

THE COLLECTION OF PRODUCTS FROM THE VAPOR-PHASE REACTION
OF PHOSPHORUS, AIR, AND AMMONIA

by

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A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Chemical Engineering

KANSAS STATE UNIVERSITY
Manhattan, Kansas

1964

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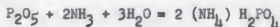
INTRODUCTION

Agriculture is the basic industry of mankind, and the prosperity of this industry depends on the fertility of the soil. Plants utilize inorganic materials from the soil. The soil becomes exhausted unless phosphoric acid, nitrogen and potash salts are supplied to it by artificial means. Ground bone was one of the first materials to be used extensively as fertilizer.

The fertilizer industry always tends to produce fertilizers having the highest possible plant nutrient content. This content is expressed as the percentages of N_2 , P_2O_5 and K_2O . Considerable research has been done and is still being carried out on different phosphorus, nitrogen and potassium carrying materials in fertilizers.

Various processes have been developed for producing ammonium phosphates (1). Such processes differ from each other in their technical aspects, however, the general principle involved in all the production methods is the same. The phosphoric acid is neutralized by anhydrous ammonia, and the chemical composition of the product depends upon the amount of acid neutralized. Phosphoric acid is produced either by the wet process or by the furnace process. Both of these processes need heavy equipment, and hence have large fixed and operating costs.

Recently much work has been done on the production of concentrated fertilizer materials by the vapor phase reaction of P_2O_5 , ammonia and water. The reaction



was assumed in the beginning of these studies, however the reaction was found to be more complex (2) (3) (4). The vapor phase reaction produces concentrated fertilizer materials with less investment.

For the vapor phase reaction molten phosphorus is supplied to the reactor. Dry air is passed into the reactor where molten phosphorus is oxidized to phosphoric oxide (P_2O_5)_x in the gaseous state. Ammonia and water are introduced into the reactor at the point of oxidation or at a point downstream. These reactions take place within the temperature range of 600-900°F. Rice (5) observed that the products of this reaction could not be collected, unless they were cooled rapidly. If the products were not cooled rapidly they decomposed into a sticky, hygroscopic and glassy material which was not collectible.

Wang (6) and McDonald (7) attempted to collect the products in an electrostatic precipitator. McDonald (7) also tried to collect the products in a fluidized bed and in a filter bag. He suggested that the fluidized bed in combination with a filter bag could be used successfully.

The studies presented in this thesis were conducted to obtain further data on fluidized bed collection using a small scale pilot plant. The effect of process variables on the collectibility of the products was investigated. Studies were also conducted on the effect of various air to phosphorus and ammonia to phosphorus molar ratios on the efficiency of collection in the bed of particles.

LITERATURE SURVEY

In 1959 Wang (6) conducted studies on the effect of various process variables on chemical and physical properties of the products of vapor-phase

reaction of phosphorus, air, ammonia and water. He investigated the possible methods of collecting the products of this reaction. A 3-inch stainless steel tubular reactor was used. Molten phosphorus was fed to the reactor and was burned in a blast of air. The combustion products were reacted with ammonia gas and water vapor. The products were carried out of the reactor by a stream of excess air and ammonia. The product gases had a smoke-like appearance.

For these studies Wang used an electrostatic precipitator to collect the products. His attempts to collect the products, using filters and cyclone collectors, proved ineffective. The products obtained from an electrostatic precipitator were white, flour-like and in some cases too hygroscopic for fertilizer use. The products contained about 14 to 15 per cent nitrogen and about 60 to 70 per cent P_2O_5 .

McDonald (7) continued Wang's work using the same basic equipment. He studied various collection systems such as an electrostatic precipitator, a bag filter and different types of fluidized beds.

The electrostatic precipitator was found to be an effective means of collecting products, but its operating difficulties limited the runs to only 20 to 30 minutes. McDonald also found that the bag filter was as efficient as the electrostatic precipitator and it was considered to be 100 per cent efficient. Considering this fact he used a bag filter in combination with various types of fluidized beds. Auxiliary air was used in all the runs, employing a fluidized bed to obtain the desired degree of fluidization or motion.

McDonald, in the runs employing a fluidized bed alone, observed channeling at the bottom of the bed and violent slugging in the upper part.

Sometimes large slugs of particles were transported up the column and into the filter. In order to reduce slugging, a packed calming section was placed, in the column beneath the bed. However, the fluidized bed with a packed calming section was not very successful because the products collected on the packing until plugs were formed near the bottom of the packing. The plugging was followed by a rise in pressure; and, in some cases, the back pressure was sufficient to lift the bed and packing up into the filter. McDonald used the fluidized bed with baffles in his later runs and this was found to be very efficient.

MATERIALS AND METHODS

Materials

Air, ammonia and phosphorus were the basic materials used in this study. The compressed air, supplied by the power plant of the University, was used. This air had a considerable amount of moisture in it, hence it was necessary to dry it by passing it through a silica gel bed. The silica gel was 3-8 mesh, from the Davison Chemical Company. The ammonia was refrigeration grade from the Spencer Chemical Company, and was contained in a conventional cylinder holding 100 pounds of liquid. The cylinder was fitted with a pressure regulator to control the pressure in the feed line.

The elemental phosphorus conformed to National Formulary Specifications and was purchased from Fisher Scientific Company. It was in the form of light yellow, wax-like sticks and was kept under water for safe storage and shipment.

Pelleted ammonium phosphate fertilizer was used in the bed for

collection of products. The fertilizer had 16 per cent nitrogen and 48 per cent P_2O_5 . It was bought from Farmers Co-op Association in 50 pound bags. The pellets, 8-10 mesh, were separated by sieving from commercial fertilizer and were used in the bed of particles.

Equipment

A schematic diagram of the reactor and associated equipment, used in this study, is shown in Plate I. Plate II shows the equipment as viewed from the front, and Plate III shows an oblique view of the equipment. The equipment consisted of the following parts:

(1) Air Flow Nozzle. A schematic diagram of the air flow nozzle is shown in Plate IV. Plate V is a photograph of the nozzle arrangement. A half-inch flow nozzle was used to measure the flow rate of air. The flow nozzle was installed in a three-inch pipe and the pressure taps were located according to the specifications given by "Compressed Air Handbook" (8). A thirty-six inch mercury manometer was used to measure the pressure drop across the nozzle.

(2) Silica Gel Dryer. Air coming out of the compressed air line was dried by passing it through a bed of silica gel. Two columns were set up so that they could be used alternately. Each column was made up of a four foot length of six-inch black iron pipe. Reducing flanges were used on both ends to connect the one-inch pipes. The bed of silica gel was supported from the bottom by a circular disk with a few small holes in it. The holes in the disk were made small enough to allow only the air to go into the one-inch pipe. Twenty pounds of silica gel were charged to one of the columns and this same initial charge was used throughout the

EXPLANATION OF PLATE I

Schematic Diagram of Reactor and Associated Equipment

- | | |
|---|--|
| A. Air flow nozzle | J. Water flowmeter |
| B. Silica gel dryer | K. Ammonia flowmeter |
| C. Constant temperature water bath | L. Manometer, gage pressure in the reactor |
| D. Electrical immersion heater | M. Conical air distributor |
| E. Mercury thermometer | N. Glass column for the bed of particles |
| F. Phosphorus melting pot | O. Shield |
| G. Electrical heating tape for phosphorus feed line | P. Filter bag |
| H. Reactor (In actual installation the reactor was tipped as shown in T ₁ , T ₂ , T ₃ . Thermocouple wells Plate III to facilitate cleaning) | ∞ Valves |
| I. Safety valve | → Relief valve or safety valve |

EXPLANATION OF PLATE II^a

Front View of Reactor and Associated Equipment

- C. Constant temperature water bath
- D. Electrical immersion heater
- I. Safety valve
- J. Water flowmeter
- K. Ammonia flowmeter
- L. Manometer, gage pressure in the reactor
- M. Glass column for the bed of particles
- N. Conical air distributor
- O. Shield
- P. Filter bag

PLATE II

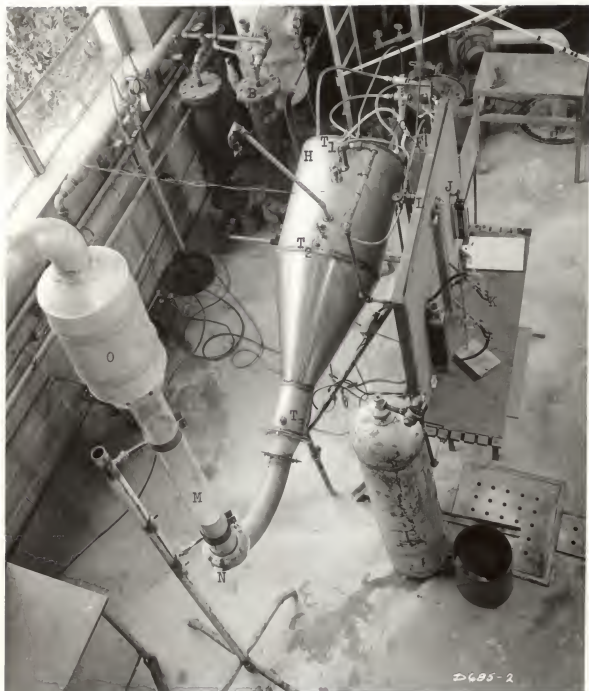


EXPLANATION OF PLATE III

Oblique view of Reactor and Associated Equipment

- A. Air flow nozzle
- B. Silica gel dryer
- C. Constant temperature water bath
- F. Phosphorus melting pot
- G. Electrical heating tape for phosphorus feed line
- H. Reactor
- I. Safety valve
- J. Water flowmeter
- K. Ammonia flowmeter
- L. Manometer, gage pressure in the reactor
- M. Glass column for the bed of particles
- N. Conical air distributor
- O. Shield
- T_1, T_2, T_3 . Thermocouple wells

PLATE III

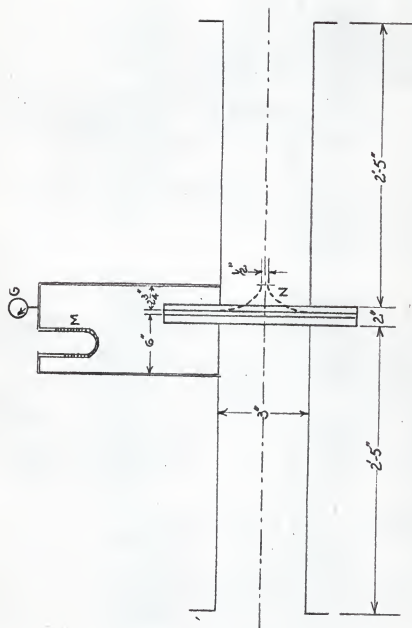


EXPLANATION OF PLATE IV

Schematic Diagram of Air Flow Nozzle Arrangement

- N. Air flow nozzle
- G. Pressure gage, pressure at the downstream
end of nozzle.
- M. Forty-eight-inch mercury manometer, pressure
drop across the flow nozzle.

PLATE IV

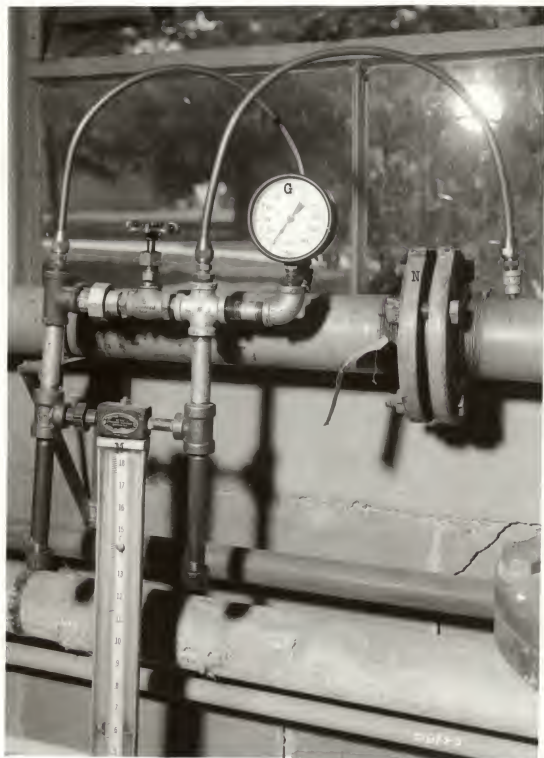


EXPLANATION OF PLATE V

Air Flow Nozzle Arrangement

- N. Air flow nozzle
- G. Pressure gage, pressure at the downstream end of nozzle
- M. Forty-eight-inch mercury manometer, pressure drop across the flow nozzle

PLATE V



studies without regeneration.

(3) Phosphorus Melter. A six-inch diameter by eight-inch long stainless steel cylinder was used as a phosphorus melter. The top of the cylinder was covered with a blind flange and this flange was tapped at three places. A flipper valve was connected to one of the three couplings on the blind flange. The phosphorus feed line was connected to one coupling and the end of the tube, at which molten phosphorus entered, was kept below the water and molten phosphorus interphase level. Water was fed from the third coupling. Since molten phosphorus is heavier than water, water was used to force phosphorus into the reactor. Yellow phosphorus was charged into the melter, and the assembly was placed in a constant temperature water bath. The water bath was a square sheet-metal tank. Water in the tank was heated by an electrical immersion heater. A powerstat was used to control the voltage supply to the heater. A plastic bucket, installed at a height of twenty feet above the melter, was used as a constant head water tank. Water was metered into the melter, displacing the molten phosphorus into the reactor. The phosphorus feed line was constantly heated by an electrical heating tape.

(4) Reactor. The reactor was made of 0.090 inch thick stainless steel plates. A schematic diagram of the reactor is shown in Plate I. Plate III shows an oblique view of the reactor. Air coming out of the silica gel dryer was supplied at four points on the upstream end of the reactor. This end of the reactor was provided with a six-inch diameter port and was covered with a stainless steel plate. This arrangement was made so that the reactor could be opened for inspection and to facilitate cleaning. The reactor was mounted on three one-inch pipes and was tipped

from the horizontal to facilitate cleaning. For temperature measurements, three iron-constantan thermocouples were located at the positions shown in Plate I and were connected to a sixteen point (converted to three point) Brown temperature indicator recorder. A twenty-four-inch mercury manometer was used to measure gage pressure in the reactor. The safety valve, used with the reactor, was a two feet long $1\frac{1}{2}$ inch pipe, one end of which was connected to the reactor and other end was sealed with a six to eight-inch long bicycle tire tube. When the gage pressure in the reactor was higher than 10 inches of mercury the tube would break and release the pressure.

(5) Bed of Particles. A four-foot long, four-inch I. D. pyrex glass column was used for the bed of particles. A four-inch flexible metallic duct was used to connect the downstream end of the reactor to the glass column. A sheet-metal conical air distributor was designed to support the bed of particles in the column. Plate VI shows the details of the conical air distributor. The top of the glass column was connected to a shield. The shield was used to keep the particles from flowing out of the column. The shield was made of two ten-inch gasoline funnels connected by a twelve-inch length of ten-inch sheet-metal duct. A 60 degree cone, nine inches in diameter, was fixed inside the duct as shown in Plate I. The conical parts of both the funnels were truncated by four inch circles and one end of the shield was connected to the top of the column, while the other end of the shield was connected to a four-inch sheet-metal duct, which in turn was used to carry the products into the filter bag.

(6) Filter Bag. Plate VII shows the filter bag in its operating

EXPLANATION OF PLATE VI

Oblique View of the Conical Air Distributor

Path of air flow through this distributor
is shown in the cross-sectional sketch on
the left. This unit is shown as unit M on

Plate I.

PLATE VI



EXPLANATION OF PLATE VII

Filter Bag in its Operating Condition

PLATE VII



condition. The filter bag was eleven feet long and about four feet in circumference. It was made of a thick coarse cotton cloth. The bottle-neck part of the bag was tied to the four-inch duct coming out of the shield.

Experimental Methods

The phosphorus melter was charged with six to seven pounds of phosphorus. This amount of phosphorus was sufficient for about fifteen hours of operation. The flange on the melter was bolted tight so that no leakage occurred. This was important in order to insure a constant and accurate flow of molten phosphorus. The melter was then placed in the water bath and was connected with the water line and the phosphorus feed line. The flipper valve on top of the melter was always kept open and was closed only when phosphorus was fed to the reactor.

Before the start up of each run the glass column was properly connected with the flexible duct and the conical air distributor was properly placed in the column. The required amount of ammonium phosphate pellets was charged to the column and the shield was placed on top of the column. The filter bag was then tied to the four-inch duct coming out of the shield.

Hot water (70°C) was poured into the water bath and the immersion heater was started. Power supply to the heater was so adjusted that the temperature of water remained constant at 70°C. White phosphorus melts at 44.1°C, therefore the bath temperature was high enough to insure its molten state. Water flow into the melter was started and water was allowed to run until the melter was completely filled. This was indicated by water flowing out of the flipper valve. Next the desired flow rate of

water was set and the flipper valve was closed. The air flow was started and its rate was adjusted to a desired value. Since the phosphorus flow rate was small, it took about 10-15 minutes for molten phosphorus to reach the reactor inlet. The entry of phosphorus into the reactor was indicated by the appearance of white smoke in the glass column. At this point the flow of ammonia was started, and its rate was adjusted to the desired value. The temperature recorder was then switched on.

The flow rates of air and ammonia remained constant, whereas, the water flow rate changed frequently and needed constant supervision.

At the end of each run, the water flow rate into the melter was stopped first. Air and ammonia were allowed to run for five or ten more minutes to make sure that all the phosphorus in the reactor had been reacted. Next the ammonia and air valves were turned off and the flipper valve was opened to let phosphorus run back into the melter. The reactor was allowed to cool for about 30 minutes.

A standard form was used to record the data for each run. This form is shown with the sample calculations in the Appendix.

The glass column and conical air distributor were cleaned with steam at the end of each run. It was not necessary to remove the products from the bag after each run. The bag was cleaned after it had been used for ten to fifteen runs and was washed with water and dried before it was used again. Some of the products were collected on the walls of reactor, moreover some red sticky material was also observed in the reactor. The formation of this material is further discussed in the section on variables affecting conversion. This red sticky material, in some instances, plugged off the phosphorus feed line. For these reasons the reactor and phosphorus

feed line were cleaned after every three or four runs. Steam was used in cleaning and it was found that the red sticky material could be easily removed by steam and hot water.

Safety

(1) Storage. Since yellow phosphorus has a low flame temperature (34°C), it was stored under water.

(2) Handling. Phosphorus is very poisonous to the human body, as well as a fire hazard. It had to be handled very carefully. Physical contact with phosphorus was avoided and a pair of tongs was used in transferring yellow phosphorus from the storage tank to the phosphorus melter. The time of exposure of phosphorus to air during transfer was kept as short as possible.

(3) Leakage. Leakage of molten yellow phosphorus in the feed line was a hazard because molten phosphorus burns spontaneously. In such cases cold water was poured on the point of leakage, and the line was checked immediately after the run.

(4) Plugging. In some instances it was found that the phosphorus feed line was plugged off by solid yellow or red phosphorus. This was noticed by a drop in the water flowmeter reading. When this occurred the valve feeding water to the melter was opened slightly to increase the water flow rate. If this did not raise the flow rate, the valve was turned off completely and no further attempts were made to force phosphorus into the reactor for fear that the sudden opening of the plug and the high water pressure would force a large excess of molten phosphorus into the reactor causing a sudden rise in temperature and pressure. The safety

valve was designed to take care of such a sudden rise in pressure, but in spite of this molten phosphorus might have leaked through the flanged connections. The molten phosphorus burns readily, and the smoke is suffocating and poisonous. Whenever such a leak occurred, the flow of air and ammonia was cut off completely, and the flanges were disconnected with great care. The molten phosphorus was allowed to run into a bucket of water, or else it was covered with sand. Because of the danger of such accidents, and in order to avoid injury, the presence of two persons during each run was required. A constant watch on the water flowmeter and the mercury manometer was maintained.

Analysis

The samples were analysed for total nitrogen and total P_2O_5 . The analysis were made by the Chemical Service Laboratory of Kansas State University using standard A. O. A. C. methods (9).

The samples for analysis were collected from the conical air distributor for the first three runs. In later runs the samples were collected from the product deposits in the shield. The shield was cleaned after each run. This insured that a given sample was not contaminated with the products from previous runs.

PRESENTATION AND DISCUSSION OF EXPERIMENTAL RESULTS

Experimental investigations were carried out to study the effect of various process variables on the collection of products and on the conversion of phosphorus. As previously discussed, the filter bag was considered to be a 100 per cent efficient collector and was used in series

with the bed of particles to study the collection of products. Ammonium phosphate (8-10 mesh) pellets were used in the bed.

Behavior of the Bed of Particles

The conical air distributor, used as a support for the bed of particles in the column, distributed air equally around the column. The conical air distributor had some advantages over other types of bed supports. The screen, if used as a bed support, would be plugged by the products whereas, the conical air distributor had a large opening, and hence the plugging in the distributor was not severe.

It was observed that the behavior of the bed depended mainly on the velocity of gases through the bed. Since the phosphorus feed rate was kept constant, the gas velocity was changed by changing the air to phosphorus molal feed ratio. The bed fluidized well in some cases, but in most of the cases violent slugging was observed in the bed.

Plate VIII illustrates a typical behavior of the bed of particles. Five pounds of particles were used in the column. An air flow rate corresponding to an air to phosphorus molal feed ratio of 500 (56.25 g-moles/min) was used to study the behavior of the bed. In Plate VIII, (A) shows the bed of particles before the air flow was started, whereas (B), (C) and (D) show the particles in motion.

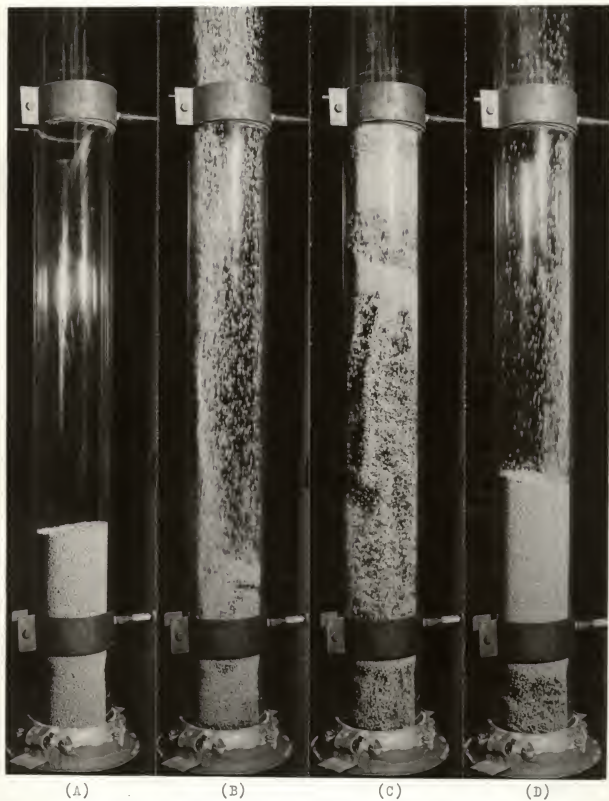
There was some continuous bubbling and channeling at the bottom of the bed. In Plate VIII, (B) shows the early stages in the formation of a slug. As the particles fall back from the top, the slug becomes larger and starts moving up the column (D). This slug when transported to the upper part of the column, starts breaking (C), and the particles on the

EXPLANATION OF PLATE VIII

Behavior of the Bed of Particles

- (A). The bed of particles before the air flow was started.
- (B). The early stages in the formation of a slug.
- (C). Breaking of a slug in the upper part of the column.
- (D). The formation of a large slug in the bottom part of the column.

PLATE VIII



bottom part of the slug start moving towards the bottom of the column. When the slug has reached the top of the column it becomes very small and most of the particles have started falling towards the bottom (B). Some particles, which are carried out of the column, hit the cone of the shield and fall back into the column. As these particles continue to fall back to the bottom of the column, another large slug forms (D). This process of slugging goes on in a continuous cycle.

Variables Affecting Collection

The effect of air to phosphorus molal feed ratio, ammonia to phosphorus molal feed ratio and weight of the bed on the efficiency of collection of products in the bed of particles was studied.

(1) Air to Phosphorus Molal Ratio. Runs 8 to 14 were made to study the effect of air to phosphorus molal feed ratios on the collection of products in the bed of particles. In all these runs 2,107 grams of 8-10 mesh ammonium phosphate pellets were used in the bed and the ammonia to phosphorus molal feed ratio was kept constant at three. The data obtained from these runs is presented in the Appendix, Table T-1.

Figure 1 shows the per cent collection of products in the bed, the conical air distributor and the pipe and shield at various air to phosphorus molal ratios. The collection of products in the conical air distributor was small in all cases. The collection of products in pipe and shield was higher than the collection in the bed for air to phosphorus molal ratios greater than 450. It was also observed that the collection of products in the bed decreased exponentially with an increase in the air to phosphorus molal feed ratio (See Figure 2). This relationship was

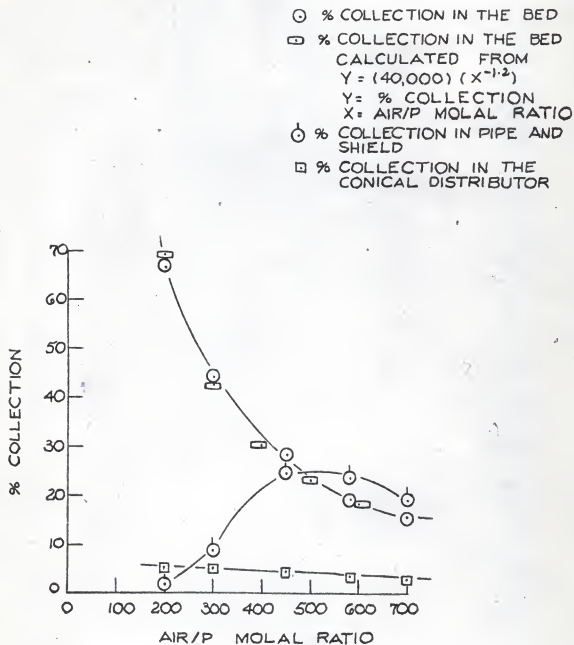


FIGURE 1. THE EFFECT OF AIR TO PHOSPHORUS MOLAL RATIO ON THE COLLECTION OF PRODUCTS IN THE BED, AT A CONSTANT AMMONIA TO PHOSPHORUS MOLAL RATIO OF 3.

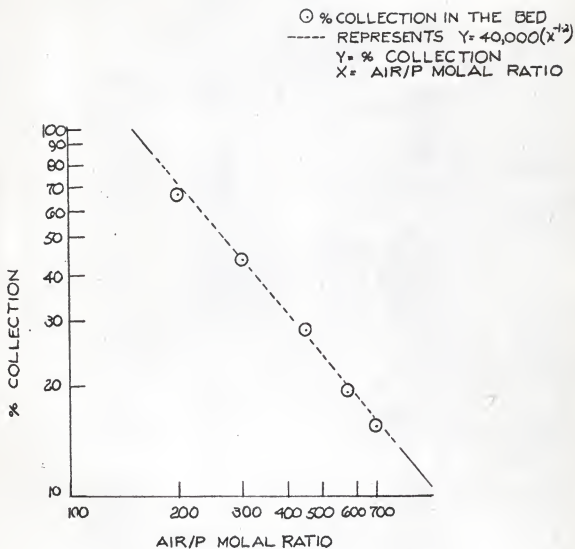


FIGURE 2. THE EFFECT OF AIR TO PHOSPHORUS MOLAL RATIO ON THE COLLECTION OF PRODUCTS IN THE BED, AT A CONSTANT AMMONIA TO PHOSPHORUS RATIO OF 3.

found to be

$$Y = 40,000 (X)^{-1.2}$$

where X = air to phosphorus molal feed ratio with $200 \leq X \leq 700$
and Y = per cent collection in the bed.

(2) Ammonia to Phosphorus Molal Ratio. Initially, runs 1 to 7 were made to study the collection of products at various ammonia to phosphorus molal feed ratios. In these runs the air to phosphorus molal feed ratio was kept constant at 489, and 2,107 grams of particles were used in the bed. It was assumed that the collection of products in the pipe and shield was negligible. However, in the later runs, per cent collection in the pipe and shield was found to exceed that in the bed of particles. Thence, it was decided to measure the weight of the products collected in the pipe and shield.

In runs 21 to 25 the operating conditions were slightly changed, as compared to runs 1 to 7. The air to phosphorus molal feed ratio was kept constant at 500, and 2,270 grams (5 lbs.) of particles were used in the bed. In these runs, the collection of products in the pipe and shield was taken into account, and it was observed that the per cent collection in the pipe and shield was equal to the per cent collection in the bed. On the basis of these observations the original data obtained from runs 1 to 7 were calculated for per cent collection in the bed. Sample calculations C-3 in the Appendix show the method used in these calculations. The data obtained from runs 21 to 25 and the calculated data from runs 1 to 7 are presented in the Appendix Table T-3.

The method of curvilinear regression (10) was used to represent

the relationship between the per cent collection of products in the bed and the ammonia to phosphorus molal ratio. The regression line, as shown in Figure 3, is

$$Y = 9.01 + 9.408X - 1.35X^2$$

where Y = expected value of per cent collection in the bed
and X = ammonia to phosphorus molal ratio.

The collection of products in the bed was a maximum for an ammonia to phosphorus molal ratio of 3 to 4. For ammonia to phosphorus molal ratios less than 2 some plugging in the conical air distributor was observed and, because of this, the per cent collection in the distributor was reasonably high at low values of ammonia to phosphorus molal ratio.

(3) The Weight of the Bed. The effect of the weight of the bed on the collection of products in the bed was studied in runs 25 to 29. In these runs the air to phosphorus molal feed ratio was kept constant at 500 and ammonia to phosphorus molal feed ratio was kept constant at 3. The data obtained from these runs are presented in the Appendix Table T-4.

Figure 4 shows per cent collection vs. weight of the bed data. A polynomial approximation was used to find the relationship between the per cent collection of products in the bed and the weight of the bed. This relationship is

$$y = 10.615 + 9.43x - 1.145x^2$$

where y = per cent collection of products in the bed
and x = weight of the bed.

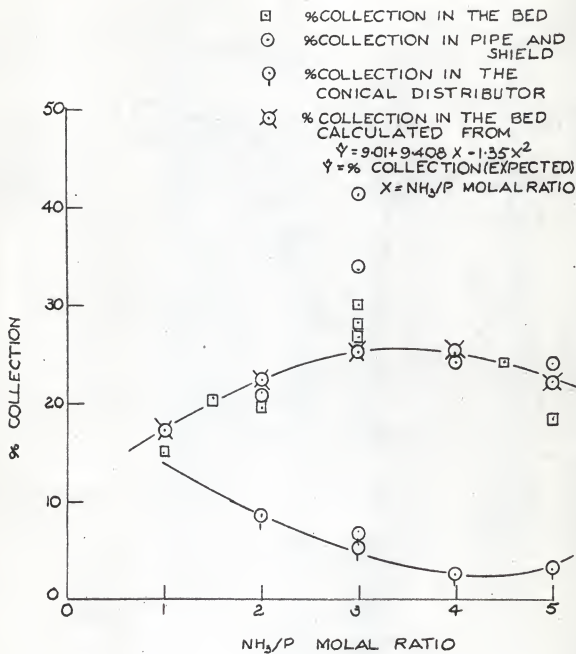


FIGURE 3.

THE EFFECT OF AMMONIA TO PHOSPHORUS MOLAL RATIO ON THE COLLECTION OF PRODUCTS IN THE BED, AT A CONSTANT AIR TO PHOSPHORUS MOLAL RATIO OF 500.

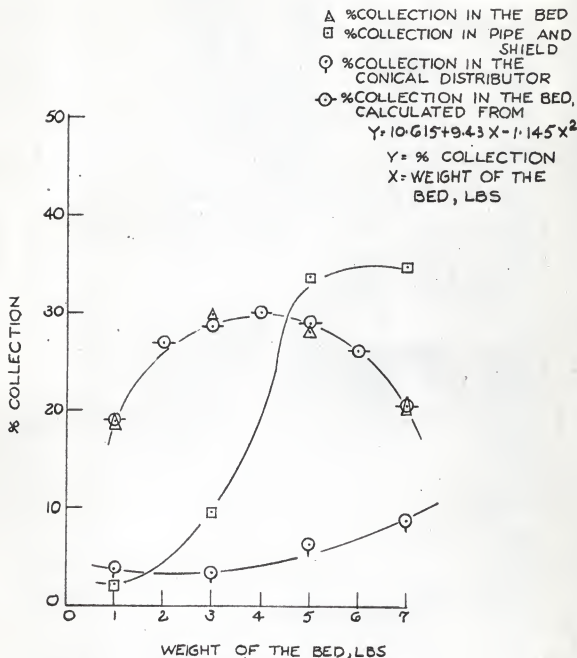


FIGURE 4.

THE EFFECT OF THE WEIGHT OF THE BED ON THE COLLECTION OF PRODUCTS IN THE BED, AT A CONSTANT AIR TO PHOSPHORUS MOLAL RATIO OF 500 AND AMMONIA TO PHOSPHORUS MOLAL RATIO OF 3.

From Figure 4 it is evident that the efficiency of collection was a maximum when 4 pounds of particles were used in the bed.

In these investigations it was found that the per cent collection of products in the bed was high when slugging in the bed was low. Moreover it was noticed, from visual observations, that the intermixing of particles in the bed was better in the runs where higher collection efficiencies were obtained.

It is possible that the efficiency of collection in the bed depended upon the extent to which the bed fluidized. Leva (11) stated that the quality of fluidization depends mainly on the particle size distribution, the gas velocity, the bed height, the height-to-diameter ratio and the design of the fluid distributor.

In all these runs the phosphorus feed rate was kept constant. Thus by varying the air to phosphorus molal feed ratio the gas velocity through the bed of particles was varied. From Fig. 1 it is evident that decreasing air to phosphorus molal feed ratio, and hence decreasing gas velocity, increased the efficiency of collection in the bed. The attempts to collect the products at air to phosphorus molal ratios less than 200 were not successful because in these cases, the gas velocities were not high enough to achieve appreciable motion in the bed.

It is possible that the efficiency of collection could be increased by the use of mechanical agitators or baffles in the fluidized bed. The collection of the products in such modified fluidized beds provides an area for future studies. The effect of particle size distribution on the collection of products is another factor to be considered for future studies.

Variables Affecting Conversion

The products obtained from runs 1 to 11 were analysed for chemical composition. The products from later runs were not analysed because the operating conditions in these runs were not much different from those in runs 1 to 11. The results of the chemical analysis are shown in the Appendix Table T-5.

The products had an average P_2O_5 content of 64 to 65 per cent. Total nitrogen content ranged from 11 to 15 per cent. These compositions, when compared with the compositions of some known ammonium phosphate materials (7), approximate those of diammonium pyrophosphate.

The product obtained from all the runs was a white flour-like material. It was quite stable and non-hygroscopic. The bulk density of the product was low (0.285 gram/cc.). For the purpose of storage the product was compressed into pellets using a Carver laboratory press.

There was no definite effect of air to phosphorus and ammonia to phosphorus molal ratios on the product compositions. However, in the runs employing ammonia to phosphorus molal ratios less than two some glassy, sticky material was observed in the conical air distributor. This might have been due to the presence of some unreacted phosphorus pentoxide.

In these studies a large excess of air was used to accomplish rapid cooling of the products and also to achieve fluidization in the bed of particles. The air to phosphorus molal feed ratios varied from 200 to 700. Since the air flow rate was large in all the runs, the temperature profiles in most of the runs were identical. Figure 10 in the Appendix shows typical profiles from run number 10.

The per cent phosphorus recovered in the products was calculated as shown in Sample Calculations C-4 in the Appendix. The calculated data is presented in the Appendix Table T-6. The per cent conversion of phosphorus data was arranged in order of size, from high to low, in an array. This arrangement is shown in Fig. 5. The data was analysed statistically (10). The mean conversion of phosphorus was 59.8 per cent and the variance about the mean was 210.4.

The per cent conversion of phosphorus data was further analysed to study the effect of air to phosphorus and ammonia to phosphorus molal ratio on the conversion of phosphorus. The statistical methods, outlined by Fryer (12), were used in the analysis of data.

(1) Air to Phosphorus Molal Ratio. Figure 6 shows the effect of air to phosphorus molal ratio on the conversion of phosphorus. The regression line is

$$\hat{Y} = 61.22 + 0.057 (X - 474.6)$$

where \hat{Y} = per cent conversion of phosphorus (expected)

X = air to phosphorus molal ratio

$\bar{y} = 61.22$, mean per cent conversion of phosphorus

$\bar{x} = 474.6$, mean air to phosphorus molal ratio

$b = 0.05703$, estimate of the slope β

with $CI_{90}: 0.00784 \leq \beta \leq 0.1062$

The calculation of the regression line was based on the data obtained from $n=11$ runs.

(2) Ammonia to Phosphorus Molal Ratio. Figure 7 shows the effect of ammonia to phosphorus molal ratio on the per cent conversion of phosphorus.

⊙ %CONVERSION OF PHOSPHORUS

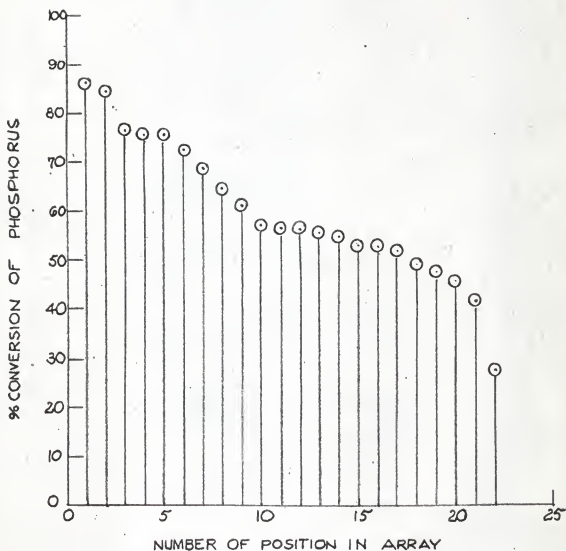


FIGURE 5. GRAPHICAL REPRESENTATION OF AN ARRAY. PERCENT CONVERSION OF PHOSPHORUS DATA.

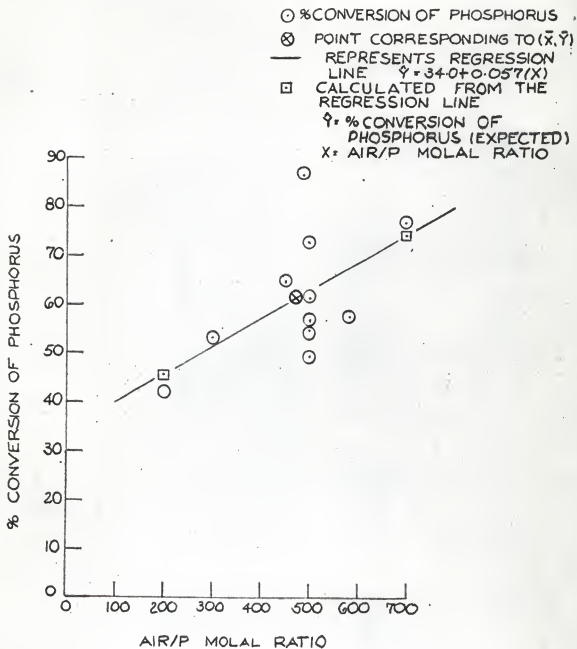


FIGURE 6. THE EFFECT OF AIR TO PHOSPHORUS MOLAL RATIO ON THE PERCENT CONVERSION OF PHOSPHORUS, AT A CONSTANT AMMONIA TO PHOSPHORUS MOLAL RATIO OF 3.

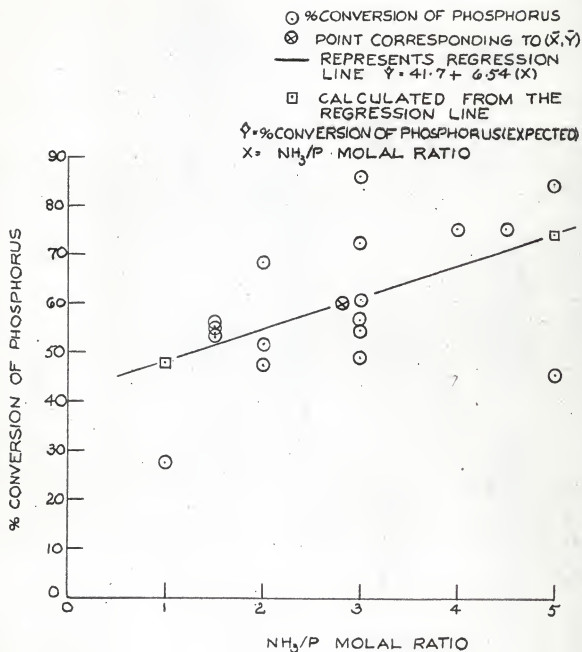


FIGURE 7. THE EFFECT OF AMMONIA TO PHOSPHORUS MOLAL RATIO ON THE PERCENT CONVERSION OF PHOSPHORUS, AT A CONSTANT AIR TO PHOSPHORUS MOLAL RATIO OF 500.

The regression line is

$$\hat{Y} = 60.16 + 6.54 (X - 2.823)$$

where \hat{Y} = per cent conversion of phosphorus (expected)

X = ammonia to phosphorus molal ratio

\bar{y} = 60.16, mean per cent conversion of phosphorus

\bar{x} = 2.823, mean ammonia to phosphorus molal ratio

b = 6.54, estimate of the slope β with

$$CI_{90}: 1.796 \leq \beta \leq 11.28$$

The calculation of the regression line was based on the data obtained from $n=17$ runs.

In both these cases the hypothesis, $H_0: \beta=0$, was tested and was rejected using the probability of a type I error of 10 per cent. This indicates that the air to phosphorus and ammonia to phosphorus molal ratios had some effect on the conversion of phosphorus. The relationships given above can be used in the preliminary design work. Detailed studies in this line may lead to more useful results.

In all these runs the recovery of phosphorus was poor. The losses of phosphorus in the system was attributed to the following causes:

(1) The first and largest contribution was the formation of red phosphorus. The conversion of yellow phosphorus to red phosphorus is discussed in detail by Van Wazer (13). Exposure of yellow phosphorus, to elevated temperatures in a closed vessel, for an extended period of time, was the main factor affecting conversion. The formation of red phosphorus in the reactor was shown by the presence of red sticky material near the point where molten yellow phosphorus entered the reactor. Exposure

of yellow phosphorus to ultraviolet radiations during storage was another factor affecting the conversion of yellow phosphorus to the red variety.

(2) The collection of products on the interior surfaces of the reactor and the flexible metallic duct, however small, was another cause for low phosphorus recovery.

(3) The third cause was the leakage of products from the various parts of the system.

The efficiency of conversion of phosphorus could be increased by avoiding the formation of red phosphorus. In order to accomplish this the method of feeding molten yellow phosphorus to the reactor should be improved. The use of an atomizing nozzle, as discussed by Rice (14), may be the next step in this direction.

CONCLUSIONS

The air to phosphorus molal feed ratio was the main factor affecting collection of the products in the bed of particles.

The product of the reaction was a white flour-like material having a low bulk density. The composition of products was not changed either with air to phosphorus molal ratio or with ammonia to phosphorus molal ratio. However, these factors had some influence on the recovery of phosphorus in the products.

RECOMMENDATIONS

Future studies in the following areas are recommended.

(1) The effect of particle size distribution on the collection of

products in the fluidized bed.

(2) The use of an agitated or baffled fluidized bed in the collection of products.

(3) The application of an atomizing nozzle for feeding molten yellow phosphorus to the reactor.

ACKNOWLEDGMENTS

The author wishes to express his sincere appreciation to Dr. Herbert T. Bates for his advice and guidance in designing the reactor. The author is also grateful to Professor Raymond C. Hall for his guidance and help in the later part of this investigation.

Acknowledgment is given to Dr. William H. Honstead, Professor and Head of the Chemical Engineering Department, whose co-operation and guidance made this work possible.

The funds for this project were provided by the Kansas State Agricultural Experiment Station.

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APPENDIX

Tables

Table T-1. The effect of the air to phosphorus molal ratio on the collection of products at a constant ammonia to phosphorus molal ratio of 3.

Run No.	Air/P molal ratio	Per cent of total products collected by			
		Bed of particles	Conical distributor	Pipe and shield	Filter bag
8	582	19.35	3.78	23.6	53.3
9	400	41.6	11.02	20.15	27.2
10	700	15.5	3.0	19.05	62.5
11	300	44.2	5.31	8.98	41.5
12	450	28.3	4.82	24.5	42.4
13	200	66.6	5.42	1.91	26.1

Table T-2. Original data obtained from runs 1 to 7. The air to phosphorus molal ratio was kept constant at 489. In these runs no data was taken on the collection of products in the pipe and shield.

Run No.	NH ₃ /P molal ratio	Length of run min.	Weight, in gms., of the products collected by		
			Bed of particles and conical distributor	Filter bag	Total (A+B)
1	2.0	53	98	171	269
2	1.5	48	104	165	269
3	3.0	60	205	261	466
4	5.0	60	70	211	281
5	2.0	58	97	169	266
6	1.0	27	26	54	80
7	4.5	39	97	176	273

Table T-3. The effect of the ammonia to phosphorus molal ratio on the collection of products at a constant air to phosphorus molal ratio of 500.

Run No.	NH ₃ /P molal ratio	Per cent of total products collected by				
		Bed of particles	Conical distributor	Pipe and shield	Filter bag	
1	2.0	20.2 *	8.9 **	20.2 *	50.7 *	
2	1.5	20.4 *	11.0 **	20.4 *	48.2 *	
3	3.0	26.8 *	5.5 **	26.8 *	40.9 *	
4	5.0	18.3 *	2.0 **	18.3 *	61.4 *	
5	2.0	20.2 *	8.9 **	20.2 *	50.7 *	
6	1.0	14.9 *	12.5 **	14.9 *	57.7 *	
7	4.5	24.2 *	2.5 **	24.2 *	49.1 *	
21	5.0	24.0	2.55	24.3	49.1	
22	4.0	24.2	3.22	24.2	48.4	
23	3.0	30.0	5.78	41.3	22.9	
24	2.0	19.7	8.6	20.4	51.3	
25	3.0	28.2	6.18	33.6	32.0	

*Calculated from the original data as shown in the Sample Calculations.

**Interpolated values from the curve drawn by using the data obtained from runs 21 to 25 (see Fig. 3).

Table T-4. The effect of the weight of the bed on the collection of products at a constant air to phosphorus molal ratio of 500 and ammonia to phosphorus molal ratio of 3.

Run No.	Weight of the bed		Per cent of total products collected by			
	gms.	lbs.	Bed of particles	Conical distributor	Pipe and shield	Filter bag
25	2270	5.0	28.2	6.18	33.6	32.0
27	1362	3.0	29.8	3.34	9.6	57.3
28	3178	7.0	20.5	8.76	34.7	36.0
29	454	1.0	18.9	3.6	2.3	75.2

Table T-5. Results of chemical analysis.

Run : No. :	NH_3/P molal ratio:	Air/P molal ratio:	% Nitrogen:	% P_2O_5	Phosphorus: Condition of sample
1*	2.0	489	12.95	65.94	Poor-sticky, contained pellets used in the column
2*	1.5	489	11.25	64.21	Very poor, very sticky and gummy
3*	3.0	489	13.92	65.34	
4**	5.0	489	14.30	64.21	
5**	2.0	489	14.16	65.44	
6**	1.0	489	13.84	63.32	
7**	4.5	489	14.10	65.44	Contained pellets used in the column
8**	3.0	582	14.28	64.70	Contained pellets used in the column
9**	3.0	400	14.89	65.10	Contained pellets used in the column
10**	3.0	700	14.14	63.71	
11**	3.0	300	14.32	64.60	

*The product sample for this run was obtained from the collection in the conical distributor.

**The product sample for this run was obtained from the collection in the shield.

Table T-6. Per cent conversion of phosphorus data.

	Phosphorus	NH ₃ /P	Air/P	Weight of: Length of: Total	Phosphorus: Conversion
Run:	feed rate,	the bed,	of run,	products	of
No.:	g-atoms/min.:	molal ratio:	gms.:	gms.:	phosphorus
			min.:		
1	0.1126	2.0	489	337*	65.94
2	0.1126	1.5	489	338*	64.21
3	0.1126	3.0	489	636*	65.34
4	0.1126	5.0	489	344*	64.21
5	0.1126	2.0	489	337*	65.44
6	0.1126	1.0	489	94*	63.32
7	0.1126	4.5	489	361*	65.44
8	0.1126	3.0	582	398	64.70
9	0.1126	3.0	400	368	65.10
10	0.1126	3.0	700	567	63.71
11	0.1126	3.0	300	301	64.60
12	0.1126	3.0	450	477	65.00**
13	0.1126	3.0	200	314	65.00**
17	0.1126	1.5	500	209	65.00**
19	0.1126	1.5	500	257	65.00**
21	0.1126	5.0	500	626	65.00**
22	0.1126	4.0	500	558	65.00**
23	0.1126	3.0	500	363	65.00**
24	0.1126	2.0	500	421	65.00**
25	0.1126	3.0	500	404	65.00**
27	0.1126	3.0	500	419	65.00**
28	0.1126	3.0	500	536	65.00**
29	0.1126	3.0	500	217	65.00**

*Calculated from the original data as shown in the Sample Calculations.

**Assumed, product not analysed.

Sample Calculations

Sample Calculations C-1

Calibration of Ammonia Flowmeter

The flowmeter was calibrated with air. The wet test meter was used to measure the volume of air. The data obtained from this calibration is given below.

<u>Temperature</u>	<u>Pressure</u>	<u>Rotameter reading</u>	<u>Flow rate</u>
94°F	736 mm.Hg.	3.0	0.0175 cu.ft.per min.
94	736	6.0	0.0675
94	736	9.0	0.125
94	736	12.0	0.179
94	736	15.0	0.238

The above data was used to calculate the flow rates of ammonia. The method given by McCabe and Smith (15), pp. 119 was used in these calculations. This method is shown below.

$$\frac{v_1}{v_2} = \sqrt{\frac{p_1 T_2}{p_2 T_1}} \quad (1)$$

where

v_1 = flow rate of ammonia, cu.ft./min.

p = gage pressure in the ammonia tank, psig.

p_1 = pressure of ammonia = 14.7 + p , psia.

T_1 = temperature of ammonia, °R.

v_2 = flow rate of air, cu.ft./min.

p_2 = pressure of air, psia.

T_2 = Temperature of air, °R.

From eqn. (1) the volumetric flow rate of ammonia was calculated, and from this the mass rate of flow of ammonia was calculated using:

$$w = v_1 A \quad (2)$$

where

w = mass rate of flow, gms/min.

and A = density conversion factor for ammonia, gms/cu.ft.

The calibration curves for various values of gage pressure in the ammonia tank are given in Fig. 8.

Sample Calculations C-2

Calibration of Air Flow Nozzle

For the type of nozzle arrangement shown in Plate IV, the following method is given by the "Compressed Air Handbook" (8, pp. 279) to calculate the volumetric flow rate of air.

The capacity is given by:

$$q_1 = \frac{(31.5) C D_n^2 Y' \sqrt{Y_n \Delta p}}{V_1} \quad (\text{Eqn. 10, pp. 291})$$

where

q_1 = capacity, volume rate of flow at inlet conditions
(pressure p_1 and temperature T_1), cu.ft./min.

D_n = nozzle throat diameter, in.

C = flow coefficient obtainable from Table 1, pp. 258.

p_1 = total pressure at inlet, psia.

T_1 = absolute temperature at inlet, °F.

γ_n = specific weight of gas, upstream side of nozzle,
lbs/cu.ft.

Δp = differential pressure across the nozzle, psi.

$$Y' = \frac{\left[\frac{k}{k-1} \left(\frac{P_{2n}}{P_{1n}} \right)^{2/k} \frac{1 - \left(\frac{P_{2n}}{P_{1n}} \right)^{\frac{k-1}{k}}}{1 - \left(\frac{P_{2n}}{P_{1n}} \right)} \right]^{1/2}}{\left[1 - \left(\frac{D_n}{D_1} \right) \left(\frac{P_{2n}}{P_{1n}} \right)^{2/k} \right]^{1/2}}$$

The values of
 Y' are given
in Table VI,
pp. 297.

P_{2n} = static pressure, downstream side of the nozzle, psi.

P_{1n} = static pressure, upstream side of the nozzle, psi.

For the nozzle used in this study

$$p = (.491) (h), \text{ psi.}$$

where h = pressure drop across the nozzle, in.Hg.

$$D_n = 0.5 \text{ in.}$$

$$P_{2n} = 14.7 + p$$

where p = pressure gage reading at the downstream end of the nozzle, psig.

$$P_{1n} = P_{2n} + p.$$

$$T_1 = 520.^{\circ}\text{R.}$$

and $D_1 = 3.068 \text{ in.}$

The calibration curves for the air flow nozzle at various values
of pressure gage reading, p , are given in Fig. 9.

Sample Calculations C-3

Calculation of Data from Runs 1 to 7 for Per cent Collection
in the Bed of Particles and the Pipe and Shield

The following method was used in these calculations.

Let a = the collection in the bed, gms.

b = the collection in the conical distributor, gms.

c = the collection in the pipe and shield, gms.

B = the collection in the filter bag, gms.

and $A = a + b$

$a+b+c+B$ = the total collection of products, gms.

a^0 = the per cent of total products collected in the
bed.

b^0 = the per cent of total products collected in the
conical distributor.

c^0 = the per cent of total products collected in the
pipe and shield.

In runs 1 to 7 only 'A' and 'B' were measured and no data on 'c' was taken. Moreover, in these runs 'a' and 'b' were not measured. In runs 21 to 25 'a', 'b', 'c', and 'B' were all measured separately. From runs 21 to 25 it was observed that

$$a^0 = c^0 \text{ or } a = c.$$

Since the operating conditions in runs 1 to 7 and in runs 21 to 25 were almost similar, it was assumed that the above relationship was valid for runs 1 to 7. From the data obtained in runs 21 to 25 a curve was drawn

to relate ' b^0 ' with the ammonia to phosphorus molal ratio and the values of ' b^0 ', for the operating conditions of runs 1 to 7, were interpolated from this curve.

Sample calculations for run number 1.

Ammonia to phosphorus molal ratio = 2.0

$$A = 98 \text{ gms.}$$

$$B = 171 \text{ gms.}$$

$$A + B = 269 \text{ gms.}$$

since, $b \ll a$ assume $b = 0.0$

therefore, $a + b = c = 98 \text{ gms.}$

and $a + b + c + B = 367 \text{ gms.}$

from Fig. 3 $b^0 = 9.0\%$

therefore, $b = 33 \text{ gms.}$

and $a = c = 98 - 33 = 65 \text{ gms.}$

now $a + b + c + B = 334 \text{ gms.}$

therefore, $b = (.9)(334) = 30 \text{ gms.}$

$a = 98 - 30 = 68 \text{ gms.}$

and $c = a = 68 \text{ gms.}$

$a + b + c + B = 68 + 30 + 68 + 171 = 337 \text{ gms.}$

from this data $a^0 = 20.2\%$

$b^0 = 8.9\%$

and $c^0 = 20.2\%$

Sample Calculations C-4

Calculation of Per cent Conversion of the Phosphorus

Per cent conversion of the phosphorus

$$= (100) \frac{\text{wt. of phosphorus recovered in products}}{\text{wt. of phosphorus fed to the system}}$$

$$\text{or,} = (100) \frac{\text{amount of } P_2O_5 \text{ in the products}}{\text{theoretical amount of } P_2O_5}$$

amount of phosphorus pentoxide in the products

$$= (\text{total wt. of the products collected}) \times (\text{P}_2\text{O}_5 \text{ content of the product})$$

Theoretical amount of phosphorus pentoxide

$$= \frac{W \rho t (\text{Molecular wt. of } P_2O_5)}{2 (\text{Atomic wt. of phosphorus})}$$

where

W = water flow rate, c.c./min.

 ρ = density of liquid phosphorus, gms/c.c.

t = length of run, min.

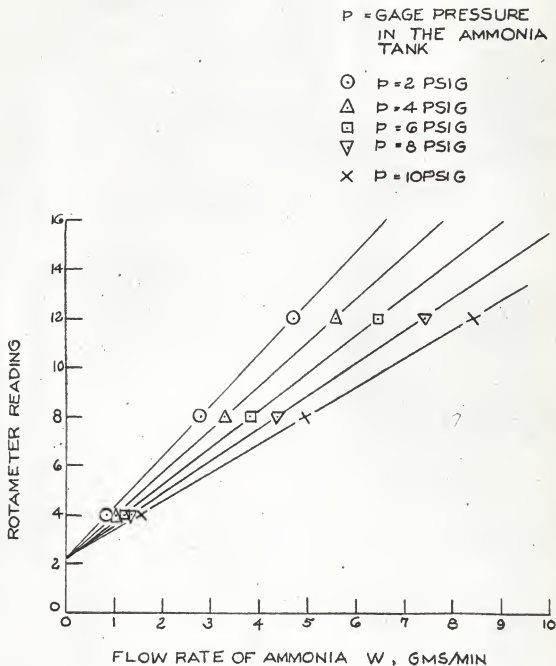


FIGURE 8. CALIBRATION CURVE FOR THE AMMONIA ROTAMETER. THIS CURVE WAS CALCULATED FROM EXPERIMENTAL DATA ON AIR. SEE CALCULATIONS FOR AMMONIA ROTAMETER CALIBRATION.

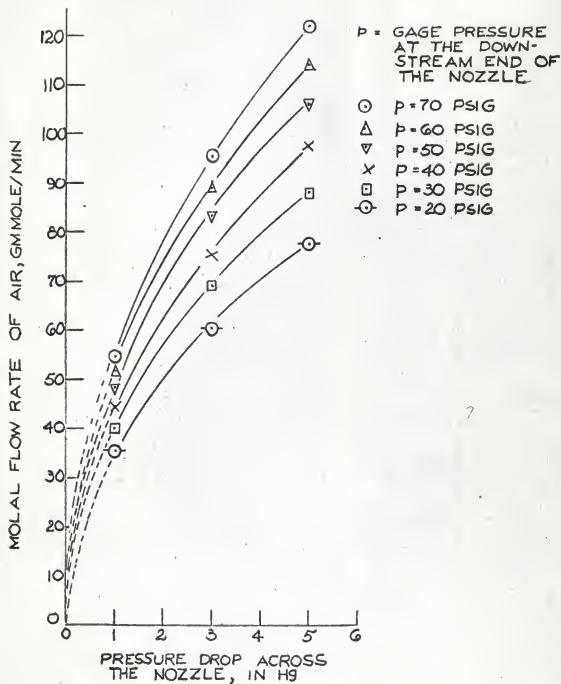


FIGURE 9. CALIBRATION CURVE FOR THE AIR FLOW NOZZLE. THIS CURVE WAS CALCULATED FROM "COMPRESSED AIR HANDBOOK".

- ◊ TEMPERATURE MEASURED AT T_1
(SEE PLATE I)
- TEMPERATURE MEASURED AT T_2
(SEE PLATE I)
- ◉ TEMPERATURE MEASURED AT T_3
(SEE PLATE I)

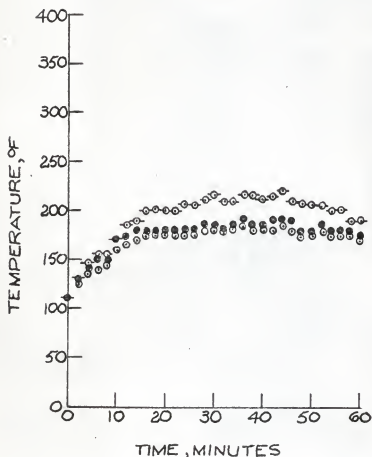


FIGURE 10. TYPICAL TEMPERATURE PROFILES (RUN NO. 10) REPRODUCED FROM THE BROWN TEMPERATURE RECORDER CHART.

Sample Data Sheet

Run # _____ Date: _____ Operator: _____
 NH₃:P Molal ratio _____ approximately.
 Air:P Molal ratio _____ approximately.
 Time: Initial _____, Final _____. Length of the run: _____ Min.

Time, Min. 2 4 6 8 10 12 14 16 18 20 22 24 26 28 30

Water flow
rate, cc/Min.

Time, Min. 32 34 36 38 40 42 44 46 48 50 52 54 56 58 60

Water flow
rate, cc/Min.

Time, Min. 0 5 10 15 20 25 30 35 40 45 50 55 60

Pressure in
the reactor, in.Hg

Air flow rate: Manometer reading _____ in. Hg
 Pressure gage reading _____ psi

Ammonia flow rate: Rotameter reading _____
 Pressure gage reading _____ psi

Weight of the particles + bucket _____ gms.
 Weight of the bucket _____ gms.
 Weight of the particles _____ gms.
 Weight of the particles + product + bucket _____ gms.
 Weight of the particles + bucket _____ gms.
 Weight of the product collected in the bed _____ gms.
 Weight of the product + conical distributor _____ gms.
 Weight of the conical distributor _____ gms.
 Weight of the product collected on the conical distributor _____ gms.
 Weight of the pipe + shield + products _____ gms.
 Weight of the pipe + shield _____ gms.
 Weight of the product collected on the pipe and the shield _____ gms.
 Weight of the product + filter bag _____ gms.
 Weight of the filter bag _____ gms.
 Weight of product collected on the filter bag _____ gms.
 Total products collected _____ gms.

% collection in the bed:

% collection on the conical distributor: % collection in the pipe and shield:

THE COLLECTION OF PRODUCTS FROM THE VAPOR-PHASE REACTION
OF PHOSPHORUS, AIR, AND AMMONIA

by

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AN ABSTRACT OF A MASTER'S THESIS

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requirements for the degree

MASTER OF SCIENCE

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1964

The purpose of this work was to study the collection of the phosphatic products obtained from the vapor-phase reaction of phosphorus, air, and ammonia. The collection was accomplished in a "fluidized" bed.

Molten yellow phosphorus was fed to a stainless steel, tank-type reactor, and was burned in a stream of dry air; the products of this combustion were then reacted with gaseous ammonia. A large excess of air was used to accomplish rapid cooling of the products. The products were carried out of the reactor in a stream of excess air and ammonia, and were then collected.

The filter bag was considered as a 100 per cent efficient collector and was used in series downstream from the bed of particles to obtain data on the collection of products. Ammonium phosphate (8-10 mesh) fertilizer pellets were employed as nuclei for collection in the bed.

Experimental investigations were conducted to study the effect of (1) the air to phosphorus molal ratio, (2) the ammonia to phosphorus molal ratio and (3) the weight of the bed on the efficiency of collection.

It was found that the air to phosphorus molal ratio was the main factor affecting collection; efficiency of collection increased with decreasing air to phosphorus molal ratios. It was also observed that the ammonia to phosphorus molal ratios and the weight of the bed had a small effect on the efficiency of collection; maximum efficiencies were observed with ammonia to phosphorus ratios of 3 to 4, and weight of the bed of 3 to 4 pounds.

The product of reaction was white, flour-like material having a low bulk density. The products had an average phosphorus pentoxide

content of about 65 per cent and the total nitrogen content ranged from 11 to 15 per cent. It was found that the product composition was unaffected by air to phosphorus or ammonia to phosphorus molal ratios.

In most of the runs only 60 per cent of the phosphorus fed to the reactor was recovered in the products. The phosphorus losses in the system were mainly attributed to the conversion of yellow phosphorus to the more stable red variety. However, an increasing trend in the per cent conversion of phosphorus was observed with increasing air to phosphorus and ammonia to phosphorus ratios.