of pollutant will be gaseous fluorides with concentrations verying from 70 to 100 mg./cu. ft. Fluorine recovery with inlet loadings this low is not practical at today's market for fluorides. Therefore, the fume scrubbers will be for pollution abatement only.

A number of different wet scrubber types have been successfully installed on wet acid plants, however in each case, a single stage system would not be sufficient to meet even the most lenient code.

The filter grade acid at this part of the process will still have 2.5 – 3.0% Fluorine.

The acid filtrate then proceeds to the evaporate section where it is concentrated to the54% P_2O_5 equivalent acid. This 54% acid will contain 1.25 to 1.50% Fluorine with the difference being evolved in the three (3) stages of evaporation. These evolved fluorides are stripped out in barometric condensers as a fluosilicic acid. Pollution abatement for the evaporator section is provided as a part of the evaporators and is not the concern of the plant or air pollution engineer.

For a 650 TPD* wet-process acid plant, the ventilation rate would be 25,000 to 35,000 CFM depending on the process equipment ventilated. For a typical flow sheet see Figure 2 on page 000.

Typical costs for a fume scrubbing system on a 650 TDP plant would be:

Purchased Equipment:	
Scrubber	$ 14,000
Pre-scrubber	2,000
Ductwork	3,000
Fan	7,000
Pump	1,000
Stack	6,000
Accessories	2,000
Piping	5,000
Power Wiring	3,000
Erection	8,000
Foundation	5,000
Total Direct Capital Costs	56,000
Indirect Capital Costs 20%	11,200
	$ 67,200
Direct Operating Costs - $/year	
Electricity**	$ 1,800
Water Treatment	750
Maintenance	1,875
	$ 4,425
Total Annual Costs:	
Direct Operating Cost	$ 4,425
Taxes and Insurance	1,345
Administrative Costs	3,450
Depreciation and Interest Costs*	13,450
	$ 22,670
Total Production 650 TPD 330 days/year	214,500 tons/yr.
Cost Per Ton	$ 0.0945/ton

NORMAL SUPER-PHOSPHATE

Normal super-phosphate is a product with a P_2O_5 equivalent of 16 to 22% and about 45% calcium sulphate. Its low phosphorus content limit the distribution area for each plant and the capacity of each plant is usually low depending upon the consumption within its economical marketing radius.

Normal super-phosphate is produced by acidulation of dried, ground phosphate rock with sulfuric acid in a batch or continuous den. Most familiar are the Sturtevant den used on newer batch processes and the Broadfield den on the continuous processes. After the den the partially reacted material is fed to a cutter and the cuttings are then sent to a curing facility where the remainder of the materials react.

Efforts have been made to reduce the curing time and eliminate the curing facilities required but these either are too costly or result in a product with too high of an acid content.

The chief pollutant from the manufacture of normal superphosphate is the gaseous fluorides evolved in the reaction of the rock and acid. The amount of fluorine evolved will range from 40 to 70% of that contained in the rock, and thiss will depend upon the type of rock and its reactivity, the fineness of the rock feed and the ratio of acid/rock employed. Usually an attenpt to achieve the higher P_2O_5 equivalent will result in greater releases of fluorides.

The emission points of the evolved fluorides are:

The acidulating cone or tower
The pan mixer or pug mill
The den, either enclosed or continuous
The cutter
Transfer points or conveyors
Curing and storage building.

Many of these normal super plants employ in-plant design scrubbers consisting of a baffled spray tower constructed of redwood. Some plants recycle in a portion of these units to concentrate a fluosilic acid. This type unit requires occasional cleaning and dredging to remove silicia deposits.

A typical mechanical den plant has a capacity of 30 to 40 tons per batch and can produce up to 12 batches per day. Since it is a step process, the amount and location of the emissions will vary greatly over the 2 hour period of the cycle. The manufacturer can produce just enough to meet demand and, therefore, the number of batches per day can vary accordingly.

The maximum fluorides evolved from such a plant running at maximum capacity and producing a 20% analysis product can be as high as 22,500 lbs./day if 60% of the fluorides are evolved. An analysis of the feed and product can actually determine the amount of fluorides evolved. Because of the open design of these plants, it is impossible to capture, in the fume exhaust system, all the evolved fluoride and therefore it becomes impractical to design a fume scrubbing system without further considerations of overall effects of the escaping fluorides.

Since it is the purpose of control regulations, in regard to fluorides, to control the ground concentrations of fluorine it would be more practical to prorate the emissions from the batch operations over the producing periods for the plant. It will also become necessary to determine the amount that escapes to that which is capable of being scrubbed in order to determine actual total emissions and whether they can meet the code requirements.

A 40 ton batch plant (20 TPH) will have a fume exhaust rate of 18,500 to 27,500 ACFM for the mixing, denning and cutting operation. Rough estimates on the percent of fluorides evolved during these operations is between 30 to 50% of the total amount evolved. The remaining 50 to 70% is released during the curing period. Since the fluorides require the presence of water vapor to affect evolving most of the fluorides released will occur when the cutter shaves the block of super releasing the excess moisture. Because of the inability to effectively enclose and hood this cutting operation, about 20% of the fluorides evolved in mixing, denning and cutting escape. This results in 100 to 200 mg/cu. ft. as F loadings to the scrubbing system. Loadings as high as 2000 mg/cu. ft. can be found in the mixing duct, however with the low ventilation rate, it becomes diluted with the larger volumes from the den and cutter areas. The remaining fluorides released during curing will exhaust in the curing building ventilation system which should have a fume scrubber even though these amounts are emitted over a long curing period.

The fume control system for both the den and curing building in a typical plant would consist of two scrubbers, one for each system. The scrubber for the den could be a two-stage unit if it is desired to recover fluorine as a byproduct. If there is too much particulate in the gas stream, it might be desirable to knock these out before recovering the fluorine. This can best be done by impinging the gases on the surface of a concentrated acid tank (20% H_2SiF_6 at 170 degrees F.). It can be followed by the two stage scrubbing system.

The curing building should have its own separate system. A typical ventilation rate for these buildings would be 30,000 to 50,000 CFMair depending upon the size building.

Typical costs of fume scrubbing systems for both the den and curing building would be:

Scrubbers - Den (2 stage)	$24,000	
Curing		$18,000
Ductwork - Den	5,000	
Curing		10,000
Fan - Den	6,000	
Curing		7,000
Pumps - Den	2,000	
Curing		1,000
Stacks - Den	6,000	
Curing		8,000
Accessories - Den	$ 1,000	
Curing		$ 1,000
Piping - Den	2,000	
Curing		2,000
Power Wiring - Den	3,000	
Curing		3,000
Erection - Den	15,000	
Curing		15,000
Foundations - Den	8,000	
Curing		6,000
	$70,000	$71,000
Total Direct Capital Costs	$142,000	
Indirect Capital Costs 20%	28,400	
Total Capital Requirements	$170,400	

Since these plants operate to meet local market demands, the period of operation over which the cost of pollution control equipment will be applied will vary greatly. Therefore, the annual cost below applies only to the fixed annual costs, and variable cost of operating on a ton basis are listed separately.

Total Annual Fixed Costs:		
Taxes and Insurance	$ 3,410	2.0% of capital costs
Administrative Costs	4,260	2.5%
Depreciation and Interest Costs	32,000	18.8%
	$ 49,670	

Varying Direct Operating Cost $/100 Tons	
Electricity	$75.00
Water Treatment	8.00
Maintenance	4.00
	$87.00/100 Tons

The manufacture of normal super-phosphate in a continuous den, such as the Broadfield, will result in similar emissions from the various steps; however, the capturing of these fumes will be more positive since the total operation can be enclosed more closely.

RUN-OF-PILE TRIPLE SUPER-PHOSPHATE

Run-of-pile triple super-phosphate is a 46% equivalent P_2O_5 produced continously by acidulation of phosphate rock with phosphoric acid. This process evolves considerably more fluorides than others, however since it is done in an almost totally enclosed or enclosable system, the control of fluoride emission is more easily accomplished. The sourses of fluoride emission are:

> The TVA mixing cone, the continuous slurry or during belt, the cutter and the storage. Each of these systems can be enclosed easily (during building).

The system in Figure No. 3 is a typical ROP flow sheet. A typical system for a 1000 TPD, as P_2O_5 ROP triple superphosphate consists of a scrubbing system for the reactor den belt and a separate system for the storage (curing) building. Also shows a three stage fluorine recovery and abatement system on such a process.

The cost of such a system with three stages of scrubbing with a ventilation system for a 2,250,000 cu. ft. storage building would be:

	Reactor/ Den/Belt 40,000 CFM	Storage (Curing) Building 300,000 CFM*
Purchased Equipment:		
Scrubbers-Venturi (RL)	$ 14,000	$ 105,000
Cyclonic (RL)	16,000	
Packed Tower (FRP)	15,000	
Fan	11,000	36,000
Pumps	3,000	3,600
Ductwork	10,000	45,000
Stack	12,000	15,000
Accessories	5,000	27,000
Piping	8,000	15,000
Power Wiring	4,000	15,000
Erection	60,000	100,000
Foundations	20,000	30,000
	$178,000	$391,000

Direct Operating Costs - $/year		
Electricity	$ 15,650	41,750
Water Treatment	18,250	36,500
Maintenance	3,000	22,500
	$ 36,900	$ 100,750
Total Annual Costs		
Direct Operating Costs	$ 36,900	$ 100,750
Taxes and Insurance	3,460	6,820
Administrative Costs	4,325	8,525
Depreciation and Interest Cost	33,400	73,500
	$ 78,085	$ 189,595

* Based upon a change of air every 7 to 7.5 minutes when the building is empty.

AMMONIUM PHOSPHATES

Monoammonium Phosphate (MAP) 11-48-0

Diammonium Phosphate (DAP) 18-46-0

The production of ammonium phosphates, although they may vary in technique, are essentially the same principle. Phosphoric acid and anhydrous ammonia are reacted to produce the ammonium phosphate material. DAP production in the United States utilizes for most plants the TVA ammoniator-granulators where the reaction and granulation take place in the same drum. These units can also be used for the addition of potash salts to manufacture NPK materials.

The crystallized material from the ammoniator granulator is then dried, cooled, washed and screened to a finished product.

If the plant is used strictly for the manufacture of DAP only, then the pollution problem is associated chiefly with release and recovery of ammonia fumes from the reactor, dryer and cooler.

The recovery of these ammonia fumes is done by scrubbing with 96% phosphoric acid. However, a number of problems arise when reacting and scrubbing with various grades of acid; particularly the filter grade (25 – 30% P_2O_5) and the 40% P_2O_5 evaporator grade acid. Gaseous fluorides released during the reaction necessitate the need for a tail gas scrubbing system for fluorine abatement.

Since the scrubbing of ammonia fumes with an aqueous acid involves some chemical reactions there also arise problems of build-up and deposits of fertilizer materials within the scrubbing system. Also, the amount of solids in the scrubbing acids will require a method of introducing the scrubbing media without clogging of the injection system.

Over the years, a number of different types of scrubbing devices have been applied to the recovery of ammonia quite successfully. However, in the past few years, the venturi scrubber has found a wider acceptance for these applications. Because of its inherent design and operating principles, it affords high ammonia recoveries without the problems of build-up and plugging. It can also utilize the entrainment separator as a cyclonic scrubber for further ammonia recovery.

The arrangement of ammonia recovery systems for a typical DAP plant will depend a lot of the operational desires of the plant engineers. However, for the most flexible operation, it is recommended that separate fume scrubbing equipment for the dryer, cooler and screens, and ammoniator-granulator be provided. On the system shown, all of the gas streams are combined into a single tail gas scrubber for fluorine abatement.

Typical costs for an ammonia recovery and fluorine abatement system on a plant producing 1200 TPD of 18-46-0 analysis DAP Equipment Costs:

Equipment Costs:	
Dryer Scrubber	$ 16,000
Cooler Scrubber	14,000
Screens & Mill Scrubber	14,000
Ammoniator-Reactor Scrubber	10,000
Fluorine Abatement Scrubber	30,000
Dryer Fan	11,000
Cooler Fan	10,000
Dust Fan	10,000
Fume Fan	8,000
Recovery Tank	4,800
Pump	2,500
Tail Gas Scrubber Pump	2,000
P_2O_5 Piping	6,000
Pond Water Piping	6,000
Stack 9' x 50'	12,000
Accessories	19,000
Power Wiring	18,000
Instrumentation	10,000
Ductwork	36,000
Flushing Piping	4,000
Utilities	4,000
Erection	100,000
Foundations	53,000
Total Direct Costs	395,000
Indirect Costs 20%	79,000
	$474,000

Direct Operating Costs - $/year	
Electricity	$33,700
Waste Treatment	23,100
Maintenance	17,250
	$74,050
Total Annual Costs:	
Direct Operating Costs	$ 74,050
Taxes and Insurance	9,480
Administrative Cost	11,830
Depreciation and Interest Costs	89,000
	$184,360
Annual Production	
1200 TPD x 330 D =	396,000 tons/year
Cost Per Ton	$ 0.465/ton (as P_2O_5)

NPK FERTILIZERS

The different grades of fertilizers it is possible to produce with the continuous ammonia-phosphoric acid plants is very important to the local agronomy of that plant. One of the products of this diversified capabilities is the NPK grade fertilizers. These are produced in much the same way as the ammonium-phosphates, but with the addition of potash salts to the ammoniator-granulator.

The location of these plants and the economies of their ammonia and material recovery, along with the air pollution control, requires extensive design considerations to satisfactorily meet the requirements of each.

The major source of pollution with this plant is gaseous ammonia, amonium chloride and fertilizer dust. The ammonium chloride NH_4Cl is a fine sub-micron fume which requires high energy venturi scrubbing. The venturi will permit the recycling of process solutions and thereby minimizes overall water consumption. Since the material can be recycled, it is possible to build up slurries and use these as process slurries for particular grades. However, it is not always possible for particular grades to consume the concentrated slurry. In these instances, it has been found feasible to have a holding tank to store the slurries untill they can be used. A typical system is shown in Figure 4. The equipment normally vented in such a plant would be the ammoniator, dryer and cooler.

The cost of a recovery and pollution control system for such a plant would be:

Venturi	$ 19,000
Cyclonic	12,000
Fans VS	11,000
CS	8,000
Stacks	22,000
Recycle Tanks	3,000
Storage Tanks	8,000
Ductwork	15,000
Accessories	10,000
Piping	20,000
Power Wiring	10,000*
Pumps (3)	4,500
Instrumentation	10,000
Slurry Treatment	5,000
Erection	30,000
Foundations	20,000
Engineering	10,000
Direct Capital Costs	$213,500
Indirect Costs 20%	42,700
Total Capital Costs	$256,200

* In many plants, new service entrance cable might be required.

TOTAL ANNUAL COST

To determine the Total Annual Cost, a number of direct operating cost factors had to be established. The cost of electricity, water treatment and maintenance are the main factors in the direct operating costs and these figures will vary from plant to plant.

The following figures are presently those being used by NSPCA for determining costs on the various pollution control studies it is conducting:

Electricity	Fixed	Operating	Total
$KWHr	$0.0018	$0.0082	$0.010
Waste Treatment:			
$/1,000 gal.*	$0.04	$0.01	$0.05
$/1,000 gal.**	$0.08	$0.02	$0.10

* This is based upon a system of pond settling where there is little or no lime treatment since the availability of land for ponds is abundant, as is the case in Florida.

** This figure is to be used where there exists a necessity to treat the settling ponds quite extensively since there is a lack of pond capacity.

The piping costs related to the scrubbing system are to within 50 feet of the unit. The remainder of the cost of delivering these effluents to the water treatment facility and back are assumed to be part of the fixed cost of the waste treatment cost figure.

TAXES AND INSURANCE

The local property tax figures in these cost sheets is based upon a 50% assessment and a 30 mils rate. This results in an annual tax rate of 1.5% of Capital Costs. An average insurance cost of 0.5% of the Capital Costs was used.

Total Taxes and Insurance -- 2.0% of Capital Costs.

ADMINISTRATIVE COSTS

This is an average cost that applies to the overall plant. It includes all salaried personnel, legal and professional services, and office supplies and expenses.

DEPRECIATION AND INTEREST COSTS

At present, the IRS allows a depreciation write-off for certified air pollution control facility over a period of eight (8) years. However, the tax reform bill, Paragraph 168, proposed a rapid depreciation for certified pollution control facilities over a period of 60 months.

For this paper, it is assumed that an allowable depreciation period of 8 years is in effect and that the interest rate is at 10%. Based upon this, the Depreciation and Interest Costs will be 18.8% of the Total Capital Requirements.

INDIRECT CAPITAL COST

The following is a breakdown of the indirect capital cost:

10%	Contingency
6%	Engineering
1%	General Construction Overhead
1%	Start-Up Cost
1%	Spare Parts
1%	Purchasing Costs and Sales Taxes***

20%	TOTAL

***In many localities and states, there is no sales tax on pollution control equipment.

SUMMARY

As the need for pollution control increases, with the desire on our part to retain a liveable environment, the need for defining the pollutants from any process and a way of effectively controlling these pollutants will become more important.

It will be the role of the Pollution Control Manufacturer to assure and guarantee to the user that the system will meet the code. No longer can he state that the unit will remove 90 to 99% of the contaminants and have the plant engineer determine that this is sufficient to meet the code.

The equipment manufacturer's pollution engineer will have to determine either by

process weight and loss calculation
field testing and/or
pilot plant studies

the nature of the pollution and the most effective control system.

If not properly approached, the selection of air pollution control can create an operational and maintenance burden, involve large capital expenditures and may become obsolete by new and stricter codes.

LITERATURE CITED:

1. Teller, A.J. – Chem. Eng. Progr., Vol. 63, No. 3, p. 78 (March, 1967)
2. Huffstutler, K. K., Starnes, W. E. – Journal Air Pollution Control Assoc., Vol. 11, No. 12 (December, 1966)
3. Rules of the Hillsborough County (Florida) Pollution Control Commission – Chapter 1–3,01–3
4. State of Illinois – Rules and Regulations Governing the Control of Air Pollution
5. State of New York – Chapter IV – Air Pollution Control

Although not specifically cited, but of extreme value in providing background material for this paper, were the books and articles listed below. Also of considerable value were the comments of the engineers and consultants with specific knowledge within the phosphate fertilizer industry.

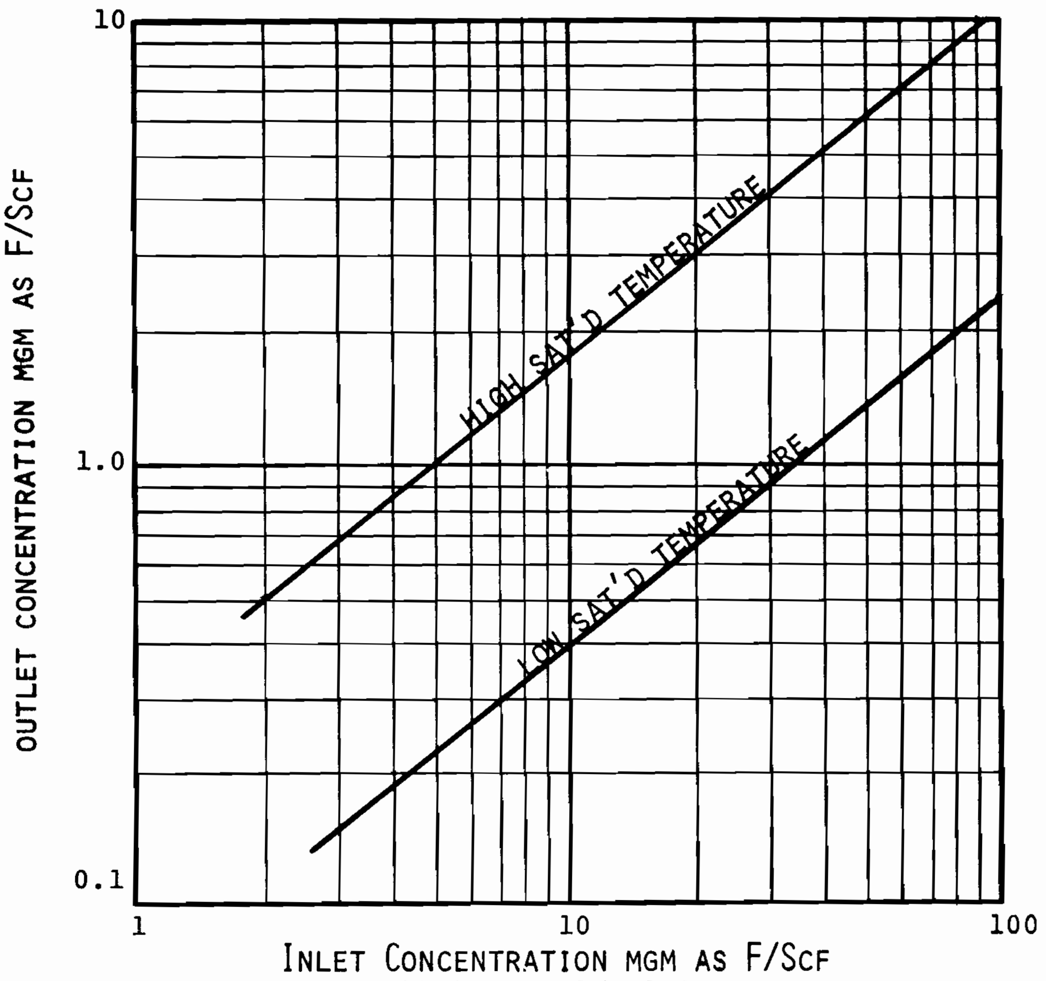
a. Phosphatic Fertilizers – The Sulphur Institute
b. New Developments in Fertilizer Technology – National Fertilizer Development Center – TVA 6th Demonstration (Oct. 4–5, 1966)
c. Gaseous Fluorides Air Pollutants from Stationary Sources – R. C. Specht, R.R. Calaceto, AICHE 59th Annual Meeting (Dec. 4–8, 1966)
d. Pollution Control in a Phosphoric Acid Plant, Chem. Eng. Progr., Vol. 60, No. 1, pages 53-55 (Jan, 1964)

I would like to thank Occidential Corporation of Florida and Agrico Chemical Company, particularly Messrs. Maywood Chesson and Allen Jaeggi for permission to use pictures of their installations.

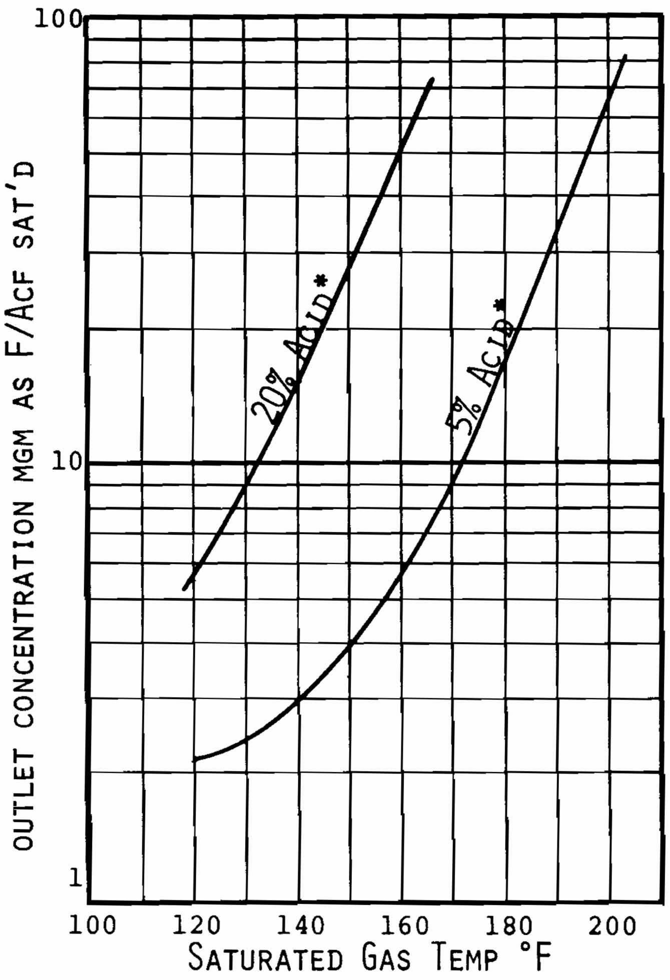
(See Figures 1, 2, 3 & 4 Pages 71, 72, 73 and 74)

MODERATOR HECK: Thank you Larry, for your thorough description on awareness and involvement management must fully understand when considering installation of pollution control equipment. I would like to announce if any of you need some additional information on the Four Papers discussed by Messrs. Renninger, Cox, Tomany and Hill, don't hesitate to contact these fine gentlemen. They will probably remain with us during the entire meeting and will be glad to answer your questions. I will now turn the meeting back to Al Spillman who has several announcements to make before we adjourn for lunch.

CHAIRMAN SPILLMAN: I will hold you for a few minutes. We will reconvene this afternoon at 2:00 o'clock. The first part of the program will be the business meeting; Secretary-Treasurer report; Nomination of the new expanded Executive Committee; Discussion on the possibility of moving our 20th Annual Meeting to another location; future proceeding printing costs and how to handle; best type of subject matter for future meetings and other pertinent data pertaining to the operation of this organization. This session should be of great interest to all of you and I am looking for a 100% attendance. The remainder of our afternoon program will be a very interesting panel on "Formulation". Mr. Grant C. Marburger, Panel Leader and Associate Panelists Jon. L. Nevins and Frank P. Achorn (paper will be given by Nevins), John T. Hailey, Robert R. Heck, R. H. Perkins and Merideth Morris. Billy E. Adams will moderate. Thank you. Meeting adjourned at 12:15 o'clock p.m.



FLUORINE SCRUBBING
WITH
LOW FLUORINE CONTENT LIQUOR



FLUORINE SCRUBBING
WITH
HYDROFLUOSILIC ACID
*% as H_2SiF_6

FLUORINE REMOVAL TRANSFER UNIT FORMULA (1)

$$Nt = \ln \frac{Y_1 - Y_a}{Y_2 - Y_a}$$

N_t = Number of transfer units

Y = Concentration of fluoride in gas
1 - at inlet
2 - at outlet
a - content based upon gas phase equilibrium with concentration of fluoride in scrubbing liquor in concentrations below 5,000 ppm as F this effect is neglected.

FIGURE 1

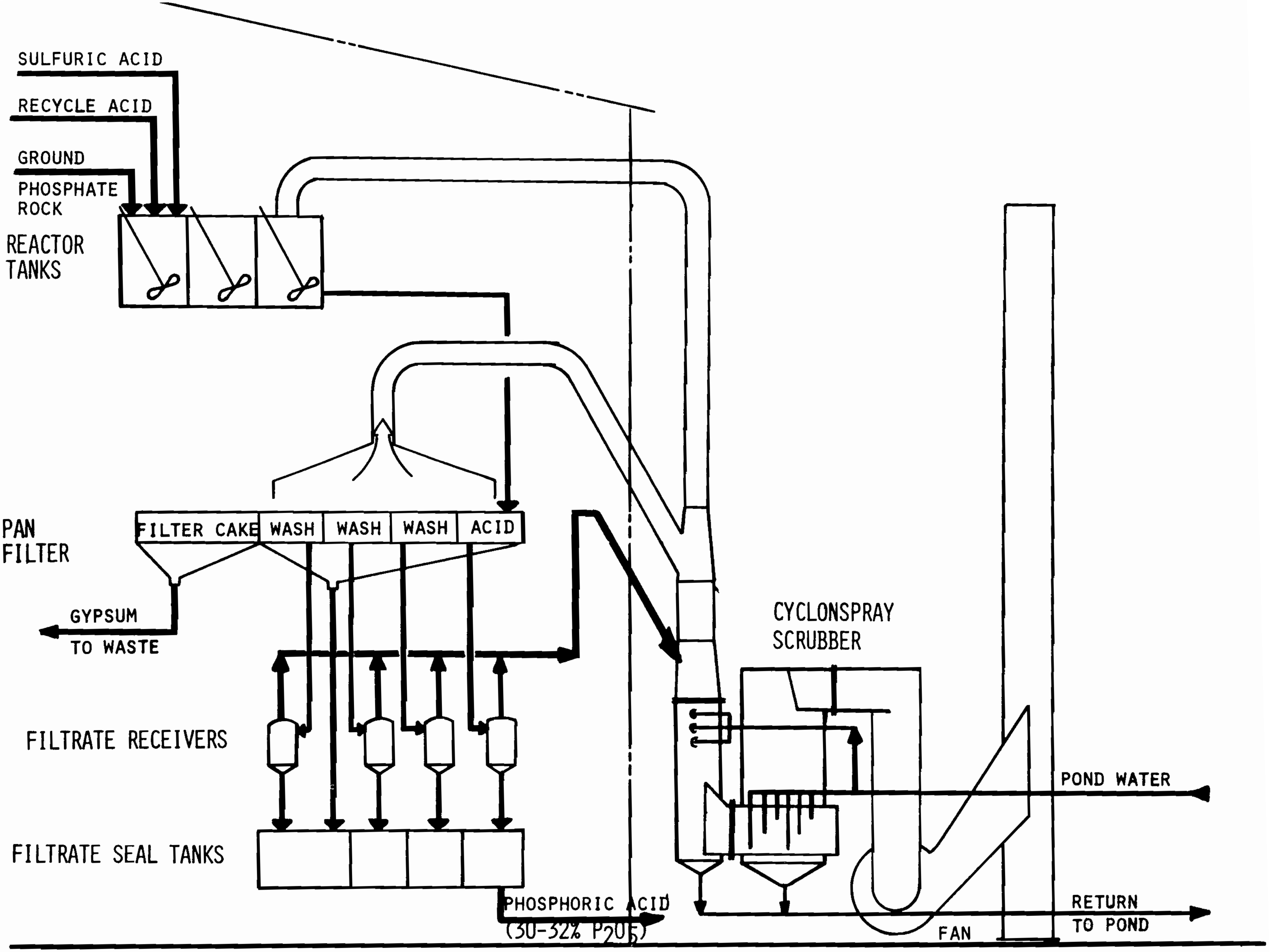


FIGURE 2 Wet Process Phosphoric Acid Flow Sheet

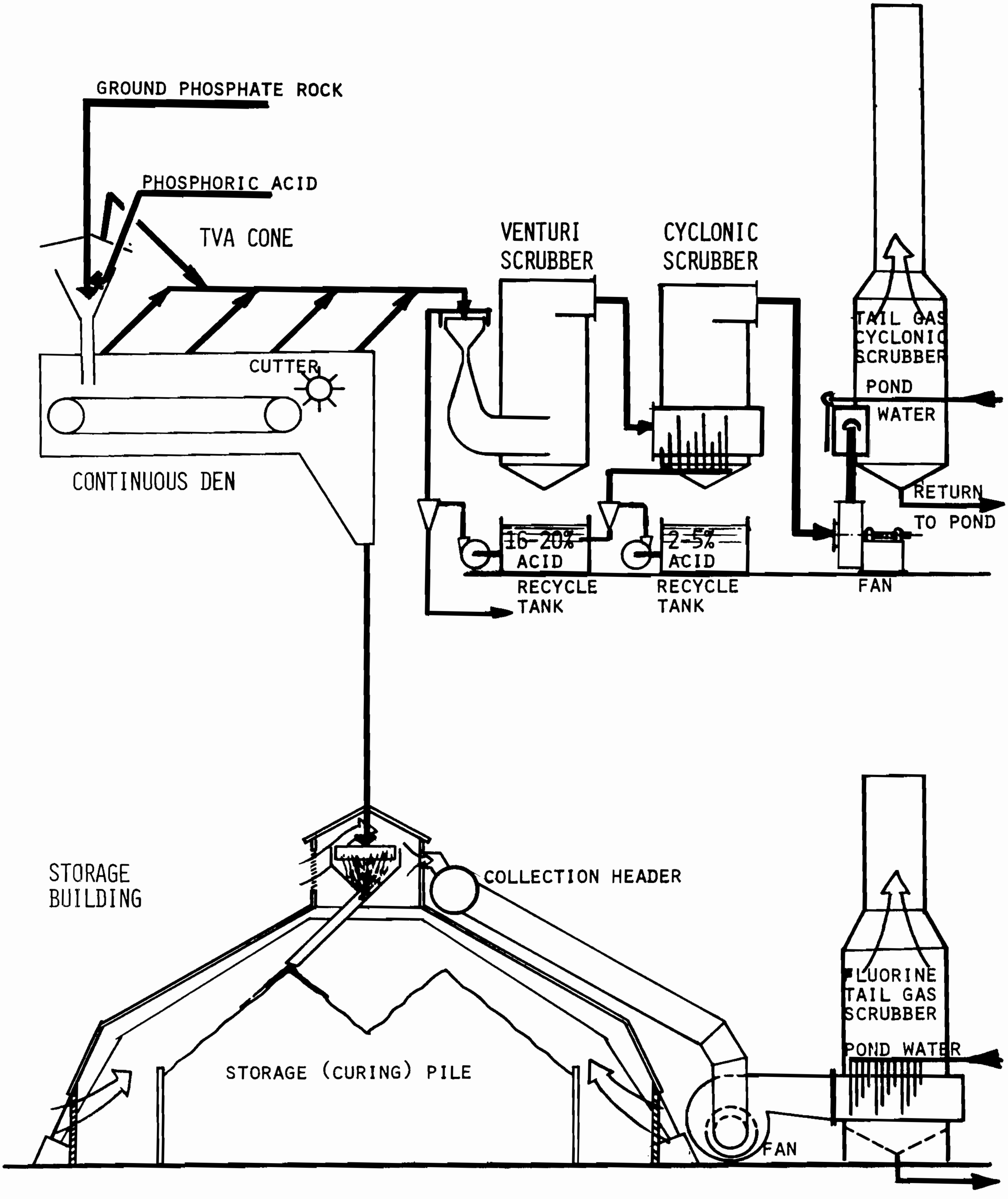


FIGURE 3 ROP TRIPLE SUPERPHOSPHATE FLOW SHEET

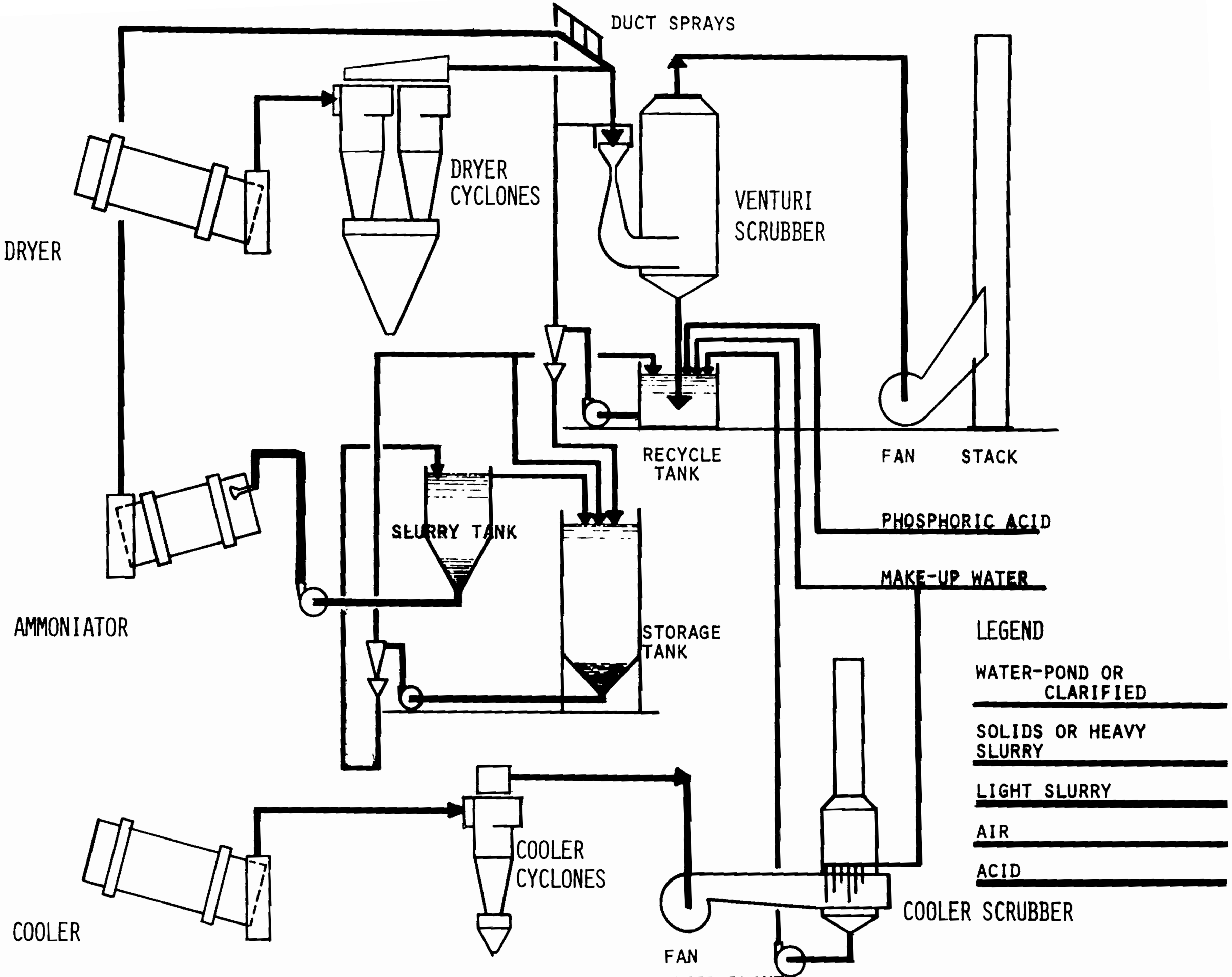


FIGURE 4 NPK FERTILIZER PLANT SCRUBBING SYSTEM FLOW SHEET

Thursday Afternoon Session, November 6, 1969

The Round Table Meeting reconvened at 2 o'clock p.m.
Albert Spillman and Billy Adams, Moderators

Business Meeting

CHAIRMAN SPILLMAN: We have a good attendance. I estimate 90% of the registration are seated and more coming in.

Our program this afternoon will cover the business session and will be followed by our panel on formulation. I call on our Secretary-Treasurer, Dr. Housden L. Marshall

Secretary-Treasurer Report

Housden L. Marshall

Your Secretary-Treasurer Office has been rather busy and active this year. We tried very hard and believe we conquered most of the problems we were confronted with.

Operating costs for 1968-69 was $10,374.45; for 1967-68, was $8,564.14. The additional operating costs, $1,810.31, represented mostly preparation and printing of The Proceedings for 1968. The 1968 proceedings had 40 pages and 45 slides more then the 1967 proceedings. We were $1,500.00 short to pay all of the costs for the year.

A letter was sent to the membership attendance for 1967 and 1968 asking that they purchase 1968 and back issues of "Proceedings". The response was real heartening and within a very short time our members from the states, Canada, Mexico, India, Europe, etc. purchased sufficient proceedings to completely cover our deficite with several hundred dollars to spare. Letters received from the members were encouraging for the Round Table to continue the excellent work it has done. Thank you for your kind expressions and contributions. The finished report is as follows:

Nov. 1,1968 –		
Cash on hand	$ 539.41	
Income during year	10,421.25	
Total cash to account for	$10,960.66	$10,960.66
Disbursements		
1968-69 Meeting Costs	$ 2,503.45	
1968-69 Membership Lists	288.95	
1968-69 Proceedings	6,327.88	
1968-69 Office, Secretarial, Postage, Equipment, Telephone, Transcript, etc.	1,254.17	
Total Expenditures	$10,374.45	$10,374.45
Balance on hand Nov. 1, 1969		$ 586.21

Our cash bank balance confirms $586.21 to our credit.

CHAIRMAN SPILLMAN: Thank you Mr. Secretary for that excellent report. Dr. Marshall has done the usual, good job.

MR. J.E. COUNTESS: I move the report be adopted and with thanks to Dr. Marshall.

MR. WAYNE KING: I second the motion.

CHAIRMAN SPILLMAN: All in favor please say "Aye". Loud chorus of "Aye's". There were no "Nays". Report was unimously accepted

Election of Expanded Executive Committee

CHAIRMAN SPILLMAN: We continue to recognize the responsibility of our Round Table to serve all segments of the Fertilizer Industry with most emphasis on "Production". The names to be recommended to serve on the expanded Executive Committee are experienced representatives of Fertilizer Manufacturers, Basic Raw Material Operations, Researchers, Agronomy, Consulting Engineering, Packaging and Machinery. We are recommending a total of 23 members representing a good cross section of United States companies, Canada, Mexico and England. This expansion will help considerably to strengthen our Round Table activities to keep pace with the continued rapid technology changes in all phases of fertilizer operations.

Our nominating committee: Wayne W. King, Chairman, Joe Whittington and J. E. Countess. Mr. King will you come forward and present the names recommended for election to the Executive Committee.

WAYNE KING: Thanks Albert. I have the list here. I did not know how important a "Guy" I was until a "Bunny", at The Bunny Club, tapped me on the shoulder advising Al Spillman telephoned requesting I attend a meeting last night in his headquarters at 11 P.M. I parted from my "Bunny's" in time to reach Al's meeting at 11:08 p.m. Your nominating committee recommends you elect the following Round Table Representatives for two year period, 1969–1971, to the Executive Committee.

Albert Spillman,
Chairman
Joseph R. Reynolds,
1st Vice President
Robert R. Heck,
2nd Vice President
Billy E. Adams,
3rd Vice President
Housden L. Marshall,
Secretary and Treasurer
Paul Prosser,
Asst. Secretary and Treasurer
Frank P. Achorn
Harold D. Blenkhorn
Charles T. Harding
William E. Jones
Quentin S. Lee
David W. Leyshon

Grant C. Mandwager
–Grayson Morris
–Ladd J. Pircon
–Henry Plate
–Herman C. Powers
–Walter J. Sakett, Jr.
–David S. Schwartz
–William F. Sheldrick
–Adolpho Sisto
–Roger C. Smith
–

Before I ask you to vote I wish to gnaw you down a little bit. I like children and always engage some conversation with them. The other day I talked with an 8 year old boy next door to my home. I said "John, how are you doing in your studies, especially that sex education thing that you have! ?" "And are you making a good grade in that?" Well he says, "Yes, but I think it is all theory and there is no lab work". –lots of laughter.

Are there other nominations from the floor. No additional nominations.

A MEMBER: I move we elect the recommended Executive Committee as outlined by Wayne King.

ANOTHER MEMBER: I second the motion.

WAYNE KING: May I have your vote by raising your hands if for. Looks like all hands. Against, no hands. Committee unanimously elected.

CHAIRMAN SPILLMAN: Thank you Wayne.

I wish to take this opportunity to introduce "The Kitchen Cabinet Advisory Committee". They have been an important cog to our Executive Committee and a listening post "Eyes and Ears" for accumulating information most helpful for programming our year to year programs. They have given up their time on many Saturday mornings to discuss, "off the record" having no voting powers, important suggestions and ideas for operating The Round Table. They will continue to help us. Will the Kitchen Cabinet please come to the rostrum: Wayne W. King, Joe Whittington, Walter J. Sackett, Jr., Paul Prosser, Thomas Athey, J.E. Countess, Elmer Leister and *John R. Remmebang. The Membership applauded their appreciation.

(*absent)

Future Handling of Proceedings

CHAIRMAN SPILLMAN: It will be appropriate at this time to work our newly appointed Assistant Secretary and Treasurer, Paul Prosser.

MR. PROSSER: Ladies and Gentlemen. First, I will be happy to work in this capacity for "A Non-Profit Organization." It is my observation there are several non-profit organizations represented in this audience.

We have had a great many discussions on operating costs and you have heard Secretary Marshall's report. The budget for this organization has been a moderate, approximate $10,000.00 per year, 60% of this has been spent for preparing and mailing the proceedings. This is a real economical operation. I think it would be fitting at this time to have some indications, from the membership here present, as to whether or not the proceedings should be continued. Dr. Marshall advised you, in his report, of the wonderful response, from the membership when they were told of the approximate $1,500.00 deficite. They purchased additional 1968 proceedings for distribution to "operating personnel".

Do you want the "Proceedings" printed as in the past 18 years. May I see your hands please.

MRS. ELEONORE DORLAND, Editor Agricultural Chemicals: How many proceedings do you print? What do they cost per issue? What do you sell them for?

MR. PROSSER: Dr. Marshall come forward and give the answers.

DR. MARSHALL: The real cost of the "Proceedings" is not the number purchased. Assembling the information into print is the real cost. You can purchase additional printed copies around $200.00 per hundred copies. We generally start off with 1000 copies. Adding additional costs for postage, mailing, other incidental costs, we figure about $6.00 per copy. The attendance membership receive a copy free. Additional copies required are sold 1968 for $10.00 each and 1969 will be sold each for $10.00 We sell approximately 400 copies for $4000.00 and we collect approximately $6000.00 dues from those attending the meeting.

MRS. DORLAND: That does mean, though, that with our membership around 300-400, we do have to get interested folks to purchase around 600 copies.

DR. MARSHALL: Oh, yes. And I might say, Eleonore, that our back issues are becoming very popular all around the world.

MR. PROSSER: I must remind you, if I may, that at this minute we have not had sufficient registrations to take care of the total cost of the Proceedings. It may well be that under present circumstances, should we not be able to sell sufficient copies we may again be faced with the problem that was faced this year, namely to raise more funds. I do not believe that will be the case, but the possibility does exist. Can we have that show of hands again, just for the record?

JOSEPH E. REYNOLDS: Paul, we are talking about $6.00 per copy for 1968. What was the cost per copy for 1967?

CHAIRMAN SPILLMAN: Joe, the printer did not charge us much more money per-set for 1968 over 1967. The difference was in the number of pages and prints required: 1967, 110 pages and 1968, 145 pages. We had more printing and there was not any way to cut it down lower. Per copy cost for 1967 around $4.75 each.

JOSEPH E. REYNOLDS: Is this a temporary thing or are we going to have about the same total printing.

CHAIRMAN SPILLMAN: Well, if we have 145 pages again this year, we are going to have to print them. I do not see how we can do otherwise. I can't see, at this time, much of a deficit for 1969. Actually we were around $1,500.00 short for paying all bills for 1968. We are running this institution for around $10,000 per year. I think this is reasonable and we are not going to ask you for another nickel unless it is absolutely necessary to do it. We do not intend to print the 1969 proceedings if we find we are in the hole for an excess amount. We will first ask the membership before we go ahead.

MR. ROBERT R. HECK: On behalf of supporting the publication of the Proceedings, I had not intended to mention this issue. My company put together the index that is available this year for sale. Just two weeks ago I went through that index trying to find in general terms what was in there and I was completely overwhelmed. Our main office at I.M. & C., Skokie, Ill. has a very substantial library. I don't think we have been able to supplement,with all of the other books and things that we have there, the information contained in the year to year proceedings during the past 18 years. My own personal opinion is that I would be willing to pay $25.00 per year to get the proceedings published.

MR. PROSSER: Make a note of that, Joe.

MR. HECK: I have already done that, Joe.

MR. PROSSER: Any other comments?

MR. COUNTESS: Are we ready to pass a motion.

MR. PROSSER: We are going to try to have a show of hands on approving or disapproving the printing of the proceedings. Those in favor of printing the proceedings as in the past please put up one hand. Most of the membership raised their hand. Those not in favor please put up one hand. No hands raised. That is pretty unanimous.

MR. PROSSER: I would like to say that the "committee" has in the past performed a fantastic service at an almost impossible, low price. They have taken no compensation and there is very little leakage out of this fund. Dr. Marshall's old school bag has got one hell of a tight lock on it, I'll tell you. (Laughter from the group.) So, we will do the best we can. One more word. I would like to suggest, on behalf of the committee that, we have your approval to make our great friend Mr. Wayne King, a member emeritus of the Executive Committee. (Applause).

CHAIRMAN SPILLMAN: We are delighted. I go for this and I am sure the membership goes for this. All we want is a show if hands to make this official. Show of hands and more applause.

MR. WAYNE KING: Mr. Chairman Spillman. He was Acting Chairman at the beginning of this meeting. I am overwhelmed and I thank all of you for the honor of your appointing me a member "Emeritus" of the Executive Committee. (Much applause).

Shall we stay in Washington for our 1970 Meeting or move to another location?

CHAIRMAN SPILLMAN: Now we come to that subject that has been discussed here for a number of years: What do we want to do for our 20th meeting, do we want to stay in Washington, do we want to move it?

There has been a lot of expressions. I have heard more expressions at this meeting so far, in the two days that I have been here, that a good many of the boys feel that we ought to move it.

I have Bob Heck here who has done a little investigation on whether or not we can get a hotel big enough, roomy enough, for our meetings and lodging. He has a proposition. I will call on Bob Heck now to give us that proposition. Bob.

MR. HECK: Thank you, Al. For the past four or five years, I think, there has been some discussion on the part of people attending the Round Table as to whether we should continue coming to Washington. I believe we have been meeting here now for 14 years, maybe 15. We feel that if we could change location and get somewhere nearer to the Central part of the United States, or maybe I should say, geographically centered in the agricultural area, that we might improve our attendance.

We have a problem, now, in that we don't have many operational people, except at the executive management level, attending FIRT.

We are talking about trying to reach that level again, at least in part, in our meeting next year.

The geographical center of the agricultural industry in the United States, appears to be somewhere between Memphis and St. Louis. So as a result of that we have looked into both locations. We have been made a tentative offer by a large hotel in Memphis that I would like to discuss with you.

The Sheraton-Peabody there has facilities that can handle up to 600 people. They have a meeting room very similar to this that has a capacity of 600 people without the podium and so forth, or about 450 people with the podium. They have conference rooms that would be available to the meeting if we wanted to break up, as was mentioned very briefly, into smaller groups to discuss specific issues maybe within a group.

They have ample rooms and suites for the number of people that attend this meeting.

There will be no charges on the part of the hotel for the use of any facilities. There will be no obligations to have any functions other than those that we plan for ourselves. In other words, we will not be required to have cocktail parties, banquets or any similar function. Obviously, they would like to have the people who are there spend their money in the hotel, but you are left with the choice.

Memphis also has, directly across the street from the Peabody, a new motel that In think has about 200 units, and they have a working arrangement with this motel to carry overflow.

The weather is usually reasonable in Memphis at this time of year and there is very little conflict with air travel schedules because of weather.

Memphis itself is a very nice town. It's large enough to accommodate a group of this size and give a lot of diversity in what you do.

We have asked for tentative dates next year during the first week of November. They have asked that we give them a confirmation by the end of this week in order for them to continue to hold the space.

They would also like, but do not require, a two-year commitment, and I think probably we would like a two-year commitment because it keeps us a little bit better organized.

And we are not proposing that Memphis replace Washington in the sense that we go to Memphis for 14 years. But we do feel that it would be a good central point and a point where we feel we could get a few more people to attend, particularly people who are located a little bit west of Memphis and a little bit south of Memphis.

I believe that's it, Al.

CHAIRMAN SPILLMAN: Thank you, Bob. Do we have any other suggestions? Any other locations? Of course it takes time. You have to have a lot of time before you contact these hotels. Most of them are booked up for a number of years. I was surprised that Bob got dates so fast. But he has them.

The question is now: Do we want to move next year to Memphis?

DR. MARSHALL: We are signed up here or have tentative reservations at the Mayflower til 1973. And we have the dates November 11th through 13th next year, if we want them. It's entirely up to you folks. But they are there. And I want to say this much. It is difficult to make reservations in any hotel of any size except two to three years in advance. And if we do start this changing business, the Executive Committee has got to get a Hotel Committee.

A MEMBER: Both Memphis and St. Louis were mentioned. Have we anything on St. Louis?

DR. MARSHALL: Yes. St. Louis could not accommodate us for 1970.

MRS. ELEONORE DORLAND: I think the presentation as made by Bob is excellent. He has made an analysis of the hotel situation in Memphis. The rates sound good, the location sounds good, and I think the membership would give it due consideration. I recommend the Committee consider an arrangements committee, of which perhaps Mr. Heck might be chairman, to continue such research for future meetings.

DR. MARSHALL: I might say this about location: Vince, Al and I many times discussed the membership list as to the location of the people who attend our meetings. We found that the majority over a period of 4 or 5 years resided in an area from Washington to St. Louis, plus 50 to 100 miles to the north or south of that line, therefore we probably have to pay attention to that area.

CHAIRMAN SPILLMAN: Are there any other comments. Are we ready to vote whether we wish to move next year's meeting out of Washington?

RODGER C. SMITH: I make a motion that we accept the recommendation made by Bob Heck's committee to have the 1970 Meeting in Memphis, Tenn. for the reasons that he has so well outlined.

CHAIRMAN SPILLMAN: Do we have a second.

MRS. ELEONORE DORLAND: I second the motion.

MR. WAYNE KING: With a reservation I also second the motion. I would say retain here, our reservation for the following year 1971. Are you with me? This does not even need to be part of the motion. But do not give up the ship. Remember what Admiral Jones said. (Laughter).

DR. MARSHALL: We have reservations for 1971-72-73 and I can hold them on the books here without any trouble. They have been tentative and are marked on the Mayflower Book. They will not take them off unless we tell them to take them off.

CHAIRMAN SPILLMAN: Will we need to change the motion?

MR. HECK: Al, it is not necessary to change the motion.

MR. SPILLMAN: All those in favor of going to Memphis for next year's meeting please raise your hands. Note: Majority of hands raised. A few hands were against moving from Washington. Motion is passed. I am sure there will be no regrets.

BOB HECK, Chairman, Joseph R. Reynolds and Dr. Housden L. Marshall are the committee for making all of the necessary arrangements and contacts for our 20th annual meeting, Memphis, Tenn., Wednesday, Thursday and Friday, November 4-5-6, 1970. Hotel-Sheraton Peabody.

Type of Agenda for 20th Annual Meeting

CHAIRMAN SPILLMAN: What type of meeting do you wish for 1970? Are you satisfided with this year's program?

It has been the policy of your Executive Committee, since the beginning of the Round Table, to bring to these meetings up-to-date technology on all phases of operations, Viz: Manufacturing, formulation, ammoniation, granulation, chemical control, conditioning, shrinkage, machinery, packaging, particle size, nomenclature, engineering for buildings and plants, safety, processes for the manufacture of ammonia, nitric acid, phosphoric acid, ammonium phosphates, sulphuric acid, high grade phosphates and other miscellaneous discussions. I recommend if you do not have an "Index" showing the many discussions and up dated discussions, on the various topics thru the years 1955 thru 1968, that you purchase a copy from our Secretary.

A MEMBER: I would like to say that it would be very good if we could have more group discussions. My experience with conferences of this type, one gains much more from the discussions following the formal deliveries and I hope in the future we will have more of these.

MR. JOE WHITTINGTON: Mr. Chairman. I would like to speak very briefly on behalf of a number of people who have suggested over the years that we consider cutting down a bit on the number of speeches and stepping up on the point of discussion.

I wasn't present at the early meetings of the Round

Table, but assume it was a small group which was a Round Table in fact, in that, as the name implies, they sat at a round table and all discussed their problems.

I would like to submit two possible procedures to encourage more general discussion.

1 – It was my privilege to attend a national meeting of oil heat men in Chicago. They decided that for the first time they would try having a one-half-day session for discussion only – no speeches –, seated at round tables in a large room, about 15 to 20 at each table. There were three or four tables on two-way radios, others on office forms, public relations, service and various other problems facing that industry, including spraying fertilizer on lawns from oil trucks, which is where I became involved.

After the meeting the chair asked, "What did you think of this meeting?" In substance typical replies were:– "Why didn't we start this long ago? . . . . . We've met more people at this session, and have learned more through an exchange of ideas with more people with common problems . . . We can read speeches – mail then to us –, but we can't have anything approximating this by mail."

2 – When discussing the foregoing at a recent Advisory Committee meeting, one of its members suggested for consideration – "I think it is tomorrow when there will be a panel on formulation. There will be seven members on that panel. Now, if we were sitting at round tables in the meeting room when the panel made their presentation, at its close the panel members could circulate among the tables to foster discussion for so long as interest lasted."

Another advantage is that sitting around a table creates a friendlier, more comfortable environment. With elbows on the table, a note pad and an ash tray in front of you, there isn't that strain of sitting all day rigidly, akin to a hard, straight-back pew in Church. That's OK for a one hour service at Church, where there is quite a bit of audience participation, but we don't sit there silently for three days for fourteen speeches, with trimmings.

The point I'm trying to make, is that I believe I am speaking on behalf of an outspoken minority, who probably also represent correctly the silent majority in feeling that we have stayed too far from the origins of the Round Table, so the sooner and further we can get back to it, the better off we'll be. Thank you. (Applause).

CHAIRMAN SPILLMAN: Thank you, Joe.

A MEMBER: Al, your committee has done an excellent job bringing important subjects to this Round Table each year. I cannot find anything wrong. Check the Proceedings for past years and also this meeting. We have had good discussions, good speakers, excellent detailed papers and volumes of valuable proceedings most helpful to all interested people responsible one way or another for fertilizer operations. If we desire more discussions, I think one way of doing this, is to have questions in the audience to address the speaker immediately after the paper is given.

CHAIRMAN SPILLMAN: I agree. I am confident, as in the past, when you gentlemen go home, you will start thinking about next year's meeting and send us your usual, many suggestions on subjects our 1970 Agenda should carry. Your cooperation in this respect has been responsible for our successful year to year 18 past meetings.

CHAIRMAN SPILLMAN: I shall now turn the meeting over to Billy E. Adams, Moderator for the Formulation Panel Discussion.

MODERATOR ADAMS: Thank you Albert. Joe Whittington stole my thoughts here as moderator. I wanted to beef up the panel discussion a little bit, however, we have used up too much time and I think we had better get going with our Panel on Forulation. Mr. Grant Marburger is the panel leader. He will introduce the panel.

Formulation Panel

Grant C. Marburger

Panel Leader

This is a formulation panel for the very simple reason that materials constitute 80% of the manufacturing cost of a ton of fertilizer, so the subject is important. Materials, men and equipment are involved in a manufacturing operation, and the panel is going to talk about materials. If manufacturing expenses in a typical plant are $8 and materials are $32, the materials contribute 80% to the total cost of the product. Formulation of course is the process by which materials are selected and used in granulation. To the blender the task is relatively simple for it's just a mathematical computation. But there's an additional ingredient called liquid phase or wetting medium necessary for the material to agglomerate. Liquid phase as used in granulation can be defined as the total amount of material existing in liquid form at a given temperature.

Formulas are historically sacred. In the early days of granulation they were like the family silver, stored away in a drawer, carefully guarded, and never changed. I'm not so sure it isn't the same today. I once co-authored a manual on granulation in the early days of the business, called the Spensol Book. It was written for the plant operator, so was full of formulas. I'll confess to using that dog-eared book once or twice last season, 15 years after it was written.

To abandon a formula that operates, and try a new one, meets great resistance on the plant's part. This is partly because formulation is not understood by many people. For instance you don't have to be a formulator – just use the ones out of the drawer – to be successful as a plant superintendent. Men and equipment and schedules and budgets are enough to give a plant high performance. If you are part of a large corporation, controls exist over most items. But formulas are a mystery – did you ever see a corporate manager get a control over that function? I've seen good ones, with MBA degrees, fail to measure the performance standards in that camp.

In addition to this historical position, formulation has changed today. A relatively few years ago we had never heard of a project aimed at lowering formula costs. Prices were set. Ammonia was the cheapest source of nitrogen, solution was next and ammonium sulfate the most expensive. We didn't even need to look at the costs, for these relations always existed in the same order. So what happened? We learned to granulate with solution because sulfate was (60c a unit) more expensive, and used as much ammonia as possible. Essentially the same relation existed on P_2O_5. But today prices have changed and previous formulation economics are out the window. For instance at a delivered price location ammonium sulfate might be the lowest cost nitrogen source, or triple and phosphoric acid less expensive than normal.

When you formulate with a set of such costs, they'll shake you up. When considering the potential dollar savings, you'll be face to face with the fundamental question, what formula will and what formula will not granulate? (The easy way out is a formula that is easy to granulate, from past experience, and ignore the costs.)

Let's take a case. If sulfate is the cheapest source of nitrogen at a plant, how much can be put into 5-10-10, 6-8-8 and 12-12-12? The 12-12-12 is easy, for it requires sulfate anyway. But your first impulse on 5-10-10, or any 4 to 6 unit nitrogen grade, is that sulfate can't be used because all 5 units are needed to achieve granulation in these low nitrogen grades.

I had to smile this last winter on the outcome of that very case. Economics said ammonia and sulfate on 6-12-12, where the old standby would have been one or two units from ammonia and the rest from solution for granulation. Would sulfate work? So I gingerly selected one unit on a "possible, but let's see" basis (Plant A might not have the operators or the knowhow or the sparger and bed depth and all of those things, but Plant B might do it) and we ended up using three successfully. That's the kind of happy ending that'll make anybody grin.

Another case arises when triple costs are lower than normal. In a low concentration grade, what happens to granulation with 800-900 pounds of filler in the formula? Round, smooth, river sand in the midwest creates a different case than the rough surfaced easy-to-granulate sand that's so abundant in Florida.

As a result of these various items that are historical in formulation, plus the changing conditions that exist today, the theme of this panel is aimed at "Unusual Formulas That Have Been Used in Granulation".

Subject matter as part of this theme will include "Operating Techniques Used in Achieving Granulation". Not only is this an interesting subject in itself, but it's difficult to separate techniques from the formula itself.

There are only two controls that are available to the ammoniator operator, water and steam, and some plants have recycle as a third. A good operator exercises skill in the manipulation of these controls, and the difference can make a formula operate or fail to operate. An example question: Can water achieve granulation in an extremely high temperature formula? What are the methods of its introduction that affect these results? (Some of you know the symptons here, the material is hot and dry). And the age old question, why does a formula work one day and not the next?

I don't believe you should ask the panel that last question, for many parts of their comments this afternoon will make up its answer.

I once had occasion to witness a very striking example of variation in achieving granulation that I'd like to share with you because it was in a batch system and most of us work with continuous. The amount of recycle returned was weighed into the system with each ton. In high liquid phase grade there were 3,200 pounds total solids (1,700 pounds formula materials, plus 1,500 pounds recycle) or a total of 3,600 pounds including the liquids, and the slight variation of only 100 pounds of recycle was enough to change it completely from granulation to non granulation. Only 100 pounds of recycle difference in 3,600 pounds was difficult to believe, and the discharge from the batch mixer was rather striking to see. In our continous systems we don't get to see the effect of this slight variation because we can't record the slight weight change that can make the difference.

With these remarks as background, I'm going to turn it over to the panel. We have one paper that's on formulation, but with a subject matter that's different from the theme of the rest of the panel. Consequently, we'll start first with it and then proceed with the other panel members. (Computer Formulation).

Formulation For A TVA-Type Ammoniator[1] Granulator By Computer

Jon L. Nevins[2] and Frank P. Achorn[3]

INTRODUCTION

Today we are in the midst of a computer revolution. Even small organizations have access to computers or can contract the service at reasonable rates. This lower cost of computer time has made many scientific computer applications an economic necessity today which might have been considered academic in the past.

At TVA, we have been using computers for almost 10 years to formulate bulk blends. During the past year or so, we have been calculating granular formulations by computer. There are a number of reasons for work in this area, but most of them can be traced to the economic changes taking place in the fertilizer industry. Ammonium sulfate prices, for instance, are so depressed in some areas that there is incentive to use as much sulfate as possible in every formulation. Last spring, triple superphosphate in some locations cost less per ton of P_2O_5 than normal superphosphate. This means that if triple is used to produce low-analysis grades, as much as one-half of the formulation may be filler. Relatively low ammonia prices and relatively high sulfuric acid prices make phosphoric or superphosphoric acid economically attractive.

With so many technical and economic factors to be considered, we have resorted to the computer as a means of calculating granular formulations. By using the computer to perform this task our field engineers devote less time to the drudgery of hand calculation and more time to consultation in areas for which they are trained and far more valuable. Furthermore, by using the computer, we can achieve the absolute minimum cost simultaneously with the desired formulation. This achievement is difficult with hand calculation.

The key to formulation by computer is to use the mathematical tool, linear programming. If the formulator has a basic understanding of setting up a linear programming problem and interpreting the linear programming solution, he can use this tool in many ways. We will omit the detailed theory of granulation and of linear programming for this discussion. Rather, we will illustrate with a simple example: (1) how to set up, and (2) how to interpret a least-cost formulation, linear programming problem.

FORMULATION OF GRANULAR FERTILIZERS

Before we get into a discussion of linear programming, let us first take a look at some of the more basic considerations of conventional formulation for a TVA-type ammoniator granulator where recycle rate is equal to the proportion of fines produced in the process. The 9 basic criteria considered at TVA in evaluating a granular formulation are summarized in Table 1 in terms of a wide variety of conventional and experimental raw material alternatives. The meaning of the table is illustrated in terms of the example 13-13-13 formula which was developed by hand calculation. The value shown for product total for each column is calcualted by multiplying the quantity of each material in the formula times the coefficient in the corresponding row and then vertically adding the column.

If the value of each of these 9 columns is sufficiently close to the criteria values, the formula would be acceptable. In choosing among equally acceptable formulas, the one with the lowest raw material cost would be selected.

Column 1 in Table 1 is the weight balance which acts more as a definition than a formulation criteria. Assuming that the desired quantity of product is 2,000 pounds, the weight balance actually defines the quantity of moisture that must be evaporated – 73 pounds in the example. The next 3 columns (2, 3, and 4) calculate the pounds of the 3 primary plant nutrients that the formula provides. As can be seen, the formula comes within rounding error of supplying exactly 260 pounds of each primary nutrient.

The fifth column calculates product moisture to be 19.9 pounds. If sufficient drying is available to evaporate the 73 pounds of moisture, product moisture should be adequate for this high nitrogen grade. Note that the moisture coefficients for the 2 superphosphoric acids are zero. In earlier work we assumed that some hydrolysis occured and negative moisture coefficients were used. Based on commercial-scale demonstrations we have not yet been able to identify such hydrolysis quantitatively.

Column 6 calculates free ammonia, which when equal to zero, indicates that the free ammonia from anhydrous or solutions has just been fixed by each input at the normal ammoniation rate. In the example, underammoniation of 0.2 pounds is close enough to zero to be called the normal balance. The positive coefficients in the ammonia column relate the portion of each ammoniating input that is free ammonia. Table 2 shows the formulation sheet used at TVA. The normal ammoniation rates which are assumed are listed at the bottom. If the ammonia balance is not near zero, the heat of reaction calculation will have to be adjusted since the heat of reaction depends largely on the degree of ammoniation.

The chemical heat of reaction produced by the formula is calculated by column 7. The coefficients in this column are expressed in BTU's per pound of each raw material input. The example formula produces 186,068 BTU's per ton of product.

The remaining considerations relate to the conditions which lead to satisfactory granulation. These conditions involve consideration of the physical properties of input materials and the amount of liquid phase in the formulation. Liquid phase coefficients for each material are shown in column 8. Each material is assigned a coefficient which basically reflects its contribution to liquid phase. Ammoniating solutions and acids are assigned a value of 1.00. Fine, solid materials such as sand or standard potash are assigned a value of zero. Other solid materials are assigned values from 0 to 0.50 depending on their contribution to the liquid phase and their general physical properties. For example, 30-10-0, which contains a high portion of ammonium nitrate, is assigned the high value 0.50; ammonium sulfate, which is more difficult to granulate, is assigned the low value of 0.10; coarse potash, which affects the granulation of other materials very little, is assigned the value 0.30; anhydrous ammonia is assigned the value 0.50; and water or steam the value 2.00

The value of column 8 is generally referred to as the pounds of liquid phase in the flrmula, though it actually is an empirical granulation index. In the example, the value 747.8 has little theoretical meaning. However, by studying the liquid phase values of several hundred formulations that are known to result in good granulation efficiencies, we have been able to rule out many formulas as being "too wet" or "too dry." Of course, liquid phase must be considered in conjunction with the total amount of heat released during granulation. Based on our experience, the liquid phase should be kept around 600 when heat is around 200,000 BTU's per ton of product and should be increased to 800 when heat decreases to 160,000 BTU's. We often try for about 700 pounds of liquid phase and 180,000 BTU's. Following this rule, the example formula would be a little hot and wet, but could be corrected by using about 14 pounds less steam. This minor correction has no significant effect on the actual formula and would probably be detected and corrected by an experienced operator.

LINEAR PROGRAMMING

The linear programming model and solutions for granular 13-13-13 are shown in Table 3. Using linear

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1. Paper prepared for presentation to the Fertilizer Industry Round Table in Washington, D.C., November 6, 1969.
2. Agricultural Economist, Distribution Economics Section, Test and Demonstration Branch, Division of Agricultural Development.
3. Head, Process and Product Improvement Section, Test and Demonstration Branch, Division of Agricultural Development.

Table 1

EVALUATION OF A GRANULAR 13-13-13 FORMULA

Raw Material	Example Formula (13-13-13)	Wt. of Product (1)	Primary Nutrients N (2)	Primary Nutrients P_2O_5 (3)	Primary Nutrients K_2O (4)	Free Moisture (5)	Free Ammonia (6)	Heat (BTU''s) (7)	Liquid Phase (8)	Material Cost ($/Ton) (9)
Anhydrous Ammonia	73	1	0.822	0	0	0	1.00	0	0.50	40.00
Ammoniating Sol. 448(25-69-0)	0	1	0.448	0	0	0.060	0.25	0	1.00	44.80
Ammoniating Sol. 470(30-64-0)	0	1	0.470	0	0	0.060	0.30	0	1.00	47.00
Ammonium Sulfate	40	1	0.210	0	0	0	0	0	0.10	21.00
Ammonium Phosphate Nitrate	0	1	0.300	0.100	0	0	0	0	0.50	55.00
Nitric Phosphate	962	1	0.200	0.200	0	0	0	0	0.30	40.00
Diammonium Phosphate	0	1	0.180	0.460	0	0	0	0	0.25	55.00
Ammonium Polyphosphate	0	1	0.150	0.610	0	0	0	0	0.25	80.00
Normal Superphosphate	338	1	0	0.200	0	0.060	-0.0580	82.8	0.10	18.00
Triple Superphosphate	0	1	0	0.460	0	0.030	-0.0874	143.6	0.20	41.40
Phosphoric Acid (wet-process)	0	1	0	0.540	0	0.170	-0.1944	431.6	1.00	56.00
Superphosphoric Acid (furnace)	0	1	0	0.760	0	0	-0.2660	738.2	1.00	125.00
Superphosphoric Acid (wet)	0	1	0	0.710	0	0	-0.2485	689.6	1.00	86.00
Potassium Chloride (standard)	0	1	0	0	0.600	0	0	0	0	27.00
Potassium Chloride (coarse)	433	1	0	0	0.600	0	0	0	0.30	29.00
Sulfuric Acid (66 Bé.)	0	1	0	0	0	0.068	-0.3234	871.9	1.00	25.00
Sulfuric Acid (60 Bé.)	199	1	0	0	0	0.224	-0.2693	726.0	1.00	19.50
Steam (50 percent heat)	28	1	0	0	0	1.000	0	486.0	2.00	3.00
Water	0	1	0	0	0	1.000	0	0	2.00	0.05
Filler	0	1	0	0	0	0	0	0	0	6.00
Evaporation	73	-1	0	0	0	-1.000	0	0	0	0
Product Totals		2,000	260.8	260.0	259.8	19.9	-0.2	186,068	747.8	32.42

NOTE: Material input costs in this and later tables are expressed in dollars per ton rather than in dollars per pound. As a result, the product total cost is actually 2,000 times $32.42.

programming terminology, the cost columa (9) is called the objective function and the other 8 columns are called constraints. In the example, all of the constraints are equalities.

For example, heat is made to equal 180,000 BTU's. Constraints can also be expressed as inequalities. For example, heat could have been permitted to range from a minimum of 160,000 BTU's up to a maximum of 200,000 BTU's. A linear programming problem is one which seeks to minimize a linear objective function subject to a set of linear constraints.

In Table 3, the indicated product totals were given to the computer and the indicated formulation was the result. The fact that this least-cost formulation results in the given product totals can be verified by evaluating each column as done in Table 4. When the cost column is evaluated, it is found to equal $29.84 which is $2.58 per ton less than the example formula in Table 1.

In addition to the least-cost formulation, called the primal solution, the computer also calculates the dual solution shown in the top row of Table 3. These dual values are dollar values assigned to each of the 8 columns or constraints. Using these individual dollar values, the total dollar-per-ton value of any raw material can be calculated as is shown in Table 5. Note that the value of each material in the least-cost solution is exactly equal to its market value or material cost. Note also that the value of every material not in the least-cost solution is less than its cost. If a material were more valuable than its cost, logic tells us that it should be used in the formula.

The values of the dual solution can also be used in conjunction with materials not considered in the original problem. For example, TVA produces another nitric phosphate product 26-13-0. Its value is calculated in Table 6 to be $36.84 per ton. This means that if 26-13-0 is produced at this value or less, then this material would enter the least-cost formula.

The dual solution values can also be helpful in evaluating small variations in product totals. Suppose heat is increased to 200,000 BTU's per ton – an increase of 10 BTU's per pound. This will increase the formula cost $0.14 per ton. At the same time, suppose liquid phase is dropped to 600 – a decrease of $0.05 per pound. This causes an increase in formula cost of $0.09 per ton. The joint result of more heat and less liquid phase is then an increase of $0.23 per ton. If changes in product totals are too large, the dual solution can actually be forced to change. Hence, some caution must be exercised in this respect.

As a further illustration of the source of raw material values, phosphoric acid is analyzed in Table 7. The first source of value is the phosphate content worth $81.80 per ton of P_2O_5 or $44.17 per ton of acid. If this product were to be used directly as a fertilizer, its P_2O_5 content might be its only source of value. Some tend to think only in terms of agronomic value in pricing fertilizers. This type of pricing, however, is disastrous in the case of a product with as many desirable chemical attributes as phosphoric acid. For example, value accrues to any raw material which has the capacity to fix ammonia. Ammonia, being the lowest cost form of nitrogen is in great demand by the least-cost formulation problem, but since no free ammonia can be in the product, all free ammonia must be fixed. In the

FERTILIZER FORMULA CALCULATION SHEET

GRADE: ____ N ____ P_2O_5 ____ K_2O

FOR: ______________________ DATE: ____________ FORMULATION NO.: ________

Material	Formula, Lb.	Analysis, %		Materials Supplied, Lb.					Ammonia, Lb.		Heat, 1,000 B.T.U.	Cost, $	
		Water	Plant Food	Water	Dry Material	Plant Food N	P_2O_5	K_2O	Supplied	Capacity		Per Unit	Total
Ammonium Sulfate			%N										
Anhydrous Ammonia			82.3%N										
Nitrogen Solution (a)			%N										
Normal Superphosphate			$\%P_2O_5$										
Triple Superphosphate			$\%P_2O_5$										
Conc. Superphosphate			$\%P_2O_5$										
Phosphoric Acid			$\%P_2O_5$										
KCl			$\%K_2O$										
Sulfuric Acid			$\%H_2SO_4$										
Product Moisture													
Total Supplied													
Loss													
Finished Product													

(a) _____% NH_3; _____% NH_4NO_3; _____% Urea.

ASSUMPTIONS:

Loss of total nitrogen is _____%

Loss of P_2O_5 by reversion is _____%

Water content of dried product is _____%

FACTORS:

66° Bé. sulfuric acid = 93.2% H_2SO_4

60° Bé. Sulfuric acid = 77.6% H_2SO_4

DEGREE OF AMMONIATION:

Lb. NH_3/unit A.P.A.

Percent of normal rate

Formulation ________ lb. underweight. overweight.

NOTE:

NORMAL AMMONIATION RATES:

0.270 lb. NH_3/lb. 100% HNO_3

0.347 lb. NH_3/lb. 100% H_2SO_4

5.8 lb. NH_3/20 lb. P_2O_5 in normal superphosphate

3.8 lb. NH_3/20 lb. P_2O_5 in triple superphosphate

3.3 lb. NH_3/20 lb. P_2O_5 in conc. superphosphate

7.2 lb. NH_3/20 lb. P_2O_5 in H_3PO_4

7.0 lb. NH_3/20 lb. P_2O_5 in superphosphoric acid

9.6 lb. NH_3/20 lb. P_2O_5 in H_3PO_4 for DP process

HEATS OF REACTION:

1,428 B.T.U./lb. NH_3 with normal superphosphate

1,643 B.T.U./lb. NH_3 with triple superphosphate

2,220 B.T.U./lb. NH_3 with H_3PO_4

2,696 B.T.U./lb. NH_3 with H_2SO_4

2,480 B.T.U./lb. NH_3 with HNO_3

2,775 B.T.U./lb. NH_3 with superphosphoric acid

Table 3

LINEAR PROGRAMMING MODEL AND SOLUTION FOR 13-13-13

	Primal Solution (13-13-13)	Wt. of Product (1)	Primary Nutrients N (2)	P_2O_5 (3)	K_2O (4)	Free Moisture (5)	Free Ammonia (6)	Heat (BTU's) (7)	Liquid Phase (8)	Material Cost ($/Ton) (9)
Dual Solution		-2.28	111.72	81.80	48.79	2.28	-48.63	0.0138	-1.86	
Raw Material										
Anhydrous Ammonia	40.53	1	0.822	0	0	0	1.00	0	0.50	40.00
Ammoniating Sol. 448(25-69-0)	0	1	0.448	0	0	0.060	0.25	0	1.00	44.80
Ammoniating Sol. 470(30-64-0)	0	1	0.470	0	0	0.060	0.30	0	1.00	47.00
Ammonium Sulfate	715.11	1	0.210	0	0	0	0	0	0.10	21.00
Ammonium Phosphate Nitrate	0	1	0.300	0.100	0	0	0	0	0.50	55.00
Nitric Phosphate	0	1	0.200	0.200	0	0	0	0	0.30	40.00
Diammonium Phosphate	425.04	1	0.180	0.460	0	0	0	0	0.25	55.00
Ammonium Polyphosphate	0	1	0.150	0.610	0	0	0	0	0.25	80.00
Normal Superphosphate	322.40	1	0	0.200	0	0.060	-0.0580	82.8	0.10	18.00
Triple Superphosphate	0	1	0	0.460	0	0.030	-0.0874	143.6	0.20	41.40
Phosphoric Acid (wet-process)	0	1	0	0.540	0	0.170	-0.1944	431.6	1.00	56.00
Superphosphoric Acid (furnace)	0	1	0	0.760	0	0	-0.2660	738.2	1.00	125.00
Superphosphoric Acid (wet)	0	1	0	0.710	0	0	-0.2485	689.6	1.00	86.00
Potassium Chloride (standard)	433.33	1	0	0	0.600	0	0	0	0	27.00
Potassium Chloride (coarse)	0	1	0	0	0.600	0	0	0	0.30	29.00
Sulfuric Acid (66 Be'.)	0	1	0	0	0	0.068	-0.3234	871.9	1.00	25.00
Sulfuric Acid (60 Be'.)	81.08	1	0	0	0	0.224	-0.2693	726.0	1.00	19.50
Steam (50 percent heat)	194.32	1	0	0	0	1.000	0	486.0	2.00	3.00
Water	0	1	0	0	0	1.000	0	0	2.00	0.05
Filler	0	1	0	0	0	0	0	0	0	6.00
Evaporation	211.83	-1	0	0	0	-1.000	0	0	0	0
Product Totals		2,000	260	260	260	20	0	180,000	700	29.84

example problem the value of this capacity is \$48.63 per ton of free ammonia. This value, which is \$59.16 per ton of nitrogen, basically reflects the cost difference between free ammonia nitrogen and solid nitrogen such as that supplied in ammonium sulfate. Since at an ammoniation rate of 7.2 pounds of ammonia per unit of P_2O_5 phosphoric acid fixes 0.19 pounds of ammonia per pound of acid, the value of this product is \$9.45 per ton more than if it fixed no ammonia at all.

Suppose that phosphoric acid is used in a DAP process and is ammoniated at 9.6 pounds of ammonia per unit of P_2O_5. Then 0.26 pounds of ammonia are fixed for a value of \$12.60 per ton of phosphoric acid. Phosphoric acid thus becomes worth \$3.15 per ton more in a DAP process, just because of the increased quantity of ammonia which is fixed.

Of course, the fixing of ammonia and the production of heat are chemically inseparable. Hence, the total value of ammoniation must also include the value of the heat which is generated. In the example problem, heat is valued at \$0.0138 per BTU. Since a pound of phosphoric acid has a heat of reaction potential of 431.6 BTU's, this heat potential is worth \$5.96 per ton of acid once the potential is realized. If phosphoric acid is priced without regard to its total ammoniation attributes, \$15.41 per ton of material would be lost.

Obviously, technical as well as agronomic attributes must be considered in pricing. All attributes must then be sold to the user of intermediate mixed fertilizer materials.

IMPLEMENTATION AND USE

While each computer prints linear programming solutions in a slightly different manner, all have the common trait that they may be difficult to read. It is, therefore, advisable to have computing service personnel write a routine that will print solution information in a self-explanatory format. The format we are now using is shown in Table 8. Materials are separated into those used and those not used in the least-cost formulation. When a material is used, its quantity, input cost, and actual cost for the amount used are recorded. When a material is not used, its substitution cost or value, input cost, and cost reduction required for use are recorded. Dual solution values presented in Table 3 are not generally printed though they are available on command.

Once the basic model and technical coefficients have been determined, the formulator need only supply to the computing service a list of materials, their prices, and the grades for which formulations are wanted.

If the TVA computer is used, Table 8 is what the formulator gets back. The formulator's first response will probably be to a sight-check the printout formula to make sure it is reasonable. For example, a typical response to Table 8 is that too much reliance is placed on steam for granulation. In Table 9, the price of steam and water has been set at \$1,000 so that neither could realistically be used. The result is a formula that would granulate without steam or water – though these may well be used for control purposes – and an imputed value for steam and water. If

Table 4

ACTUAL EVALUATION OF PRIMAL LINEAR PROGRAMMING CONSTRAINTS AND OBJECTIVE FUNCTION

	Primal Solution (13-13-13)	Wt. of Product (1)	Primary Nutrients			Free Moisture (5)	Free Ammonia (6)	Heat (BTU's) (7)	Liquid Phase (8)	Material Cost (\$/Ton) (9)
Raw Material			N (2)	P_2O_5 (3)	K_2O (4)					
Anhydrous Ammonia	40.53	40.5	33.3	0	0	0	40.5	0	20.3	0.81
Ammoniating Sol. 448(25-69-0)	0	0	0	0	0	0	0	0	0	0
Ammoniating Sol. 470(30-64-0)	0	0	0	0	0	0	0	0	0	0
Ammonium Sulfate	715.11	715.1	150.2	0	0	0	0	0	71.5	7.51
Ammonium Phosphate Nitrate	0	0	0	0	0	0	0	0	0	0
Nitric Phosphate	0	0	0	0	0	0	0	0	0	0
Diammonium Phosphate	425.04	425.0	76.5	195.5	0	0	0	0	106.3	11.69
Ammonium Polyphosphate	0	0	0	0	0	0	0	0	0	0
Normal Superphosphate	322.40	322.4	0	64.5	0	19.3	-18.7	26,695	32.2	2.90
Triple Superphosphate	0	0	0	0	0	0	0	0	0	0
Phosphoric Acid (wet-process)	0	0	0	0	0	0	0	0	0	0
Superphosphoric Acid (furnace)	0	0	0	0	0	0	0	0	0	0
Superphosphoric Acid (wet)	0	0	0	0	0	0	0	0	0	0
Potassium Chloride (standard)	433.33	433.3	0	0	260.0	0	0	0	0	5.85
Potassium Chloride (coarse)	0	0	0	0	0	0	0	0	0	0
Sulfuric Acid (66 Be'.)	0	0	0	0	0	0	0	0	0	0
Sulfuric Acid (60 Be'.)	81.08	81.1	0	0	0	18.2	-21.8	58,866	81.1	0.79
Steam (50 percent heat)	194.32	194.3	0	0	0	194.3	0	94,439	388.6	0.29
Water	0	0	0	0	0	0	0	0	0	0
Filler	0	0	0	0	0	0	0	0	0	0
Evaporation	211.83	-211.8	0	0	0	-211.8	0	0	0	0
Product Totals		2,000.0	260.0	260.0	260.0	20.0	0	180,000	700.0	29.84

Table 5

ACTUAL EVALUATION OF DUAL LINEAR PROGRAMMING CONSTRAINTS AND OBJECTIVE FUNCTION

	Weight of Product	Primary Nutrients			Free Moisture	Free Ammonia	Heat (BTU's)	Liquid Phase	Material	
		N	P_2O_5	K_2O					Cost ($/Ton)	Value ($/Ton)
Dual Solution	-2.28	111.72	81.80	48.79	2.28	-48.63	0.0138	-1.86		
Raw Material										
Anhydrous Ammonia	-2.28	91.83	0	0	0	-48.63	0	-0.93	40.00	40.00
Ammoniating Sol. 448(25-69-0)	-2.28	50.05	0	0	0.14	-12.16	0	-1.86	44.80	33.90
Ammoniating Sol. 470(30-64-0)	-2.28	52.50	0	0	0.14	-14.59	0	-1.86	47.00	33.92
Ammonium Sulfate	-2.28	23.46	0	0	0	0	0	-0.19	21.00	21.00
Ammonium Phosphate Nitrate	-2.28	33.52	8.18	0	0	0	0	-0.93	55.00	38.49
Nitric Phosphate	-2.28	22.34	16.36	0	0	0	0	-0.56	40.00	35.87
Diammonium Phosphate	-2.28	20.11	37.63	0	0	0	0	-0.46	55.00	55.00
Ammonium Polyphosphate	-2.28	16.76	49.90	0	0	0	0	-0.46	80.00	63.92
Normal Superphosphate	-2.28	0	16.36	0	0.14	2.82	1.14	-0.19	18.00	18.00
Triple Superphosphate	-2.28	0	37.63	0	0.07	4.25	1.98	-0.37	41.40	41.28
Phosphoric Acid (wet-process)	-2.28	0	44.17	0	0.39	9.45	5.96	-1.86	56.00	55.84
Superphosphoric Acid (furnace)	-2.28	0	62.17	0	0	12.94	10.19	-1.86	125.00	81.17
Superphosphoric Acid (wet)	-2.28	0	58.08	0	0	12.08	9.52	-1.86	86.00	75.56
Potassium Chloride (standard)	-2.28	0	0	29.28	0	0	0	0	27.00	27.00
Potassium Chloride (coarse)	-2.28	0	0	29.28	0	0	0	-0.56	29.00	26.44
Sulfuric Acid (66 Be'.)	-2.28	0	0	0	0.15	15.73	12.04	-1.86	25.00	23.79
Sulfuric Acid (60 Be'.)	-2.28	0	0	0	0.51	13.10	10.03	-1.86	19.50	19.50
Steam (50 percent heat)	-2.28	0	0	0	2.28	0	6.71	-3.71	3.00	3.00
Water	-2.28	0	0	0	2.28	0	0	-3.71	0.05	-3.71
Filler	-2.28	0	0	0	0	0	0	0	6.00	-2.28
Evaporation	2.28	0	0	0	-2.28	0	0	0	0	0
Product Totals	-2.28	14.52	10.63	6.34	0.02	0	1.24	-0.65		29.84

Table 6

Calculation of 26-13-0 Value

Source of Value	Calculation	Value ($/Ton)
Nitrogen (N)	.26 x 111.72 =	29.05
Phosphate (P_2O_5)	.13 x 81.80 =	10.63
Liquid Phase	.30 x -1.86 =	-0.56
Weight	1.00 x -2.28 =	-2.28
Total value		36.84

Table 7

Calculation of 0-54-0 Phosphoric Acid Value

Source of Value	Calculation	Value ($/Ton)
Phosphate (P_2O_5)	.54 x 81.80 =	44.17
Ammonia Capacity	-.1944 x -48.63 =	9.45
Heat Potential	431.6 x 0.0138 =	5.96
Liquid Phase	1.00 x -1.86 =	-1.86
Weight	1.00 x -2.28 =	-2.28
Moisture	.17 x 2.28 =	0.39
Total Value		55.84
Market Cost		56.00
Cost Reduction Required for Use		0.16

the plant has no steam but is considering such an investment, this inputed value can be helpful in making such a decision.

Suppose the formulator is more interested in a particular type of formulation rather than just economics. He could price all materials but the ones he wants used at $1,000, or in the case of Table 10 where superphosphoric acid formula is desired, simply price superphosphoric acid at one cent below its substitution cost. With practice, the formulator can usually get the formulation he desires. As a last resort, a specified quantity of one or more materials can be forced into the solution by adding the appropriate constraints.

The formulations calculated by our computer help to "bracket" a likely workable least-cost formulation. These formulations must then be proved and often adjusted in practice. As more field experience is gained, it will then be possible to come back and adjust model coefficients or even the model itself.

Formulation by computer is mot a sterile, ivory tower, derive-it- theoretically- and forget-it, computer application. It is more akin to a living growing organism. The key to successfully inplementing this tool is people. People as individuals working together as a team. The engineer, plant operator, economist, mathematician, computer programmer, and many other disciplines must all do their "thing."

Most important of all in this or any other operations research project is the managerial element that is able to successfully bring all disciplines together. Without this element the team cannot function and formulation by computer can never be successfully implemented.

MODERATOR MARBURGER: Are there questions for Jon Nevins or Bud Balay? Would you stand up so that everyone knows who you are in case there are discussions or questions on any application in engineering or in granulation techniques.

MR. LARRY LORTSCHER, NSS Agri-Chemicals, Div. NSSteel: Can you put a separate constraint column in for forcing materials in or out, rather than fudging the price?

MR. NEVINS: Excuse me? For forcing materials in or out, did you say?

MR. LORTSCHER: Where you want to specifically say Well, we want to use superphosphoric acid regardless of these other factors, or, in the one case, example you gave, let us eliminate steam and water, instead of actually doctoring the price column, couldn't you put a go or no-go constraint column on materials?

MR. NEVINS: Right. We do this. The standard program we run, we include perhaps 30 or 40 materials in it and we use this high priced thing just to knock a lot of them out. But we carry two columns for restriction materials. For example, in some of the work that we have been doing with pollution control, through the use of superphosphoric acid, we might want to place a constraint that there be, let's say, at least, or exactly, 150 pounds of superphosphoric acid in the formula. Of course this statement would be made almost regardless of the economics involved. So, to answer your question, yes, you can do this and we do, very often.

MODERATOR MARBURGER: Other questions? Anything on the constraints or linear programming?

MR. Hwa C. Ai, Lummus Company: I want to ask whether this computer technique could be extended to include the nitrophosphate type of formulation where you have to consider the water solubility of the phosphate.

MR. NEVINS: Yes, it can be. We have just finished doing this for every conceivable combination of mixed acid nitric phosphate processes. In other words, where a conventional ammoniation granulation plant would be modified with a preneutralizer and extractors to use phosphate rock, nitric acid, sulfuric acid, phosphoric acid in any combination, and then it would also be subject to partial acidulation where you could put some materials into the drum.

We have done this and this is really helpful because, as you know, if you have tried to formulate mixed acid nitric phosphates, it's almost impossible, by the time you

Table 8

TVA GRANULATION FORMULATION--STEAM AND WATER AVAILABLE

Nitrogen	Phosphorus	Potash
13.00	13.00	13.00

Total Units	Unit Cost	Total Cost
39.00	0.765	29.84

Var. No.	Materials Used	Quantity (Lbs.)	Input Cost ($/Ton)	Actual Cost ($/Ton)
1	Anhydrous Ammonia	40.5	40.00	0.81
4	Ammonium Sulfate	715.1	21.00	7.51
7	Diammonium Phosphate	425.0	55.00	11.69
9	Normal Superphosphate	322.4	18.00	2.90
14	Potassium Chloride (standard)	433.3	27.00	5.85
17	Sulfuric Acid (60 Be.)	81.1	19.50	0.79
18	Steam (50 percent heat)	194.3	3.00	0.29
21	Evaporation	211.8	0.00	0.00
22	Quantity of Product	2,000.0	0.00	0.00

Var. No.	Materials Not Used	Substitution Cost	Input Cost ($/Ton)	Cost Reduction Required for Use
2	Ammoniating Sol. 448(25-69-0)	33.90	44.80	10.90
3	Ammoniating Sol. 470(30-64-0)	33.92	47.00	13.08
5	Ammonium Phosphate Nitrate	38.49	55.00	16.51
6	Nitric Phosphate	35.87	40.00	4.13
8	Ammonium Polyphosphate	63.92	80.00	16.08
10	Triple Superphosphate	41.28	41.40	0.12
11	Phosphoric Acid (wet-process)	55.84	56.00	0.16
12	Superphosphoric Acid (furnace)	81.17	125.00	43.83
13	Superphosphoric Acid (wet)	75.56	86.00	10.44
15	Potassium Chloride (coarse)	26.44	29.00	2.56
16	Sulfuric Acid (66 Be.)	23.79	25.00	1.21
19	Water	-3.71	0.05	3.76
20	Filler	-2.28	6.00	8.28
23	RHS	2,984.17	0.00	-2,984.17

Table 9

TVA GRANULATION FORMULATION--STEAM AND WATER UNAVAILABLE

Nitrogen	Phosphorus	Potash
13.00	13.00	13.00

Total Units	Unit Cost	Total Cost
39.00	0.789	30.76

Var. No.	Materials Used	Quantity (Lbs.)	Input Cost ($/Ton)	Actual Cost ($/Ton)
1	Anhydrous Ammonia	72.2	40.00	1.44
2	Ammoniating Sol. 448(25-69-0)	61.6	44.80	1.38
4	Ammonium Sulfate	815.7	21.00	8.56
7	Diammonium Phosphate	9.9	55.00	0.27
9	Normal Superphosphate	313.6	18.00	2.82
11	Phosphoric Acid (wet-process)	356.9	56.00	9.99
15	Potassium Chloride (coarse)	433.3	29.00	6.28
21	Evaporation	63.2	0.00	0.00
22	Quantity of Product	2,000.0	0.00	0.00

	Materials Not Used	Substitution Cost	Input Cost ($/Ton)	Cost Reduction Required for Use
3	Ammoniating Sol. 470(30-64-0)	44.69	47.00	2.31
5	Ammonium Phosphate Nitrate	42.36	55.00	12.64
6	Nitric Phosphate	37.98	40.00	2.02
8	Ammonium Polyphosphate	63.56	80.00	16.44
10	Triple Superphosphate	39.17	41.40	2.23
12	Superphosphoric Acid (furnace)	70.33	125.00	54.67
13	Superphosphoric Acid (wet)	66.68	86.00	19.32
14	Potassium Chloride (standard)	24.73	27.00	2.27
16	Sulfuric Acid (66 Bé.)	13.26	25.00	11.74
17	Sulfuric Acid (60 Bé.)	13.42	19.50	6.08
18	Steam (50 percent heat)	20.05	1,000.00	979.95
19	Water	28.48	1,000.00	971.52
20	Filler	0.57	6.00	5.43
23	RHS	3,075.94	0.00	-3,075.94

Table 10

TVA GRANULATION FORMULATION--SUPERPHOSPHORIC ACID COMPETITIVELY PRICED

Nitrogen	Phosphorus	Potash
13.00	13.00	13.00

Total Units	Un : Cost	Total Cost
39.00	0.789	30.76

Var. No.	Materials Used	Quantity (Lbs.)	Input Cost ($/Ton)	Actual Cost ($/Ton)
1	Anhydrous Ammonia	19.2	40.00	0.38
2	Ammoniating Sol. 448(25-69-0)	234.5	44.80	5.25
4	Ammonium Sulfate	621.9	21.00	6.53
7	Diammonium Phosphate	47.8	55.00	1.32
9	Normal Superphosphate	459.1	18.00	4.13
13	Superphosphoric Acid (wet)	205.9	66.66	6.86
15	Potassium Chloride (coarse)	433.3	29.00	6.28
21	Evaporation	21.6	0.00	0.00
22	Quantity of Product	2,000.0	0.00	0.00

	Materials Not Used	Substitution Cost	Input Cost ($/Ton)	Cost Reduction Required for Use
3	Ammoniating Sol. 470(30-64-0)	44.69	47.00	2.31
5	Ammonium Phosphate Nitrate	42.36	55.00	12.64
6	Nitric Phosphate	37.98	40.00	2.02
8	Ammonium Polyphosphate	63.56	80.00	16.44
10	Triple Superphosphate	39.17	41.40	2.23
11	Phosphoric Acid (wet-process)	55.99	56.00	0.01
12	Superphosphoric Acid (furnace)	70.31	125.00	54.69
14	Potassium Chloride (standard)	24.73	27.00	2.27
16	Sulfuric Acid (66 Bé.)	13.23	25.00	11.77
17	Sulfuric Acid (60 Bé.)	13.40	19.50	6.10
18	Steam (50 percent heat)	20.04	1,000.00	979.96
19	Water	28.48	1,000.00	971.52
20	Filler	0.57	6.00	5.43
23	RHS	3,075.77	0.00	-3,075.77

get the calcium nitrate shifted out of the product and all the other constraints that have to go into it. So it is this type of application that some day we hope to see this linear programming going to, because it really will answer questions that we can't answer today.

MODERATOR MARBURGER: I think we will go on. There is a great amount of work behind the paper just presented and I know that a great number of you over the coming year or maybe for several years are going to be involved in such a thing.

Our next speaker is also a granulator. And without further background on him, I am going to introduce him to you. A. Merideth Morris, Manager Southern States Fertilizer Plant, Russellville, Ky. Merideth, please.

Formulation For 7′ x 14′ Ammoniator—Granulator

A. Merideth Morris

When Grant called and asked me to take part on the formulating panel, my first reaction was to say "no", as it is a lot better feeling, as far as I am personally concerned, to be in the audience. Then I realized the valuable information that has been imparted by others in the past years, so I could not say "no" without a guilty feeling if the small contribution I can make will be of any help. My remarks will be pointed mainly to a plant with a 7x14 ammoniator granulator, 7x50 dryer (co-current) and cooler (counter-current).

In dealing with this thing we call formulating, there is a lot to consider. The first thing that comes to mind is cost. This is one thing I believe has gotten plant people in more trouble than meets the eye. What will work on paper will not always hold true when we start it through the mill. It is more critical in a granular plant than a pulverized one. But the problems are there just the same. I do not believe we ever overcame the bag set or reversion problem in pulverized fertilizer, and if your formulation is not watched in the granular plant the same problem of set will arise in fertilizer that is stored in bags for a long period. We can formulate a little differently if we know it will be handled in bulk and the consumer will get the same plant foods. (I am referring to N. P. & K. in the previous statement), but most of us have to handle both in our operations.

Geographical location has some bearing on formulating. This is due partly to freight rates and weather, as you all know. The solutions that really do the best job in the ammoniator, are not always used. For example, we use a 471 solution – 30-64-0 for simplicity, with a salting out temperature of -32 degrees F. We can use it in all seasons at our location and the adjustments for seasonal changes are nil. as the vapor pressure is not critical. For granulating, a higher ammonium nitrate solution would be to an advantage, providing the proper heat could be attained.

The No. 1 problem is the variable in raw materials, the floats or dust that runs down in front of the triple super, sulphate of potash, and to some degree muriate bins, when unloading are very hard to handle in a granulator or mixer (especially the granulator), and can cause some restless nights when production is slowed up during the peak season. Most all normal superphosphates are easy to handle, yet expensive in lots of cases.

We find wet phosphoric acid hard to control, especially the finished product in the dryer. When we find a way to get it to react in the ammoniator like it does in the dryer, we will really make a good, high analysis fertilizer. In watching the discharge from the dryer, first you have dry, white-like product, then it turns brown, next it will come out the nicest granules you want to see. In a few minutes, watch out – it starts to melt and roll up in balls. We are in the process of getting a closer control on the drying temperature and this will be a big help, along with more heat in the ammoniator.

In considering formulas, what goes into the bin, and at what rate, has to be given consideration. Condition of spargers, and location in the ammoniator is of utmost IMPORTANCE. When making changes from high nitrogen to low nitrogen grades during the operating day without stopping, has a bearing on the formula for the change. In my opinion there is a lot more than meets the eye in formulating granular fertilizer.

I cannot help but be a bit concerned about computer formulating, yet we have this to think about. Since our last meeting, most of us have witnissed "Man on the Moon". This was accomplished mainly by computers – yet just before impact, man had to take over and steer the "Eagle" to a safe landing place. Maybe the computers will tell us what direction to take, and at the critical point man can take over and steer on the right course.

May I say I am proud to be a small part in the fertilizer industry. Where could you work that does more for humanity? Another thing, if other industries had operated like the fertilizer segment and farmers we serve, we would not be bothered with inflation as it is today. Thank you.

MODERATOR MARBURGER: Thank you, Meredeth We will not accept your apology for the shortness of your valuable paper.

Out next paper will be given by Mr. John Hailey, Manager, mixed goods supply, Agricultural Chemical Co., Memphis Tenn. John has a great amount of past experience in a number of granulation and fertilizer plants across the United Ststes.

Formulation

John T. Hailey

When we consider formulation, what are we really talking about? Isn't the prime interest in how we can put raw materials of all kinds together in a chemically compatible fashion and come out with a good homogenous, granular fertilizer?

If all we wanted was a mixture of plant food, we could make a pulverized fertilizer with little effort. If granular consistency was all that matters, we could blend granular raw materials in a physical mix that would satisfy this condition. What we really are considering is granulation when we talk formulation. This is what the consumer wants and this is what has caused the bulk of our problems in production of mixed goods, however the biggest problem people face is convincing themselves that the rules we practiced in our fertilizer plants a few years ago are as obsolete as the horse drawn wagon.

We're basically a conservative group of people, when it comes to production planning and operations. A great deal of this conservatism has developed as a balance to some of the optimistic planning generated by our marketing departments. What Sales Manager hasn't faced a new season and honestly felt he was going to make a large, or tremendous increases in his profits and volumes. And what Plant Manager hasn't taken these same estimates, checked his records on past performance and thrown a large amount of Kentucky Windage into his production planning.

Let's give our Marketers credit, however. The reason we're meeting here today is because they have forcibly generated a forward thrust to our direction and production can no longer sit in their plants turning out 3-12-6 and 5-10-5 because that's what moved last year and the year before that.

There's a well informed, knowledgeable group of customers out there who have voiced their discontent with what we had to offer and our sales people have gotten the message if we haven't. The survivors, in our industry, are going to be those companies who are willing to abandon the old patterns of manufacturing and marketing and make some innovations in what Grant Marburger has referred to as "historically sacred areas."

Now, what we've been doing for many years has not been bad. On the contrary, the production groups have come up with some remarkable results in prodding the old, basic TVA granulation system into performing as it was never designed to do. Grades such as 15-15-15 and 8-24-24 were thought to be in the realm of the prereactor or other exotic methods until the pressures of a changing market forced us to try and adapt our systems to meet these new demands.

To some people this attitude or philosophy of being forced to make changes might sound like production has been anti-progress and reluctant to move off the status quo. This is decidedly not so. In all companies these men are charged with the responsibility for holding costs in line and are keenly aware of the expenses that can be incurred in the development of new grades and methods. Despite well laid plans with bench tests, pilot plant runs and plant size runs a good number of promising projects winds up as a bin full of offanalysis material. This can be heart-breaking in the time and money expended.

In the same vein, however, too much emphasis on possible losses can lead to a "rocking chair" philosophy where we remain frozen at an acceptable level. While the Marketers are impatiently waiting for production to develop grades on which they can base sales programs, the market is drifting away to the newer processes, the blends or any other product which promises to fulfill the customers desires.

This is a challenge that must be met. Instead of passively waiting to be pressured or actively resisting this change, we need to be promoting these innovations ourselves. We don't honestly know what limits our plants and machinery have, but we must find out.

Viewing formulation from this direction means taking the wraps off all the built-in constraints and restrictions we have developed over the years and allowing ourselves the freedom of new and fresh thinking.

This is an area where computer formulation can help you immensely by pointing the direction to go or it can hurt you badly if the input data is warped by the human opinions and restrictions you are trying to escape.

For example, we originally set up a computer formulation program and asked all of our plant managers to contribute their knowledge in establishing minimum and maximum limits on physical and chemical rules. There were large variations in answers depending on each individuals experience, grades and plant equipment. Attempting to be conservative we took a middle-of- the-road approach and said we would set average limits that some plants had already exceeded, but others could reach. The result was a program that was comfortable for some, attainable by others – and expensive to the company.

Normal superphosphate was considered mandatory in most grades to achieve good granulation. Filler was considered detrimental and most plant men said 400 pounds was all you could handle.

Liquid phase was critical. Four hundred pounds of total liquids was high and 500 pounds was an absolute limit.

Ninety pounds of free ammonia was all that could be handled. You'd lose the rest. So what are we doing now? How about:

1350 pounds of filler in 3-12-6
1250 pounds of filler in 4-12-8
1100 pounds of filler in 5-10-10
900 pounds of filler in 10-10-10

and what's happened to Normal Superphosphate? In some plants it's no longer produced or used in granular formulas.

What about liquid phase? Eight hundred pounds of phosphoric acid, solution and anhydrous in 8-24-24. One thousand and thirty pounds of phos acid and anhydrous in 7-28-28.

Anhydrous ammonia at five to six units with losses not excessive.

This is not theory, but working formulas that have been used successfully. How was this done when our experience said it couldn't or shouldn't be possible. Like most new things the formulas were derived by necessity. In some cases, materials were temporarily short during the peak of the season or raw material costs started oscillating so badly you could no longer afford to use the old standby formulas. At this point you'd go to the experts, the plant managers and ask them to try anything that worked.

These men came up with some outstanding innovations. Consider the formula with high amounts of filler and no superphosphate. One Plant Manager tried several different kinds of dolomitic limestone and various river sands. He finally hit on one that screened about 50% between 20 and 30 mesh and had very few fines through 60 mesh. When additional freight was considered, it was still much cheaper than staying with superphosphate.

This doesn't mean we advocate abandoning normal superphosphate and putting in fillers. Normal super is still an excellent plant food material and a good aid to granulation. In some sulfur deficient areas it is the best source for supplying this element. We fully intend to continue its manufacture and use in mixed fertilizer where the economics and usage prove beneficial.

It does mean, however, that we are not tied to this material or a number of others in building our formulas. All materials have a value from an agronomic or economic standpoint. The old conception of a filler as a non-plant food material that took up available space in a formula is gone. In some formulas Normal super may be the cheapest and best filler and we even have formulas where triple super acts as a filler.

The problem with liquid phase was one of identification. In the early days of granulation we lumped Sulfuric and Phosphoric Acid, Solution and Anhydrous together as liquids and turned up the water when the bed ran too dry. As experience was gained and materials changed, we began to realize that Anhydrous did not behave as a liquid, but actually dried the bed; that acids in correct ratios with free ammonia formed solid salts and that almost every plant food material had a certain melting point which made it behave like a liquid. We now use the term "Salt Phase" in formulas as well as liquid phase.

This brings up an interesting point in the operating techniques of our units. The ammoniator operator is normally considered to control water, recycle dust and sometimes steam. He also has another control in his dryer temperature which can aid or destroy his granulation.

We were running some granular normal superphosphate a few years ago and in starting up we had a period of several minutes when the bed of pulverized super ran extremely dry. At the same time a probe malfunction on our dryer temperature control allowed the temperature to rise above 1500 degrees before the limit switch shut down the main burner. We relit the burner and controlled it manually and started watching our screens for product to appear. When 40 minutes had gone by, we pulled the door off the discharge end of the dryer and out flowed an almost molten mass of mud.

From that little experience, along with similar pieces of information from other runs, we began to relize that heat was a more critical matter in granulation than merely drying the product. With careful logging of information from successive runs, most of the plants developed operating data that allowed them to achieve improved granulation by careful control of their dryer heat and air flow.

Some of the units found their overgranulation was the result of high front end temperatures creating this "melt" condition and fusing of large lumps of material. Others found their air flow too strong with material being pulled through the dryer so fast that coarse particles remained wet and fine granules went to the cyclones as recycle.

Using judicious dampening of airflows most plants established set criteria for heat levels to be used on various grades. Small temperature adjustments frequently brought about that fine shade of control that meant the difference between a good run and one that was marginal at best.

The same held true for reaction heat in the ammoniator. We'd known for a long time that temperatures below 200 degrees with low liquid phases were difficult to granulate and the more water we added the colder they became. This was the period when normal super was considered indispensable and filler was not even mentioned. As liquid phases went up, it was necessary to keep proper ratios between acids and ammonia and before long we were in a reverse position of having excess heat in the ammoniator causing overgranulation and a semi-melt condition of potash and the salts of sulfates and phosphates.

To cure this we could either cut back on our acids and solutions or bring in additional recycle. By electing to bring in recycle we were able to maintain the cost savings of some of the high liquid formulas while keeping the bed temperature under control with recycle dust and water. In a sense, the entire reacted bed became our liquid or molten salt phase and the recycle dust became dry base.

As we became more experienced we tried some of these formulas on lower analyses and found they worked extremely well. For example a 5-10-10 was made with 1100 pounds of filler, potash, phosphoric acid and a combination of 3 units of anhydrous and two units of sulfate of ammonia. This could have been made with all anhydrous and additional sulfuric acid, but the heat of reaction would have been high. This also happened to be the lowest cost formulas at this location with the materials available.

All of these changes didn't come about overnight nor were all of them planned changes. Many of our improved formulas and techniques were the results of observations made by good, conscientious operators and foremen who reported what they saw even when they didn't understand what was occuring. From the start of the TVA granulation systems in our company, we have maintained daily running logs on every grade made. These have been modified, expanded and improved several times and are analyzed thoroughly at the plant and headquarters to determine not only what caused malfunctions, but what went right, and could be applied elsewhere. With over 20 manufacturing units to draw from, this cross indexing of information resulted in uncovering the best methods more rapidly,then assessing each unit separately.

This is a key point. One of the most effective aids and techniques to improve your process and product lies in a well trained observant operator who notes down everything that occurs. Don't let him depend on memory after the shift is over, but provide some form of logical log sheet to report all details of the run.

Another thing concerning operators. In the early days of granulation, he was a normally intelligent person who was told where the water valve was and don't worry too much about anything else. Now he is a man who has control of the entire unit and all its functions and can recognize when something is going wrong and take corrective steps to bring it back in line. This is probably the most critical job in any plant and the man holding it should be several steps above the average.

Another time an analysis was being made of down-time on the log sheets which revealed one plant had no problem with their dryer chute plugging. This particular problem had caused costly delays and lost production at other locations. The answer was simple. When the granulation unit was installed, it had been necessary to convey the hot, wet material from the ammoniator on an 80 foot long conveyor belt. This short delay of 20 seconds before hitting the dryer chute was enough to cool the tacky salt condition and allow most grades to slide down the chute with little sticking.

This piece of knowledge and many others that were gleaned from the log sheets were incorporated in the engineering and modification of all our plants.

In conclusion let me restate a few basic ideas. As production people with a wealth of experience and information, let's not sit back and wait for somebody else to force us into action. We're in a tough market and the creative use of our knowledge can help the progress of our own companies and the Plant Food Industry as a whole.

Let's take a fresh look at outmoded procedures and methods. We don't expect the present granulation process to exist forever, but it still is a good method and the things we learn now will pave the way towards the future methods we'll be using.

Last, but not least, listen to your operating people. These are the men who can really make things go, but they are not going to if they are hancuffed by inflexible rules. Give them a little room and stand back for results.

MODERATOR MARBURGER: I am sure there are going to be some questions raised. John, you commented, but I am not sure I quite caught what you said about the maximum ammonia that could be used.

MR. HAILEY: I would say about 6 units of anhydrous is about all we would care to put in at the normal rates of recycle, normal heat and with the handling equipment that we presently have. That is while trying to obtain full production on the unit at the same time. Now, if you wish to drop back to 10 tons per hour on a 40 ton hour unit, you can put in more anhydrous.

MODERATOR MARBURGER: Do I also get as a fact, or an overall impression from what you have said that the existing process, the TVA type granulator and granulation process are more capable of performance than what we are presently doing over all?

MR. HAILEY: Grant, I don't think we have fully explored all the possibilities with the TVA granulation system. We have frozen ourselves on a kind of set of values that were established maybe ten or 15 years ago. And there are still a lot of operating techniques that we don't know. We admit we don't know all there is to know about formulation. How can we know all there is to know about the units we are working with? There are things that we haven't even tried yet that we probably should be trying.

MODERATOR MARBURGER: I guess saying this another way is the fact, that instead of now or five years from now or sometime in the future, changing the process completely for new equipment or a new plant, there is still room for being better in all ways than we presently are.

MR. LORTSCHER: In describing your high liquid phase and sensitive use of recycle, what manner of control have you over the recycle brought in, as far as keeping away from surges in it, which would take you out of balance?

MR. HAILEY: Most of our plants have been equipped with surge hoppers and automatic feeders where the operator has a control over the amount of recycle he is feeding. He also can control the amount of recycle he brings back by adjustments on the screen, by changing his oversize flow off the screen into his mills and regrinding some cold recycle he can bring it back intentionally.

MODERATOR MARBURGER: Our next speaker will be Dick Perkins of the W. R. Grace Company, also from Memphis. The title of his paper is Consider Formulation in the Correct Perspective. And actually formulation really depends on your viewpoint. If you are a corporate manager, you don't care about the formula. If you are an ammoniator operator, it might be something sort of hazy in the background but right now you've got to do something else. If you are a plant superintendent, you've got schedules and men and equipment and that neighbor's phone call this morning choking from from pollution. So it really depends on your viewpoint exactly what you say and how you are going to think about a formula. It's like the question that was posed to a number of men asking "What would you do if a burglar breaks into your house at night?" And one man replied, who was the father of six children, all under six. He said, "I would probably get out of bed, take him by the hand and take him to the bathroom." So it really depends on your viewpoint.

Mr. Richard Perkins, please.

"Consider Formulation In The Proper Perspective"

R. H. Perkins

While some resistance to change and reluctance to attempt formula revisions exist in our plants, we have made a major effort for many years to avoid such inflexible thinking. Over the years we have not allowed our formulas to become rigid standards, but have used formulation as the means of producing granular fertilizers using lowest cost raw materials, having the greatest practical quality and optimum rates of production.

How can you avoid the inherent rigidity of thinking so common to plants in earlier years? Much can be accomplished by working closely with plant operating groups; making certain they are aware of the reasons flexibility is needed, emphasizing that being flexible can help their operation and by providing them technical assistance regarding new approaches to be tried. The following have proven of benefit to us in establishing a high degree of flexibility in formulation within our plants over the years.

1. Frequent studies are made of prevailing raw material costs by geographical area and our formulations are re-evaluated in light of any significant raw material cost changes.

2. Plant tests are made frequently to determine if new combinations of materials offering greater economy can be utilized. Detailed information as to how the test should be made must be supplied the plant and a careful review of results of each plant trial is essential.

3. Plants should be provided with equipment suitable for handling and processing a wide selection of raw materials in order to take greatest advantage of formulation benefits.

II. Perhaps some examples of formulation and process development in our plants would be of interest and serve to illustrate the vital part these factors play in our mixed fertilizer operation. The examples were selected from data going back as far as the early 1950's and up to the present time.

1. When continuous granulation systems were installed in our plants beginning about 1950, the the double-shaft pugmill was selected as the means to accomplish our objective. The ability of this equipment to form a hard dense granule and the ability to utilize large amounts of nitrogen solutions with relatively high ammonia content were important factors in this choice. Economics favored use of as much anhydrous or ammonia containing nitrogen solutions as possible.

Continuous trials and development took place for sometime to learn the effect on granulation and nitrogen recovery resulting from factors such as depth of bed in the pugmill, retention time in the pugmill and temperature-moisture relationships. The depth of bed and retention time were controlled by pitch of the paddles and by amount of recycle. The study of temperature-moisture relationships was essential to learn how to control liquid phase, granulation size and to obtain optimum production rates on each different grade produced. Variations in formulation were frequent as different raw materials were tried to accomplish lower cost, higher production rate or better quality of product.

One of the better developments during these early days was that of mixing anhydrous ammonia, nitrogen solution and water outside the pugmill in a pre-mixer. This system develops heat in the pugmill very well with resulting increased granulation as compared to adding each material to the pugmill separately. We still utilize this sytem in several plants today.

Early liquid distribution systems in the pugmills consisted of various arrangements of top entering pipes. This gave problems due to holding back material or the pipes building up with material and provided only limited efficiency of distribution. Some nitrogen loss and, to a degree, evolution of fume was experienced. The pugmill lends itself to a great variety of sparging arrangements to achieve liquid distribution. A constant effort to improve sparger design has been followed due to the continuously greater need to use more economical raw materials. As more and more anhydrous was used, better sparger design was critical to avoid nitrogen loss. Separate acid spargers were necessary, but in close proximity to nitrogen spargers for rapid and well distributed mixing. Some spargers were designed to enter through the end of the pugmill and others designed to enter through the bottom. Pipe spargers of all types were used and machined block spargers with very narrow slot openings were tried and used. Present practice in most of our plants has two or more machined sparger blocks located one directly over the other with all liquids entering pipes through the bottom of the pugmill. The acid sparger is located above the nitrogen sparger. The spargers have narrow slots from two to four feet in length on each side of the block.

Materials of construction of spargers have required change as raw material patterns were altered.

At first stainless stell was used. As phosphoric acid was added, better materials of construction became essential. Hastalloy spargers were designed and tested. At the present time in most of our plants the acid sparger is constructed of Hastalloy "C" and the nitrogen sparger is constructed of T316 stainless steel.

2. Experimental formulations were worked out in the early 1950's to use electric furnace phosphoric acid on one of our granulation plants to produce high analysis grades of fertilizer. Plant tests followed and grades such as 8-24-8, 20-20-0, 13-39-0, 15-15-15 and 16-20-0 were produced. Although the economics of using electric furnace phosphoric acid were not favorable, the data from these tests was very useful in prodicting the recycle capacity and other factors required in a plant to be used in producing such grades. This data has been of great value a number of times in the past 10 to 12 years. It was found such grades were practical to produce if raw materials were available at economical cost and a plant was designed for production of such grades.

3. The use of urea-ammonia- ammonia nitrate solutions was pioneered prior to 1958 in our plants. The basic economics of formulating mixed fertilizers with nitrogen solutions containing anhydrous ammonia promoted the use of solutions containing the highest fixed-to-free nitrogen ratio. During the 1955 period, ammonia - ammonium nitrate solutions which most closely met this need were an 83% ammonium nitrate solution with no free ammonia which salted out at 154 degrees F., a 32% nitrogen solution which salted out at 93 degrees F., or a 37% nitrogen solution which salted out at 48 degrees F. We selected the 37% solution due to its more practical salting out temperature.

Shortly after adoption of the 37% solution, the total nitrogen content of most solutions was increased through production of lower moisture content nitrogen solutions. This reduced the delivered cost of the nitrogen, but changed the salting out properties. The comparable lower moisture content 41.4% solution had a 65 degrees F. salt temperature. The grade pattern and tonnages at several plants justified the installation of heat exchangers, recirculation lines and heat traced piping to permit using the 41.4% solution in cold climates. Smaller plants continued to rely on 37% nitrogen solution.

It was noted that an ammonia - ammonium nitrate solution containing 11% urea was available with a fixed-to-free nitrogen ratio very similar to the original 37% solution. One great advantage, however, was the salting out temperature of 7 degrees F. This was significantly lower than regular nitrogen solutions in use. Since very little information was available on limitations as to the quantity of urea which could be used without effecting quality of product, a limit of 50 lbs. urea per ton of product was tried. This proved satisfactory on our product and the urea solution was used. The use of the 11% urea solution was tried in high 1-1-1 ratio grades without success. The granulation was satisfactory, but the granules later broke down in storage because of the higher percentage of urea they contained.

The property of the urea containing solutions of lower salting out temperature warranted use of these materials. Eventually solutions containing 6 to 7% urea were available and these were used very successfully in several plants so long as a maximum of about 50 lbs. of urea per ton of product was formulated.

4. During the late 1950's formulation studies showed wet process phosphoric acid should be used for greater economy. The use of wet process phosphoric acid and combinations of anhydrous ammonia and nitrogen solutions was begun in most of our plants. Studies of raw material costs at individual plants dicated the amounts of single superphosphate, triple superphosphate and phosphoric acid to be used to achieve a least cost formula. The ability of the pugmill to handle a relatively wet bed of material was a real asset during this period.

5. Limitations in the amount of phosphoric acid and anhydrous ammonia which could be used in the pugmill without sacrificing too much in production rate dicated the need for further process change as analysis of grades increased. The production of 16-48-0 and 18-46-0 was begun and the demand for these products in addition to other high analysis grades caused us to install a preneutralizing tank system in one plant in 1960. A T316 stainless steel tank was installed for reaction of anhydrous ammonia and phosphoric and sulfuric acids. This slurry was gravity fed onto the bed of material in the pugmill. The production rate was increased in this manner and lower cost achieved through use of more economical raw materials. Nitrogen recovery was better on the high analysis grades.

The original pre-neutralizing system is still in use producing grades such as 14-28-14, 16-20-6, 18-46-0, 8-32-16 and 6-24-24.

6. A complete new plant was designed in 1966 to achieve the highest practical grades using anhydrous ammonia and regular 54% wet process phosphoric acid. The plant has operated very well producing grades such as 7-28-28, 9-36-18, 7-18-33 and lower analyses such as 6-24-24 and 8-32-16.

We have found the 7-28-28 to be considerably easier to granulate than 6-24-24 due to the higher liquid phase during granulation.

The slurry from the pre-neutralizing system is pumped and sprayed onto the bed of material being granulated. By providing a flexible system for spraying the slurry into the granulator a close control of granulation is possible. We are able to adjust the spray nozzle location and by adding steam to the spray nozzles some control of slurry distribution and droplet size is obtained.

This plant also was designed to produce high nitrogen to phosphate ratio grades utilizing nitrogen solutions. Production of 17-17-17 and 20-10-10 using 37% nitrogen solution, phosphoric acid and sulfuric acid in the pre-neutralizing system is regularly scheduled.

The recycle requirement is relatively high for such grades but this capability was designed into the plant.

The use of ammonia - ammonium nitrate solutions in the pre-neutralizer with a mixture of phosphoric and sulfuric acids has proven to be very easy to handle. The resulting slurry is considerably more fluid and easier to handle than a mixture of ammonia and phosphoric acid alone.

7. Changing raw material prices the past year or two have caused us all to carefully review past formulation practices. In some cases continued use of a standard raw material can become prohibitively expensive. This was the case in one of our modern plants where the cost of sulfuric acid to produce single superphosphate became a serious matter. We had already begun a program to switch as much phosphate as possible from the single superphosphate source to more economical triple super and phosphoric acid. The use of single superphosphate was being limited to low analysis grades. Trial formulations looked less than promising due to the large amount of filler to be granulated on low analysis grades when only triple super was used to supply the phosphate. Plant tests were made and the operating people learned to handle the more difficult granulation on lowered production rates. The rate was increased gradually and the plant now operates at production rates as high as those previously used without the use of any single superphosphate. Fairly large amounts of filler can be used when a plant has learned to handle the more difficult granulation technique.

III. Any study of raw material costs will show a less expensive way to product mixed fertilizer than you are using. However, actual formulation must take into consideration many factors other than the simple cost of raw materials. Aside from the limitations of basic formulation practice, a compromise must always be reached by considering most of the following factors.

1. *Geographic location and grade patterns* – In an area where low analysis grades are predominant, the use of large amounts of economical phosphoric acid and anhydrous ammonia is not practical because too much filler will be required. In states where large amounts of tobacco are grown you cannot formulate muriate of potash or anhydrous ammonia just because they are lower in cost than sulfate of potash and other nitrogen materials. A major problem exists for anyone who has a plant which is tightly restricted as to the grade ratios they can produce. For instance, a nitric phosphate plant that is restricted to

high nitrogen to phosphate ratio grades may have trouble in several areas of the country today where these ratios are not used in large quantity.

2. *Effect on product physical quality* — We know many materials are not compatible when used together or when used in certain proportions in mixed fertilizers. Experience has taught us that there are limits in our choice of materials used in various grades because of problems caused by the mixture being very difficult to granulate or showing poor storage characteristics. If we use lower cost materials that cause the product to set hard in bulk piles or bag storage we may lose some customers. Some grades will pickup moisture in bulk storage resulting in product deterioration and others may experience degradation of the granule in storage or handling if the wrong raw materials are used or used in the wrong proportions.

3. *Production rate* — Care must be taken in selecting the amount of certain raw materials to be used. They may result in satisfactory physical quality and meet chemical analysis, but cause the production rate to drop sharply. The requirement for recycle may increase with less product being sent to storage. This gives higher processing costs which can offset savings in raw material cost.

4. *Plant equipment limitations* — Many times a plant facility finds that it cannot take advantage of some lower cost material because no provisions were made for handling or storing such a material. Thus some otherwise desirable materials may be ruled out at particular locations. Many plants have lacked facilities for unloading, storage and metering of phosphoric acid. Some lack anhydrous ammonia capability. Some have marginal facilities which permit the use of materials, but in only limited quantities. Such limitations can be corrected, but it usually requires time to accomplish.

Even more serious limitations are often found in the processing equipment in a plant. Many times a plant may lack overall load capacity to carry high enough recycle ratios to use certain materials which cause higher liquid phase during processing. Frequently the drying or cooling capacity of a plant is a major problem. Higher analysis materials are often heat sensitive and require much lower drying temperature and longer drying retention time. The motor size on specific equipment may be too light for heavier loads required by use of different materials. Oversize crushing equipment is frequently inadequate when some raw materials are used. If recycle requirement is increased substantially the screening surface may not be sufficient to size product properly. Some types of granulating and ammoniating equipment are more flexible than others and allow use of a greater choice of raw materials.

5. *Operating personnel limitations* — Unfortunately, in many areas today it is almost impossible to replace experienced operating personnel. Over a period of time in such areas, the quality of the operating labor and their overall capability decreases to the point that much of the flexibility in raw material choice is removed. As experienced men are lost, the replacements often are not able, or will not perform the work as well as the men they replace. Less effective operation may result with a lower plant throughput or decrease in quality. In such a situation the plant needs as few problem raw materials as possible.

6. *Increased losses in raw material handling and processing* — At times the anticipated savings from use of lower cost materials may vanish because of increased losses in handling or storing the materials. Some raw materials are subject to high loss unless a plant is provided with efficient means of handling them. A good example is wet process phosphoric acid. If stored in an unagitated storage tank a high percentage of loss can occur as sludge precipitates out in the storage tank. Bulk and ammonium nitrate are subject to loss due to moisture pickup and caking in storage.

Greater shrinkage during processing is another area where formulated savings can quickly disappear. A change in raw materials may result in greater loss of nitrogen during ammoniation or drying. Plants having high losses of particulate from air handling systems may experience greater losses if higher analysis raw materials are used. Improper formulation of some materials can cause higher insoluble phosphate results with reduced availability of phosphate.

7. *Pollution Control* — Changes in formulation can have a great deal to do with the nature of gases and waste water discharged from a plant. It is readily seen that imprudent formulation of excess anhysrous ammonia can cause loss of more nitrogen to the atmosphere. Many high analysis raw materials are extremely heat sensitive and are easily overheated in the drying operation. This may result in driving off nitrogen and cause a fume condition.

Some increase in particulate loss can occur in plants having inefficient air handling systems if more dusty materials are introduced to the system. In the case of plants equipped with wet scrubbers this greater loss may take place in the form of contaminated waste water rather than as airborne particulate. Usage of large amounts of sulfuric acid and ammonia can cause serious generation of ammonium-chloride fume.

These and similar results of unwise formulation are becoming causes of increased concern today.

In summary, a plant today must be operated by supervisors who are highly flexible in attitude and must have the processing capability to constantly strive to utilize to the greatest degree possible the most economical selection of raw materials. Frequent reviews of material costs are necessary. Don't hesitate to consider use of any low cost material, but realize that there are many limitations involved and contemplated savings do not always materialize if an important factor is overlooked.

MODERATOR MARBURGER: Thank you, Dick.

Our next speaker needs no introduction. Bob Heck, with International Minerals and Chemicals. Bob, I will turn it over to you.

MR. HECK: He said I needed no introduction and I got none.

Gross Heat Effect In Granulating N-P-K Fertilizers

Robert R. Heck

One of the questions that our sales and production people ask each year is, "How can we increase the tonnage throughput in our granulation plants?". Sometimes these increases are needed to increase total output and at other times, they are desirable to meet heavy seasonal demands.

In attempting to increase tonnage, we usually start by testing the effects of a small increase first – say from 20 TPH to 22 TPH. If this is successful, then we will try another small increase – say from 22 TPH to 25 TPH. These tests are made on the assumption that incremental increases will measure the success without creating problems such as the build-up of excessive recycle or excessive over-agglomeration.

In order to better understand the conditions under which our plants could be operated, we conducted a series of tests, starting in 1967, to measure the effect of throughput increases on granulation characteristics.

These tests were based upon a hypothesis formulated by John Medberry and Frank (Slugger) Nielsson of IMC and resulted from an observation that some of our plants were using less nitrogen solution or less BTU's per ton than others to formulate the same grade of fertilizer.

In an attempt to determine the differences between the plants, an analysis was made of three variables that existed in the ammoniator-granulator drums of the different plants, i.e., bed volume, bed surface, and heat loss area.

Data on three different units is tabulated as follows:

	A	B	C
Ammoniation Section Dimension	8'x10'x20"	7'x10'x14"	6'x10'x14"
Volume of Bed, Cubic feet	76.0	42.2	38.7
Bed Surface, Square feet	65.0	52.2	47.5
Heat Loss Area Square feet	91.1	72.2	62.6

The two different formulas that were being used in our plants are as follows:

	FORMULA 1			FORMULA 2		
RAW MATERIAL	**POUNDS**	**BTU's**	**H_2O**	**POUNDS**	**BTU's**	**H_2O**
66° H_2SO_4	172	151,250	12	91	79,750	6
448 Solution	458	---	27	356	---	21
Sul. Ammonia	---	---	--	214	---	0
R/P Super	1,025	88,800	62	1,025	88,800	62
60% Mur. Pot.	334	---	0	334	---	0
Dry Filler	92	---	0	49	---	0
Evaporation	-81	---	-81	-69	---	-69
	2,000	240,050	20	2,000	168,550	20

Based upon many years of experience at a plant using the 6' drum, we felt that the practical production limit for a 10–10–10 made with 10 units of N from solution 448 was 10 TPH in this 6' drum.

At 10 TPH the capacity of the drum was 2,400,500 BTU's per hour when using Formula 1. If reaction volume governs bed behavior, the limit through a 38.7 cubic foot bed is 62,028 BTU's/ft.^{3}/hr. $\frac{(2,400,500)}{(\ 38.7\ )}$ If bed surface volume governs bed behavior, the limit is 50,527 BTU's/ft.^{2}. If heat loss surface area governs bed behavior, the limit is 38,347 BTU's/ft.^{2}/hr. At this time we were not aware of the exact effect of any of these factors but felt that they were interrelated and that one was probably dominant.

Having established the three limiting factors, we applied them to other ammoniating drums.

A. With Bed Volume as the limiting factor:

1. **Using 7' drum.**
   62,028 BTU's/ft.^{3}/hr. x 42.2 ft.^{3} = 2,617,582 BTU's/hr.
   - **(a) Using Formula 1 (10 units N from solution)**

     $$\frac{2,617,582 \text{ BTU's/hr.}}{240,050 \text{ BTU's/T}} = 10.9 \text{ TPH}$$

   - **(b) Using Formula 2 (8 units N from solution)**

     $$\frac{2,617,582 \text{ BTU's/hr.}}{168,550 \text{ BTU's/T}} = 15.5 \text{ TPH}$$

2. **Using 8' drum.**
   62,028 BTU's/ft.^{3}/hr. x 76 ft.^{3} = 4,714,128 BTU's/hr.
   - **(a) Using Formula 1**

     $$\frac{4,714,128 \text{ BTU's/hr.}}{240,050 \text{ BTU's/T}} = 19.6 \text{ TPH}$$

   - **(b) Using Formula 2**

     $$\frac{4,714,128 \text{ BTU's/hr.}}{168,550 \text{ BTU's/T}} = 28.0 \text{ TPH}$$

B. With Heat Loss Area as the limiting factor:

1. **Using 7' drum.**
   38,347 BTU's/ft.^{2}/hr. x 72.2 ft.^{2} = 2,768,653 BTU's/hr.
   - **(a) Formula 1 = 11.5 TPH**
   - **(b) Formula 2 = 16.4 TPH**
2. **Using 8' drum.**
   38,347 BTU's/hr./ft.^{2} x 91.1 ft.^{2} = 3,493,412 BTU's/hr.
   - **(a) Formula 1 = 14.6 TPH**
   - **(b) Formula 2 = 20.7 TPH**

C. With Bed Surface Area as limiting factor:
50,537 BTU's/ft.^{2}/hr. x 52.2 ft.^{2} = 2,638,031 BTU's/hr.

1. **Using 7' drum.**
   - **(a) Formula 1 = 11.0 TPH**
   - **(b) Formula 2 = 15.7 TPH**
2. **Using 8' drum.**
   50,537 BTU's/ft.^{2}/hr. x 65 ft.^{2} = 3,284,905 BTU's/hr.
   - **(a) Formula 1 = 13.7 TPH**
   - **(b) Formula 2 = 19.5 TPH**

Based upon the "guesstimates" of our technical personnel, we assume that the effect of the three factors on production rates could probably be weighted as follows:

Bed Volume	60%
Bed Surface	30%
Shell Area	10%

Now the combination parameters for the 8' drum can be calculated as follows:

(.60 x 4,700,00) + (.30 x 3,500,000) + (.10 x 3,300,000) = 4,200,000 BTU's/hr.

Using this calculated maximum heat and release, we can now estimate the maximum production rate using the two formulas given earlier:

$$\text{FORMULA 1} = \frac{4{,}200{,}000 \text{ BTU's/hr.}}{240{,}050 \text{ BTU's/Ton}} = 17.5 \text{ TPH}$$

$$\text{FORMULA 2} = \frac{4{,}200{,}000 \text{ BTU's/hr.}}{168{,}550 \text{ BTU's/Ton}} = 25.0 \text{ TPH}$$

These rates agree closely with the experience we have had in granulation plants using the two formulas in 8' x 10' x 20' ammoniating drums.

Estimates for the plants using a 7' x 10' x 14' drum shows the following:

1. 7' x 10' x 14' drum.

$$\text{FORMULA 1} = \frac{2{,}660{,}000}{240{,}050} = 11.0 \text{ TPH}$$

$$\text{FORMULA 2} = \frac{2{,}660{,}000}{168{,}550} = 15.8 \text{ TPH}$$

These rates also agree closely with the experience we have had in granulation plants using 7' x 10' x 14' ammoniating drums.

Based upon our previous assumptions and realization of the predicted tonnage rates, we considered another application for the ammoniator heat parameter, i.e., determining production rate of low-heat formulae to attain optimum granulation.

A test was run in a granulation plant using an 8' drum. The two formulas tested were as follows:

5-10-15

	FORMULA	BTU's
1.	72 lbs. NH_3 equivalent from 490 solution	---
	80 lbs. 60° Be' H_2SO_4	51,660
	51 lbs. NH_3 neutralized by normal super	75,480
	TOTAL . . .	127,140 BTU's/T

$$\text{Optimum production rate calculated} = \frac{4{,}200{,}000}{127{,}140} = 33 \text{ TPH}$$

	FORMULA	BTU's
2.	57 lbs. NH_3 equivalent from 440 solution	---
	40 lbs. N from $(NH_4)_2SO_4$	---
	80 lbs. 60° Be' H_2SO_4	51,660 BTU
	36 lbs. NH_3 neutralized by normal super	53,280 BTU
	TOTAL . . .	104,940 BTU's/T

$$\text{Optimum production rate calculated} = \frac{4{,}200{,}000}{104{,}940} = 40.0 \text{ TPH}$$

These rates are almost identical to the rates realized in a granulation plant using an 8' drum.

In granulating products which had a low heat release per ton we found that we achieved a bed temperature condition more conducive to formulation of granules in the proper size range when the ammoniator is "crammed" with reactants. The chemicals reacting under these conditions generate the amount of heat per unit of volume or per time interval which produces the optimum granulation condition in the ammoniator. As we have developed earlier, this optimum condition is thought to prevail when the heat output from the ammoniator is 4,200,000 BTU's/hr.

In the development of the ammoniator heat parameter, we have disregarded effects on granulation attributable to formula water, raw material particle size, soluble salts contained in the formula, etc. We have also ignored another facet which may be of interest to you regarding the relative cost of the two formulas.

At the time that Formula 1 and 2 were used in the tests, the raw material cost for Formula 2 was $0.11 higher than for Formula 1. In out plants we found that the increased production rate provided an increase in profit far in excess of $0.11 per ton. In addition, we also found that we could reduce the amount of steam required in granulating low nitrogen (low heat) grades. If our assumption that 4,200,000 BTU's/hour are required is valid, then a plant producing 5-10-15 with 57 pounds of ammonia (104,940 BTU's/hr.) at a rate of 25 TPH would only generate 2,624,000 BTU's/hr. The additional heat (4,200,000-2,624,000 = 1,576,000 BTU's) would have to be provided with steam. Sixteen hundred and forty five pounds of steam/hr. at 212 degrees F. would be required to provide 1,576,000 BTU's. This could very well require 2,000 cubic feet of gas per hour in the boiler. If gas costs $0.40 per 1,000 cubic feet, the additional cost would be $0.032 per ton of product. The steam also adds water which must be removed with expenditure of fuel in the dryer.

In closing, I think it only reasonable to state that we haven't discovered anything new, nor have we duplicated Aladdin's Lamp. One rub of the hypothesis predicated in this paper doesn't cast a magic spell over our everyday problems. At the most, we have exposed a new way to look at an old problem.

In the future, I can visualize tying together, through the "miracle" of the computer, the concepts proposed in this paper and that of other speakers today; TVA's work on acid addition rates and sparger lengths; Payne and Daniel's work on computer formulation; Gilliams' work on liquid phase and many, many other works of equal value.

When that time comes, we can honestly say that we have changed our methods from trial and intuition to the basic methods of science. Until that time comes, however, let's give a vote of thanks to the plant personnel whose curiosity and intuition continues to advance the art of granulation and whose patience leads us to new methods of sciences.

MODERATOR MARBURGER: Thank you, Bob. Are there any questions?

MR. HECK: May I say something before we take questions?

MODERATOR MARBURGER: By all means.

MR. HECK: I just talked to the Sheraton-Peabody Hotel in Memphis and they have confirmed November 4th, 5th, and 6th of 1970 for the Fertilizer Industry Round Table meeting. So that will be the same week in November next year as we are having this year.

MODERATOR MARBURGER: I believe there was a question back there.

A MEMBER: I would like to know whether the last speaker arrived at the optimum granulation temperature. Did you arrive at the optimum granulation temperature?

MR. HECK: No, I don't think so. Only through the principle that we could ger 4,200,000 BTUs per hour through our ammoniating drum.

I think this is going to vary. The temperature is going to vary with the grades we are making and the raw materials we are using.

MEMBER: At the time at which you granulated the best or your optimum granulation, you are not aware of the temperature at that point. Is that right?

MR. HECK: I'm sure that temperature was recorded but I don't have it with me. Mr. Medbery, who is the co-author of the hypothesis may be able to help us there.

MR. MEDBERY: The measurement of the temperature was right at 200 degrees as it exited the ammoniator, 200 Fahrenheit.

A MEMBER: Mr. Perkins mentioned the problem of compatibility with materials. I wonder if anyone has found any problem with the interrelationship between DAP, phos acid, triple and super, possibly all used at the same time. Is there any problem involved here, or should there be any limits in the use of either one of these materials in such instances?

MR. PERKINS: We have had one case where this was a problem. This was during the production of 6-24-24 using a preneutralizer. It seems that the particular production of run-of-pile superphosphate that we used,and overflowed the ammonium phosphate slurry onto, caused a sticky condition on this particular grade. We were able to get away from this by changing the proportions of run-of-pile superphosphate.

MODERATOR MARBURGER: Another question?

MR. JOHN MEDBERY: I'm John Medbery with IMC. I would like to ask the TVA gentlemen: On running grades which contain no nitrogen, have you tested these with the computer method to evaluate and prove the necessity for the heat and the moisture. in other words, the 700 pounds of liquid phase giving an equivalent amount of steam or water or the two in combinations?

MR. H. L. BALAY (TVA): I think the answer is no we haven't really tried to formulate any grades with no nitrogen. However, in my own experience, I do know that it takes a little heat and a little liquiphase, from whatever source, to granulate these grades. And we do allow a little flexibility. Now, we show 180,000 BTUs up there on Jon's paper. However this is not a hard and fast number. We can vary this.

I believe that we could maybe pick 165,000 BTUs or something like this. But these variations could be made. All we would have to do is ask the computer for these variations. Now, these formulas, I kind of like to think of the stage we are in now as something like a TV set. We have a channel knob and we have a fine tuner. Now, the computer will get us on the channel but sometimes it takes, from the engineer's standpoint, a little fine tuning to get one in the plant that will operate. However, from my personal point of view, this computer has relieved me of a lot of trial-and-error formulation. In other words, it does get the channel for me. All I have to do is the fine tuning. And I don't think Jon or anyone else says that we have the final answer. But it is an advance from a practical point of view.

This is one thing that we haven't investigated. If we did, I think the approach would be, as Mr. Heck has approached it, to find formulations that have worked in the past and base our changes on the chemical and physical properties of these and rather approach it from a practical standpoint. I do not know whether that is any answer or not but it does maybe point up the state of flux that we are in right now.

MR. MEDBERRY: Yes, that is about the kind of answer I expected. Let me say this. It makes a difference, if the theory works, if the 180,000 BTUs is a key number and the 700 pounds of liquid phase is a key number. Then you can see how steam and water can be used interchangeably or in combination and an economics factor applied here, depending on the cost to generate steam or the cost to evaporate water, in which case a great deal more water would be needed than steam.

MR. BALAY: In Jon's paper he pointed out one formulation and said that the engineer looked at this and said that it had too much steam in it. Now, I think maybe that engineer he was talking about may have been me. And it has been my experience that if you overdo the steam and the water, your granules are not hard and they tend to break down.

Now, we make 0-20-20 or 0 grades from steam; we do a pretty good job, but still these granules are not hard, as are the granules produced when more salts, more soluble salts, are present. I think we have tended to steer away from this for this reason. And another reason is that 0 grades are something we just haven't worked on. And maybe we need to do more work here. As you say, this is probably the answer you expected, but this is the way it is. And I think the value of this, of a meeting of this sort, is to pinpoint this sort of thing, the problems, and to move on and not to stand still and to get to work on these problems if we don't have the answers.

MR. HECK: I would like to ask a question too on this computer formulas. One of the problems we have with our computer formula program is that we don't know enough about the effect of particle size on granulation characteristics. So if we offer our computer both standard and coarse potash, for instance, it will always reject coarse because it is highest cost. Or if we go to even what we call special standard and standard, it will always reject the highest cost element without any other considerations being made. Have you done any work on the effect of particle size of raw materials on granulation to determine if you can force in a more expensive item and get either increased production or increased quality?

MR. BALAY: Do you want to answer that, Jon?

MR. JON NEVINS: Let me just make one comment. Basically this. I think the term liquid phase coefficient for the number that we had is a little bit unfortunate. After all, this is an empirical index.

Now, we chose zero and .3 as the spread on the two types of potash. Of course this would depend a lot and I think this is something you would adjust. But if you have steam and water or have steam availalbe, you're right, it will take the cheapest. However, there are cases where, if you price the steam out, the liquid phase value of the coarse potash will make it come in.

MR. BALAY: I would like to say this. If you noticed these tables that Jon had, we have given coarse potash a granulation index of 2/10ths; fine potash got a zero. And on this basis sometimes the computer will bring coarse potash in. Now, as Jon says, we call this liquid phase. How you relate whether something is coarse or fine or not too liquid phase, there is no real relation here. However, we called it something, is what we called it. But it is a granulation index.

MR. NEILD (Kerr-McGee): If I might ask another question I do not believe this was discussed today. In our plant we find that some trace minerals, or minor elements or whatever you want to call them, in fine powdery condition seem to interfere with granulation. Specifically manganous oxide and magnesium oxide. I wonder if you can give us any idea of what might be used to overcome this. We're talking about rates up to about 60, 80 to 100 pounds per ton. We found that at 100 pounds per ton of some of these materials, granulation was almost impossible in grades that would otherwise granulate pretty well.

MODERATOR MARBURGER: John Hailey, would you like to answer that one, or do I have another volunteer?

MR. HAILEY: I'll take a whack at it. Any of the oxides are going to behave like any of the other basic materials such as ammonia and you have to figure in your acid and your heat ratio similar to the way you would figure it for ammonia or for limestone being in the formula. If you don't allow for a certain amount of excess acid in there, yes, you are going to have difficulty. You can produce some heat with these materials also.

I don't say that is a total answer. We have trouble too, when you get too much of these materials. You can adjust your formula with a little excess acid sometimes and come out with some granulation that is better than what you were doing before.

MODERATOR MARBURGER: Dick Perkins do you have any further comment?

MR. PERKINS: I don't think so, Grant.

MR. BALAY: I would like to ask Mr. Hailey and Mr. Perkins if, when they use large amounts of filler, as we are tending to do these days because of the low cost of phosphoric acid and triple, they run into any problems with analysis, due mainly, I would think, to segregation. I have run into those and I would like to know what their experience has been.

MR. PERKINS: I think Mr. Hailey used the most filler. I'll pass to him and then stand by for rebuttal.

MR. HAILEY: Getting back to the filler. I think it depends on your sizing of your filler as much as anything as to whether you achieve good granulation and/or segregation of your particles. The example I gave was where this plant manager had examined various fillers and then finally found one that was about the right size. He is achieving very good granulation and he is achieving very little segregation and getting good analysis using as much as 1,000 to 1,200 pounds of filler.

He is coating each particle. Actually the filler is acting as a seed and he's coating each particle with a very high liquid phase formula. So no, actually we have gotten very good analytical results with high filler rates without serious separation problems.

MODERATOR MARBURGER: I would think this would be the same, really the same question as when in a grade, particularly a high-phosphate, high-potash grade like 5-20-20, the analysis is under on one and over on the other and why? The why would be generally answered by the fact that it had not completely granulated and therefore the segregation showed up in analysis.

MR. HAILEY: Just as a side issue here, which would you recommend when you are starting into a computer program, training a fertilizer man to learn to be a programmer or a programmer to be a fertilizer man?

(Laughter.)

MR. NEVINS: I made the comment toward the end and I'm quite serious about it. The whole concept of operations research revolves around this team approach. And you have to admit right away that nobody knows the whole ballgame here. And it really does take the working together of quite a few disciplines to come up with something that plant operators can use and engineers can read and what-have-you. So far as training goes, I think many of the larger companies have an operations research group and they can do a linear programming problem for you. It's quite simple, of course. To get it more to the operational stage and to advance it somewhat more, they are obviously going to have to be trained somewhat in the formulation of fertilizers.

MR. BALAY: Yes. I think it's teamwork. If we have got a little time, a minute here, Grant, I think it might be sort of interesting how we got into this. We are sort of a tech service group. We work with all kinds of granulators. And we generally have to come up with a standard formulation that we can take to a granulator.

We are using many materials that TVA develops. We ask ourselves will these granulate? Are they economical? And sometimes you have to formulate, like I do, by trial and error. And I have tried simultaneous equations and I soon decided that trial and error was probably a little better. You may run into two full day's work trying to resolve some of these questions, in other words, to get on channel. And the way we are now formulations, I might add, have been successful beyond my expectations. Not 100 per cent but the batting average is pretty good.

I had the opinion that the thing wouldn't work, but Jon and others have shown me that it would. And this has saved many, many hours of my time, by getting us in the ball park in a very short time on some of these things. Now, we have to add quite frequently another factor for a plant, because every plant we work in, as you well know, is different.

I think this sort of approach might have some real value especially the way prices fluctuate, in coming up with a study almost immediately of whether some new lower priced material is feasible and worth further study.

MODERATOR MARBURGER: I would like to

comment, Bud, that most of us don't know a constraint from a miniskirt.

MR. NEILD: I'm a plant operator myself and I can't help but ask this question. You fellows that run the computers, when do you shoot these to the plants, during the busy season or do you provide a little time off-season to get some of these science and art things combined?

MR. BALAY: I think we're fortunate in a way. The plant operators won't have anything to do with us in the busy season. We're able to approach this thing from a Let's-try-it and-see standpoint, it's worthwhile, because there is a chance we can save you some money, and we can do this without really costing you too much. It is generally an off-season sort of thing, because, as you said in your question, you may do anything, cost or otherwise, to get production during the season and you certainly don't want people like us interfering with production. However in off-season it is worthwhile to try these things. Usually there is time, personnel and there is equipment available to try it, and next year it might result in a saving in formulation.

MODERATOR MARBURGER: I think that was further brought out by the diversity of the papers this afternoon in that it is equipment, time, personnel, and a great number of other things that have to be considered with the formula. Pat, I think what you were just bringing out is we can do this some time when we are not pressed for seasonal production.

MR. MARBURGER: I have a great deal of personal appreciation for the great amount of work that has gone into this program by each panel member and I would like to thank them for it. I also appreciate the time on the program and I would like to turn it back now to Billy Adams.

MR. ADAMS: Thank you, Grant, for this very stimulating discussion. We think this was what the group wanted and our Committee has worked very hard to work these panels back in the picture and we will continue more of it next year, that is, if you think in that direction, and I'm sure you do, from the remarks that were made earlier.

One thing I would like to put my two cents in on, when they were talking about the computer, I would like for them to put a number in there for pollution,along with it,so that all these formulas come out as non-polluting.

If there is no more business to be taken care of at this time, I would like for us to give all of the panel a hand.

(Great applause.)

Meeting adjourned at 5:15 o'clock p.m.

Friday Morning Session, November 7, 1969

The Round Table Meeting reconvened at 9 o'clock a.m.
Albert Spillman and Billy Adams, Moderators

CHAIRMAN SPILLMAN: Good Morning Ladies and Gentlemen: First, I wish to make several announcements. We are priveleged to have visitors from outside the United States. We have quite a number from Canada, who have, from the beginning of The Round Table, shown considerable interest in our Proceedings. In addition we have with us visitors from France, England, Mexico, Australia, Porto Rico, Kenya, Sweden, Germany and Costa Rico. Total, 23 representatives. Thank you for coming and contributing to the cause. Hope you had an enjoyable stay. We are looking forward to having you at our 20th Meeting at Memphis, November 4-5-6, 1970. Most of these people were present. They stood up and received a good round of applause.

During this meeting all messages received were placed on the bulletin board at the registration desk. If you have not already acknowledged your calls please check.

I wish to take this opportunity, on behalf of all of us, to thank our entertainment committee: Tom Athey, Chairman, Paul Prosser and Walter J. Sakett, Jr. for arranging that excellent cocktail hour Thursday evening. Everyone enjoyed the party and the opportunity to chat and meet one another. Thanks to our hosts who very kindly financed the costs:

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It is my privelege to call on Mr. John L. Rodgers, Manager of Engineering, Central Farmers Fertilizer Co., Chicago, Ill. Materials, mixed goods, etc., shrinkage in the fertilizer plants has bugged us many times. Numbers of studies have been made individually by many of us who carried the responsibility for inventory and quality control. There are many angles to help control the shrinkage problem. Mr. Rodgers will give us some of his highlights on this vital subject.

Shrinkage In Fertilizer Plants

John L. Rodgers

It is a privilege to be asked to elaborate on a topic as old as the fertilizer industry itself – shrinkage – and its measurement and control. For as surely as we have endeavor which through any manufacturing process results in a saleable product, there will be somewhat less of this product than was received in the constituent raw materials. Shrinkage is a matter of fact, as well as matter of degree.

You will note that the following discussion concerns in a particular type of fertilizer plant which supposedly reached its peak popularity a few years back but which continues to thrive, despite prophesies to the contrary, because it satisfies a demand. I refer of course to the chemical mix granulation plant. In this regard, Central Farmers member companies own 40 of these granulation plants, including two just recently built, and we know that not only is the operation of some of them economically justified but also that the companies who operate them are holding their own in the marketing areas they serve. Therefore, in examining reasons why some granulation plants are profitable while others are certainly not, the effect of shrinkage must not be overlooked as a factor. Moreover, while potential shrinkage in bulk blend operations may not be as great as in chemical mix plants, neither should it be overlooked; and some of the basic rules for measurement and control apply, with modifications, to each type of operation.

I would like to outline a statistical approach to shrinkage measurement. The objective of this program was and is to correlate shrink data from several granulation plants in such a manner as to be able to compare the performances of individual plants and, of even more importance, to be able to place proper emphasis on reducing losses of fertilizer materials. The success of the program depends on gaining a knowledge of the true operational experiences of the plants involved; and this, in turn, requires that measuring bases must be uniform according to established definitions:

Number One: Shrink is the difference between the materials received in a plant and the materials removed from the plant, including the change in internal inventory.

Number Two: Materials bought and materials sold are N, P_2O_5 and K_2O. Shrinkage is measured in terms of unit-tons of these plant foods.

Number Three: The total measure of materials in and out of a plant during a specified time period constitutes the material balance. A nominal material balance is based on grade numbers of materials shipped and nominal analyses of raw materials received. An actual material balance is based on the true chemical analyses of raw materials, inventories and products.

Number Four: Economic loss is the loss of N, P_2O_5 and K_2O due to the difference between the actual quantities of raw materials available for use less the nominal quantity shipped as grade numbers during the same time period.

With the use of the above definitions in our shrink program, forms were prepared for recording the activities of receiving materials, manufacturing, shipping and taking physical inventories at yearly intervals at the several plants included in our program. The top of figure (1) is an example of the materials received report. Note that each of the common materials is already listed, with blank spaces provided for others. In use, yearly tonnage receipts of individual materials are totalled from plant records and recorded on this sheet. At the same time, the nominal and the actual units of N, P_2O_5 and K_2O are calculated from the receipts, totalled at the end of the year, and also recorded on this sheet. The second heading shows the materials shipped report. It is very similar to the materials received report, except that the list of raw materials is replaced with grade numbers shipped. The third heading shows the ending inventory sheet, again quite similar, which becomes the beginning inventory sheet for the next year.

These four sheets are completed after the close of inventory generally corresponding to the end of a fiscal year, and are then used to calculate yearly plant food losses for a single operating plant. Figure (2) shows this calculation sheet and its relative simplicity. Totals from the four work sheets are transferred to this sheet and the loss of each plant food calculated as the difference between the units received and in the beginning inventory, less the units shipped and in the ending inventory. Finally, the percentage loss is calculated by dividing the loss into the material shipped. Considerable attention and effort is required to successfully carry on a continuing shrink program of this nature:

First of all, the calculations engendered by material receipts and shipments must be done routinely and are dependent on the availability of analytical information from suppliers and from internal or commercial laboratories. Second, physical inventories must be as accurate as possible, both as to quantity and plant food content, putting additional stress on sampling and analysis. The point is that in order for a detailed program of shrinkage measurement to be successful, a plant manager must be convinced of its value and must be prepared to expend the effort and money to do a thorough job.

As would be expected, this mode of management shows that the nitrogen losses are highest, P_2O_5 next highest and K_2O least. Another significant point is that operating on the basis of close analytical control of raw materials and products can reduce the economic loss somewhat but that the major part of shrinkage is still within the plant. To further point out the persistance of plant food losses, even after continued measurement there is no marked tendency for a continuing decline in shrinkage of any of the plant foods except in the case of some specific plants on specific nutrients. The good plant manager reduced his nitrogen losses and maintained them at low levels, despite the averages of several plants over a period of years. This again means taking positive steps to determine what is causing the loss, making a thorough study of the situation, including physical measurements, examining formulations and process procedure, developing recommendations and following through with the necessary action to promote the improvement.

Although we don't mean to imply that the losses of P_2O_5 and K_2O are unimportant, there is obviously a greater potential benefit in determining the reasons and extent of nitrogen losses, and we have concentrated our efforts in this area. Knowing that nitrogen losses do not regularly show up as high nitrogen analyses in products, we concluded, along with plant managers and most of the residents within a 5-mile radius of fertilizer plants, that most nitrogen is lost as ammonia from granulation stacks. To pursue this suspicion on a more quantitative basis we developed a procedure and assembled test apparatus for measuring the ammonia and solids content of gas streams. Figure (3) shows the essential apparatus, which consists of a replaceable, sharp-edged probe tip, a 1/2" diameter stainless steel probe, a thimble holder connected through plastic tubing to a bottle rack, a small rotameter and a vacuum pump. The thimble holder contains a replaceable paper filter. The bottle rack contains first a dry bottle, two bottles of measured quantities of standardized sulfuric acid containing methyl red indicator, followed by another dry bottle. In use, gas is drawn through the assembly at a pre-determined rate and carefully timed, until either the first acid bottle is neutralized and changes color or for 30 minutes, whichever is shorter. The acid bottles are removed and back-titrated with a standard base to determine, by difference, the amount of ammonia absorbed from the air stream. Figure (4) is an example of the sheet used for the basic calculations. Knowing the weight of ammonia absorbed and the quantity of gas sampled, the concentration of ammonia in the gas stream is calculated. The volume of gas in the stack is also calculated from its velocity and the diameter of the stack, and this volume is multiplied by the concentration figure to give the weight of ammonia per unit time escaping. This is then finally related to the formula and production rate for a percentage result. If the solids content of the gas stream is being measured, the paper thimbles are pre-weighed, then dried and weighed and after uses to determine the weight of solids collected from the gas stream.

The equipment is rugged, and we have used it to measure not only ammonia and solids losses but also, with standard base in the bottle, to measure flourine emissions from superphosphate plants. As previously mentioned we have access to a number of granulation plants, and a side benefit of our work in this area has been to develop individual patterns of losses.

We have measured somewhat over 60 ammoniation stacks and, in more recent times, a number of dryer stacks. Some losses from ammoniator stacks were as high as 20%. In the tests performed on dryer stacks, the losses were found not to exceed 6% in measurements made over a period of 8 years, including tests made at various times on the same stacks.

The significance of these measurements is that they help to explain the major reason for nitrogen shrinkage experienced in granulation plants. If the stack losses of NH_3 are subtracted from the overall nitrogen shrink, it would leave a residual shrink which is in line with the P_20_5 and K_20 shrink.

Despite this conclusion however, fertilizer plants do not have to sustain high losses of ammonia from plant stacks. Plants employing proper formulation procedures, operating conditions and ammoniator design, including good sparger arrangement, have solved the fundamental problems of ammonia loss. There is room for error in the above, however, where the exhaust gases from ammoniators and dryers are passed through efficient wet scrubbers. In addition, motivation to control losses may be influenced by economics and, to an increasing extent, because of the consequences of air pollution. To illustrate what can, in fact, be accomplished, we would like to draw on the experience of a specific fertilizer plant, that of Cotton Producers Association in Cordele, Georgia. This plant is located within a few blocks of the downtown section of Cordele, a prosperous town of 10,000 people, and is itself a prospering plant, producing approximately 150,000 tons per year of mixed goods. Figure (5) shows a view of the general vicinity punctuated by the ammoniator exhaust stack; and Figure (6) shows a general view of the granulation plant, including the dryer scrubbers and exhaust system to the right of the building. At the request of CPA, we started a program to determine their ammonia and solids losses, to study modifications for reducing the losses and for periodic control measurements. Their motivation was two-fold; to satisfy pollution control ordnances and to recover the cost of ammonia losses. Helped by the fact that our initial tests showed an ammonia loss of 6½% from the ammoniator stack, CPA completely replaced their scrubbing systems with wet scrubbers on the ammoniator, the dryer-cooler and acidulation exhausts. During the time the modifications were being made, their formulations and their ammoniator spargers were adjusted for efficient ammonia absorption. The plant was tested after the modifications were made and was re-tested again this September. The ammonia losses were less than 1/10 of 1% in both the ammoniator and the dryer-cooler exhaust stacks. Moreover, since the scrubbing equipment is operating well, losses should remain low.

The remaining loss of nitrogen, as well as the losses of P_2O_5 and K_2O are not as readily analyzed as the ammonia loss, which sticks out like a sore thumb. On the assumption that these plant food losses represent the weight loss of their respective carriers, a plant shipping 30,000 tons per year, could lose over 500 tons of fertilizer.

In the pursuit of our stack testing, we have made dust loss measurements on several ammoniator, dryer and cooler stacks. These show that these losses are significant and controllable with the use of efficient wet scrubbers or bag collectors, assuming the collected material is returned to process. Referring to our tests at CPA, total measured solids losses were reduced from 46 pounds per hour to 8.3 pounds per hour after modification of their collection system to include wet scrubbers on the ammoniator, dryer and cooler stacks.

What other sources of loss are there? One source of error is in the weights of raw materials received in which there are several possibilities of short weight. These include inaccurate scales during shipping from the vendor's facility, loss of material enroute, and returing heels for which credit is not received. We have observed rail cars of material arriving with leaky relief valves, ruptured discs and dome covers. These are obvious signs of shrinkage which, while they may not be prevalent, do contribute to shrink. The ideal procedure is of course to weigh all incoming raw materials and to tare empty cars. This being impractical if not impossible in most cases, it is recommended that cars selected at random from each supplier be weighed in and out.

Another source of shrink is in accurately weighing products shipped. In order to give customers the benefit of full measure the tendency is to overweigh rather than underweigh. It is also a reflection of some mistrust of weighing devices. In either event, it contributes to shrink, and whether or not it is planned, sufficient emphasis should be placed on product weights to allow a judgment based on factual information as to exactly what is being shipped.

Shrinkage should be placed in its proper perspective in the operation of any plant, whether it be chemical mix granulation, or other. As a guide in finding the perspective, the economic loss, to a granulation plant making the equivalent of 30,000 tons per year of 12-12-12, from raw materials valued at $1.00 per unit, can easily amount to $50,000. For those who are in this situation and, where applicable for other type of plants, the following suggestions are offered:

1) Examine the records of material receipts, inventories and shipments to develop a history of experienced shrinkage over a period of at least the previous year.
2) Obtain and use test equipment to help determine the specific sources of loss initially and for a continuing program to reduce and control the losses.
3) Measure all effluent streams for plant food losses. Where losses are found to exist, evaluate

them in terms of their cost to you in money and pollution; where it is practicable and advisable, make process alterations to strip the effluents of their plant food value.

4) Carefully analyze both incoming raw materials of N, P_2O_5 and K_2O, and operate on the basis of these analyses. Periodically check the weights of raw materials purchased and the tare weights of empty cars. Make sure you have received what you purchased.

5) Shipping scales deserve the utmost respect and attention. Not only should they be properly maintained, they should also be calibrated as frequently as necessary to maintain the accuracy for which they were designed. It isn't difficult to give away material through an inaccurate scale.

6) Errors in chemical analyses are prevalent. A dependable laboratory is an important asset, but even the most dependable are subject to error and their results should be compared routinely to those of similar laboratories.

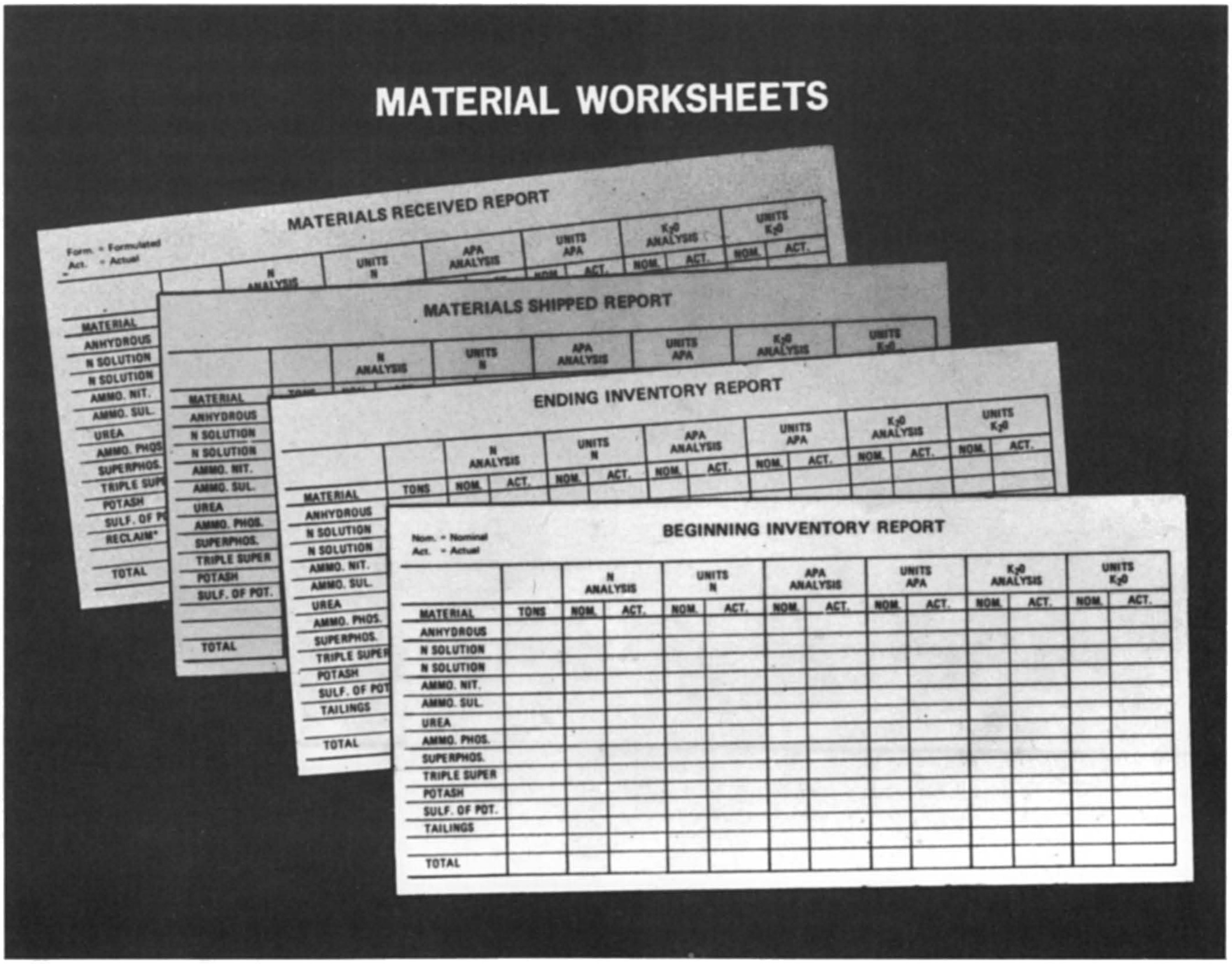
7) and last, where shrinkage becomes questionable, any part of the measurements or the operation should be suspect and systematically checked until the cause is found and corrected. The extent of shrinkage is a function of the time it is allowed to continue.

CHAIRMAN SPILLMAN: Thank you, Mr. Rodgers. You have given us a lot of food for thought. Many factors are responsible for excessive shinkage. I agree with you, in most cases, each plant requires thorough, searching studies to eliminate costly shrinkage. Basically you have given us very good advice.

MR. BILLY ADAMS: I would like to ask Mr. Rodgers concerning the weights of loss of materials. What would this be percentage wise? It was mentioned at 46 lbs. per hour before and 8 pounds per hour after the scrubber was installed

MR. RODGERS: I believe that the 46 lbs. per hour in the case of CPA was quite small percentage wise.

MR. ADAMS: Can you remember how many tons per hour approximately they run it.



MATERIAL WORKSHEETS

MATERIALS RECEIVED REPORT

Form. = Formulated
Act. = Actual

MATERIAL
ANHYDROUS
N SOLUTION
N SOLUTION
AMMO. NIT.
AMMO. SUL.
UREA
AMMO. PHOS.
SUPERPHOS.
TRIPLE SUPER
POTASH
SULF. OF POT.
RECLAIM*
TOTAL

MATERIALS SHIPPED REPORT

MATERIAL
ANHYDROUS
N SOLUTION
N SOLUTION
AMMO. NIT.
AMMO. SUL.
UREA
AMMO. PHOS.
SUPERPHOS.
TRIPLE SUPER
POTASH
SULF. OF POT.
TOTAL

ENDING INVENTORY REPORT

MATERIAL
ANHYDROUS
N SOLUTION
N SOLUTION
AMMO. NIT.
AMMO. SUL.
UREA
AMMO. PHOS.
SUPERPHOS.
TRIPLE SUPER
POTASH
SULF. OF POT.
TAILINGS
TOTAL

BEGINNING INVENTORY REPORT

Nom. = Nominal
Act. = Actual

		N ANALYSIS		UNITS N		APA ANALYSIS		UNITS APA		K_2O ANALYSIS		UNITS K_2O	
MATERIAL	TONS	NOM.	ACT.	NOM.	ACT.	NOM.	ACT.	NOM.	ACT.	NOM.	ACT.	NOM.	ACT.
ANHYDROUS													
N SOLUTION													
N SOLUTION													
AMMO. NIT.													
AMMO. SUL.													
UREA													
AMMO. PHOS.													
SUPERPHOS.													
TRIPLE SUPER													
POTASH													
SULF. OF POT.													
TAILINGS													
TOTAL													

Figure 1

CALCULATION OF PLANT FOOD LOSSES

	1. Nominal			2. Actual		
	N	P_2O_5	K_2O	N	P_2O_5	K_2O
A. Units Received						
B. Units in Beginning Inventory						
E. Total						
C. Units Shipped						
D. Units in Ending Inventory						
F. Total						
G. Units Loss (E – F)						
H. Loss, Units per Unit Material Shipped (G ÷ C)						
J. % Overall Loss (100H)						

Figure 2

MR. RODGERS: I do not have that information.

CHAIRMAN SPILLMAN: Can you give us an idea what the shrinkage is in bulk blend plants verus the granular plant, percentage wise?

MR. RODGERS: It is quite small except for errors in weight measurements and the possibilities of short weight car loads coming in and overweight going out.

MR. CHARLES T. HARDING – Wellman Lord: When you formulate, say a 10-20-10, do you allow overages in your formulation in ammonia?

MR. RODGERS: We do not have strict formulation procedures, but would allow the flexibility of overformulation to compensate for losses.

MR. HARDING: When you get your analyses would your shrinkage be the difference between a 10-20-10 or actual put in viz: 10-20-10.20 10-20-10.30, etc. I mean, would you give or take? How would you keep your unit balance? Would it be based on what the formula actually calls for or the actual 10-20-10? Do you get my point?

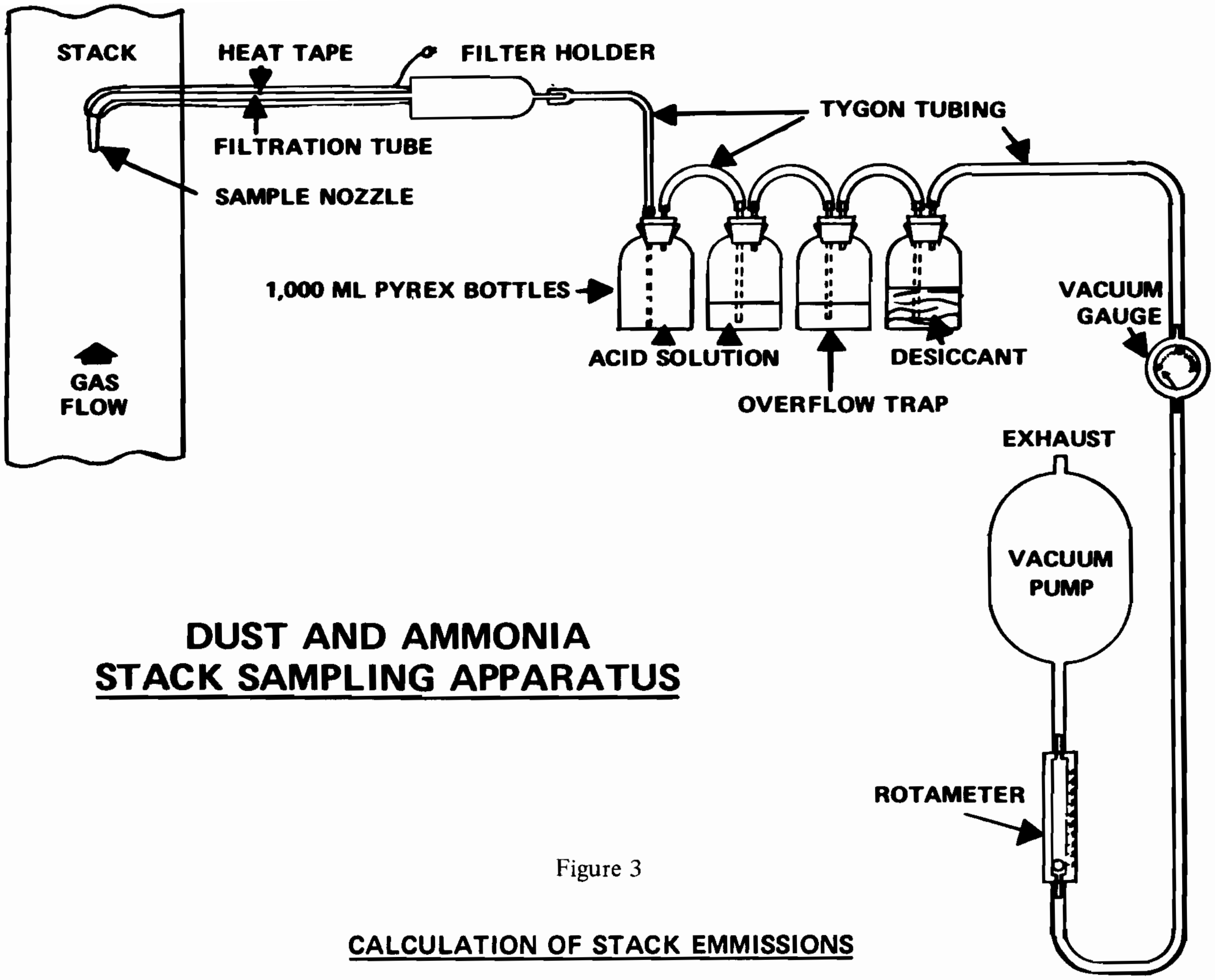
MR. RODGERS: Yes, computing actual shrinkage, you would make it on the basis of put in, 10-20-10.20 or 10-20-10.3. This is a distinction between nominal analysis and an actual analysis. Calculate your shrinkage on the basis of what ever you formulate to. However, on a nominal basis, many plants operate, they take their materials in on a nominal basis also, a 0-0-60 for example. Of course their shipments out of the plant are by grade number. This is the reason we made this distinction between a nominal and an actual material balance.

MR. HARDING: We found many times that a major problem loss was in the weighing of the bag. The biggest problem we had was the over weight because there was no criticism if the bag weighed too much; only when it was short.

MR. RODGERS: That is right. And I will say that even if some of these are planned that you should know exactly what you are doing and whether your plan is being carried out.

CHAIRMAN SPILLMAN: Thank you Mr. Rodgers. We appreciate your coming here and giving us your advice and experience.

Our next topic "Diaphragm Valves will be discussed be a very good friend of the Round Table. He has been with us 2 times and has given us some valuable technical information on various types of equipment. His paper this morning on valves should be of interest to all of us. It is a real pleasure for me to again present to you Mr. Donald L. Warren of The Grinnell Co. Don, please.



DUST AND AMMONIA STACK SAMPLING APPARATUS

Figure 3

CALCULATION OF STACK EMMISSIONS

Company______________________________ **System**________________ **Number**__________

1. $$\frac{.7854 \times (\overset{D^2}{\qquad\qquad})}{144} = \rule{3cm}{0.4pt} \text{ area of stack in sq. ft.}$$

2. $$\overset{\text{FPM}}{\rule{3cm}{0.4pt}} \times \frac{530^\circ\text{ F}}{460+\underset{\text{measured temp.}}{\rule{1cm}{0.4pt}}} \times \overset{\text{area}}{\rule{3cm}{0.4pt}} = \overset{\text{CFM}}{\rule{3cm}{0.4pt}} \times 60 = \overset{\text{CFH}}{\rule{3cm}{0.4pt}}$$

3. $$\overset{\text{CFM}}{\rule{3cm}{0.4pt}} \times \overset{\text{min.}}{\rule{2.5cm}{0.4pt}} \times \overset{\text{Vacuum Correction}}{\frac{29.9 - \rule{1cm}{0.4pt}}{29.9}} = \overset{\text{cu. ft. sampled}}{\rule{2.5cm}{0.4pt}}$$

4. $$\frac{(\overset{\text{ml acid}}{\qquad} \times \overset{\text{N.}}{\qquad}) - (\overset{\text{ml base}}{\qquad} \times \overset{\text{N.}}{\qquad})}{26{,}656 \times \underset{\text{cu. ft. sampled}}{\rule{3cm}{0.4pt}}} = \rule{3cm}{0.4pt} = \overset{\#NH_3/\text{ cu. ft. air}}{\rule{3cm}{0.4pt}}$$

5. $$\overset{\text{CFH}}{\rule{3cm}{0.4pt}} \times \overset{\#NH_3/\text{ cu. ft. air}}{\rule{3.5cm}{0.4pt}} = \overset{\#NH_3/\text{ hr.}}{\rule{3.5cm}{0.4pt}}$$

6. $$\frac{\#NH_3/\text{ hr. lost} \times 100}{\#NH_3/\text{ hr. fed}} = \rule{3.5cm}{0.4pt} \text{ \% } NH_3 \text{ loss}$$

Figure 4

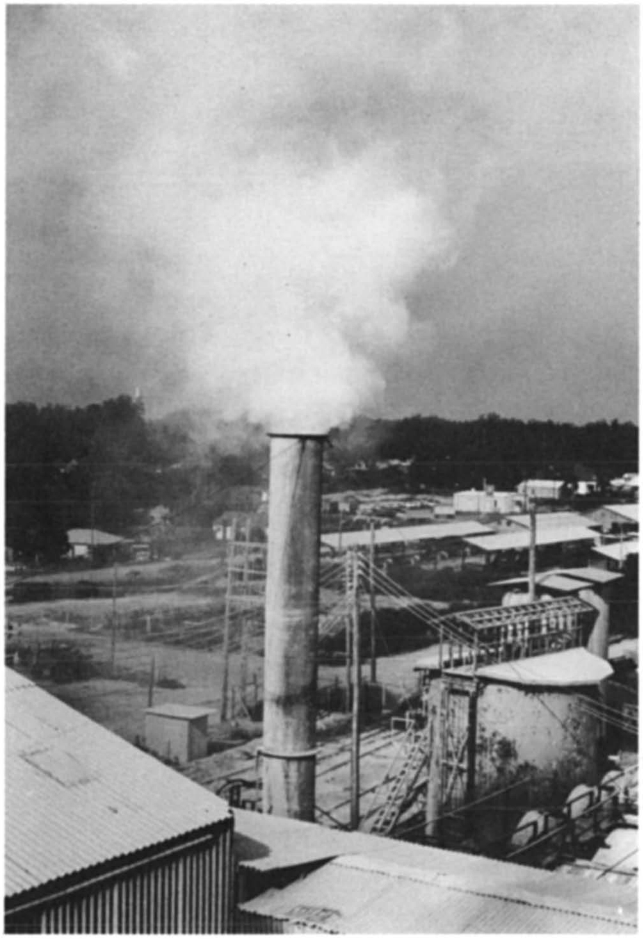


Figure 5

General View Ammoniator Exhaust Stack

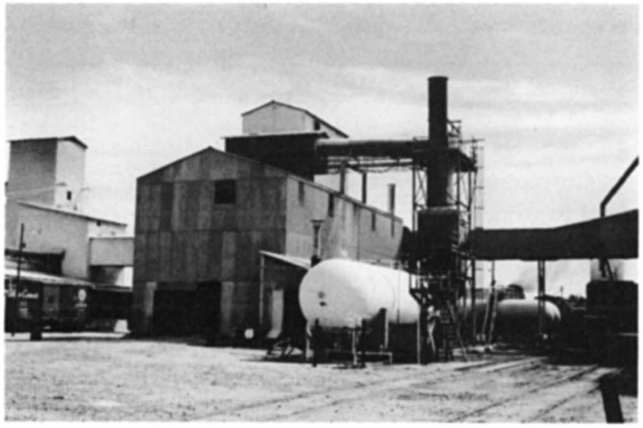


Figure 6

General View Granulating Plant

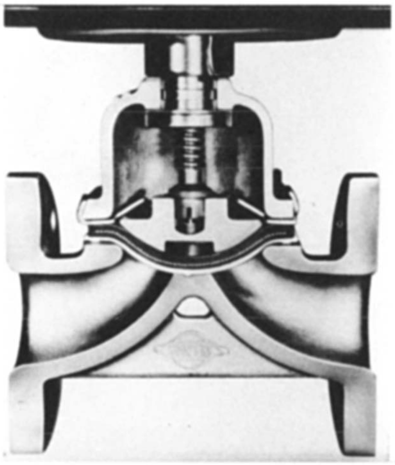
Grinnell Diaphragm Valves Selection, Sizing and Maintenance

Donald L. Warren

Gentlemen, I'm very happy to talk to you at your 1969 Round Table. This is my third appearance over the years. This time I will talk about a class of equipment which is sometimes taken for granted, and occasionally applied with not enough thought to sizing, materials of construction, types of operation and maintenance – those all important components – the valves in your plant.

This paper is limited to diaphragm valves although we give due credit to other good valves, such as ball, butterfly, pinch, gates, and globes.

Slide 1.



First, let's take a quick look at two common types of diaphragm valves, the Weir and the Straightway.

Slide 1. The secret of this well known style, the weir type, is the lack of sliding or rotating parts in the liquid handled, be it wet process phosphoric acid, slurry fertilizer or sulphuric acid. The diaphragm is the seal and heart of the valve. There will be no stem leakage.

The body can be cast iron, any one of several alloys or choice of plastics, solid, lined or coated; also glass lined.

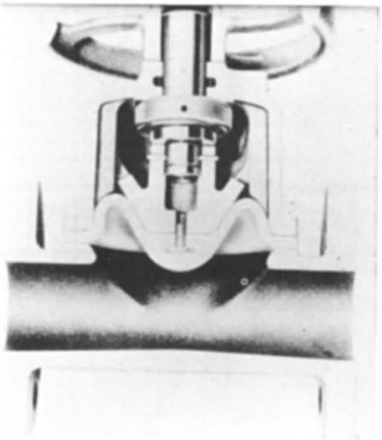
It has good flow characteristics, a cross sectional area 75% to 80% that of schedule forty pipe, is self draining when mounted with the stem 15 degrees from the vertical and is simple to maintain.

More than 20 types of diaphragms can be furnished ranging from soft natural rubber thru thick TFE with an elastomer backing cushion. Available in 1/4" to 20" sizes.

Slide 2. Here's another work horse, the Grinnell Straightway, shown closed. It can be C.I., Stainless, Rubber, or Glass lined and has several types of elastomer diaphragms. It can be rodded. Slide 2 not available.

Slide 3. Here is the open position. You can visualize the huge capacity and the ability to handle slurries, abrasives, viscous, materials and, yes, even finely pulverized solids; like phosphate rock dust, attapulgite clay, potash, etc. Available in 1/2" to 12" sizes.

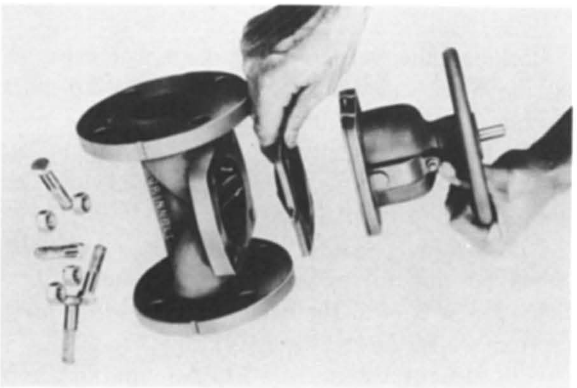
Slide 3.



Available in 1/2" to 12" sizes.

It's bore is 120% that of schedule 40 pipe. Of course, as well as hand sheel operated, they can be furnished with a choice from our broad line of cylinder or diaphragm type operators – all the way from a little 7 square inch beauty on up to a 400 square inch job, which easily holds 100 PSI air and gives thrusts to 40,000 lbs. The weir type can be furnished with manual lever operators. More later on operators.

Slide 4.

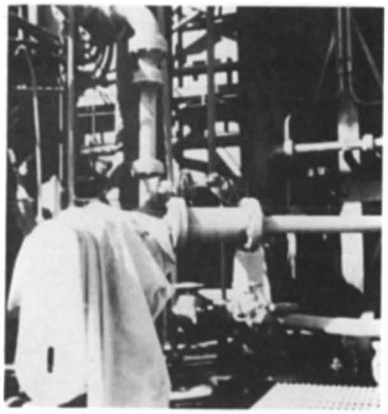


Slide 4. Maintenance shouldn't require much of your time and will not be given much here. All bonnet parts are isolated. Here is all that usually is required – a simple, quick inline change of diaphragms, and this infrequently. A lubrication program should be set up and I recommend a silicone grease in fertilizer plants. It seems to hang on in the face of tough service in corrosive atmosphere. In request, at extra charge when ordering the valve a sealed bonnet can be furnished. Don't allow cheater bars to be used when closing these valves. They are not necessary and will shorten diaphragm life.

Slides 5 and 6 show some typical installations in fertilizer plants. Let me assure you that these installations exist now – these pictures were all taken in the last month. Slides 5 and 6 not available.

Slide 7. This No. 25 double acting operator is doing yeoman service in a strong atmosphere. We build the diaphragm type operator in 12, 25, 50, 100, and 200 nominal square inch sizes. They are available in double acting, spring-to-close or spring-to-open versions. They are close coupled with no moving parts exposed to atmosphere. Accessories include position indicators, open or closing stops, pnumatic positioners, solenoid air pilot valves.

Maintenance is simple and infrequent – the diaphragm is noted for its long service life. Field reports of several million cycles are common. Slide not available.



Slide 8. This is our Dualrange valve on a slurry line. The D.R. is our "valve within a valve." This hand operated model is used for wide range control of slurries and abrasive material. Of greater importance, however, is its use as a control valve when coupled with a Grinnell valve actuator.

The blue brochure you have gives technical information on its operation and the flow curve. Note particularly the shape of the opening when the valve is from 5 to 40% open. The eliptical orifice maintains flow efficiently while giving a nearly equal percentage output curve. A good control valve with all the advantages of a diaphragm valve. The range is wider than any meter we know if, so it's an ideal control valve for closed loop systems where magnetic, turbine or differential producer type flow meters are used.

Slide 9. This weir type valve is doing a good job on the suction of a pump. This brings to mind the fact that our weir type valves are recommended for vacuum service – they're good down to 1 Microns of vacumn *as they come from the factory.* No special trim or seals required. They are great on the section side of your vacumn phosphonic acid filters. The Straightway can be used on vacuum also

but the bonnet must be evacuated to a pressure near that in the valve.

Various shots of the valves in their working clothes fighting corrosion and wear. Slides 10, 11, 12, 13, 14 15 and 16 not available.

Slide 17. This shows a G.D.V. with another brand of operator. If required for any reason, we furnish bonnets to fit all major (and most minor) makes of valve actuator. Slide not available.

More G.D.V. faithfully handling their appointed goodies. There's a closed loop metering system with a G.D.V. as the final control element although its nearly out of sight. Slides 18, 19 and 20 not available.

Slide 21.

Slides 21,22, 23 and 24. These 1/4 turn and hand wheel valves are handling phos acid, nitrogen solution, clay slurry going into a hot mix system. The valves in this plant were installed 3 years ago and have had absolutely no maintenance. We received an order for 20 more (and this is not a large plant) earlier this week. Slides 22, 23 and 24 not available.

This same plant is protected by out sprinklers, fire control and alarms. Just thought I'd throw that in. Now about materials of construction. Slide 25 not available.

Grinnell diaphragm valves

SERVICE	BODY			DIAPHRAGM			REMARKS
	MATERIAL	MAXIMUM		CODE	MAXIMUM		
		CONC. BY WT.	TEMP. °F.		CONC. BY WT.	TEMP. °F.	
AMMONIA Anhydrous NH_3	C. ST. GLASS KYNAR PENTON PVDC ALUMINUM C. I.	ALL ALL ALL ALL ALL ALL ALL	ALL ALL 275 220 190 ALL ALL	R-2 Z-2 B M	ALL ALL ALL ALL	350 280 150 150	Aluminum for dry service only. Watch Vapor Pressures
AMMONIUM HYDROXIDE NH_4OH Aqua Ammonia	C. I. ST. ST. GLASS CTFE KYNAR PENTON	ALL ALL ALL ALL ALL ALL	ALL ALL ALL 325 275 220	M B T	ALL ALL ALL	275 225 200	
AMMONIUM NITRATE NH_4NO_3	C. ST. GLASS KYNAR PENTON ALUMINUM PVDC #10 PVC	ALL ALL ALL ALL ALL ALL ALL ALL	ALL ALL 275 250 200 190 180 140	M B C T P	ALL ALL ALL ALL ALL	275 225 200 200 180	
CARBON DIOXIDE CO_2	ANY METAL KYNAR PENTON PVDC POLYPROP PVC	ALL ALL ALL ALL ALL ALL	ALL 275 250 190 150 140	M B C T	ALL ALL ALL ALL	275 250 225 200	
FERTILIZER SOLUTIONS complete For Direct application	C. I. ST. ST. PENTON ALUMINUM PVC	ALL ALL ALL ALL ALL	ALL ALL 250 200 140	M B C T	ALL ALL ALL ALL	250 225 200 200	Watch Vapor Pressures
NITRIC ACID HNO_3	GLASS ZIRCONIUM CTFE CTFE ALLOY-20 ST. ST. PVC PENTON ALUMINUM	ALL ALL FUMES 70 To 70 To 50 To 50 30 82-100	350 B.P. 325 325 190 190 140 150 100	R-2 Z-2 B C M	ALL ALL 30 30 30	350 280 100 100 100	
NITROGEN SOLUTIONS	C. I. D. I. ST. ST. PENTON ALUMINUM #10 PVC	ALL ALL ALL ALL ALL ALL ALL	ALL ALL ALL 250 200 200 140	M B C T	ALL ALL ALL ALL	250 225 225 200	Watch Vapor Pressures

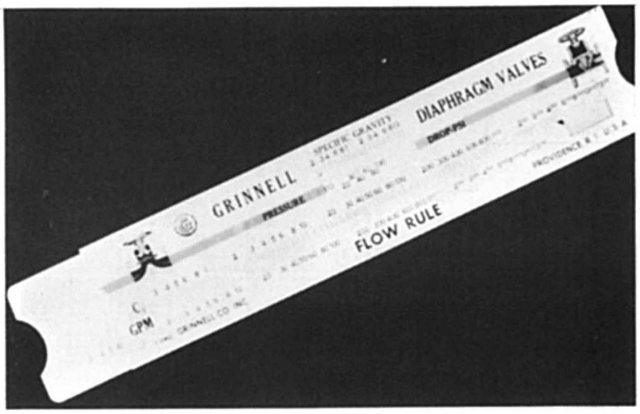
SERVICE	BODY			DIAPHRAGM			REMARKS
	MATERIAL	MAXIMUM		CODE	MAXIMUM		
		CONC. BY WT.	TEMP. °F.		CONC. BY WT.	TEMP. °F.	
PHOSPHORIC ACID H_3PO_4	GLASS* PENTON ALLOY-20 KYNAR KYNAR #10 PVDC ST. ST. #7 PVC POLYPROP	ALL ALL 85 30 85 ALL 85 85 ALL 50 10-85	ALL 250 Boiling 275 215 200 190 160 150 140 140	R-2 Z-2 B C M	ALL ALL ALL ALL ALL	350 280 200 200 200	"R-2" or "Z-2" if no discoloration permissible. *Check for fluorides. Always ask if customer is using "furnace" or "wet" process acid. The latter contains many impurities such as hydrofluoric acid and gypsum. The Gypsum tends to plate out on pipes, valves, etc. Straightway has worked well
SULFURIC ACID H_2SO_4	GLASS ALLOY-20 KYNAR PENTON #10 #7 C. I. PVC POLYPROP	ALL ALL ALL 50 50 50 85 up 75 93	ALL 175 200 200 170 150 140 140 100	R-2 Z-2 C B M	ALL ALL 93 60 60	325 280 150 150 150	Boumé 66´ Boumé is 93%
Super Phosphoric Acid Same materials as Phosphoric acid.							
UREA $CO(NH_2)_2$	ANY METAL PENTON PVDC POLYPROP PVC	ALL ALL ALL ALL ALL	ALL 250 190 150 140	M C T P	ALL ALL ALL ALL	250 225 200 180	

Here's another version of the diaphragm valve which we call the direct loaded bonnet. It has a molded diaphragm which can be installed either molded closed or molded open. The bonnet is actually an air tight dome and can be exposed to pressures up to 110 P.S.I. and will close against line pressures up to 100 P.S.I.

With low line pressure the diaphragm is assembled in the open position for minimum flow resistance. In this configuration it is ideal for batch systems where material comes from storage tanks by gravity.

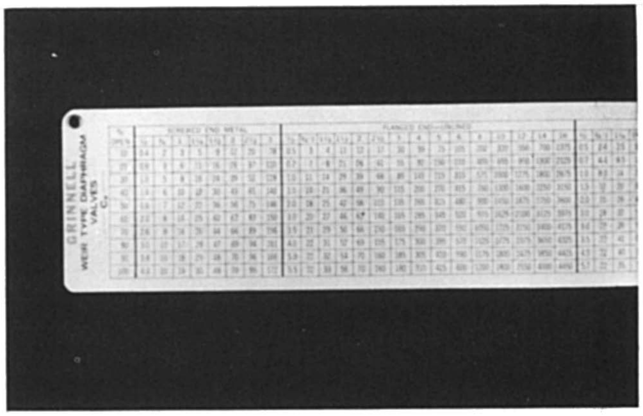
With higher pressures, say 30 P.S.I. and up it can be assembled in the closed position in which case it is "self closing." By that we mean with, say 40 P.S.I. line pressure in the valve, it can be closed with 40 P.S.I. in the dome. The dome pressure could be from the liquid in the line in which case it's an ideal valve on irrigation lines where the water or fertilizer and water mixture could be piloted into the top of the valve to effect shut-off. Air or other gases are also used for closing.

With a regulated air pressure in the dome it can be used as a safety or by-pass valve and is useful for inserting in a line near a control valve when the control valve is taking too much pressure drop in a system. It is available with all the regular Weir type bodies. The diaphragm is always elastomer. Where applicable it is a very inexpensive air operated valve – as an example a 2" screwed C.I. bodied valve would cost $30.00 including air actuation. It is not recommended for throttling and is difficult to make fail-safe. TFE diaphragm not available.



Slide 26. You have all received a valve sizing slide rule. This will make you a valve expert in 1 easy lesson. You can use it for sizing any manufacturers valve as most of them use the CV system of rating valve capacities. A CV represents a valve capacity unit of 1 G.P.M. of water at a 1 P.S.I. pressure drop (Delta P) across the valve. Lets do an example, say, on Sulphoric Acid.

The specific gravity is 1.5 Set 1.5 over 60 which is the pressure drop across the valve. Say we want a flow rate of 400 G.P.M. Looking above the 400 G.P.M. on the bottom scale we find a CV valve of 64.



Slide 27. Now look at the CV table which is with the slide rule. With the little metal grommet in the upper left hand corner look at the tables under "flanged end unlined". This could be a cast iron, stainless or other alloy body. The bottom line gives the maximum CV rating of the valves. Under the 2" valve this is 70. Our requirement is 64. Looking higher on the table we will see our rate will be produced with the valve a little over 60% open.

Slide 28. Here we show a ΔP of 30 P.S.I. and a Sp. gr. of 1.7 which could be Phosphoric acid. Let's not take the time to do an example but lets say we need a CV of 1200. Slide 28 not available.

Slide 29. Turn your CV table so the grommet is in the lower left corner which shows us the Straightway tables. The No. 5 and No. 10 are rubber lined and would be one of our recommendations for phos. acid. Note that this flow is easily handled by a 6" S.W. between 8-90% open. Slide not available.

One of the most common errors particularly in control valves is oversizing. This means that many times a valve is operating, even at medium or maximum flow in a nearly closed position.

This leads to cavitation, excess noise, too high velocities, rapid wear and control problems.

Many times these control problems are blamed on the instrumentation when a properly sized valve would have eliminated the trouble.

We hope you will use these sizing tools. They will contribute to longer valve life and a smoother running plant.

In conclusion a word on pressure and temperature limitations.

The Weir type can handle up to 200 P.SI.G. depending on size. Temperatures to 350 degrees. The two extremes cannot be achieved together however. The higher the temperature the lower the pressure and vice versa.

The Straightway can be used up to 100 P.S.I. or 225 degrees F. Again – never simultaniously. Charts are available showing the Pressure/Temperature range for the various materials of construction.

Gentlemen, you've been a great audience. Thanks for listening. If time permits I'll try to answer any questions you may have.

CHAIRMAN SPILLMAN: Thank you, Don, for that excellent explanation of valves. You gave us real good advice and pointers on types to use for specific conditions, liquids used, minium requirements and maintenance. I note nearly everyone outthere in the audience are checking the slide rules you passed out. I am sure we will all be "Slide Rule Experts on Valves" by the time this meeting is over! Thank you again, Don.

I shall turn this meeting over to Billy Adams. Billy, please.

MR. ADAMS: We do not want to work our new chairman too hard here, so we will switch around a little bit.

Our next two speakers will talk to you on a new granulation process. Several of you folks have already asked me "What is the new granulation process". I will turn this over to Mr. Jim Madigan, President, Fertilizer Engineering and Equipment Company and Mr. Cam Bolduc, Brockville Chemical Company.

The "C. B. Granulation Process"

James E. Madigan and Cam Bolduc

The technology of granulating N–P–K fertilizer has not witnessed too many changes since the advent of the well known TVA system some 10 or 15 years ago. Many refinements of the art have, however, been introduced through the years, but, basically, the principles involved in the TVA ammoniator-granulator, are still those utilized now to pelletize a very large portion of the N–P–K granular fertilizers in North America and abroad.

In the present paper, we wish to illustrate a different approach to granulation which, as you will see, reaches a high degree of efficiency and gives a product of unusual uniformity of chemical composition. As a matter of fact, the homogeneity of the products manufactured by this technique, is such that it solves one of the problems that has plagued the fertilizer industry for years: segregation.

But, before describing this new process, let us consider for a while what is happening in commonly used granulating drums: ordinarily, mixtures of particulate ingredients of various nature, size and shape are fed at one end of the drum, mixed and/or reacted with liquid constituents in the reaction zone and expected, in theory at least, to exit the other end as pellets or granules of homegeneous chemical composition and uniform size. In practice, we all know that products resulting from the granulator, are generally characterized by a large range of size and analysis. As a direct consequence, it is necessary, in most cases, to classify the product and reprocess a large portion of this material. In conventional rotary granulating drums, one can say that each constituent tends to agglomerate at a specific rate, in a manner only slightly influenced by the presence of the other constituents and at a rate relatively independent of the basic ratio of N–P–K in the fertilizer formula.

The lack of uniformity of the products ex. the granulator, is reflected throughout the entire granulation system, as well as in the finished product. This, we are all well aware, is the direct cause of segregation, one of the most difficult problem the fertilizers industry has always been faced with.

The non-homegeneous characteristics of the fertilizer products can easily be demonstrated by chemical analysis of various screen fractions. Tables 1 and 2 show this kind of results obtained on samples of 0-20-20 and 5-20-20 from three (3) different sources of commercially available farm fertilizers.

TABLE I

COMMERCIALLY AVAILABLE PRODUCTS

0-20-20

PRODUCER NO. 1

MESH	%	P_2O_5	K_2O
6	0.2	-	-
10	50.2	22.2	15.7
20	43.5	19.9	21.6
-20	6.1	17.3	21.8
	100.0		
Av.		20.8	18.6
Range of Variation:		4.9	6.1

F.W.T.: NONE

PRODUCER NO. 2

MESH	%	P_2O_5	K_2O
6	0.8	23.5	12.6
10	52.6	22.6	17.2
20	42.9	15.5	23.2
-20	3.7	18.9	22.5
	100.0		
Av.		19.4	19.9
Range of Variaton:		8.0	10.6

F.W.T.: NONE

PRODUCER NO. 3

MESH	%	P_2O_5	K_2O
6	5.0	8.2	41.3
10	64.7	25.6	15.7
20	26.2	17.4	29.7
-20	4.1	11.3	9.7
	100.0		
Av.		21.9	20.4
Range of Variation:		17.4	31.6

F.W.T.: NONE

TABLE II

COMMERCIALLY AVAILABLE PRODUCTS

5-20-20

PRODUCER NO. 1

MESH	%	N	P_2O_5	K_2O
6	2.6	4.1	25.3	12.4
10	48.1	4.4	21.1	17.2
20	38.4	4.5	18.7	21.5
-20	10.9	4.6	19.7	21.7
	100.0			
Av.		4.4	20.1	19.2
Range of Variation:		0.5	6.6	9.3

F.W.T.: NONE

PRODUCER NO. 2

MESH	%	N	P_2O_5	K_2O
6	1.0	5.4	21.4	11.7
10	49.7	5.2	26.0	19.6
20	46.6	5.7	19.1	21.6
-20	2.7	3.9	19.5	21.5
	100.0			
Av.		5.4	22.6	20.6
Range of Variation:		1.8	6.9	9.9

F.W.T.: NONE

PRODUCER NO. 3

MESH	%	N	P_2O_5	K_2O
6	0.4	-	-	-
10	32.1	6.4	23.2	16.0
20	49.0	4.1	16.6	27.6
-20	18.5	3.2	21.3	27.5
	100.0			
Av.		4.7	19.5	23.7
Range of Variation:		3.2	6.6	11.6

F.W.T.: NONE

Examination of our own production also indicated similar conditions and lack of uniformity. Table 3 shows fraction analysis on typical production samples.

TABLE 111

5 - 20 - 20

			ANALYSIS	
MESH	%	N	P_2O_5	K_2O
8	4.0	5.2	26.3	13.0
10	28.1	5.6	25.6	11.9
20	40.4	5.4	17.4	23.2
20	27.5	5.0	15.8	25.8
	100.0			
Av.		5.3	20.1	20.0

F.W.T.: NONE

6 - 12 - 12

MESH	%	N	P_2O_5	K_2O
6	1.5	4.9	14.0	6.2
10	19.2	5.9	14.8	7.0
20	34.9	6.0	12.1	13.4
- 20	44.4	6.2	10.8	14.5
	100.0			
Av.		6.0	12.0	12.5

F.W.T.: NONE

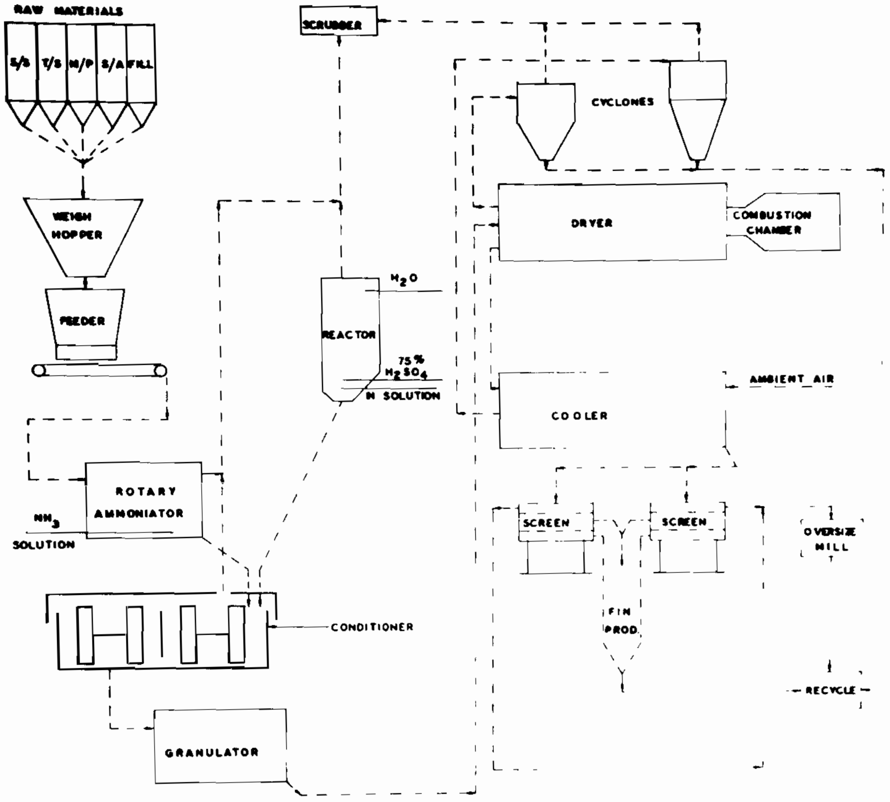
10 - 10 - 10

MESH	%	N	P_2O_5	K_2O
6	13.5	10.0	13.4	7.0
10	52.4	9.6	12.9	8.1
20	22.4	10.9	9.2	12.0
- 20	11.7	12.2	8.8	11.0
	100.0			
Av.		10.5	11.7	9.3

F.W.T.: NONE

These results clearly demonstrate the lack of uniformity of many of the fertilizers products on the market, and illustrate what happens when particles of these compositions are allowed to segregate in piles.

As a solution to this problem, we have developed a granulation process now designated as the "C.B. Granulation Process", covered by Canadian and U.S. Patents which introduces the use of a muller as an intermediate step between ammoniation and granulation. This process is now in full scale operation at our Cornwall plant. Mr. Madigan will comment on the next chart which shows a flow diagram of the C. B. Granulation Flow Diagram at the Cornwall Plant.



C. B. GRANULATION FLOW DIAGRAM
CORNWALL PLANT

Let's have a look now at some of the results obtained with this new system. On the next Table (IV) are compared analytical results on pilot run of 0-20-20 and on a sample of the same grade made on a plant scale by our former granulation system.

TABLE IV

PILOT VS PLANT RESULTS (Old System)

0-20-20

PILOT

MESH	%	P_2O_5	K_2O
6	1.9	20.8	19.7
10	68.6	20.2	20.1
20	29.3	19.7	22.3
-20	0.2	-	-
	100.0		
	Av.	20.0	20.7

FRACTION WITHIN TOLERANCES: 97.9

PLANT

6	8.1	23.0	17.7
10	32.8	23.6	16.5
20	38.6	20.4	21.8
-20	20.5	20.5	23.7
	100.0		
	Av.	21.6	20.1

FRACTION WITHIN TOLERANCE: 0.0

C.B. GRANULATION PROCESS

TABLE V

PILOT VS PLANT RESULTS (Old System)

5-20-20

PILOT

MESH	%	N	P_2O_5	K_2O
6	11.1	4.6	19.9	19.8
10	69.6	4.7	20.0	19.8
20	19.0	4.9	18.7	21.6
-20	0.3	-	-	23.9
	100.0			
	Av.	4.7	20.4	20.1

F.W.T.: 80.7

PLANT

6	4.0	5.2	26.3	13.0
10	28.1	5.6	25.6	11.9
20	40.4	5.4	17.4	23.2
-20	27.5	5.0	15.8	25.8
	100.0			
	Av.	5.3	19.6	20.3

F.W.T.: NONE

10-10-10

PILOT

6	1.2	9.4	11.4	9.9
10	61.4	9.5	11.2	9.8
20	36.4	10.2	10.4	11.2
-20	1.0	10.6	9.0	15.5
	100.0			
	Av.	9.8	10.8	10.3

F.W.T.: 99.0

PLANT

6	15.8	9.6	13.3	7.7
10	50.5	9.4	12.9	8.1
20	23.0	10.9	9.2	11.8
-20	10.7	11.9	8.8	10.8
	100.0			
	Av.	10.0	11.7	9.2

F.W.T.: NONE

A similar comparison is made on grade 5-20-20 and 10-10-10 as noted on Table 5.

With these encouraging results on the pilot operation, it was decided to modify one of our granulation plants, located at Cornwall, Ontario. The equipment engineered and supplied by Feeco was installed in August 1968, and after an easy start up, has been kept fully operative since that date.

On the next two slides, analytical results on 6-12-12, 5-20-20, 10-20-20 and 16-8-8 regular production samples are compared with pilot plant results. It will be seen that duplication was excellent.

C.B. GRANULATION PROCESS

PLANT RESULTS

6-12-12

	(C.B.) PLANT				PILOT			
MESH	%	N	P_2O_5	K_2O	%	N	P_2O_5	K_2O
6	0.1	-	-	-	3.7	5.9	13.0	11.3
10	52.5	6.4	12.5	11.5	77.0	6.1	13.4	11.9
20	40.3	6.4	11.7	12.3	17.8	6.0	12.7	12.9
-20	7.1	6.3	11.3	13.6	1.5	5.0	11.3	16.2
	100.0				100.0			
	Av.	6.4	12.1	12.0	Av.	6.0	13.2	12.1
F.W.T.:	92.8				94.8			

5-20-20

6	-	-	-	-	11.1	4.6	19.9	19.8
10	50.1	4.8	21.8	20.6	69.6	4.7	20.0	19.8
20	41.6	5.0	21.4	21.1	19.0	4.9	18.7	21.6
-20	8.3	4.9	19.8	23.6	0.3	-	-	-
	100.0				100.0			
	Av.	4.9	21.4	20.9	Av.	4.7	20.5	20.2
F.W.T.:	91.7				80.7			

Next chart shows similar results: our grades 10-20-20 and 16-8-8.

The high degree of uniformity demonstrated so far on production samples, is maintained in piles and bags, the following slide compares analytical results on samples originating from process, bins and bags. In all cases, the high percentage of particles meeting guarantee, is maintained.

C.B. GRANULATION PROCESS

PLANT RESULTS

10-20-20

MESH	%	N	P_2O_5	K_2O
6	0.3	-	-	-
10	57.7	10.1	21.0	19.0
20	39.4	9.6	19.7	20.7
-20	2.6	8.3	16.0	23.7
	100.0			
	Av.	9.8	20.3	19.7

F.W.T.: 97.1

16-8-8

6	0.4	-	-	-
10	51.6	16.0	9.2	8.1
20	36.4	15.6	8.9	8.9
-20	11.6	15.9	5.5	8.4
	100.0			
	Av.	15.8	8.6	8.4

F.W.T.: 88.0

Now, what are the practical implications of this process? The high efficiency of agglomeration and the high degree of uniformity of the product, presents two main advantages:

1) Low recycle.
2) Product virtually independent or segregation.

The average recycle on a production of 30,000 tons is 15% of total feed, with minimum as low as 5% and maximum of 20%. More than 30 different grades were processed, including 0 grades. No steam is used, and the additional water required for granulation is also cut down.

With this low recycle, the production capacity of a given installation, can be increased markedly and important savings are possible on fuel, power and labour.

The uniformity of chemical analysis between particles of different size has made possible the elimination of formula overages generally included to minimize the segregation effects. Sampling becomes extremely easy and guarantee performance with government control agencies is much improved.

In summary, the advantages of the C. B. Granulation Process are the following:

a) uniform rate of agglomeration for all constituents.
b) uniformity of chemical composition between particles of different size.
c) elimination of costly overages in formulation.
d) elimination of pile trimming to avoid coning effect.
e) ease of obtaining representative sample.
f) improved performance with government control.
g) low recycle – higher plant capacity.
h) lower fuel consumption.
i) lower power consumption.
j) lower labour bills.
h) reduced dust loss in dryer and cooler.

C.B. GRANULATION PROCESS

PLANT RESULTS

GRADE	T.P.H.	RECYCLE %	N	P_2O_5	K_2O	F.W.T.	ORIGIN
0-20-20	9.0	14.7	-	20.2	20.1	95.1	A.S.
			-	20.6	20.5	88.2	BIN
			-	19.7	20.1	93.4	BAGS
5-20-10	9.6	11.8	5.2	20.5	10.4	93.6	A.S.
			4.9	20.0	9.9	96.5	BIN
			5.4	19.7	10.5	97.3	BAGS
5-20-20	9.2	18.0	5.1	20.1	19.9	97.8	A.S.
			5.2	19.7	20.4	88.7	BIN
			5.1	19.8	20.3	92.5	BAGS
8-16-16	9.0	12.0	8.5	16.2	16.5	94.2	A.S.
			8.8	16.0	17.0	86.2	BIN
			8.2	16.4	16.0	94.5	BAG
10-10-10	9.9	18.8	10.0	10.1	10.0	96.6	A.S.
			10.0	10.1	10.3	95.7	BIN
			9.9	10.8	10.9	94.6	BAGS
10-20-20	9.2	8.0	9.9	20.1	20.6	97.7	A.S.
			9.7	19.8	22.0	97.7	BIN
			9.6	20.0	20.0	97.0	BAGS

MR. ADAMS: Thank you Jim and Cam for that nice presentation on the "C.B." Process.

MR. PERKINS: I would like to ask either of the gentlemen, do you have any experience on higher analyses grades than shown in your presentation?

MR. BOLDUC: The maximum concentration is about 10-20-20 formulated with diammonium phosphate. We do not use phosphoric acid.

MR. PERKINS: This was the next question and you answered it. Thank you.

MR. BOLDUC: Using phosphoric acid is not economical yet in our area.

MR. HWA C. Ai: Could you explain how this conditioner works or the mechanical construction of the conditioner, what it looks like?

MR. MADIGAN: Yes, the conditioner is, in essence, a mixer-muller. It consists of big, very heavy wheels rotating around a central point and in essence compressing and breaking down the particles before it goes to the granulation operation. This is adjustable and gives the excellent results you have seen.

CHAIRMAN SPILLMAN: Jim, can you give us the minimum and maximum capacities, production capacities, per hour on this equipment? I stepped out for a few minutes and maybe I missed it.

MR. MADIGAN: Fine Al. Actually the muller that we have is capable of 30 tons per hour continuous, with one unit and the unit is approximately 7'x7'x7'. If you have an operation that is running 20 tons an hour, there is no problem in installing a unit which, with the decrease in the recycle rate, should give you increased production.

It is also very easy to go to a 60 ton an hour unit because you would just put two muller conditioners in parallel and of course you would need an ammoniator–granulator with capacity to handle 60 tons per hour. Does that answer your questions, Al.

CHAIRMAN SPILLMAN: Very well. Thank you Jim.

MR. PERKINS: Did you mention the power consumption on the conditioner? What power requirements does it have?

MR. BOLDUC: The unit we have is a 10 ton per hour and there is a 30 horsepower motor used.

MR. PERKINS: Thank you.

MR. BOLDUC: Yesterday mention was made of the high proportion of filler in formulations. We have tried with the system, just for curiosity, a mixture of 85 per cent sand and 15 per cent superphosphate and it granulated perfectly.

MR. MADIGAN: I think one more comment that might be in order is that the Brockville Chemical Industries Limited holds the patents. The licensing arrangement is rather simple. It is a nominal fee, a one time fee. And, in exchange for the fee you get three years of information pilot plant and actual plant operation, plus an exchange of information in the future of all licensees and free interchange of information.

MR. ADAMS: Thank you again Jim and Cam. This has been a real, good discussion.

Our next speaker on the program is Mr. Robert Clarke, Project Manager, Reynolds, Smith and Hills, Architects–Engineers–Planners. His paper will discuss low fluorine triple super phosphate. This should be very interesting in light of our pollution problems today. Mr. Clarke, please.

Low Fluorine Triple Superphosphate

Robert W. Clark

Most phosphates occur in nature in the form of an apatite with fluorapatite being the predominant form. Due to its low solubility, the phosphate content of apatite is not in a form readily available as plant food. A number of processes have been developed to convert the unavailable phosphates to a soluble or an available form. These processes fall under two general classes: thermal and acidulation.

The thermal process consists of reducing the phosphorus in the apatite to elemental phosphorus in an electric arc furnace. The phosphorus is then burned in air to form phosphorus pentoxide (P_2O_5) which is hydrated to form phosphoric acid. This process produces very pure acid which is used primarily in the food and fine chemical industries. Its application to the plant food industry has been limited due to its higher cost.

Soluble phosphates for the plant food industry are usually manufactured by acidulation of phosphate rock with a mineral acid. While sulfuric acid, phosphoric acid, nitric acid and hydrochloric acid can be used, only the first two have been of commercial significance in the United States.

Normal or single superphosphate is produced by acidulating phosphate rock with sulfuric acid. This gives a product high in calcium sulfate with approximately 20 percent P_2O_5. This product was the first commercially significant form of superphosphate produced. Although it is still produced in large quantities, its use is declining due to the increasing freight rates associated with handling a product with a relatively low plant food content.

Triple, or concentrated superphosphate, is produced by acidulating phosphate rock with phosphoric acid. The product generally contains about 46 percent available P_2O_5. Due to the obvious freight advantage of higher analysis materials, triple superphosphate has increased in importance and is a major source of phosphorus for mixed fertilizers.

A further step in this direction has been the development of high analysis superphosphates produced by acidulation of phosphate rock with superphosphoric acid. This product contains about 54 percent available P_2O_5. It is expected that this product will gain increased acceptance in the face of ever increasing freight and handling charges which comprise about 40 percent of the delivered cost of fertilizers.

Phosphate production has increased rapidly during the last twenty years. Figure 1 shows the data for superphosphates (1). Production of triple superphosphate increased almost 450 percent between 1950 and 1966. Since 1966, production has slowed some but 1968 totals were still 40 percent above 1960, but 18 percent below 1966. This temporary setback should be overcome and world production of triple superphosphate in 1971 should be four times the 1960 total. This curve also shows the decreasing production of normal superphosphate.

In the face of this expansion, phosphate producers have been confronted with ever tightening restrictions on air pollutants being emitted. Most phosphate rock contains a significant quantity of fluorine, with Florida rock averaging about four percent fluorine. In the production of superphosphates, some 10 to 30 percent of this fluorine is evolved. With the current processes, this evolution does not stop at the manufacturing step, but continues for several weeks while the products remain in the large curing sheds.

In Florida, where some 30 percent of the world's phosphate is mined and some 40 percent of this rock is converted to high analysis phosphates, state regulations limit fluoride emissions. New plants constructed after the regulations were established are limited to 0.4 pound of fluorine per ton of P_2O_5 produced as acid. Existing plants are allowed 0.6 pound of fluroine per ton of P_2O_5.

These regulations made it mandatory for all processors to either scrub the gases vented from the huge curing sheds or adopt some alternate process which would not occasion fluoride emissions from the curing sheds. Due to lack of feasible alternates and to time limitations to meet the new regulations, most companies in Florida installed scrubbers on the curing buildings at a capital cost of up to $1 million per installation.

Another disadvantage to the present process is the dust created by fines. Not only does this represent a potential loss of product, but it also creates an air pollution problem and poor working conditions.

Finally, the residual fluorine in the product may react with the available phosphates during subsequent ammoniation causing reversion to the unavailable fluorapatite.

II. NEW PROCESS

There are two avenues to approach the fluorine evolution problem. The first of these is to in some way tie up the fluorine so that it is not liberated from the product. A considerable effort was made in the industry some few years ago along this line and several additives, such as sodium hydroxide and ammonia, met with limited success chemically. For several reasons, primarily economic ones, this alternate was not commercially accepted.

The other avenue would be to, in some manner, increase the fluorine evolution during the acidulation step where the gases could be more easily scrubbed and thereby reduce the residual fluorine content of the product to a level where additional fluorine would not be evolved during curing and storage. This approach formed the basis for the new process being offered by Reynolds, Smith and Hills.

The essential feature of the process is the use of hot phosphate rock along with hot acid, followed by denning, cooling, crushing and screening of the product. A series of bench scale tests and pilot plant runs have been made to develop and evaluate this process. Some work was done using nitric and hydrochloric acids which demonstrated the processes applicability using these reactants. However, the majority of the work has been done with sulfuric and phosphoric acids and it is this work that will be discussed here.

The basic work has included investigations of various acid supplies, various rock supplies, ranges of acid and rock temperatures, ratios of acid to rock, additives, and varying time and temperatures of denning.

The bench scale work was carried out using a kitchen mixer with stainless steel bowls for the acidulation step. The acid was heated on a hot plate while the rock was heated in aluminum foil "boats" in a muffle furnace. This method assured that the rock was at a uniform temperature. The bowl was preheated to reaction temperature in an oven. The hot acid was dumped into the bowl and the hot rock added during mixing, taking about 10 seconds to complete rock addition. Mixing was then continued until the product had begun to "set up" at which time the bowl was dumped and the material broken up with a spatula. The crushed product was then immediately transferred to a beaker and placed in an oven for denning at a specified temperature and time interval, after which the beaker was removed, lightly covered and allowed to cool to room temperature. Analyses were performed after twenty-four hours. Standard A.O.A.C. analysis methods were used throughout.

A. NORMAL SUPERPHOSPHATE

A series of runs were made to produce normal superphosphate using hot phosphate rock. Runs were also made under identical conditions except that ambient temperature rock was used. Figure 2 shows the comparison of four such runs. Runs 1 and 2 were made with ambient rock and Runs 3 and 4 with rock preheated to 600 degrees F. The increase in fluorine evolution is very obvious with the hot rock process. Also, the increased evolution of water due to the increased reaction temperatures leads to more rapid curing and higher initial available P_2O_5. No fluoride evolution was detectable after the hot rock--hot acid acidulates reached ambient temperature.

B. TRIPLE SUPERPHOSPHATE

The majority of our work has been directed toward the production of triple superphosphate, using wet process phosphoric acid of about 52–54 percent P_2O_5 and 1.2 – 1.5 percent fluorine. The phosphate rock used contained about 34 percent P_2O_5 (74 BPL) and 3.9 percent fluorine. Acid temperatures used ranged from 180 degrees F. to 260 degrees F. and rock temperatures ranged from ambient to 800 degrees F. Figure 3 shows a graph of the percent fluorine in the product after twenty-four hours versus the rock temperature. All results were converted to the dry basis for comparison. As can be seen, the fluorine content drops rapidly as the rock temperature increases up to about 250 degrees F. and then only decreases slightly up to 800 degrees F. Product analyses after 24 hours averaged 49.4 percent TPA, 3.3 percent IPA, 46.1 percent APA, 13.5 percent F.A., 7.5 percent H_2O and 1.6 percent fluorine on a wet basis. The increased evolution of fluorine and water at the high reaction temperatures creates higher than normal TPA and APA values with the same raw material input.

Other acid sources were used ranging from reagent grade acid to wet process acids with high solids content. It was discovered, not surprisingly, that the major factor influencing the fluorine evoluation achieved was the fluorine content of the acid used. Figure 4 shows this relationship. Here we have plotted percent fluorine evolution versus the percent fluorine in the acid. In the range normally encountered with wet process acid used for triple superphosphate manufacture, 1.2 to 1.5 percent fluorine, the fluorine evolution could be expected to range from 33 to 29 percent of the input value. With furnace grade acid, this value exceeds 60 percent. We have consistently achieved higher fluorine evolution rates in our pilot plant work than in the bench scale tests. Thus, we feel that the results reported here are probably conservative.

For acids with higher fluorine contents, a series of tests were made with the addition of two percent of the rock weight of amorphous silica. This addition increased fluorine evolution to the equivalent of acids with lower fluorine contents. This silica addition is not required for acids with fluorine contents below about 1.5 percent. Where required, this would cost about $0.75 per ton of product. An attempt was made to use other less expensive sources of silica such as ground tailings obtained from a phosphate mine. No effects were seen even with eight percent addition of the sand.

An attempt was also made to determine the curing characteristics of the hot rock – hot acid products. It has been established by TVA that it is very difficult, if not impossible, to simulate pile curing on a small scale. However, it was felt that some data was required so an attempt was made. Small piles of about 200 pounds were produced in the pilot plant and periodically sampled as well as resampling small samples stored in sample bottles. The results of these tests indicated that initially about 94 percent of the total P_2O_5 is in the available form. This increases to about 96 percent after 28 days. This compares to about 95 percent for the standard process which increases to about 98 percent after six weeks.

Previous work by TVA on so called "quick cure" processes indicated that an increase in the speed of the conversion reaction could be accomplished only at the sacrifice of the degree of final conversion achieved. Apparently, this same effect occurs to an extent with the hot rock–hot acid process. However, this is offset by the increased grade achieved by the reduction of the fluorine content of the product. Thus, the same final grade may be achieved with the same quantities of raw material.

The process results in a semigranular material with good physical condition. Although the free acid analysis of the fresh product is relatively high, it does not appear wet or sticky. Samples only six hours old were ground in a mill for analysis without difficulty. Several hundred pounds of screened product were stored for a month with no evidence of caking. Figure 5 shows a photograph of a sample of product from the process and Figure 6 shows a typical particle size analysis. This material should load much faster and with considerably less trouble than R.O.P. triple. Also, it should offer advantages in mixed goods manufacture since the particle size is still small enough and porous enough for ammoniation without the caking and dusting problems inherent to R.O.P. triple.

In order to directly measure the fluorine evolution after manufacture, a series of tests were made in which 200 pounds of material were placed in a 27 cubic foot box and sealed. Air was drawn in through small holes around the bottom and exhausted from the top through a series of impingers by a vacuum pump.

During these tests we did not provide for maintaining the product temperature at the 140 degrees F. range, that would be the case in large piles, so we terminated the tests after five hours. A later test was run in which the product was placed in 55-gallon drums immersed in a water bath maintained at 140 degrees F. In this test, some 200 pounds of fresh product were continuously monitored for seven days.

The results of these tests are shown in the next two figures. Figure 7 shows the pounds of fluorine evolved per day per ton of product versus the time after the test was started.The top line indicated the 0.6 pound of fluorine per day per ton that has been measured in several cases with product produced in actual operating Florida plants. Tests 1 and 2 indicate the results from the five-hour tests and Test 3 is the seven-day test. It is apparent that the hot rock–hot acid process does essentially eliminate fluorine evolution after manufacture. Due to equipment breakdowns during these tests, this material was collected just after the disintegrator and had not been through the cooler and mill. We know that some additional fluoride evolution does take place in this equipment and, therefore, these data are probably conservative. You will note the very rapid drop in evolution after about twenty-four hours and that evolution reached essentially zero at about forty-eight hours.

Figure 8 shows another comparison of these data. The basis chosen was a 700 TPD P_2O_5 phosphoric acid plant, 1,000 TPD triple superphosphate production, and the existing Florida regulation permitting 0.6 pound fluorine emission per day per ton of acid P_2O_5 production. Emissions for the first twenty-four hours after manufacture are compared. This size acid plant allows us a fluorine emission of 420 pounds per day. At the current emission rate of 0.6 pound of fluorine per day per ton of product, we have an emission of 600 pounds of fluorine per day, well above the allowable level without consideration of other units in the plant complex, such as the acid plant and the granular triple plant if applicable. Using the data from the previous chart, we calculate three levels of emission for the hot rock–hot acid process. These range from a high of 37 pounds to a low of 14 pounds of fluorine per day, giving a range of 94 to 98 percent reduction over the current process. Thus, the hot rock–hot acid process does reduce fluorine evolution after manufacture to an insignificant level.

In addition, tests have been made to determine the ammoniation characteristics of this product. I'm sure you are aware of the difficulties involved in performing such tests on a small scale. One producer ran a series of tests for us in their laboratory using equipment they normally use to measure ammoniation rates. Identical runs were made using our material and their own run-of-pile triple. The results indicated that the hot rock–hot acid process product ammoniates at least as well as standard process product.

C. PILOT PLANT

A pilot plant was constructed to demonstrate the process on a larger scale, to provide scale-up data for a full scale design and to determine the applicability of the process with standard fertilizer plant equipment.

The nominal design capacity was 400 pounds of product per hour. Previous studies by TVA have indicated that the so called "TVA cone" will not operate at a rate of less than one ton per hour. For this reason, a cone was not installed in the pilot plant. Figure 9 shows the general layout of the pilot plant. Phosphate rock is fed from a storage hopper to an elevator to a constant speed screw. The screw delivers a measured quantity of rock to the rock heater. This unit is a 12-inch diameter cylinder with a 4-inch diameter pipe through the center. The rock is fed into the annular space where it is tumbled over the inner pipe by flights welded to the shell. A propane fueled burner supplies hot gases countercurrent through the center pipe. The shell is insulated on the outside to hold the heat. The hot rock then discharges into a double shafted pubmill.

Phosphoric acid is delivered from storage to a lead-lined heating tank where it is heated by four electric emmersion heaters, then pumped to the pugmill by a diaphragm pump. The acid is distributed through nozzles onto the pugmill bed.

The product discharges to an enclosed steel conveyor for denning. At the end of the den a standard "Squirrel cage" disintegrator breaks up the product before it is conveyed to a rotary cooler. The cooler discharges to a mill where it is further broken up and fed to a single deck screen. Oversize material is recycled to the cooler inlet.

All components are enclosed and connected by ducts to a standard water spray scrubber for removal of the fluorine.

A wide range of particle sizes may be achieved by varying the screen size and the mill discharges. We have experienced no dust problems with the product whatever.

Several designs of rock heaters have been proposed. We have discussed this unit with some ten equipment manufacturers and have several choices of equipment ranging from rotary indirect fired kilns to fluidized bed units. This area is still under investigation and the particular piece of equipment used will be determined on an individual basis according to available space, types of fuel, etc.

In a large scale plant the acid would be heated in a carbate shell and tube heat exchanger which is standard in the industry today.

Although we do not have actual operating experience with a cone, we expect a cone to improve mixing.

Thus, all of the equipment used is standard and proven in the industry today and should present no operating problems.

D. FLUOSILICIC ACID BY-PRODUCT

One of the major advantages claimed for this process is the possibility of the recovery of salable by-product fluosilicic acid. This is possible since the amount of fluorine evolved is almost doubled and all fluorine evolution occurs in essentially closed equipment where it can be easily and efficiently collected.

Using typical formulations and assuming the use of Florida rock with 3.9 percent fluorine and acid with 1.6 percent fluorine, a 30 percent evolution and recovery of fluorine would produce 19.9 pounds of fluosilicic acid per ton of product produced. This is equivalent to 86.5 pounds of 23 percent solution per ton of product or about 8,600 tons per year for 200,000 TPY production.

Obviously, the first question that arises is just what is the market situation now for fluosilicic acid? Reynolds, Smith and Hills has done a fairly extensive market survey in an attempt to answer this question. Our responses ranged from extremely optimistic to extremely pessimistic.

The *Oil, Paint and Drug Reporter* in 1967 (2) estimated the market at some 50,000 tons per year of 23 - 25 percent solution, plus another 50,000 tons per year converted to the solid sodium salt. The acid is used by most larger cities and the salt by the smaller areas. The ten largest users consumed about 30,000 tons of solution in 1967 at a delivered price ranging from $31.40 to $45.00 per ton of solution. The weighted average price was $34.44 or $143.50 per ton of 100 percent. With a freight rate of $16.60 per ton of solution for Jacksonville to New York, this leaves a price of $17.84 per ton of solution or $74.33 per ton of 100 percent fluosilicic acid F.O.B. plant.

However, recent inquiries have indicated that short term contracts are available at considerably higher prices, in the neighborhood of $200 per ton of 100 percent acid. Applying the same freight rate, this leaves a price of $130.83 per ton of 100 percent acid F.O.B. plant. For purposes of this evaluation, we have assumed an F.O.B. price of $100 per 100 percent ton.

The use of fluosilicic acid as a raw material for synthetic Cryolite for the aluminum industry is also increasing. The zinc and magnesium salts are used as hardening agents in the cement industry.

With these additional uses and with several expansions in fluosilicic acid production facilities announced recently, we feel that the market is sufficiently well established to justify allowance of a credit for fluosilicic acid sale.

E. ECONOMICS

Obviously, the economics of this process depend to a large degree on the individual application. This is particularly true for the rock heater. If a long cylindrical kiln is used, a considerable amount of open space will be required. As most of you know, space is at a premium, especially in the older plants. The distance from the rock heater to the acidulator will set the size and cost of equipment for conveying the hot rock. Also, the size of existing scrubbing equipment may be sufficient so as not to require a new installation. Obviously, the least cost situation would be to include required equipment as part of a new plant design where the full extent of savings in size of storage facilities and reduced leading equipment could be realized.

The next four figures present a preliminary engineering estimate of the operating costs for this process compared to the cost of operating a scrubber complex on a large curing building. Two cases have been chosen for illustration. Case 1 uses a capital cost of $400,000 and Case II $800,000. This is not to say that these are necessarily the upper and lower limits. All cases are based on 200,000 tons per year of production and reflect only the cost above that already existing in the current process.

It should be pointed out that no credit has been allowed for labor savings during loading of the semigranular product which could be significant. Also, it should be noted that if production is restricted or the plant is down for some time, this system would be down with no direct operating cost, whereas building scrubbers must be operated continuously regardless of production demands or plant operations.

Figure 10 presents the estimated operating cost for a $400,000 capital investment as $0.41 per ton of product. Credit for fluosilicic acid sale is $1.00 per ton of product leaving a net savings of $0.59 per ton. Figure 11, for $800,000 capital, shows an operating cost of $0.73 per ton and a net savings of $0.27 per ton. Figure 12 is the estimated cost of operating building scrubbers showing a cost of $0.86 per ton. Figure 13 is a comparison of these three cases. Even without any credit for fluosilicic acid recovery and with the higher capital costs, the hot rock–hot acid process shows a 15 percent lower operating cost than storage building scrubbers. With credit for fluosilicic acid recovery, an air pollution control system is converted from a loss to a potentially profit making operation.

III. SUMMARY

In summary, the hot rock–hot acid process utilizes phosphate rock heated to 250 degrees F. and up and wet process phosphoric acid heated to about 250 degrees F. to produce a semigranular triple superphosphate which does not evolve significant quantities of fluorine during curing or storage. The process will cost from $0.40 to $0.75 per ton above current cost. Credit for by-product fluosilicic acid can convert this to a $0.30 to $0.60 per ton savings. Even without such credit, the process offers a lower cost alternate to installation of scrubber complexes on curing buildings.

For those plants using very low grade acid with high fluorine contents, it may be necessary to add some silica to achieve the desired fluorine evolution. This cost, however, can be offset by the by-product, fluosilicic acid, produced.

In addition, there are other benefits that are difficult to affix dollar values to, such as, essentially eliminating a dust problem, increased loading rates and improved working conditions due to reduced fluorine in the air.

Work on this process was begun by Mr. Albert Henderson in 1962. Reynolds, Smith and Hills became involved in 1967 providing engineering evaluation and design services. A patent application was filed in December, 1967. The pilot plant was constructed in early 1968, and work has been continuous through this period and is still continuing.

We welcome inquiries from any interested parties concerning further details on this process. Inquiries should be directed to: Mr. Robert W. Clark, Project Manager, Reynolds, Smith and Hills, Post Office Box 4850, Jacksonville, Florida 32201, Phone A/C 904 - 396-2011.

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(See Figures 1 thru 13, Pages 120 thru 133)

MR. ADAMS: Thank you for that excellent, thorough paper on "Low Fluorine Triple Superphosphate". I am sure when the final paper is published, in "The Proceedings" you will have many interested Round Table members and others, who will be purchasing the 1969 Proceedings, thoroughly interested in your paper when they digest its contents.

I will now turn the meeting over to our chairman, Albert Spillman.

CHAIRMAN SPILLMAN: I think we have had a very good meeting. Our speakers have done a superb job presenting their respective papers. Discussions from the floor to speakers, and back to the floor, were excellent. Thanks to our speakers, both from U.S. and outside of U.S., for giving their time and money to be with us to share their knowledge with our membership.

We thank the Hotel Mayflower personnel for all the courtesies extended to our group. Everyone that was contacted for services were prompt and courteous.

We thank the press for the coverage given to the Round Table thruout the year and for coming to our meeting. You have done an excellent job and we always appreciate your interest.

I thank my Associates on The Executive Committee and many of our members for coming to our assistance and helping with the additional burdens immediately after our Dear Chairman, Vince Sauchelli, passed away, last October 1, 1969. Vince worked with us on most of the planning for this meeting until a few days before his sudden passing away. Vince, we missed you and we thank you for your devoted work with the Round Table for the past 19 years. We thank you, Vince, for the guidance and advice you have given to us during this period. We intend to do our best to keep the Round Table operating on the highest principal possible and shall always be thinking of you. God Bless.

Continued Page 134.

FIGURE 1.

U.S. PRODUCTION OF SUPERPHOSPHATES

(MILLIONS OF SHORT TONS OF P_2O_5)

SOURCE: FERTILIZER TRENDS, 1967, T.V.A.

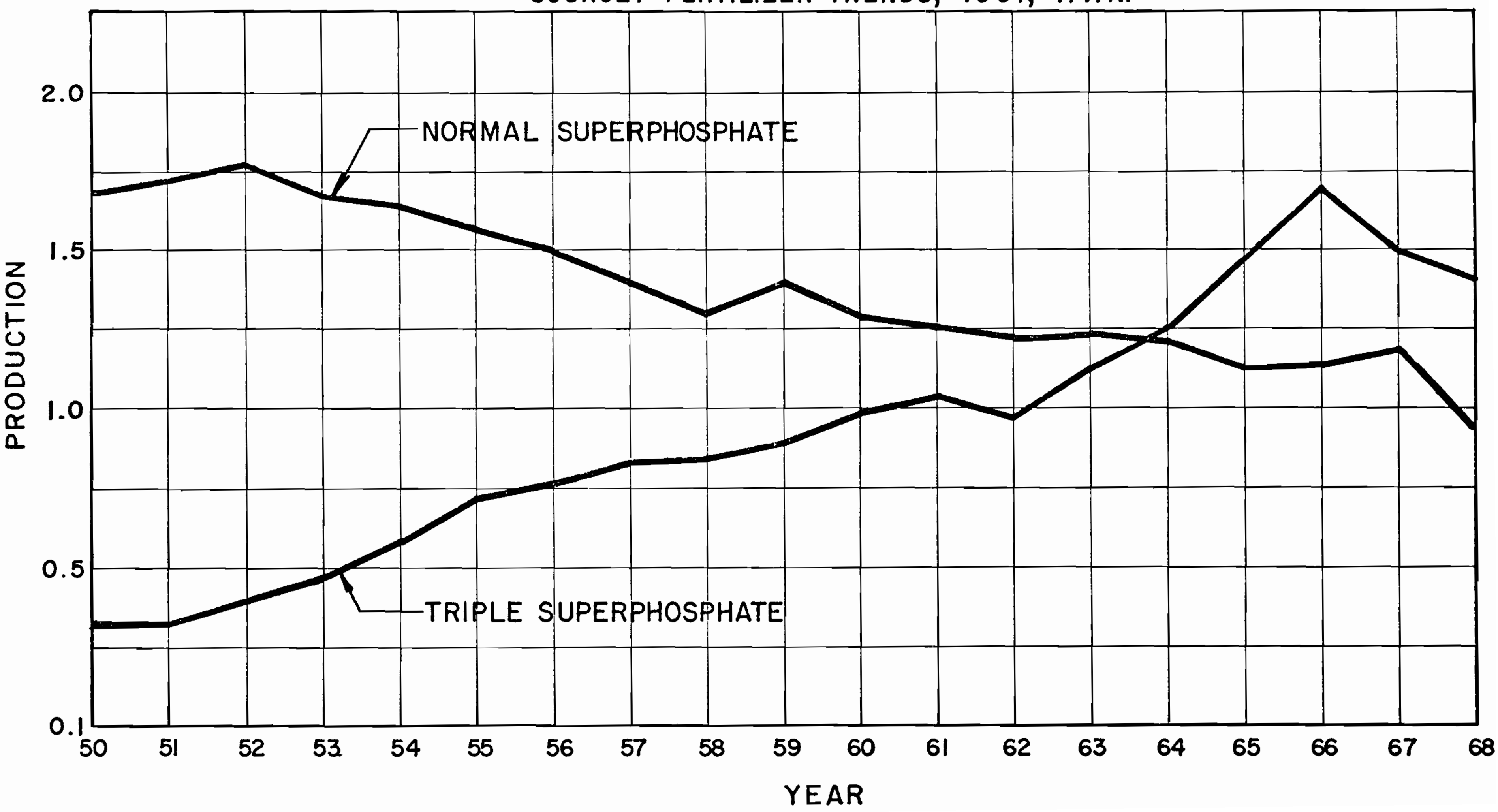


FIGURE 2

NORMAL SUPERPHOSPHATE TESTS

Process	Percent TPA	Percent IPA	Percent APA	Percent FA	Percent H_2O	Percent F	Percent F Evolved
Standard	20.2	1.5	18.7	4.4	10.2	1.7	22
Standard	20.2	1.5	18.7	4.5	10.4	1.7	22
Proposed	21.8	2.0	19.8	5.5	3.6	0.8	67
Proposed	21.7	1.8	19.9	5.6	3.2	0.8	69

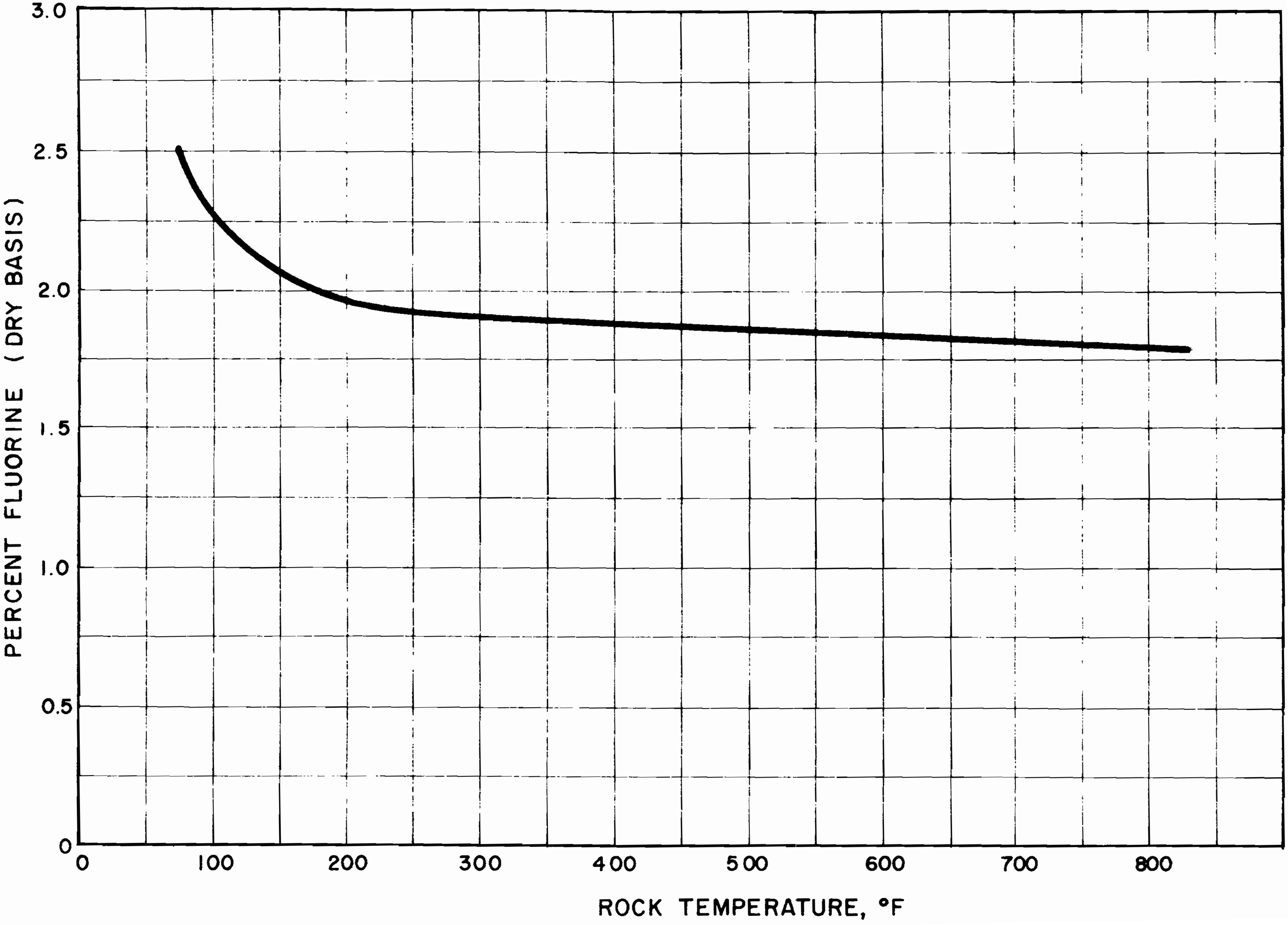
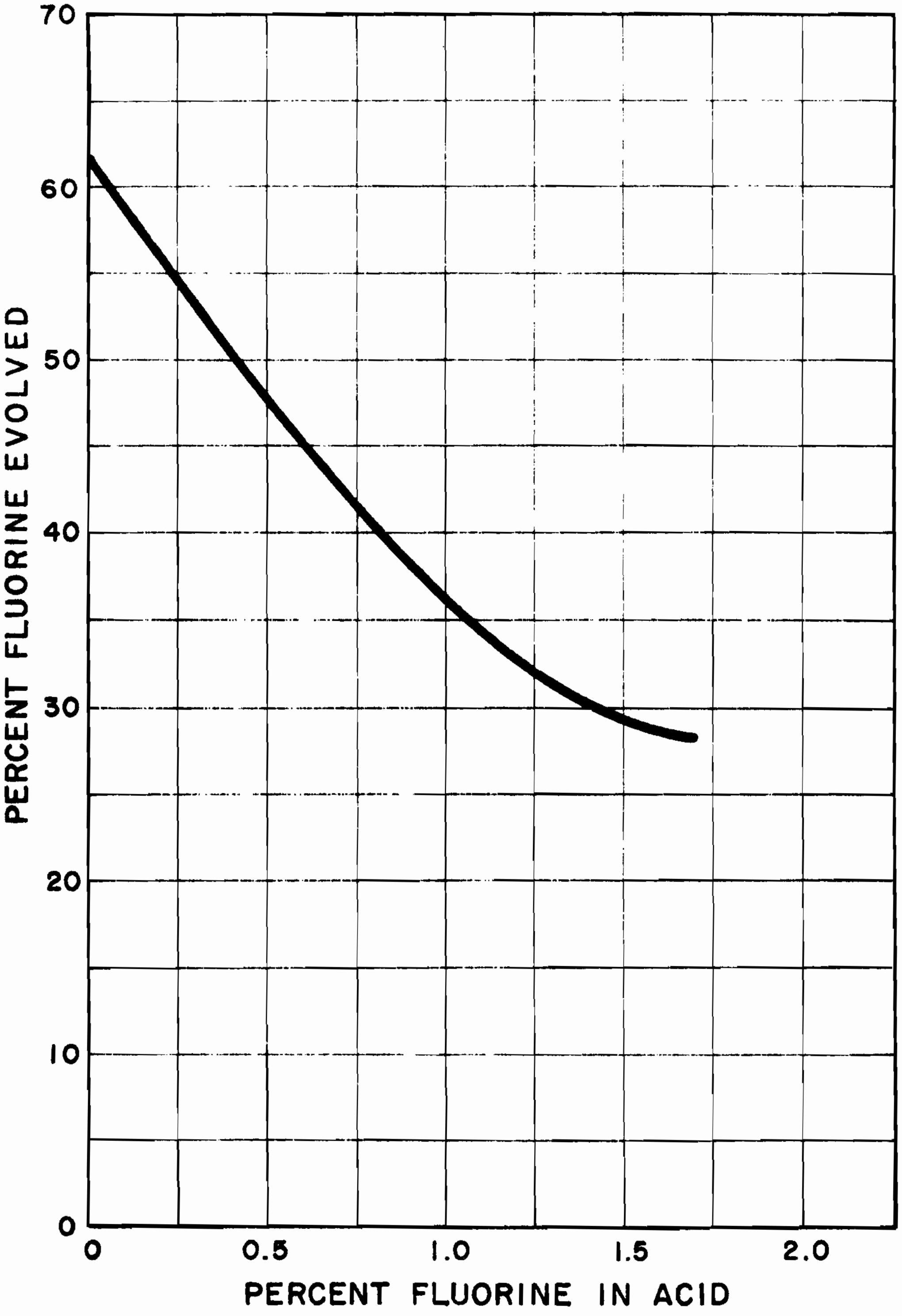


FIGURE 3. FLUORINE IN PRODUCT VS ROCK TEMPERATURE

FIGURE 4.

FLUORINE EVOLUTION VS FLOURINE IN ACID



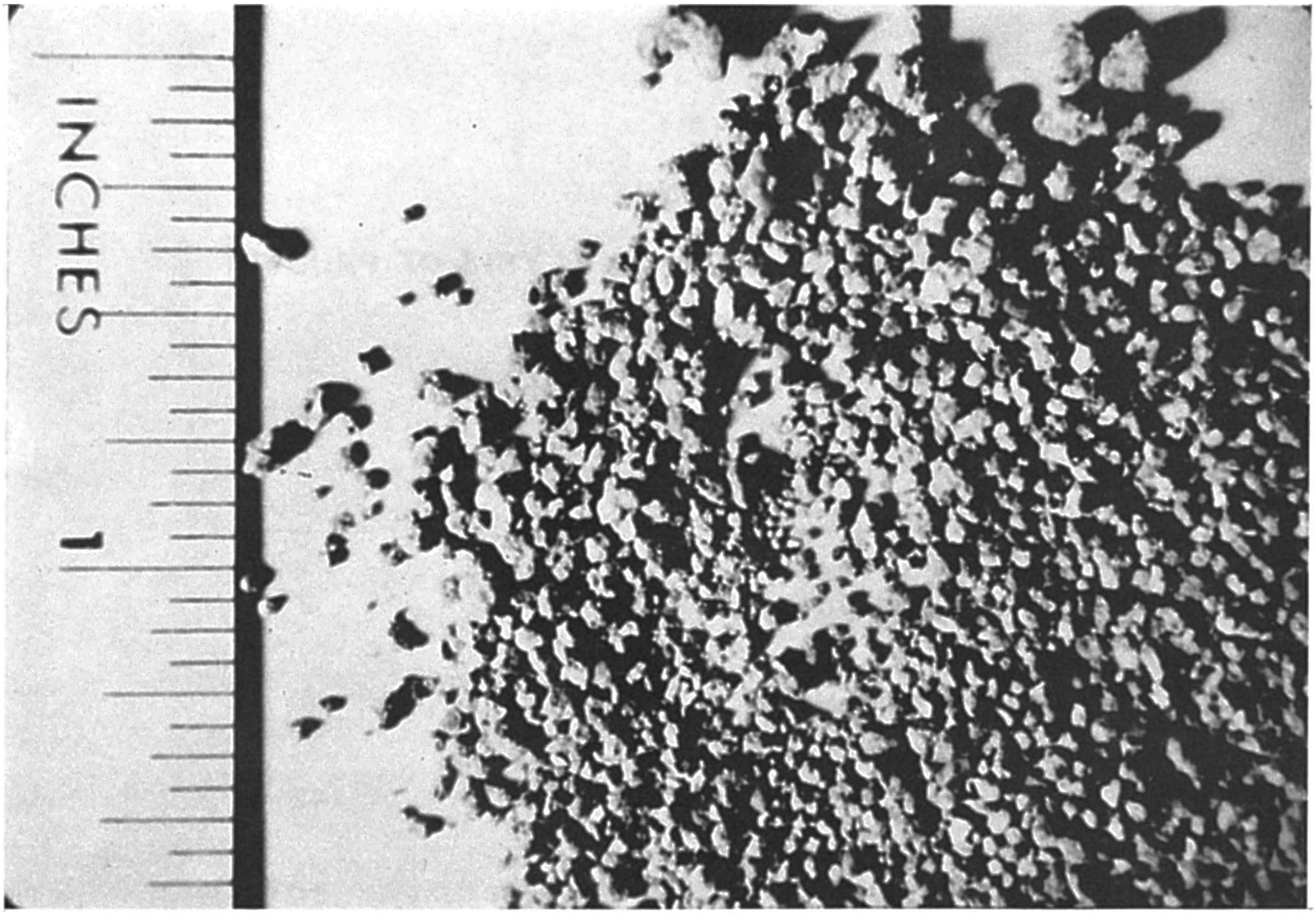


FIGURE 5

SAMPLE PRODUCT

FIGURE 6.

TYPICAL SCREEN ANALYSIS OF PRODUCT

TYLER STANDARD SCREEN SIZE	% OF PRODUCT
+ 7 MESH	2
− 7 + 10 MESH	6
− 10 + 16 MESH	42
− 16 MESH	50

FIGURE 7. FLUORINE EVOLUTION TESTS

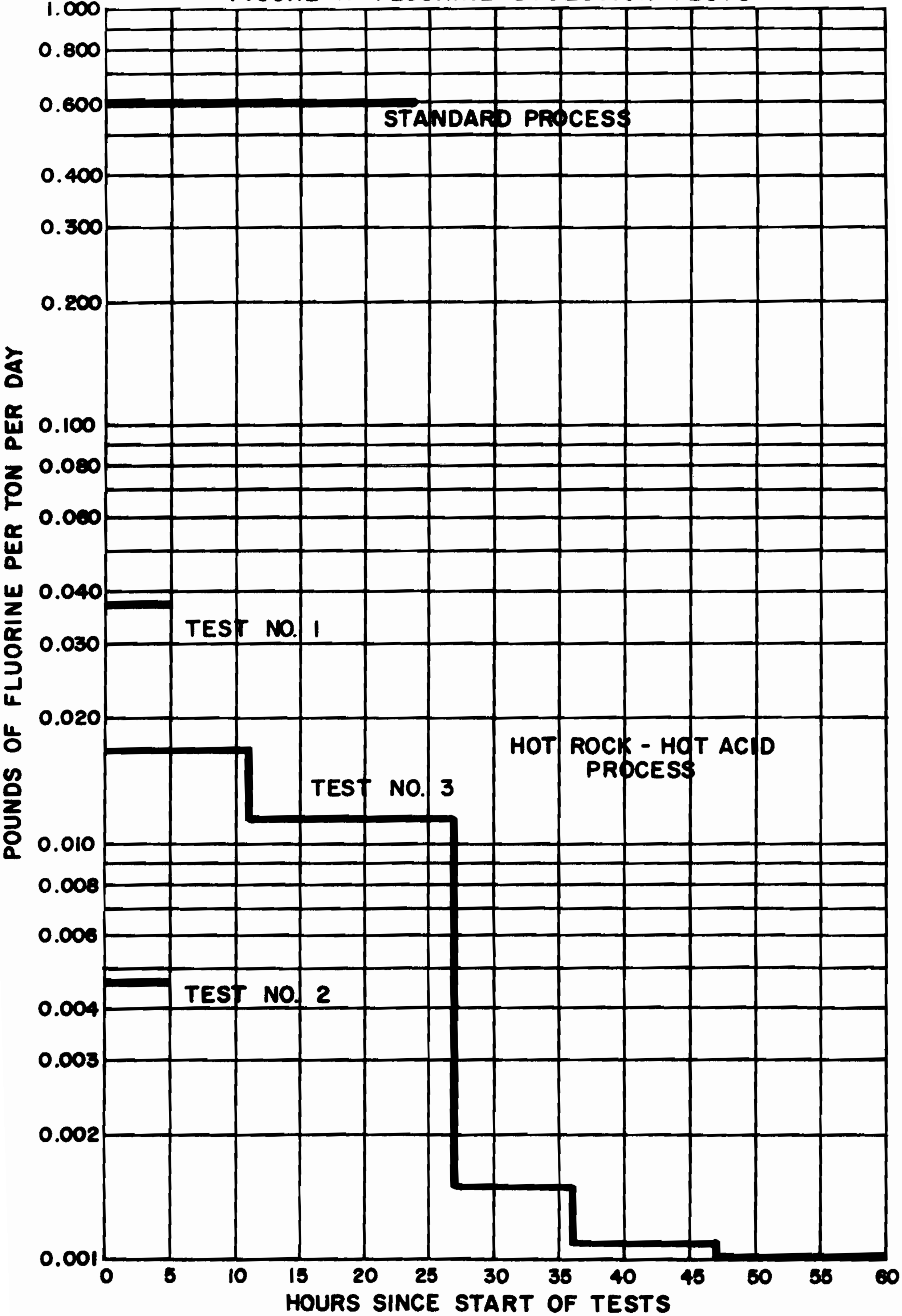


FIGURE 8

COMPARISON OF FLUORINE EVOLUTION RATES

Basis: 700 TPD P_2O_5 Acid Plant

1,000 TPD TSP Production

Florida Regulation: 0.6 lb F per day per P_2O_5 TPD acid capacity
First 24 hours after manufacture

Allowable total fluorine emission = 700 x 0.6 = 420 lbs/day

Emission at present level of 0.6 # F per day per ton = 0.6 x 1,000 = 600 lb F/day

Emission with hot rock--hot acid process =

High: 0.037 # F per day per ton x 1,000 tons = 37 lb F/day

$$\text{Percent reduction} = \frac{600 - 37}{600} \times 100 = 93.8 \text{ percent}$$

Low: 0.0046 # F per day per ton x 1,000 tons = 4.6 lbs F/day

$$\text{Percent reduction} = \frac{600 - 4.6}{600} \times 100 = 99.2 \text{ percent}$$

Median: 0.014 # F per day per ton x 1,000 tons = 14 lb F/day

$$\text{Percent reduction} = \frac{600 - 14}{600} \times 100 = 97.7 \text{ percent}$$

FIGURE 9.

GENERAL ARRANGEMENT OF PILOT PLANT

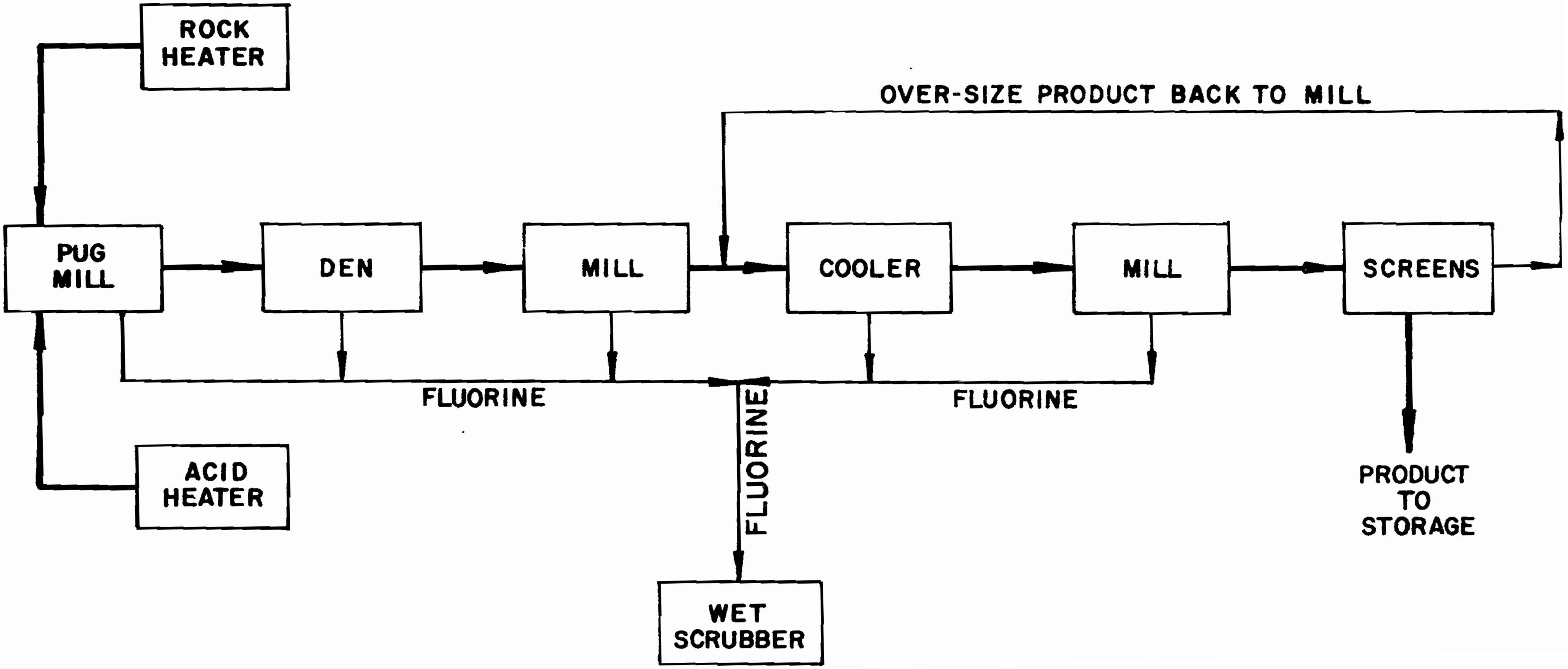


FIGURE 9.

GENERAL ARRANGEMENT OF PILOT PLANT

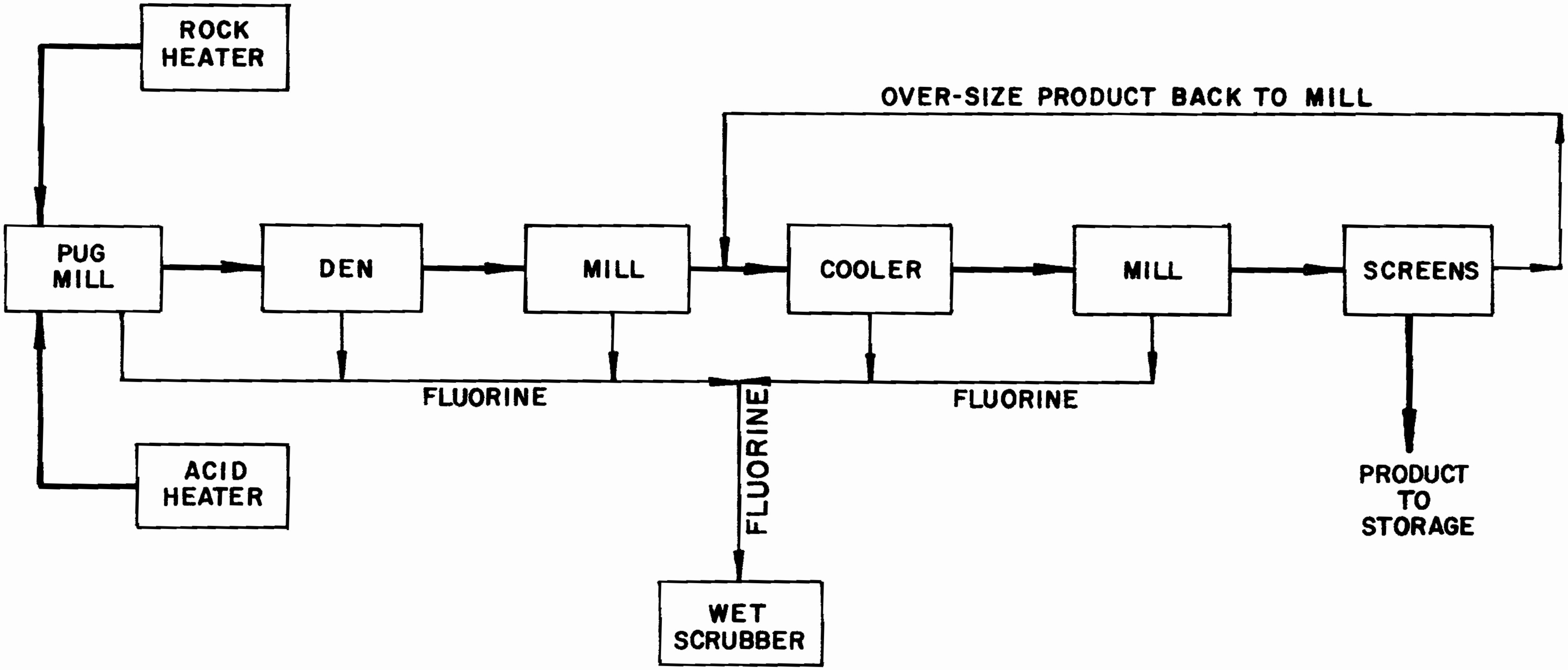


FIGURE 10

OPERATING COST - CASE I

Capital: $400,000

Production: 200,000 TPY

		$/Ton Product	
Direct Cost:			
Electricity:	200 hp @ $0.01/KWH	$0.03	
Natural Gas:	9 x 10^6 Btu/hr @ $0.35/$10^6$ Btu	0.06	
Maintenance:	6 percent of capital	0.12	
	Total Direct Cost		$0.21
Indirect Cost:			
Depreciation:	10 Yr - straight line	0.20	
	Total Indirect Cost		$0.20
	Total Operating Cost		$0.41
	Credit for H_2SiF_6 Recovery		(1.00)
	NET COST		(0.59)

FIGURE 11

OPERATING COST - CASE II

Capital: $800,000

Production: 200,000 TPY

		$/Ton Product	
Direct Cost:			
Electricity:	200 hp @ \$0.01/KWH	$0.03	
Natural Gas:	9×10^6 Btu/hr @\$0.35/10^6 Btu	0.06	
Maintenance:	6 percent of capital	0.24	
	Total Direct Cost		$0.33
Indirect Cost:			
Depreciation:	10 Yr - straight line	0.40	
	Total Indirect Cost		0.40
	Total Operating Cost		$0.73
	Credit for H_2SiF_6 Recovery		$(1.00)
	NET COST		$(0.27)

FIGURE 12

OPERATING COST - CURING BUILDING SCRUBBERS

Capital: $800,000

Production: 200,000 TPY

		$/Ton Product	
Direct Cost:			
Electricity:	500 hp @ $0.01/KWH	$0.16	
Labor:	2 men/day--5 days/week	0.05	
Payroll Charge:	20 percent of labor	0.01	
Maintenance:	6 percent of capital	0.24	
	Total Direct Cost		$0.46
Indirect Cost:			
Depreciation:	10 Yr - straight line	0.40	
	Total Indirect Cost		$0.40
	Total Operating Cost		$0.86

FIGURE 13

COMPARISON OF OPERATING COST
($/Ton Product)

Case:	I	II	Building Scrubbers
Capital:	$400,000	$800,000	$800,000
Production:	200,000 TPY	200,000 TPY	200,000 TPY
Direct Cost:			
Electricity	$0.03	$0.03	$0.16
Natural Gas	0.06	0.06	
Labor			0.05
Payroll Charges			0.01
Maintenance	0.12	0.24	0.24
Total Direct Cost	$0.21	$0.33	$0.46
Indirect Cost:			
Depreciation	0.20	0.40	0.40
Total Indirect Cost	$0.20	$0.40	$0.40
Total Operating Cost	$0.41	$0.73	$0.86
Credit for H_2SiF_6 Recovery	($1.00)	($1.00)	0
NET COST	($0.59)	($0.27)	$0.86

Thanks to our members for promptly coming to our rescue and contributing the necessary deficite to pay last year's bills.

Your Executive Committee and your Advisory Committee is already deep in studies for a good program for our 20th annual meeting to be held in Memphis Tenn., Wednesday, Thursday and Friday, November 4-5-6, 1970. Don't forget. Put these dates on your calendar and keep the appointment. Also, please send your suggestions on subject matters you think will be appropriate for our 1970 Meeting. Contact our Secretary; also for additional 1969 proceedings and back issues.

Thanks to our hard working Secretary Treasurer, Dr. Marshall for his usual hard work and interest to carry on the details necessary thruout the year. Doc is a real pusher. When he calls on you and wants something done, you better drop everything and start performing. Doc will stand on your door steps until you take care of the work he wants done. We thank you Doc for a real job well done.

If you have no further business to discuss I will call for motion to adjourn this 19th Annual Meeting.

A MEMBER: May I congratulate all of you dedicated people who have spent so much of your time to plan and organize this great Round Table. You have helped the Industry more than anyone, for so little cost, to keep it informed of up-to-date technical information in all phases of Fertilizer Operations. I have attended 5 of your meetings and have all of the proceedings for the past 18 years. I can honestly say I have been educated on fertilizer production matters, more from your meetings and proceedints than any other educational material I could find. Good luck for many more successful Round Table years. I recommend we all stand and applaud these dedicated people. A long standing ovation took place.

CHAIRMAN SPILLMAN: Thank you sir. Meeting was adjourned at 11:25 a.m.

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