PILOT PLANT PRODUCTION OF TRIPLE SUPERPHOSPHATE USING A CENTRIFUGAL-PUMP-TYPE MIXER

By

James E. Gantt

A thesis submitted in partial fulfillment of the requirements for the degree of Bachelor of Science in Chemical Engineering

The University of Tennessee

August 1938

CONTENTS

	page
INTRODUCTION	1
GENERAL SURVEY OF THE FIELD	4
STATEMENT OF PURPOSE	8
GENERAL THEORY	10
APPARATUS	14
Description	14
Calibration	20
EXPERIMENTAL RUNS	23
Experimental Runs 1 and 2	23
Experimental Run 3	24
Experimental Run 4	27
Experimental Run 5	29
ANALYTICAL METHODS	32
DISCUSSION OF RESULTS	37
CONCLUSIONS	47
BIBLIOGRAPHY	49
APPENDIX	50

PILOT PLANT PRODUCTION OF TRIPLE SUPERPHOSPHATE USING CENTRIFUGAL-PUMP-TYPE MIXER

INTRODUCTION

The agronomical importance of the phosphatic fertilizers is a matter of common knowledge. There are abundant supplies of natural phosphates and phosphatic materials, but Nature has not seen fit to provide these supplies in a form immediately available for plant food. Hence, they must be processed before they attain their maximum value for agricultural use.

The most complete reference available at the present time on the chemistry and manufacture of phosphates and phosphorous compounds[1] was published in 1927. Although adequate then, it has now become quite antiquated due to rapid advances in recent years along these lines.

Thus the only source of information concerning the present state of the art is in the technical literature of the past few years.

Several factors are evident from these sources:

1. The art is ahead of the science.
2. The science is ahead of itself.

By the latter observation it is meant that the more theoretical aspects, although naturally not entirely worked

---

1. Waggaman, W.H., and Easterwood, Henry W., Phosphoric Acid, Phosphates, and Phosphatic Fertilizers, New York, The Chemical Catalog Co., Inc. (1927)

out, have been given considerable attention, both in the United States and abroad, especially in Germany. The system, $CaO - P_2O_5 - H_2O$, has been quite thoroughly investigated.[2] Accurate methods for the various essential determinations have been worked out for the ideal cases using pure chemicals. But, unfortunately, for the study of actual superphosphates these theoretical principles and analytical procedures are found to be pitifully inadequate, due to the effects of the impurities necessarily present in all commercial phosphatic fertilizers.

The problem, then, is to attempt in some logical manner to close the gap between the art and the science.

This necessarily involves a study of actual fertilizer mixtures, and such a study is fully capable of presenting some quite troublesome problems, some of which are brought out in the following pages.

With superphosphate production running into eight figure tonnages per year and affecting such a vital cross-section of the population, any research which might benefit the industry, either by increased economies or quality, certainly has ample justification.

This study was carried out as part of the research program of

The Tennessee Valley Authority
Chemical Engineering Department
The Chemical Engineering Laboratory

The project was entitled W.O. 162-New Fertilizer Combinations

2. Bassett, H.J., Z.Anorg.Chem. 59, 1-55,(1908)

and Equipment.

At this point it is desired to gratefully acknowledge the cooperation of the Estabrook Research and Laboratory Staff and the provision of equipment and materials by The Tennessee Valley Authority.

GENERAL SURVEY OF THE FIELD

The process for the manufacture of water soluble phosphates by acidulation of natural phosphatic materials had been known and practiced since the middle of the nineteenth century.

The first methods consisted of the sulfuric acidulation of bones, and later this treatment was extended to other phosphatic materials[1]. The first product tended to be somewhat irregular in composition and quite often in rather poor mechanical condition.

A century of experience in superphosphate manufacture has resulted in more economical and efficient methods.[2] However, while the mechanics of the process have apparently been pretty thoroughly worked out, the application of chemical principles has fallen far behind. As superphosphate has been manufactured for a century it would naturally seem that all the chemical reactions which occur in the process are thoroughly understood. The technical literature, however, does not show this to be true, particularly as regards the chemical changes undergone by the iron, aluminum, and other rock impurities. These changes are of special interest because they appear to exert a large effect on both the chemical and physical properties of the product and must account for the large deviations from the theoretical as worked out with pure chemicals.

---

1. Waggaman and Easterwood, Loc. Cit., p. 138
2. Jacob, K.D., Proc. Natl. Fert. Assoc., p. 55, June, 1929.

Until quite recently practically all the water soluble superphosphates on the market were made by the action of sulfuric acid on phosphatic materials, in the case of phosphate rock or apatite giving a product carrying from 16% to 20% available P_2O_5 as determined by standard citrate digestions.

As recent advances in volatilization methods have made possible the large scale economical production of phosphoric acids of any desired concentrations, it has become feasible to produce more concentrated superphosphates with a P_2O_5 availability of 45% to 50% in the case of the triple superphosphate to over 60% for the new metaphosphates.

Thus the trends in the phosphatic fertilizer industry point toward more concentrated sources of available phosphorous compounds.

The present study concerns the construction and test of a semi-continuous superphosphate mixing unit, using a high-speed, centrifugal-pump-type mixer for mixing phosphoric acid and finely ground rock phosphate to obtain triple-superphosphate*.

It was early observed in the manufacture of triple-superphosphate that the initial mixture is quite fluid, remaining so for a period depending on several factors[1], and then thickens, going through a plastic stage, and finally reaching the state of a hard solid.

1/ Copson, R.L., Newton, R.H., and Lindsay, J.D., Ind. Eng. Chem. 29, p. 175-179 (1937)

* The terms "double superphosphate" and "triple superphosphate" seem to be interchangeable in present usage, as applied to the product resulting from the acidulation of rock phosphate with phosphoric acid.
"Triple superphosphate" will be used in this discussion.

It was further determined that after the plastic stage had begun the gain in the quality of the product by further mixing did not justify the necessarily heavy machinery and large power consumption for kneading the tough, hardening mass.

Thus, as far back as September, 1935, an "ideal" process for superphosphate manufacture was outlined[1].

The process, due to the general advantages of such methods, would necessarily be continuous.

The logical equipment for such a process is outlined as follows:

1. Continuous high-speed mixer.
2. Slow speed conveyor, receiving the still fluid mixture and carrying it until the crumbling stage is reached.
3. A simple preliminary disintegrator--such as an inclined plate or tumbling barrel.
4. A slow conveyor to the storage heap-allowing time for set in transit thus preventing setting of pile as a whole.

The last three phases of the operation apparently would present no insurmountable difficulties, but the development of a simple, economical, dependable, and efficient high-speed mixer has presented a number of exceedingly troublesome problems.

---

1. Curtis, H.A., Chem. Met. Eng. 42, 488-489 (1935)

Considerable work has been done by the T. V. A. and others on the development of a continuous rapid mixer.[1] One type was tried in which the rock was fed by a horizontal screw into a two stage centrifugal pump. Good mixing was obtained but the acid concentration was limited to 70% H_3PO_4, and power requirements were excessive. Of a number of types of mixers tried, the most satisfactory was one in which two sets of blades travelled in a hypocycloidal path around a set of fixed blades. The mixing was satisfactory, but sufficiently strong construction for plant operation would be quite expensive.

---

1. Continuous Mixing of Superphosphate in Fluid Form-, W.O. 172, Report No. 4. June, 1936. (T. V. A. Fertilizer Works Report)

STATEMENT OF PURPOSE

In carrying out this work several purposes were in view.

1. To develop a high speed mixer for superphosphate making preliminary tests and adjustments.

2. To test this mixer under a variety of working conditions in order to determine the feasibility of its commercial use.

(This study is briefly outlined as follows:)

a. Variation of acid-rock ratio at constant rock feed.

(1) Determination of approximate optimum conditions.

b. Variation of feed rates, acid-rock ratio remaining constant.

(1). Determination of capacity of mixer.

c. Continuity of operation.

(1) Operation at the optimum conditions determined above as long as possible, or feasible.

3. To study the performance of the mixer and properties of the product obtained using low acid-rock ratios and low acid concentrations, and to note the effect of heating the acid and mixture.

a. Mixing in approximately the proportion corresponding to the product $CaHPO_4$, dicalcium phosphate.

(1) Study of effect of heating materials before mixing and the mixture after mixing.

(2) Observation of properties of samples treated in the usual manner.

4. To make a study of the effect of acid-rock ratio on the physical and chemical properties of the product on the working range (70%-80%) of acid concentrations, and to note the effect of the addition of penetrants to the acid.

a. To utilize the range and flexibility of the mixing unit in obtaining a variety of superphosphate samples for the following observations:

(1) Setting times

(2) Setting temperatures

(3) Maximum temperatures reached

(4) Physical properties

(5) Periodic analyses for kinetic study

(6) Final (30 day) analyses and stoichiometric treatment of results.

b. Determination of optimum conditions from economic considerations.

The primary intention of this investigation was to make a thorough test of the mixing unit under a wide variety of mixing conditions to ascertain the feasibility of its commercial use. Obviously, however, the data so obtained lend themselves to correlation of physical and chemical properties as well as an investigation of certain economic factors which can not be entirely overlooked in such a test.

GENERAL THEORY

In outlining the general theory involved in these investigations it is found rather difficult to draw a sharp line of demarcation between fact and opinion. On the other hand, the reacting system is so affected by the presence of various impurities as to render the relations derived from purely theoretical considerations of but little value in correlating the actual results.

The relations are especially obscure in regard to the kinetics of the actual reactions undergone by the iron and aluminum compounds and the peculiar behavior of the mixture on aging.

The process of making superphosphate involves ideally the following reactions, assuming the active components of the rock to be as noted.

Major reaction:

$$3Ca_3(PO_4)_2 \cdot CaF_2 + 14H_3PO_4 + 10H_2O = 10((Ca(H_2PO_4)_2 \cdot H_2O)) + H_2F_2 \qquad (1)$$

Minor reactions:

$$Fe_2O_3 + 2H_3PO_4 = 2(FePO_4) + 3H_2O \qquad (2)$$

$$Al_2O_3 + 2H_3PO_4 = 2(AlPO_4) + 3H_2O \qquad (3)$$

$$CaCO_3 + H_3PO_4 + H_2O = CaHPO_4 + 2H_2O + CO_2 \qquad (4)$$

$$CaCO_3 + 2H_3PO_4 = Ca(H_2PO_4)_2 + CO_2 \qquad (5)$$

$$CaF_2 + 2H_2O + H_3PO_4 = CaHPO_4 \cdot 2H_2O + H_2F_2 \quad (6)$$

$$CaF_2 + H_2O + 2H_3PO_4 = Ca(H_2PO_4)_2 \cdot H_2O + H_2F_2 \quad (7)$$

$$Ca_3(PO_4)_2 + 6H_2O + H_3PO_4 = 3(CaHPO_4 \cdot 2H_2O) \quad (8)$$

The reactions indicated by (4), (6), and (8), although apparently not impossible, are not likely to take place under the conditions imposed in these preparations.[1,2]

The principal reaction is exothermic, but the reaction as a whole does not lend itself conveniently to thermochemical or thermodynamical treatment.

The equilibrium diagram, system $CaO-P_2O_5-H_2O$, indicates that the stable form under the conditions existing in the fluid stage is monocalcium phosphate. However in the semi-fluid, plastic and solid stages the difficulty of ascertaining equilibrium conditions is obvious.

It must not be overlooked that, under the conditions used, the system in the fluid stage did not at any time attain stable equilibrium; hence for quantitative use the equilibrium data for the liquid state, as available, are of little value in this case. They do, however, furnish certain limiting conditions.

As noted above the reaction between phosphoric acid and rock phosphate takes place in three stages: a fluid stage, a plastic stage, and a solid stage.

---

1. Copson and Newton, Ind. Eng. Chem. 28, Loc. Cit. p.1182
2. Bassett, Loc. Cit.

It is generally conceded that the setting and hardening of superphosphate (both ordinary and triple) is due to the hydration of monocalcium phosphate. Monocalcium phosphate monohydrate is unstable above 100°C.; thus it should be possible to ascertain quite easily by observing the temperature on setting whether the setting is due to hydration or evaporation of the water.

It was found[1] that with varying acid concentration the length of the fluid stage was a minimum for about 80% H_3PO_4. Its length was found to decrease with increasing range of particle sizes, increasing temperature and increasing fineness of rock sizes. These factors, along with preliminary solution of some rock phosphate in the acid, offer some methods of shortening the fluid stage should such a necessity arise; that is, in a spray tower or similar process.

A thorough study has been made of the effect of variations in acid-rock ratio, acid concentration, and rock quality, using a batch-type mixing unit.[2] Due to the shorter mixing time and almost instantaneous homogeneity obtained with the present mixer, that study and the present investigation (Run 5.) can be considered as having been made under quite different conditions.

1. Copson, Newton, and Lindsay, Ind. Eng. Chem. 29. Loc. cit. p. 175 (1937)
2. Copson and Newton, Ind. Eng. Chem. 28., Loc. cit., p. 1182

It was found in this study, among other things, that most of the iron and aluminum compounds present in the original rock react to an available form; that little dicalcium phosphate is formed, and that the citrate insoluble residue is almost entirely unreacted apatite. No explanation is offered, however, as to the mechanism of reversion. (The term "reversion" in this study is applied to the increase in citrate insoluble P_2O_5 observed during the aging of superphosphates.)

The above findings, along with other evidence noted in these investigations, indicate that it is reasonably accurate for correlation purposes to assume that all the citrate insoluble P_2O_5 originated in the rock.

APPARATUS

Description:

Rock Feed: Two rock feed systems, with variations, were used. The first consisted of a small conical hopper of approximately 1/4 cubic foot capacity with a 2-inch diameter screw (constructed from a 2-inch wood auger and a 2-inch standard iron pipe) that emptied rock from the bottom of the hopper into the mixer chute. The screw was arranged with a cone-pulley, V-belt drive for operation at several set speeds. The hopper was equipped with a rotating set of scrapers for agitation.

The second feed-system consisted of a W.J. Savage, screw-conveyor feeder with a 4-inch diameter screw(Savage-Gauntt type 2B, size No. 430). It was equipped with a specially made hopper of 13.5 cubic feet capacity. This hopper was built with two vertical sides in an attempt to prevent bridging over of the rock. A short vertical chute leading from the hopper to the feeder was equipped with two rotary agitators. The variable-rate, ratchet drive with which the feeder came equipped was removed and replaced with spur gears, the feed variation being regulated by a variable-speed drive (New Departure "Transitorq" variable-speed unit). The speed was reduced for operation of the screw over the range from 5.25 to 18.9 revolutions per minute.

A tachometer was connected to the drive for use in calibration to avoid fluctuations resulting from voltage and frequency variations in the electric supply. The "Transitorq" unit was equipped with a speed indicator which could be used under normal conditions.

In the last run, as the rock feed rate was to be held constant, the 1/4 HP. motor "Transitorq" unit was replaced by a 1/2 HP. motor driving the feeder at a constant speed. A voltage regulator was placed in the electric supply line to insure absolutely constant voltage.

Tanks, Piping, and Pumps: To resist corrosion by phosphoric acid of the concentrations used it was decided to use a lead lined tank and brass piping, pumps and orifice accessories. A brass strainer was installed in the acid line ahead of the pumps. Wooden barrels were used for storage of the acid.

Two pumps were used. The first was a Viking, Model ECH2, of 2/3 gpm. rated capacity, and of brass except for a steel rotor. It was used in connection with the smaller rock feeder noted above, The second pump was a Micro-Westco, turbine type pump, model SR4K8, of brass construction (except stainless steel shaft). It was used with the larger rock feeder noted above.

Orifice Meters: Two meters were used. The first meter consisted of a square-edged, brass orifice plate in a 3/8 inch standard brass pipe line. Flange taps were used, and the meter mounted in a vertical position. This meter was used in connection with the Viking pump noted above. The second consisted of a square-edged orifice in a $1\frac{1}{2}$ inch heavy brass pipe. Flange taps were used, and the orifice meter was mounted in a vertical position. This meter was used with the Micro-Westco Pump noted above.

Manometers: Mercury manometers with a range of 100 cm. were used with both orifice meters.

Three materials of construction were used, brass, glass, and steel. The problem was to design the manometer so as to prevent contact between brass and mercury and acid and steel. This proved quite difficult in practice. Two methods were used to eliminate the contact between acid and steel (which resulted in evolution of gas and inaccurate indications). An all glass U-tube was tried, but due to the frequent drainage necessary it was found quite inconvenient. The original steel reservoir base was acid-proofed with rubber-base paint and this appeared to prevent acid action, at the same time maintaining the original convenience.

As the action of mercury on the brass header, although quite destructive, was not so rapid it was thought better to renew these parts from time to time than to attempt replacement by another material of perhaps doubtful properties.

Acid Heater: In Run 4 it was necessary to heat the acid before mixing. An acid heater was constructed from a 4-inch pipe about 20-inches long containing a copper coil about 18-feet long of 3/8-inch tubing, the ends of the tubing being flanged in brass unions. The heat was supplied by steam entering near the top of the 4-inch pipe, the condensate being removed by a Yarway steam trap. High pressure steam (110 lbs. per square inch gage) was available for heating, and suitable valves were provided for throttling and for pressure release. The piping provided for by-passing the heater, heating only part of the acid, or all, as might be desired. A thermocouple well was installed a short distance farther along the acid line to determine the acid temperature on entering the mixer.

Mixer: A detailed drawing of the mixer is shown on plate 1. The cast iron body and rotor were part of a water pump for a 1926-27 Nash car (Nash Motor Company part No. 9972). The pump was mounted with the shaft in a vertical position and driven at a no-load speed of 3750 rpm.

Two motor sizes were used, a one-third horsepower motor was found sufficient for usual operating conditions, but a one-half horsepower motor was installed for the last runs to handle the stiff mixture at very low acid-rock ratios.

Three different acid inlets were tried, the acid was first introduced by means of a hose directed through the rock chute, the next step was to feed the acid through the mixer head by means of four slanting holes, located as shown by the dotted lines of plate 1. After annular slots proved unsatisfactory, for the acid used, the final arrangement was eight 5/16-inch holes (shown on Plate 1, by solid lines) drilled vertically slightly farther removed from the center than the previous holes.

The first arrangement provided a bearing at the top of the rotor carried by a spider in the rock inlet (shown as broken lines in Plate 1.). This was later removed.

Superphosphate Bin: The mixer discharged into a wooden bin with the inside dimensions of 6-feet by 5-feet by 4.5-feet high.

The bin as a whole was demountable, and one side was arranged for easy removal.

Exhauster: To remove evolved gases a steam exhauster was fitted in a five-inch diameter flue that led from one corner of the bin out a window.

Sample Boxes: Wooden boxes, 12 in. X 12 in. X 4 in. and 8 in. X 8 in. X 4 in. deep were used for receiving samples from the mixer in Runs 3 and 5, respectively. The mixture was left in these boxes throughout the aging period.

Instruments for Taking Setting Times: Three instruments were used for taking setting times. They were small weighted rods:

		dia. end of rod
1.	1.41 oz.	0.112 in.
2.	3.03 oz.	0.112 in.
3.	4.06 oz.	

Needle No. 3 is the standard Gilmore Needle for taking the setting time of concrete. Needles one and three were used in Run 3; needles one and two were used in Run 5.

Calibrations:

Rock Feed: The first feeder was calibrated at several set speeds. The rate was found to vary with the bulk density of the rock. This in turn depended on the amount of aeration that the rock had previously undergone.

The Savage-Gauntt feeder was calibrated against both the tachometer and indicator on the "Transitorq" drive. The ¼ HP. motor allowed a feed range from 4 to 24 lbs. of rock per minute. The limits of error were about 3 percent at the lowest feed rate and about 1 percent at the highest feed rate. In the last run, as noted above, the drive was changed and the speed of the feeder held constant. Three carefully conducted calibrations on different days gave an apparent accuracy of 0.1 percent.

Working on experience with the previous feeder several devices such as scrapers and air agitators were tried in the hopper to prevent bridging and variation of bulk density. However, it was later found, contrary to the results with the first feeder, that the discharge was practically independent of bulk density. This was likely due to the action of the agitators in the chute connecting the hopper and feeder trough. Furthermore only occasional prodding was sufficient to keep the rock flowing freely from the hopper.

The speed of the spiral ring pick-up that removed the rock from the end of the primary screw was found to affect the uniformity of the discharge rate and was adjusted to give as uniform a discharge as possible. In order to obtain an even discharge at extremely low feed rates, however, it was found necessary to admit a small amount of air through a jet into the top of the trough near the outlet.

Acid Feed: The acid manometers were calibrated both with water and with the acid to be used, with good agreement between the calibrations.

The orifice meter used in the first system in connection with the Viking Pump gave a maximum differential of 40 cm. of mercury with a 1 cm. pulsation. The second meter used in connection with the Micre-Westco pump gave a manometer differential of 100 cm. (the manometer capacity) at an acid rate of 32.75 lbs. per min. (79% H_3PO_4-27°C) practically free from pulsation and capable of being held constant within 0.2 cm. over a long period of time.

The latter orifice meter was calibrated using water and 30%, 40%, 50%, 60%, 70%, 75%, 78%, 79% and 80% H_3PO4.

When manometer difficulties were eliminated, the calibrations were consistent within ½%.

Mixing Time: The free mixing space in the mixer used was found by stopping the outlet, filling the mixer to an arbitrary "mixing line" with water and measuring the volume contained. This volume was found to correspond to approximately 1.2 pounds of a typical mixture of rock and acid in the fluid stage.

Therefore, based on the actual output of 53 to 15 pounds per minute the "average" mixing time is found to vary from 1.3 to 4.8 seconds.

EXPERIMENTAL RUNS

Preliminary Runs, Runs 1, and 2.:

Purpose: The purpose of these runs was to make preliminary tests and adjustments.

Apparatus: The small rock feeder and the Viking pump noted above were used with the centrifugal mixer.

Materials: The rock of batch No. 1 and 78% H_3PO_4 from The Victor Chemical Co. were used.

Rock-acid Ratio: Acid was admitted in 95% the theoretical requirement for conversion to $Ca(H_2PO_4)_2$, $FePO_4$, and $AlPO_4$.

Acid Inlets: In Run No. 1 the acid was introduced by means of a hose directed into the rock inlet. In Run 2 the acid was introduced through the four 1/8 inch holes mentioned in the above description (Shown by dotted lines on Plate 1.)

Operation and observations:

Run 1: This run was continued for 20 minutes until the acid in the feed tank was depleted.

Run 2: The mixer was operated for a period of approximately one and one half hours at a total feed rate of 13.8 pounds per minute. The termination of the run was due to the depletion of the acid supply.

Results and conclusions:

As the purpose of these runs was essentially to make a preliminary investigation of the mixer operation, extensive data were not taken.

A sample was taken from the bin after Run 2. The analyses and calculations are included in Table V.

The physical operation gave evidence of a larger capacity than had been anticipated, and the next step seemed to be the provision of larger and more accurate rock and acid-feed systems.

Experimental Run 3.:

Purpose: The purpose of this run, using more finely ground rock than that of Batch 1, was to make a test of the mixing unit under a variety of controlled working conditions to determine the feasibility of its commercial use.

Apparatus: The larger rock and acid feed systems were used in this study.

Materials: The rock of Batch 2 and 80% H_3PO_4 from Wilson Dam were used.

Acid Inlets: The acid inlet consisting of four 1/8 inch holes, as used above, proved unsatisfactory in handling higher acid rates, due to splashing. To remedy this situation several types of acid inlets were tried. An annular slit worked well with water, but the slit was necessarily so

narrow, in order to give sufficient downward velocity, that it was plugged by the acid impurities. The final arrangement consisted of eight 5/64 inch holes as shown on Plate 1. This appeared to operate quite satisfactorily with but a small amount of splashing. At this time the top bearing spider was removed to decrease the danger of plugging of the rock inlet.

Operation and Observations:

For an outline of the runs made and the variables studied see Table III.

Eight tests were made studying the effect of varying acid-rock ratio and varying capacity. The acid and rock feeds were set on the desired values; a short time was allowed for constant conditions to be obtained, and some of the product was caught in a shallow wooden box. As soon as the sample was obtained the feed rates were changed as required and the procedure repeated.

An attempt was made to determine the maximum capacity of the mixer. This was not reached because at the capacity of the rock and acid feeders, a combined feed of 1.58 tons per hour, the mixer gave no indications of failure. About thirty minutes were required for these tests.

After taking eight test samples the feed was set on a low rock rate, 6.7 pounds per minute, using 90% of the theoretically required acid. The mixer was operated in this

continuity of operation study until the acid supply was depleted, or for two hours, making a total of two and one half hours of continuous operation.

Setting times were obtained by starting a stop clock at the instant the sample box was withdrawn and noting the time at which the weighted needles, described above, could first be placed on the material without making a noticeable impression.

The power consumption was obtained from periodic wattmeter readings.

Results and conclusions:

A detailed account of the mixer operation and properties of the product will be given with the Discussion of Results; in Tables III and IV and Figures 1 and 2.

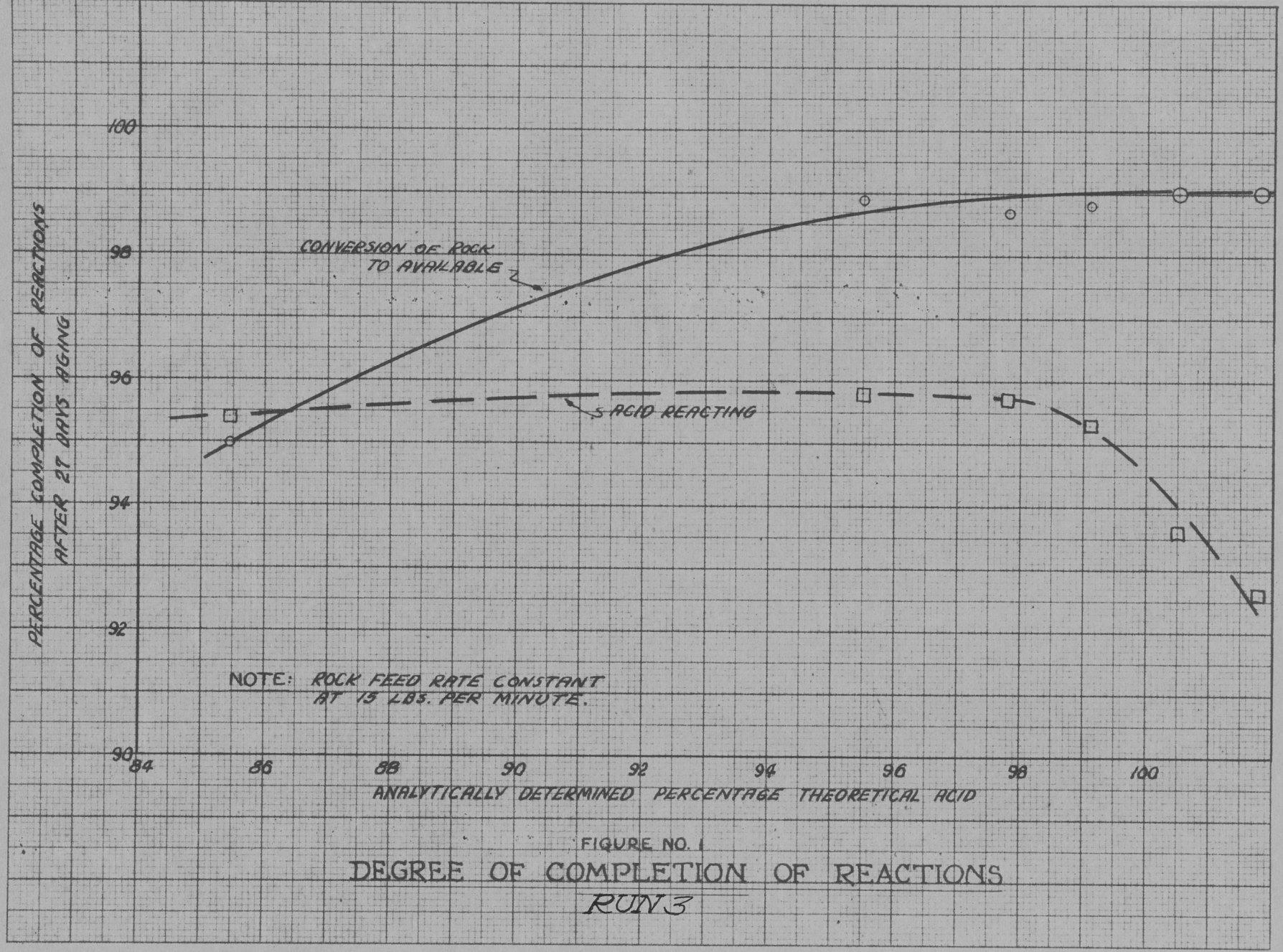


FIGURE NO. 1

DEGREE OF COMPLETION OF REACTIONS

RUN 3

FIG. NO. 1

KEUFFEL & ESSER CO. N.Y.

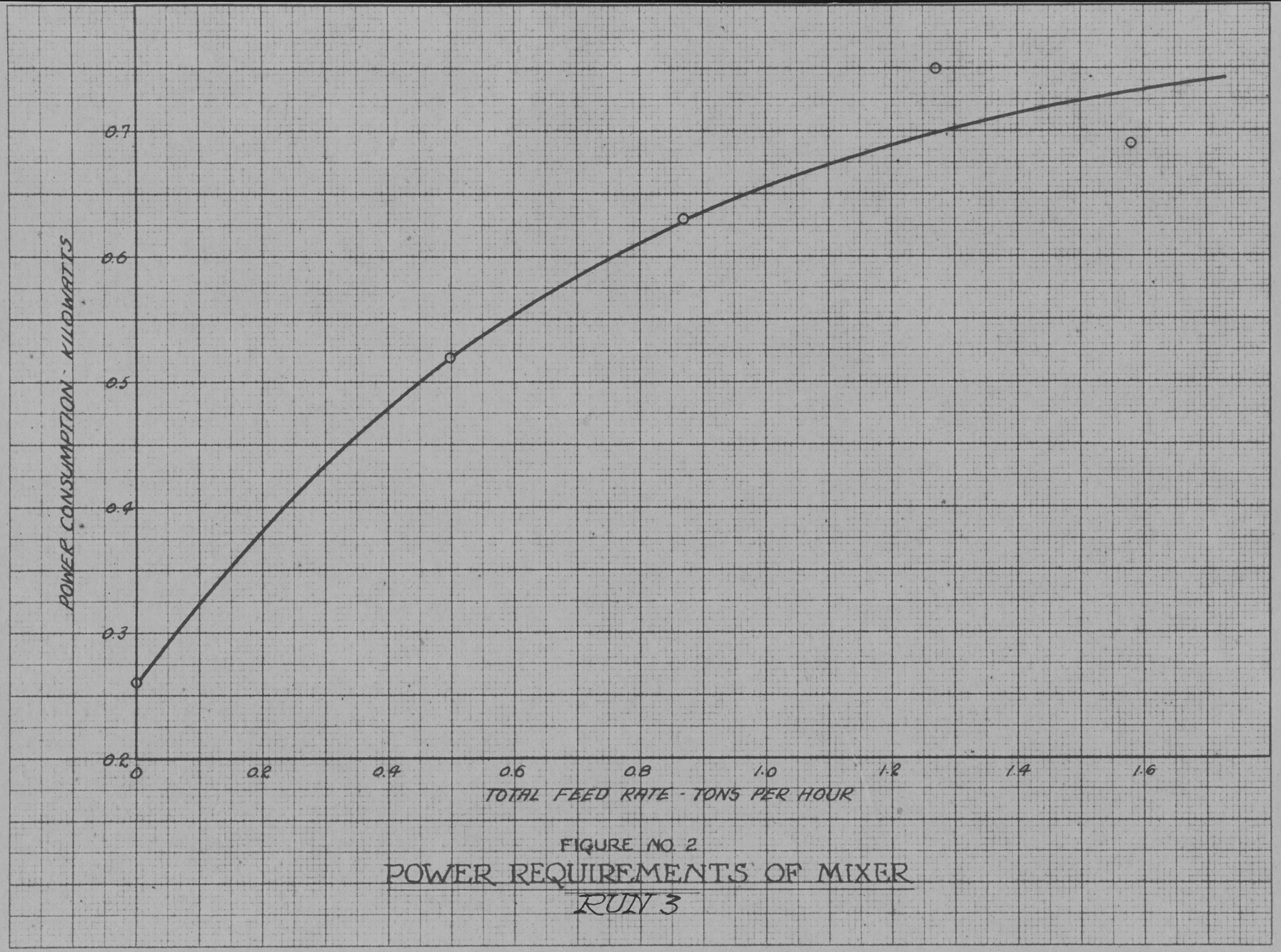


FIGURE NO. 2

POWER REQUIREMENTS OF MIXER

RUN 3

FIG. NO. 2

KEUFFEL & ESSER CO. N.Y.

Experimental Run No. 4. :

Purpose: As the results of Run 3. considered elsewhere, indicated good conversion at low acid rock ratios, it was recommended that the effect of larger acid deficiencies be studied. At the same time it was thought worthwhile to note the effect of heating the materials before mixing and the mixture immediately after mixing.

Apparatus: Essentially the same apparatus was used as in Run 3 with the addition of the acid heater noted above.

Materials: In order to obtain a sufficiently fluid mixture at the high acid deficiencies, acid concentrations of 40% and 60% H_3PO_4 were used. The rock of Batch No. 2 was used.

Rock-acid Ratio: The rock feed was constant at 15 lb. per minute and the acid feed was regulated to give an acid-rock ratio slightly higher than that stoichiometrically required for the conversion of the calcium content of the rock to dicalcium phosphate, $CaHPO_4 \cdot 2H_2O$.

Operations and observations:

The feed rates were set on the desired values and after time was allowed for constant conditions to be reached a sample was taken. This procedure was followed using first acid at room temperature and then making a similar run heating the acid.

Samples run with cold 40% H_3PO_4 were still a gray fluid after several weeks. Crystals were noticed separating on the surfaces of these mixtures. Complete water solubility indicated that these crystals were essentially monocalcium phosphate.

Samples were taken from the mixer in casseroles and placed in an oven at 125°C to dry.

The mixture made with 60% acid at room temperature set comparatively rapidly to a brown, grainy mud. Samples of this mixture were placed immediately in an oven and dried at 125°C., the remainder being set aside to air-dry.

Another set of runs were made heating the acid. The 40% acid was heated to 100°C. and the 60% acid was heated to 116°C. The product with 40% H_3PO_4 was brown in color, a crust forming on the surface immediately, and set slowly throughout to a stiff paste. The product with the 60% acid was gray in color, had considerable froth on the surface and the mass set to a pasty solid after several hours. Samples of these mixtures were placed in an oven at 125°C and the remainder set aside to air-dry.

Results and conclusions:

The analyses of both the oven-dried and the air-dried samples are given in Table VIII., as well as a stoichiometric treatment of the results.

Run 5:

Purposes: 1. To study the effect of acid-rock ratio and acid concentration within the working range.

2. To study the effect of the addition of wetting agents.

Apparatus: Essentially the same apparatus was used as in Run 3, with additional piping to facilitate calibrations.

Materials: A new shipment of 80.3% and 79.3% H_3PO_4 (Sp. g. Analyses) from Wilson Dam was used with the rock of Batch No. 2.

Operation and observations:

The rock rate was held constant at 13.86 pounds per minute.

The procedure was essentially the same as in Run 3, with slightly more time allowed for constant conditions to be reached.

Using 79% H_3PO_4 the acid proportion was varied from 105% to 64% theoretical, based on conversion to $Ca(H_2PO_4)_2$, $FePO_4$, and $AlPO_4$. Four samples were taken in this range. Using 75% H_3PO_4 the proportion was varied from 98% to 50% of the theoretical, six samples being taken in this range. Using 70% H_3PO_4 (by sp. g. analysis) the proportion was varied from 100% to 56% theoretical, and six samples were taken.

The effect of the addition of small amounts of wetting agents was also studied. Using 79% H_3PO_4, 15 cc. of "Tergitol" No. 4 were added to 300 lbs. of acid. A mixing study was made in which the acid proportion was varied from 100% to 53% theoretical, five samples being taken. Using 70% H_3PO_4, about 5 cc. of "Tergitol" No. 7 were added to about 100 lbs. of acid, and three samples were obtained at acid proportions from 55% to 65% theoretical.

The setting times, setting temperatures and maximum temperatures reached were observed by methods noted above. These are presented in Table VIII.

Results and Conclusions:

Mixer Operation: The minimum rock-acid ratio in each case represents the limiting conditions under which the mixer could be operated. At these low acid-rock ratios the mixture extruded was of a mud-like consistency and set almost immediately after being discharged from the mixer.

Failure was due in some cases to the plugging or cementing over of the mixer outlet while sometimes operation had to be stopped because of the accumulation of rock in the mixer chute, as only that portion would fall through the rock inlet that would form a sufficiently fluid mixture with the acid.

It must be further noted that failure under these conditions was no discredit to the mixer for in all cases it handled lower acid-rock ratios than had been expected. At the normal working ratios the performance was quite satisfactory.

Although the addition of wetting agents to the acid had an appreciable effect on the elimination of foaming, it had no noticeable effect on the operation of the mixer.

Temperature observations: All the mixtures were observed to set in the region of 65°C. (Table VIII.). The maximum temperatures reached, in all cases after the mixture had set, approached 100°C. for the mixtures made with 79% acid, 90°C. for the mixtures made with 75% H_3PO_4 and 78°C. for those with 70% acid.

Thus it is observed that the setting temperatures are by no means high enough to cause such rapid drying by the mechanism of evaporation; furthermore, with the exception of the 79% acid run, the maximum temperatures reached by the solid were hardly high enough to give this effect. Hence, it is inferred that the drying is for-the-most-part due to hydration of the monocalcium phosphate, as is now, but has not always been, accepted to be the case.

ANALYTICAL METHODS

Samples:

Sampling: The samples were taken from the wooden sample boxes, except in the case of several taken from the bin, by the following procedures:

Run 3. Samples taken from the upper portion of the box.

Run 5. Samples taken by drilling out cores at corresponding positions in the boxes.

Preparation: The samples were ground as follows:

Run 3. Through 65 mesh.

Run 5. Through 40 mesh.

The latter mesh was used because of the rather poor grindability of some of the samples in the wide range studied.

Methods of Analysis:

The water soluble and citrate soluble P_2O_5 determinations were made by the official Association of Official Agricultural Chemists methods.[1]

Moisture was determined by drying 24 hours in a desicator over concentrated sulfuric acid.

Free acid was determined by the acetone extraction method.[2,3] Determinations were made on both the dried and undried samples.

1. Official and Tenative Methods of Analysis, Bulletin, Amer. Assoc. of Official Agric. Chemists.
2. Swift and Company.
3. Hill and Beeson, J. Assoc. Agr. Chem., 18, 244-260 (1935)

The total P_2O_5 and CaO were determined according to the method furnished with Standard Sample No. 56 by the Bureau of Standards.

Acid concentrations were determined by the following methods:

1. Specific gravity and specific gravity-temperature-concentration charts.
2. Gravimetrically-precipitation of P_2O_5 as Ammonium phosphomolybdate.
3. Titration to bromo-cresol green end point.
4. Titration to phenolphalein end point.
5. Back titration to brom-cresol green end point.

DISCUSSION

Acid concentrations:

The acid concentrations as determined from specific gravities were consistently higher than the values obtained from chemical analyses. This is attributed to the presence of impurities in the acid. Furthermore, the acid concentrations as determined by gravimetric chemical analyses were found to be from one to two per cent higher than those determined by volumetric methods, due to the presence of phosphate other than that making up the acid.

Moisture:

There seems to be no standard method for making the determination of moisture in superphosphate. The method used, drying over H_2SO_4, is quite a slow process and tends to give low results. It was found to require up to 72 hours to remove the "free moisture" in some cases.

Free Acid:

This determination, used directly in calculation of acid reacting, is made by an acetone extraction of the material. This extraction is so affected by several factors such as moisture and proportion of free acid as to give results in some cases several times too high. Compare columns 7 and 8- Tables V, VI, and VII, where column (7) is free acid determined without drying sample and column (8) is free acid determined after drying samples.

Although obviously in error, the method of (7) is generally used in determinations made on samples of this nature.

Due to the buffer effect, the end point in this titration was also quite uncertain, varying somewhat with different amounts of free acid present.

Water Solubility:

Although duplicate analyses and numerous checks indicated that this determination was reproducible, it is known that monocalcium phosphate is unstable in the presence of water, breaking down into solids containing more lime than the original solid (eg. dicalcium phosphate) and phosphoric acid[1]. Thus, due to the insolubility of dicalcium phosphate in water, there is always danger of a low water solubility determination due to its retention on the filter in the A.O.A.C. analyses. This would account somewhat for the difference between Citrate Soluble P_2O_5 and Water Soluble P_2O_5 over that provided by the phosphates of iron and aluminum (and possible some dicalcium phosphate already present, see page 10.).

For the erratic nature of this determination compare other curves on Curve Sheet 4.

---

1. Mellor, Loc. Cit. p. 866.

Citrate Solubility:

As a basis for a quantitative study, the shortcomings of this method are obvious. This arbitrary determination is especially dependent on manipulation rather than quantitative reaction.

As this is the criterion by which superphosphates are judged it is indeed unfortunate that such a condition should exist.

Sampling:

Although samples were taken by carefully removing cores from the sample boxes there is some evidence that uniform sampling was not obtained in all cases. There seems no obvious method, however for improving this procedure.

DISCUSSION OF RESULTS

Mechanical:

Mixer Operation: After the elimination of preliminary difficulties with the acid inlet, the centrifugal mixer was found to perform quite satisfactorily with acid concentrations from 40% to 80% and with acid rock ratios as low as 25% theoretical using 40% acid, and as low as 60% theoretical with the most concentrated acid used.

The maximum feed rate the mixer will handle has not been determined because it exceeds the present capacity of the rock and acid feed systems. However at the maximum rate tried the mixer gave excellent performance handling a total feed of 1.58 tons per hour.

Operating in the continuous run (Run 3) under quite unfavorable conditions, i.e. low feed rate, hence long mixing time and greater probability of setting up in the mixer, there were no indications of failure of the mixer.

Acid Feeder: After elimination of the manometer difficulties and venting air from the system the second acid feeder was found to operate quite well. The 3/16-inch orifice placed in the 1½-inch section of acid line afforded large differentials at the ordinarily used feeds and at the same time had ample capacity within the range of the manometer.

The Micro-Westco pump, due to factors inherent in its design, was found to be ideally suited for this application, in so far as a uniform and flexible discharge was concerned.

Rock Feeder: The rock feeder did not at all times give the performance which might have been desired. The requirements for this application, instantaneously constant feed with accurate control, seem rather difficult to meet with the types of feeders generally available.

At the higher feed rates used in commercial operation, however, this factor could perhaps be quite easily eliminated.

The bridging over and bulk density variation of the phosphate rock are ever-present sources of difficulty and probably could be better worked out for each specific application.

Miscellaneous: Although under the conditions of these runs the process could hardly be termed continuous because the mixer discharged into a batch type den, the general characteristics of the mixer seem to fit it into a continuous outlay as noted in the General Survey. This type of mixer might fit equally well into a tower drying unit (3), or any other combination capable of handling the reaction mixture through the remainder of its course.

Corrosion: The lead-lined tank showed considerable attack by the phosphoric acid, while the brass parts of the pump and acid lines showed little effect of corrosion. The shaft of the Micro-Westco pump was corroded considerable however, while the steel idler on the original Viking pump was almost completely destroyed.

The cast-iron housing showed little signs of corrosion as a result of the runs. The blades of the impeller, however, were corroded about 3/32 inch deep near the point of acid entrance. This trouble could be largely eliminated by building the impeller of brass or more simply merely by coating the parts subject to the action of the acid with brass by brazing. The cast iron impellers, however, are quite inexpensive and easily installed.

The most serious corrosion encountered throughout the series of runs was not due to acid but rather to the action of mercury on the brass and copper equipment. This mercury, blown over from the manometer in Run 3, amalgamated with the copper and destroyed a number of brass fittings, necessitating a complete renewal of the acid feed piping. Improved mercury traps were installed to prevent recurrence of this damage.

Although untreated wooden barrels are apparently satisfactory for shipping concentrated phosphoric acid for short distances, they are unsatisfactory for long time storage. On standing in the wooden barrels the acid became quite discolored, and the barrels had a tendency to leak, probably due to the dehydration properties of the acid.

Discrepancies:

Discrepancy between proposed acid-rock ratios and actual ratio as calculated from analyses:

In Run 3, when the rock acid ratios and feed rates were changed, sufficient time was not allowed for constant conditions to be reached, hence the analytically determined results are not in close agreement, in all cases, with the proposed values.

In Run 4, the discrepancies are attributed to the overloading of the rock feeder drive and its failure to deliver the rock corresponding to the calibration value. The ¼ HP. "Transitorq" unit was replaced for this reason.

In Run 5, at the higher acid-rock proportions close agreement exists, but with the low ratios there is a marked discrepancy. This is attributed to piling up of the rock in the mixer chute.

Chemical Properties: These results are best observed by reference to Figures 1,3,4, and 5 and Tables II,III,IV, V,VI,VII,VIII, and IX.

In brief, the stoichiometric treatment of the analytical results shows a general high degree of conversion in the working range. Using 78% H_3PO_4 in Run 3, a conversion of 95% using 85% of the theoretically required acid is observed while in Run 5 a conversion of 94% was obtained, using the same rock-acid proportions. With the higher acid concentrations used, 99% conversion was obtained consistently, using the theoretically required acid.

The rock reacting after 30 days as a function of acid-rock ratio for the acid concentrations used in Run 5, as presented by Figure 3, indicates that the best conversion is obtained with the most concentrated acid used. This fact is also brought out by Figures 1 and 5 and Tables IV,V,VI, andVII.

The data from Run 5, using three acid concentrations, as shown on Figure 1, indicate that, within the range studied, the per cent of acid reacting is practically independent of concentration. However, as these calculations are based on the free acid determinations they must be accepted as true only within the numerical accuracy of this method. It is further shown by this curve that the amount of acid reacting is substantially constant up to the region of 90% theoretical where it begins to drop off rapidly from 96% at 90% theoretical

20 × 20 to the inch, 10th lines heavy.
MADE IN U. S. A.

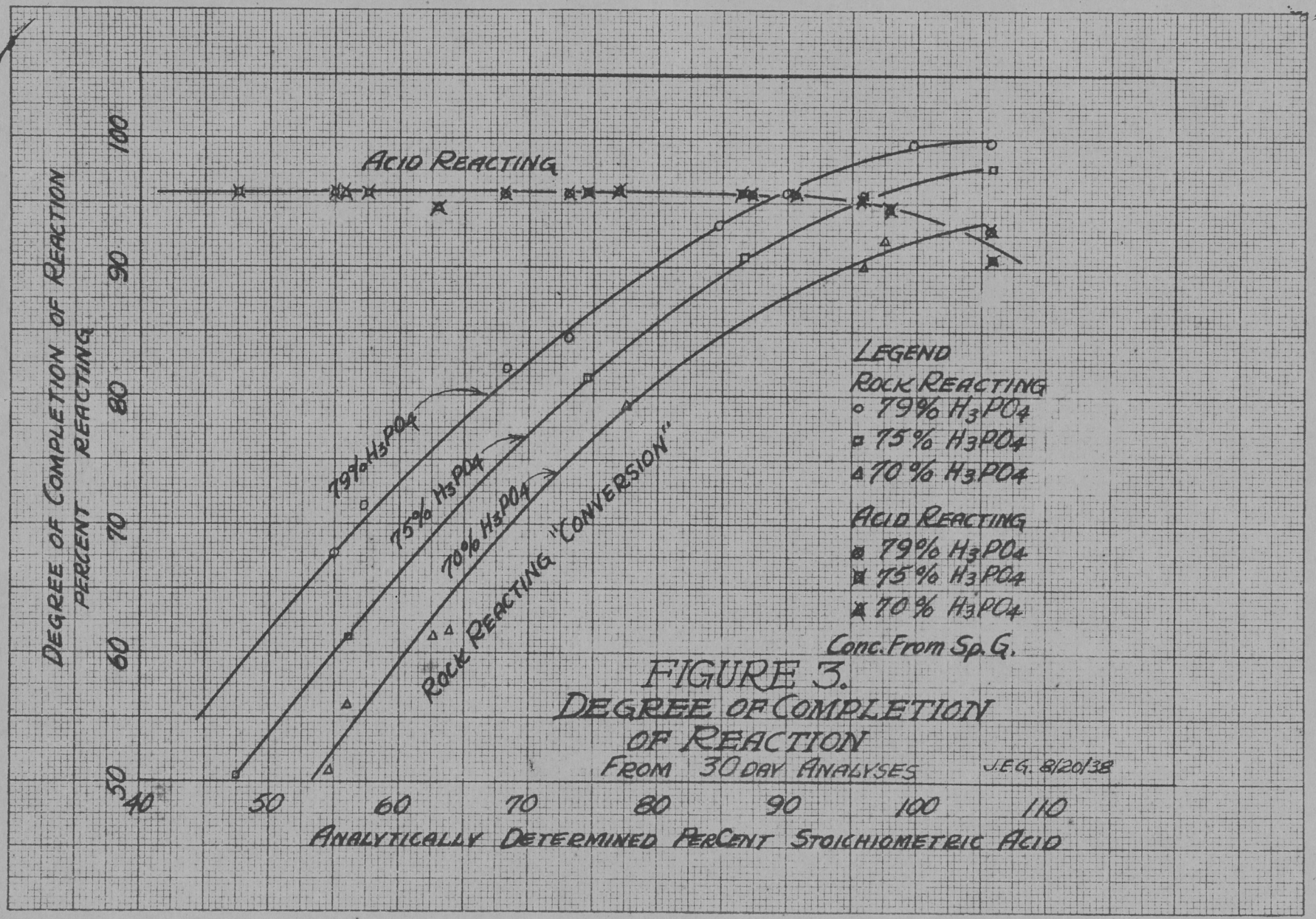
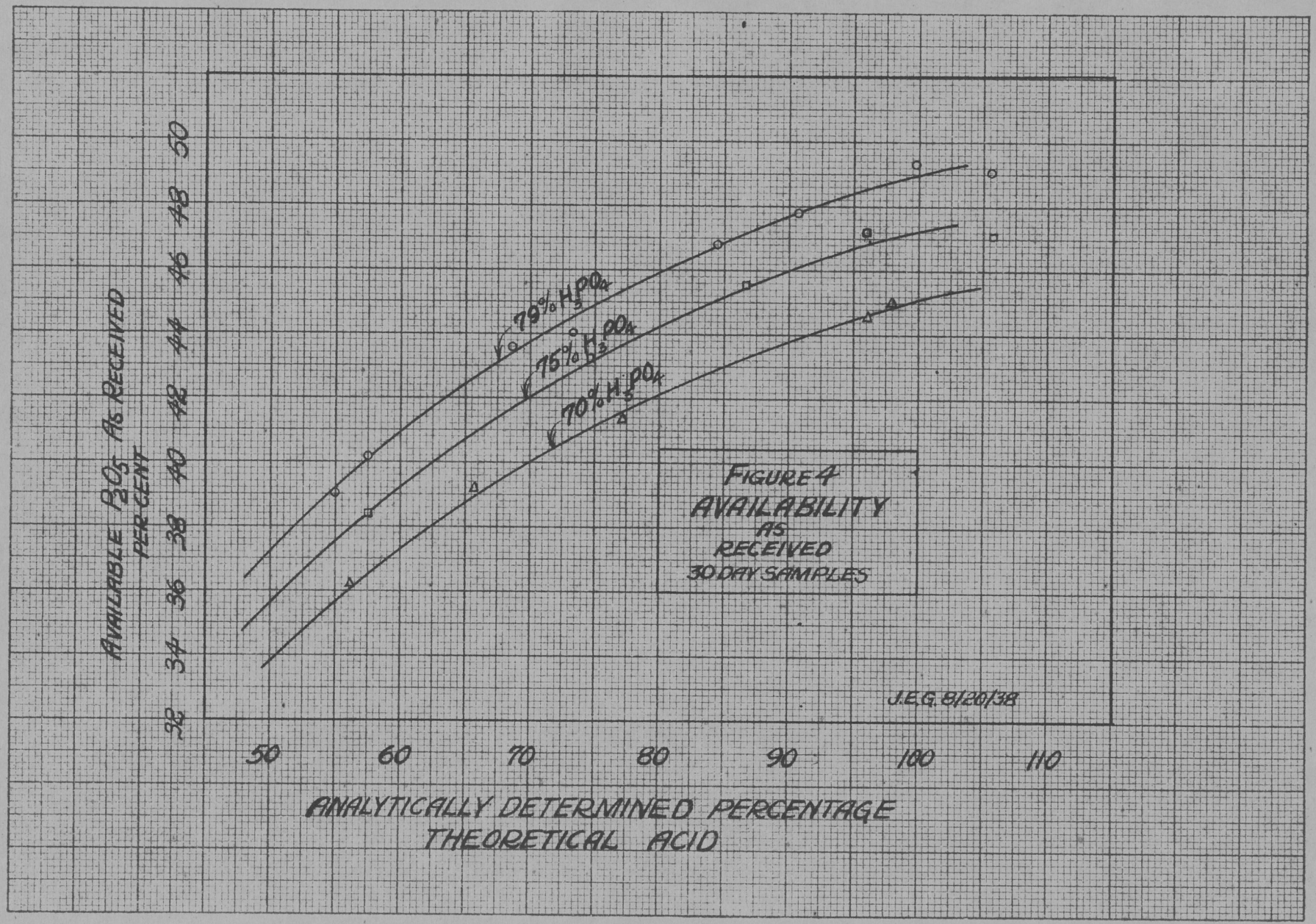


FIGURE 3.
DEGREE OF COMPLETION OF REACTION
FROM 30 DAY ANALYSES

J.E.G. 8/20/38


AVAILABLE P_2O_5 AS RECEIVED PERCENT
32
34
36
38
40
42
44
46
48
50
79% H_3PO_4
75% H_3PO_4
70% H_3PO_4
FIGURE 4
AVAILABILITY
AS
RECEIVED
30 DAY SAMPLES
J.E.G. 8/20/38
50
60
70
80
90
100
110
ANALYTICALLY DETERMINED PERCENTAGE
THEORETICAL ACID

to only 90% at 105% theoretical; this is as might be expected from other considerations.

In the results from Run 4 as presented in Table 8, although containing some relations which do not lend themselves to simple explanation, there is no conclusive evidence of any gain in using the low-acid rock-ratios, neither is there any conclusive indication of the formation of any appreciable amounts of dicalcium phosphate.

Effect of Addition of "Tergitol": The effect, if any, of the addition of "Tergitol" to the acid is not apparent from either the Tables or the Curves in the case of the 79% H_3PO_4 Run using "Tergitol" No. 4. The samples in which "Tergitol" No. 7 was used (9-D'-E'-F') carried a slightly higher moisture than similar mixtures without "Tergitol", but the methods used in this determination are quite erratic.

Time-Conversion Observations: In regard to the kinetics of the reaction, the following facts are observed from the data presented in Tables V, VI and VII.

Using 79% H_3PO_4 the percent conversion is found to drop off between the three day and the fourteen day analyses and rise again after 30 days to a percentage in most cases higher than the original.

Using 75% H_3PO_4 the conversion is observed to rise between the third and the fourteenth day and then drop after thirty days to a percentage roughly equal to the original values.

Using 70% H_3PO_4 the conversion was found to rise during the first period and then drop off to a value in most cases considerably lower than the original determination.

These phenomena, due to inadequate data and information, can not be definitely explained, but it would seem quite logical to expect this action to be due to two simultaneous reactions which might be called "conversion" in the desired direction and "reversion" in the other, preceding at different rates and towards different equilibria. Thus acid concentration could be expected to affect the reaction rates in such a manner as is indicated by a rough graphical representation of the time-conversion data as noted above and presented in the tabular summaries.

Summarizing in regard to the chemical results, there are many points brought out by a careful study of the data that are not discussed above or elsewhere in this report, both because of lack of space and inadequate knowledge of the mechanisms involved. The data are presented as taken but are not yet considered complete in every detail. Where doubtful points arise there are in general several possible explanations, and it is for this reason that no conclusions have been drawn concerning such factors as the true nature of reversion, the form of the P_2O_5 which makes up the difference between the water soluble, and citrate soluble, the effect of moisture and the magnitude of the free acid on the free acid estimation

by the acetone extraction, the effect of hydrolyses on the water-solubility determination, the final condition of the iron and aluminum, and many others of equal or greater importance which have arisen in the course of the many analytical determinations and subsequent attempt of correlation.

As these questions have arisen they have been set aside for future laboratory investigation.

Physical Properties of Products: Some indication of the physical properties of the product may be noted from the arbitrary setting times (Table III) and the moisture contents.

Using these criteria, as well as the observations made during the runs and grinding of the product, it is evident that the most desirable physical properties are obtained in the runs using the most concentrated acid (78-80% H_3PO_4) with the products ranging from dense, hard, dry solids in these runs through sticky solids and stiff muds to pasty masses in the case of the runs using dilute acids.

The superphosphate collected in the bin from the continuous run was gray in color and extremely hard and difficult to break. Two one-foot cubes were sawed out of this material and their density determined to be 56 lbs. per cubic foot. After grinding in a hammer mill the bulk density was found to be 60 lbs. per cubic foot.

Power required: The wattmeter readings pulsated considerably. The average results are shown by Fig. 2. and in Table XI.

With the maximum feed rate of 1.58 tons per hour an average of 0.69 KW. was required. The no load power consumption was observed to be 0.26 KW.

Economic Factors:

These calculations were made, based on the actual chemical analyses, with the following general purposes in view:

1. To ascertain the material cost distribution in superphosphate.

2. To obtain a relation between these costs and the acid-rock ratio for a number of acid concentrations.

3. To consider the effect of freight, and other tonnage costs outside of materials on the optimum operating conditions.

These relations are brought out in Figures 6 and 7 and Tables IX and X.

The following appear to be the most important observations from this standpoint:

1. When the cost per ton of available P_2O_5 in the product is plotted as a function of the acid-rock ratio a relatively flat curve is obtained. Thus there is no sharply defined optimum in evidence.

2. The main cost is acid, both because more is used and because P_2O_5 in this form is more expensive than that in rock.

3. The most economical acid concentration is the highest used.

4. When freight charges are considered, the optimum operating conditions are found to shift toward the higher acid-ro

operating conditions are found to shift toward the higher acid-rock ratios, as would be expected.
(Note broken line on Figure 7.) for trend of optimum conditions.)

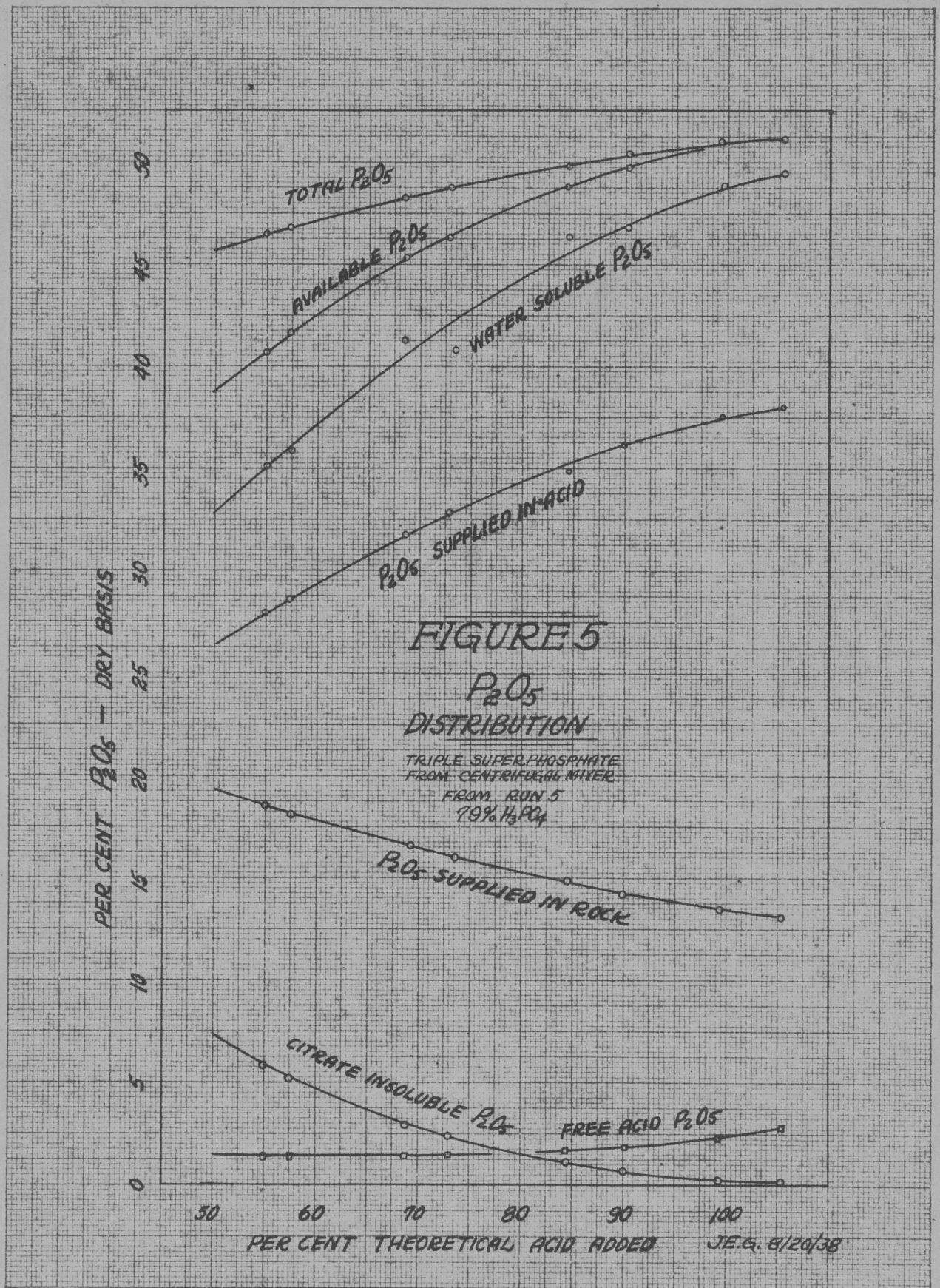


FIGURE 5

P_2O_5 DISTRIBUTION

TRIPLE SUPERPHOSPHATE FROM CENTRIFUGAL MIXER FROM RUN 5 79% H_3PO_4

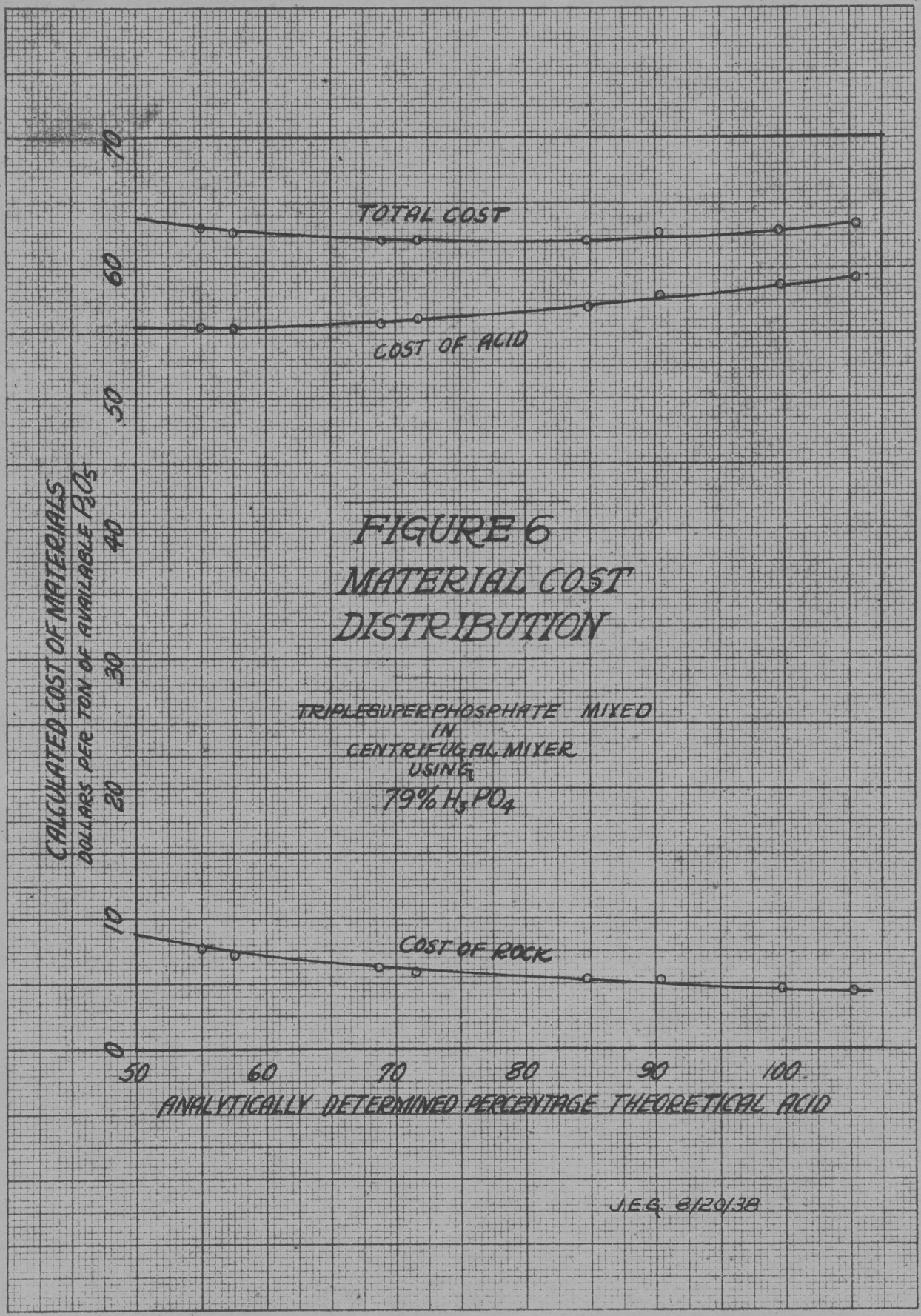


FIGURE 6

MATERIAL COST DISTRIBUTION

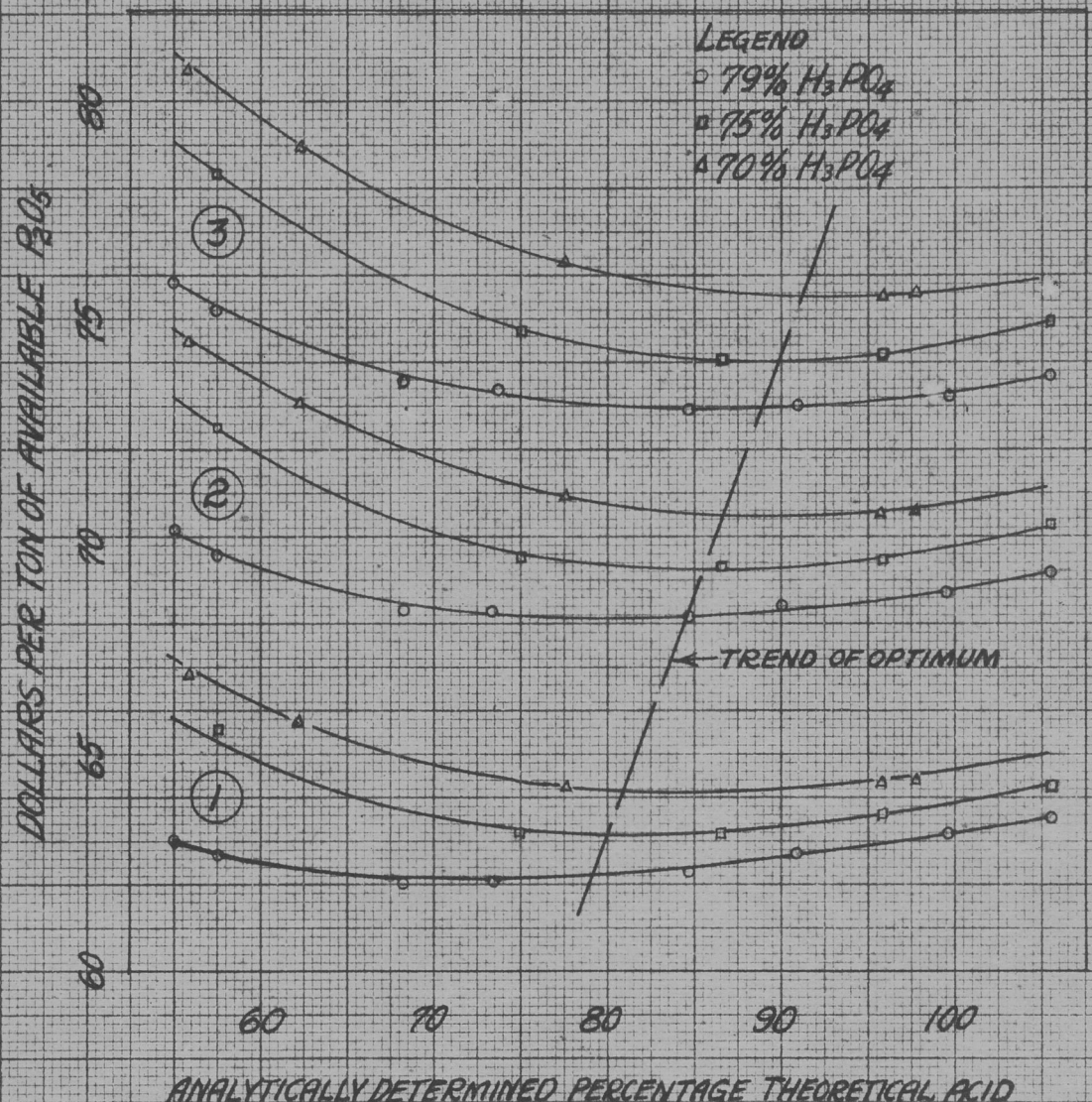


FIGURE 7
COST VARIATION

(1) Cost in Storage Pile

(2) Cost Including Bagging and Freight - 100 Mi.

(3) Cost Including Bagging and Freight - 200 Mi.

James E. Gantt - 8/20/38

MADE IN U.S.A.

CONCLUSIONS

Mechanical

Mixer: The mixer was found to perform very satisfactorily throughout the normal working range of acid concentrations and acid-rock proportions, and to be capable of successful operation with higher acid concentrations and over a wider range of acid-rock ratios than is apparently possible with most other types of mixers in general use.

The very short mixing time and continuous discharge would seem to fit this mixer into any of several types of continuous units for super phosphate manufacture, such as a slow conveyor[1] or tower or spray process, or to make it highly satisfactory as an initial or intermediate step in a multistage process.

The possibilities of such a mixing unit need not be confined to superphosphate, for, although it seems especially fitted to such a reaction, it could apparently be used on any other process requiring the rapid and continuous mixing of a finely ground solid and a liquid.

Accessories: As the size of a commercial unit would have considerable effect on the type of acid and rock feed systems used, these will not be noted in detail at this point.

---

1. Curtis, H.A., Chem. Met. Eng. 42, loc. cit., p. 491

However, it suffices to note that, while after elimination of certain initial difficulties these parts of the present system performed quite satisfactorily, the higher feeds of a commercial unit should make possible even more accurate control than was obtainable with the present systems.

Properties of Product: From the results noted in detail above, it appears that the product in the normal working range is quite satisfactory from a chemical standpoint. The consistently high degree of completion of the reactions as well as the definite effect of the variables studied indicate that, in spite of the very short mixing time, the nature of the final product can be quite accurately controlled.

From the standpoint of physical properties the present mixer has the desirable capacity of handling higher acid concentrations than generally possible with other types, hence giving a drier product. Furthermore the mixture, being discharged as a fluid, may be handled economically in such a manner to give a final product with the most desirable physical properties, which is quite important to both the manufacturer and ultimate consumer.

The most striking overall observation is that, unlike most other types of mixing units, the present mixer gives the most desirable product, from the chemical, physical, economical standpoints, with the highest acid concentration tried, (80% H_3PO_4).

These factors seem to make a centrifugal-pump-type mixer fit well into the modern trend of superphosphate manufacturing.

- o -

BIBLIOGRAPHY

1. Baskervill, W.H., "A Centrifugal Type Superphosphate Mixer," T.V.A. W.O.162, Report No.4, Mar. 1938.
2. Bassett, H. Jr., Z. Anorg. Chem. 59, 1-55, (1908)
3. Brand, C. J., Mfg. Record, 20, June (1938)
4. Copson, R.L., Newton, R.H, and Lindsay, J.D., Ind. Eng. Chem. 29, 175-9 (1937)
5. Curtis, H. A., Chem. Met. Eng. 42, 488-491 (1935)
6. Curtis, H. A., Miller, A. W., and Junkins, J. N., Chem. Met. Eng. 42, 647-50, (1936)
7. Hill and Beeson, J. Assoc. Agr. Chem, 18, 244-260 (1935)
8. Jacob, K. D., Proc. Natl, Fert. Assoc., June, 1929, p. 55.
9. Larison, E. L., Proc. Natl. Fert. Assoc., June, 1929, p. 67.
10. Meller, J. W., Comprehensive Treatise on Inorganic and Theoretical Chemistry, Vol. 3., p. 865-70
11. Newton, R. H., and Copson, R. L., Ind. Eng. Chem. 28, 1182-6 (1936)
12. Newton, R. H., "Continuous Mixing of Superphosphate in Liquid Form", T.V.A. W.O. 172, Report No. 4, June, 1936.
13. Waggaman, W. H., and Easterwood, H. W., Phosphoric Acid, Phosphates and Phosphatic Fertilizers, The Chemical Catalog Co, Inc., New York, (1927) p. 132.

Based on original data recorded on T. V. A.

notebook pages-

2326-K - 2337-K

2599-K - 2625-K

2776-K - 2850-K

5850-K - 5900-K

APPENDIX

Appendix A. Derivations and Calculations

Table I. Screen Analyses of Rocks Used.

Table II. Chemical Analyses of Rocks Used.

Table III. Feed Rates and Variables, Runs 1, 2, and 3.

Table IV. Data and Calculations, Run 3.

Table V. Data and Calculations, Run 5, 79% H_3PO_4.

Table VI. Data and Calculations, Run 5, 75% H_3PO_4.

Table VII. Data and Calculations, Run 5, 70% H_3PO_4.

Table VIII. Setting Times and Temperatures, Run 5.

Table IX. Data and Calculations, Run 4.

Table X. P_2O_5 Distribution- (dry basis)

Table XI. Cost Distribution.

Table XII. Power Consumption, Run 3.

Plate 1. Sectional Drawing of Mixer.

APPENDIX A

Derivations:

1. Per Cent Theoretical Acid Added:

Basis: 100 lb. of rock

Let x = lb. of P_2O_5 added

Let k = lb. of P_2O_5 required

then; $$\frac{(P_2O_5)_o + x}{(CaO)_o} = \frac{(P_2O_5)_f}{(CaO)_f} = \frac{(P_2O_5)_o}{(CaO)_o} + \frac{x}{(CaO)_o}$$

% Theoretical acid added = 100 x/k

$$\% = \left[\frac{(P_2O_5)_f}{(CaO)_f} - \frac{(P_2O_5)_o}{(CaO)_o}\right] \frac{(CaO)_o}{k}$$

2. Per Cent Rock Reacting:

Basis: 100 lb. superphosphate

$$P_2O_5 \quad \frac{(CaO)_f}{(CaO)_o} = \text{lb. } P_2O_5 \text{ furnished by rock}$$

Assuming the limiting case; that is, that all citrate insoluble P_2O_5 comes from the rock.

(See General Theory)

Then; $$100\left[1 - \frac{(P_2O_5)_{c.i.}}{\left(\frac{P_2O_5}{CaO}\right)_o \quad (CaO)_f}\right] = \text{\% rock reacting or "conversion"}$$

Where; Sub o = per cent from rock analyses

Sub f = per cent from product analyses

3. Acid Reacting:

Basis: 100 lb. superphosphate

$$(CaO)_f \left(\frac{P_2O_5}{CaO}\right)_o = \text{lb. } P_2O_5 \text{ added in rock}$$

$$(P_2O_5)_t - (CaO)_f \left(\frac{P_2O_5}{CaO}\right)_o = \text{lb. } P_2O_5 \text{ added in acid}$$

$$\frac{(P_2O_5)_{f.a.}}{(P_2O_5)_t - \left(\frac{P_2O_5}{CaO}\right)_o (CaO)_f} = \text{acid not reacting}$$

$$100\left[1 - \frac{(P_2O_5)_{f.a.}}{(P_2O_5)_t - \left(\frac{P_2O_5}{CaO}\right)_o (CaO)_f}\right] = \% \text{ acid reacting}$$

4. P_2O_5 Distribution:

Basis: 100 lb. superphosphate

(a). P_2O_5 from rock = $\left(\frac{P_2O_5}{CaO}\right)_o (CaO)_f$

(b). P_2O_5 from acid = $(P_2O_5)_t - \left(\frac{P_2O_5}{CaO}\right)_o (CaO)_f$

Calculations:

(For rock analyses see Table I.)

Acid Required:

Basis: 100 lb. or rock

Containing:

CaO = 45.4 lb.	/ 56.08	=	0.8090 lb.mols.
Fe_2O_3 = 2.8 "	/ 159.68	=	0.0175 " "
Al_2O_3 = 2.14 "	/ 101.94	=	0.0210 " "
			0.8475 " "

P_2O_5 = 32.1 lb. / 142.04 = 0.2260 lb mols.

a. For conversion to $Ca(H_2PO_4)_2$, $FePO_4$, and $AlPO_4$.

Total P_2O_5 req'd.	=	0.8475 lb. mols.
P_2O_5 present	=	0.2260 " "
To be added in acid	=	0.6215 " "
	=	88.25 lb.

b. For conversion of Ca to $Ca(H_2PO_4)_2$.

Tota l P_2O_5 req'd.	=	0.80 90 lb. mols.
P_2O_5 present	=	0.22 60 " "
Ta be added in acid	=	0.58 30 " "
	=	82.8 lb.

Numerical adaptation of above formulas to the rock used:

1. Per cent theoretical acid added:

a. For conversion to $Ca(H_2PO_4)_2$, $FePO_4$, and $AlPO_4$.

$$\% = 51.4 \left(\frac{P_2O_5}{CaO}\right)_f - 36.4$$

Columns (11) in Tables V, VI, and VII.

b. For conversion of Ca to $Ca(H_2PO_4)_2$.

$$\% = 54.8 \left(\frac{P_2O_5}{CaO}\right)_f - 38.75$$

Column (12) in Tables V, VI, and VIII.

2. Rock Reacting:

$$\% = 100 - 141.4 \frac{(P_2O_5)ci.}{(CaO)_f}$$

3. Acid Reacting:

$$\% = 100 - \frac{100\ (P_2O_5)fa.}{(P_2O_5)_f - 0.707\ (CaO)_f}$$

4. P_2O_5 from rock:

$$\% = 0.707\ (CaO)_f$$

5. P_2O_5 from acid:

$$\% = (P_2O_5)_t - 0.707\ (CaO)_f$$

TABLE 1.

SCREEN ANALYSES OF ROCKS USED

SCREEN SIZE	BATCH NO. 1. ESTABROOK ANALYSIS	BATCH NO. 2. ESTABROOK ANALYSIS	BATCH NO. 2. WILSON DAM ANALYSIS
MESHES PER INCH	CUMULATIVE PERCENT THROUGH	CUMULATIVE PERCENT THROUGH	CUMULATIVE PERCENT THROUGH
50	--	--	99.9
65	95.5	99.3	--
80	93.5	98.8	--
100	89.6	97.3	97.9
150	77.0	90.5	92.5
200	64.8	79.8	83.3

TABLE II

CHEMICAL ANALYSES OF ROCKS USED

COMPONENT	BATCH 1. (RUNS 1.&2.) %	BATCH 2. (RUN 3.) %	BATCH 2. (RUNS4.& 5.) %
MOIST.	1.38	0.78	--
P_2O_5	29.3	32.3	32.1
CaO	41.5	45.6	45.4
Fe_2O_3	3.03	2.80	2.80
Al_2O_3	1.56	2.14	2.14
F	--	--	3.53

TABLE III

RUN 3. RATES OF FEED OF ACID AND ROCK

RUN NO.	TEST NO.	ROCK RATE	ACID RATE	ACID PROPORTIONS		VARIABLE STUDIED
		lbs/min	lbs/min	A	B	
1	Continuous period	6.0	7.9	95 ± 8	100 ± 8	Continuity of Operation
2	Continuous period	6.0	7.9	95 ± 8	100 ± 8	Continuity of Operation
3	1	15.0	23.3	99 ± 3	105 ± 3	Acid-Rock Ratio
3	2	15.0	22.3	95 ± 3	101 ± 3	" " "
3	3	14.8	21.2	91 ± 3	97 ± 3	" " "
3	4	14.8	18.8	81 ± 3	86 ± 3	" " "
3	5	6.7	10.0	95 ± 6	101 ± 6	Feed Rate
3	6	11.5	17.4	97 ± 4	103 ± 4	" "
3	7	16.7	25.5	97 ± 3	103 ± 4	" "
3	8	20.7	32.1	99 ± 2	105 ± 2	" "
3	Continuous period	6.7	9.45	90 ± 6	96 ± 6	Continuity of Operation

A Per cent of the amount required to convert all Ca to $Ca(H_2PO_4)_2$, Fe to $FePO_4$, and Al to $AlPO_4$.
B Per Cent of the amount required to convert all Ca to $Ca(H_2PO_4)_2$.

TABLE IV

DATA SHEET RUN 3. *

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
Run No.	Test No.	Elapsed Time	Moist.	P_2O_5 Total	P_2O_5 W.S.	P_2O_5 C.I.	P_2O_5 F.A.	P_2O_5 Avail.	CaO	Acid Supplied % Theoretical		Acid Reacting	Rock Reacting
		Days	%	%	%	%	%	%	%			%	%
2	Cont.	20	7.02	45.8	41.4	1.32	2.97	44.5					
3	1	3	4.03	49.95	46.95	0.33	4.13	49.62	18.5	100.5	107	88.7	97.5
3	1	6				0.25	3.41					90.7	98.1
3	1	19			48.6	0.17	2.75	49.78				92.5	98.7
		27				Trace	2.36					93.6	99
3	2	3	4.48	50.15	47.65	0.22	4.05	49.93	18.4	101.8	108.5	89.1	98.3
		27				Trace	2.72					92.6	99
3	3	3	4.32	49.70	46.25	0.55	3.04	49.15	19.2	99.1	105.7	91.6	95.9
3	3	6				0.30	2.25					93.8	97.3
		27				0.16	1.67					95.3	98.8
3	4	3	4.54	49.20	44.75	0.94	2.24	48.26	20.5	85.4	90.0	93.5	93.4
3	4	19			45.20	0.73	1.45	48.47				95.8	95.0
		27				0.75	1.16					95.4	94.9
3	5	3	3.82	49.7	47.25	0.39	4.67	49.76	18.1	102.9	109.5	88.3	97.0
3	6	3	4.56	49.8	47.10	0.55	4.09	49.25	18.4	100.8	107.3	88.8	95.7
3	7	3	5.40	49.95	47.50	0.50	5.03	49.45	17.8	106.0	113	86.5	96.1
3	8	3	4.85	50.15	47.90	0.44	5.57	49.71	17.6	108.1	115.4	85.0	96.5
3	Cont.	6		49.40	46.25	0.41	3.50	49.09	19.0	95.5	101.7	90.6	97.0
		19			47.05	0.20	1.63	49.20				96.6	98.5
		27				0.15	1.49					95.8	98.9

(11) Acid supplied for conversion to $Ca(H_2PO_4)_2$, $FePO_4$, and $AlPO_4$. (Per cent theoretical)

(12) Per cent theoretical acid for conversion of calcium content to $Ca(H_2PO_4)_2$.

* Includes one sample from Run 2.

Identification of Samples

Run 5.

Because of the large number of runs, a two-symbol numbering system was used. According to this scheme, the first number indicates both the acid concentration used and the time elapsed between mixing and analysis, while the letter following is an indication of the acid-rock ratios used.

Thus 1-A, 4-A, and 7-A are the 3-day, 14 day and 30 day samples, respectively, of the mixture using 79% H_3PO_4 in approximately the 100% theoretical proportion. 2-A, 5-A, and 8-A are then the 3, 14, and 30 day samples, respectively, of the mixture using 75% H_3PO_4 in the same proportion; 3-A, 6-A, and 9-A correspond using 70% H_3PO_4.

The letters following the numbers may be taken as an indication of the theoretical acid added. This relation is best observed by comparing columns (1) and (11) in Tables V, VI, VII, and VIII, or columns (1) and (2) in Tables IX and X.

"Primes" (1-A' etc.) signify that "Tergitol" was used in the acid with these mixtures.

This nomenclature applies to Tables V, VI, VII, VIII, IX, X, and XI.

TABLE V

DATA SHEET * RUN 5. * SERIES 1. USING 79% H_3PO_4

(1) SAMPLE NUMBER	(2) ELAPSED TIME	(3) MOIST.	(4) P_2O_5 TOTAL	(5) P_2O_5 W.S.	(6) P_2O_5 C.I.	(7) P_2O_5 F.A.	(8) P_2O_5 F.A.	(9) P_2O_5 AVAIL.	(10) CaO	(11) ACID SUPPLIED % THEORETICAL	(12)	(13) ROCK REACTING	(14) ACID REACTING
	DAYS	%	%	%	%	%	% DRIED	%	%			%	%
1-A	3	7.8	48.15	45.3	Trace			48	17.72	103.1	110	99	
4-A	14	6.5		47.55	0.16	4.42		48				98.7	
7-A	30	4.2	49.0	47.35	Trace	3.70	2.49	49	17.76	105.6	113	99	93.16
1-B	3	6.3	47.05	43.6	1.23			46.8	20.15	83.6	89.1	91.3	
4-B	14	5.4		43.3	1.41	2.67		45.6				90.1	
7-B	30	4.0	47.8	43.55	0.97	2.24	1.41	46.8	22.4	84.6	90.2	93.3	95.78
1-C	3	6.3	45.7	40.4	3.28			42.4	22.47	67.9	72.4	79.4	
4-C	14	4.9		38.1	3.68	2.63		42.0				76.9	
7-C	30	3.6	46.5	39.6	2.90	2.17	1.26	43.6	22.76	68.6	73.1	82.0	95.86
1-D	3	6.0	45.12	36.1	5.03			40.1	23.78	61.3	65.4	70.1	
4-D	14	4.2		35.6	5.59	2.16		39.5				66.8	
7-D	30	3.3	47.55	35.4	4.54	2.10	1.10	43.0	24.38	64.0	68.1	73.7	96.38
4-A'	13		47.5	46.7	0.45			47.0	18.40	96.3	102.7	96.5	
7-A'	30	3.1	49.5	47.35	0.13	2.19	2.15	49.4	1867	99.8	106.3	99	90.58
4-B'	14		47.5	44.5	1.18			46.3	18.91	93.1	99.3	91.2	
7-B'	30	4.1	48.4	44.9	0.63	2.67	1.44	47.8	19.53	90.8	96.8	95.5	95.84
4-C'	14		45.5	39.4	3.38			42.1	21.51	72.4	77.1	77.8	
7-C'	30	4.9	46.4	38.75	2.45	2.83	1.41	44.0	21.73	73.3	78.2	84.1	95.46
4-D'	14		44.2	33.75	6.85			37.4	24.30	56.9	60.6	60.2	
7-D'	30	3.5	45.15	34.50	4.92	1.98	1.12	40.2	24.70	57.4	61.2	71.9	95.96
1-E'	3	6.4	43.65	32.8	6.57			37.1	25.02	53.1	56.6	62.9	
4-E'	14			33.2	7.12	2.55		36.5				59.8	
7-E'	30	3.7	44.8	33.7	5.78	2.09	1.19	39.0	25.22	55.0	58.6	67.6	95.41
1-F'	3	6.7	44.7	36.2	4.95			39.7	23.14	62.8	67.0	69.7	

TABLE VII

DATA SHEET RUN 5. SERIES 2. 75% H_3PO_4

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
SAMPLE NUMBER	ELAPSED TIME	MOIST.	P_2O_5 TOTAL	P_2O_5 W.S.	P_2O_5 C.I.	P_2O_5 F.A.	P_2O_5 F.A.	P_2O_5 AVAIL.	CaO	Acid Supplied % Theoretical		Rock Reacting	Acid Reacting
	Days	%	%	%	%	% Undried	% Dried	%	%	(See Table 4.)		%	%
2-A	3	8.0	46.15	44.6	0.36			45.8	16.85	104.5	111	97.0	
5-A	14	9.3		45.7	0.15	5.77		46.0				98.7	
8-A	30	7.3	47.35	44.8	0.28	4.98	3.30	47.0	17.07	105.8	112	97.7	90.62
2-B	3	7.7	45.5	40.7	1.80			43.7	19.9	81.1	86.5	87.3	
5-B	14	8.3		41.9	1.07	4.49		44.4				92.4	
8-B	30	6.4	47.0	42.1	1.31	3.62	1.41	45.7	19.61	86.6	91.2	90.6	95.75
2-C	3	7.7	42.0	38.5	3.74			39.3	21.06	65.8	70.1	74.9	
5-C	14	8.0		38.75	2.40	4.00		39.6				83.9	
8-C	30	5.7	46.1	38.26	2.83	3.14	1.26	43.3	21.30	74.6	79.5	81.2	95.90
2-D	3	7.1	41.2	33.25	5.28			35.9	22.87	56.1	59.8	67.2	
5-D	14	6.0			4.68	3.03		36.5				71.0	
8-D	30	4.2	45.25	31.20	6.85	2.18	1.12	38.4	24.75	57.5	61.3	60.9	95.97
2-E	3	6.3	46.15	26.80	9.33			36.8	27.45	50.0	53.3	52.0	
5-E	14	6.1		26.82	8.00	2.62		38.1				58.8	
8-E	30	4.2	46.5	27.0	10.00	2.13	1.08	32.4	28.50	47.5	50.6	50.5	95.89
2-F	3	8.0	44.94	42.95	0.89			44.0	18.42	88.6	94.5	93.2	
5-F	14	8.4		43.5	0.55	4.33		44.4				95.8	
8-F	30	5.6	47.9	44.45	0.58	3.18	1.70	47.3	18.57	95.9	102.1	95.6	95.10

(11) Theoretical acid proportion for conversion to $Ca(H_2PO_4)_2$, $FePO_4$, and $AlPO_4$.
(12) Per cent theoretical acid added for conversion of calcium content to $Ca(H_2PO_4)_2$.

TABLE VII

DATA SHEET RUN 5. SERIES 3. USING 70% H_3PO_4

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
SAMPLE NUMBER	ELAPSED TIME	MOIST.	P_2O_5 TOTAL	P_2O_5 W.S.	P_2O_5 C.I.	P_2O_5 F.A. UNDRIED	P_2O_5 F.A. DRIED	P_2O_5 AVAIL.	CaO	ACID SUPPLIED % THEORETICAL		ROCK REACTING	ACID REACTING
	DAYS	%	%	%	%	%	%	%	%			%	%
3-A	3	8.2	46.75	44.25	0.54			46.2	17.34	102.1	108.5	95.6	
6-A	14	11.2		42.3	0.71	6.35		46.0				94.2	
9-A	30	6.8	46.0	42.2	1.00	5.27	1.92	45.0	17.58	97.9	104.2	92.0	94.29
3-B	3	9.7	45.35	42.55	0.85			44.5	17.35	97.8	104.1	93.1	
6-B	14	11.4		41.7	0.79	6.30		44.5				93.6	
9-B	30	5.6	45.9	41.4	1.30	5.21	1.52	44.6	17.92	95.9	102	89.8	95.43
3-C	3	9.1	45.23	40.55	1.70			43.5	20.68	76.0	81	88.4	
6-C	14	11.8		39.4	1.59	6.74		43.6				89.1	
9-C	30	6.1	44.32	38.9	2.95	5.41	1.30	41.4	20.0	77.4	82.4	79.2	95.68
3-D	3	8.7	44.2	36.4	4.36			39.8	21.07	71.1	75.8	70.7	
6-D	14	10.9		35.2	3.71	5.79		37.2				75.1	
9-D	30	4.4	43.25	36.6	4.92	3.79	1.05	37.0	21.79	65.7	70.0	68.0	96.23
3-E	3	8.1	43.3	32.8	6.09			36.2	24.11	55.8	59.5	64.4	
6-E	14	10.1		31.3	5.95	4.88		37.3				65.2	
9-E	30	5.0	43.78	32.0	7.58	3.14	1.19	37.5	24.36	56.1	59.8	56.0	95.52
3-F	3	8.4	42.6	34.72	5.34			37.5	22.8	59.7	63.5	66.9	
6-F	14	10.7		33.5	5.13	5.60		37.5				68.2	
9-F	30	6.8	43.7	33.8	6.19	4.26	1.48	37.5	22.7	62.6	66.7	61.6	94.65
9-D'	30	9.1	43.4	33.6	6.02	4.51	1.45	37.4	22.28	63.6	67.8	61.8	94.75
9-E'	30	9.1	42.2	30.4	8.28	4.30	1.45	33.9	23.80	54.8	58.3	50.8	94.89
9-F'	30	8.7	43.8	34.35	6.00	4.19	1.19	37.8	22.25	64.7	68.9	62.0	95.67

(11) Theoretical acid proportion for conversion to $Ca(H_2PO_4)_2$, $FePO_4$, and $AlPO_4$.
(12) Per cent theoretical acid added for conversion of calcium content to $Ca(H_2PO_4)_2$.

TABLE VIII
SETTING TIMES AND TEMPERATURES

RUN 5.

SAMPLE NUMBER (#)	SETTING TIME SECONDS		TEMPERATURES DEGREES CENTIGRADE	
	40 g. NEEDLE	86 g. NEEDLE	SETTING	MAXIMUM
1-A'	36	58	66°	100°
1-B'	45	65	67°	99°
1-C'	31	43	65°	98°
1-D'	10	12	60°	95.5°
1-E'	6	10	--	--
1-F'	--	--	--	--
2-A	75	125	71°	89°
2-B	58	95	65°	88°
2-C	50	70	70°	87°
2-D	30	50	65°	85°
2-E	21	30	63°	85°
2-F	8	13	61°	82°
3-A	160	300	65°	78°
3-B	118	235	61°	77°
3-C	196	228	59°	77°
3-D	60	118	57°	76°
3-E	43	65	55°	76°
3-F	48	70	52°	71°

TABLE IX

RUN 4.

DATA AND CALCULATIONS * (USING LOW ACID CONCENTRATIONS AND ACID*ROCK RATIOS)

SAMPLE NUMBER	ACID CONC. %	ACID TEMP. °C.	TREATMENT OF PRODUCT	P_2O_5 TOTAL %	P_2O_5 C.I. %	P_2O_5 W.S. %	CaO %	$CaHPO_4$ #	$Ca(H_2PO_4)_2$ ##	% CONVERSION
1a*	40	27	DRIED IMMEDIATELY 125 - 130° C.	43.6	15.6	16.9	32.1	115.1	32.6	32.2
2a*	40	100	"	43.6	16.0	16.6	32.4	111.1	32.0	31.2
2b*	40	100	"	43.7	15.7	.6.1	32.4	111.5	32.1	32.5
3a*	60	27	"	43.4	15.3	17.4	32.6	108.5	31.2	34.7
4b*	60	116	"	44.7	12.3	24.3	29,7	138.0	40.0	42.2
5a**	60	27	AIR DRIED 2 WKS. DRIED OVEN 75° C.	46.2	15.7		33.4	117.6	34.0	34.3
6a**	60	116	"	44.7	15.0		31.3	125.6	36.3	33,2
6b**	60	116	"	46.3	14.3		31.6	132.4	38.2	37.3

*Analyses made 3 days after mixing.

**Analyses made 14 days after mixing.

Per cent theoretical acid added for conversion to $CaHPO_4$.

Per cent theoretical acid added for conversion to $Ca(H_2PO_4)$, $FePO_4$, and $AlPO_4$.

TABLE X

P_2O_5 DISTRIBUTION (DRY BASIS)

SAMPLE NUMBER	ACID ADDED	P_2O_5 AVAIL.	P_2O_5 W.S.	P_2O_5 F.A.	P_2O_5 TOTAL	CaO	P_2O_5 SUPPLIED #/# SUPERPHOSPHATE	
#	%	%	%	%	%	%	ROCK	ACID
7-A	105.6	51.1	49.4	2.73	51.1	18.55	0.131	0.380
7-B	84.6	48.8	46.4	1.58	49.8	21.20	0.150	0.348
7-C	68.6	45.3	41.1	1.42	48.3	23.60	0.167	0.316
7-D	64.0	44.5	36.6	1.25	49.2	25.20	0,178	0.314
7-A'	99.8	51.0	48.9		51.1	19.3	0,1365	0.375
7-B'	90.8	49.8	46.8	1.60	50.5	20.4	0,1441	0.361
7-C'	73.3	46.2	40.8	1.60	48.7	22.82	0,1641	0.326
7-D'	57.4	41.6	35.8	1.27	46.8	25.6	0.181	0.287
7-E'	55.0	40.5	35.0	1.35	46.5	26.2	0.185	0.280
8-A	105.8	50.7	48.4	3.67	51.0	18.41	0.1304	0.380
8-B	86.6	48.8	45.0	1.62	50.2	20.96	0.1482	0.354
8-C	74.6	46.0	40.6	1.45	48.9	22.60	0.160	0.3295
8-D	57.5	40.1	32.5	1.30	47.2	25.8	0.1825	0.290
8-E	47.5	33.8	28.2	1.24	48.5	29.55	0.2090	0.276
8-F	95.9	50.2	47.1	1.92	50.8	19.7	0.1394	0.369
9-A	97.9	48.3	45.3	2.06	49.4	18.88	0.1335	0.361
9-B	95.9	47.3	43.9	1.61	48.7	19.01	0.1347	0.352
9-C	77.4	44.1	41.4	1.37	47.3	21.30	0.1508	0.322
9-D	65.7	41.6	38.7	1.11	45.7	23.0	0.1627	0.2943
9-E	56.1	38.1	33.7	1.60	46.1	25.63	0.1815	0.280
9-F	62.6	40.3	36.3	1.59	46.9	24,4	0.1726	0.296

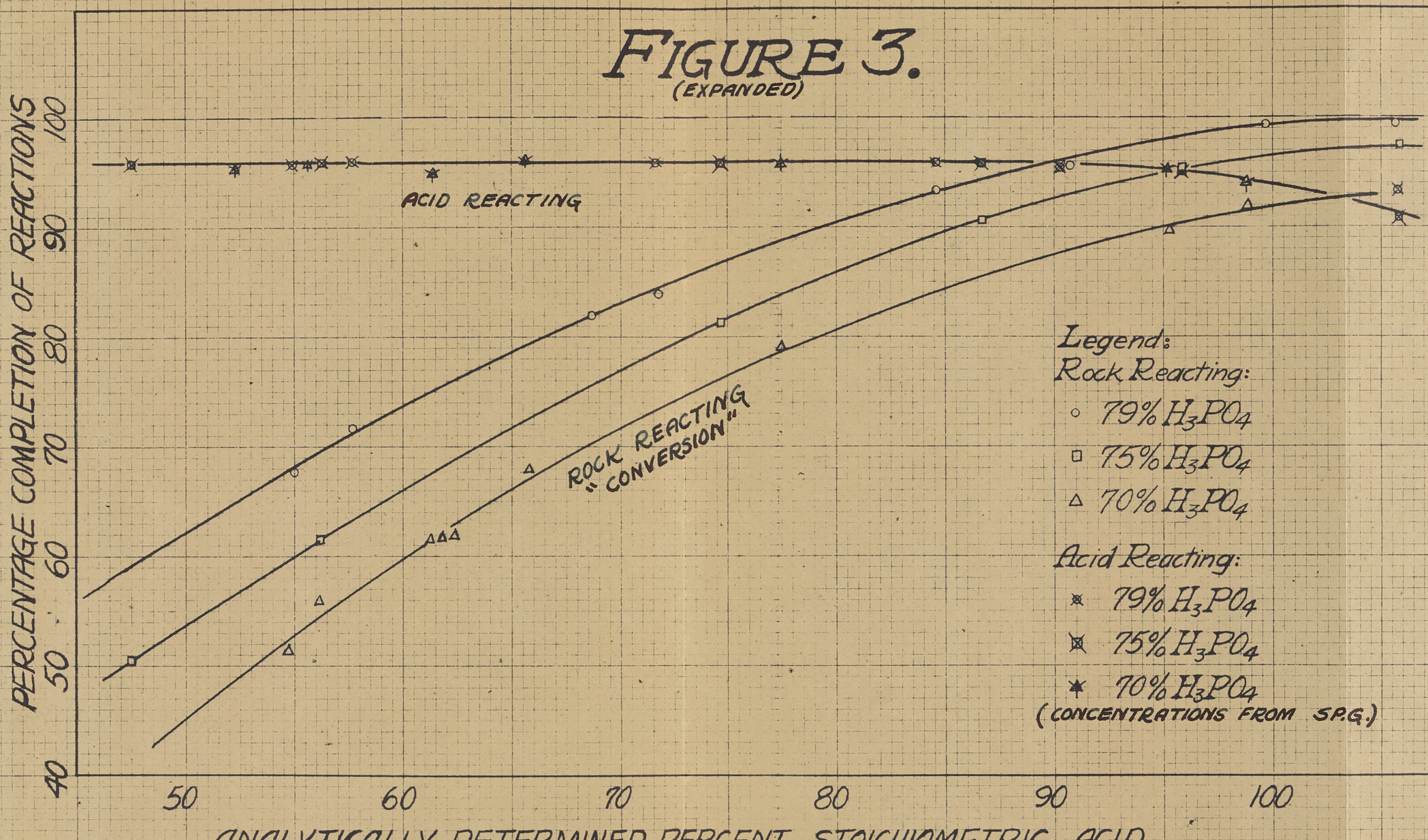
TABLE XI

ESTIMATED COSTS PER TON OF AVAILABLE P_2O_5

SAMPLE NUMBER	SUPER-PHOSPHATE P_2O_5 AVAIL.	ROCK	ACID	TOTAL	FREIGHT ETC.	
					100 Mi.	200 Mi.
#	TONS/TON	$	$	$	$	$
7-A	2.04	4.36	59.16	63.52	5.61	10.20
7-B	2.13	5.22	57.00	62.22	5.85	10.65
7-C	2.29	6.26	55.80	62.06	6.30	11.45
7-D	2.33	6.80	56.40	63.20	6.40	11.65
7-A'	2.02	4.55	58.80	63.35	5.55	10.10
7-B'	2.09	5.91	58.00	62.91	5.75	10.45
7-C'	2.27	5.96	56.60	62.56	6.25	11.35
7-D'	2.49	7.40	55.30	62.70	6.85	12.45
7-E'	2.56	7.77	55.30	63.07	7.06	12.82
8-A	2.13	4.32	60.00	64.32	5.85	10.65
8-B	2.18	5.20	58.00	63.20	6.02	10.92
8-C	2.31	5.90	57.30	63.20	6.35	11.55
8-D	2.60	7.70	57.90	65.60	7.15	13.00
8-E	3.09	10.12	65.20	75.32	8.50	15.45
8-F	2.11	4.72	58.80	63.60	5.80	10.55
9-A	2.22	4.70	59.80	64.40	6.10	11.10
9-B	2.24	4.84	59.40	64.24	6.16	11.20
9-C	2.42	5.80	58.40	64.20	6.65	12.10
9-D	2.54	6.64	56.00	64.24	7.01	12.72
9-E	2.76	8.10	58.80	66.90	7.60	13.80
9-F	2.67	7.27	58.60	65.87	7.34	13.34

FIGURE 3

FIGURE 3.
(EXPANDED)



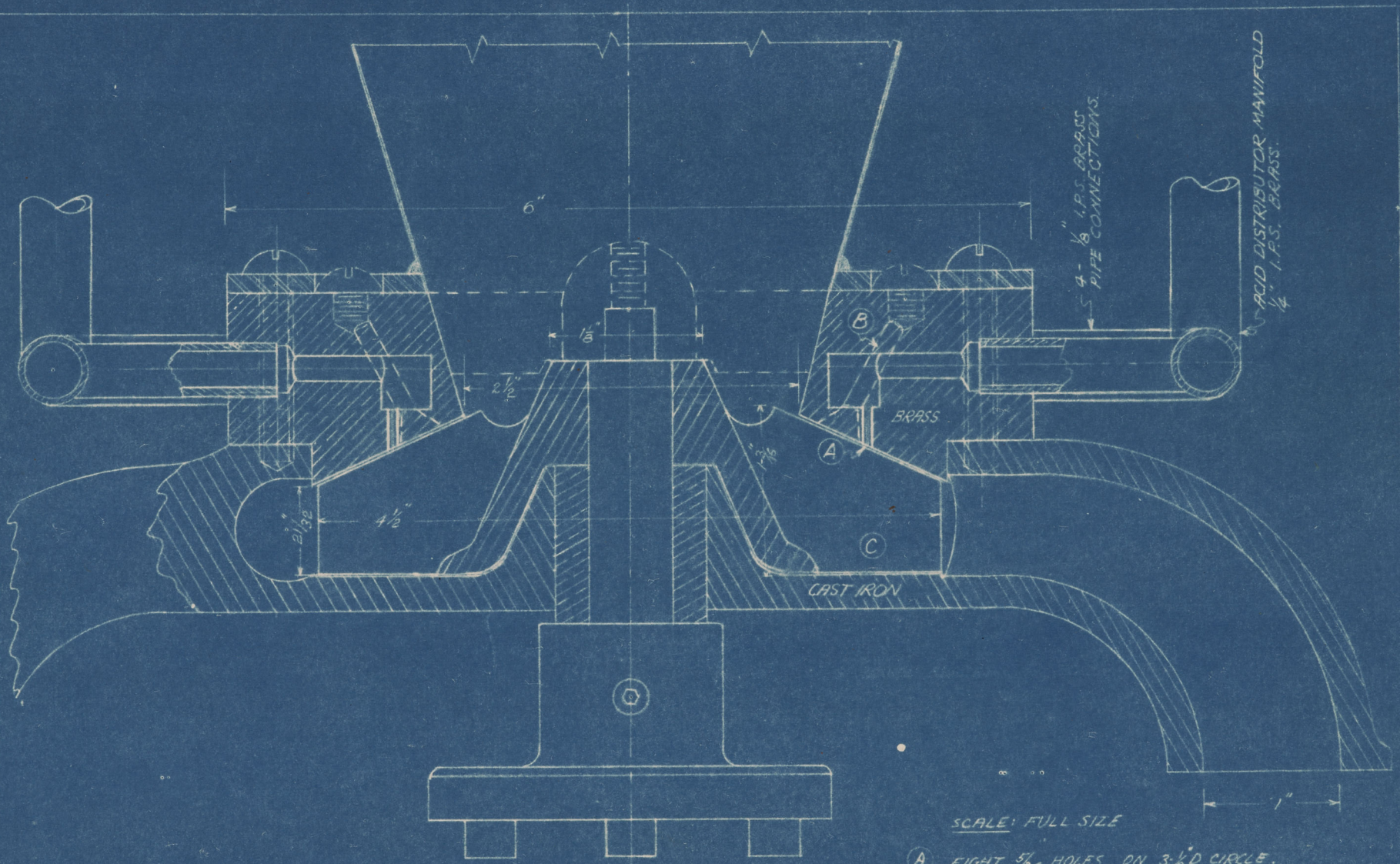
DEGREE OF COMPLETION OF REACTIONS
AFTER 30 DAYS

J.E.G. 8/20/38

TABLE XII

POWER REQUIRED RUN 3.

TEST NO.	TOTAL FEED RATE	ACID PROPORTION	POWER CONSUMPTION		PRODUCT*POWER RATIO	
			RANGE	AVERAGE		
	TONS PER HOUR	THEORETICAL %	KILOWATTS	KILOWATTS	KW. HR. PER TON	TONS PER. HP. HR.
1	1.15	99	0.48-0.58	0.5	0.435	1.71
2	1.12	95	0.48-0.54	0.5	0.447	1.67
3	1.08	91	0.52-0.64	0.58	0.536	1.39
4	1.01	81	0.50-0.60	0.57	0.566	1.32
5	0.50	95	0.44-0.68	0.52	1.038	0.72
6	0.87	97	0.58-0.82	0.63	0.727	1.03
7	1.30	97	0.68-0.70	0.75	0.593	1.26
8	1.58	99	0.68-0.85	0.69	0.436	1.71
CONTINUOUS PERIOD	0.48	90	0.36-	0.58	1.193	0.62
	0		Constant	0.26		0



SCALE: FULL SIZE

(A) EIGHT 5/64 HOLES ON 3-1/2" D CIRCLE.

(B) OLD ACID INLET - FOUR 1/8" D. HOLES

(C) SIX RADIAL BLADES.

SECTIONAL VIEW OF MIXER

PLATE NO. 1

BY J.E.G. - K-1163

APPROVAL

The foregoing thesis is hereby approved as a creditable study of an engineering subject, carried out and presented in a manner sufficiently satisfactory to warrant its acceptance as a prerequisite to the degree for which it has been submitted. It is to be understood that by this approval the undersigned does not necessarily endorse or approve any statement made, opinions expressed, or conclusions drawn therein, but approves the thesis only for the purpose for which it is submitted.

Name R. W. Boarts

Title Prof. Chem. Engr.